IN SITU ELLIPSOMETRY OF SURFACES IN AN ULTRAHIGH VACUUM THIN FILM DEPOSITION CHAMBER

by

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Submitted to Brigham Young University in partial fulfillment

of the graduation requirements for University Honors

Department of Physics and Astronomy

June 2000

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Acknowledgments

This thesis has been possible through the support and guidance of Dr. David Allred, of the Department of Physics and Astronomy, Brigham Young University. I am also grateful for those who have helped in the building of the vacuum chamber and the measurements of films- Aaron Fox, J.J. Warriner, Greg Harris, Zach Barton, Bryan Schramm, Joseph Young, Wesley Lifferth, Scott Daniel, Nan Ellen Ah You, Matthew Squires, Cort Johnson, and Michael Newey. Training Luke Bissell, Richard Sandberg, and Tyler Smith on this UHV system while gaining feedback and comments have tremendously helped this thesis to be polished in its written form. The support and kindness of my parents, Soo-Young Choi and Ock-ja Choi, family members, Dr. Justin Peatross, Matthew Tollestrup and many others have also been valuable in completing this work.

Generous financial support from the Department of Physics and Astronomy, Honors Department, ORCA Scholarship, Multi-cultural Student Services Scholarship of Brigham Young University, and SPIE Scholarship have been significant and much appreciated.

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Abstract

As current microtechnology advances toward building nanosize components for computers and aerospace devices, oxidation of thin films can have dramatic effects on the electronic and optical properties of these devices. In order to understand optical properties of thin film materials and their oxides in a high purity environment, we have been building an ultrahigh vacuum deposition chamber to achieve a base pressure under 10^{-9} torr. We have incorporated evaporation and sputtering capabilities for thin film deposition in vacuum, and an in situ ellipsometer to analyze the optical properties and oxidation of the films without exposure to atmosphere. This combined with an oxygen leak allows for control over the amount of oxidation on the films. Ruthenium was initially investigated with the chamber in high vacuum to understand initial oxidation of thin films, and the applicability of our chamber.

Chapter 1

Introduction

I have been working with a group of five BYU undergraduates, under the direction of Dr. David Allred, my research advisor, in designing a vacuum system for thin film deposition and characterization. In April 2000, we began building the vacuum system from a stainless steel vacuum chamber body made by Varian, Inc. The chamber is designed to achieve UHV (ultrahigh vacuum) with a base pressure $< 10^{-9}$ torr. The system is designed to create thin films by two deposition methods- sputtering and thermal evaporation. Through an oxygen leak valve and an in situ ellipsometer, measurements of initial oxidation of thin films can be made. The chamber will allow BYU's XUV (extreme ultraviolet) Research Group in the Department of Physics and Astronomy to analyze oxidation rates, measure optical constants, and model the oxidation of thin films. Currently the UHV system is placed on the second floor of the Physics Underground Laboratory of Brigham Young University. (See Figure 1)

1.1 Definition of Terms

In order to understand the research, some basic definitions are given. Additional terms used in later chapters will be included under the proper chapter heading.

Chamber Port The opening or arm extending out from the main chamber body. Devices used inside the vacuum chamber are usually mounted on the ports.

Deposition Act of depositing material onto a surface, hence creating a thin film layer.

Ellipsometry Technique of measuring optical properties of film by reflecting polarized light off of the film. Measurement of reflection, transmission, optical constants, and thickness of layers can be determined.

Evaporation Deposition of material onto film substrate by using boats (usually metal plates with high resistance) to thermally heat and 'evaporate' the material.

Flange Metal plate that provides sealing of any open ports of the chamber.

HV (High Vacuum) Base pressure less than 1×10^{-3} torr, and greater than 10^{-9} torr.
(Atmospheric pressure is $7.6 \times 10^{+2}$ torr.)

In situ Measurement inside the chamber while under vacuum.

Optical Constants Constants that describe the behavior of light inside a given material. It is a complex number that relates the speed of light in vacuum ($c = 3.0 \times 10^8 \frac{m}{sec}$) to the speed of light in the material, and specifies the rate of absorption.

Reflectance The amount of light that reflects off an optical film in relation to the amount of light initially emitted.

Sputtering Deposition of material onto film substrate by bombarding positive ions onto the material, and physically removing atoms from the material.

Substrate The underlying surface layer on which material will be deposited to create a thin film.

Transmission The amount of light that passes through an optical film in relation to the amount of light initially emitted.

UHV (Ultrahigh Vacuum) Base pressure less than 10^{-9} torr.

1.2 Thesis Description

My research goal is two fold. First, to build a UHV chamber with in situ ellipsometry, sputtering, and evaporation capabilities. Second, to study thin films by measuring and developing models for thin film oxidation.

A ruthenium thin film of Ru thickness $\approx 243\mathring{A}$ was created in high vacuum. (in the $1\times10^{-7}-1\times10^{-6}$ torr range) In situ ellipsometric measurements to analyze oxidation rates, and to improve optical constants for ruthenium thin films was made and analyzed.

1.3 Motivation

1.3.1 Oxidation of Thin Films

The motivation behind building a UHV deposition chamber with an in situ ellisometer is primarily to understand oxidation. From a computer model of a thin film of silicon oxide on a silicon film, (See Figure 2), it can be seen that reflectance of a silicon film can vary from 70% to below 5% depending on the amount of initial oxidation. In the micro-electronic industry where nano-scale effects of electro-optical films are important, such difference in reflectance alone can have dramatic effects on devices.

Films begin to oxidize immediately as soon as they are taken out of the vacuum chamber in which they are created. Traditionally at BYU, these films were taken out of the chamber and measured by an ellipsometer in atmospheric air. The very initial oxidation of films could not be measured or studied because the film had already oxidized some before any measurements could be made. In the new chamber we will be able to measure, with in situ ellipsometry, the oxidation and optical properties of thin films as they are created and from the time they begin to oxidize, without removing the film.

1.3.2 High Purity Thin Films

A UHV chamber will allow 100 - 10,000 times better vacuum than all but a few vacuum chambers at BYU. Most of the vacuum chambers at BYU are currently HV (high vacuum) chambers, which have a base pressure of about 1×10^{-6} torr.

The base pressure of a vacuum system is proportional to the number of gas molecules in the system. Because of the presence of these gas molecules,

deposition onto a film occurs naturally even in vacuum. For example, a film can still oxidize in vacuum over a period of time. At HV vacuum, one layer of gaseous atoms impact onto the surface of the film every second. In UHV, for one layer of gas to deposit onto the film, it will take longer than 1000 seconds. This also reduces the amount of oxygen molecules binding onto the film surface, hence reducing the oxidation rate inside the chamber tremendously. Thus, through UHV vacuum, high purity films with little oxidation may be created.

1.4 Building History

In building our UHV chamber, I have worked with a total of five BYU undergraduates who were part of Dr. Allred's 'Building Group.' These five are Aaron Fox, J.J. Warriner, Greg Harris, Zach Barton, and Bryan Schramm. Detailed description and definition of most of the components we built are provided in later chapters of the thesis.

During the summer of 2000, from April to August, Aaron Fox, a mechanical engineering major, and I began the project. He and I completed most of the overall design and mechanisms for the vacuum system. For the vacuum pumping mechanism, we designed it using two sorption pumps in parallel, along with the cryopump, and the positioning of the gate valve and roughing line valves. For the deposition system, we required the sputter gun to be placed at the bottom of the chamber, and contemplated using a bellow with a linear motion guide for a 3 boat evaporator. The ports for argon and oxygen gas lines, the crystal monitor, thermocouple gauges, and the in situ ellipsometer were also determined for the initial stage. We sent in the cryopump to be fixed to full capacity, and ordered two sorption pumps and dewers, con-flat flanges, electrical feedthrough for the evaporator, and the sputter gun from US Inc.

From September 2000, for all of Fall semester and most of Winter semester, J.J. Warriner and Greg Harris helped me in finalizing our design, improving our components, and the actual building and assembling of necessary components. Several changes were made for the final design of the vacuum system. I oversaw the design and mechanism of each component, cleaning of parts, and the precautions necessary to obtain a UHV vacuum system. J.J. and Greg built many of the parts and accomplished much of the connections and practical workings to allow the system to function.

J.J. built the sample stage and with Greg he designed the shutter system to protect the film substrate. J.J. also set up the argon and oxygen gas lines with appropriate gas valves, and built the voltage reader for the argon mass flow controller. He installed the air lines and the connections for the water cooling lines, as well as the operation of the pneumatic gate valve with a solenoid and an electrical switch.

The evaporator was designed to be at fixed position without any linear motion, and with an optional shield to protect the sputter gun that it would sit above. Greg built the main structure of the 3-boat evaporator with the copper lines. He also provided connections for and obtained the power supplies for both the sputter gun and the evaporator with the help of Scott Daniel in the department. Greg designed and built the heating system for the chamber bake-out and provided the steel support for the gate-valve and the cryopump.

All necessary parts were ordered, including the crystal monitor, thermocouple gauge, copper gaskets and o-rings, sieve materials for the sorption pumps, and additional flanges. All necessary parts inside the chamber were inspected, cleaned, reassembled, and installed by myself to ensure UHV compatibility.

Beginning in January 2001, Zach and Bryan joined our group. Zach finished the shields for the evaporator and provided connections for the water cooling system. Bryan completed the building of the shutter and the bake-out system for the cryopump.

Even after more than a year of designing and building by our group, there remains improvements to our system, as well as additional vacuum components to be installed. However, we have been able to construct a working vacuum system with all the basic and necessary components for thin film deposition and in situ ellipsometric analysis.

Chapter 2

Ultrahigh Vacuum

2.1 Additional Chapter Definitions

Bake out Method of eliminating gas molecules on surfaces by thermally heating, or 'baking out' the surface to high temperatures. Increased thermal energy provides gas molecules energy to leave the surface.

Conflat (CF) Vacuum sealing method that uses a gasket in between two knife-edge surfaces to provide the seal. The gasket used is usually copper shaped as a washer. (Refer to Figure 4)

Gasket Any material, usually in the shape of a ring or a washer, that is pressed against to provide sealing.

High Vacuum Pump Pump used to obtain high vacuum pressure or below ($< 10^{-3}$ torr).

Leak Detection Testing to detect leaks in a vacuum system.

Outgassing The action of gas molecules leaving the walls and components inside the vacuum chamber, when under vacuum.

Partial Pressure The pressure of a particular type of gas, excluding the pressure of all other types of gases in the system.

Roughing Pressure ranging from atmospheric pressure down to 1×10^{-3} torr. This is the pressure range that must be achieved by a roughing pump before any high vacuum pumps usually can be operated.

Roughing Pump Pump used to obtain roughing pressures (atmospheric pressure down to 10^{-3} torr).

Seize The fusing with other parts as a result of high pressure or high temperature.

Virtual Leak Leak from a trapped pocket of air or contaminants, inside of a vacuum system. The leak is not from a source outside the vacuum system, hence it is called a 'virtual leak.'

2.2 Background

Ultrahigh vacuum has been defined as base pressures below 10^{-9} torr. Prior to 1950, there were no known methods of measuring such pressure, although there are indications of some UHV achievements by experimenters. In 1950 Bayard and Alpert were able to produce an ionization gauge that could measure UHV pressures down to 1×10^{-10} torr relatively easy. With simple means of pressure measurement, UHV technology and equipment developed and became readily available by the 1960's.[1]

There are about 3×10^{19} molecules/cm^3 at atmospheric pressure (760 torr). In UHV this would be reduced by ~ 12 orders of magnitude or to $\sim 3 \times 10^7$ molecules/cm^3. Comparisons with the vacuum in space are helpful. The pressure at the midpoint between the earth and the moon is $\sim 1 \times 10^{-14}$ torr, and outside of the solar system, into interstellar space, the pressure drops to $\sim 1 \times 10^{-16}$ torr (~ 3 molecules/cm^3). Each order of magnitude decrease in pressure corresponds to an order of magnitude decrease in molecules in the volume. Although there are researchers with vacuum systems achieving pressures in the 1×10^{-13} torr range, for most industrial and experimental applications the purity and reliability of measurements in 1×10^{-10} torr range seem sufficient.[2]

Various interactions are involved within a chamber under vacuum, including the chamber walls themselves. Some of these include molecule-molecule, molecule-surface interactions, charged particle interactions with surfaces, scattering by projectiles with various energies, and in some cases radiation. Theoretical modelling and experiments have been conducted to understand these interactions in detail using thermodynamics, classical mechanical behaviors, and analysis of their respective energy and potentials.

Detailed theoretical treatment can be found in *The Physical Basis of Ultrahigh Vacuum*.[1]

This chapter is an overview of some of the known theories, facts, and design practices of ultrahigh vacuum systems.

2.3 Relevant Theory

2.3.1 Ideal Gas Law and Dalton's Law

In a vacuum chamber, there are many atoms and molecules in the gas phase interacting with each other. Most of these gases are those from atmospheric air, consisting of N_2, O_2, H_2, water vapor, and others. The behavior of gas molecules in a vacuum can be understood through the ideal gas law. The relationship between pressure, p, volume, V, number of molecules, N, and temperature, T, can be written as:

$$pV = Nk_BT, or \tag{2.1}$$

$$p = nk_BT \tag{2.2}$$

,where $n = \frac{N}{V}$ is the number of molecules per volume of gas, the molecular number density, and $k_B = 1.38 \times 10^{-23} \frac{J}{K}$ is Boltzmann's constant. Since in most vacuum systems, the volume of the chamber or components are fixed, Equation 2.2 will be preferred in this thesis.

Another important equation is Dalton's Law which states that the total pressure of a mixture of gases, is the sum of the partial pressure of each gas:

$$p_T = \sum_i p_i \tag{2.3}$$

The summation index i is for each species of gas.

According to these two equations, the pressure inside a chamber due to a mixture of gases is proportional to the number of molecules in the volume considered, and to the temperature. Thus, there are at least two methods to reduce the pressure inside a vacuum chamber:

1. Reduce the number of gas molecules inside the chamber, by removing the gas from the chamber.

2. Reduce the temperature.

There is a reason why 'reducing the pressure' was considered, over 'reducing the number of molecules.' Although the ultimate goal in a vacuum system is to reduce, if not eliminate, the number of gas molecules, this is not the practical solution. Materials in vacuum chambers outgas at certain rates that decrease as they are pumped down over time. In order to compensate for these outgas rates and to vacuum the chamber down from higher base pressure, the vacuum pumps need to pump faster. Thus, high pumping speed is essential to achieve low vacuum. 'Pressure' can also be thought as the kinetic energy or the speed of the gas molecules inside the chamber. Thus, by reducing the 'pressure', the pumping speed of the vacuum pump is increased relatively, and hence the vacuum is improved.

2.3.2 Steady State Conditions

Achieving low vacuum is possible by reducing the pressure of the system. The partial pressure of an ideal gas in a vacuum system at steady state is given by:[2]

$$p_i = \frac{\sum Q_i}{\sum S_i} \tag{2.4}$$

where

$p_i =$ Equilibrium pressure in system of species i.

$Q_i =$ Outgassing rate from all sources in system of species i. This is dependent on the material type and proportional to the surface area of each outgassing source.

$S_i =$ Pumping speed from all sources for species i. It is the volume of gas that flows past a reference point per unit time.

In essence, to minimize p_i, the pumping speed for the particular gas should be maximized and the outgassing rate by the gas must be minimized. Pumping speed can be maximized by acquiring effective vacuum pumps that can be very expensive. In most cases, this is limited due to funding and availability, hence vacuum systems are usually designed to work with available vacuum pumps. So most designers of vacuum systems attempt to minimize the outgassing rates of materials and components inside the chamber, because over this is what they have most control.

2.3.3 Dynamic Conditions

The dynamic conditions require analysis of vacuum phenomena in relation to time. This would require analysis of vacuum pressure and molecular interactions in relation to the energy and momentum inherent in the system and of the gas molecules. This section will only analyze some useful rules and surface interactions under vacuum.

Useful Rules

Ronald Outlaw, in *UHV Design and Practices*[2], provides some rules of thumb that are readily derived and used for understanding the basic dynamics of vacuum systems.

Gas Number Density The number of ideal gas molecules in unit volume at a given pressure. It is directly proportional to pressure.

$$\boxed{\eta_p \sim 3 \times 10^{16} \text{ molecules/cm}^3\text{/torr}} \tag{2.5}$$

Surface Density The average number of atoms on a solid ordered surface in unit surface area. This is approximately how many atoms are on a thin film surface.

$$\boxed{\rho_s \sim 1 \times 10^{15} \text{ atoms/cm}^2} \tag{2.6}$$

Bulk Density The average density of atoms in an ordered solid bulk.

$$\boxed{\rho \sim 2 \times 10^{22} \text{ atoms/cm}^3} \tag{2.7}$$

Incident Flux The number of incident gas molecules on a unit cross-sectional area at 20°C. This is directly proportional to the pressure.

$$\boxed{I_a \sim 1 \times 10^{15} \text{ molecules/sec/cm}^2 \text{ at } 1 \times 10^{-6} \text{ torr}} \tag{2.8}$$

or using the surface density rule (2.6),

$$\boxed{I \sim 1 \text{ monolayer/sec at } 1 \times 10^{-6} \text{ torr}} \tag{2.9}$$

Rule 2.9 is very useful for understanding dynamic vacuum conditions. For thin film depositions, the flux I represents the number of gas molecules that can contaminate or oxidize the outer layer of film per unit time. Since I is directly proportional to pressure, lower pressure implies lower I, hence less chance for contamination or oxidation. With this rule it is easy to see that for 1×10^{-9} torr, which is the high end of UHV pressure, $I \sim \frac{1}{1000}$ monolayer/sec. Thus at UHV pressure, on average it takes many minutes or hours for one layer of gas to form on a surface of thin film, compared to the seconds it takes to form in HV.

Surface Interactions

There are various molecule-surface interactions involved in vacuum systems that affect the outgassing rate of gas molecules. In order to minimize outgas rates it is helpful to understand the mechanisms behind molecule-surface interactions. A short list of some of the main interactions are described below.[2]

Adsorption The bonding of atoms and molecules to a surface. There are two types- physisorption and chemisorption. In most cases the surface is affected by both physisorption and chemisorption.

- **Physisorption** Physical bonding without exchange of electrons. Characterized by van der Waal's forces. In general the bonding force is weak enough to be overcome by increasing the temperature of the surface.
  Example) Dipole-dipole interactions such as water molecules adsorbed into stainless steel walls.

- **Chemisorption** Chemical bonding with a reactive surface through electron transfer (ionic bonding) or electron sharing covalent bonding. The resulting bonding force is strong enough to result in a long residence time, the time that the molecules stay bonded to the surface.
  Example) Metal oxidation of surface walls.

Desorption The emission of surface adsorbed molecules or atoms back into the gas phase of the vacuum space. These gases may be emitted intact or may form a new molecule at the surface and then be emitted. This process is usually considered as outgassing.

Desorption can be separated into two conditions- baked and unbaked. Baking a system at high temperatures removes much of the physically adsorbed layer. So in a baked system most of the desorption comes from the chemisorbed molecules that were not removed during bake out. On the other hand, in an unbaked system the desorption comes mostly from the physically adsorbed layer which lies on top of a chemisorbed layer.

Absorption The subsequent dissolution of gases into the bulk surface after adsorption takes place. The dissolution depends on the solubility of the gas.

In general hydrogen is very conducive to absorption because its small size allows hydrogen to fit well in the lattice of all metals and move easily from site to site in the bulk. Hydrogen however does not diffuse easily through glass. OH molecules on the other hand fit more readily in the open structure of amorphous solid like glass. Helium is also well known to dissolve into the lattice and move through a concentration gradient in glass systems. He transport mechanism is primarily due to no chemical bonding at the relatively large and open sites.

Permeation The movement of gases from high density side of a solid barrier, through the solid, and to the low density side of the solid barrier. Usually there is very small permeation of gases from atmosphere (high density) to the inside (low density) of the vacuum chamber. Permeation does play a significant role when a pressure of 1×10^{-12} torr is achieved or when o-rings are used. This is due to the large difference in pressure from one side of the barrier to the other side, or because of the relatively porous polymers that make up o-rings.

Ultimate Versus Operational Pressure

The ultimate pressure is the steady state pressure achieved after a vacuum system is pumped down. This pressure is usually the lowest pressure achieved by the system and is often used when describing the pressure of a given vacuum chamber. When vacuum pressure of our UHV chamber is described in this thesis, ultimate pressure is implied.

When a system is actually in operation, and all the appropriate tools are operating, the actual pressure of the chamber usually increases. This

is the operational pressure of the vacuum system. For example when the ion gauge, evaporator, or the sputter gun is on, contaminants from these devices are emitted into the vacuum space, and hence the pressure rises. Also the location of the gauge that measures the chamber pressure may be located close to the pump, hence will not be able to measure the pressure of the actual process.[2] In order to understand the vacuum environment of a given process, it is important to realize these facts of operational pressure in comparison to the tolerance level of the process.

2.4 Achieving Ultrahigh Vacuum (UHV)

Achieving HV (high vacuum) is very routine in terms of conventional vacuum technology. However, to achieve UHV requires much care and planning. Some of the major issues involved with achieving UHV will be listed in this section. (Refer to Figure 3)

2.4.1 Cleanliness

No oil must be inside the chamber. Vacuum oil for o-rings must be minimally applied. A thumbprint can jeopardize achieving UHV altogether.

Cleaning agents must be used. Use reagent or purer type to avoid contamination from the cleaning agents themselves. Hexane or acetone is first used to eliminate oil, and then methanol is used to remove any debris that hexane or acetone may leave. Hexane is usually stronger at eliminating oil than acetone. When cleaning, be sure to wear nitrile gloves (or other special gloves that provide adequate protection), especially when using hexane or acetone. It is a good practice to wear a pair of disposable latex or vinyl gloves underneath the nitrile gloves, so as to provide additional precautions for cleanliness.

Parts that are cleaned should be placed on a new sheet of Kimwipes® from Kimberly-Clark®, or similar delicate wipes. Clean aluminum foil underneath the sheet of Kimwipes is also useful. If storing the clean parts, ensure that they are covered with Kimwipes and aluminum foil and stored away.

It is important to recognize that whatever someone has touched with his/her skin or clothes is also a source of oil or dirt. Touching such areas even with gloves can contaminate the gloves. In such cases, the gloves should

either be cleaned or replaced before using the gloves to touch parts that will go inside the vacuum chamber. It is always better to be sure and safe instead of compromising the system with questionably clean parts.

2.4.2 Bake out

Chamber walls, screws, any parts, and any material inside the chamber are sources of outgassing. They are similar to sponges that soak up air when exposed to atmosphere. In order to facilitate quick degassing of these components, baking out the chamber to temperatures above 150 ^{o}C (in our case, above 250 ^{o}C) is recommended. The temperature should be increased or decreased at no more than about 20-25°C per hour to prevent the straining of the components of the vacuum system. The bake out temperature of the system should be as high as the system can handle. This is because there are almost always gases that degas better at higher temperatures than lower temperatures. The bake-out is to be done continuously for three to four days under vacuum, with a roughing pump pumping out the gas. This allows for efficient degassing of water and hydrogen vapors.

Because of the high temperatures that a vacuum system may be baked out, the screws and nuts may seize to each other or to the screw holes in the flanges. To prevent the seizing, anti-seize lubricants should be used on the threads of the screws for flanges that are baked out. The lubricant used in our chamber is a Permatex anti-seize lubricant that protect metals from seizing, galling, and corrosion for temperatures in -65 - 1600 ^{o}F (-55 - 870 ^{o}C).

It is important to only use a thin film of anti-seize lubricant. Also gloves should be worn and care should be taken as the anti-seize lubricant can damage both the skin and clothes upon contact. Because the lubricant on the screws may be wiped on the inside of the vacuum chamber when installing a flange or taking the flange off, care should be taken to avoid this and clean the affected areas with acetone (acetone seems to work better than hexane for anti-seize lubricant) and methanol.

2.4.3 Sealing methods

It is important to minimize, if not eliminate, any o-rings or polymers. To seal the chamber from atmosphere, use of metal-to-metal seals is necessary, such as using conflat flanges.

2.4.4 Material

All materials inside the chamber should provide minimal outgassing, including at bake-out temperatures. Materials that have low outgas rates are necessary to achieve UHV.

