Master’s Thesis of Sekye Jeon

Construction of an ultra low temperature STM using a Dilution Refrigerator

희석냉각기를 이용한 초저온 주사 터널링 현미경의 구축

February 2018

Graduate School of Seoul National University
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이 논문을 이학석사 학위논문으로 제출함
2018년 2월

서울대학교 대학원
물리천문학부
전 세 계

전세계의 이학석사 학위논문을 인준함
2017년 12월

위원장 최석봉

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Construction of an ultra low temperature STM using a Dilution Refrigerator

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A Dissertation
Submitted to the Faculty of
Seoul National University
in Partial Fulfillment of
the Requirements for the Degree of
Master of Philosophy

February 2018

Graduate School of
Seoul National University
Department of Physics & Astronomy
Abstract

A scanning tunneling microscope (STM) is widely used in the condensed matter physics which can visualize a surface of a solid and accomplish a spectroscopy imaging with differential conductance measurements. For our STM, a dilution refrigerator is integrated to investigate novel phenomena in the ultra low temperature (DR-STM).

With a dilution refrigerator (DR), the scanning tunneling microscopy is carried out at the ultra low temperature. A helium mixture of $^3$He and $^4$He is used for a DR cooling and circulated continuously by a closed cycle circuit. The DR has several components and involves perplexing operation methods. A description and an operation procedure of the DR are specified to establish a basic operation of the DR.

A target sample of the STM is prepared at an ambient condition and transferred into a cryogenic and an ultra high vacuum (UHV). A special sample transfer rod is used for transferring the sample to the STM by a top-loading method. The sample is transferred from the top of the sample transfer passage to the STM head located at the bottom of the sample transfer passage. In the middle of a sample transfer passage, a sample cleaving stage is placed to prepare a clean and flat target sample surface. An entire sample
transfer procedure and a fine-tuning list of the sample transfer components are described to perform the sample transfer operation proficiently.

An experimental room has a vibration isolation system and the scanning tunneling microscopy is accomplished in the ultra low vibration. Experimental equipment is designed to be easily operated and to efficiently utilize the experimental room. For instance, a gas panel makes easy evacuation control for the dewar and a vacuum chamber of the STM. Also, a dewar is stored at the base of the experimental room and lifted up to utilize the experimental room as much as possible.

In this thesis, general equipment operations and explanations will be described to accomplish the STM at ultra low temperature and ultra low vibration with DR and the vibration isolation system.

Keywords : Scanning tunneling microscopy, sample transfer, cleaver, dilution refrigerator, leak-detect, dewar lifting, gas panel

Student Number : 2016-20318
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Chapter 1. Scanning Tunneling Microscopy

1.1. Physics of Scanning tunneling microscopy

Scanning tunneling microscopy (STM), invented by Binnig and Rohrer in 1980s\textsuperscript{1-3}, is one of the most powerful tools to investigate the atomic scale phenomena\textsuperscript{4-6}. The basic principle associated with the STM is an electron tunneling between the sample surface and the STM tip. The STM has achieved atomic resolution imaging by the exponential dependence of the tunneling current on the tip-to-sample distance. The STM measures the physical surface corrugation of the sample (topography) by measuring the distance between the tip and the sample via the tunneling current\textsuperscript{7}. The STM can also be viewed as a spectroscopic tool by measuring the locally resolved electronic structure with the differential conductance measurement\textsuperscript{7}. Furthermore, STM has a capability for a spectroscopic imaging in real space by measuring the differential conductance at every point on the surface and is called the spectroscopic imaging – scanning tunneling microscopy (SI-STM)\textsuperscript{8}. 
**Figure 1.** Electron tunneling between a sample and a tip with a negative bias voltage \(-V\) applied to the sample.

The electron tunneling between the tip and the sample can be described by a simple model, which is called the one-dimensional Bardeen model\(^7\). As the tip and the sample gets closer, the Schrödinger equation of the combined system with the tip and the sample is
\[ i\hbar \frac{\partial \psi}{\partial t} = \left( -\frac{\hbar^2}{2m} \frac{\partial^2}{\partial z^2} + U_{\text{tip}} + U_{\text{sample}} \right) \psi \]  \hspace{1cm} (1) 

The \( \psi \) is a wave function of the combined system with a linear combination of wave functions with the sample and the tip. The wave functions of the tip and the sample are assumed as orthogonal for each other. It is also assumed that an electron of the sample tunnels to the tip, and the wave function of the sample evolves with the wave function of the tip.

\[ \psi = A_x(t)\psi_x^{\text{sample}} e^{\frac{iE_x^{\text{sample}} t}{\hbar}} + \sum_y B_y(t)\psi_y^{\text{tip}} e^{\frac{iE_y^{\text{tip}} t}{\hbar}} \]  \hspace{1cm} (2) 

Inserting (2) into (1), and projecting on a \( \psi_y^{\text{tip}} \) state,

\[
\begin{align*}
 i\hbar \frac{dB_y(t)}{dt} &= A_x(t) \langle \psi_y^{\text{tip}} | U_{\text{tip}} | \psi_x^{\text{sample}} \rangle e^{\frac{i(E_x^{\text{sample}} - E_y^{\text{tip}}) t}{\hbar}} \\
 &\quad + \sum_z B_z(t) \langle \psi_y^{\text{tip}} | U_{\text{sample}} | \psi_z^{\text{tip}} \rangle e^{\frac{-i(E_z^{\text{tip}} - E_y^{\text{tip}}) t}{\hbar}} \hspace{1cm} (3)
\end{align*}
\]

At first, the electron starts to tunnel from the sample. So \( A_x(t) = 1 \), and all \( B_y(t) = 0 \). The equation (3) can be solved in the first-order time dependent perturbation.

\[ i\hbar \frac{dB_y(t)}{dt} = \langle \psi_y^{\text{tip}} | U_{\text{tip}} | \psi_x^{\text{sample}} \rangle e^{\frac{i(E_x^{\text{sample}} - E_y^{\text{tip}}) t}{\hbar}} \]  \hspace{1cm} (4)

A tunneling matrix \( M_{xy} \) is defined as \( \langle \psi_y^{\text{tip}} | U_{\text{tip}} | \psi_x^{\text{sample}} \rangle \).
Integrating the equation (3) with time yields

\[
B_y(t) = \frac{e^{-(E_x^{sample} - E_y^{tip})t/h}}{E_x^{sample} - E_y^{tip}} M_{xy}^{-1}
\]

(5)

