This thesis titled
Operation of Cold STM System In Conjunction With In Situ Molecular Beam Epitaxy

by

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Abstract

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Operation of Cold STM System In Conjunction With In Situ Molecular Beam Epitaxy (69 pp.)

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Procedures for operating an STM designed to operate with the surfaces being imaged at temperatures between 4.2 – 323 K were adjusted to be applied to the system, the system’s performance was tested, and aspects of the system were modified. The procedures include the outline of methods for cooling the system, an outline of many of the parameters for annealing tips, and a method for leak detection. Aspects of the system tested for performance were tip annealing, isolation of the system from noise, vacuum quality, system cooling, sample growth and STM scanning. The modifications included a new filament for annealing, an additional noise dampener made of rubber, and removal of vacuum leaks.
This thesis is dedicated to my family for their continued support in all things.
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1 Introduction

The intended purpose of the system is the growth and study of nitride based compounds at temperatures between $4.2 - 325 \ K$. Future plans for the system include the use of pulsed laser deposition (PLD) and/or molecular beam epitaxy (MBE) to grow a wide range of samples for study. However, only MBE growth will be discussed. The system is designed to study the atomic ordering found in these materials using reflection high energy electron diffraction (RHEED) and scanning tunneling microscopy (STM). Additionally, the magnetic ordering can be studied using spin polarized STM (SP-STM) techniques. However, only RHEED and STM techniques will be discussed. Certain techniques are not being discussed because they have not been applied in this system yet.

1.1 System Outline

The chapters following this one will contain a discussion of portions of the system and their individual components. This chapter will serve as a discussion of the design of the system as a whole to assist in the visualization of the interconnectedness of the system. The system consists of a modular design, which includes three main vacuum chambers and one load lock chamber. These chambers are depicted in Fig. 1.1.

The three main chambers are the distribution chamber, growth chamber, and analysis chamber. Each is associated with its own specific functions and is needed for the intended purpose of the system.

The growth chamber is where samples are prepared. To allow this growth, a cryo-pump is used in this chamber because it is able to operate at the higher pressures generated during growth[17]. Sources of growth material release enough particles to raise the pressure into the $10^{-5} \ Torr$ range. Further growth discussions can be found in section 2.5.
The distribution chamber is designed to serve as a place to store samples and STM tips. As well, it serves in the preparation of tips. Discussion of the tip annealing process can be found in sections 2.1 and 3.1. This chamber uses an ion pump in combination with a titanium sublimation pump (TSP).

The analysis chamber is where the STM occurs. It contains a cryostat for low temperature study of samples. The chamber uses an ion pump and TSP combination like the distribution chamber. Within this thesis, a dewar will always refer to a chamber for storing cryogen inside the cryostat and a storage tank will refer to something that stores cryogen outside the system.

Throughout the system exist a series of transfer arms. They allow for the transfer of samples throughout the system. The modular design and transport arms together allow samples and tips to be transferred throughout the system without contaminating the tips, samples or chambers.
The load lock chamber is used for bringing samples in and out of the higher vacuums in the remainder of the system. A turbo pump backed by a scroll pump pulls a vacuum in this chamber down into the $10^{-8}$ Torr range. When pumping, the pressure is low enough to allow transfer into the rest of the system without contaminating the other chambers when they are under ultra high vacuum (UHV). The load lock can also be vented to allow transfer between it and the outer atmosphere. The load lock is a necessary intermediary between the other chambers and the outside air to prevent contamination of the main vacuum chambers. The need for the avoidance of contamination is discussed in section 2.3.
2 Procedures

2.1 Tip Annealing

2.1.1 Background

Tungsten is a popular material for use in STM tips. There are two main reasons for this. Firstly, tungsten wire is relatively easy to form into an atomically sharp tip using various electro-chemical etching techniques. Secondly, tungsten is a stiff durable material.

Atomic sharpness has the advantage of creating a small single site from which electrons will tunnel from the tip. This property is desirable in a tip because it increases the attainable image resolution in an STM. The resolution is increased because the current comes from a consistent point on the tip. This consistency results in any displacement of the tip being more likely to result in imaging at an equal displacement on the surface. If the current is shifting around relative to the tip in an unpredictable manner, then it becomes more uncertain where on the surface the tip is observing.

Secondly, a stiff durable material will maintain its shape longer while scanning. While STM scanning is designed to prevent direct contact between the tip and the surface, contact can still occur sometimes. Whenever there is contact, it is beneficial to have a tip that will minimize blunting on the surface even when it comes in strong direct contact with the surface.

However, tungsten has a significant disadvantage. It oxidizes quickly enough that oxide layers will form on it before it can be placed under vacuum. Oxidized tungsten is a poor conductor. It is poor enough that the layers formed in only a few minutes in air under atmospheric pressure can reduce the achievable tunneling current to the point that no image can be obtained by the STM when viewing a surface.

In order to access the advantages of tungsten this disadvantage must be overcome. This can be done by annealing the tip under vacuum. Most commonly this is achieved
with field emission or electron beam (e-beam) annealing. Due to the design of this system, field emission can only be done in the STM and risks damaging the STM head. So, the distribution chamber is used for tip preparation.

The pre-existing tantalum e-beam filament was found to be unable to provide enough power to properly anneal a tungsten tip. It shorted to the base of the annealing station because the filament was not stiff enough to support its own weight and would sag down to touch the base. Additionally, the majority of its surface area did not have line of sight to the tip due to its shape. When held in the station the tip is in a recessed cavity above the filament to prevent the tip from touching anything during transport. As well, due to the design of the annealing station, the walls of the cavity are held at the same electric potential as the tip during operation. These two factors made the filament unable to emit enough electrons that could find a path to the tip during annealing. This layout is shown in Fig. 2.1.

This lack of a proper filament meant that etched tungsten tips could not be cleaned properly and thus achieve enough tunneling current. So, etched tungsten tips could not be used for STM scanning in this system. This type of tip has many properties making it the desired choice for scanning. So, a new filament was designed.

2.1.2 Design

Due to the annealing process only working under vacuum, the large time necessary to vent and then pull a vacuum on the chamber, it was essential to plan the design of the filament as much as possible before final testing. The first consideration for making the filament was finding the material that should be used. Tungsten was selected because it was a readily available wire material, has a high recrystallization temperature, and is stiff enough to support its own weight when heated. Crystallization temperature is the temperature above which a material can become amorphous. The stiffness of a material
drops off sharply when it passes its crystallization temperature. The power supply can only deliver a current up to 14 A and tungsten has a crystallization temperature of around 1300 – 1500°C depending on the pressure and purity[7][12].

Figure 2.1: Side view of annealing station with tip loaded in. Wires 1 and 2 carry the current, which also goes through the filament. Wire 3 holds the tip shuttle and tip at a voltage separate from the filament circuit to drive annealing current.

Figure 2.2: Diagram of filament and annealing station. Left: Side view of filament. The top two cone structures attach together at the red dots to form the structure on the bottom, in which the top structure is nested inside of the middle structure. The double cone structure on the bottom is the full filament. The green dots indicate where the filament feeds to the wire leads. Right: Top view of annealing station without tip loaded in. Shows a top view of the filament and its connections to the wire leads on either side of it.
The filament design was selected in order to maximize the filament’s surface area near the tip. When the tip is in the annealing station the opening below the tip is roughly a 10 by 15 mm rectangle, as seen in Fig. 2.1. A coiled double cone design, illustrated in Fig. 2.2, was chosen. The circular edge reduced the available surface area, but was necessary to prevent any kinks that would cause uneven heating or weak points that would have been in a rectangular edged filament design. Having the coils out of plane with their neighbors allowed a greater length of wire to fit directly under the opening. The double cone allowed the center loops that would have been farther away in a single cone design to be closer to the tip.

The diameter of the wire was limited to those which were commercially available. Further, the smallest diameter that would support its own weight was desired in order to increase resistance. This desire results from the previous filament not being able to support its own weight and shorting to the base of the annealing station. A .015 in diameter wire was tested and was found to be able to support its own weight when in the shape of a filament. A .01 in diameter, the next available size, wire was then tested and was found to be unable to support its weight when in the shape of a filament. From these two tests it was found that a .015 in diameter wire would be the smallest available diameter wire, which would support its own weight when in the shape of a filament.

The desired length needed to reach and connect to the electrical leads, which were 35.5 mm apart, was estimated. The leads each required around 12 mm of wire to achieve a solid electrical and mechanical connection. The length of wire coming from the edge of the double cone to a lead needed to be 12.75 mm and the length of wire coming from the center of the double cone to a lead needed to be 17.75 mm long.