Examples of materials that should be avoided in vacuum systems are cadmium and zinc. Examples of materials with low outgas rates are aluminum, and stainless steel. Any components or device used inside the UHV part of the vacuum system should be made exclusively of aluminum or stainless steel, or other materials with low outgas rates that are safe for vacuum usage. This includes all the metal components such as bolts, nuts, and washers.

A very useful method of determining whether a metal is stainless steel is to use a magnet. If the metal is magnetic, then the metal is not stainless steel, and if it is not magnetic there is a high probability that it is stainless steel.

2.4.5 Surface Area

All surface areas are sources of outgassing. Smaller total surface area inside a vacuum system allows for less outgassing. The surface area of all the components should be minimized, as much as it does not hinder the efficiency of the operation and construction of the component.

Electrochemical Polishing

Surface area can be minimized by creating clean surfaces. Polishing the surface is also very helpful. Electrochemical polishing, or commonly 'electropolishing,' is one of the most efficient methods to polish metal surfaces for vacuum systems. Electrochemical polishing employs an electric field to improve the selective removal of peaks and protuberances on surfaces. Generally, the metal to be polished is immersed in an electrolyte at some distance away from the cathode electrode. Electric field lines are concentrated at the peaks of the surface, and preferentially the metal from these peaks dissolve into solution. It is by far the best way to reduce the surface area, establish a uniform oxide layer, and provide the maximum passivation of the surface. The final oxide surface has minimum defects that minimize the diffusion paths from the bulk metal to the vacuum space, hence reducing outgassing.[2] The main disadvantage of electrochemical polishing is that it is very expensive to

implement, usually requiring the surface that needs polishing to be sent in to be professionally electropolished.

2.4.6 Machining

When machining components that will be used inside the chamber, much care to reduce the surface area and provide a vacuum-tight seal should be given. Machining of parts by inert gas welding or vacuum brazing is required.

When metals are pressed against each other, space between the two surfaces can cause a virtual leak. This is apparent when a screw is screwed inside a metal surface. Usually there is a small pocket of air between the end of the screw and the bottom of the screw hole inside the metal piece. This pocket of air is a source of 'virtual leak,' because it provides a source for air to enter into the vacuum system. To eliminate this virtual leak, holes that go all the way through the screws can be drilled. It is important to ensure that no virtual leaks are created by way of design or construction of the components inside the vacuum system.

2.4.7 Leak Detection

Perhaps the most challenging yet important method in achieving UHV is to properly conduct a leak detection test. If the ultimate pressure ($< 1 \times 10^{-9}$ torr for a UHV system) cannot be achieved, or when the rate of pressure rise is greater than the normal outgassing rate of the system (there are various methods of determining this and can be looked up in most vacuum system literatures), it is *probable* that there is a vacuum leak in the system.

Procedures

(Joseph Young, the vacuum expert in the Department of Physics and Astronomy, is very knowledgable and experienced in detecting vacuum leaks and should be consulted for more practical information, directions, and help.)

1. Leak detection must be done carefully so as not to contaminate the already UHV clean system. If any area has been contaminated through the test, then the area(s) should be noted and cleaned appropriately.

2. Think of the possible reasons for a leak in the system. Sealing methods, structural design, and contamination could be possible reasons. Determine which areas may be possible leaks.

3. It is helpful, if not imperative, to separate different sections of the vacuum system through closing appropriate valves. Then each section should be pumped on to maintain a fixed pressure. The pressure together with the rate of pressure rise should be measured. (With fixed valves, pressure measurement gauges, and pumps in the system, this procedure requires some creativity to be done properly.)

   (See Figure 5)

4. For roughing pressure leaks and even high vacuum leaks, methanol may be sprayed through the suspected leaks. If it is a leak, then usually the pressure remains stable at first, and then suddenly increases at a fast rate.

5. For high vacuum leaks, using helium (utilizing its small molecular size) instead of methanol is helpful.

In reality, there is no step-by-step procedure to detect all leaks. Experience and knowledge of the vacuum system and its design should be used to effectively detect leaks. Sometimes the reason that UHV vacuum pressure is not achieved may not be leaks. It may be the fact that better bake out procedures, better cleaning, better structural components are necessary to achieve UHV.

Once leaks have been detected, appropriate measures to fix the cause of the leaks should be taken. This may require replacing parts, cleaning, or improving sealing techniques.

Chapter 3

In Situ Ellipsometry

3.1 Additional Chapter Definitions

Ex situ Ellipsometry Ellipsometry conducted outside of the original environment in which the sample was created.

Incident Angle The angle at which the light beam or wave reflects from the sample surface. (See Figure 6)

Incident Wave The original wave that is emitted to a surface, before any reflection or transmission occurs.

Plane of Incidence The plane containing the incident and reflected beams and the plane that is perpendicular to the sample surface. (See Figure 6)

Propagation The movement or travelling of an electromagnetic wave in a certain direction.

Viewport Port used to view the inside of the chamber.

3.2 Principle of Ellipsometry

An ellipsometer emits polarized light from its input unit, or light source, and reflects the light at one of several angles off the surface of the film to be measured. The optical properties and thicknesses of the different layers in the film will change the polarization of the incident light that was emitted.

The changed light is reflected and then received by the output unit, or the detector and analyzer, of the ellipsometer, and in turn is measured by the ellipsometer. Using a computer program such as that developed by J.A. Woollam Co., Inc., the measured data for various samples and angles can be processed to generate optical constants, reflectance, transmission, oxidation, and thicknesses of the different materials composing the film. (See Figure 7)

3.2.1 Light- Electromagnetic Wave

Light is an electromagnetic wave that is described by Maxwell's equation for electromagnetic fields. In a non-conducting, non-dispersive medium, one solution of Maxwell's equation for the electric field is the electromagnetic plane wave:

$$\vec{E}(\vec{r},t) = \vec{E_0}exp(\frac{i2\pi\tilde{n}}{\lambda}\vec{q}\cdot\vec{r})exp(-i\omega t) \tag{3.1}$$

where $\vec{q}$ is a unit vector along the direction of propagation, $\tilde{n}$ is the complex index of refraction $n + ik$, λ is the wavelength of the light in vacuum, ω is the angular frequency of the wave, t is time, $\vec{r}$ is the position in space that is written as a vector, and $\vec{E_0}$ is a complex vector constant specifying the amplitude and polarization state of the wave.

A similar form as in Equation 3.1 can be obtained for the magnetic field also, with it being perpendicular to the electric field. Thus, light can be represented as a combination of an electric field and a magnetic field that oscillate perpendicularly to each other. Both fields are also perpendicular to the direction of propagation. (See Figure 8)

Because of the relationship between the E-field (electric field) and the B-field (magnetic field), only the E-field and the direction of propagation are necessary to completely describe a beam of light. [3]

If the imaginary part (k, or the 'extinction coefficient') of the complex index of refraction ($\tilde{n}$) is nonzero, then the amplitude (the real part of $\vec{E}$) will change. From Equation 3.1,

$$|\vec{E}| = E \propto exp(-\frac{2\pi kz}{\lambda}) \tag{3.2}$$

where z is the distance of propagation of the wave. (See Figure 8) Because of the negative sign in the exponential argument, the amplitude of the E-field

will decay as z increases. This implies that as the E-field travels through an absorbing medium, it will become weaker.

The distance, D_P, known as the penetration depth is the distance the wave travels before it decays 1/e from its original amplitude:

$$D_P = \frac{\lambda}{2\pi k} \tag{3.3}$$

The penetration depth is an important concept in ellipsometry, as many materials exhibit large extinction coefficients such that a light beam may only penetrate a few tens of nm (10^{-9}m) or less. No information can be gained from a sample unless the light beam penetrates it sufficiently and then is able to reflect back out of the sample. This is why typically it is impossible to measure the thickness of metal films greater than about 50 nm thick with an ellipsometer.[3]

3.2.2 Polarized Light

The electric field described in Section 3.2.1, on page 20, lies on a plane perpendicular to the direction of beam propagation. Convention has created two directions when describing the reflection and transmission of light relative to a surface. The p-direction is defined as lying on the plane of incidence. The s-direction (from *Senkrecht*, German for perpendicular) lies perpendicular to the p-direction such that the p-direction, s-direction, and the direction of propagation (in the same order) define a right-handed Cartesian coordinate system. (See Figure 6)[3]

Both p- and s- directions are used to represent the polarization states of an electromagnetic wave. The electric field can be defined using the p-direction and the s-direction since they form orthogonal vectors:

$$\vec{E} = \begin{bmatrix} \tilde{E}_p \\ \tilde{E}_s \end{bmatrix} \tag{3.4}$$

A beam of light reflected from a surface can be written in terms of the component of the light lying in the p- and s- directions, similar to Equation 3.4. Ellipsometry ultimately compares these two components, the polarization of the light to analyze the optical properties of the reflected (or even transmitted) surface.

3.2.3 Measurement

An ellipsometer measures the change in polarization state of the light reflected from the surface of a sample film. This sample film can be considered as an 'optical system' that modifies the polarization state of the beam of light.

The actual values measured are expressed as psi (Ψ) and delta (Δ). These values are related to the ratio of the Fresnel reflection coefficients $\tilde{\mathrm{R}}_p$ and $\tilde{\mathrm{R}}_s$ for p- and s- polarized light respectively.[3]

$$\rho = \frac{\tilde{R}_p}{\tilde{R}_s} = \tan(\Psi)e^{i\Delta} \tag{3.5}$$

$\tilde{\mathrm{R}}_p$ and $\tilde{\mathrm{R}}_s$ represent the complex valued reflections for p- and s- polarized light respectively. From Equation 3.5, it can be seen that Ψ and Δ correspond to the amplitude and phase of ρ. By measuring Ψ and Δ and fitting a model to it, the optical properties of a material can be deduced using a computer program. We use a WVASE32^{TM} software by J.A. Woollam Co., Inc.

3.2.4 Optical Constants

Ellipsometry can determine thin film thicknesses, optical constants, and in many cases both thickness and optical constants of the same film. Ellipsometry has also proven to be a valuable technique in determining optical constants in the near-UV, visible, and near-IR wavelength ranges.[3]

Optical constants are important in understanding the optical properties of the material. They vary over differing wavelengths of light propagating through the material. The optical constant can be represented as a complex index of refraction, $\tilde{n} = n + ik$. The real part n corresponds to the speed of light inside the material compared to the speed of light in vacuum. The imaginary part k corresponds to the amount of light absorbed through the material as the light passes through it.

Optical constants are especially important in ellipsometry. By using the correct optical constants over a range of wavelengths, an accurate and precise model of the sample film can be developed. (Section 3.2.5 on page 23) This allows accurate film thicknesses to be obtained by the ellipsometer. However, an ellipsometer can also obtain optical constants of a material. The accuracy of such constants depend on how many unknown variables there were when fitting for the constants. (Section 3.2.5 on page 23) For example, if the

thicknesses of each film layer was measured accurately by x-ray diffraction, then these thicknesses can be entered into the WVASE, and the optical constants can be fitted for these known values. The resulting optical constants could then be considered to be reasonably correct. Although it is possible to determine both optical constants and thicknesses, doing so would not yield highly reliable results as these two properties tend to be strongly correlated. (Section 3.2.5 on page 24)

3.2.5 General Experimental Procedures

Optical experiments never directly measure the sample parameters of interest (thickness, optical constants, and others), rather they measure some quantity that is a function of the parameters of interest. J.A. Woollam Co., Inc.[3] suggests four steps in conducting an optical experiment. These are the basic steps that our ellipsometer uses to obtain optical constants and film thicknesses.

Measurement

The parameters of interest (film thickness, optical constants, etc.) must be determined. Determine the optical quantities that can actually be measured (reflectance, transmission, Ψ, Δ), from which the parameters of interest can be derived. Then devise an optical experiment and measure beam intensities (reflectance and transmission) or polarization states (Ψ, Δ), possibly as functions of the light beam wavelength, angle of incidence, and/or polarization states.

Model Development

Construct an accurate model from which we can predict what we should measure from a sample with known properties. The model should contain some known parameters, such as the incident wavelength, incident polarization state, and the incident angle. It should also include unknown physical parameters, such as layer thickness and optical constants.

Fitting of Model

The unknown physical parameters can then be varied and the known parameters can be calculated from the model, until they closely match the actual

known parameters. The goal is for the model to be accurate enough, that through this process of fitting to the known parameters, the true values of the physical parameters are obtained. WVASE uses the Levenberg-Marquardt multivariate regression algorithm.

Evaluation for Best-fit

The best-fit set of physical parameters must be determined to be parameters that are unique, physically reasonable, and not strongly correlated. If these criteria are met, then the best-fit model probably represents the physical reality of the sample reasonably well. Statistics can also be used to evaluate the accuracy and precision of the fit results.

3.2.6 Advantages

Ellipsometry is very sensitive for thin film measurements. The sensitivity is actually greater than a simple reflectance measurement. The sensitivity of an ellipsometer is derived from determining the relative phase change (Δ from Equation 3.5) of the beam of reflected polarized light.[3]

Another advantage of an ellipsometer is that the absolute intensity of the reflected light does not have to be measured. This allows ellipsometric measurements to be more accurate than simple intensity reflectance measurements. From Equation 3.5, an ellipsometer measures the ratio of the two reflectance values for p- and s-. This provides accurate and reproducible results unique to ellipsometers.[3]

Because the ellipsometer measures relative phase and intensity rather than their absolute values, it can be operated effectively in most any type of environment. Conveniently, it is not necessary to maintain an ellipsometer in a dust-free area.

3.3 In Situ Ellipsometer

In situ ellipsometry refers to ellipsometry conducted in the original setting in which the sample was prepared. If a sample film is created inside a vacuum chamber, then in situ ellipsometry refers to ellipsometry conducted on the sample film inside the chamber, without removing the film.

3.3.1 Building

We have used an M-44® spectroscopic ellipsometer with UV option from J.A. Woollam Co., Inc. together with their WVASE^{TM} software to run the measurements, modelling, fitting, and other calculations. (The components of the M-44® ellipsometer are discussed in Sections 3.3.2, 3.3.3 on pages 25, 26)

Two Bomco windows are mounted, one on each of the two angled 2.75" flange ports that are on the side of the chamber body. The flange ports themselves form an angle of approximately 75°. Two window mounts, standoffs, and a steel window protection cage were installed over and around each Bomco window. The Input and Output Unit Tilt Stages were installed on top of these setups, one on each side. The Input and Output Units were mounted on their respective tilt stages. Minimal strain, if any, should be applied to the Bomco windows, especially during the mounting process, so as to eliminate birefringence on the windows.

The Input Unit installed is able to produce light from the in the IR (infrared) to UV (ultraviolet) range. Wavelengths of the light range from 280 nm to 600 nm. The incident angle of the light beam onto the sample film varies according to the position of the sample film in the chamber (controlled by the sample stage in Section 7.2 on page 78), the adjustments made to the Input and Output Tilt stages, and the original angle of the two ports on which the ellipsometer units are mounted. With the sample film down 6" from the top of the chamber (details in Section 7.2 on page 78), the tilt stages adjusted to correctly align the sample, the incident angle lies in the range of 69-73°. These were values measured from the ellipsometer by performing a normal fit for the angle using a ruthenium film inside the chamber. The range is approximate and may change depending on the sample and accuracy of the model used to fit the angle.

3.3.2 General M-44® Components

The M-44® ellipsometer consists of the following major components:[4]

Lamp LPS-300, which integrates the lamp housing and lamp power supply, is used. It is a highly regulated DC power supply that provides stable power to the arc lamp. A +12 kV and -12 kV are applied to the anode and the cathode of the system upon ignition. The Arc Lamp is a high pressure Xenon discharge point source lamp. Xenon gas above

atmospheric pressure is filled inside the lamp. The lamp provides the beam of light that enters the Input Unit through a fiber optic cable.

Input Unit The Input Unit conditions the beam from the Arc Lamp before it encounters the sample. It consists of an SMA Connector, Collimator, Shutter, and a Polarizer mounted in a rotational stage. (See Figure 9)

Output Unit The Output Unit consists of an analyzer and a detector that further conditions the beam from the sample, and converts the beam into electrical signals. (See Figure 10)

Analyzer During calibration and data acquisition the beam from the sample enters the Analyzer through a fixed aperture. The purpose of the aperture is to reject stray light and control the amount of light entering the Detector.

Detector The detector box contains a small grating that disperses the desired spectrum of light across a diode array. Additional circuitry conditions the signals from the diode array to signals that can be analyzed by the computer. There is a four-quadrant detector inside the detector box used for alignment.

Computer The computer controls all electrical systems of the ellipsometer. It initiates pulses to the stepper motor drivers in the EC-110. The computer controls all stepper motors using digital I/O cards and an analog to digital (A/D) card.

During the Alignment Procedure, signals from the Detector are sent to the computer and displayed as a cross hair on the computer screen. During the Calibration and Data Acquisition, the signal from the Detector is coupled to the A/D card within the computer. The software uses this information for calibration and analysis.

Electronics Control Box The two stepper motors are controlled by the Electronics Control Box, EC-110. The EC-110 contains all the power supplies and drivers for these motors. It also contains the circuitry for the shutter within the ellipsometer.

3.3.3 In situ M-44® Components

The following parts are unique to the in situ set up of the M-44® ellipsometer:[4]

Alignment Jig The Alignment Jig is mounted on the Output Tilt Stage during the Alignment Procedure if necessary. The reflected light beam from the sample should enter through the center of the entry-hole of the Alignment Jig. Then the light beam should be centered through the glass reticule (cross hair) at the end (bottom) of the Alignment Jig.

The Alignment Jig allows the path of the light beam through the Output Unit to be visible. This makes it easier to align the beam than using the Output Unit directly.

Bomco Window The Bomco windows, manufactured by Bomco, Inc., are made with fused quartz and are shaped like a bottle. They have a quartz to moly seal between the window and the 2.75" UHV flange. Bomco windows can be heated to 400°C to remove typical growth deposits on the windows. Retardation of Bomco windows are substantially and consistently less than conventional viewports.

Note: Because ellipsometers are very sensitive to birefringence of windows, no strain should be caused in the windows.

Input Unit Tilt Stage The stage on which the Input Unit is be mounted. The Input Unit Tilt Stage provides the tilting of the Input Unit so that the beam encounters the surface of the sample and reflects into the entry-hole of the Output Unit. (See Figure 9)

Output Unit Tilt Stage The stage on which the Output Unit is be mounted. The Output Unit Tilt Stage provides the tilting of the Output Unit so that the beam entering the entry-hole of the Output Unit passes straight into the Analyzer and Detector. (See Figure 10)

3.3.4 Operation

The system should be powered. Usually it is recommended that 30 minutes elapse from the turning on of the lamp before operating the ellipsometer. The system should then be initialized, aligned, and calibrated before taking actual measurements.

Detailed procedures of initializing, aligning, and calibrating the system and the sample may be found in *Hardware Manual M-44^{TM} Ellipsometer*[4]. Details on taking ellipsometric measurements, modelling, fitting, and analysis

can be found in *Guide to Using WVASE32^{TM} - Software for VASE and M-44 Ellipsometers*[3].

System Alignment

System Alignment is the adjustment of the input and output components relative to the fixed angle base of the M-44^{TM} system. It is required when the system is first delivered, first mounted on the chamber or base, or whenever the integrity of the alignment is in question.[4]

The System Alignment is done with the Input Unit and the Alignment Jig mounted on the Input Unit Tilt Stage and the Output Unit Tilt Stage respectively. The beam should be reflected off of the sample and enter straight through the Alignment Jig. The Input Unit Tilt Stage can be adjusted to allow the beam to reflect off the sample and center into the entry hole of the Alignment Jig. The Output Unit Tilt Stage can then be adjusted to allow the reflected beam to come out from the center of the cross hair on the bottom of the Alignment Jig.

System Calibration

System calibration is required when the M-44^{TM} is operated for the first time or the computer malfunctions, causing the hard drive to be corrupt. The system calibration is performed to determine the polarization sensitivity of the grating, the electronic attenuation of the amplifier circuitry, and phase shifts induced in the detector channel signals by the detector amplifier circuitry.

The system is calibrated by using the 'straight-through' mode. This requires that the Input and the Output Unit face each other directly. This is only possible in ex situ, so for in situ, the 'straight-through' is not quite plausible.

Sample Alignment

Sample alignment is required for each new sample. It is necessary in order to align the sample surface with respect to the probe beam and align the reflected light beam on the grating within the Output Unit.[4]

With the Output Unit on the Output Unit Tilt Stage, instead of the Alignment Jig, the sample can be aligned. The Input Unit Tilt Stage should be adjusted to ensure that the beam reflects off the sample and is centered into the entry hole of the Output Unit. With the computer and WVASE on,

by selecting Hardware|Align, a cross hair appears on the computer screen. The Output Unit Tilt Stage should then be adjusted so that the cross hair is centered onto the origin of the x, y-axes as close as possible. Usually $x = 0 \pm 0.5$ and $y = 0 \pm 0.5$ or better can be achieved fairly easily.

Sample Calibration

Sample calibration must be performed every time the M-44^{TM} hardware is initialized. This is to acquire data to determine the azimuthal angles of the polarizer, the analyzer, and the grating.[4]

First a Coarse calibration should be performed for the sample by selecting Hardware|Calibrate|Coarse calibration. After a successful Coarse calibration, a Normal calibration should be performed by selecting Hardware|Calibrate|Normal calibration.

Whenever the Electronics Control Box, EC-110, is turned on after being off, the sample has been replaced, or there has been a major change to the sample inside, it is *recommended* that a Coarse calibration be performed. This generates a calibration in a general 'ball park' area. The Normal calibration refines this 'ball park' and should be performed for each sample after the hardware has been turned on and initialized. If the ellipsometer and WVASE has been continuously on, then it is not necessary to perform subsequent calibrations after the first one.

3.3.5 Comparison with Ex Situ

The principles of ellipsometry and the procedures for measuring films are basically the same with in situ as that with ex situ (ellipsometry outside of the vacuum chamber). There are a few points to consider when conducting in situ ellipsometry.

Restrictions in In Situ Ellipsometry

- Usually incident angles cannot be varied to obtain more optical data of the sample. Data from varying angles allows for a tighter fit on the model. This option is not readily available for in situ analysis, since to change angles to obtain useful data would require changing the vacuum chamber ports on which the ellipsometer is mounted. This means that the ports would have to be bent at varying angles, which usually is

impossible for fixed stainless steel chambers.

- The sample cannot be easily taken out, since doing so requires the vacuum system to be brought back to atmospheric pressure. Thus analysis of multiple films is not efficient.

- Alignment and calibration must usually be done before the deposition of the sample occurs, i.e. on the silicon substrate. When metal is deposited on the film, the calibration may give erroneous values that could affect the accuracy of the measurements. It is recommended that the EC-110, the Electronics Control Box, remain on when measuring the same sample. This is to preserve the original calibration data that was obtained from the silicon substrate.

Advantages of In Situ Ellipsometry

- The sample can be measured continuously in real time as it is created or developed.

- Analysis can be made of films and layers of films that are not oxidized.

- The initial oxidation of films can be analyzed since ellipsometric measurements may be made without removing the sample from the chamber when exposing the sample with oxygen.

Chapter 4

Vacuum Pumps

4.1 Additional Chapter Definitions

Backstream The process of molecules or atoms leaving the unit that should contain them and travelling to a cleaner area of the vacuum system. Oil may sometimes backstream from a mechanical pump into a vacuum chamber if the pressure of the chamber is at lower pressure than the pump and the line between the two is open. Backstreaming can be a severe source of contamination in a vacuum system.

Cross Fitting Fitting shaped like a cross.

Crossover The point when pumping of a vacuum is switched from roughing pumps to high vacuum pumps. Usually around 1×10^{-3} torr.

NPT (National Pipe Thread) Type of threading that is angled so that the outer diameter decreases towards the end/tip of the threading. Because of the angle, as an NPT screw/shaft is tightened, the gap between the wall of the female fitting (the screw hole) and the wall of the male fitting (the screw/shaft) decreases. This provides a seal as the male fitting is screwed in the female fitting.

Reducing Flange Flange that allows a larger port to be connected to a smaller port.

Regeneration The process of emitting all the gases contained inside a vacuum pump out into atmosphere. Pumps that store the gases inside

require regeneration when the gas load inside is saturated. Saturation causes the pump to lose its capacity to pump.