The total probability that an electron of the sample state tunnels into all available tip states at time \(t\) is

\[
p_{xy}(t) \equiv \sum_y |B_y(t)|^2 = \sum_y |M_{xy}|^2 \frac{2 \sin^2 \left( \frac{(E_x^{sample} - E_y^{tip})^2}{2h} \right)}{(E_x^{sample} - E_y^{tip})^2}
\]

(6)

The summation with discrete tip states of the right side in (6) can be replaced with an integration with a density of states of the tip over the energy.

\[
p_{xy}(t) = \int_{-\infty}^{\infty} |M_{xy}|^2 n_{Tip}(E_y^{Tip}) \frac{4 \sin^2 \left( \frac{(E_x^{sample} - E_y^{tip})^2}{2h} \right)}{(E_x^{sample} - E_y^{tip})^2} dE_y^{Tip}
\]

(7)

The \(B_y(t)\) is a function which has a maximum value when \(E_x^{sample} = E_y^{tip}\), and approaches to zero for \(E_x^{sample} \neq E_y^{tip}\). So the integrand of the (7) only gives non-zero value around \(E_x^{sample} = E_y^{tip}\) region which is a condition of the elastic tunneling. In that region, the tunneling matrix \(|M_{xy}|\) and the density of states of tip \(n_{Tip}(E_y^{Tip})\) is assumed as a constant. In
that region, \( n^{Tip}(E_y^{Tip}) \) becomes \( n^{Tip}(E_x^{Sample}) \). With using a relation \( \int_{-\infty}^{\infty} \frac{\sin^2 ax}{\pi ax^2} \, dx = 1 \), equation (7) can be calculated as

\[
p_{xy}(t) = \left| M_{xy} \right|^2 n^{Tip}(E_x^{Sample}) \int_{-\infty}^{\infty} \frac{4\sin^2 \left( \frac{(E_x^{sample} - E_y^{Tip})t}{2\hbar} \right)}{(E_x^{sample} - E_y^{Tip})^2} \, dE_y^{Tip}
\]

\[= \frac{2\pi}{\hbar} \left| M_{xy} \right|^2 n^{Tip}(E_x^{Sample}) t
\]

(8)

The tunneling current is given by the product of the total probability that an electron of the sample state tunnels into all available tip states and the total number of electrons which can tunnel from the sample. Moreover, all possible states for electron tunneling also need to be taken into account. First, occupied states of the sample are described by the Fermi-Dirac distribution as

\[ f(E) = \frac{1}{1 + \exp \left[ \frac{E - E_F}{k_B T} \right]} \].

Second, unoccupied states of the tip are described by \( 1 - f(E) \). Third, since one electron state must be considered in the spin up state and spin down state, every single state of the electron tunneling must be considered twice. When the bias voltage is \(-V\),
\[ I_{\text{Sample to Tip}} = \frac{4\pi e}{h} \int_{-\infty}^{\infty} d\varepsilon \ f\left(E_F^{\text{Sample}} + eV + \varepsilon\right) \left[1 - f\left(E_F^{\text{Tip}} + \varepsilon\right)\right] \]
\[ \times \ n^{\text{Tip}}(E_F^{\text{Tip}} + \varepsilon)n^{\text{Sample}}(E_F^{\text{Sample}} + eV + \varepsilon)|M_{xy}|^2 \] (9)

The tip-to-sample current also need to be considered as the sample-to-tip current is considered. The total tunneling current can be described with the difference of the two currents.

\[ I = \frac{4\pi e}{h} \int_{-\infty}^{\infty} d\varepsilon \ [f\left(E_F^{\text{Tip}} + \varepsilon\right) - f\left(E_F^{\text{Sample}} + eV + \varepsilon\right)] \]
\[ \times \ n^{\text{Tip}}(E_F^{\text{Tip}} + \varepsilon)n^{\text{Sample}}(E_F^{\text{Sample}} + eV + \varepsilon)|M_{xy}|^2 \] (10)

If the $k_B T$ is smaller than the energy resolution, the Fermi-Dirac distributions are replaced as step-functions.

\[ I = \frac{4ne}{h} \int_{-eV}^{0} d\varepsilon \ n^{\text{Tip}}(E_F^{\text{Tip}} + \varepsilon)n^{\text{Sample}}(E_F^{\text{Sample}} + eV + \varepsilon)|M_{xy}|^2 \] (11)

The tunneling matrix $M_{xy}$ defined as $\langle \psi_y^{\text{Tip}} | U_{\text{tip}} | \psi_x^{\text{Sample}} \rangle$ can be calculated when the junction is planer and the elastic tunneling occurs.

\[ \langle \psi_y^{\text{Tip}}(0)|\exp(-\kappa_{xy} z) U_{\text{tip}} \exp[\kappa_{xy}^{\text{Sample}}(z-s)]|\psi_x^{\text{Sample}}(0+s) \rangle = me^{-\kappa s} \] (12)

The $m$ is the tunneling matrix constant. The $s$ is the distance between the tip to the sample. The $\kappa = \sqrt{2mE_x^{\text{Sample}}/h}$ is the decay
constant. Because of the elastic tunneling condition, $\kappa = \kappa_{y}^{Tip} = \kappa_{x}^{Sample}$. In the planar junction, the tunneling current can be described in a simple proportional equation as

$$I \propto \int_{-eV}^{0} d\varepsilon \quad n^{Tip}(E_{F} + \varepsilon) \quad n^{Sample}(E_{F} + eV + \varepsilon) \quad e^{-2\kappa s}$$ (13)

For the topography, the tunneling current $I$ in the left side of the equation (13) is determined with a tunneling current constant. The distance between the tip and the sample $s$ is adjusted to maintain the same tunneling current as the tunneling current constant. As the tunneling current has the exponential dependence on the tip-to-sample distance, the STM can achieve a high resolution topography$^7$.