The remainder of the length of the filament wire composes the double cone section. Length is proportional to the surface area of the filament wire. The power output is proportional to the surface area. So, more length would increase the power output. For
this reason, the double cone section was wound as tightly as possible to use as much length as possible. The double cone section was able to use 265.5 mm.

2.1.3 Filament Testing & Operation

2.1.3.1 Burning In The Filament

To begin operation, the filament was first degassed while under vacuum. This needs to be done before using any filament for annealing to insure that the best vacuum can be maintained during annealing. To accomplish this degassing an 8 A current was run through the filament.

Using a paper outlining the relevant properties of tungsten in combination with the Ohmic Resistor Power law,

\[ P = I^2R \]  \hspace{1cm} (2.1)

and the Stephan-Boltzmann law,

\[ P = A\epsilon\sigma T^4 \]  \hspace{1cm} (2.2)

, the temperature of the filament can be estimated from the current. Where I is the current through the filament, R is the resistance of the filament, \( \epsilon \) is the emissivity, \( \sigma \) is the Stefan-Boltzmann constant, A is the surface area of the wire and T is the temperature.

From these equations, an 8 A current should raise the temperature of the filament to approximately 1150\(^\circ\)C[19]. It is necessary to hold at this temperature in order to degas the filament. This temperature exceeds the normal operating temperature, 700 – 800\(^\circ\)C, so that any degassing at normal operating temperature will be minimal. Minimizing the amount of degassing during normal operation will result in there being a better vacuum during normal operation. However, it is important to note 1150 – 1200\(^\circ\)C approaches the recrystallization temperature 1204\(^\circ\)C of the material[12]. Therefore, a temperature of 1200\(^\circ\)C should not be used in general operation because doing so will greatly diminish the lifetime of the filament. The lifetime would be reduced because exceeding the
recrystallization temperature increases how brittle the material is\cite{10} \cite{11}. Going over the recrystallization temperature more than is necessary to degas the filament increases this effect, which is why it is only done during the initial degassing.

The pressure in the chamber rises as the filament degasses. If the pressure exceeds $10^{-6}$ Torr for too long, the ion pump pulling a vacuum on the chamber can be damaged\cite{16}. As well, sudden large changes in current can damage the filament. So, the filament current was increased slowly in order to protect the ion pump and filament. The filament current was raised by $.5$ A at a time and the pressure in the chamber was then allowed time to come down to under $5 \times 10^{-7}$ Torr before the next increase. In this instance, the base pressure was measured to be $1.7 \times 10^{-7}$ Torr. The pressure in the chamber would typically rise to the upper $10^{-7}$ Torr range after each current increase. Each step took an average of 2.5 min.

Then, the filament current was held at 8 A until the pressure stabilized. The final stable pressure was $2.4 \times 10^{-7}$ Torr. A pressure somewhat over the base pressure was expected because the filament was slowly heating the rest of the chamber. This heating would cause the rest of the chamber to bake out slightly and be expected to cause a noticeable pressure increase even after the filament had finished degassing. The filament current was lowered slowly at a rate of around 1 A/min.

2.1.3.2 Testing Power Output

After burning in the filament, a tip was put into the annealing station. Using the procedure of raising or lowering the filament current by $.5$ A increments every 30 sec, the filament current was brought to 5.9 A using a voltage of 7.5 V. Then, a voltage of 1 kV was applied between the tip and filament. The e-beam emission current was then raised to 20.1 mA, corresponding to an e-beam power of 20.1 W. This power was held for roughly 30 sec before dropping the emission current back to 0. Afterwards, under magnification of
×10 the end of tip was observed to have formed into a small spherical bead. This
observation indicated that the experimental setup used is able to deliver an e-beam to the
tip far exceeding the necessary power output for annealing.

2.2 Noise Isolation

2.2.1 Background

This STM (Scanning Tunneling Microscope) system is intended to provide atomic
resolution. Additionally, it is intended to study nitride based compounds such as GaN and
$Mn_xN_y$. The atomic spacing associated with these materials is around $0.1 - 0.6 \text{ nm}$, and
the change in STM signal current typical over such horizontal displacements is
approximately $0.5 \text{ nA}$ for common STM operation[2]. Typical operation occurs when the
tip remains about $0.3 - 0.5 \text{ nm}$ from the surface. So, for atomic resolution to be reached
noise must be significantly lower than this value.

There is one further complication that must be considered in order to appreciate the
effect of noise on an STM image. The complication is that different noise frequencies will,
depending on the scanning frequency, interfere with the signal being acquired. The
scanning mode employed by this system to map out a 2-dimensional area involves
sweeping the tip back and forth in one dimension and moving a constant increment in the
second dimension after each sweep. This causes the tip to sweep an entire area line by line
at a periodicity (line time) resulting from the length of a line and the scan speed. If a peak
frequency of the noise is around 10-20 times the line frequency, the peak frequency will
produce periodicities in the image which may look like atomic resolution. This type of
signal must be understood and distinguished from any true signals from atomic features.
Figure 2.3: **Left:** A noise spectrum with frequency peaks. **Right:** Scan of a manganese nitride film with noise resulting in a false image of regularly occurring bumps.

An example of a noise spectrum is shown on the left in Fig. 2.3. A spectrum typically consists of background noise with peak frequencies distributed through the background noise.

A frequency peak will produce regularly occurring bumps. An example of these periodic bumps is shown on the right in Fig. 2.3. Peak frequencies of different frequency, but equal amplitudes will produce bumps of different widths along the surface and equal heights above the surface.

As already indicated, if the bumps are near the same width as the atoms, then they can provide false images and more effectively obscure the true image. True signals are easier to pick out when next to much more wide or thin signals. The line time can be adjusted to help to raise or lower the size of the noise relative to the true signals. However, certain peak frequencies will limit the available scan speeds and scan areas to a large degree.

Peak frequencies typically have the greatest effect on obscuring true images. This is because the false images they produce typically have significantly higher amplitudes than the signal of other frequencies and the highest amplitude signal tends to dominate an image.
The magnitude in which different peak frequencies affect an image’s resolution is related to the line time. Typical widths of scan areas, which are used to achieve atomic resolution, are within the sizes of $5 - 100 \text{ nm}$. Typical scan speeds capable of reaching atomic resolution for this system are found roughly between $50 - 100 \text{ nm/s}$. However, larger scan areas and speeds can be used for topographical images. This gives a range of line times, which might provide atomic resolution, of around $.05 - 2 \text{ sec}$.

Within the mentioned parameters, a peak frequency of $1 \text{ Hz}$ or lower will produce at most about two bump signals per line. Even at the low end of typical scan lengths, about $5 \text{ nm}$, the signal is much larger than the periodicity of atoms, about 12 atoms per line for large spacings. So, peak frequencies lower than $1 \text{ Hz}$ are not noticeable. A peak frequency of $1000 \text{ Hz}$ or higher will produce a minimum of 50 signals per line at the smallest typical line time. A peak frequency of $1000 \text{ Hz}$ or higher would then be noticeable at large scan areas with fast scan rates. However, peak frequencies over $1000 \text{ Hz}$ are manageable at smaller scan lengths and scan speeds. For example, consider the limitation of scan lengths less than $40 \text{ nm}$ and scan speeds slower than $80 \text{ nm/s}$. Under these limitations $1000 \text{ Hz}$ or greater would give at least 500 signals per line. Under these limitations true signals of the smaller atomic spacings used in this system would give 133 signals per line. So, the noise becomes manageable without limitations being too prohibitive to scanning. So, as long as a peak frequency is not much greater than the strength of the real signals, the peak frequencies outside of around $1 - 1000 \text{ Hz}$ will not produce signals that will unavoidably obscure signals of true images. For this reason, in the noise isolation experiment the main consideration is peak frequencies between $1 - 1000 \text{ Hz}$. 
2.2.2 Description Of Noise Isolation Measures

As mentioned previously a low noise level is desired. To achieve this low noise level, multiple techniques are employed. These techniques are intended to reduce either mechanical or acoustical noise.