Roughing Line The section of the vacuum system that contains the lines, valves, and pumps used to bring the chamber to roughing pressure. Usually the roughing line itself is not UHV leak tight since it is unnecessary, however the valve connecting the roughing line to the rest of the UHV system should be UHV leak tight.

Tee Fitting Fitting shaped like the letter 'T.'

Viton Polymer that may withstand temperatures as high as 150°C. Viton o-rings were preferred in our vacuum system over regular o-rings because of this property.

Zeolite/Molecular Sieve Material used to adsorb gases. Usually used in sorption pumps. When cooled to liquid nitrogen temperature, gases are bonded to the surfaces by Van der Waals forces.

Two types of pumps are used in our chamber- sorption pumps, and a cryopump. The operation of these pumps rely on two main principles:

1. One principle is to use cold traps, by which the material or components inside the chambers are cooled to very low temperatures to attract the gas from the relatively hot chamber. Sorption pumps go down to liquid nitrogen temperatures ($\sim$ 70 K), while cryopumps can go down to liquid helium temperatures ($\sim$ 4-10 K).

2. Another principle is to use material or components with very large surface areas. Sorption pumps use sieve materials, while cryopumps use activated charcoal that can have surface areas of several football fields.

Two sorption pumps are used to bring the chamber from atmospheric pressure to roughing pressure (1×10^{-3} torr). The cryopump then takes the chamber down to UHV pressure (under 1×10^{-9} torr).

4.2 Sorption Pumps

The term 'sorption pumps' includes all types of pumps that remove gases and vapors through sorption means- temperature-dependent adsorption forces

(van der Waals forces), chemisorption, absorption, or by becoming embedded during the formation of new sorbing surfaces. Some of the different types of sorption pumps are adsorption pump, getter pumps, evaporator pumps, and sputter pumps.[5] Only the adsorption pump will be described in this section as it is the type of roughing pump that is employed in our vacuum system. When 'sorption pump' is used in this thesis, it will be assumed to be an adsorption pump.

4.2.1 Advantages

- Sorption pumps were used in our vacuum system mainly because sorption pumps do not require any oil to operate. This allows the vacuum system to be clean from any accidental backstreaming of oil from the roughing pumps to the rest of the UHV system.

- Sorption pumps, although their usage in the vacuum industry has decreased over the past years, are relatively inexpensive.

- Sorption pumps are easy to maintain. There have hardly no mechanical failures. Regeneration of sorption pumps to their original pumping capacity only requires the molecular sieve to be baked out, and rarely requires the replacement of the molecular sieve.

4.2.2 Building

Two Varian sorption pumps (Model number 941-6501) are used in parallel in our system. The two are both standard size (15" in length) The sorption pumps were bought used. The zeolite, or molecular sieve material inside was first emptied, then the inside of the sorption pumps was cleaned with water, acetone, and methanol. After the pumps were air dried, the sorption pumps were filled with new molecular sieve. The new molecular sieve material used is $\frac{1}{16}$" molecular sieve pellets (MS-5A-23) from Duniway. Two half gallons were used to fill both sorption pumps.

The conical screen to protect the molecular sieve from leaving the sorption pumps was inserted back. Each sorption pump is mounted on a right-angle high vacuum valve from MDC. (See Figure 11) These valves in effect open or close the sorption pumps to the rest of the vacuum system. These valves can withstand temperatures up to 150°C because of the o-ring that

provides the seal. (Technically there are two o-rings that provide the seal. One seals against atmospheric air, and the other seals against the gas that passes through the line that the valve is mounted on. The former seal is what distinguishes between UHV and HV, UHV valves requiring a copper gasket seal for this. The former seal is also what affects the maximum temperature that the valve can withstand.)

The two MDC valves were then mounted on a three-way tee. This tee was mounted on a four-way cross. A thermocouple gauge to measure pressure is mounted on one end of the cross. Another end of the cross is connected to a UHV right-angle valve that is connected to the roughing line of the cryopump by a steel-reinforced rubber hose. The other end of the cross is connected to a UHV in-line valve that is mounted on to the main chamber body through a reducing flange. Both of these UHV valves are made by A&N Corporation, can withstand up to 204°C, and are rated to seal down to 10^{-10} torr.

All components and lines between the UHV in-line valve and the roughing line of the cryopump (including this line) is considered as the complete 'roughing line' for our vacuum system. The roughing line of the system does not have to be UHV leak tight nor UHV clean. However, it must be able to be completely isolated from the UHV section through a UHV valve, especially when the UHV section is under lower vacuum pressure than the roughing line (i.e. when the cryopump is pumping on the chamber). This is so that the UHV section of the chamber is not contaminated by the roughing line. Hence, the in-line valve connected to the chamber is the most crucial valve as it provides the UHV seal necessary to separate the roughing line from the UHV section of the chamber.

A thermocouple gauge is mounted in an $\frac{1}{8}$" NPT (National Pipe Thread) hole which is threaded on a 2.75" conflat flange. This flange is mounted on the top end of the four-way cross of the roughing line. The NPT threads of the thermocouple gauge was wrapped with teflon tape to provide a HV safe seal. The seal has been able to provide and adequate seal down to $10^{-3} - 10^{-2}$ torr. However if there is any leak in the roughing line, it is most likely from the thermocouple gauge as all other seals, besides that of the hoses connecting to cryopump, are HV leak tight (copper gaskets or o-rings).

4.2.3 Components

(Refer to Figure 12[5]) Pumped gases enter the sorption pumps through the inlet port (1). These gases are then adsorbed by the zeolite (6) inside the pump. Vanes (5) to enhance the cooling of the zeolite are contained inside the pump. The degassing port (2) should remain closed when the pump is operating, and open when it is regenerating.

Stainless steel dewars should be mounted on the sorption pumps. These dewars are where the liquid nitrogen is poured in to operate the sorption pumps. Heaters are also mounted around the outside of the pump bodies. The screws that tighten the heaters on the bodies were lubricated with anti-seize lubricant because of the high temperatures that the heaters reach ($\sim 250^o$C). (See Figure 11)

4.2.4 Principles of Operation

Adsorption pumps work according to the principle of adsorption of gases at the surfaces of molecular sieves. Zeolite is frequently used, which is molecular sieve.

Zeolite is an alkali aluminosilicate that possesses a very large surface area, about 1000 m^2/g of solid substance. The pore diameter of a zeolite 13X is about 13Å which is within the size of water vapor or oil vapor. Assuming that the mean molecular diameter of the average gas molecule is about 5Å, about 5×10^{18} molecules are adsorbed in a monolayer on a surface of 1 m^2.[5] A standard size sorption pump by Varian (which is what we have) contains nearly 300 acres of surface area.[6]

The adsorption of gases at surfaces depend on both the temperature and more importantly on the pressure above the adsorption surface. Typically if this pressure is larger, the adsorption is greater. By immersing the body of the pump in liquid nitrogen, the pump is activated. Best values are achieved for nitrogen, carbon dioxide, water vapors, and hydrocarbon vapors. However molecules with small particle diameters, such as hydrogen, helium, and neon, are poorly adsorbed by sorption pumps. As the pumped gases cover the zeolite surfaces, the pumping speed decreases.[5]

4.2.5 Operation

Two pumps are used in the system to provide ideal pumping to roughing pressure. One pump is first used to pump out the first stage of air inside the vacuum chamber. This first stage of air usually may be less clean (more contaminants) than later stages, so by pumping initially with one pump, the less clean air can be contained inside one of the pumps alone. Also as the air is pumped in, it creates a type of 'wind' that causes helium, hydrogen, and neon to flow into the pump initially. If the pump is kept open, then these gases may leak out from the pump since they are difficult for the zeolite to adsorb. However if one pump is used to initially pump on the chamber and then if this pump is closed, then much of the helium, hydrogen, and neon may be trapped inside this one pump. Thus by using one pump initially and then closing it, lower vacuum pressure and relatively cleaner vacuum can be achieved, than when using only one sorption pump for the whole process. Typically opening the first pump for 1-1$\frac{1}{2}$ minutes and then closing it is sufficient for a standard size sorption pump.

The following is the manufacturer's recommended procedure for using a single sorption pump in a vacuum system.[6] A standard operating procedure for pumping down with the sorption pumps in our system is found in Section A.2.

1. Make sure the roughing valves are closed, so the sorption pump is sealed off from the rest of the vacuum system.

2. Ensure that the Viton stopper is firmly inserted in the degassing port. A *light* film of vacuum grease on the inside of the Viton relief-valve (on the degassing port) and on the Viton stopper will insure a leak-tight seal.

3. Place the liquid nitrogen container (the dewar) around the pump and fill it. After a few minutes of boiling of liquid nitrogen, refill the container so that the liquid nitrogen surface reaches the top of the 4.5" diameter portion of the pump body.

4. Allow the sorption pump to chill for at least 10 minutes or longer. After one hour of chilling, most of the molecular sieve will be near liquid nitrogen temperature.

5. Open the valve between the sorption pump and the vacuum system. Observe pressure decrease on the meter (thermocouple gauge in our system).

6. Leave the pump open to the system until the pressure is less than 10 mtorr. (It is beneficial to go as low as possible and then hold that pressure for an hour or two before closing the system from the sorption pump) Then close the roughing valve and begin pumping of the chamber by a high vacuum pump.

7. When roughing is complete, remove the liquid nitrogen container and allow the pump to warm to room temperature. Complete warmup requires 1-1$\frac{1}{2}$ hours. Pressure inside the pump will increase, causing the Viton stopper to come off the relief valve on the degassing port. The pump may also be warmed to room temperature in 30 minutes or less by using the wrap-around heaters.

8. When the pump is not used, keep the Viton stopper in the relief valve so that vapor is not accumulated in the molecular sieve. Also valve off the pump to keep out water vapor and other gases.

Caution

It is important that when the roughing line is closed to the rest of the vacuum chamber, it remains closed unless it can be verified that the roughing line is at lower vacuum pressure than the section of the chamber that it may open to. If the roughing line is open to a section of the chamber that is at lower vacuum pressure, then contaminants from the relatively dirty roughing line may enter the UHV clean section of the chamber. Probably most damaging may be possible molecular sieve dust that could escape the roughing pump because of the pressure differential.

One way of doing this is to place liquid nitrogen in the dewars and start pumping on the roughing line. The pressure of the section of the vacuum chamber that the roughing line will open to must be above 1×10^{-3} torr. After the roughing line reads below 1×10^{-3} torr, the roughing line may then be opened to that part of the chamber.

4.2.6 Regeneration

Most gases desorb, or leave, from the molecular sieve surfaces at room temperature. However water vapor remains with great tenacity. Once a considerable quantity of water vapor is accumulated in the molecular sieve, it is necessary to remove the water vapor by baking out the sorption pumps, hence regenerating the pumps.

The wrap-around heaters, when turned on, can heat the pumps up to about 250°C after about $2\frac{1}{2}$ hours. A standard operating procedure for regenerating the sorption pumps in our system is found in Section A.3.

4.3 Cryopump

The cryopump that is installed in our vacuum system is a Cryo-Torr® 8 High Vacuum Pump, made by CTI-Cryogenics. It is a refrigerator type cryopump, which can become 'cold upon demand,' like a refrigerator. (See Figure 13)

Some of the characteristics of the Cryo-Torr® 8 High Vacuum Pump are listed in Table 4.1.[7]

Table 4.1: CTI-Cryogenics Cryo-Torr® 8 High Vacuum Pump Capacity

Pumping Speeds - liters/sec	
Water Vapor	4,000
Air	1,500
Hydrogen	2,500
Argon	1,200
Capacity - std. liter	
Argon	1,000
Hydrogen(5×10^{-6} torr)	12
Cooldown time	
	1.5 hrs.
Dimensions	
Length - in. (mm)	20.7 (526)
Weight - lbs. (kg)	45 (20.4)

4.3.1 Components

A refrigerator cryopump consists of a compressor unit, cold head, pump casing, and flexible pressure lines. (See Figure 14).

Compressor Unit

The compressor unit provides the power for the refrigerant to circulate throughout the cryopump. Helium is used as the refrigerant because of the low condensation temperature it can achieve.

Cold Head

The cold head is the unit where the gases from the vacuum system cools within the cryopump. The gases bond to the cold surfaces of the panels of the cold head and condense. The cold head together with the casing are mounted directly onto the vacuum chamber with a gate valve in between.

Pump Casing

The metal casing that surrounds the cold head. It provides the structural frame of the cryopump.

Pressure Lines

The helium flows through the flexible pressure lines in a closed cycle. The pressure lines connect the cold head with the compressor unit.

4.3.2 Principles of Operation

The general principles behind the operation of a refrigerator pump will be described.[5] Much of the pumping from a cryopump relies on condensation that occurs in vacuum. This is similar to how water condenses on cold glass surfaces, and ice forming on the evaporator unit of a refrigerator. Layers of frozen gases are stacked on to the panels of the cryopump due to bonding forces from mostly thermal interactions.

Figure 15 shows the design of a refrigerator cryopump. (adapted from *Fundamentals of Vacuum Technology*[5]) Cooling within the cryopump is provided by a two-stage cold head. The thermal radiation shield (5) and

the baffle (6) are closely linked thermally to the first stage (9) of the cold head. For pressures below 10^{-3} torr thermal radiation causes most of the heating within the cryopump. For this reason the second stage (7) with the condensation and cryosorption panels (8) is surrounded by the thermal radiation shield (5) which is black on the inside and polished and nickel plated on the outside.

With no load the baffle and the thermal radiation shield (first stage) attain temperatures in the 50-80 K range at the cryopanels. At the second stage the temperature is about 10 K. The surface temperatures of these cryopanels are important in the actual pumping process. These surface temperatures are dependent on the refrigerating power supplied by the cold head, and the thermal conduction properties in the direction of the pump's casing. During operation of the pump, loading of the gas and heat from condensation causes further warming of the cryopanels. The surface temperature depends on both that of the cryopanels and that of the gas frozen on to the cryopanel.

The cryopanels (8) attached to the second stage (7) of the cold head are coated with activated charcoal on the inside. The activated charcoals allow gases that do not easily condense to be pumped by cryosorption.

4.3.3 Bonding of Gases

As the condensate (solid) of the gases on the panels increase in thickness, thermal resistance and thus the surface temperature increase. This reduces the pumping speed and capacity of the cryopump. The bonding process for the various gases in the cryopump is performed in three steps:[5]

1. First the mixture of different gases and vapors meets the baffle whose temperature is about 80 K. Mostly H_2O and CO_2 are condensed here. The partial pressures of these gases drop below 10^{-9} torr at T$\approx$ 77 K.

2. The remaining gases penetrate the baffle and impinge on the outer surface of the cryopanel of the second stage, whose temperature is about 10 K. Here gases like N_2, O_2 and Ar will condense. The partial pressures of these gases drop below 10^{-9} torr at T$\approx$ 20 K.

3. H_2, He and Ne remain after these two stages. These gases, after several impacts with the thermal radiation shield, pass to the inside of the cryopanels which are coated with an adsorbent (cryosorption panels)

where they are finally bonded by cryosorption. The partial pressures of these gases drop below 10^{-9} torr at T< 4.2 K.

There are differences in the bonding mechanisms involved in the operation of a cryopump:[5]

Cryocondensation

The physical and reversible bonding of gas molecules through van der Waals forces caused by sufficiently cold surfaces of the same material. The bond energy is the energy of vaporization of the solid gas bonded to the surface. The bond energy decreases as the thickness of the condensate and the vapor pressure increase.

Cryosorption

Physical and reversible bonding of gas molecules through van der Waals forces caused by sufficiently cold surfaces of other materials. The bond energy is equal to the heat of adsorption which is greater than the heat of vaporization.

In cryosorption gas molecules are adsorbed into adsorbent materials such as activated charcoal, silica gel, alumina gel and molecular sieve. These materials have very large specific surface areas of about 10^6 m^2/kg.

As soon as a monolayer of adsorbed gases form, the gases condense on to surfaces of the same kind, hence returning to cryocondensation. The higher bond energy of the cryocondensed layer further restricts the capacity for the adsorbed gases. This is why the adsorbent materials must have large surface areas.

Cryotrapping

The inclusion of a low boiling point gas which is difficult to pump (such as hydrogen), in the matrix of a gas with higher boiling point (such as Ar,CH_4 or CO_2) which can be pumped easily. At the same temperature the condensate mixture has a saturation vapor pressure which is several orders of magnitude lower than the pure condensate of the gas that has the lower boiling point.

4.3.4 Operation Procedures

The following procedures are recommended by the manufacturer- CTI-Cryogenics, Helix Technology Corporation.[8] A standard operating procedure for pumping down with the cryopump in our system is found in Section A.2.

Before Start-up

The compressor unit, cold head unit, lines, and power source should be properly installed.

Operating Log An operating log will assist in troubleshooting if problems arise. The log should include the cooldown time to 20 K, the roughing time to 50 mtorr, the time to base pressure at crossover, the time between regeneration, and the compressor pressure reading.

Rough Pumping

Roughing pressure between a minimum of 50-75 mtorr should be achieved before the cryopump begins to pump on the vacuum system.

Rate-of-Rise (ROR)

Rate of the pressure rise is an important measure of the tightness of the vacuum system. To measure the rate of pressure rise, close the roughing valve when the pressure has reached 50-75 mtorr. Observe the rate of pressure rise over a five-minute period.

$$\boxed{ROR < 10 \text{ mtorr/minute over a five-minute period}} \tag{4.1}$$

The rate of rise in Equation 4.1 (50 mtorr total) is an indication of the integrity and cleanliness of the cryopump and the vacuum system for high vacuum usage. If the total ROR is greater than 50 mtorr, the cryopump should be repurged, leaks should be checked, and the roughing cycle and ROR procedure should be repeated.

Start-up and Cooldown

1. Confirm that the gate valve connecting the cryopump to the chamber is closed. Confirm that the pressure in the cryopump is approximately

100 mtorr. **Most importantly ensure that the roughing valve is closed so that the roughing line is completely sealed from the cryopump. If this is not done then sieve dust may backstream into the chamber and the cryopump, contaminating and even compromising the whole vacuum system.**

2. Turn on the system power ON/OFF switch for the compressor and then the cold head. (See Figure 16)

3. Note the helium pressure and the temperature reading during the initial cooldown. The typical values that should be achieved are shown in Table 4.2.[8] Refer to the Troubleshooting section of the Cryo-Torr 8 manual[8] if problems arise.

4. When the cooldown temperature of 20 K or less is reached, the cryopump is ready for normal vacuum operation. An additional 30 minutes will allow the cryopump to reach the bottom temperature.

5. Record the time that was required to reach 20 K in the log. Also record the compressor return gas pressure at 20 K. These values may be useful for future evaluation of cryopump performance.

Table 4.2: Typical pressure variations during cooldown and normal operation (all values are nominal)

Cryopump Model	Compressor Model	Time	Nominal Helium Pressure Psig (kPa)	Temperature Indicator Reading	$H_2 V_P$ Reading (Psia)
CT-8	8200	Before start-up 90 mins. after start-up	250 (1725) 280 (1930)	300 10-20	— 20

4.3.5 Regeneration

Because the cryopump condenses and stores all of the gas molecules pumped inside the pump, after some time the cryopump becomes saturated. This saturation reduces the capacity of the cryopump to maintain high vacuum

pressures. Gases that are captured inside the cryopump can be released by bringing the cryopump to room temperature or higher and opening it to atmosphere.

If the cryopump becomes incapable of maintaining high-vacuum (typically an increase of the vacuum chamber base pressure by a factor greater than 10, despite satisfactory performances by the compressor and cold head), the cryopump needs regeneration.[8] Also a ring of ice or condensation around the exterior of the cryopump are signs that the cryopump is saturated and must be regenerated.

After saturation, a regeneration of the cryopump that is complete and thorough will increase the pumping ability of the cryopump, hence lowering the base vacuum pressure of the overall vacuum system. There are several methods to regenerate the cryopump. One is to simply turn the cryopump off, close the gate valve, let the pump come to room temperature, and pump out the gas from the cryopump through the roughing pumps. Another is to bake out the cryopump while regenerating (this is what we have implemented; procedures are described in detail in Section A.3). The most effective may be an assisted regeneration that uses dry inert gas to purge the cryopump.

4.3.6 Assisted Regeneration

Assisted regeneration incorporates the use of heated dry inert purge gas (nitrogen or argon) to regenerate the cryopump.

Advantages

The advantages of assisted regeneration over unassisted regeneration are:[8]

1. Minimizes the time required to bring the condensing and cryo-adsorbing arrays to room temperature.

2. Reduces the time required to pump the cryopump to roughing pressures. This is because the dry inert purge gas will minimize the amount of residual water vapor in the 15 K array.

3. Dilutes hazardous gases and ensures their removal from the cryopump housing.

Procedures

The following are the steps to conduct assisted regeneration:[8] (Refer to Figure 17)

1. Isolate the cryopump from the vacuum chamber by closing the gate valve.
2. Shut off the cryopump by turning off the ON/OFF switch of the cold head and the compressor.
3. *Immediately* introduce heated dry purge gas through the vacuum vessel purge fitting at approximately 150°F (66°C) and at a flow rate of 1-2 cfm. Allow the purge gas to vent through the 'poppet' relief valve.
4. Halt the gas purge when the condensing arrays reach 80°F (26°C) (300 K).
5. When the condensing arrays reach ambient temperature, rough the cryopump to 50-100 mtorr. After roughing, perform a rate-of-rise (ROR) test to ensure that the cryopump regeneration has been thorough and that no leaks are present in the system.
6. Close the cryopump from the roughing line and start the cryopump.
7. The cryopump is ready for use when the second stage cold head reaches a temperature of 20 K or lower.

Chapter 5

Vacuum Components

5.1 Additional Chapter Definitions

Backfill Filling a volume with material; usually a closed chamber is filled with gas.

Dewar Container used to hold liquid gases at low temperatures. Dewars are thermally insulated so that the gas can last and so the container itself does not become cold.

Inner Diameter (ID) The diameter size of the inner boundary.

Load-lock Isolating one particular volume in a vacuum chamber from the rest of the chamber. This is done to open this particular volume to atmospheric pressure while maintaining the rest of the chamber under vacuum pressure. Load-lock provides an efficient method of changing samples since changing samples would require only a small volume to be pumped down to vacuum instead of the whole chamber.

Mass Spectrometer Instrument used to determine the composition of the gas inside the vacuum system.

Outer Diameter (OD) The diameter size of the outer boundary.

5.2 Chamber

The main body of the vacuum chamber was custom built by Varian, Inc. The chamber body is made of stainless steel. The total volume enclosed by the main body, excluding all additional components installed, is approximately 15-20 liters. The chamber sits on a steel cart to provide support and mobility. All the flanges on the original body are conflat, sealed with copper gaskets. An ion gauge was already installed on one of the ports of the chamber, presumably by Varian, before I started working on the system in April 2000. Many of the components that are mentioned in this section will be described with more detail in later chapters of the thesis.

In Table 5.1, each port of the main chamber body, its diameter, and corresponding usage are listed.

5.3 Gate Valve

A gate valve is used to separate the vacuum chamber from a pump, typically a high vacuum pump. It provides partial or full sealing of the chamber from the high vacuum pump.

5.3.1 Advantages

When the gate valve is closed, the high vacuum pump can first pump on itself, meaning the high vacuum pump can reach optimal vacuum pressure with no load to pump, before pumping on the chamber. For example, cooling of the cold head for a cryopump can be achieved much faster with a gate valve closed against the chamber.

The gate valve allows gas flow in the high vacuum pump to be separated from the gas flow in the chamber and the rest of the vacuum system. (It should be noted that the port on the end of the cryopump, which is connected to the roughing line, must be sealed in order to completely separate the cryopump from the entire vacuum system.) This allows separate testing of the cryopump and the rest of the chamber when conducting a leak detection test.

The gate valve can be closed, so that when the chamber is open to atmospheric pressure, the cryopump can still maintain high vacuum pressure. This eliminates the need, and hence saves time, for the cryopump cold head

to cool down each time the chamber is to be pumped down from atmospheric pressure.