STM can measure the local density of states on the sample. The right hand side of (11) is written again after changing the interval of integration to $\left[-\frac{1}{2}eV, +\frac{1}{2}eV\right]$.

$$I = \frac{4\pi e}{h} \int_{\frac{-1}{2}eV}^{\frac{1}{2}eV} d\varepsilon \quad n^{Tip}(E_{F}^{Tip} - \frac{1}{2}eV + \varepsilon)n^{Sample}(E_{F}^{Sample} + \frac{1}{2}eV + \varepsilon)|M_{xy}|^2$$ (14)

The decay constant can be written with the work function.

$$\kappa = \frac{\sqrt{2mE^{sample}_{x}}}{\hbar} = \frac{\sqrt{2m(\phi_{work \ function}^{sample} - \varepsilon)}}{\hbar}$$ (15)
Assuming the bias interval $|eV|$ is always smaller than the work function $\phi_{\text{work function}}^{\text{sample}}$, the decay constant can be approximated as

$$\kappa \approx \sqrt{\frac{2m\phi_{\text{work function}}^{\text{sample}}}{\hbar}} \left(1 - \frac{\varepsilon}{2\phi_{\text{work function}}^{\text{sample}}}\right) \equiv \kappa_0 \left(1 - \frac{\varepsilon}{2\Phi}\right) \quad (16)$$

Following (12), the tunneling current is given by

$$I \propto \int_{-\frac{1}{2}eV}^{\frac{1}{2}eV} d\varepsilon \quad n^{\text{tip}} \left(E_F^{\text{tip}} - \frac{1}{2}eV + \varepsilon\right) n^{\text{Sample}} \left(E_F^{\text{Sample}} + \frac{1}{2}eV + \varepsilon\right)$$

$$\times e^{-2\kappa_0 s} \quad e^{\frac{\kappa_0 \varepsilon}{\Phi}} \quad (17)$$

The integrand has an exponential dependence on the energy $\varepsilon$, and the main contribution of the integral comes from around $\varepsilon \approx \frac{1}{2}eV$. Replacing $\varepsilon$ as $eV'$ on (17) and differentiate with $V'$ yields

$$\left.\frac{dI}{dV'}\right|_{V'=-\frac{1}{2}V} \propto n^{\text{tip}} \left(E_F^{\text{tip}}\right) n^{\text{Sample}} \left(E_F^{\text{Sample}} + eV\right) \quad (18)$$

Assuming that the density of states of the tip is a constant, the local density of states of the sample is obtained with a $\frac{dI}{dV}$ curve\textsuperscript{7}. A bias voltage is swept within a certain range while pausing the tip. (i.e., -100 mV to +100 mV). The tunneling current measurement with the bias sweep results in a $I(V)$ curve.
Numerical differentiation of the $I(V)$ curve gives a $\frac{dI}{dV}$ curve. However, a lock-in technique can produce more clear spectra, and the spectra are less affected by noise. In the lock-in technique, small voltage modulation applies to the sample bias as $V = V_0 + V_{\text{modulation}} \cos(\omega t)$. Expanding $I(V)$ by Taylor series,

$$I(V) = I(V_0) + \left( \frac{dI}{dV} \right)_{V = V_0} V_{\text{modulation}} \cos(\omega t) + O^2 \quad (19)$$

By choosing a sinusoidal current with at frequency $\omega$, the local density of states is obtained with a $\frac{dI}{dV}$ curve which is called a differential conductance. SR830 DSP Lock-In Amplifier by Stanford Research Systems is used for measuring the $\frac{dI}{dV}$ value.

A map of the local density of states can be produced over the sample surface. At each point on the map, the tip is suspended at a constant height and the bias voltage is swept within a certain range. The differential conductance measurement is repeated until all differential conductance measurement are completed on all points. The spectroscopic image can be obtained as a function of $(x, y, V)$. For example, a field of view (FOV) of the map is divided as 256*256 points in a 500Å square area. An interval of the bias
sweep is divided by 101 points. The spectroscopic image is produced with a 256*256 (spatial)*101 (energy) data map. The spectroscopy is measured in 3 seconds in one point. Imaging one spectroscopic image takes at least three days. If the tip is not stable during the spectroscopic imaging, the differential conductance measurement cannot be proceeded and started again from the beginning. It is most important to isolate the STM from the external noise and to prepare a stable tip as not to deform the tip during the spectroscopic imaging.

In the spectroscopic image, there can be a modulations which can be explained with elastic quasi-particles scattering\(^8\). A definition of the local density of states (LDOS) is described as

\[
\text{LDOS, } n(r, E = eV) = \sum_k |\psi_k|^2 \delta(\varepsilon_k - eV) \tag{20}
\]

For an ideal metal surface, the local density of states contains modulations due to the periodicity of the lattice structure. Since the wave function is described as a Bloch function \( \psi_k = e^{i\mathbf{k} \cdot \mathbf{r}} u_k(\mathbf{r}) \), the local density of states would be homogenous on the entire sample surface. However, an actual metal surface has the crystal imperfection such as impurities or line defects\(^8\) which make
the spatial LDOS modulations and the modulations are also called as the Friedel oscillations. The crystal imperfection such as impurities make an elastic scattering between Bloch states as

\[ |\psi_{k}\|^2 = \left| a e^{i \vec{k}_1 \cdot \vec{r}} u_{\vec{k}_1}(\vec{r}) + b e^{i \vec{k}_2 \cdot \vec{r}} u_{\vec{k}_2}(\vec{r}) \right|^2 \]

\[ = \left| a u_{\vec{k}_1}(\vec{r}) \right|^2 + \left| b u_{\vec{k}_2}(\vec{r}) \right|^2 + a e^{i \vec{k}_1 \cdot \vec{r}} u_{\vec{k}_1}(\vec{r}) \cdot b^* u_{\vec{k}_2}^*(r) e^{-i \vec{k}_2 \cdot \vec{r}} \]

\[ + a^* e^{-i \vec{k}_1 \cdot \vec{r}} u_{\vec{k}_1}^*(\vec{r}) \cdot b u_{\vec{k}_2}(r) e^{i \vec{k}_2 \cdot \vec{r}} \]

(21)

The LDOS modulations can be described with this mixed states and observed in the spectroscopic image.

1.2. Head of Scanning Tunneling Microscopy

The STM Head is a core part where an actual STM experiment is accomplished. In the head, a tip and a sample receptacle are located. In the sample receptacle, the stud can be inserted by the sample transfer rod. The concept of the stud and the sample transfer rod will be presented in section 2.1. On a stud surface, a sample is prepared with a clean surface by the cleaving method. The concept of the cleaving method will be presented in section 2.2.
A walker and a piezoelectric tube are used for a tip-approach to the sample. The walker is used for a coarse tip-approach to the sample and the piezoelectric tube is used for a fine tip-approach to the sample. The STM tip is attached to the top of the piezoelectric tube. The length of piezoelectric tube finely increases until the tunneling current flows between the tip and the sample. If there is no current at the maximum length, the piezoelectric tube contracts to its original length.

![Diagram](image.png)

**Figure 2.** The STM head schematics. Left: 3D modeling of the STM head. Right: sketch of the piezoelectric tube.

If there is no tunneling current, the tip should coarsely approach to the sample in one step, which is a purpose of the walker. The walker has six piezo stacks. The walker takes one step to the sample by applying a high voltage with saw-tooth waveform to the piezo stacks. The coarse tip-approach and the fine tip-approach approach.
are repeated until the tunneling current flows between the tip and the sample. The z position of the tip can be adjusted by the tip-approach process. An x-y position of the tip can be adjusted with the four piezoelectric actuators (+x, -x, +y, -y) which are used for scanning on the sample surface by a lateral direction as the figure 2 (Right).