The techniques intended to reduce mechanical noise are as follows. Two stages of laminar flow isolators exist. The first stage consists of 4 laminar flow isolators (commonly referred to as air legs), which float the entire system off the lab floor. The second stage uses 3 laminar flow isolators to float the cryo-insert off the main analysis chamber. However, the cryo-insert is still connected to the main analysis chamber by a bellows. After study, a layer of soft rubber material was wrapped around and securely pressed against the bellows to serve as a dampener to minimize conductance of noise through the bellows. These measures are shown in Fig. 2.4. Further, all electrical, gas, and water lines to the chamber are hung with slack either from walls or a tray hung from the ceiling. The laminar flow isolator technique is intended primarily for mechanical noise reduction of high frequencies since the resonance frequency of the laminar flow isolators is very low and therefore they damp high frequencies very well.

The technique intended to reduce acoustical noise is as follows. To achieve this, every air vent in the room is covered with sound dampening material. In addition, a sound dampening wall and set of doors separates the STM controller and experimenters from the system. To isolate from the remaining noise, the cryo-insert itself is surrounded by an acoustical box as shown in Fig. 2.5. The box is in the shape of a triangular prism around the upper part of the analysis chamber.

Going further, the noise sources themselves are reduced when scanning. All non-essential devices within the lab are turned off. In addition, efforts are made to scan in
Figure 2.4: Noise isolation measures in analysis chamber.

The evening when other labs are inactive within the building. The effort is made especially when a helium liquefier, which is down the hall from the lab, is not running.

The mechanical and acoustical shielding combined with noise source elimination are intended as a comprehensive means to eliminate noise detrimental to scanning. In the results section, the effectiveness of these measures will be analyzed.
2.3 Vacuum Leak Detection

2.3.1 Background

Vacuum quality is a major concern for experiments involving the study of materials with a scanning tunneling microscope (STM). A pressure below $10^{-10}$ Torr is necessary to insure negligible contamination of a clean surface[20]. STM measurements are only of the surface of materials. Accurate studies of a material cannot be performed if the material is covered by unknown contaminants. For this reason, ultra high vacuum (UHV), pressures lower than $10^{-10}$ Torr are desired for the type of experiments this system is designed for, namely the atomic and magnetic ordering of nitrogen based compounds such as GaN and Mn$_x$N$_y$.

Pressures below $10^{-10}$ Torr are low enough so that the amount actively deposited on the surface approaches the amount that is leaving the surface. These pressures allow a sample to remain clean enough for study for as long as a few days. Due to the time it takes
to scan a surface, sometimes an experiment lasts for up to a few days. UHV is thus necessary for this system, but UHV can only be achieved if the leaks in the system are sufficiently small.

Various particles, especially water molecules, have the behavior, when approaching UHV to constantly desorb and reabsorb on the chamber wall before they can be pumped out. This behavior creates a background pressure inside the chamber that cannot be pumped down into low UHV, \(10^{-11} - 10^{-10}\) Torr range. Bake-outs consist of heating a vacuum chamber for an extended period of time. Bake-outs are designed to quickly desorb the particles from the chamber walls so that they can be pumped away.

A bake-out must occur after every time the chamber is exposed to atmosphere in order to reach UHV again. The reason for this is that the particles in the air, such as water vapor, will recoat the chamber walls. A chamber can even be partially contaminated by being exposed to pressures over \(10^{-6}\) Torr.

### 2.3.2 Leak Detection Process

After baking the system out for two weeks the pressure in the analysis chamber did not drop below \(10^{-8}\) Torr. The pressure would be expected to drop much more after this amount of bake-out time. This result led to the suspicion that there was a leak in the chamber.

Typical methods for measuring a leak include releasing an agent outside a chamber and then using a device inside the chamber to detect the agent. The preferred agent is often helium gas, which can be detected by a residual gas analyzer (RGA). Helium is typically used for leak detection because it is inert and small in size. These factors mean it can travel easily through any leak that larger or more reactive particles might not be able to pass through.
An alternative type of leak detection when no RGA is available is to spray large molecules such as isopropanol in the vicinity of a suspected leak. This can result in the isopropanol molecules getting stuck inside the leak for a while and partially sealing the leak, which can then be detected as a small pressure drop inside the chamber. Thus the leak can be found.

The process applied by spraying the isopropanol alcohol on every flange, bellows and welds on the chamber. These are the places where leaks have a much higher chance of happening. Each of these was sprayed systematically. Starting at the top of the chamber and moving counterclockwise around the chamber towards the bottom, each potential leak location was checked.

2.4 Cryogenics

2.4.1 Background

There are many reasons to operate a cold temperature STM as opposed to a room temperature STM. First, there is lower thermal drift at cold temperatures. Thermal drift is reduced for two reasons. The primary reason is the thermal expansion coefficient is smaller at lower temperatures. The secondary reason is that a cryogen tends to have a more stable temperature than the air in a room. This results in less temperature variation within a microscope that is submerged in a cryogen. Over time the atoms being scanned will move less relative to the reference point of the tip. Any measurement of the relative positions of all features then requires less correction at lower temperatures.

Second, certain phenomena are easier to observe at lower temperatures. For example features such as dimers, which can be mobile on a surface are easier to observe and monitor at colder temperatures because their surface diffusion is reduced. Sometimes, the features will freeze out and become stationary on the surface. Due to this effect, low temperatures make it possible to study such features.
Thirdly, certain structural and magnetic phase transitions occur at lower temperatures. For example, Si(001) has been observed to transition from a $2 \times 1$ to a $c(2 \times 4)$ structure near 200 $K$ [23]. As well, the Néel temperature, the temperature above which an antiferromagnetic material transitions to a paramagnetic one, is often below room temperature.

Finally, it is likely that interesting unknown phenomena exist at low temperatures. In the past multiple phenomena such as quantum corrals were first discovered by using a low temperature STM [22]. This type of discovery will likely continue. This likelihood of further discovery and the previously mentioned advantages are all reasons for studying materials at low temperature.

However, there is a downside to operating a cold temperature STM compared to a room temperature STM. Using cryogenics in an STM system requires much greater time and money to obtain reasonable measurements. Cryogens can boil off in a matter of hours while STM scans often go on for many hours at a time. Therefore, scanning must be stopped periodically in order to replace spent cryogen. The tip must be retracted from the surface every time the system is being refilled. The refilling itself can take a few hours. To resume scanning the tip must reapproach the surface, which can take 30 minutes to an hour. Therefore, the use of cryogens reduces the number and length of experiments that can be performed in a single work day. All cryogens are expensive. Liquid helium especially is an expensive cryogen that must be used to achieve stable temperatures near 4.2 $K$. Additionally, liquid helium boils off continually. The boil-off must either vent to atmosphere or a liquefier. When vented to atmosphere the helium is lost and must be replaced by purchasing new helium. When vented to a liquefier the helium can be recycled between uses. A helium liquefier can be just as expensive to build and operate. However, recycling with a liquefier is the likely to be less expensive in the long term.
2.4.2 Cryogenic System Outline

This subsection consists of a description of the system. This description serves as a reference, which is intended to assist the reader in understanding the procedure related to operating the cryogenic aspect of the system. This procedure will be outlined in the following subsection.

In the operation of the system three main dewars are used to store cryogenic liquids. The first dewar is at the very center of the cryo-insert as shown in Fig. 2.6. The dewar is called the flow cryostat and has a 1 L capacity. This dewar has the option of either continuously flowing a cryogen to assist in cooling the substrate or to be held under vacuum to act as an insulating layer. The second dewar from the center is the main helium dewar with a total capacity of 15 L. This dewar has direct thermal contact to the STM head. The connection is made through a copper plate, which is bolted to both the base of the dewar and the STM head. A diagram of the STM head is in Fig. 2.7. The third dewar is the nitrogen dewar, which is in thermal contact with the nitrogen cooled radiation shield. These two sit between the helium dewar and the vacuum jacket. The nitrogen dewar has a capacity of 8 L. The nitrogen cooled shield is intended to emit a lower level of thermal radiation towards the helium dewar than a room temperature vacuum jacket would.

Additionally, the STM cryostat contains sensors, which assist in monitoring the operation of the cryogenics system. There are 2 Cernox thin film resistance thermometers with an effective temperature range between 4.2 K and 400 K. One is on the bottom surface of the superconducting magnet and the other is inside of it. As well, there are 3 liquid helium level monitors. One is in the main helium dewar and has a 28 in active length with a second backup meter. The third meter is in the flow cryostat and has a 17 in active length.
The two supply lines into the main helium dewar have rubber washer seals as shown in Fig. 2.8. These washers are pressed down, which forces them to, in turn, press sideways against the stainless steel tube. This forms an effective seal at the transfer point. The supply line into the nitrogen dewar does not have a seal. It is not as necessary to have a
seal when transferring into this dewar because only nitrogen which, is less expensive, dangerous and cold than liquid helium, is ever contained in this dewar.