5.3.2 Building

(Refer to Figure 22)

The gate valve installed is a pneumatic aluminum gate valve. The gate valve is mounted on an 11" diameter reducing flange that fits on the 8" diameter port of the main chamber body. This reducing flange was custom ordered to allow the gate valve to mount on to the chamber properly. The seal between the reducing flange and the main chamber uses a conflat copper gasket. The seal between the gate valve and the reducing flange uses a Viton o-ring. The cryopump is mounted on the other side of the gate valve with a Viton o-ring seal.

The gate valve was taken apart, and all of its parts were cleaned with hexane, acetone, and methanol. All o-rings, including those in the shaft that opens/closes the gate valve, were replaced with new Viton o-rings. Viton o-rings were used so that the gate valve and all its seals could withstand up to 150°C temperatures during bake out. As we baked out the chamber up to 270°C, the outside of the gate valve was well below 150°C. We assume that the temperature of the inside of the gate valve to be similar to the outside, especially because no heating tapes were directly placed on the gate valve. Caution should be taken, however, so that the temperature of the gate valve never exceeds 150°C.

One important note on the design of the gate valve is that both sides of the gate valve has o-ring grooves that are the same size. For our system it is imperative that only one of these grooves on the gate valve be used. A Viton o-ring should be used only on the side facing the reducing flange on the chamber body. The o-ring groove on the other side of the gate valve should not be used. Instead a Viton o-ring should be placed on the inlet port of the cryopump, which itself contains an o-ring groove. (Refer to Figure 17) This is because the o-ring groove of the gate valve is too small to provide a seal between the cryopump and the gate valve. The reason that our initial vacuum pump down would not achieve 10^{-3} torr was because we placed an o-ring on the o-ring groove of the gate valve instead of the one on the cryopump. This o-ring was not in contact with the mounting flange of the cryopump. Instead it was positioned inside the open inlet of the cryopump, where the 80K condensing arrays are. After this leak was detected and fixed,

the chamber was able to achieve below 10^{-3} torr.

Although the gate valve is not itself quite UHV clean (because of the use of polymers, vacuum grease, and seemingly perpetual dirt that could not be completely eliminated), because it is right next to the cryopump, the contamination should not affect the main UHV part of the chamber.

5.3.3 Components and Operation

A steel support to hold the gate valve was built and installed on the steel cart on which the chamber sits. The gate valve, due to the bolt positions of the reducing flange (error in the custom design of the reducing flange), is tilted at a small angle. To compensate for this angle, an incline metal support piece was also needed. This is mounted directly below the gate valve and is held by two bolts (with 5 markings, which denotes the bolts are some of the strongest bolts). (Refer to Figure 23)

The steel support provides mechanical support primarily for when the cryopump in mounted on the gate valve. The flange of the chamber that the gate valve is mounted on is strong enough to support the gate valve alone. However, the cryopump will provide too much torque for the flange to support both the gate valve and the cryopump.

An electrical switch to operate the pneumatic gate valve was built with a solenoid and pressurized air lines. When the switch is turned off, the gate valve closes. An aluminum handle was built to allow the partial opening of the gate valve when backfilling the chamber with argon, in order to create plasma for sputtering. (Refer to Figure 23)

The pneumatic component of the gate valve has been taken off. It was observed that the gate valve would not completely seal when using the pneumatic operation. A wrench about one foot in length is needed to provide enough torque manually to close the gate valve completely. Until improvements or fixes are made to the pneumatic capability of the gate valve, a foot long 1" wrench should be used to close and open the gate valve. It is suspected that the mechanical structure of the gate valve inside may be the cause of the defective pneumatic seal. When the gate valve is completely closed, it makes a clicking sound that can be both felt and heard when manually closed by the wrench.

The sealing of the gate valve is provided by a flange inside. (Refer to Figure 24) This flange has a Viton o-ring installed on its side facing the cryopump. When the flange is completely closed, by a mechanical force

coupled with the o-ring, the flange provides a high vacuum seal that can seal off even atmospheric pressure.

5.4 Gauges

There are two gauges to measure the pressure in our vacuum system. A TC (thermocouple) gauge is used to measure from atmospheric pressure down to roughing pressure, and an ion gauge is used to measure from roughing pressure down to UHV vacuum pressure. Both gauges are connected to a Varian senTorr^{TM} gauge controller which displays the pressure readings for both gauges.

Vacuum gauges to monitor the pressure within the chamber are important in evaluating the vacuum capability of any vacuum systems. It is important to understand the limitations, range, and reliability of the gauges that are used in order to assess the vacuum system properly and determine the actual vacuum of the system.

5.4.1 Thermocouple Gauge

A thermocouple gauge, Model DST-531 from Duniway Stockroom Corp., is mounted on the top end of the four-way cross of the roughing line. This is the only gauge in our vacuum system used to measure from atmospheric pressure down to roughing pressure. (See Figure 25)

The thermocouple gauge is mounted on the roughing line because of its NPT (National Pipe Thread) thread which is not UHV leak tight. The NPT threads on the thermocouple gauge provide some vacuum type sealing, however clean teflon tape must also be wrapped around the NPT threads to provide high vacuum tight sealing.

A cable to connect the thermocouple gauge to the senTorr^{TM} gauge controller was made by Scott Daniel. The senTorr^{TM} gauge controller displays the pressure read by the thermocouple gauge. In atmospheric pressure, before pumping the system (or at least the roughing line), the thermocouple gauge reading on the senTorr gauge should be calibrated to atmospheric pressure (typically 7.6×10^2 torr). This provides a point of reference for future vacuum pressure readings by the thermocouple gauge.

Limitations

It should be noted that the thermocouple gauge nor the senTorr gauge controller is able to accurately and reliably read pressures near atmospheric pressure (7.6×10^2 torr). Varian states that their 531 Thermocouple Gauge (which is what Duniway's model is similar to, if not exact.) provides accurate indication of total pressure from 1×10^{-3} torr to 2.0 torr.[9]

In our first attempt with the vacuum system, the thermocouple gauge reading on the senTorr gauge read $3.6 - 3.9 \times 10^2$ torr even though the pressure of the roughing line was at least at atmospheric pressure. This was even after calibrating the thermocouple gauge reading on the senTorr gauge to 7.6×10^2 torr before pumping on the system. This is simply the mechanical limitations of a thermocouple gauge and the gauge controller.

Operation

Because there is only one roughing pressure gauge installed on the vacuum system, careful and creative methods must be used to measure the roughing pressure of a given section of the vacuum system. Luckily there are 4 valves immediately next to the thermocouple gauge to provided some control. Also the gate valve provides additional control.

Usually there are only three sections in our vacuum system that requires the roughing pressure to be measured by the TC gauge.

1. The main chamber body: The roughing pressure must be measured before opening the main chamber to the cryopump. This can be measured by closing all valves but the one valve connecting the roughing line to the main chamber body. (So 3 roughing line valves and the gate valve should be closed.)

2. The cryopump: The pressure inside the cryopump should be monitored to determine whether an appropriate regeneration of the cryopump has been achieved. This can be measured by closing all valves but the one valve connecting the roughing line to the cryopump roughing line. (So 3 roughing line valves and the gate valve should be closed.)

3. The roughing line: The pressure of the roughing line must be measured so as to determine when to open the roughing line to any other sections of the vacuum chamber. Typically it is safest to only open the roughing line to any other section when the roughing line is below 1×10^{-3} torr.

The pressure of the roughing line should be measured by closing all 4 valves.

Perhaps the most important precaution to take when using the thermocouple gauge is to ensure that no contaminants from the roughing line backstream into the section of the vacuum chamber that the TC gauge is trying to measure. If the section that the TC gauge is trying to measure has been open to the roughing line, then there is no problem in using the TC gauge. However, if the roughing line has been closed to this section of the chamber, then before opening it to the roughing line to use the TC gauge, it must be ensured that the roughing line is at lower vacuum pressure than the pressure of this particular section.

For example, suppose that the main chamber body is closed to the roughing line and is at a pressure above 1×10^{-3} torr. (Since the ion gauge cannot read pressures above 1×10^{-3} torr, when the ion gauge does not read the pressure of the main chamber, assuming it being still functional, the main chamber can be though to be at pressures above 1×10^{-3} torr.) To measure the pressure of the chamber, the TC gauge must be used because it is above high vacuum pressure. If the roughing line is at 1×10^{1} torr, then the valve connecting the chamber and the roughing line should not be opened. This is because there is a possibility that the main chamber may still be at pressures lower than 1×10^{1} torr. Opening the roughing line in this case may cause the relatively dirty roughing line to contaminate the UHV clean main chamber, and may even cause molecular sieve dust to enter into the chamber. This is due to the pressure differential between the roughing line and the main chamber. It is always better to be overly cautious than to not be careful and contaminate a UHV vacuum system.

5.4.2 Ion Gauge

A D571-K2471-303 Bayard-Alpert type ionization tube gauge is mounted on a 2.75" conflat flange on the port to which the roughing line is connected. It is directly connected to the main UHV section of the vacuum chamber. The ion gauge is connected to the senTorr^{TM} gauge controller through a cable. (See Figure 25)

The ion gauge can withstand long periods of degassing or accidental exposure to atmosphere at operating temperature and still recover its original characteristics. The sensitivity of the ion gauge is typically 10 $(\text{torr})^{-1}$. The

operating pressure of the ion gauge is 2×10^{-10} torr to 1×10^{-3} torr.[9]

Limitations

The ion gauge can only be operated at pressures below 1×10^{-3} torr. Hence above this pressure, the ion gauge should not be operated. The senTorr gauge actually provides a safety mechanism that shuts the ion gauge off when the pressure is above 1×10^{-3} torr. So unless the ion gauge is malfunctioning, when the ion gauge cannot read the pressure and the senTorr gauges shuts it off, the pressure may be assumed to be greater than 1×10^{-3} torr.

Also the thermocouple gauge reading and the ion gauge reading usually do not coincide. In our initial pump down, it took some additional pumping by the roughing pumps for the ion gauge to read below 1×10^{-3} torr; this was after the thermocouple gauge had already read below 1×10^{-3} torr.

5.4.3 Gauge Controller- senTorr^{TM}

(See Figure 26)

The senTorr^{TM} Gauge Controller by Varian has been installed to display the pressure readings from both the thermocouple gauge and the ion gauge. Up to two thermocouple gauges and one ionization tube may be connected to the senTorr gauge. The setpoint option has not been installed on our senTorr gauge. This option could allow automatic control of pneumatic valves that open or close depending on the programmed pressures of the thermocouple or ionization gauges. It is called a gauge controller because of this ability to control the operation of the vacuum system according to the measured pressures from the gauges.

The senTorr^{TM} Gauge Controller supplies the power for the ionization gauge. To protect the ion gauge, the senTorr gauge automatically turns the ion gauge off when pressures are too high (usually above 1.0×10^{-3} torr). Whenever a thermocouple gauge is connected to the senTorr gauge, and the senTorr gauge is turned on, the pressure measured by the thermocouple gauge will automatically be displayed on one of the TC1 or TC2 displays.

The configuration, functions, and options of the senTorr^{TM} Gauge Controller can be found in the Instruction Manual.[10] There are three pressures that can be displayed- the top one for the ion gauge and the two lower displays for two thermocouple gauges (TC1 and TC2).

The senTorr^{TM} Gauge Controller has a programmable option to auto-

matically turn the ion gauge on when the TC1 gauge reads between either 1.0×10^{-3} to 5.0×10^{-3} torr or 1.0×10^{-2} to 5.0×10^{-2} torr.

Some of the basic functions of the senTorr^{TM} Gauge Controller are listed below:[10]

Option Select Puts the senTorr into programming mode.

Units Pressing this key toggles the pressure measurement between Torr, mBar and Pascal for all pressure readings.

Cal This calibrates the vacuum and atmospheric readings for the thermocouple gauges.

Emis Turns the high vacuum gauge (the ion gauge for our system) on or off. The high vacuum gauge emissions will come on only if the appropriate vacuum has been achieved.

It has been advised that all vacuum chambers can possibly become charged to lethal voltage levels if they are not properly grounded with the common ground of the controller of the ionization tube.[10] The chamber has been grounded using braided copper wires connecting to the ground of the electrical lines in the Physics Underground Laboratory.

5.5 Flanges

Flanges are the metal plates that cover and seal the open ports of vacuum chambers. There are many different methods of providing the seal. Our chamber utilizes two main sealing methods- conflat copper gaskets and o-rings.

Where possible in our vacuum system, all flanges are conflat flanges because of the UHV tight seal that conflat flanges provide. O-rings have been avoided. Any of the areas where o-rings are used to seal atmospheric air from the inside of the vacuum system can be potential leak sources for UHV vacuum pressure.

5.5.1 Conflat (CF) Flanges

Conflat flanges are the industry standard for achieving UHV vacuum. Two flanges with knife edges are screwed together with a copper ring gasket placed

in between. (See Figure 4[11]) The knife edges press annular grooves into the soft copper gaskets, and the extruded fills all voids or defects, providing a very tight high vacuum seal. A typical CF flange can withstand temperatures in the range of -196°C (-321°F) to 450°C (840°F).[11]

There are two basic CF flange designs: fixed (non-rotatable) and rotatable. The rotatable flange is very useful when the component that will be mounted on has to be turned at certain angles to be placed properly (such as the tee used for the oxygen gas lines). In the rotatable flange, the knife edge and bolt hole ring are separate pieces. During welding, the knife-edge piece is welded to a tube or component, leaving the bolt-hole-ring free to rotate. This allows the component to be mounted in any orientation regardless of the bolt hole orientation of the flange to which it is attached.[11] (See Figures 27, 28[11])

Two conflat flange surfaces are bolted together. To allow the bolts to also withstand high bake out temperatures, a light layer of anti-seize lubricant should be applied. It is important to be aware of the lubricant so as to not contaminate the inside (any area within the copper gaskets of the vacuum chamber) of the chamber when opening or closing the ports with the conflat flanges. Caution should be taken to avoid or clean lubricant contamination.

5.5.2 O-ring Fittings

O-rings have been used in our system for the gate valve and the cryopump which cannot have conflat fittings due to their manufactured designs. An o-ring is also used for the quick-coupler fitting on the flange of the sputter gun. All o-rings used in the vacuum system have been replaced with new Viton o-rings, which can withstand up to 150°C. If possible, o-rings should be eliminated in the system because of the inability of o-rings to provide UHV tight seals.

An o-ring is pressed against two mating surfaces to provide a seal. There are various types of mechanisms to provide o-ring sealing. One type uses an o-ring inside a quick-coupler fitting, which is pressed against a shaft when the quick-coupler fitting is tightened. This type was used for the sputter gun. The most commonly used o-ring sealing methods are those of QF and ASA flanges.

QF Flanges

A QF assembly consists of two identical ('sexless') flanges, a clamp, and a centering ring to align the flanges and to prevent the o-ring from collapsing. The o-ring is mounted around the outer diameter of the centering ring. The two QF flanges are typically tightened through a thumbscrew clamp. A QF system is ideal for small diameter tubing that needs frequent reassembly. QF fittings work in rough or foreline vacuum plumbing and simple HV (10-6 torr) chambers.[11]

A QF flange is used at the end of the cryopump, connecting to the rubber hose of the roughing line. (See Figure 29

ASA Flanges

An ASA flange has either a flat surface or an o-ring groove machined in it. A series of holes for bolts encircles the edge of the flange. The pair of flanges are bolted together, one flat and the other grooved with an o-ring installed. If the mating pair has both o-ring grooves of identical ID's (inner diameter) , then o-rings should be places in both grooves with a ring of thin metal sheet to separate them. If both flanges are smooth, flat rubber gaskets or o-rings molded into carriers between the flange faces can be installed. Finally, when mating a normal flange pair, it is important that the flat flange has a smaller ID than the o-ring, or else the o-ring will not provide a seal.[11] (See Figure 30[11])

ASA flanges with Viton o-rings are used for both the gate valve and cryopump flanges in our vacuum system. Because these o-ring flanges are close to the pumping source, we assume that the pumping rate would be greater than the leak rate through these flanges. Also, if the vacuum grease flows out from the o-rings, they will most likely be pumped toward the cryopump, hence away from the UHV clean chamber.

Ordering O-rings

To order o-rings for ASA flanges, first the inner diameter (ID), outer diameter (OD), and thickness (width) of the o-ring or the o-ring groove should be measured. These measurements can then be used to order o-rings. However it is more efficient, if possible before ordering, to look in an industry standard manual for o-rings and determine the number that represents the size of the o-rings. Joseph Young in the Physics Department can provide much help in

determining the appropriate number for the size of the o-rings based on the measurements. These numbers can then be used to order the appropriate o-rings. (See Figure 31[11])

Vacuum Grease

It is generally advised that a light film of vacuum grease be used to provide enough lubrication for the o-rings to seat properly in the o-ring grooves. Dow Corning® high vacuum grease was used in our vacuum system. Typically vacuum grease has low outgassing rate that is compatible for high vacuum systems.

The following is a guideline when applying vacuum grease on to o-rings used for ASA flange type fittings.

1. The o-ring should be cleaned with some methanol and Kimwipes.

2. With clean gloves, a thin film of vacuum grease should be applied with the fingers evenly around the o-ring.

3. Place the lubricated o-ring inside the o-ring groove.

4. Carefully bolt the flanges together, avoiding contact of the o-ring on to other surfaces. The grease should be thought of as a contaminant when placed on unnecessary surfaces.

According to Kurt J. Lesker Company, leak-free joints depend as much on the absence of dust or lint particles on the o-ring and imperfection on the flange surfaces as on the presence or absence of grease. The choice between ‘to grease or not to grease’ probably relates to how oil-free the user wishes the system to be. Greases, in general, have very low vapor pressures, but any measurable vapor pressure means the grease will (very slowly) migrate from the O-ring to other surfaces.[11]

Hence it is important to be wise in the usage of vacuum grease in a vacuum system. If it can be avoided, vacuum grease should not be used. It should also be noted that vacuum grease tend to flow out from the o-ring surfaces and may eventually contaminate the system, compromising the system’s vacuum capability. This is one reason why the o-ring on the quick-coupler fitting of the sputter gun flange should be replaced as soon as it is feasible.

5.6 Bake Out

A thorough bake out of the system over a period of three to four days at temperatures above 250°C speeds up the outgassing rate of surfaces in the vacuum system. This allows a faster and cleaner pump down to vacuum pressures. To achieve UHV pressures, it is a standard practice to bake out the vacuum system.

It is important to be able to control the rate of change of temperature throughout the bake out process and the cool down process of the vacuum system. The temperature should increase or decrease at no more than 20-25°C per hour so that the strain on the components of the vacuum system is minimized.

Our vacuum system is baked out by using heating tapes. The heating tapes are powered by controls that can vary the voltage level of the power supply. The cryopump and the main chamber body have independent bake out systems. (See Figure 32)

Because of the high bake out temperatures, it is important that people are aware when the system is being baked out. A sign and/or notes may be helpful, but not sufficient.

5.6.1 Heating Tapes

There are two types of heating tapes on the system. One is used for the Cryopump and another for the main chamber body.

Cryopump

The heating tape on the cryopump is a continuous tape wrapped around the outside of the cryopump. It should rise to 150°C for the regeneration process of the cryopump. (See Figure 33)

Main Chamber Body

The heating tapes on the main chamber body were custom ordered from Clayborn Labs, Inc. These adhesive tapes were attached to the outer surfaces of the chamber walls. Aluminum adhesive tape and high temperature silicon glue were also used to keep the tapes on the surfaces. As electrical power is sent through the tapes through an electrical power supply from the

wall, the resistance of the tapes allow the tapes to be heated to the desired temperatures. (See Figure 32)

Because of the different geometry of the various areas of the chamber, and the fact that the heating tapes cannot effectively be wrapped continuously on the whole chamber at varying angles and surfaces, the characteristics of the heating tape had to be customized for each port and area. The resistance and power required for each heating tape for each section was calculated so that a maximum temperature of at least 250°C could be achieved. The calculated data for each heating tape for each section of the chamber can be found in Table B.3 in Section B.2.

5.6.2 Power Control

Cryopump

The power to the cryopump heating tape is controlled by the smaller Variac controller which is cylindrically shaped and painted with a blue greyish color. (See Figure 34) This Variac controller should be able to control the voltage across the cryopump heating tape so that temperature changes of 20-25°C per hour, and a maximum temperature of 150°C may be maintained.

Main Chamber Body

The power to the main chamber heating tapes are controlled by the larger Variac controller which is hexagonally shaped. (See Figure 34) This Variac controller should be able to control the voltage across all the heating tapes so that temperature changes of 20-25°C per hour, and at least a bake out temperature of 250°C may be maintained.

The heating tapes on the ports and those on the main body may be separately powered if desired. There are two electrical lines, one for each, coming off of the heating tapes. These two lines go through a plastic casing that is mounted on one of the steel support bars, right next to the gate valve. (See Figure 32)

The fuse inside either the Variac or the transformer behind the Variacs has blown a couple of times. The blown fuses have been replaced, and the transformer has been fixed, however there still may be problems when trying to achieve high bake out temperatures.

5.6.3 Temperature Monitoring

Two K-type thermocouple temperature gauges have been mounted on the chamber walls. The thermocouples measure the temperature of the chamber walls, providing a way of monitoring the bake out temperature.

Aluminum tape and high temperature silicon glue have been used so that the thermocouples are firmly attached to the surface of the chamber walls. This is important because it minimizes the amount of contact with the relatively cool air outside of the chamber walls, hence providing an accurate reading of the temperature of the chamber walls.

A portable thermocouple and reader has been used also. This is useful in monitoring the temperature of the various parts of the vacuum chamber. For example, it can be used to monitor areas where excess heat may damage the components, such as areas where Viton o-rings are used.

5.6.4 Aluminum Foil

Aluminum foil provides insulation when baking out the vacuum system. Aluminum foil should be tightly wrapped around all areas of the vacuum system that require baking out. These sheets of aluminum foil can be reused when baking out.

5.7 Power Supply Racks

There are two racks used for our vacuum system. (See Figure 47) One contains the RF power supply for the sputter system (left rack in Figure 47). The other (right rack in Figure 47), from top to bottom, contains a power switch for the fan in the rack, voltage display for the amount of gas flow through the mass flow controller, heating tape switches and the thermocouple temperature readers, the crystal monitor, evaporator power supply, and the Variac controllers.

5.8 UHV Cleaning

Creating and maintaining a UHV clean environment is essential for achieving UHV vacuum. Dust and dirt must be removed from all components inside the vacuum system. Perhaps it is most important to eliminate any oil inside

the vacuum chamber. Overcoming outgassing from oil in the system can take days or weeks of constant pumping. In most cases oil completely compromises the vacuum system from achieving UHV altogether.

5.8.1 Principles and Precautions

- **If any part of the body comes in contact with any harmful chemicals, immediately wash affected area with flowing water and soap. If irritations arise or persists, contact proper help or authority immediately.**

- Be careful so that prolonged contact with fume, gases, or vapor from the chemicals are avoided. Always provide adequate circulation and venting of air in the room.

- Anything that will be placed inside the vacuum system must be made of UHV safe material (aluminum, stainless steel, or other materials with low outgassing material).

- Anything that will be placed inside the vacuum system must be UHV clean- free from oil, dirt, contaminants, or coated with material that is not UHV safe.

- Two steps should be used at all times when cleaning. First, hexane (or acetone) should be used to remove oil. Second, methanol should be used to remove any remaining residue left by hexane of acetone. Use reagent or purer type to avoid contamination from the cleaning agents themselves.

- Always use clean gloves and clean Kimwipes when cleaning surfaces or handling any parts that have been cleaned. Gloves, after worn, should be cleaned with hexane (or acetone) to eliminate oil (if the gloves are safe for these chemicals, such as the green nitrile gloves or latex gloves, although these chemicals should be used carefully on the latex gloves), and then with methanol using Kimwipes.

- Anything in indirect and/or direct contact with human skin contaminates it from UHV usage, and hence should be cleaned properly. For example, gloves that touch clothes, telephone, tables, or even the hexane, acetone or methanol bottles should be considered as 'contaminated.'

- Be careful when taking off or installing flanges, especially if the bolt holes may have some traces of anti-seize lubricant. Clean the areas on the flange and the mating port that may be considered part of the 'inside' of the vacuum system (usually anywhere inside of the diameter where the knife edges clamp onto the copper gaskets). Usually acetone followed by methanol works well for removing the anti-seize lubrication.

- It is a good practice to place clean parts separately on clean sheets of Kimwipes.