For the spectroscopic imaging, the tip stability is critical to obtain the reproducible spectroscopy data for all pixels because it takes at least three days to produce only one spectroscopic image. A tip treatment can be a solution to make the reproducible tip\(^7\). For the treatment, a high voltage is applied to the gold target and field emission (FE) current flows to the tip. As the current ramps up, the high voltage field emission applies to the tip and a sharp decrease of the current can be observed at moment. The sharp decrease of the current can be interpreted that the tip is prepared as a reproducible tip to produce the spectroscopic image.

Even with the advantage of the reproducible tip, the field emission technique is somewhat challenging. For conventional STM experiments, a sample should be replaced with a gold target before proceeding the FE. After a tip treatment, the sample should
be loaded at the receptacle again. There are several problems with the conventional method. Whenever reinserting the sample, a sample cleaving should be done again to get a clean and flat surface. It also takes lots of time to wait for the UHV, cryogenic condition, and tip-approach.

![Figure 3. in-situ field emission stage in a 3D modeling. Left: Sample approach mode. Right: Gold approach mode.](image)

With the improvement of the head, *in-situ* tip treatment is performed via the built-in field emission stage\(^{17}\). The field emission stage has a gold target for the field emission. A high voltage can be applied to the gold target for the field emission. The field emission stage is located between the tip and the sample receptacle. When the field emission is needed, the tip is retracted completely from the sample. Then the field emission stage is rotated along with a vertical axis of the head, and the gold target is

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brought out to a tip path. Tip-approach ends at the gold target which enables the special tip treatment.

Chapter 2. Sample Transfer system

2.1. Components of sample transfer rod

In our STM experiment, a sample is prepared at the ambient pressure and room temperature and transferred into the STM chamber with the UHV and the cryogenic state. The sample can be loaded into and unloaded from the sample receptacle by a special sample transfer rod. The sample transfer rod is composed of three parts: a stud, a grabber, and rods.

Figure 4. The end of the sample transfer rod. The size of the sample is smaller than the diameter of a cleaving post.
Figure 5. The stud and the grabber.

The stud is used for loading the sample into the STM head. The sample is attached on a stud surface with silver epoxy. A post is set up on the sample surface and attached with silver epoxy. This post is used to cleave the sample. The stud has a wing-like shape. As the stud passes through a transfer passage, there are several keyholes following one after another. The keyhole has the same wing-like shape of the stud. The keyhole is depicted in the figure 6. During the sample transfer, the stud wing can press the keyhole when the stud wing and the keyhole are perpendicular to each other.
For example, the sample is cleaved by pressing a keyhole of the push plate and the sample can be loaded on the head by pulling out the sample transfer rod with orienting the stud wing and the keyhole of the head in perpendicular. The sample cleaving and the sample loading on the head is discussed in section 2.3. The stud can pass through the keyhole when the stud wing and the keyhole are parallel to each other. Pressing or passing the keyhole repeatedly, the stud can finally be loaded into the head.

**Figure 6.** Keyholes in the sample transfer passage. Oxygen free copper transfer guide has a keyhole. Below the guide, the STM head is located. A keyhole also exists at the top of the head.
The height of the sample transfer passage with the insert is about 2.5m. However, the height of the acoustic room is limited. The sample transfer rod is separated into three pieces. Each rod can be fastened by the screw mechanism. For this reason, the transfer rod is always rotated in the clockwise direction for every keyhole operation. If the transfer rod is rotated in the counter-clockwise direction, rods can be loosen inside the sample transfer passage. There is a vent hole at the female threads hole of each rod. Because the air inside the threads can break the UHV, all vent holes must be evacuated during the sample transfer.

If the rod and the stud are engaged by the screw mechanism, the stud and the rod cannot be engaged inside of the transfer passage. A special adaptor is required between the rod and the stud and is called a grabber. The grabber is fastened with a rod by the screw mechanism. The grabber and the stud is joined with the tongue and groove joint mechanism and the grabber holds the stud with a spring. If a stronger force is applied than the spring, the stud can be separated or engaged with the grabber.

There are two holes on both sides of the grabber and two SUS balls are engaged with the two holes. Two balls are slightly larger
than the holes on both sides, so they cannot pass through the holes. The two balls acts as the tongue of the joint. A BeCu ring holds two balls by covering the sides of the grabber, which acts as a plate spring. The end of the stud has a horn-like shape which acts as the groove of the joint.

**Figure 7.** The Grabber structure. (a) Grabber with a BeCu ring holding two SUS ball. (b) Bottom view of a grabber. (c) The tongue part of the joint. (d) Engaged joint between a stud and a grabber
If the spring force is too strong, the grabber and the stud cannot be engaged or separated by hand. If the spring force is too weak, the BeCu cannot act as a spring and the grabber cannot hold the stud. A 0.6mm thickness BeCu ring satisfies the appropriate condition. There are three transfer guides with a keyhole to ensure the sample transfer rod to be straight down in the transfer passage. Any part of the sample transfer rod should not intrude tidy keyholes for all guides. So two wingless grabber sides of BeCu ring are grinded not to intrude keyholes. A silver epoxy block is formed between the ends of the BeCu ring, since the BeCu ring can rotate and two balls can escape from the grabber without the silver epoxy block.

2.2. Cleaver

For our STM, the sample is prepared at an ambient condition and transferred into a cryogenic and an ultra high vacuum (UHV). Loading the sample directly to the STM head causes the bad surface states. The cleaver is used for the *in-situ* sample surface preparation in the UHV and cryogenic state\(^{17}\). After the sample cleaving with the *in-situ* cleaver, the sample is prepared with a clean and flat surface.
Figure 8. Cleaver schematics

After preparing the sample on the stud, silver epoxy is applied on the sample and a cleaving post is set up on the stud. The cleaver knocks off the cleaving post and the sample surface in contact with the silver epoxy is cleaved in one atomic layer as the post falls off from the sample surface. The cleaver consists of an upper keyhole, a push plate, a cleaving basket and radiation blocks\textsuperscript{17}.
The upper keyhole is adjoined to the 4 K plate and is called as the keyhole of 4 K plate. The keyhole of the 4 K plate prevents the sample from being transferred without sufficient cooling. After the keyhole of the 4 K plate, there is a push plate with a key hole which is connected with a cleaving basket knocking off the cleaving post. The knocking off operation is depicted in the figure 9. When the push plate is pressed down, the cleaving basket begins rotating downside. The cleaving basket is equipped with a horizontal post and it is called a hammering pin. This hammering pin knocks off the cleaving post on the stud. A spring connects between the cleaving basket and the cleaver body. When there is no force to press down the push plate, the cleaving basket rotates upward to its original position by the spring.