Figure 2.7: Diagram of STM Head

Figure 2.8: Cross-section of washer seal and nitrogen supply line.
There are two separate types of transfer lines into the dewars. One is for the transfer of liquid nitrogen into the system and the other is for the transfer of liquid helium into the system and liquid nitrogen from the system. The procedure for transfer is discussed in following subsections.

The end of the liquid nitrogen transfer line, which is inserted into the supply line of the dewar, consists of a stainless steel tube. The stainless steel tube has outer and inner diameters of \(0.375\) and \(0.363\) in respectively. It is used to bridge the gap between the main transfer line and the dewar. A Tygon tube, which makes up the majority of the transfer line’s length, fits snugly around the stainless steel tube and two pull ties secure the two tubes together, as shown in Fig. 2.8. This seal can be maintained under the typical operating pressures of \(20 - 30\ psi\).

The Tygon tube piece of the liquid nitrogen transfer line is unable to attach directly to the supply tanks available in the lab. Therefore, an adapter is required in order to attach the supply line to the tanks. A threaded adapter was made using a female lead with \(3/4-20\) UNEF screw threads, which can be fitted to the supply tanks, on one end and a brass tube with the same dimensions as the stainless steel tube on the opposite end of the fill line. The Tygon transfer line connects to the brass tube end of the adapter by fitting snugly around it and being tied down with 2 pull-ties in the same manner as the stainless steel tube connection on the opposite end of the Tygon tube.

A different transfer line is used to transfer liquid helium. The transfer line consists of two layers of metal with a vacuum in between the two layers. The vacuum is needed because insulating the line with a material would be impractical because a thick material would be required in order to provide sufficient insulation. A thick material due to its size would have a high heat capacity. Therefore, much more helium would need to be boiled off so that the line would be cool enough to transport the helium as a liquid.
2.4.3 Fill Procedure

The system has three temperature modes of operation. They are room, liquid nitrogen and liquid helium temperature modes. In the room temperature mode the entire system is equilibrated with room temperature, generally around 298 K. In the second-mode, liquid nitrogen is used to bring the STM and sample near 77 K, the boiling point of nitrogen at atmospheric pressure. In the final mode, liquid helium is used to bring the STM and sample to near 4.2 K, its boiling point. However, before filling can occur, some preparations must take place.

2.4.3.1 Preparation

Three steps must be taken before any cooling is performed on the system. Without taking these steps the system may become damaged. The analysis chamber must be brought under its desired operating vacuum level, all dewars must be pumped and then flushed with nitrogen gas. If the flow cryostat is going to be used as a vacuum insulator, it must be brought under vacuum.

The analysis chamber needs to be brought to its desired operating vacuum level for one main reason. If the chamber is not under at least $10^{-4}$ Torr, then there is a risk of contaminating the inner surface of the vacuum chamber. Many particles will condensate onto a surface colder than the air next to it when exposed to normal atmosphere. Ice can even form. Any water on a vacuum chamber wall can greatly increase the difficulty of achieving ultra high vacuum (UHV) conditions in the future. This problem occurs because water molecules have the property when approaching UHV to constantly desorb and reabsorb with a chamber wall before they can be pumped out. This property creates pressure in the chamber that cannot be pumped down into low UHV, $10^{-11}$ – $10^{-10}$ Torr range. When a vacuum pressure under $10^{-4}$ Torr is present in the analysis chamber there are not enough particles to adsorb to the cold surface in a way that might damage the
surface. This step of pulling a vacuum will keep the chamber clean and able to achieve UHV.

A secondary reason the analysis chamber should be brought to its desired operating vacuum, not just $10^{-4}$ Torr, is that the final vacuum will be lower than could be achieved otherwise. The lower the pressure before cooling the fewer particles adsorbed onto the sources and the better that the colder surface will pump down the chamber.

The cryo-insert cannot be completely baked out because a superconducting magnet in the cryostat would be damaged if it was heated over 323 K [8]. Therefore, UHV must be achieved without baking the cryo-insert at typical bake-out temperatures. However, when cooled, the surface of the cryo-insert can act as a getter pump and effectively trap any contaminants on itself, which causes the surface to behave as if it was baked out at typical bake-out temperatures.

The second step before cooling is to use a vacuum pump to draw a vacuum in the dewars and then to flush the dewars with nitrogen gas. This procedure should be repeated at least 3 times. Pumping and flushing is a commonly accepted procedure for removing impurities from a system. In this case it is intended to remove any excess water or water vapor that has formed in the chamber. As has been mentioned before, water vapor in the air condenses and freezes on surfaces that are cold enough. The dewars are quickly sealed in between transfer of cryogens to prevent moisture from the fill lines, and the outflow of boil-off prevents moisture from entering the dewars through the exhaust lines. However, there is still some exposure while cooling and even more when left at room temperature, when there is no boil off.

Within an enclosed space, freezing water can sometimes rupture its container because water expands when freezing. There are narrow tubes and spaces in the dewars that can become filled with only small amounts of water relative to the dewars’ total size. As a result, the presence of water must be limited within the dewar to prevent clogging the flow
of cryogens and possible damage to the dewars from water freezing and expanding. By pumping and flushing 3 times with nitrogen gas the amount of water in the dewar can be lowered enough to make the amount of moisture in the dewars far too small to cause clogs or damage to the dewar.

A third step must occur if the flow cryostat is used as a dewar instead of a vacuum insulator. If this is the case, it must be pumped and flushed like the other dewars. As in the other dewars, the amount of water in flow cryostat needs to be minimized. If it is not minimized, ice may form inside of it clogging or damaging the flow cryostat like the other dewars would be.

2.4.3.2 Nitrogen Filling

Before any liquid nitrogen filling begins, the exhaust from the dewar should be fed to outside the lab’s air supply. Nitrogen gas is an asphyxiant. Liquid nitrogen at 77 K has a density of 808 kg/m$^3$ and nitrogen gas at room temperature has a density of 1.25 kg/m$^3$. When exposed to a room of air at room temperature 1 L of liquid nitrogen can rapidly expand to 646 L or .646 m$^3$ [13]. Oxygen typically composes 20.9% of air by volume. When this drops below 19.5% ill effects begin to occur for any persons breathing the air. Additionally, the lab doors should be opened to insure good ventilation in case of spills. As well, gloves and goggles should be worn to protect against direct exposure to cryogens.

The liquid nitrogen supply is typically delivered from a storage tank. Most liquid nitrogen storage tanks store their contents under pressure. This pressure is useful in pushing the liquid into the dewars. However, if the pressure is too great it creates the risk of cracking the Tygon supply lines. To decrease this risk, the vent valve of the tank should be opened until the tank pressure is lowered below 30 psi.

Once the tank pressure is low enough, the nitrogen fill line is connected to the tank using the adapter. The fill line should then be supported so that the mechanical strain on
the line is limited in order to prevent cracking. Before filling the line it should be dried. Any water on the line will often freeze after the line has hardened. When the water freezes the layer closest to the fill line will freeze and bond to the fill line. As the rest of the water freezes it will increase in volume. This puts strain on the hardened fill line and often will result in cracking the fill line.

The terminal end of the fill line should be place near the nitrogen dewar fill port. Then, the liquid flow is turned on slowly. Gas will eventually begin to flow out. Once this occurs, the fill port can be opened. The gas should be allowed to blow into the port for a few seconds before inserting the fill line. This is done to push any air, which enters upon opening the dewar, out of the dewar. This is done to prevent moisture from getting into the dewar. Once the air is pushed out, the fill line can be inserted into the port. The fill line should continue to be supported until it hardens and can support its own weight.

At the base of the fill line, the opening into the rest of the nitrogen dewar is significantly smaller the fill line itself. The liquid will back up at the pinch point so the port will often overflow because there is not a seal between the transfer and fill lines. Due to varying amounts of the liquid evaporating, the liquid flow rate cannot be measured. This fact prevents the ideal rate being measured. As a result, to adjust the flow rate, a specific procedure should be followed.

The first step is to slowly increase the flow from the tank until the port overflows. After this happens the flow rate is slowly decreased until the rate is just low enough for the port to stop overflowing. The liquid flow rate will then be as high as possible without overflow occurring. Once the dewar is full, the port will begin to overflow again. The flow from the tank can then be shut off because filling is completed.