- If storing the clean parts, wrap them in clean sheets of Kimwipes, and then wrap this around with aluminum foil, before storing.

- It is a safe practice to store all clean parts separately, with notes or signs that ensure others know the parts are clean.

- Contaminated gloves should be replaced and/or cleaned before the final cleaning components. For example, when cleaning a metal plate, the plate can be dipped in a container containing hexane, together with the hands with the gloves on. This allows the gloves to be cleaned while cleaning the plate simultaneously. The plate should then be wiped with Kimwipes, and then dipped in methanol together with the gloves. Kimwipes should be used again to wipe off the methanol, at the same time being careful that the gloves are not contaminated until the clean metal plate has been placed on a clean sheet of Kimwipes.

- It is always better to be overly cautious than to be semi-cautious.

- Assume that whatever has not been cleaned by yourself or is not known to be clean, is 'contaminated.'

5.8.2 Procedures

1. First put on disposable latex or vinyl gloves. Be careful not to touch the fingers of the gloves. Touching these areas contaminate them with body oil. Clean the gloves with methanol and Kimwipes. Latex gloves can be cleaned with acetone which will eliminate the oil.

2. When cleaning with hexane or acetone, put on a pair of nitrile gloves or other gloves that can withstand these chemicals, over the disposable gloves.

3. Repeat the following for first hexane (or acetone) and then methanol:

   (a) Clean the gloves with hexane (or acetone) and methanol.

   (b) If possible, use an ultrasonic cleaner when cleaning, as this will vibrate and get much of the dirt from the small areas, holes, and components that are difficult to clean otherwise.

   (c) Soak the parts to be cleaned adequately (5-15 minutes) in the ultrasonic cleaner, or a container with the chemical. Soaking is not necessary, and if desired the parts may just be wiped with the chemical.

   (d) Wipe off the chemical and dirt with clean sheets of Kimwipes.

   (e) Place the clean parts on adequate sheets of Kimwipes.

4. Install, mount, or assemble the clean parts together. Always ensure that clean parts are not contaminated by any contact with 'contaminated' items or gloves. If 'contaminated,' clean the affected areas again as necessary.

5. If parts are not installed immediately after being cleaned, store in clean sheet of Kimwipes, and wrap around with aluminum foil.

6. Dispose of used chemicals properly. BYU Waste Management is vary helpful in the proper disposal of these chemicals.

7. Dispose of gloves and clean the working area.

Table 5.1: Usage of the Vacuum Chamber Ports

Port	**Diameter**	**Usage**
Top of the Chamber (See Figure 18)		
Center port	6”	Sample Stage
2 Angled ports	4.5”	May be used in the future for a mass spectrometer or load-lock system
2 Angled ports	2.75”	Oxygen line, crystal monitor
Offside port	2.75”	Ion gauge
Left, Side of the Chamber (See Figure 19)		
Short, lower port	8”	Evaporator
Left, upper port	8”	Gate valve and cryopump
Right, upper port	8”	Roughing line, sorption Pumps
Angled port	4.5”	Ellipsometer Output Unit (Can be interchanged with the Input Unit if desired)
Right, Side of the Chamber (See Figure 20)		
Long, lower port	8”	Not currently in use
Angled port	4.5”	Ellipsometer Input Unit (Can be interchanged with the Output Unit if desired)
Right, upper port	2.75”	Shutter system for the sample stage
Bottom of the Chamber (See Figure 21)		
Bottom port	8”	Sputter gun

Chapter 6

Deposition

6.1 Additional Chapter Definitions

Breaking Vacuum Reference to opening the vacuum chamber at atmospheric pressure.

Cross-contamination Mixing of two or more different materials, thus compromising the purity of the sample.

Evaporation Boat Casing, plate, or wire used to hold and heat the material source for evaporation. Usually made of tungsten to withstand the high temperature created by the current applied and the resistance of the boat.

Mean Free Path The average distance that gas molecules travel before colliding with other gas molecules. Lower pressures increase the mean free path since there are less gas molecules for collision to occur than that of higher pressures.

mtorr 1×10^{-3} torr. The typical pressure range for sputtering is about 1-100 mtorr.

RF (radio frequency) An oscillating electrical signal much like an ac (alternating current) signal.

Target The source containing the material that deposits onto a film.

Our UHV chamber allows for two different methods of deposition- RF magnetron sputtering and thermal evaporation (physical vapor deposition). These two methods allow flexibility and versatility in the material type and composition of thin films created in the chamber. The deposition pattern for both sputtering and evaporation typically follow the line of sight, i.e. the atoms leaving the source deposit onto whatever surface they 'see' if they had eyes.

6.2 Sputtering

Sputtering refers to the method of deposition that uses ions impacting a surface to create a train of collisions in the target material, thus emitting atoms from the target material into the chamber. It is analogous to a cue ball (ion) breaking up a set of billiard balls (a pool of atoms), scattering the balls in all directions. The efficiency of sputtering is related to the energy transfer that takes place in these collisions.[13] There are two main types of sputtering, categorized by the source of the power supply. These are dc sputtering and RF sputtering which use either a dc (direct current) or an RF (radio-frequency) electrical power source.

6.2.1 Design and Building

The sputtering system we implemented uses a Mighty MAK 4" from US Inc. It is a planar magnetron sputtering system, powered by an RF power supply. This will be described in more detail in Sections 6.2.4, 6.2.5.

The sputter gun is placed inside the chamber from underneath, through an 8" flange. The flange initially used is a conflat flange with a quick-coupler, o-ring sealed fitting. The quick-coupler fitting contains a small Viton o-ring inside which tightens around the shaft of the sputter gun as the quick-coupler nut is tightened. This nut should be no more than hand tightened.

The steel cart that the vacuum chamber sits on was an obstacle when installing the sputter gun. To provide enough space for the sputter gun to be installed, and to allow the shaft of the sputter gun to extend below the bottom flange after it has been installed, a hole of about 8" diameter was cut from the metal plate below the bottom flange. The sputter gun can then be installed through this hole, although some hard work may be required so that the UHV clean sputter gun does not touch external areas.

There are two stainless steel collars, one right below the quick-coupler nut (on the outside of the chamber) and another that is placed against the inside of the 8” flange (on the inside of the chamber). The outer collar (relative to the chamber) was mounted to hold the sputter gun from being sucked in further into the chamber, which would be caused by the pressure differential between atmosphere and vacuum when the system is under vacuum pressure. The inner collar was mounted to hold the sputter gun from falling down because of the force of gravity.

The quick-coupler, o-ring sealed fitting should be a temporary fitting. The reason for using this o-ring seal initially was to first ensure the appropriate, working position of the sputter gun in the chamber before permanently brazing or welding it to the flange, in which case the stainless steel collars may be unnecessary. (See Figure 35)

6.2.2 Plasma

Plasma, or discharge, is the collection of ionized gas composed of ions, electrons, and neutral particles that is electrically neutral overall. The density of the charged particles in a plasma is large enough to promote Coulombic interactions.

A plasma must be created in order for sputtering to begin. Argon is typically used as the medium of the plasma. Argon is a noble gas, so it remains relatively unreactive in the deposition process. This keeps the film deposited by the desired material without any impurities from the plasma source.

The charged particles in the plasma allow for Coulombic interactions to take place among the ions and electrons, creating a fluidlike behavior in the plasma. Typically there are about 1-10 charged particles per 10,000 neutral particles in a plasma. At pressures of 10 mtorr, in room temperature there will be about 3×10^9 ions and electrons in 1 cm^3 volume. The average measured electron energy is about 2 eV, which corresponds to an extremely high temperature of some 23,000 K. However there are relatively few electrons to raise the chamber temperature by much. Neutral atoms and ions in the plasma have energies of 0.025 eV (T = 290 K) and $\sim$0.04 eV (T = 460 K) respectively. Ions have higher energies due to that obtained from the electric field.[13] When the ions are accelerated to the target, their energies increase to several hundred eV, sufficient to break off atoms from the target source.

The creation of plasma is evident from the noticeable sudden tempera-

ture increase of the chamber, which could be seen from the thermocouple connected to our chamber wall. Because the plasma is positioned in vacuum space away from the chamber walls, and the temperature increase is not sustained well enough, the chamber does not remain hot. However, electrical and thermal cautions should be taken when creating a plasma. The bright light that is emitted from the plasma source is another evidence of plasma that is easy to see. In our chamber with an argon plasma source, we were able to see noticeably bright, purplish light that was emitted through the windows of the in situ ellipsometer ports.

6.2.3 Procedures and Principles of Operation

Each step in operating a sputter gun can be broken down in their respective order to target preparation, gas medium backfilling, plasma formation, and sputter deposition.

Target Preparation

First, a target plate is placed on the sputter gun parallel to the film substrate. The target must have a smooth contact with the sputter gun, a contact that is both electrically and thermally conductive.

Thermal conduction is required to allow for appropriate cooling, which is provided by the sputter gun water cooling lines. Thermal conductivity can be provided by applying a very thin film of vacuum safe paste between the target and the sputter gun block. (US Inc. provides Thermal Contact Paste which is essentially a type of vacuum grease.)[14]

We did not use any paste with the ruthenium target that was sputtered. (Ruthenium measurements will be discussed in Chapter 8) This was to avoid any possible outgassing of the paste that may compromise UHV vacuum. Instead, we soldered the ruthenium target with high purity (99.97%) indium onto a copper backing plate provided by US Inc. This copper plate was then coated with a thin indium foil, which provided both the electrical and thermal contact between the sputter gun block and the target. Indium was chosen because of its low outgassing, ideal for vacuum systems, its low melting point ($\sim 250^{o}$C), and malleability. It was soldered so that the ruthenium target could later be taken off of the plate for use in other sputter systems. (Refer to Figure 38)

Gas Medium (Argon) Backfilling

The chamber should be backfilled with the gas medium for the plasma to be created, usually argon. There should be enough of this gas medium in the chamber to provide the discharge. At the same time the gas should be in constant flow. To accomplish this, the gate valve for the pump should be slightly open, usually about $\frac{1}{4}$ *open.* The recommended operating pressure for Mighty MAK, is 0.6 - 600 mtorr of argon, while the most efficient operation occurs between 2 - 15 mtorr.[14] The argon pressure should be raised high first to create the plasma. Once the plasma is created, the argon pressure can be lowered.

Plasma Formation

A DC sputter gun, powered by DC power, produces a plasma through a large voltage difference. An external potential difference is applied from the sputter power source. In our case the power source is RF. (The RF power source is itself difficult to manage. It's operation is discussed in detail in Appendix A.) The maximum power that can be handled by the Mighty MAK 4" is DC power of 3000 watts, or RF power of 1200 watts.

In DC sputtering, the target is charged to a high negative voltage (3 to 5 kV)[12] while grounding the outer parts of the sputter gun. In RF sputtering, the target is kept at a negative potential most of the time, and at periodic intervals it becomes positive for a very brief time. The argon is ionized and plasma is created above the target. The collision of argon ions from the plasma begins the deposition process.

Sputter Deposition

(Refer to Figure 36)
The ions created from the plasma source collide with the target material on the sputter gun to initiate and maintain the sputter deposition process. These collisions transfer kinetic energy to the atoms from the target material, which break the bonds from the target source and are emitted into the vacuum chamber. These atoms from the target which are mostly neutral, scatter in all directions away from the target. Many of these atoms will deposit onto the substrate, creating a thin film. A typical sputter deposition rate is $\sim 3 \times 10^{16}$ atoms/cm^2/sec.[13]

6.2.4 RF Sputtering

When sputtering, insulating materials tend to accumulate positive ions when kept at a negative voltage. This saturates the target so that the target becomes electrically neutral as a whole, eliminating any sputter deposition.

RF sputtering was invented to deposit insulating materials. DC sputtering of an insulating material would require an impossibly large voltage, in the order of 10^{12}V, to operate.[13] RF sputtering uses an oscillating radio frequency signal similar to an ac signal. The oscillating signal allows the target to remain at negative potential for most of the time, while switching to positive potential for relatively brief periods of time. This brief switch allows the insulating material to remove positive ions that accumulated on it. When the target potential becomes negative again, the ions can then bombard the target, allowing the sputter deposition process to continue.

Two effects of RF sputtering are notable. First, ionizing collisions due to oscillating electrons reduce the need for additional electrons to sustain the plasma. Second, RF voltage can couple through any kind of impedance, which makes sputtering of material possible regardless of its resistivity. Typical RF frequencies used range from 5 to 30 MHz. Because the Federal Communications Commission has reserved 13.56 MHz for plasma formation, this is the frequency most widely used.[13]

6.2.5 Magnetron Sputtering

Magnetron sputtering uses electric and magnetic fields to enhance sputtering. It is the most widely used commercial method of sputtering. The magnetic fields prolong the life of the electrons in the plasma. This enhances the probability of ion collisions, leads to a larger discharge current, and increases the sputter deposition rate.[13] More importantly it allows sputtering to be possible at much lower pressure (1-3 mtorr) than that of a non-magnetron sputter gun.

Sputtering at low pressures produce dense and tightly bound thin films. The mean free path is inversely proportional to pressure. So at low pressures (1-3 mtorr), sputtered atoms can travel a relatively large distance ($\sim$ 1-10 meters) without colliding with other atoms or gas molecules. This allows the sputtered atoms to arrive at the substrate with much of the initial energy they had when they left the target. Because of the high energy with which the sputtered atoms arrive at the substrate, the film produced tend to be

dense and tightly bound.

In magnetron sputtering, magnetic fields are created by magnets placed behind the target, with their south poles toward the center of the sputter gun. This creates a magnetic field pointing toward the center, and parallel to the target plate. (Refer to Figure 37) Because the cathode, which is the target material, is kept at negative voltage, the electric field points down, and into the target. Electrons that are created from the cathode begin to accelerate toward the anode, and away from the target source. As they accelerate, the presence of the magnetic field causes the electrons to return back to the target in a helical path. This is due to the Lorentz Force on the electron:

$$F_{Lorentz} = -q(\mathbf{E} + \mathbf{v} \times \mathbf{B}) \tag{6.1}$$

where q, $\mathbf{v}$, $\mathbf{E}$, and $\mathbf{B}$ are the electron charge, velocity, the electric field, and the magnetic field respectively. Thus the electrons 'hop' along the surface of the target, rotating along this track. This track of the electrons is where most of the sputtering occurs, since the ionization of the gas is most intense there.[13] This is evident by the circular ring that forms on the target when we have sputtered. (Refer to Figure 38)

6.2.6 Advantages

- Sputtering allows for the composition of the target material to be mostly preserved in the deposition process of films. The ability to deposit metal alloy films with the same composition as the target is one of the main reasons for the wide use of sputtering. This is an advantage over evaporation, in which a composite material breaks into its individual component materials in separate stages of evaporation.[13] This property of evaporation is analogous to water and alcohol in liquor boiling out at different temperatures.

- High kinetic energy of sputtered atoms (2 - 30 eV)[13] allows for fast deposition. Sputter deposition rates are improved with an RF sputter system because of increased density of the plasma.[12]

- A sputter gun allows for good control over deposition initiation and completion. When a plasma is created or destroyed, sputtering begins and ends almost immediately without any lingering deposition that is typical in evaporation.

6.3 Evaporation

Evaporation is much more simple in its operating principle than a sputter system. The basic idea is that current is sent through an evaporation boat whose resistance coupled with the current produces extreme heat. When appropriate temperature and vapor pressure have been reached, the deposition begins.

The first known evaporations were conducted by Faraday who exploded metal wires in vacuum. His attempt led to the discovery of thermal evaporation in the late 1880's. These techniques were used to study properties of thin films for many years since then. However, most of the applications of this technique arose in the 1940's and 1950's.[12]

6.3.1 Design and Building

A three-boat evaporator using four medium current electrical feedthroughs from MDC Vacuum Products Corporation was used. We custom built four high purity copper extensions and connections to attach three evaporation boats. We built the evaporator to handle up to three separate evaporations in a single vacuum session (without breaking vacuum), two from each of the two alumina coated boats and one from the tungsten boat. These evaporation boats were obtained from R.D. Mathis Company. However, any boats drawing less than 150 amps may be used, the current limitation being the limitation of the current feedthroughs. The bottom copper wire is the common return line of the current while the other three copper wires may be turned on one at a time, not simultaneously. This system provides thin film deposition of up to three multilayers. (See Figures 39, 40)

Three alumina ceramic shields and two aluminum shields, mounted on an aluminum plate, are used to protect cross-contamination from the three evaporation sources, if more than one boat were to be used together in the same vacuum session. Alumina was used to prevent any shorting of the electrical circuit in case contact is made with any of the three possible 'hot' copper wires. The two aluminum shields on the side are to protect the chamber walls, and the bottom aluminum mount to protect the sputter gun below. The two aluminum plates on the side should have enough distance to avoid contact with any of the copper wires. The top of the shields are approximately 1.5-2.0" above the evaporation boats.

To provide additional support for the copper extensions and the alu-

minum and alumina shields, a small alumina piece shaped as a keyhole is mounted on the bottom copper extension. The alumina support lies between the back alumina shield and the 2.75" flange. (See Figure 39) This support lies on the floor of the 8" port that the evaporator goes in.

Alumina is a very hard material, ranked right below diamond. It may be brittle, however, so care should be taken when machining alumina. A high-pressure water jet, from the Engineering Department, was used to cut the alumina into the desired shapes and design. Although the surfaces that were cut by the water jet were rough and inaccurate, it suited our purposes.

The evaporator is to be mounted on the shorter of the two lower, side, 8" diameter ports. The electrical feedthroughs, built on a 2.75" conflat flange, is mounted on a 8" to 2.75" reducing flange. The approximate distance from the evaporation boats to the sample stage is about 6-8". The center evaporation boat was designed to be directly below the sample stage.

6.3.2 Principles of Operation

Current sent through the evaporation boat will produce heat. This will heat the material to increase its temperature, thus its vapor pressure. Two modes of evaporation can be distinguished in this process of heating. They depend on whether the vapor from the material emanates from the liquid state or solid state of the material.

Most metals require a melt into their liquid phase for evaporation to begin. Usually the element to be evaporated must achieve a vapor pressure greater than 10^{-3} torr. Thus, if the element doesn't achieve this vapor pressure before its melting point then melting is necessary for evaporation. If the element is able to achieve this vapor pressure before its melting point, then effective deposition is possible in its solid phase. Some elements that fit the latter case are Cr, Ti, Mo, Fe, and Si that reach sufficiently high vapor pressures below melting, hence they sublime. A typical evaporation deposition rate is $\sim 1.3 \times 10^{17}$ atoms/cm^2/sec.[13]

6.3.3 Advantages

- Simple operating principle. Only sufficient current and power should be supplied to turn the system on.

- Relatively cheap to obtain evaporation source material, boats, and equipment.

Chapter 7

Deposition Control

Effective deposition requires correct monitoring and control of the deposition rate, amount, and duration. From the point of view of our thickness monitors, oxidation is like deposition. The mass of the film increases and its thickness changes, usually increasing (with rare cases of thickness decreasing). Thus monitoring and control of oxidation is important and the same measurement tools can also be used. In our vacuum system there are about six components that factor into the monitoring and controlling of the deposition and oxidation of thin films. These include the sample stage, shutter, quartz crystal monitor, oxygen line, argon line, and water cooling lines.

In addition to these components, perhaps the most effective monitor in the system is the in situ ellipsometer discussed in Chapter 3. The in situ ellipsometer allows real-time measurement of deposition thickness and oxidation thickness relatively accurately.

7.1 Additional Chapter Definitions

VCR Fitting Fitting that requires the male fitting screwed into the female fitting with a metal gasket/washer in between. Mainly used for gas lines, a VCR fitting is similar to a conflat flange in its sealing method and effectiveness.

7.2 Sample Stage

(See Figures 41, 42)
Vertical and horizontal adjustments are made to hold the substrate/film in the correct position for material to be deposited. The adjustments also provide additional degrees of freedom to align the ellipsometric beam. By the design of the sample stage the width of the film must be less than 2 inches, while the length can be up to 6 inches long due to the flange size of the sample stage port. The width of the film is to fit the 2" movement provided by the shutter in Section 7.3

7.2.1 Installation

(See Figure 43)

The sample stage is mounted from the top facing down. It is directly above the sputter gun and the evaporator. The height and lateral adjustments should first be made accurately outside of the chamber with clean gloves. These adjustments should position the film in the center and 6" from the surface of the flange the whole stage is mounted on. The substrate/film should be placed in the stage with the spring holding it well in place. The long side of the stage and the film should be perpendicular to the linear feedthrough of the shutter.

7.3 Shutter

(See Figure 44)
A shutter is necessary in order to protect the substrate/film from the impurities of the initial stages of sputtering or evaporation. This is because the surface of the target material usually contains oxidation or impurities which are sputtered or evaporated before the pure material. The shutter also allows for the deposition rate to stabilize before actually opening the film for deposition. By stabilizing the actual deposition rate, control over the amount and quality of the deposited film can be established.

We have placed an aluminum plate 0.25" underneath the sample stage. The distance between the sample stage and the shutter should be minimal, yet provide enough room for no accidental bumping of the shutter into the stage. This aluminum plate is attached to another aluminum mount that

provides $\sim$ 0.50" vertical movement and $\sim$ 4" forward/backward movement. This setup is attached to a 2.00" linear motion feedthrough from MDC. This feedthrough provides a 2" sideways movement of the aluminum plate through a bellow that preserves vacuum. 2" was the most cost effective movement range for our shutter system. The 2" length in turn regulates the width of the sample film. An aluminum casing and spring held cap provide protection of the shaft of the linear feedthrough from evaporated or sputtered atoms.

7.3.1 Installation

1. If the shutter has not been installed, then remove the sample stage first.
2. The aluminum mount and shaft casing should then be attached to the linear feedthrough.
3. Insert this set up through the 2.75" port on the side of the chamber and tighten the flange bolts.
4. After the flange has been attached, the long aluminum plate can then be placed on the mount through the sample stage port above the shutter. The plate should then be placed appropriately and the screws tightened.
5. The sample stage can now be installed after any necessary adjustments are made.

This is a very difficult process where care should be given so that UHV safe cleanliness is achieved. All parts should first be cleaned, clean gloves used, and any area where the arm or clothing has touched should be thoroughly cleaned.

7.3.2 Usage

Before pumping the chamber down to vacuum, the aluminum plate of the shutter must be positioned directly below the sample stage. It should adequately cover the sample film and be about 0.25" below the film. At this point, the correct position of the linear feedthrough should be noted by reading the position of the mark on the ruler of the feedthrough. This is generally not at 2.00" mark. After this is completed, and when the chamber is in

vacuum, the following steps are recommended for appropriate usage of the shutter.

1. For each vacuum session, the shutter (the aluminum plate, not the whole shutter system) must first be removed from underneath the sample stage. This is accomplished by pulling the linear feedthrough handle completely away from the chamber.

2. The initial alignment, calibration, and measurements of the substrate/film should be made through the in situ ellipsometer at this stage.

3. Move the shutter back into its position directly underneath the sample stage by using the ruler mark noted before. The shutter should be protecting the sample film before any deposition takes place.

4. When the deposition rate has stabilized, which can be monitored through the quartz crystal monitor, and after some time has passed for all the oxidation and impurities of the target surface to leave, the shutter may be removed from underneath the sample film by pulling the linear feedthrough away from the chamber.

5. After the desired amount has been deposited, the shutter may be placed underneath the sample film to protect the film from any more deposition.

7.4 Quartz Crystal Monitor

(See Figure 45)
It is helpful to monitor the deposition rate and deposition amount in order to control the thickness of each layer and the quality of the film created. A quartz crystal monitor measures both the deposition rate and the film thickness.

A quartz crystal monitor by MaxTek, Inc. was installed. The crystal and electrical feedthrough are mounted on a 2.75" conflat flange. The length of the feedthrough from the inner surface of the flange to the crystal is 7". This was to allow the crystal to be placed 1.5" away from the sample from the flange port that is 65° from the horizontal plane. The feedthrough was vacuum brazed by MaxTek, Inc. Metal water cooling lines allow the crystal monitor to be baked out without overheating the system and to provide a

cool operating temperature. The monitor is placed on the 9" wide rack that seats the evaporator power supply, the thermocouple gauge reader, and the mass flow controller voltage reader.