The tension of the spring is important for rotating the cleaving basket. If it is too weak, the gravity of the cleaving basket is strong than the tension, and the cleaving basket keeps rotated downward without pressing down the push plate. In this case, the cleaving basket always blocks the transfer passage and the sample transfer cannot be proceeded. If it is too strong, the cleaving basket does not move no matter how hard the push plate is pressed by hand, so the cleaver does not work.
**Figure 9.** The sample cleaving process. (a) Before the sample cleaving. The stud is on the push plate, and does not press down the push plate. (b) The sample cleaving is in progress. The cleaving basket rotates downside by pressing down the push plate. (c) Front view during the cleaving process. The hammering pin in the cleaving basket knocks off the cleaving post. (d) Side view during the cleaving process.

The fallen off cleaving post enters the cleaving basket. In the actual sample cleaving operation, it is important to put this fallen post into the basket, since the fallen post may fall in the transfer
passage. In the worst situation, the fallen post enters into the keyhole of the head, and the stud cannot be loaded into the head. The cleaving basket should rotate downward fully so that there is no gap between the cleaving basket and the cleaver body and the fallen post enters into the cleaving basket properly. The proper operation of the cleaver is achieved by fine-tunings such as adjusting the spring tension and smoothing the cleaver body sides and the cleaving basket.

Also, when the cleaving post is too long, it cannot enter the basket after the sample cleaving. However, when it is too short, the hammering pin cannot knock off the cleaving post. The length of the cleaving post is confirmed as 14mm for the optimal length.

2.3. Sample transfer operation

The sample is transferred with the sample transfer rod by hand. A fatal failures may occur if the sample transfer operation is done without sufficient skill by hand. For example, the sample may not be properly cleaved and a condition of the sample surface becomes bad. If the sample is taken out in a wrong way, the stud may fall and block the transfer passage.
The sample is loaded into the STM head via the transfer passage. At the top of the transfer passage, the sample transfer rod is installed and at the bottom of the transfer passage, the STM head is located. It is called a top-loading method for the sample transfer. The sample transfer passage also acts as a STM chamber which is maintained as UHV.

Before the beginning of the sample transfer, straightness of the sample transfer rod should be ensured by rolling rods on a flat surface. If rods do not roll, the transfer rod does not go down straight but goes down obliquely. In this case, the stud is directed outward from the head. The entire sample transfer rod is depicted in figure 10.

A Teflon piece is engaged with the transfer rod. The Teflon piece ensures that the transfer rod is fixed at the center position of the transfer passage. When the Teflon piece is dropped from the top of the rod and to the end of the rod, the Teflon piece must not be caught in the middle of the transfer rod. If the Teflon piece does not fall to the fixture, the Teflon piece would be stuck at the middle of transfer passages. The transfer rod would be biased as off-center. It causes the transfer rod goes down obliquely and an
irreproducible sample transfer. In that case, an inner diameter of the Teflon piece should become larger.

Figure 10. The sample transfer rod. Left: installed on the insert. Right: engaged transfer rods.

A 4-way-cross is engaged with the transfer rod up to the Teflon
piece. The transfer rod passes through the upper and down flanges. At both sides, Viton O-rings are engaged to maintain a vacuum level of the 4-way-cross during the sample transfer. On the left and the right sides there are NW16 flanges. One side is connected with a rotary pump by bellows and the other side is connected with a vacuum gauge to check the pressure of 4-way-cross. The pressure of the STM is maintained as UHV and exposing the STM with the ambient circumstance breaks the UHV. So a small buffer with low vacuum level by a rotary pump prevents the sudden break of the UHV.

A Bakelite rod holder is engaged with the top of 4-way-cross. Tightening the holder can maintain the height of the transfer rod and prevent the transfer rod from accidentally falling due to the gravity or pressure differences between upside and downside of the sample transfer rod in the transfer passage. The Bakelite rod holder is used for changing an orientation of the stud wing at the keyholes or evacuating the vent holes of the sample transfer rod.

Vacuum grease may be lightly applied at the top of the 4-way-cross. As the transfer rod passes 4-way-cross, vacuum grease is applied to the O-ring of the top flange and the O-ring applies
vacuum grease to the entire rods. Apiezon N grease is preferred in the cryogenics. If the grease is applied too much, the sample transfer rod becomes slippery and the sample transfer operation becomes difficult.

Since the cleaving post can fall off while engaging parts of the transfer rod, the grabber and the stud are always engaged at the last time. The transfer rod is finally installed on the insert. It must be ensured that the cleaving post is firmly attached to the stud viewed through the Viewport. After evacuating the 4-way-cross vacuum buffer with a rotary pump, the gate valve of the insert is opened. The sample transfer begins when the transfer passage pressure is 10E-6 mbar.

Three separated rods should be engaged into one rod while pushing down the transfer rod to the inside of the sample transfer passage. The two vent holes of the rods must be evacuated during the sample transfer. After engaging all three rods, a laser pointer is engaged with the top of the transfer rod. The laser point records transfer positions and directions at each the keyholes and provides a reproducible sample transfer.
Figure 11. Keyhole description. Left: Keyhole mechanism at the 4 K plate. Right: locations of keyholes of the insert. There are six keyholes in the transfer passage.

When the sample transfer rod stops to go down, the sample transfer rod reaches the first keyhole at the 4 K plate. The stud wing and the keyhole of the 4 K plate are perpendicular to each other so that the transfer rod shall not pass. The sample needs cool
down about one hour. After cooling down the sample, the transfer rod passes through the keyhole of the 4 K plate. The sample transfer rod enters the cleaver adjoined just below the 4 K plate.

Inside the cleaver, a push plate with a keyhole exists which operates the sample cleaving. The keyholes of the 4 K plate and the push plate are perpendicular each other, so the sample cleaving is proceeded as pressing down the push plate by the sample transfer rod without changing the orientation of the stud wing. The sample is cleaved as a hammering pin knocks off the cleaving post in one direction. Since the other side of the sample may not be cleaved, the sample cleaving should be done again by rotating the wing orientation by 180 degrees.

After the sample cleaving, the orientation of the wing is rotated by 90 degrees and the sample transfer rod passes through the keyhole of the push plate. After the push plate, radiation blocks are engaged with the cleaver. The radiation blocks keep off the transfer passage. The radiation blocks are unfolded by gently pushing down the transfer rod and the transfer rod can pass by the cleaver.