After filling stops, the transfer line will heat up unevenly. The section closest to the adapter will remain hardened for a longer time than the rest of the tube because it is in thermal contact with the outflow line of the tank. The outflow line takes longer to heat up
because it is better insulated than the fill line. The weight of the fill line will be applying force to the joint where the adapter and fill line are connected. This joint will sometimes crack as a result. A heat gun should be used to thaw out the entire fill line to prevent the fill line cracking. Once the line is thawed out, it can be removed from the dewar port.

Whether cooling to helium mode or nitrogen mode, the main helium dewar should now be filled with nitrogen. This is the final step to reaching nitrogen mode and a necessary step to reach helium mode. Otherwise, the helium boil-off caused by cooling the dewar will be too great to fill effectively. This limitation results from the process this lab uses to recycle the helium. There is limited storage for the helium gas before it is liquified. If the helium were not recycled, the limitation would also apply because too much of the expensive helium would evaporate to atmosphere.

The process for filling the main helium dewar with liquid nitrogen starts similarly to that of filling the nitrogen dewar. The tank pressure should be below 30 psi. The fill line should be dried. The adapter should be attached to the tank. The fill line should be supported. The flow should be turned on and then the port opened. After blowing nitrogen gas into the port for a few seconds, the stainless steel tube can be put into the port.

The procedure differs after these steps. After inserting the stainless steel tube into the helium fill line, the washer valve needs to be screwed down to create a seal. Then, as before, the transfer line must be supported while it hardens. After waiting for the line to harden enough to support its own weight, the flow rate can be increased. The flow can be slowly increased until the transfer line begins shaking. Once it begins shaking, the flow should be decreased until it is no longer shaking in order to avoid the line cracking.

The main chamber has two thermometers inside of it. One is exposed to the rest of the dewar at the base of the magnet. The other is shielded from the rest of the dewar by the magnet and is near the top of the magnet. When the lower thermometer reads 77 K the liquid nitrogen level has reached the bottom of the magnet. After reaching the magnet, the
fill should continue for another 30 min. This is necessary to provide a 48 hour hold time in the dewar.

Once the dewar is filled, the fill line should be thawed out as it was following the filling of the nitrogen dewar. The line can then be removed from the port. The system is now in liquid nitrogen mode.

2.4.3.3 Helium Filling

Before cooling the system down to liquid helium mode the system must be in liquid nitrogen mode. More liquid helium than would be practical would need to be used to cool down the system, if it is not in liquid nitrogen mode.

There are two fill ports for the main helium dewar. The initial fill port empties all the way at the bottom of the dewar. The refill port empties mid-dewar. Nitrogen freezes at 63 K. Frozen liquid nitrogen could potentially clog the fill ports. For this reason the liquid nitrogen in the chamber must be removed.

A tank of pressurized nitrogen gas is used to push out the bulk of the liquid nitrogen. First, the tank is connected to the refill port. Next, the helium transfer line is connected with the initial fill port with the opposite end placed in a bucket, which will collect the liquid nitrogen. The nitrogen tank is set to release nitrogen gas with a pressure of 4 psi into the refill port while the exhaust port is closed. The liquid nitrogen will then be pushed out of the dewar and into the bucket. Care must be taken to seal the port afterwards in order to minimize moisture from getting into the dewar. The flow of nitrogen gas into the dewar should be stopped once liquid stops coming from the transfer line.

There will still be a small amount of liquid nitrogen in the dewar. Due to this, the remaining liquid nitrogen needs to be allowed to boil off for 10 min. After this is done, the transfer line should be removed from the bucket and the initial fill port. The exhaust line should be reopened. Helium filling can now begin.
Helium storage tanks must constantly release helium gas as it boils. Otherwise, the pressure will continue to build until the storage tank ruptures. Additionally, helium should be saved due to its high cost. Therefore, the boil-off of the storage tank is typically fed into a gas bag to be recycled later. Before filling, the tank should be cut off from the gas bag so that pressure can build in the storage tank. This pressure will allow the liquid helium to be pushed out of the storage tank and into the dewar. To start the fill, the transfer line can be inserted into a helium storage tank. Once gas begins exiting the other end of the transfer line, it can be inserted into the initial fill line. The initial fill line is used because the refill line will pour liquid helium over much of the chamber increasing the initial boil-off by contacting more of the dewar initially. If this is done before the dewar is cooled down enough there will be extra boil-off and the fill will be less efficient because the gas bag, which collects the boil-off, will reach capacity sooner and the fill must stop.

Four things need to be monitored while filling the chamber. The pressure in the storage tank, the pressure in the helium exhaust line, the fullness of the gas bag collecting the boil-off, and the liquid level in the dewar all must be monitored. To insure that liquid is transferred from the storage tank, the end of the transfer line must be kept below the liquid level in the storage tank. Inserting the transfer line in further will heat the liquid helium. This will increase the pressure in the tank. The pressure should be kept near 4 psi. This will maintain the flow at a rate suitable for filling. Some of the gas transfer lines exiting the dewar are unable to deal with higher pressures. A heating filament is built into the helium tank to raise the pressure after the transfer line is inserted completely into the chamber.

There is a pressure gauge that monitors the pressure in the exhaust line. It is depicted in Fig. 2.9. If liquid helium begins to flow past the pressure gauge, it will drop down to the gauge and rapidly boil off because that lower section will not have cooled yet. This
Figure 2.9: Depiction of the pressure gauge setup in the exhaust line of main helium dewar.

sudden boil off will cause a sudden rise in pressure. The transfer line should be immediately pulled above the liquid level in the storage tank. The exhaust line cannot support liquid helium flow and would break.

The gas bag, which the boil off feeds into, can burst if it becomes too full. So, its fullness must be constantly monitored. A gas compressor can pull gas from the bag and put the gas into storage tanks. However, due to its limitations the compressor can only slow the filling of the gas bag. Once the gas bag nears capacity, the transfer line should be pulled above the liquid level in the storage tank. After removing the transfer line from the liquid the rate of flow into the dewar and gas bag will be greatly reduced. Before refilling continues the gas bag needs to be pumped empty by the compressor.

There is a liquid level monitor in the dewar that indicates the liquid helium level in the dewar. When the liquid level reaches 20 in the transfer line should not be inserted into the storage tank any further and the heating filament should be turned off if it is on. Once the level reaches 28 in the dewar is full. When this happens the transfer line should be
pulled from the liquid level in the storage tank. The pressure in the storage tank should then be allowed to drop down so helium does not pour out uncontrollably when the transfer line is removed from the dewar and storage tank.

Once filling is complete, the transfer line is above the liquid in the storage tank and the pressure has dropped, the transfer line can be removed from the dewar. Once removed, the initial fill port should be sealed quickly. The transfer line can now be removed from the storage tank. After this the storage tank should be quickly sealed and allowed to vent to the gas bag. The initial fill line screws together at a joint below the liquid level. To decrease the heat going into the chamber it should be unscrewed and pulled above the liquid level in the dewar. The helium is now isolated at the bottom of the dewar with the boil off flowing into the gas bag.

### 2.4.3.4 Refilling

All nitrogen refilling involves the same procedure as the nitrogen filling procedure. The helium refilling is modified somewhat from the initial helium filling. The only difference between the two types of refilling is that a separate refill port to the main helium chamber is used for helium refilling. This is done because the first part of the fill usually pumps in only gas until the thermal load of the transfer line is overcome. Unlike the He initial fill, which occurs soon after liquid nitrogen was drained from the main helium dewar through the transfer line, the transfer line is not precooled to near 77 $K$ for the He refill. So, the time it takes to overcome the thermal load of the transfer line is greater for the refill than the initial fill.

If the hotter helium gas is pumped through the bottom of the dewar, the gas will boil off a substantial amount of liquid, which remains from the previous fill. If the refill port is used, the initial gas flow will boil off less liquid helium because it will not pass through
the liquid before going out the exhaust. Otherwise, all the procedure is the same as for an initial fill.

2.5 Sample Growth And Study

2.5.1 Background

This system is primarily designed to grow and study nitride based semi-conductors and metallic nitrides. There are three growth sources in the growth chamber. Two of the sources are home built Knudsen effusion cells. One cell is a manganese source and the other is a gallium source. In addition, there is a radio-frequency (RF) nitrogen plasma source.

This thesis will discuss the preparation and inspection of one sample each of graphite, $\epsilon$-phase Mn$_4$N and $\eta$-parallel Mn$_3$N$_2$. The $\epsilon$ and $\eta$-parallel phases are depicted in Fig. 2.10. The red dots within the $\epsilon$-phase depict the N-vacancy lattice and the dotted red lines in the $\eta$-parallel phase depict the N-vacancy planes.