A shutter was not installed to protect the crystal monitor from the initial deposition process. Maxtek recommends that a shutter be installed for the sensor head which contains the crystal. This is to protect the crystal from small droplets of molten material that may hit the crystal.[15] If the crystal is damaged by such material, the oscillation would cease and the crystal monitor would not function appropriately.

7.4.1 Principle of Operation

The crystal used is a resonating quartz crystal which oscillates at its natural frequency. One of the crystal's surface is exposed to deposition. The mass of the oscillator is one of the factors which influences the natural frequency of the crystal. As material is deposited onto the crystal, the mass of the oscillator (the crystal together with the deposited material) increases. This changes the resonant frequency of the crystal which can be detected. By using the acoustic impedance and density of the known material that is deposited (which should be input in to the monitor before deposition begins), the monitor then calculates the deposition rate and thickness.

Tooling Factor

Careful calibration of the monitor is initially required. The calibration is performed by comparing the apparent thickness of the crystal monitor with the thickness of the film measured by a known and accurate instrument, such as an ellipsometer.[12] This calibration corrects for the position of the crystal, which is at some distance away from the actual film, and any errors in the calculation. From this initial calibration, all future measurements by the crystal monitor will yield the thickness and rate of the deposition at the position of the film, not at the position of the crystal.

The calibration is accomplished by adjusting the Tooling Factor of the monitor. The tooling is entered in percent units. Theoretically, the Tooling Factor depends only on the relative position of the source, the crystal, and the substrate film as follows:[15]

$$\text{Tooling\%} = \left(\frac{\text{Distance from the source to the crystal}}{\text{Distance from the source to the substrate}}\right)^2 \times 100\% \tag{7.1}$$

Equation 7.1 shows that a 100% Tooling Factor implies that the crystal is placed exactly where the substrate film is. However, the tooling factor is not exactly equal to that in Equation 7.1. The Tooling Factor does not correct for position alone, but all other factors contributing to the mis-calculation of the monitor. So the Tooling Factor should be adjusted to calibrate the monitor reading to correspond to an accurate measurement of the film by an independent source. The positive side of this is that the calibration may be done only once, and no Tooling Factor adjustments should be necessary afterwards.

Acoustic Impedance

The shear wave acoustic impedance of the material is required to accurately establish the sensor scale factor when the crystal is heavily loaded by deposition. The unit of the acoustic impedance is entered in 100,000 gm/cm^2/sec. This should be obtained from a handbook of physics or other reliable sources. The shear wave acoustic impedance can be calculated from the shear modulus or the shear wave velocity and the density:

$$\text{Acoustic Impedance} = PC = PG, \tag{7.2}$$

where

$$\begin{aligned} P &= \text{Density (gm/cm}^3) \\ C &= \text{Transverse (shear) wave velocity (cm/sec)} \\ G &= \text{Shear modulus (dynes/cm}^2) \end{aligned} \tag{7.3}$$

If the crystal is not heavily loaded, then using $8.83 \times 100,000$ gm/cm^2/sec can be used to obtain sufficient accuracy.[15]

7.4.2 Usage

A crystal monitor can be used as an additional thickness and oxidation measurement device that is independent of the in situ ellipsometer. Although not quite accurate and reliable by itself, it can give us a relatively accurate range for the thickness of the film layers along with the oxidation thickness. This can contribute to evaluating the results from the ellipsometer by providing what to expect. In fact, by using the thickness obtained from the crystal

monitor restrictions of the model fittings by the ellipsometer can be made to obtain more accurate optical constants or film thicknesses.

- First, the appropriate acoustic impedance, density, and tooling factor should be input, and accurate calibration should have been completed.
- To measure the thickness of each layer, restart before each layer is deposited. Record the value of the crystal monitor thickness. Repeat for each layer.
- To measure the oxidation of the top surface of the film, keep the crystal monitor on after deposition. The thickness may be reset or kept. Record the change in thickness when any oxygen is entered in the chamber. A recommended method is to measure the change of thickness over time as the chamber is brought back to atmospheric pressure. Careful monitoring should take place as soon as the chamber is opened to atmospheric air, or as soon as any oxygen enters the chamber. The initial oxidation may occur too quickly if care is not taken.

After repeated deposition, the life of the crystal will eventually expire. The monitor can show when to replace the crystal. The crystal can be replaced by removing its container at the bottom of the feedthrough inside the chamber.

7.5 Gas Controls- O_2 and Ar

There are two gas lines installed in the system that affects deposition and oxidation. The argon line is used to provide argon for sputter deposition and to repressurize the system; the controlled oxygen leak gives control over the total amount of oxidation possible. (See Figure 46)

7.5.1 O_2 Gas line

Oxidation can occur naturally on a film surface by atmospheric air which contains 21% oxygen by volume. For this to happen, the film in the chamber only needs to be exposed to atmospheric air by opening the chamber to atmospheric air. Another method of oxidation is through a controlled leak valve, which is installed in our system.

The controlled oxygen leak makes it possible to control the amount of oxidation on the surface of the film before breaking vacuum. Also it is possible to create multilayer films with each layer oxidized if desired. This can be accomplished by sending in oxygen into the chamber after each layer of film is created. Thus the oxygen leak provides variety and control over the type of film created in the chamber.

The oxygen goes through a stainless steel 1/4" line. Stainless steel gaskets are used to provide the vacuum-tight seal in the VCR fittings. Two valves control the flow of oxygen through the line. One of the valve is a UHV variable leak valve, by Varian, Inc., and the other is an open/close valve by Nupro Company. Both valves are connected through VCR fittings on a tee fitting. This tee has 2.75" conflat flanges on its ends and is mounted on one of the ports on the top of the chamber body.

Oxygen Line Valves and Flow Control

The Nupro open/close valve (model SS-4H) is placed before the variable leak valve in reference to the flow of the oxygen from the gas tank. It is helium leak tight, meaning molecules as small as helium cannot leak through the seal. However, if the knob is not tightened fully, there has been evidence of vacuum leak through the valve.

If no oxygen is to flow into the chamber, then the Nupro valve should be completely closed. If any oxygen is to flow into the chamber, then this valve should be completely open. The amount of flow is then controlled by the variable leak valve.

The Varian, Inc. variable leak valve controls the amount of oxygen flow into the chamber when the Nupro valve is open. Leak rate adjustments are controlled by the finger knobs on the side of the valve. The seal inside the valve is provided by an optically flat sapphire. The sapphire is controlled by a threaded shaft-and-lever mechanism that has a mechanical advantage of 13,000 to 1. The sapphire compresses a metal gasket inside the valve which results in a permanent change in the gasket. However, the change is very small, and when the knob is returned to its previous position the leak rate remains approximately the same.

To open the valve from a closed, leak-tight condition, and provide a leak, the finger knobs must be turned a minimum of two full turns counterclockwise. By turning the knobs (together) clockwise to the stop position against the handle, the valve is closed. Any state in between would provide a pro-

portional leak.

Some of the specifications of the variable leak valve are provided in Table 7.1.[16]

Table 7.1: Variable Leak Valve Specifications

Minimum leak rate	1×10^{-9} to 1×10^{-10} torr-litres/sec
Rate of change of leak	Rate of change increases as the size of the leak increases, giving precise control in proportion to the size of the leak
Vacuum range	Atmospheric pressure to below 1×10^{-11} torr
Temperature range	Up to 450°C in either open or closed position
Inlet gas pressure	500 psi maximum
Gasket life	$\sim$ 300 closures for unbaked systems; 20-30 closures for baked systems. Gaskets are replaceable

7.5.2 Ar Gas Line

Argon is an abundant inert gas which is ideal for vacuum systems since it does not chemically react with other molecules. Argon is used for two reasons.

1. Argon is used as the gas medium for creating a plasma, whose ions provide sputtering of a target material onto the films substrate.

2. Because argon does not react with the components and materials in the chamber, it is well suited for venting the chamber back to atmospheric pressure. Argon is much easier for a vacuum pump to pump out than other gases in atmospheric air. Typically hydrogen, helium, and oxygen from air become difficult to pump out when adsorbed and absorbed by the chamber walls or devices in the chamber.

   By filling the vacuum chamber with argon first, the chamber walls and components will first soak in argon. *Then* when the chamber is opened to atmospheric air, the argon will prevent much of the gases from air to adsorb and absorb into these parts. Thus pumping the chamber down

into low vacuum becomes much more efficient after venting with argon than without.

The argon goes through a stainless steel 1/4" line. Stainless steel gaskets are used to provide the vacuum-tight seal in the VCR fittings. The mass flow controller is used to control the amount of flow of the argon. There are two open/close valves, one for the bypass line and another that provides the seal between the argon line and the chamber.

Argon Line Valves and Flow Control

A model FC-260 mass flow controller from Tylan Corp., with a maximum flow of 150 psig, is used. The purpose of the mass flow controller is to control the bulk flow of argon into the chamber when creating a plasma. The mass flow controller is connected to a Simpson Electric voltage display that is mounted on one of the racks. (See Figure 47) There is also a knob next to the display that controls the opening within the mass flow controller. This controls the amount of argon flow through the mass flow controller.

With the gate valve 3/4 way closed against the cryopump, the flow of argon can be controlled well enough for the chamber base pressure to remain in the 10^{-4} torr range. Of course when creating a plasma, the base pressure can also be raised to the $10^{-3} - 10^{-1}$ torr range necessary and also lowered back. (Refer to Section 6.2 for details)

The Nupro Company open/close valve (model SS-4H) is placed before the chamber in reference to the flow of the argon from the gas tank. It is the final valve before any argon goes directly into the chamber. It is helium leak tight, meaning molecules as small as helium cannot leak through the seal. However, if the knob is not tightened fully, there has been evidence of vacuum leak through the valve.

The Nupro valve should be either completely open or completely closed. If no argon is to flow into the chamber, then this valve should be completely closed. If any oxygen is to flow into the chamber, then this valve should be completely open. If the mass flow controller does not function well, then the Nupro valve may be used to provide some control over the flow rate of argon into the chamber.

When venting the chamber with argon, in order to open the chamber back to atmospheric pressure, the bypass line should be used. The bypass line is controlled by an open/close valve made by Veriflo Corp. Because the Nupro valve is helium leak tight, the bypass valve does not have to be quite

as vacuum tight, although if it is it would be beneficial for the purity of argon flowing through the line.

The mass flow controller does not allow argon to flow freely, so it takes days or sometimes weeks to fill the chamber with argon at atmospheric pressure. The bypass line allows the argon to bypass the mass flow controller and directly enter the chamber, as long as the Nupro valve is opened.

7.6 Temperature Control- Cooling Lines

The RF power source for the sputter gun, the crystal monitor, and the sputter gun need to be cooled. The RF power supply needs to be cooled whenever in operation due to heat generated inside the source. The sputter gun and the crystal monitor require cooling especially when the system is at temperatures above 100-150°C.

It is recommended that water cooling of the sensor head be provided all the time, except during short depositions at low temperatures. The sensor head containing the crystal should not be operated above 100°C. Sufficient cooling can be provided by $\sim$ 0.2 gallons per minute water flow, in an environment up to 300°C.[15] Thus, when baking out the chamber, the sensor head and crystal should be appropriately cooled.

The sputter gun should also be cooled. For the Mighty MAK 4.0", a minimum water flow of 0.75 gallons per minute should be provided. The Maximum inlet temperature should not exceed 70°F/21°C.[14] The cooling of the magnetron sputter gun is mainly to protect the magnets since at high temperatures magnets lose their ferromagnetic properties.

Cooling is provided by the water lines that connect the RF power supply, the sputter gun, and the crystal monitor. Distilled water from water cooling pipes in the Physics Underground Laboratory is used. (Refer to Figure 48)

7.6.1 Operation

1. First, the valves from the pipe source on the ceiling should be opened for both the in-line and out-line. (See Figure 49)

2. Both control valves on the wall for the in-line and out-line should be opened. When opened, the yellow valve is aligned vertically and the bottom black valve is aligned horizontally. (See Figure 50)

3. It may be difficult, yet it is possible to know if water is flowing by paying careful attention to the slight movement of the lines and the small noise produced when the valves are opened. (See Figure 48)

4. Check to see if there are any water leaks from the lines.

Chapter 8

Measurement and Analysis

8.1 Additional Chapter Definitions

Mean Square Error (MSE) The average sum of the square of the errors. MSE is generated by WVASE when fitting measured data to a particular model. MSE shows how well the model fits the measured, experimental data. Generally MSE less than 1.0 in WVASE implies a good fit. Higher MSE may demonstrate that the model (optical constants, thicknesses, etc.) may be incorrect.

8.2 Background

Dr. Allred's XUV Research Group in Brigham Young University has been interested in understanding optical constants of ruthenium, ruthenium oxide (thought to be RuO_2), the oxidation rate and amount of oxidation for ruthenium thin films. I have been able to study and improve the optical constants of ruthenium, ruthenium oxide with the help of Cort Johnson, Michael Newey, and others in Dr. Allred's XUV Research Group. With the UHV thin film deposition chamber, I have been able to analyze ruthenium thin films in more depth, and at the same time understand the initial oxidation of ruthenium thin films from high vacuum pressure up to atmospheric pressure.

After a successful high vacuum pressure of 1.5×10^{-7} torr (according to the ion gauge connected to the senTorr gauge) was achieved on April 12, 2001, several attempts were made to create a plasma above the sputter gun.

On May 3, 2001, a ruthenium thin film was successfully sputtered from a base pressure of 1.0×10^{-6} torr (pressure had steadily risen in the vacuum system during this time period).

The sputtered ruthenium thin film was then analyzed with the in situ ellipsometer (M-44® spectroscopic ellipsometer with UV option from J.A. Woollam Co., Inc. together with the WVASE^{TM} software) through many dynamic real time scans and single spectroscopic scans. Ellipsometric analysis was then performed to improve the known optical constants for ruthenium, and measure the oxidation rate and amount of the ruthenium thin film.

8.3 Sputtered Ruthenium Thin Film - History

May 3, 2001 Ruthenium was deposited onto the prepared substrate using the Mighty MAK 4" sputter gun. Base vacuum pressure inside the chamber was 1×10^{-6} torr.

Before sputtering, the in situ ellipsometer was aligned and calibrated with the silicon substrate on the sample stage. The silicon oxide layer on the substrate was measured to be about 31.6Å. with a mean square error (MSE) of 0.9064. The incident angle was also fitted by WVASE to be about 69.25^o.

Sputtering started at 1:18pm with a deposition rate of about 1Å per second until 1:24pm. The crystal monitor read 350Å total thickness. It should be noted that the crystal monitor was used for the first time, so the tooling factor could not be calibrated properly and the ruthenium acoustic impedance could not be found. The tooling factor was set to 100%, the acoustic impedance set to $8.83 \times 100,000$ gm/cm^2/sec, and the density was correctly set to about 12.45 gm/cm^3. Hence the readings of the crystal monitor could not be taken to be correct until further calibrations and corrections were made.

Real time continuous ellipsometric measurements were made during the sputtering process, however the data was lost due to computer failure. The last read data and fit, using optical constants obtained earlier (in Tables 8.1, 8.2), fitted the thickness of the ruthenium layer as 243.4Å.

Beginning at 2:55pm on May 3, real time continuous ellipsometric mea-

surements were again made for each 1-2 minute periods for about 7+ hours.

May 10, 2001 Crystal monitor read 348Å, chamber pressure read 2.810^{-6} torr. The chamber was prepared to be open to air by first using argon to backfill it to atmospheric pressure. At 12:01pm, gate valve was closed and the pressure rose to 2.7×10^{-5} torr in about 15 seconds. Beginning at 12:04pm the argon line was open to the chamber, and argon flowed through the mass flow controller (the bypass line for argon had not yet been installed). At 12:45pm, the chamber pressure rose to 1.1 torr and the crystal monitor read 350Å.

May 11, 2001 At 4:00pm, the crystal monitor read 356Å, indication some oxidation of the ruthenium film that was deposited on the crystal. At 4:38pm, chamber pressure read 2.2 torr.

May 15, 2001 At 3:05pm, chamber pressure read 9.9 torr, and the crystal monitor read 356Å.

May 17, 2001 At 3:40pm, chamber pressure read 7.2×10^1 torr, and the crystal monitor read 355Å.

May 18, 2001 At 4:35pm, chamber pressure read 1.5×10^2 torr, and the crystal monitor read 355Å.

May 23, 2001 At 4:00pm, chamber pressure read 3.6×10^2 torr, and the crystal monitor read 359Å. After opening the chamber on a future date (May 29), it was apparent that 3.6×10^2 torr was atmospheric pressure, as the thermocouple gauge was not reading the pressure correctly in this range.

May 24, 2001 At 3:30pm, chamber pressure read 3.6×10^2 torr, and the crystal monitor read 360Å.

May 25, 2001 Chamber pressure read 3.8×10^2 torr, and the crystal monitor read 360Å.

May 29, 2001 Chamber pressure read 3.6×10^2 torr, and the crystal monitor read 361Å. The argon bypass line was installed on this day. Up to this day, single spectroscopic scans were made of the film, once a day for about the past week.

The chamber was flooded with argon through the argon bypass line. The thermocouple reading decreased to about 2.1×10^2 torr. It had been inside the chamber reading the pressure incorrectly. The flange on the left side of the chamber, above the senTorr gauge, was opened and air at atmospheric pressure was introduced into the chamber.

Real time continuous ellipsometric measurements were made from the time of opening the chamber to air for about 30 minutes every 1-2 seconds. Real time continuous ellipsometric measurements were again made from an hour after opening the chamber to air until the following day, every 1-2 minutes.

May 30, 2001 At 6:30pm, the open chamber pressure read 3.9×10^2 torr, and the crystal monitor read 357Å.

Another spectroscopic measurement was made of the ruthenium film. In situ measurements of the film up to this day were analyzed with WVASE by Michael Newey and I. After comparing several in situ data and by fitting for the ruthenium thickness, using optical constants derived from literature (in Tables 8.1, 8.2), the thickness of the ruthenium layer alone averaged out to be about 290Å while the apparent thickness of ruthenium oxide ranged from 0-67Å with an MSE around 14. The errors and the thickness of the ruthenium oxide were so high that we decided to determine our own optical constants for ruthenium, in order to obtain a more realistic model than what we had before. (See Table 8.3)

May 31, 2001 The ruthenium film was taken out of the sample stage. The surface of the film showed visible blemishes. After showing it to Dr. Allred several days later, he stated that delamination or lift off of the surface had occurred. This was due to compressional stress. (See Figure 51) More water vapor was introduced by blowing on the film, which caused the top layer of the film to be lifted up by too many bubbles. The film may have been ruined.

8.4 Ruthenium and Ruthenium Oxide Optical Constants

8.4.1 Initial Optical Constants

During the summer of 2000, Cort Johnson had created some ruthenium thin films. He and I measured these and other old ruthenium thin films (one to two years after they had been deposited) using the M-44® spectroscopic ellipsometer from J.A. Woollam Co., Inc. Measurements were made with the variable angle option that had been installed previously in April 2000, as part of my research.

Cort Johnson and I had previously adapted the ruthenium[17][18] and ruthenium oxide (RuO_2)[19] optical constants that had been published. Cort extrapolated these published constants over the range that the M-44 ellipsometer covered, with the help of the WVASE program.

These optical constants that Cort Johnson obtained were then improved. Michael Newey and I, using WVASE, attempted to find the best fit based on these optical constants for six different ruthenium sample measurements. The optical constants were fitted by WVASE and were then initially used for the modelling of the newly created in situ ruthenium thin film.

The optical constants initially used when modelling the newly sputtered ruthenium film, which was created on May 3, 2001, are shown in Tables 8.1, 8.2.

8.4.2 Improved Optical Constants

Michael Newey and I did not attempt to recalculate the optical constants for thin ruthenium oxide because the ruthenium oxide layer on the sample was very thin. We did recompute the optical constants for ruthenium thin films based on the in situ ellipsometric measurements made of the ruthenium thin film in high vacuum. We assumed that the thickness of the ruthenium oxide layer on top of the film was negligible. This assumption was safe to take because the measurements were made under the following conditions:

1. Measurements were made within an hour of sputtering.

2. The film was in high vacuum ($\sim 1 \times 10^{-6}$ torr) during the measurement.

3. After the XUV group has analyzed previous ruthenium thin film samples, oxidation of ruthenium has been known to be slow and relatively

thin, even after the film has been exposed to air at atmospheric pressure for a long period of time (months or years).

On May 30, 2001, Michael Newey and I, with this assumption and using the previous in situ measurements, improved the ruthenium optical constants of Table 8.1. The silicon oxide layer was set to 33.8Å with an MSE of 1.054 after fitting for the silicon oxide thickness previously that day. The incident angle was fixed to 69.25^o after trying different angles that ranged from 62-73°. 69.25^o was chosen as it is what WVASE fitted when the uncoated silicon substrate was measured inside the system before sputtering.

In computing optical constants of a metal thin film, knowing the thickness of the film can be important. This can be done iteratively. We took three thicknesses suggested by the measurements and determined the optical constants for all three.

We set the ruthenium oxide layer of the model in WVASE to be nearly 0Å (0.1Å). The previous ruthenium optical constants were adapted to several of our in situ measurements with the thickness of ruthenium fixed to 243Å, 290Å, and 352Å to fit the constants. Only the measurements made on May 3, when the chamber was in high vacuum (in the 1×10^{-6} to 1×10^{-5} torr range) were taken into account.

243Å and 290Å thicknesses were chosen because when the WVASE fitted for the thickness of ruthenium on the sputtered film using the constants in Tables 8.1, 8.2, the fitted values were 243Å and 290Å on May 3 and May 30 respectively. (Refer to Section 8.3). 352Å was used because this was the initial crystal monitor reading for the amount of sputtered ruthenium on the crystal.

After comparing various data that were measured after the deposition, we noticed that the optical constants did not vary much over the range of ruthenium thicknesses we had used. So we fixed the Ru thickness to be 352Å and the RuO_2 thickness to nearly 0Å (as before). We used the measurements made on May 3, which began an hour and a half after sputtering, while still in high vacuum. Out of these measurements, data from 2, 200, and 400 minutes into the measurement was used to fit for the optical constants of ruthenium. These improved optical constants for thin film ruthenium are shown in Table 8.3.

8.5 Ruthenium Oxidation

8.5.1 In High Vacuum

In high vacuum (1×10^{-6} to 1×10^{-5} torr range), there seems to be minimal amount of oxidation, which was one of our assumptions when developing the ruthenium optical constants in Section 8.4.2. After analyzing the measurements made in high vacuum, with WVASE using the initial optical constants in Tables 8.1, 8.2, the ruthenium oxide layer would either remain at 0Å or jump to about 67Å (with an MSE of about 14 or more) when the incident angle was fixed at 69.25^o or allowed to be fitted respectively. The latter is highly unlikely from our understanding that ruthenium film oxidizes very slowly and forms a rather thin oxide layer. It is very likely that 0Å of RuO_2 layer was formed in high vacuum.

The crystal monitor did not show any change greater than 1-2Å even after it remained in high vacuum for a week. (See Section 8.3) Although the crystal monitor may not have been completely accurate or reliable, any oxidation of the ruthenium film on the crystal should affect the crystal to some extent that could be noticed by a change in the crystal monitor reading. Hence, the readings of the crystal monitor also support that very little (0-1Å), if any, oxidation of ruthenium occurred in high vacuum.

8.5.2 During Argon Backfill of Chamber

Both the crystal monitor and the ellipsometric measurements show a change of the film layer. This is due to the small amount of oxygen that may have entered together with the argon, or through outgassing or leaks (though leaks are probably unlikely) in the vacuum system.

The crystal monitor reading changed from 348Å to 360Å over the two week period of filling the chamber with argon from May 10 to May 24. During this period, the chamber pressure rose from 2.8×10^{-6} torr to atmospheric pressure. Air at atmospheric pressure was *not* introduced at this stage.

The ellipsometric data was not quite conclusive, however. Using the improved Ru optical constants, the ruthenium oxide thickness after about 30 minutes of backfilling the chamber with argon came out to be about 0.7Å. In fact the oxide layer seem to decrease from 1.6Å to 0.7Å over this initial 30 minute period of argon backfilling. The ruthenium layer remained at about 312Å. The average MSE was 1.8. (See Figures 52, 53)

8.5.3 In Air At Atmospheric Pressure

The vacuum system was open to air at atmospheric pressure on May 29, about three and a half weeks after sputtering the ruthenium film. Two stages of the oxide development were measured initially- the first 20-30 minutes and the next 24 hours after the first hour.