After the sample transfer rod passes by the cleaver, there are three transfer guides with a keyhole. They ensure the end of rod
reaches at the center of the head. The orientations of three keyholes are different each other, so the orientation of the stud should be adjusted for each key hole of the sample transfer guide.

![Images](image1.png)

(a) Before the sample transfer  (b) Inserted stud on the head  (c) Detached stud after rotating grabber by 90 degrees

**Figure 12.** Inserting the sample in the keyhole of the head

The stud now reaches at the top of the head. The stud is inserted into the keyhole of the head. If the rod is pulled out, the stud will come out from the head with unloaded. So before pulling out the rod from the head, the orientation of the stud wing should be rotated by 90 degrees. By pulling the transfer rod out of the head, the stud is loaded in the keyhole of the head. In other words, the stud is stuck by the keyhole of the head and the stud is loaded at the sample receptacle.

For taking out the sample from the head, the transfer rod is pushed down to the head while orienting the grabber with all
keyholes in the transfer passage because there is also a wing on the grabber. The loaded stud and the grabber are engaged together by pushing down the sample transfer rod and rotating the sample transfer rod in clockwise.

After the stud and the grabber are engaged together, the sample can be unloaded from the sample receptacle by aligning the keyhole of the head and the stud wing in parallel and pulling out the transfer rod. If the orientations are not parallel, the transfer rod and the stud are separated again. The orientations of the stud wing and the keyhole of the head can be in parallel by the laser-point records.

(a) Push plate with upper position   (b) Push plate with original position

**Figure 13.** Failure while taking out the sample
While taking out the stud, the stud may fall off in the cleaver. The failure arises from a poor operation of the push plate. For an unknown reason, the push plate is stuck in the middle of the cleaver and does not keep its original position. The original position of the push plate is designed to the lowest position where the push plate can be located in the cleaver. Sometimes, the push plate is located at the upper position than its original position. In this case, the stud and the grabber are separated and stud falls off by rotating the sample transfer rod when the stud wing is placed at the middle of the keyhole of the push plate. The push plate with upper position can be corrected by gently rotating the transfer rod on both directions with a small movement not to extend over the keyhole. The push plate can return to its original location, and the stud can be taken out safely. This is achieved by a fine-tuning of the push plate. The original push plate has the right edges. They make a hard friction between the cleaver body and the push plate. By smoothing the push plate edges, the push plate can move smoothly.
Chapter 3. Dilution Refrigerator

3.1. Physics of Dilution refrigerator

In 1962, the dilution refrigerator (DR) is first made by H. London, G.R. Clarke, and E. Mendoza. DR is a powerful experimental tool for the cryogenic states. The base temperature of our DR can be lowered less than 7 mK by the mixing two phases of the mixture of $^3$He and $^4$He. The DR is the optimum cryogenic device for the STM because there is no mechanical drive part, so it has a very low vibration.

![Diagram of phase transition in $^3$He and $^4$He mixtures.](Image)

**Figure 14.** Mixture in the cryogenic state. Left: the phase diagram of the mixture. Right: The separation of the mixture with two layers. There is a phase boundary between the concentrated phase, and the diluted phase.
DR uses liquid $^3$He and liquid $^4$He mixtures for the cooling. The DR is equipped with a closed cycle circuit filled with the mixture. The DR cooling occurs when a mixing chamber in the closed cycle circuit is cooled down below than the triple point of the mixture at a temperature of 0.87 K$^{14}$. The mixture is separated into two layers as the $^3$He rich state (concentrated state) and the $^3$He poor state (diluted state). Because the $^4$He is heavier than the $^3$He, the concentrated state floats up and the diluted state sinks down.

When the phase separation occurs in the mixture, $^3$He molecules moves from the concentrated state to the diluted state at the phase boundary. There is an enthalpy difference of the $^3$He molecules between the concentrated state and the diluted state and it produces the cooling power of the DR.

The DR cooling can be understood with the $^4$He evaporation cooling where the liquid $^4$He evaporates into a vacuum background to obtain the cooling power by the latent heat of evaporation$^{14}$. The comparison between the $^4$He evaporation cooling and the DR cooling is depicted in the figure 15. In the dilution cooling, concentrated $^3$He molecules moves to diluted states where the liquid $^4$He corresponds to the vacuum background.
It can be interpreted as the phase of the $^3$He molecules changes from the $^3$He liquid state to the $^3$He gas state$^{15}$. The latent heat of mixing the $^3$He molecules between the concentrated state and the diluted state produces the cooling power.

**Figure 15.** Comparison between evaporation cooling and DR cooling. Left: The latent heat of evaporation produces the cooling power of the $^4$He evaporation refrigerator. Right: The latent heat of mixing the two states produces the cooling power of the DR.

$^{14}$The cooling power of liquid $^4$He evaporation is given as $\dot{Q} \propto e^{-1/T}$. The exponential decays of the cooling power to the temperature can be interpreted as there are no evaporated
molecules and the evaporation cooling ends up.  

The cooling power of DR is given as $\dot{Q} \propto T^2$. Contrary to the evaporation cooling, the DR cooling has continuous molecule flows between the concentrated state and the diluted state no matter how temperature is lowered. For this reason, DR has lower base temperature than the liquid $^4$He evaporation cooling.

The cooling power of DR can be described by mixing the two states

$$\dot{Q} = \bar{n}_3 [H_{diluted} - H_{concentrated}] \quad (22)$$

For the novel phenomena of the mixture, the solubility of $^3$He molecules in the diluted state is maintained as 6.4%\(^{15}\). If the solubility of the $^3$He becomes lower than 6.4% in the diluted state, than $^3$He molecules are supplemented to the diluted state from the concentrated and the $^3$He molecules flow rate $\bar{n}_3$ produces the cooling power. The solubility of the diluted state can be decreased than 6.4% by evaporating only $^3$He molecules from the mixture by a fractional distillation. The evaporated $^3$He molecules are condensed and return to the concentrated phase layer of the mixing chamber. The $^3$He molecules flow rate $\bar{n}_3$ is maintained and the DR cooling can occur continuously compared to the evaporation
cooling\(^{15}\). A cycle of \(^3\)He molecules will be presented in the next section. The enthalpy difference between the diluted state and the concentrated state is described as

\[
H(T) - H(0) = \int_0^T dT \ C(T)
\]

(23)

By the isotope characteristics of Helium, heat capacity inequality is always given as\(^{14}\)

\[
C_{diluted}(T) > C_{concentrated}(T)
\]

(24)

The enthalpy difference is always positive, and \(C(T)\) is proportional to \(T\) at low temperature because of the fermi characteristic of \(^3\)He. So the cooling power of DR is given as\(^{14}\)

\[
\dot{Q} = n_3 \Delta H \propto T^2
\]

(25)

The cooling power is produced until the DR is cooled down to zero kelvin. However, the thermal resistance called Kapitza resistance and viscous heating inhibit the temperature falls to zero temperature.\(^{14}\)

The higher heat capacity and the finite solubility of \(^3\)He molecules in the diluted state can be explained with the isotope properties of the \(^3\)He and the \(^4\)He. The ground state energy of the
helium is described as\(^{14}\)

\[ E_0 = \frac{\hbar^2}{8ma^2} \quad (26) \]

The \(^3\)He has higher ground state energy than the \(^4\)He because of its smaller mass, and the vibration amplitude of the ground state is high in \(^3\)He. So \(^3\)He has a larger molar volume than the \(^4\)He.