Figure 2.10: Model of two manganese nitride structures. Gray circles represent N atoms and black circles represent Mn.
2.5.1.1 Graphite

Graphite is non-reactive enough to be used in STM experiments conducted outside of vacuum. The preparation consists of placing a piece of scotch tape on its surface, applying an even pressure to the back of the tape across the whole surface and then pulling the tape off the surface in one smooth motion. A thin layer of graphite will be pulled away with the tape leaving behind an atomically smooth surface behind. Then, the sample can put under vacuum and inserted into the STM for study.

2.5.1.2 Manganese Nitride Preparation

Both manganese nitride samples were grown using molecular beam epitaxy (MBE). MBE consists of delivering a molecular beam of particles onto a substrate, which has the same crystalline structure as the desired sample. When the right substrate temperature and molecular beam fluxes are used the particles will self assemble into the desired crystal using the substrate as a scaffold. In the case of manganese nitride growth in this system, Mn is heated inside of a crucible to the point of its sublimation. The Mn is stored at the bottom of the crucible. From there Mn gas will radiate away from the source and onto the substrate. The nitrogen is flowed, as a gas, past a copper coil. The copper coil transmits a radio frequency power signal, which results in the creation of a plasma. The plasma consists of $N$ atoms, excited $N_2$ molecules and some nitrogen ions, which radiate out toward the substrate.

Before growth an MgO substrate was heated to 1000°C for 30 min while the nitrogen plasma source is active. This cleans and prepares the surface for growth. Then, the Mn source is activated to begin growth.

A calibration was done for the Mn flux using a quartz crystal thickness monitor inside the chamber. From the Mn flux and comparison with a previous study the nitrogen flux was then estimated for each growth[3]. During growth in this system the atomic
spacing on the surface of the sample is observed using reflection high-energy electron diffraction (RHEED) with an incident electron beam energy of 20 keV. The diffraction pattern was compared with a past study to determine the phase of manganese nitride present in the samples. After growing, each sample was scanned in the STM.
3 Results And Discussion

3.1 Normal Operation Of Annealing Station

Using the same procedure for raising and lowering the filament current as when testing the power output, tips have been annealed using various power outputs for different lengths of time. The power outputs used were either 0.8 – 1 W. The annealing times between 5 – 10 min.

After preparation, these tips were tested in the STM at low temperatures. Each time a tunneling current has been achieved. When the annealing time or power output was lowered to 0.8 W the resolution of STM images were qualitatively observed to have better resolution. Images and the annealing parameters are of the associated tips are available in following sections of the results chapter. This observation has led to the confirmation that the new design of the filament is able to deliver the majority of its e-beam power to the tip as opposed to the tip shuttle.

3.2 Testing of the Noise Isolation

Previous experiments were conducted on the system showing a noise spectrum with the highest RMS frequency peak to be below 0.7 pA for all frequencies between 1 – 1000 Hz at room temperature. This noise was associated with the taking of what are suspected to be atomic resolution images of a graphite sample. Both an image and the associated noise and frequency peaks can be seen in Fig. 3.1. The image in Fig. 3.1 may or may not be atomic resolution. The scale was not calibrated when the image was taken. As a result, the 2 nm sized features may indeed be atoms. There is also a possibility that it is a Moiré pattern, which results from a misalignment of the top graphite layer with the one beneath it. This misalignment can result in peaks and troughs from the two atomic structures adding constructively to create larger than atomic sized features. It is uncommon to scan a Moiré pattern by chance, but because the length scales of the system
had not yet been calibrated a Moiré pattern cannot be ruled out. However, the features should be within an order of magnitude of atomic size even if the image is a Moiré pattern. This level of noise serves as a benchmark for the necessary noise reduction in the system for other temperature modes. If the level of noise is above the benchmark further measures must be taken to reduce the level of noise.

Figure 3.1: Suspected atomic resolution and associated benchmark noise. Unpublished data courtesy of Wenzhi Lin.

### 3.2.1 Liquid Nitrogen Mode Testing

Various spectra were taken while operating in liquid nitrogen mode on a graphite substrate using a tungsten tip annealed for 10 min at a power of .8 W. All the spectra were taken using the same settings used in the benchmark spectrum. This was done to allow for a better comparison to be made between the two. However, the benchmark spectrum was achieved with a Pt/Ir tip. The spectrum analyses in Fig. 3.2 were taken by using a FFT algorithm[6]. So, the graphs are scatter plots with lines connecting each data point. Each data point is the root-mean-squared current over a small frequency interval. The interval
each data point measures is \(0.953 \text{ Hz}\). So, the root-mean-squared current in the interval around each data point is the square root of \(0.953 \text{ Hz}\) times the value at the data point. For sake of visualization there is roughly a 1:1 correspondence between a data point and the root-mean-squared current at that frequency.

![Figure 3.2: Noise spectrum analysis of the system in liquid nitrogen mode with acoustical box on and no bellows dampener. Top Left: Air legs floating base and cryostat. Top Right: Air legs floating base and not floating cryostat. Bottom Left: Air legs not floating base and floating cryostat. Bottom Right: Air legs not floating base or cryostat.](image)

The graphs all have a peak at \(460 \text{ Hz}\) that is around \(7 \text{ pA/}\sqrt{\text{Hz}}\). This is an order of magnitude above the peak in the benchmark. This suggested the noise was much too high to achieve atomic resolution.
Further, as seen in Fig. 3.2 the magnitude of the 460 Hz peak is somewhat lower without floating the base or cryostat than when floating both. As well, the background noise without floating the base or cryostat is within 50% of the background noise when both are floating at every frequency measured. For some frequencies the noise level is lower when neither section is floating. This is unexpected because the air legs have a transmission coefficient of mechanical noise below 5% for all frequencies over 10 Hz\[15\]. The low amount noise reduction suggests the primary noise source is not mechanical noise coming from outside the system.

The air leg’s lack of effectiveness led to two suspected noise issues. Firstly, that acoustic noise was getting through the acoustical box either through the box itself or by being conducted through the bellows. Secondly, that the boiling nitrogen was bubbling and generating noise inside the system.

Next, the system was allowed to equilibrate with room temperature and a sheet of soft rubber material was constructed in order to serve as a dampener of the bellows. When the temperature measurement on the STM magnet was 234 K noise spectra in Figs. 3.3, 3.4 and 3.5 were taken using the same substrate and tip. At this temperature all of the liquid nitrogen is boiled off and should no longer be a noise source.

The noise spectra in Fig. 3.3 indicate that the acoustical box has the effect of removing the two peaks at 19 Hz and 774 Hz from the spectra. As well, it shows that the 7 pA/√Hz at 460 Hz is most likely due to the liquid nitrogen. This relatively large peak is completely lost in the background noise and the only change in the system between the measurement on the left spectra in Fig. 3.3 from the bottom right spectra in Fig. 3.2 is that there is no liquid nitrogen.

The noise spectra in Fig. 3.4 show that both stages working together reduce the two peaks at 19 Hz and 774 Hz similarly to the acoustical box. However, the stage at the
Figure 3.3: Noise spectrum analysis of the system in room temperature mode without the bellows dampener. **Left:** Acoustical box is off and all air legs are down. **Right:** Acoustical box is on and all air legs are down.

cryostat appears more effective at reducing the peak at 774 Hz and the stage at the base appears better at reducing the peak at 19 Hz. Additionally, there is an unexplained peak which arises at 120 Hz when only the base stage is floating. As this peak can be removed from the spectrum it was not given significant consideration.

Next, the effectiveness of the bellows dampener was tested. Figure 3.5 indicates that including the dampener will reduce the background noise at higher frequencies. This desired effect warrants its inclusion into the system.
Figure 3.4: Noise spectrum analysis of the system in room temperature mode with acoustical box off and bellows dampener off. **Top Left:** All air legs are on. **Top Right:** Air legs are on at the base stage and off at the cryostat stage. **Bottom Left:** Air legs are off at the base stage and on at the cryostat stage. **Bottom Right:** All air legs are off.
Figure 3.5: Noise spectrum analysis of the system in room temperature mode with acoustical box on and all air legs floating. **Left:** Bellows dampener is on. **Right:** Bellows dampener off.
3.2.2 Liquid Helium Mode Testing

After testing the noise isolation system with no cooling liquid, the system was tested in liquid helium mode due to the suspected noise resulting from the liquid nitrogen. A new tungsten tip was put into the chamber and annealed at the same parameters as the last. Then, using the same scanning parameters the spectra in Fig. 3.6 were taken.