The crystal monitor increased about 1Å initially from 360Å, however decreased to 357Å the next day on May 30. The flux of gases inside the chamber after opening it to air may have caused the slight drop in value. The crystal monitor reading has been mostly disregarded in analyzing the oxidation of ruthenium in air at atmospheric pressure. Instead the ellipsometer has been used more extensively to understand the initial oxidation of the ruthenium thin film in air.

8.5.4 First 20-30 Minutes of Exposure

When the chamber was open to atmosphere, real-time ellipsometric measurements were made every 1-2 seconds for approximately 20-30 minutes. The fitted Ru and RuO_2 layers were 280Å and 3.2Å thick respectively. There were slightly noticeable changes (slope of Psi data changed) in the ellipsometric measurements, over the first 20-30 minutes of exposure. (See Figure 54) This demonstrates the high probability of some initial oxidation in air at atmospheric pressure.

Fittings for Ru and RuO_2 thicknesses fluctuated over this period of time. (See Figures 55, 56) The spike/dip in the figures may have been caused by the flux of gases present after the chamber was open to atmosphere.

8.5.5 First 24 Hours of Exposure

After about an hour had passed since opening the chamber to atmosphere, real-time ellipsometric measurements were made every 1-2 minutes for approximately 24 hours, until the following day on May 30. The fitted Ru and RuO_2 layers were 280.7Å and 3.1Å thick respectively. The average MSE was about 6.8. There seem to have been little change in the ellipsometric measurements, over this 24 hour period of exposure. (See Figure 57)

Fittings for Ru and RuO_2 thicknesses fluctuated over this period of time also. (See Figures 58, 59)

8.6 Aftermath

After additional water vapor was introduce on May 31, 2001, and about a week after it was taken out of the vacuum system, the film was remeasured. Michael Newey obtained measurements at five different angles (60°, 65°, 70°, 75°, 80°) using a better version of J.A. Woollam's ellipsometer in the Chemistry Department than the M-44 ellipsometer in our UHV system.

This data has been analyzed by WVASE on June 8, 2001. The fitting resulted with a ruthenium layer that is about 313.3Å thick, a ruthenium oxide layer that is about 24.6Å thick, with an MSE of 31.35. Dr. Allred and I believe that the MSE is too high. The surface of the ruthenium film was damaged too much for the ellipsometer to be able to make correct measurements. This would be the cause of these results.

Future improvements to obtain better Ru and RuO_2 optical constants could have been made. Further data obtained through measuring the thicknesses of each layer of the film by X-ray diffraction could have also provided useful and relevant data in improving the optical constants for Ru and RuO_2. A new ruthenium film should be deposited and analyzed in the future.

8.7 Ruthenium Thin Film Conclusions

From the in situ analysis of a sputtered ruthenium thin film of about 280Å ruthenium thick layer, it appears that there is some noticeable oxidation. The RuO_2 layer seems to form even before the vacuum chamber is exposed to air at atmospheric pressure, while the chamber is being backfilled with argon. The ultimate RuO_2 thickness appears to stabilize to about 3.1Å after the initial 24 hours of exposure of the 280Å ruthenium layer to air. This is a very small amount, in the order of 1-2 atomic layers of RuO_2.

It appears that the known optical constants of both Ru and RuO_2 can be substantially improved to better fit the ruthenium thin film model when using ellipsometry to determine the thin film properties and thicknesses. The improved Ru optical constants that we were able to develop seem to work relatively better than the initial published Ru optical constants. The initial constants would not allow WVASE to give a reasonable RuO_2 thickness, whereas the improved constants provided a fit for a 3.1Å thick RuO_2 layer.

Table 8.1: Ruthenium optical constants N and K - Fitted using six different Ru thin film measurements; Based on published optical constants

Wavelength (Angstrom)	**N (real part)**	**K (imaginary part)**
2861	1.617622	3.596361
2931	1.636191	3.686386
3001	1.673544	3.756396
3070	1.746131	3.816332
3141	1.825034	3.889203
3212	1.89865	3.946771
3284	1.985777	4.013282
3358	2.053746	4.074445
3432	2.132705	4.139677
3506	2.200425	4.206789
3581	2.297176	4.276969
3654	2.402717	4.293381
3726	2.45048	4.311091
3794	2.551048	4.383743
3886	2.698917	4.427622
3957	2.782718	4.447131
4033	2.891218	4.4345
4110	2.9555	4.446743
4185	3.024968	4.453993
4259	3.089138	4.456133
4337	3.159788	4.465329
4412	3.218202	4.471216
4491	3.277843	4.486873
4567	3.334018	4.495767
4649	3.368167	4.522273
4718	3.410582	4.536077
4799	3.461029	4.548926
4872	3.504962	4.562753
4946	3.551499	4.577219
5024	3.588483	4.606395
5100	3.633274	4.631534
5177	3.681743	4.650655
5251	3.729378	4.669709
5327	3.775189	4.698285
5401	3.828698	4.712176
5473	3.873171	4.735036
5549	3.930596	4.749538
5624	3.977997	4.773323
5697	4.033931	4.784562
5770	4.095135	4.793333
5840	4.151261	4.794891
5911	4.196892	4.805849
5981	4.257076	4.799964
6052	4.301175	4.808517

Table 8.2: Ruthenium oxide (RuO_2) optical constants N and K - Fitted using six different Ru thin film measurements; Based on published optical constants

Wavelength (Angstrom)	**N (real part)**	**K (imaginary part)**
2861	1.222544	1.002123
2931	1.600873	1.002353
3001	1.384585	0.9168893
3070	1.382675	0.9621751
3141	1.420025	0.8894264
3212	1.430229	0.8805887
3284	1.439517	0.8756795
3358	1.444712	0.8753325
3432	1.445715	0.8812263
3506	1.452538	0.9151114
3581	1.450349	0.9285933
3654	1.438749	0.9561812
3726	1.446778	1.008696
3794	1.49304	1.044372
3886	1.547467	1.049235
3957	1.574388	1.055827
4033	1.598108	1.040644
4110	1.614351	1.042523
4185	1.632137	1.033783
4259	1.646106	1.030018
4337	1.659933	1.026506
4412	1.671002	1.022292
4491	1.684256	1.01999
4567	1.693332	1.013962
4649	1.700616	1.014594
4718	1.70845	1.006428
4799	1.71745	0.9998324
4872	1.72465	0.9885945
4946	1.730432	0.97796
5024	1.734762	0.9668088
5100	1.736629	0.9564642
5177	1.738297	0.9384346
5251	1.737419	0.9236766
5327	1.730304	0.9126321
5401	1.727696	0.8852185
5473	1.715055	0.864433
5549	1.701118	0.8476734
5624	1.682353	0.8320531
5697	1.655688	0.8081419
5770	1.623477	0.8010651
5840	1.591355	0.7827886
5911	1.55581	0.7855957
5981	1.517066	0.7784875
6052	1.479561	0.7887052

Table 8.3: Improved ruthenium optical constants N and K - Fitted using 3 different Ru thin film in situ ellipsometric measurements in high vacuum

Wavelength (Angstrom)	**N (real part)**	**K (imaginary part)**
2861	1.414168	3.909593
2931	1.452633	4.025786
3001	1.488577	4.086054
3070	1.543297	4.180354
3141	1.611849	4.315892
3212	1.676743	4.391868
3284	1.748177	4.48859
3358	1.832845	4.592326
3432	1.904305	4.66412
3506	1.997548	4.754883
3581	2.07482	4.831154
3654	2.169967	4.908803
3726	2.256361	4.991684
3794	2.334881	5.035164
3886	2.443673	5.111683
3957	2.561181	5.200842
4033	2.663018	5.25141
4110	2.772643	5.297512
4185	2.878374	5.333275
4259	2.980662	5.359329
4337	3.068179	5.36844
4412	3.158358	5.388476
4491	3.210132	5.388798
4567	3.279537	5.40675
4649	3.341914	5.42078
4718	3.385379	5.461017
4799	3.430885	5.492202
4872	3.484785	5.537263
4946	3.529718	5.578005
5024	3.591648	5.611994
5100	3.651888	5.643077
5177	3.71899	5.70043
5251	3.776683	5.729577
5327	3.851703	5.757141
5401	3.911889	5.83272
5473	3.991945	5.838867
5549	4.070646	5.851487
5624	4.153063	5.903648
5697	4.230457	5.938151
5770	4.326889	5.978906
5840	4.377103	5.975037
5911	4.480794	5.973563
5981	4.542005	5.968662
6052	4.669601	5.985336

Chapter 9

Current Status and Future Enhancements

(As of June 21, 2001)

9.1 Current Status

9.1.1 Vacuum Results

The first vacuum session of our UHV system was conducted from March through April, 2001. The lowest pressure that the senTorr gauge controller read was 1.5×10^{-7} torr on April 12, after about two days with the cryopump pumping on the chamber.

The chamber had been baked out previous to turning the cryopump on for 3-4 days at $250 - 270^{o}$C. During this bake out, the sorption pumps pumped out the chamber 2-3 times. On the third day of bake out the power source for the heating tapes blew out, due to a failure of a fuse. Greg Harris and Scott Daniel fixed the problem, although the fix may not allow the heating tapes to reach 250°C (as we have yet to test). With the fix, the chamber was again baked out to 150°C, so as not to blow the fuse again, for an extra day. During this time the chamber was also pumped out by the sorption pumps again.

By pumping out the chamber and cryopump several times each, the cryopump was able to achieve high vacuum pressures. High vacuum of 1.5×10^{-7} torr was achieved on April 12. After a week, the base pressure of the chamber

rose to 4.0×10^{-7} torr. By the end of April, the base pressure had risen to 1.0×10^{-6} torr. The chamber pressure then stabilized in the low 10^{-6} torr range until argon was introduced on May 10 to bring the chamber back to atmospheric pressure.

From this rise of pressure, it could be thought that there may be a small high vacuum leak. It is possible that the outgassing of the material inside the chamber may be the sole cause of the rise in pressure also. Although the lowest base pressure was not quite UHV, with HV pressure in the low 10^{-6} torr range, high purity thin films can still be created and analyzed.

9.1.2 Deposition Results

Sputtering

A ruthenium thin film of about 280Å was sputtered using the Mighty MAK 4" sputter gun. Plasma formation and the sputtering was successful after several attempts. Delamination of the film occurred which shows that stress on the sputtered film must be reduced.

Evaporation

The evaporator has been installed. Several boats, including two alumina boats, several tungsten boats, and a chromium boat have been obtained. One of each of the three boats, with the chromium boat in the middle, have been mounted on the three copper extensions of the electrical feedthrough. Three alumina shields have been mounted on the evaporator. The two aluminum side shields have not been installed yet, as they had to be bent in order to fit inside the port. The length of the support needs to be fixed in order to fit properly inside the port.

The next vacuum session will be conducted to study an evaporated chromium thin film. The positioning of the shields, support, and the boats will require some adjustments and testing. Also the evaporator power supply and control over the amount of current and power sent through the evaporator feedthroughs will require some attention.

9.2 Future Enhancements

9.2.1 Ultrahigh Vacuum

There may or may not be high vacuum leaks in the current system. After discussing the system with Joseph Young, he thinks that there may *not* be a leak, but that the rise of pressure in the chamber while under vacuum is rather caused by outgassing. The following are suggested improvements that may help the vacuum system to achieve improved vacuum pressure, eventually in the UHV range.

Cleaning of the Inside of the Chamber

The inside of the chamber should be cleaned again. There are pieces of Kimwipes and aluminum foil that accidentally entered the vacuum chamber when it was below atmospheric pressure.

Before the chamber was able to be pumped down to roughing pressure, we had discovered a large leak together with the fact that the gate valve would not seal. The cryopump was then taken off of the system, and the gate valve was analyzed. Joseph Young found out that the gate valve just had to be tightened with a wrench that would provide enough torque.

Once the gate valve had been reinstalled, the sorption pumps were used to see if the chamber could hold roughing pressure. The chamber was able to hold roughing pressure, however we learned that the mass flow controller would not allow argon to flow through it fast enough to bring the chamber to atmospheric pressure. To keep the gate valve clean, while trying to find a way to bring the chamber back to atmospheric pressure, the gate valve was wrapped with Kimwipes and aluminum foil while the chamber remained under vacuum.

Although the chamber pressure was close to atmospheric pressure, it was below atmospheric pressure. One of our builders attempted to find a mechanism for the gate valve so that it could seal better. He did not realize that the chamber was below atmospheric pressure and accidentally opened the chamber, which caused pieces of Kimwipes and aluminum foil to enter into the system. The extent of the problem had not been noticed until after the chamber brought back to atmospheric pressure after our first high vacuum session. Eliminating all the particles may be a very effective means of helping the vacuum system achieve UHV.

Replacing Sputter Gun Flange

The o-ring of the quick-coupler unit in the sputter gun flange may provide extensive outgassing, if not a high vacuum leak. It is positioned at a fair distance away from the cryopump, hence the leak or outgassing rate from the o-ring may exceed the high vacuum pumping speed of the area where the o-ring is placed.

First, the exact permanent position of the target of the sputter gun relative to the bottom flange should be determined. This would depend on the position of the evaporator and the sample stage, together with the ideal distance of the target from the substrate film.

Once the position of the sputter gun is determined, the sputter gun should be vacuum brazed to the bottom flange with the quick-coupler unit. The o-ring can be eliminated because the shaft will be brazed onto the flange. This would provide a UHV leak tight seal.

Improved Bake Out

The heating tapes have been installed for the cryopump and the cryopump was successfully baked out at 150°C on June 7-8, 2001. This should allow the cryopump to pump more effectively.

The main chamber body can also be baked out more effectively than that of the first vacuum session. The bake out should be continuously conducted for 3-4 days, or more, at temperatures above 250°C. Joseph Young recommended a bake out temperature of 300-400°C, as this would speed up the degassing of gases that are difficult to degas at 250°C. While the maximum possible temperature should be achieved during bake out, care should be taken so that all components in the vacuum system can withstand the high bake out temperature by ensuring that adequate cooling is provided if necessary.

In addition to increasing the bake out temperature, adequate pumping of the chamber by the roughing pumps should be maintained throughout the bake out process. During the first vacuum session we achieved, the chamber was not pumped out regularly by the sorption pumps. Pumping the chamber out to roughing pressure once a day may be sufficient in achieving UHV after the bake out process.

Also aluminum foil can be used more extensively than before. The aluminum foil should be wrapped around the chamber walls tightly, and as much

of the chamber surface as possible should be insulated using the aluminum foil. Using a better insulating material than aluminum foil will also increase the bake out effectiveness.

Gate Valve

Joseph Young recommended that the aluminum gate valve currently installed in the system be replaced by a stainless steel gate valve. Apparently the same cryopump installed in our system was able to achieve UHV vacuum with a stainless steel gate valve.

9.2.2 Component Design

There are many improvements that can be made to the current design of our UHV thin film deposition system. Improvements in design and components of the system may allow the system to be more efficient in deposition, analysis, and vacuum pump down.

Sputtered Film

The stress applied to the substrate film when sputtering should be reduced. The compressional stress on a sputtered film was apparent with the delamination of the sputtered ruthenium thin film. The solution may be to extend the distance from the target to the substrate, reduce the energy of the plasma when sputtering, or to modify the sample stage to eliminate some of the stress.

Mass Flow Controller

The current mass flow controller does not seem to effectively control the flow of argon passing through it. The maximum flow of argon through the mass flow controller into the chamber, when attempting to bring the chamber to atmospheric pressure, was very slow compared to typical mass flow controllers. (Although the mass flow controller is not meant to be used to vent a whole chamber with argon, typical mass flow controllers should take no longer than couple days to vent the whole chamber with argon.) Also the argon flow controlled by the knob next to voltage display of the mass flow controller, was somewhat too large and random when backfilling the chamber to create a plasma. The mass flow controller may need to be fixed

or replaced in order to achieve accurate and reliable control over the flow of argon through it.

Mass Spectrometer

A 4" to 2.75" reducing flange has already been purchased. The mass spectrometer, mounted on a 2.75" flange on the sputter chamber on the bottom floor of the Physics Underground Laboratory, may be available for our UHV system. The mass spectrometer could be installed on one of the top 4" flanges of chamber. (Refer to Section 5.2 on page 48) A mass spectrometer will allow analysis of the various gases present in the vacuum system when under vacuum. This could help determine the gases present in the system that pose possible leak or outgassing problems.

Load-lock System

A load-lock system for the sample stage would be effective in reducing the amount of time required to pump the system to UHV, between each vacuum session. Such a load-lock system would allow the isolation of the sample stage from the rest of the chamber. Thus the rest of the chamber could remain under UHV vacuum while the sample stage alone could be brought to atmospheric pressure for the sample film to be replaced or taken out.

A load-lock for the sample stage may be too difficult of a task to implement because of the position of the sample stage in our current design. The sample stage is currently on the top port which is relatively small in its flange size (6") and too crowded with the other flanges surrounding it. Also the sample stage is about 6" in height and 2-4" in width and length, which makes it difficult to be moved through the ports in order for the stage to be isolated. Thus any type of gate valve to take out and isolate the sample stage would be difficult to install. This could be overcome by moving the sample stage to a less crowded, larger port, but then the deposition equipment (sputter gun and the evaporator) would have to be adjusted accordingly. If all these issues could be overcome by some clever design, it may be worthwhile to install a load-lock system.

Chapter 10

Summary

10.1 Ruthenium Thin Film

Ruthenium can be used as a good XUV mirror because of its low oxidation in air. The ellipsometric measurements show that a very small layer of ruthenium oxide ($\sim 3 - 4$Å) may form on a ruthenium thin film after the film's exposure to air in atmospheric pressure.

10.2 System Capabilities

The UHV thin film deposition system that has been designed and built can achieve low high vacuum, down to 1×10^{-7} torr. With improved vacuum techniques and cleaning, UHV pressures below 10^{-9} torr should be achievable. Thin film sputtering and real-time in situ ellipsometric measurements have been made. Future sputtering and in situ measurements should pose little difficulty, with the RF power source operating correctly. Thin film evaporation should also be available once the evaporator has been correctly installed. Oxidation of deposited films can also be controlled through the oxygen line, together with the variable leak valve.

Even after a year of designing, planning, building, and researching on how to build a UHV vacuum chamber, actually achieving UHV is itself a formidable task. However, the full benefit of this system is yet to be realized. This in situ ellipsometry UHV chamber, even with its basic functions, can produce many publishable experiments and data that will improve current understanding of thin film optics, especially controlled thin film oxidation.

Through this research, I have developed extensive knowledge in vacuum systems, experience in leading a small research group, some machining knowledge, and experience in a real research environment that have required much work while producing much satisfaction.

Figure 1: View of the UHV deposition system with in situ ellipsometry

Figure 2: Reflectance of Silicon film versus thickness of SiO_2 layer

Figure 3: Achieving UHV

Figure 4: Conflat flange- sealing

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Figure 7: In situ ellipsometer operation principle

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Figure 23: Gate valve components

Figure 24: Gate valve sealing flange

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Figure 30: ASA type o-ring flange

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Figure 32: Bake out system

Figure 33: Cryopump heating tape

Figure 34: Variac voltage controllers for bake out

Figure 35: Sputter gun - Mighty Mak 4, by US Inc.

Figure 36: Sputter gun mechanism in vacuum chamber

Figure 37: Applied fields of planar magnetron sputtering

Figure 38: Target of the sputter gun

Figure 39: Evaporator with shields and support- Side view

Figure 40: Evaporator with shields and support- Front view

Figure 41: Sample stage

Figure 42: Sample stage inside the chamber

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Figure 44: Shutter

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Figure 49: Water cooling source- pipes in the Physics Underground Laboratory

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Figure 51: View of the sputtered ruthenium thin film (Taken out of the chamber on May 31, 2001)

Figure 52: Sputtered ruthenium film during first 30 minutes of argon back-filling of the chamber- Ru thickness fit (Å)

Figure 53: Sputtered ruthenium film during first 30 minutes of argon back-filling of the chamber- RuO_2 thickness fit (Å)

Figure 54: Sputtered ruthenium film during first 20-30 minutes of exposure to atmospheric air- Ellipsometric measurements and fit of Psi

Figure 55: Sputtered ruthenium film during first 20-30 minutes of exposure to atmospheric air- Ru thickness fit (Å)

Figure 56: Sputtered ruthenium film during first 20-30 minutes of exposure to atmospheric air- RuO_2 thickness fit (Å)

Figure 57: Sputtered ruthenium film during first 24 hours of exposure to atmospheric air- Ellipsometric measurements of Psi

Figure 58: Sputtered ruthenium film during first 24 hours of exposure to atmospheric air- Ru thickness fit (Å)

Figure 59: Sputtered ruthenium film during first 24 hours of exposure to atmospheric air- RuO_2 thickness fit (Å)

Appendix A

Standard Operating Procedures

This appendix contains procedures, precautions, and principles in addition to the main body of the thesis. The procedures contained in this appendix should be used in conjunction with appropriate precautions and principles described in both this appendix and in the main body of the thesis. Whereas the procedures in the main body of the thesis may apply generally and/or specifically to our vacuum system, those described in this appendix are specifically designed for our UHV system as of June 2001, while the principles therein may still be applied generally.

A.1 Notes

Refer to Figure A.1 for the labelling of the valves and sorption pumps on the roughing line. This is helpful when following the procedures for vacuum pump down and bake out.

In general, know and follow the cleaning procedures and precautions laid out in Section 5.8 on page 61. When handling any parts that will or are placed inside of the vacuum system, especially be aware of the following:

- Use *clean* gloves at all times.

  When working with internal parts, after gloves have touched any external parts or anything other than the chamber such as after using the telephone, pens, etc., either clean the gloves or replace the gloves.

- If skin or body touches anywhere internal to the chamber or gloves, clean with hexane or acetone (use protective nitrile gloves) and then

wipe residue off with methyl alcohol. (Use reagent or purer type to avoid contamination)

- **Caution: Never open roughing valve and cryo valve unless the chamber and the cryopump has been vented and is in atmospheric pressure. The only other instance when the roughing valve or the cryo valve is safe to open is when the chamber or the cryopump, respectively, is at higher pressure than the roughing line for certain.**

  Opening these valves under unwarranted circumstances may cause the relatively dirty roughing line to contaminate the UHV clean cryopump or chamber, and may even cause the molecular sieve dust from the sorption pumps to backstream into these UHV clean areas.

A.2 Vacuum Pump Down

(Refer to Figure A.1)

A.2.1 Liquid Nitrogen Dewar

A liquid nitrogen dewar is needed for the sorption pumps. Any dewar will suffice. The following describes how to operate the large dewar that is currently used for our system. (See Figure A.2)

Filling an Empty Dewar

1. Unscrew the clamp and disconnect the hose that connects the dewar to the pressurized air line from the ceiling.

2. Fill the dewar with liquid nitrogen from Chem-Stores (Chemistry Central Stockroom) in the Nicholes Building.

   Be careful with transporting dewar, especially if the dewar contains liquid nitrogen, so it does not accidentally tilt over.

3. Connect the air line back to the proper fitting. (See Figure A.2)

Pouring Liquid Nitrogen Out From Dewar

By sending pressurized air through the dewar, the liquid nitrogen inside will come out of the dewar. However, it is important to realize that too much air pressure going through the dewar may cause the ∼3" diameter black cork lid to pop off the dewar. Hence it is important to be able to control the air pressure entering the dewar so that sufficient liquid nitrogen flows out without compromising safety.

The air that entered in may condense to very low temperatures, and when it comes out, it may freeze anything immediately near it. Also liquid nitrogen may leak out or spray out sporadically. After liquid nitrogen begins to come out of the dewar, the hoses will freeze and hence harden. Frozen parts become very brittle which allow them to break easily if not carefully handled. Be aware of these and take the proper precautions.

Repeat these steps for each dewar of the sorption pumps:

1. Close all four roughing valves.
2. Ensure that the air line hose is connected properly to the fitting of the dewar.
3. Safely place the end hose of the dewar (from where the liquid nitrogen will come out) inside the container that is to be filled with liquid nitrogen.