**Figure 16.** Phase boundary explanation at the mixing chamber. Left: \(^3\)He at the boundary moves to \(^4\)He concentrated phase because the van der Waals force is strong as the molecular distance becomes shorter. Right: Chemical potential of \(^3\)He in Diluted phase. Equilibrium can be achieved at \(x=6.4\%\) by the fermi character of the \(^3\)He.
The heat capacity of the concentrated state and the diluted state can be explained by the binding energy with the \( ^3\text{He} \) molecule. Suppose, the mixture is separated into two layer as a \( ^3\text{He} \) concentrated state and a \( ^4\text{He} \) concentrated state at 0 K. A \( ^3\text{He} \) atom at the phase separation boundary can diffuse into the \( ^3\text{He} \) concentrated state or the \( ^4\text{He} \) concentrated state. Because of the ground state vibration, the distance between a \( ^3\text{He} \) molecule and a \( ^4\text{He} \) molecule is shorter than the distance between two \( ^3\text{He} \) molecules.

The binding energy comes from the van der Waals force, so the binding energy between the \( ^3\text{He} \) molecule and the \( ^4\text{He} \) molecule is higher than between the \( ^3\text{He} \) molecules. The \( ^3\text{He} \) molecule moves to the \( ^4\text{He} \) concentrated state, and the \( ^4\text{He} \) concentrated state becomes diluted state as more \( ^3\text{He} \) molecules are introduced to the \( ^4\text{He} \) concentrated state. The heat capacity of the diluted state is always larger than the concentrated state because of the strong bonding\(^{\text{15}}\)

Even at the zero temperature, \( ^3\text{He} \) molecules diffuse in the \( ^4\text{He} \) concentrated state. It can explain the finite solubility of the \( ^3\text{He} \) molecules in the diluted state. But with this explanation, the phase
separation of the mixture cannot occur because the $^3\text{He}$ and $^4\text{He}$ can be mixed each other even at 0 K. 15 The separation of two layers is due to the fermi character of the $^3\text{He}$.

As the fermi $^3\text{He}$ molecules move into the diluted state, they occupy from the ground state and stack up to the fermi state due to the Pauli exclusion principle. The fermi energy of the dilute state increases with the $^3\text{He}$ concentration of the diluted state. Therefore, the chemical potential of the $^3\text{He}$ in the diluted state decreases as more $^3\text{He}$ molecules are introduced to the diluted state by the increase of the fermi energy.

Because of the weak binding between $^3\text{He}$ molecules, the $^3\text{He}$ of the concentrated state has lower chemical energy than the $^3\text{He}$ of the dilute state. Chemical potential of the diluted state is lowered to the chemical energy of the $^3\text{He}$ concentrated state as the $^3\text{He}$ concentration of the diluted state becomes 6.4%15.

The solubility of the $^3\text{He}$ molecules in the diluted state has a limit. If the chemical potential of the diluted $^3\text{He}$ molecule becomes lower than the chemical potential of the pure $^3\text{He}$ molecule, then the $^3\text{He}$ molecules of the diluted state move back to the concentrated state. The phase separation of the mixture and
the finite solubility of the $^3$He in the diluted state can be explained with the chemical potential of the $^3$He.

3.2. Components of Dilution refrigerator

The DR is based on the closed cycle circuit to prevent a loss or a contamination of the mixture and to produce a continuous DR cooling power. The phase separation of the mixture requires several pre-cooling steps for the DR cooling. For these two major reasons, the DR consists of many components.

Figure 17. Components of the DR. The height of dewar engaged with the insert is around 2.5m. Our DR is the KelvinoxMX400 with the 14T superconducting magnet by the Oxford-Instruments
One of the major components of the cryogenics is the dewar. The helium bath of the dewar is filled with the liquid $^4\text{He}$ approximately every week. The insert is dip into the helium bath and maintains a cryogenic state of 4.2 K, the boiling point of liquid $^4\text{He}$. The dewar has a super-insulation jacket to prevent unnecessary heat exchanges between the cryogenic and the ambient environment. The dewar jacket needs to be evacuated every six months to maintain the performance of insulation.

A superconducting magnet is mounted inside the dewar. In the cryogenic state, a magnet coil becomes a superconducting state. There is a superconducting wire with a heater as a persistence switch in parallel with the magnet coil. When the heater is turned on, the superconducting wire near the heater acts as a small resistor. As an electrical source is connected to the magnet circuit, the current begins to ramp up by the small resistor. The heater is turned off after the magnet coil has been energized and the small resistor becomes a superconducting state. The ramped current is persisted in the superconducting magnet after disconnecting the electrical source$^{16}$. 
There is a cold trap with a liquid nitrogen dewar. The mixture always passes through the cold trap before entering the insert and it also removes contaminants from the DR unit. The liquid nitrogen dewar should be filled with liquid nitrogen every week. The cold trap should be cleaned up before the cool down.

Other major component of the cryogenics is the insert. At the insert, there is the STM chamber with the UHV state. Inside of the STM chamber, the closed cycle circuit is attached to the insert for the DR cooling (DR-STM). The STM head is attached below of the bottom plate of the insert. The bottom plate is the mixing chamber plate where the DR cooling occurs. For this reason, the STM is carried out in the ultra high vacuum and ultra low temperature.
Figure 18. The dilution refrigerator\textsuperscript{14}. It is called a wet refrigerator, because of the 1 K pot. \textsuperscript{4}He evaporation occurs to cool the mixture in 1 K pot.
**Figure 19.** The dry refrigerator schematics of the kelvinoxMX400. The 1 K pot is replaced with the Joule-Thompson stage for the isenthalpic expansion. STM head is attached below the plate of the mixing chamber.
After passing through the cold trap, the mixture enters to the condensing line. The condensing line pass through the liquid $^4$He bath of the dewar, so the mixture is cooled to 4.2 K. The first stage of the condensing line is the Joule-Thompson stage (J-T stage) with a heat exchanger. First, the heat exchanger cools the mixture. Next, a primary impedance cools the mixture by the isenthalpic expansion. The mixture is cooled to 1 K after the J-T stage with a heat exchanger$^{15}$.