Figure 3.6: Noise spectrum analysis of the system in liquid helium mode with all noise isolation measures in place. **Left**: Same scan parameters as previous spectra. A/D Gain is 1. **Right**: A/D Gain adjusted to 8.

Figure 3.6 shows that the background noise significantly dropped after entering the liquid helium mode. The spectrum on the left is still above the benchmark noise. Next, a measurement was taken where the gain was multiplied by 8 because this setting is more common when attempting atomic resolution with this system[6]. This setting was not used before because it did not match the settings during the benchmark noise. This further supports the hypothesis that the liquid nitrogen was a major source of noise in the system. With the benchmark achieved, attempts were then made to achieve atomic resolution on other substrates by adjusting scan parameters.
3.3 Leak Detection

Sudden fluctuations in pressure were observed when isopropanol was sprayed on three potential leaks. Two were in flanges. One was the ion gauge flange and the other was in a transport arm flange. The third was in the wobble stick depicted in Fig. 3.7. The wobble stick is used to transfer samples and tips in and out of the STM.

![Figure 3.7: Wobble Stick](image)

The chamber was vented and the gaskets in each flange were replaced and the knife edges were checked for nicks to repair the leaks. No nicks were visible. After pumping down the system it was checked for leaks again. A leak was found to still be in the wobble stick. After spraying the travel line with isopropanol and sliding it in and out of the flange the leak became sealed and the pressure in the chamber began to slowly drop down and then rise back to the original pressure. This pressure fluctuation indicated a leak in the bellows of the wobble stick that would be partially sealed temporarily by the isopropanol. The wobble stick was then removed and the chamber pumped down. The pressures achieved after this were in the in the mid $10^{-9}$ Torr range with no bake out. This lower
pressure indicates that the wobble stick is the only remaining leak in the chamber. The leak in the wobble stick must be repaired or the wobble stick replaced in order to achieve the desired vacuum level. Successfully finding all the leaks in the chamber suggests that the method of using isopropyl alcohol is effective on this system.

### 3.4 Cryogenics

#### 3.4.1 Boil Off

To estimate how much helium and nitrogen this system requires the filling boil off and the hold time must be estimated. Filling boil off is the amount of liquid that boils away when cooling down the system from a temperature above the boiling temperature. The filling boil off results mainly from cooling the dewar. The hold time is the amount of time taken for the liquid in a full dewar to completely boil away. The hold time is most influenced by how well the system is insulated.

This system typically requires $50 - 60 \text{ L}$ to fill the $15 \text{ L}$ main helium dewar with liquid helium when the system starts at $77 \text{ K}$. $35 \text{ L}$, the difference between $50$ and $15 \text{ L}$, is used as the best estimate for boil off because the lowest amount of boil off should have the minimized amount of wasted liquid helium. There is always a small amount of waste due to the procedure used even though attempts are made to minimize waste. For example, in this system the helium boil off is collected into a gas bag in order to later be compressed into storage tanks. The gas can then be reliquefied and stored for later use. So, the majority of gas can be saved. However, after the transfer valve is removed from a port helium will leak out in the time it takes to reseal the port.

The latent heat of vaporization is $8.29 \times 10^{-2} \text{ kJ/mol}$ for helium. One mole of helium corresponds to $2.76 \times 10^{-2} \text{ L}$[14]. Meaning the removed heat needed to lower the $15 \text{ L}$ system from $77 \text{ K}$ to $4.2 \text{ K}$ is $105 \text{ kJ}$. 
The amount of metal associated with this amount of heat and temperature change was roughly estimated in order to check the estimate for helium filling boil off. This was done because helium gas can be lower than 77 $K$ in temperature and may contribute significantly to cooling. The metals cooled in the dewar include bronze, copper, niobium and stainless steel. These all have specific heat capacities of around 0.4 $kJg^{-1}K^{-1}$. At lower temperatures below 77 $K$ these heat capacities should be scaled down by roughly a factor of 10 for the purposes of an estimate. This gives a heat capacity of 0.04 $kJg^{-1}K^{-1}$. Using this value gives an estimate of 36 kg, which is a reasonable estimate for the amount of metal being cooled.

The amount of liquid nitrogen needed is unknown because there are no accurate gauges on the liquid nitrogen tanks. However, the average heat capacity of the metal in the dewar from 77 to 298 $K$ can be approximated to be around 5 times lower than the heat capacity at room temperature. This approximation predicts 638 $kJ$ need to be removed to cool the main helium dewar from 298 $K$ to 77 $K$. The heat of vaporization of $N_2$ is 5.56 $kJ/mol$. One mole of $N_2$ corresponds to $3.47 \times 10^{-2} L$[13]. This gives 4 L of filling boil off. This much lower volume of boil off is primarily the result of the much higher heat of vaporization for nitrogen over helium. As the main helium dewar is much larger than the nitrogen dewar, the filling boil off for the nitrogen dewar is expected to be much less than the larger dewar.

The hold time for the main helium dewar in liquid helium mode is about 10.5 hr. This is much lower than the manufacturers claim of a 36 hr hold time[8]. There are holes in the vacuum jacket, which is shown in Fig. 2.6. The holes are needed in order to insert and remove tips and samples from the STM. This discrepancy may result from the absence of a radiation shield designed to slide over these holes during STM operation. Additionally, the company estimate assumes that the flow cryostat has liquid helium flowing through it.
The heat needed to boil off 15 L of helium is 45 kJ. A hold time of 10.5 hr supports an estimate of 1.2 W for the rate that heat enters the main helium dewar.

The hold time for the nitrogen dewar was observed to be around 48 hr. A sudden increase in the boil off rate in the main helium dewar is observed in the nitrogen dewar after around 48 hr of not refilling the nitrogen dewar. A loss of 8 L of liquid nitrogen every 48 hr suggests heat comes into the dewar at a rate of 7.4 W. This rate is higher than the rate of heat into the main helium dewar as expected. The expectation exists because the nitrogen dewar is less insulated than the main helium dewar.

The total amount needed to fill the nitrogen dewar is unknown because of there is no accurate way to measure the amount used. However, the filling boil off would be expected to be much less than that of the main helium chamber because it is a much smaller mass. So, it should be safe to estimate the filling boil off of the nitrogen dewar to be less than a liter. The estimates for filling boil off combine to give the total liquid nitrogen needed to enter liquid nitrogen mode from room temperature mode is 27 L.

The measurement for filling boil off suggests that 50 L of liquid helium are needed to enter liquid helium mode from liquid nitrogen mode. This measurement and the estimate for the amount of liquid nitrogen needed to enter liquid nitrogen mode suggest 50 L liquid helium and 27 L of liquid nitrogen are needed to reach liquid helium mode from room temperature mode. The estimates for heat flow rates suggest 4 L of liquid nitrogen and 35 L of liquid helium are needed every 24 hr to maintain liquid helium mode.

3.5 Sample Growth And Study

3.5.1 Graphite

The image in Fig. 3.8 was taken using a tungsten tip annealed with 1 W of power for 5 min. The step visible in the image was the only feature successfully seen while in liquid nitrogen mode. The many horizontal streaks present in the image are the result of noise.
On many of the bright streaks the tip is likely contacting the surface. The tip would quickly become damaged as a result of the relative hardness of the graphite sample.

Graphite is a popular material for calibrating a system. This popularity results from the ease of preparation of large atomically smooth areas. Finding a step, such as the one in Fig. 3.8, when scanning is unusual.

An atomically smooth surface will only have individual atoms as features. When far from achieving atomic resolution these features will be a complete blur. Any small changes in resolution will also produce a blur. So, if small incremental changes are made to the settings neither noticeable improvements nor noticeable decreases in image quality will be observed. Calibration consists of making small incremental changes that improve image quality. For this reason, a material with a greater number of larger features, more than 1 nm, was needed in order to know if calibrations were having a positive or negative effect on image quality.
So, a softer sample with a greater number of larger features was needed. There were three options available to be produced by the growth chamber: manganese nitride, gallium nitride, and manganese gallium. Manganese nitride was chosen because it is more conductive than gallium nitride. Images are produced by current so it was more likely to produce an image.