   It is a good idea to place the hose so that sudden jerking (caused by the liquid nitrogen coming out) is minimized. Also it is helpful for the tip of the hose to be visible so as to determine the amount of flow of liquid nitrogen coming out of the dewar.
4. If closed, open the bronze-colored knob several turns; this fitting is located on the dewar fitting for the air line hose.
5. Slightly open the air valve on the ceiling, until some air flow through the hose can be heard.

   Be careful so that enough air flows through the line, at the same time regulate the flow so that the pressure reader does not exceed around 3 psi.
6. Open the top knob of the dewar while ensuring proper insulation between the knob and hands.

Try to avoid direct contact with the knob. The knob may become cold as liquid nitrogen flows from out of the dewar. Using a long metal piece such as a wrench may be helpful, unless there is adequate insulation on the knob itself.

7. Wait initially until liquid nitrogen begins to come out.

   This may take a little time because the hoses of the dewar must come close to equilibrium temperature with the liquid nitrogen.

8. Control the air pressure until enough liquid nitrogen is flowing out of the dewar, in a safe and controlled manner.

   Violent boiling of liquid nitrogen will occur initially, but it will slow down as the sorption pumps reach equilibrium temperature with liquid nitrogen. Because of the boiling, it may be helpful to go back and forth between the two dewars, filling each dewar as much as possible, partially initially, until both dewars are full with liquid nitrogen. The dewars may be considered 'full' when the top surface of the liquid nitrogen reaches the bottom of the neck of the sorption pumps. However, full operation of the sorption pumps does not necessarily require that the dewars are completely 'full.'

9. After filling the container, close the top knob.

10. Close the air valve from the ceiling.

11. Carefully remove hose and the dewar away.

    This can be done after the hoses have warmed up.

A.2.2 Roughing Vacuum

The following procedures describe how to operate the two sorption pumps that are connected in parallel on the roughing line.

Preparing the Sorption Pumps

The sorption pumps must be cooled to liquid nitrogen temperature before they are ready to pump on the system.

1. Have enough liquid nitrogen to fill both sorption pump ready in a dewar.

2. Ensure that the sorption pumps are close to room temperature, especially after baking out.

   (The pumps may be cooled with liquid nitrogen even when the pumps are initially above room temperature; however, if possible, it is always more energy efficient to wait until the pumps are at room temperature.)

3. Close all four roughing valves.

4. If the Viton stoppers are already placed inside the gas release valve, then proceed to the next step. If not:

   (a) Put on clean gloves.

   (b) Clean the stoppers, and the gas release valve if needed, with methanol and Kimwipes.

   (c) With the fingers, spread a thin film of vacuum grease on the Viton stoppers, on the area of the stoppers that come in contact with the release valves. Wipe off the excess grease.

   (d) Carefully place the Viton stoppers in the gas release valves.

   They only need to placed lightly on the valves, as the pressure drop in the sorption pumps will naturally tighten the seal between the stoppers and the valves.

5. Ensure that the stainless steel dewars are placed on the each sorption pump.

6. Carefully pour liquid nitrogen into each dewar, one at a time. (Refer to Section A.2.1 if necessary)

   Violent boiling of liquid nitrogen will occur initially, but it will slow down as the sorption pumps reach equilibrium temperature with liquid nitrogen. Because of the boiling, it may be helpful to go back and forth between the two dewars, filling each dewar as much as possible, partially initially, until both dewars are full with liquid nitrogen. The dewars may be considered 'full' when the top surface of the liquid nitrogen reaches the bottom of the neck of the sorption pumps. However, full operation of the sorption pumps does not necessarily require that the dewars are completely 'full.'

7. Let the sorption pumps reach equilibrium temperature with liquid nitrogen.

   When the boiling of liquid nitrogen stops, and the base pressure of the pumps are below 1×10^{-3} torr, the sorption pumps are ready for operation. Usually right before the sorption pumps reach this point, a sudden increase of noise from the boiling occurs, and then stops immediately.

Operation

After the sorption pumps are cooled to liquid nitrogen temperature, the following steps are recommended for operating the sorption pumps.

1. Close all four roughing valves.
2. Make sure chamber body (or the cryopump, whichever is to be pumped) is in atmospheric pressure, or at least at higher pressure than the roughing pumps.
3. Open either the roughing valve or the cryo valve, depending on whether the chamber or the cryopump is to be pumped, respectively.
4. Open valve #1 for 1-$1\frac{1}{2}$ minute and then close valve #1.

   This initial 'wind' will absorb and trap much of the hydrogen, helium, and neon that is usually hard to absorb. Also much of the 'dirt' will be absorbed and trapped inside only sorption pump #1.
5. Open valve #2. The pressure should eventually read below 1×10^{-3} torr.
6. Pump for as long as the sorption pump can maintain pressures below 1×10^{-3} torr, or until the rate of pressure rise of the chamber or cryopump is sufficiently low.

   Usually, with a 'full' dewar, the sorption pump can last for 3-4 hours.
7. Close valve #2 and roughing valve.
8. The roughing valve or the cryo valve may remain open. However, it should be eventually closed when necessary.

A.2.3 Ultrahigh Vacuum

After the chamber has been able to *maintain* adequate roughing pressure ($\leq 1 \times 10^{-3}$ torr), the cryopump may then be used to pump the chamber down to UHV pressure.

Preparation

1. Close the gate valve and the cryo valve.
2. Turn the power on with the two switches for the compressor and then the cold head, on the compressor unit. (See Figure 16)
3. Wait for about $2\frac{1}{2}$ hours, enough for the cold heads to cool, so that the cryopump can pump to or below high vacuum pressures.

Operation

1. Make sure all four roughing valves are closed completely.
2. Ensure that the chamber can maintain a base pressure $\leq 1 \times 10^{-3}$ torr for an adequate time period.

   If the chamber cannot maintain low roughing pressures in the 10^{-3} torr range for about five minutes, it is best that the roughing pumps pump on the chamber longer. This will preserve the pumping capacity and speed of the cryopump.
3. Open the gate valve.
4. Turn on ion gauge and wait until it reads below 1×10^{-9} torr for UHV vacuum, or until the desired vacuum pressure is reached.

A.3 Bake Out

(Refer to Figure A.1) **Precautions:**

- Ensure that everyone is aware of the high temperature bake out when baking out.
- Ensure that all cables, wiring, and anything else that cannot stand high temperatures are removed away from the heat.

Each of the following parts may be baked out separately. The sorption pumps must be baked out before the chamber or the cryopump can be baked out, since the sorption pumps provide the vacuum source for both bake outs.

A.3.1 Sorption Pumps

To regenerate the sorption pumps, they should be baked out to 250°C for about 24 hours, and then cooled. This will emit most, if not all, of the gases that were trapped inside the sorption pumps.

Heating

1. Close the roughing and cryo valves.
2. Open valves #1 and #2 all the way.
3. Take both dewars off from the sorption pumps. (optional)
4. Ensure that any wires, hoses, or material are removed away from the heaters.
5. Turn heaters on sorption pumps by plugging the electrical cords into the proper power outlet.
6. When the pressures of the sorption pumps are close to atmospheric pressure, take the Viton stoppers off both sorption pumps and place them in a safe place.

   If the pressures of the sorption pumps are below atmospheric pressure, then the Viton stoppers will be difficult to take off. If the pressures of the sorption pumps are above atmospheric pressure, then the Viton stoppers will usually pop off on its own.
7. The heaters should heat the sorption pumps to 250°C in about $2\frac{1}{2}$ hours. It is recommended that this temperature be maintained for 24 hours.

Cooling

1. Turn the heaters off.

2. Allow the sorption pumps to begin cooling. Proceed to next step.

3. When needed, clean and lubricate the Viton stoppers: (These sub-steps should be performed right before placing the stoppers inside the release valves, which is the next step.)

   (a) Put on clean gloves.

   (b) Clean the stoppers, and the gas release valve if needed, with methanol and Kimwipes.

   (c) With the fingers, spread a thin film of vacuum grease on the Viton stoppers, on the area of the stoppers that come in contact with the release valves. Wipe off the excess grease.

4. After about 1-2 hours of cooling, or when the valves are below 150°C, *without* touching the area of the stoppers that come in direct contact with the release valves, place the Viton stoppers lightly inside the release valves.

   They only need to placed lightly on the valves, as the cooling will cause the pressure to drop in the sorption pumps, and will naturally tighten the seal between the stoppers and the release valves.

5. When the sorption pumps reach room temperature, place the dewars on the pumps.

Note: Because of the Viton o-rings inside valves #1 and #2, if the temperature of these valves exceed 150°C, provide adequate cooling of these valves. However, the two valves have never exceeded 150°C when we baked out the sorption pumps.

A.3.2 Cryopump

For this procedure, the sorption pumps should be prepared beforehand and be ready to pump on the cryopump. The gases trapped in the cryopump will be dumped into the sorption pumps when regenerating the cryopump.

Heating

1. Close the gate valve completely.

2. Close all four roughing valves.

3. Shut down the cryopump by turning the power off with the two switches for the cold head and the compressor, on the compressor unit. (See Figure 16)

4. If possible, vent the cryopump with argon, or other suitable, dry, noble gas.

5. Allow the surface temperature of the cryopump to rise to room temperature.

6. Open only valve #1 to bring the roughing line pressure below the cryopump pressure. (Usually $< 1 \times 10^{-3}$ torr for the roughing line will suffice)

7. Open the cryo valve.

8. Pump on the cryopump with the sorption pumps. (Follow the procedures in Section A.2.2) While still pumping on the cryopump, proceed to the next step.

9. Turn on the cryopump heating tape with the Variac controller.

10. The cryopump should be baked out at **150°C**. Slowly increase the Variac voltage so that the temperature increases +25°C, each hour, up to **150°C**. Maintain this temperature for about 24 hours, or for a suitable time.

11. When the base pressure of the sorption pumps begin to increase, or when they cannot maintain good roughing pressures (in the 10^{-3} torr range), be sure to close the sorption pump valves (valves #1 and #2).

12. In order to adequately regenerate the cryopump, repeat the following for at least 1-2 times, in addition to the pump down completed above:

    (a) Regenerate the sorption pumps.

    (b) Use the sorption pumps to pump out the cryopump over the course of the bake out process (including when it is being cooled down).

Cooling

1. Slowly decrease the Variac voltage so that the temperature decreases by no more than 25^oC, each hour, down to room temperature.

2. Close all valves.

A.3.3 Chamber Body

For this procedure, the sorption pumps should be ready to pump on the chamber, no later than when the chamber bake out temperature reaches 250^oC. The gases baked out from the walls of the chamber, together with all other gases inside the chamber, will be dumped into the sorption pumps when baking out the chamber body.

Preparation

1. Take off the Input and Output Units, together with the tilt stages of the ellipsometer from the chamber.

2. Remove cords, lines, and anything that could be burned away from the heating tapes and chamber surfaces.

3. Close the gate valve and all four roughing valves.

4. Turn on the water cooling lines by opening all four water line valves, two from the ceiling and two on the wall. (See Figures 49, 50)

   (The sputter gun and the crystal monitor need to be cooled when baking out the chamber.)

5. Wrap the surfaces to be baked out with aluminum foil to provide some thermal insulation. Do not wrap aluminum foil around components that contain o-rings or material that may not withstand high bake out temperatures. Monitor these areas so that they do not exceed critical temperatures.

Heating

The chamber body should be baked out to temperatures $\geq 250^o$**C**:

1. Turn ON the electrical switch (on the evaporator power supply rack) for the chamber heating tapes.

2. Slowly increase the Variac voltage for the chamber heating tapes, so the temperature increases $+25^o$C, each hour, until $\geq 250^o$**C** has been achieved. Use the top of the two thermocouple thermometers located on the evaporator power supply rack.

3. Maintain this temperature for 3-4 days or more. While baking out, proceed to the next step.

4. Open only valve #1 to bring the roughing line pressure below the chamber pressure. (Usually $< 1 \times 10^{-3}$ torr for the roughing line will suffice)

5. Open the roughing valve.

6. Pump on the chamber with the sorption pumps. (Follow the procedures in Section A.2.2)

7. When the base pressure of the sorption pumps begin to increase, or when they cannot maintain good roughing pressures (in the 10^{-3} torr range), be sure to close the sorption pump valves (valves #1 and #2).

8. In order to adequately bake out the chamber, repeat the following for at least 2-3 times, in addition to the pump down completed above:

    (a) Regenerate the sorption pumps.

    (b) Use the sorption pumps to pump out the chamber over the course of the bake out process (including when it is being cooled down).

Cooling

1. Slowly decrease the Variac voltage so that the temperature decreases by no more than 25^oC, each hour, down to room temperature.

2. Close all valves.

A.4 Deposition

A.4.1 Sputter Gun- Plasma Formation

Plasma is created by backfilling the vacuum chamber, while under vacuum, with a gas medium (usually argon), and then applying potential differences between the target source (on the sputter gun) and the rest of the chamber with a power supply. Our system uses an RF power supply to create a plasma above the sputter gun target.

The following procedures describe how to use the RF power supply for the magnetron sputter gun. The RF power supply is contained in the left rack in Figure 47. Operating the RF power supply to create a plasma is not easy. In fact, even after following these procedures, a plasma may not be created. If any difficulty arises, consult with Scott Daniel, in the Physics Department.

Preparation

1. The vacuum system should be initially in UHV or HV vacuum.
2. Close the gate valve until $\frac{1}{4}$ is *open*. Make sure that the gate valve can maintain this position until the sputtering is completed.

   A 1" wrench may be used to get it stuck on the gate valve, thus holding the position of the gate valve.
3. Tightly close the valve on the argon bypass line.
4. Open the valves on the argon gas tank to appropriate pressure readings (around 10-20 psi on the line may be sufficient).
5. Turn the mass flow controller voltage display on and set the knob to the minimum 0 value.
6. Slightly open the Nupro valve on the argon line.

   Be careful that the Nupro valve is not opened too much, causing too much argon to flow through it.
7. Slowly turn the mass flow controller knob and increase the argon flow through the mass flow controller. Backfill the chamber with argon to a pressure in the $(10^{-3} - 10^{-1})$ torr range.

There should be enough argon in the chamber to provide the discharge. At the same time the gas should be in constant flow.

Operation of RF Power Supply

1. Check to see that all plugs and connections are secure and safe.
2. Turn on all switches on the back.
3. Turn on all power switches on the front, including the Control and Line switches, all but the HV (high voltage) power switch and the RF On switch.
4. Wait 2-3 minutes for the power source to warm up.
5. Turn RF Power control knob to a minimum.
6. Turn the RF power switch on.
7. It may be helpful to check that the proper voltages are on.
8. Turn HV power on.
9. Flip Power Control switch to Auto Power Control.
10. Manually set Loading and Tuning to about 108.5 and 150.5 respectively. (These are the values that the RF power supply was tuned to, when plasma was created.)
11. Turn RF Power knob to get about 200 Watts of Incident power.
12. Tune the Plate Current with the PA Tuning knob so that the Incident power is maximized while the Reflective power is minimized. Eventually when a plasma is actually created, the PA Tuning knob tends to lie on 65.

    The Incident and Reflective power represent the power going into the impedance source (the sputter gun) and the power coming out from this source back into the RF power supply, respectively. By maximizing Incident power and minimizing Reflective power, the output power from the RF power source is efficiently consumed.
13. Set the Loading and Tuning to Automatic tuning.

14. Try the following until a plasma is created.

    When a plasma has been successfully created, a bright purplish light can be seen from the view ports of the in situ ellipsometer. Also the crystal monitor can detect the plasma, as a creation of plasma implies the immediate sputter deposition of the target material.

    (a) Adjust the RF Power knob.
    (b) Adjust the PA Tuning.
    (c) If the Loading and Tuning tend to go off the limits, or are far away from 108.5 and 150.5 respectively, and a plasma has not been created, manually reset Loading and Tuning to about 108.5 and 150.5 respectively.
    (d) Adjust the RF Power knob to provide enough Incident power.
    (e) Adjust the PA Tuning to maximize and minimize Incident and Reflective power respectively.
    (f) Set the Loading and Tuning to Automatic tuning.
    (g) The argon pressure may not be appropriate. Increase or decrease the argon flow adequately.
    (h) Carefully check to see if all connections on the RF power supply are sound. The inside of some wiring may have burned due to errors in the plasma creation process.
    (i) Scott Daniel is very knowledgeable with the RF power supply. Seek his help if necessary.

15. After the plasma has been created, the argon flow should be reduced to the recommended operating pressure for Mighty MAK- between 2 - 15 mtorr.[14]

16. The power can be controlled manually by switching to Manual Power Control, and by adjusting the RF Power knob. Controlling the output power will allow control over the sputter deposition rate and the energy of the sputtered material.

Shutting Down

1. After the deposition has been completed, turn RF Power knob to the minimum, and turn off *all* power switches on the RF power supply.

2. Close all valves on the argon gas tank.
3. Wait for several seconds so argon from the line is vented into the chamber, eliminating an unnecessary high pressure differential between the argon line and the chamber.
4. Turn off the mass flow controller voltage display and knob.
5. Tightly close the Nupro valve.

   If the Nupro valve is not closed tight enough, it provides a UHV leak.
6. Completely open the gate valve.

Appendix B

Documentations and Records

B.1 Cryopump Performance Operating Log

Tables B.1, B.2 contain the performance log of the cryopump when it was first used to pump the initial vacuum session of our UHV system.

B.2 Adhesive Heating Tapes Data

Greg Harris calculated the data in Table B.3 to order the adhesive heating tapes for baking out the main chamber body. The heating tapes were ordered from Clayborn Labs, Inc.

Greg based his calculations on achieving a ΔT of 430°F (500°F total) by applying 142 watts per square foot. (If these resistances are not available in stock, they should be substituted with the next hottest tapes.) All calculations were based on 1/2 inch tape.

The position of the tapes is relative to the perspective of facing the vacuum system with the sorption pumps in the front, as in Figure A.1.

B.3 Mass Flow Controller- Voltage Display

The voltage display used to display the voltage reading across the mass flow controller is a product of Simpson Electric Company. J.J. designed a circuit connecting the knob that controls the voltage applied, the voltage display, and the mass flow controller. This circuit design is shown in Figure A.3.

The pin-out for the mass flow controller is shown on the controller itself. The pin-out for the Simpson voltage display is shown in Figures A.4, A.5.

Table B.1: Cryopump performance operating log- Initial vacuum session during April - May 2001

Date / Time	ETM	H_2 Vapor Pressure	Compressor Unit Pressure (psi)	Chamber Base Pressure (torr)
April 10 @11:00pm	13040.9	Device not available	221	2.1×10^{-4}
11:12pm				$> 1.0 \times 10^{-3}$ (Cryopump not quite prepared to pump)
11:17pm			225	2.4×10^{-4} (Chamber was roughed again, with gate valve closed)
11:57pm	13041.9		231	
12:25am	13042.3		230	
12:39am			230	
12:51am	13042.7		230	
1:12am			227	9.8×10^{-5} (Gate valve opened again)
1:15am				8.8×10^{-5}
April 11 @11:50am	13053.8		223	2.4×10^{-6}
12:00pm				Decrease of bake out temperature began from $\sim 160^oC$
3:22pm				1.1×10^{-6}
4:08pm			223	9.9×10^{-7} @93^oC
4:25pm				8.8×10^{-7} @73^oC
4:57pm	13058.9		222	7.5×10^{-7} @54^oC - Bake out completely turned off at this temperature
April 12 @12:24pm	13078.3		224	5.2×10^{-7} @24^oC
1:06pm				2.2×10^{-7} (Gas valves were closed tighter than before)
1:50pm				1.7×10^{-7} (Gas valves were closed even tighter, together with the UHV variable leak valve)
3:30pm				1.5×10^{-7}

Table B.2: Cryopump performance operating log- Initial vacuum session during April - May 2001 (continued)

Date / Time	**ETM**	**H_2 Vapor Pressure**	**Compressor Unit Pressure (psi)**	**Chamber Base Pressure (torr)**
April 16 @4pm				3.2×10^{-7}
April 17				3.5×10^{-7}
April 19 @1pm				3.9×10^{-7}- Unsuccessful sputter attempt on this day
May 3				1.0×10^{-6}- Successful sputtering of ruthenium on this day
May 10			222	2.1×10^{-6}- Venting of chamber with argon began
May 11 @4:38pm			222	2.2×10^{0}- Continuing attempt to vent chamber with argon
May 17 @3:40pm				7.2×10^{1} @24°C- Continuing attempt to vent chamber with argon
May 18 @4:35pm			220	1.5×10^{2} @24°C- Continuing attempt to vent chamber with argon
May 23 @4pm			220	3.6×10^{2} @24°C- This is about atmospheric pressure. The thermocouple gauge was reading incorrectly.

Table B.3: Adhesive heating tapes calculated data

Position	**Quantity**	**Length (ft)**	**Total Watts**	**Watts / Foot**	**Ohms / Foot**
Right hand, back side	2	5.5	94	17.1	23.4
Left side	1	4.0	56	14.0	54.0
Front port	1	5.5	130	23.6	16.9
Bottom port	1	3.0	56	18.7	72.0
Body	4	2.0	95	47.5	63.7
Top body ring	1	3.0	142	47.3	28.4
Bottom body ring	1	6.5	186	28.6	10.0

Appendix C

Company References

Table C.1: Company References

Address	Phone Number	Fax Number	Webpage	Contact
Company Name			**Description**	
A&N Corporation			Vacuum products	
707 Southwest 19th Avenue Williston, FL 32696	(800) FLANGE1	(352) 528-3441	www.ancorp.com	
Clayborn Labs, Inc.			Heating tapes	
12219 Business Park Drive, Suite 11 Truckee, CA 96161	(530) 587-4700	(530) 587-5255		
Duniway Stockroom Corp.			Vacuum products	
1305 Space Park Way Mountain View, CA 94043	(800) 446-8811	(650) 965-0764	www.duniway.com	
Helix Technology Corporation			Cryo pumps	
3350 Montgomery Drive Santa Clara, CA 95054	(408) 727-8077	(408) 988-6630	www.ctivacuum.com	
Huntington			Vacuum products	
1040 L'Avenida Mountain View, CA 94043	(650) 964-3323	(650) 964-6153	www.huntvac.com	
Inficon			Thin Film Deposition Control	
Two Technology Place East Syracuse, New York 13057-9714	(315) 434-1100	(315) 437-3803	www.inficon.com	
J.A. Woollam Co., Inc.			Ellipsometry	
645 M Street, Suite 102 Lincoln, NE 68508	(402) 477-7501	(402) 477-8214	www.jawoollam.com	
Kurt J. Lesker Company			Vacuum products	
1515 Worthington Ave. Clairton, PA 15025-2700	(800) 245-1656	(412) 233-4275	www.lesker.com	
Maxtek, Inc.			Quartz crystal monitor	
11980 Telegraph Rd. Santa Fe Springs, CA 90670-6084	(562) 906-1515	(562) 906-1622	www.maxtekinc.com	
MDC Vacuum Products Corporation			Vacuum products	
23842 Cabot Blvd. Hayward CA 94545-1661	(800) 443-8817	(510) 887-0626	www.mdc-vacuum.com	

Table C.2: Company References

Company Name			**Description**	
Address	**Phone Number**	**Fax Number**	**Webpage**	**Contact**
R.D. Mathis Company			Evaporative boats	
PO Box 92916 Long Beach, CA 90809-2916	(562) 426-7049	(562) 595-0907	www.rdmathis.com	
Simpson Electric Company			Voltage meters	
853 Dundee Avenue Elgin, IL 60120	(847) 697-2260	(847) 697-2272	www.simpsonelectric.com	Tom Franta (ext. 1479)
US Inc.			Sputter gun	
6280 San Ignacio Ave. Suite E San Jose, CA 95119-1365	(408) 363-6906	(408) 363-6996	www.us-incorp.com	Todd Johnson
Varian, Inc.			Vacuum systems	
121 Hartwell Avenue Lexington, MA 02421	(800) 882-7426		www.varianinc.com	Bill Horton

Figure A.1: Roughing line and pumps - labeling

Figure A.2: Liquid nitrogen dewar currently used for the system

Figure A.3: Circuit design- Mass flow controller, voltage control knob, and voltage display

Figure A.4: Pin-out for Simpson voltage display

Figure A.5: Pin-out for Simpson voltage display (continued)

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