### 3.5.2 Manganese Nitride

#### 3.5.2.1 Source Calibration

Before growth began, the Mn source was calibrated in order to determine the dependence of particle flux on source temperature. The rate of growth was measured for different temperatures, as shown in table 3.1, using a quartz crystal thickness monitor. Then, flux was calculated for each growth rate using the relation,

$$J = \frac{d\varrho}{mt}$$

(3.1)

, where \(J\) is the flux density, \(d\) is the thickness of sample, \(\varrho\) is the density of the material, \(m\) is the mass of an individual particle, and \(t\) is the time of growth. Growth rate is equal to \(d\) divided by \(t\). The calculated fluxes versus their corresponding temperatures are shown in Fig. 3.9. An exponential fit line of the data, also shown in Fig. 3.9, is used to define the relationship between temperature and flux. This fit is used to predict the temperature to be used in an effusion cell to obtain the desired flux during a growth. The fit has a high r-squared value, 0.999, which means that the data lines up very closely with the fit and suggests that the fit should make accurate predictions.

#### 3.5.2.2 \(\epsilon\)-phase Manganese Nitride Growth

After annealing an MgO substrate the RHEED image shown in the top left of Fig. 3.10 was taken on the [100] face of the substrate. The temperature of the substrate was
Table 3.1: Mn Source Flux Calibration Data

<table>
<thead>
<tr>
<th>Temperature (Celsius)</th>
<th>Rate (nm/second)</th>
</tr>
</thead>
<tbody>
<tr>
<td>940</td>
<td>.0028</td>
</tr>
<tr>
<td>980</td>
<td>.0061</td>
</tr>
<tr>
<td>1020</td>
<td>.0131</td>
</tr>
<tr>
<td>1060</td>
<td>.0291</td>
</tr>
</tbody>
</table>

then brought to 450°C. The flux of the N-plasma source was set so the pressure in the chamber 1.0 × 10^{-5} Torr and a forward power of 450 W was used. The Mn source was set to a temperature of 1080°C, which from calibration corresponds to a flux of 3.56 × 10^{14} n sec^{-1} cm^{-2}. Comparison between RHEED images in other studies indicated ε-phase manganese nitride was grown[3]. The flux ratio for ε-phase growth, $J_{Mn}/J_N$, is 1.67. This suggests the N flux used was around 2.13 × 10^{14} n sec^{-1} cm^{-2}.

3.5.2.3  $\eta_{||}$-phase Manganese Nitride Growth

After annealing an MgO substrate the RHEED image shown in the top right of Fig. 3.10 was taken on the [100] face of the substrate. The temperature of the substrate was then brought to 450°C. The flux of the N-plasma source was increased from the previous experiment so that the pressure in the chamber was 2.0 × 10^{-5} Torr. A forward power of 450 W was used. This was done because the $\eta$-phase requires a lower $J_{Mn}/J_N$ than the ε-phase growth. In order to target $\eta_{||}$-phase the Mn source flux was lowered to contribute to lowering the flux ratio as well. The Mn source was adjusted to obtain the desired RHEED pattern. A temperature of 1040°C, which from calibration corresponds to a flux of 1.60 × 10^{14} n sec^{-1} cm^{-2}, was used. This provided the desired flux ratio of 0.87. Comparison between RHEED images in other studies indicated $\eta$-phase manganese nitride was indeed grown[3]. This suggests the N flux used was around
$1.84 \times 10^{14} \text{ n sec}^{-1} \text{ cm}^{-2}$. The nitrogen flux found for this sample is lower than the nitrogen flux estimated from the ideal flux ratio in $\epsilon$-phase sample. It is not expected to be lower because the pressure in the chamber from the nitrogen flux was higher. Likely the flux ratio in the previous growth was not ideal and thusly higher than 1.67. Growth occurred for 1 hr 33 min 26 sec. The nitrogen flux in the chamber needs to be better calibrated for experiments in order to be sure of the flux ratios. However, the RHEED patterns match well enough with the previous studies to suggest the quality of growth should be sufficient when the experimental goal is achieving atomic resolution with the STM.

Figure 3.9: Plot of Mn flux rates at varied temperatures and extrapolated fit line.
3.5.2.4 Images

The image in Fig. 3.11 was taken of the $\epsilon$-phase sample in liquid helium mode using a W tip annealed at a power of 0.8 W for 5 min. This image is of a region with varied and uneven topography. The first true features seen by the STM in liquid helium mode were in this scan. However, the uneven topography in this area of the substrate was preventing seeing higher resolution features. So after retracting the tip the substrate was moved slightly in order to scan a new area.

With the tip over a different area on the same substrate as in Fig. 3.11 the images in Fig. 3.12 were taken. This scan provided the first imaging of individual steps. Evident in both images are angles in the step formation associated with the expected type of
tetrahedral growth in the material. These features were likely original to the sample immediately following growth.

However, there was a problem with the scan. Figure 3.13 shows the noise associated with the scan in Fig. 3.12. The background noise in Fig. 3.13 is below 1 \( pA \) and comparable to the benchmark noise. However, there are three peaks exceeding 5 \( pA \) and two over 1 \( pA \) in Fig. 3.13, which far exceeds any of the benchmark noise. This comparison with the benchmark indicates that the noise level is too high to reach atomic resolution.

There is another problem with the scan. The image on the right in Fig. 3.12 was taken before the image to its left over the area indicated by the blue box. As can be seen from the disparity between the two images, the area in the blue box was damaged during scanning. Further, the area along a 100 \( nm \) tall strip across the full length of bottom of the larger area in Fig. 3.12 was scanned before the full image of that area shown on the left
was taken. Rounded deformities appear to be deposited on the surface in the strip on the bottom of the left image. The combination of this and the two visible punctures on the left image suggest the tip was picking up and putting down sections of the surface during scanning some time before the image on the left was taken.

Also, evident in the scan is a buildup of impurities on the surface in the right image. They are most likely caused by the vacuum level in the chamber. The vacuum level is above the desired amount because of the leak mentioned in section 2.3. The right image in Fig. 3.12 was from an initial scan so it was unlikely that these built up impurities were due to damage from scanning.

Due to the surface interacting with the tip to this extent and being coated with a substantial amount of impurities, the tip and surface were thought most likely to be too damaged to achieve better resolution. As a result, scanning was halted so that the tip could be replaced. Another sample was prepared as well because of the impurity build up on the sample.

Figure 3.12: Topographical STM image of ε-phase manganese nitride
Figure 3.13: Noise associated with the topographical STM image of $\epsilon$-phase manganese nitride in Fig. 3.12

The images in Fig. 3.14 were taken of the $\eta$-phase sample using a different tip as in Figs. 3.11 and 3.12 using the same annealing parameters. The images in Fig. 3.14 were of similar resolution to the scan in Fig. 3.12. However, there appeared to be much less damage to the tip and surface from scanning as there were no noticeable punctures or deposits from the tip. Impurities covering the surface continued to be an issue as evident in the bottom left image in Fig. 3.14. Figure 3.15 shows the noise associated with the scan in Fig. 3.14. The background noise in Fig. 3.15 is below 1 pA and comparable to the benchmark noise. However, there are six peaks exceeding 2 pA in Fig. 3.15, which are much higher than any of the benchmark noise. These peaks are an improvement on the scan of $\epsilon$-phase manganese nitride, but are still too high. In order to achieve better resolution the tip and vacuum quality both likely need to improve. The issue with noise level is such that it might be overcome by adjusting scan parameters in order to reach atomic resolution.
Figure 3.14: Topographical STM image of $\eta$-phase manganese nitride
Figure 3.15: Noise associated with the topographical STM image of $\eta$-phase manganese nitride in Fig. 3.14
4 Conclusion

Modifications to the filament in the annealing station allowed the system to achieve the necessary tunneling current using tungsten tips. In order to check the hypothesis that the majority of the e-beam power from the filament is reaching the tip, a much lower power output of 0.2 W will be tested. Achieving a high resolution would indicate this hypothesis is correct. Having a poor tunneling current will indicate otherwise.

The benchmark noise level associated with achieving atomic resolution was reached. To achieve atomic resolution in liquid helium mode more scanning should be performed with similar scanning and annealing parameters with small corrections. Vacuum pressure continues to require significant improvement. By adjusting scan, annealing and vacuum parameters it is reasonable to expect that the system can achieve atomic resolution.

Keeping the system in liquid helium mode requires 2 fillings a day. This amount is too high for the system to be maintained at liquid helium mode practically because measurements must be taken as well as cooling the system. To achieve constant liquid helium mode the issues of the heat flow into the system must be addressed. Future experiments must be attempted with the radiation shield on the vacuum jacket and flowing liquid helium through the flow cryostat.

STM images with step resolution were achieved. Making further advances towards atomic resolution will most likely require improvement to the tip and vacuum quality. The annealing parameters must be optimized and the wobble stick must be prepared or replaced to accomplish this.
REFERENCES