There is a dilution unit after the J-T stage. There are three plates in the dilution unit. The first plate is called a still plate where a still is placed. The mixture enters a heat exchanger attached to the still. After the heat exchangers of the still, the mixture enters a tube-in-tube heat exchanger. The tube-in-tube heat exchangers end at the cold plate. The mixture is cooled to 0.1 K at the cold plate by the two heat exchangers$^{15}$.

Following from the cold plate, there is a mixing chamber plate. Before the mixture enters into the mixing chamber, there are several step heat exchangers consisted of sintered silver. The step heat exchangers effectively depress the kaptiza resistance and the viscous heating for the milli-kelvin temperature$^{14}$. The mixture
enters into the mixing chamber, and the DR cooling occurs.

Figure 20. The heat exchangers in the dry refrigerator with pseudo schematics for more clearance.
After the DR cooling, the osmotic pressure difference pulls up the $^3\text{He}$ molecules from the mixing chamber. The outgoing mixture line from the mixing chamber is attached with several steps heat exchangers and the tube-in-tube heat exchanger. The outgoing cold $^3\text{He}$ molecules from the mixing chamber produce the cooling power of the two heat exchanger. The mixture is pulled up to a pot called a still.

A (de)still is a component for a fractional distillation. There is a heater at the still. When the heater is turned on, the partial pressure difference between $^3\text{He}$ and $^4\text{He}$ is generated. The $^3\text{He}$ molecules are selectively evaporated from the still\textsuperscript{14}. The outgoing $^3\text{He}$ gas has a cooling power for the heat exchanger attached to the still. The $^3\text{He}$ solubility of the still is lowered after the fractional distillation. The difference of the $^3\text{He}$ solubility between the still and the mixing chamber generates the osmotic pressure to pull the $^3\text{He}$ molecules up from the mixing chamber to the still.

The still is connected to a turbo pump for evaporating the $^3\text{He}$ molecules from the still. The outgoing line from the still is attached with the heat exchanger of the J-T stage. The outgoing $^3\text{He}$ molecules generate a cooling power for the heat exchanger of the
J-T stage.

After the evaporation, the $^3$He molecules are liquefied by a compressor at room temperature. The $^3$He of the mixture may return to the cold trap for a continuous DR cooling, or to the mixture tank to stop the DR cooling.

All the closed cycle circuit operation is managed by a control rack. Before the beginning of the DR cooling, the control rack checks whether the temperature of the mixing chamber and the still are below 20 K. The mixture begins to fill the dilution unit. When the mixture fills the dilution unit sufficiently, the turbo pump and the still heater are turned on and the mixture circulates the closed cycle circuit. At first, the evaporation cooling occurs at the still. The DR cooling occurs as the mixing chamber is cooled below the phase separation temperature. It takes 3-5 hours to reach the base temperature after the temperature of the mixing chamber is reached at 20 K.
3.3. Operation of Dilution Refrigerator

![Diagram of Gas Handling System]

Figure 21. Closed cycle circuit. Left: an explanation of the closed cycle circuit. Right: Evacuation of the closed cycle circuit

1. After an IVC indium sealing, lift up dewar and engage with the insert.

2. Evacuate
   
   A. Dewar, and IVC (Inner vacuum chamber)
   
   B. Cold trap cleaning.
      
      i. Collects all the mixture by the triton software
ii. Close all valves. All pneumatic valves by the triton software, three manual valves of the pump rack, two manual valves of the mixture tank, and a gate valve of a still line and a swage-lock valve of condensing line at the insert should be closed.

iii. Lift up cold traps from liquid nitrogen dewar.

iv. Connect a backing pump at V10. Evacuate cold traps by Still turbo pump with V10 and V6 opened, until P3 (Still line pressure) becomes 10E-4 mbar.

C. Condensing line and pre-cool line

i. Open valves of the insert with still line, and condensing line (manual V10 and pneumatic V6, V7, V8)

ii. Evacuate the condensing line until P3 (Still line pressure) is 10E-4 mbar, P4 (Turbo backing pressure) below 0.1 mbar
3. Throughput test to leak check between IVC – PC lines.

A. Confirm the valves V1,V2,V3,V9 are closed at the gas handling system and Close V10, V12 and open V11 manually at the pump rack

B. Open V14 manually at the mixture tank.

C. Open V4, V5 at the gas handling system and turn on the fore pump and the compressor. Wait until P1 and P2 become stable, and record the P1 and P2 values

D. Open only V5 pneumatic valve, pulsing V9 until P2 (Condensing line pressure) becomes 2.5mbar.

E. Open V2, and leak check between PC-IVC. If there is no leakage, close V2.

F. Open V8 to lower the pre-cool line pressure.
Figure 22. Throughput test preparation of the closed cycle circuit

Figure 23. Pre-cool operation
Figure 24. Dilution Unit operation

4. Throughput test to leak check between IVC – DU lines.

   A. Open a gate valve of a still line and a swage-lock valve of condensing line at the insert.

   B. Open V1, V5 pneumatic valve, pulsing V9 until P2 (Condensing line pressure) becomes 2.5mbar.

   C. Keep track of the increasing P3 value per minute and confirm throughput test value with a factory test result. It is on the last page of the hard-copy manual.

   D. Close V1, open V6 and leak check between DU-IVC

5. Collect mixture. Close V14 of the mixture tank.

6. Put $^4$Helium gas to dewar, and leak-check between IVC –
Bath.

7. Dip the cold traps into the Liquid nitrogen dewar.

8. Connect dewar vent line to liquid nitrogen exhaust, transfer liquid nitrogen

9. After reach 77 K, blow up liquid nitrogen with \(^4\)Helium gas of room temperature.

10. Evacuate dewar while checking the temperature. Evaporation of liquid nitrogen makes the temperature slope higher than before.

11. Throughput test to leak check between IVC – PC lines at cryogenic state do same as 3.

12. Throughput test to leak check between IVC – DU lines at cryogenic state do same as 4.


14. Connect the dewar vent to \(^4\)helium recovery line.

15. Put \(^4\)Helium gas to dewar, and leak-check between IVC – Bath.

17. Transfer liquid $^4$Helium.

A. Slow initial transfer needs to make all uses of cooling power, only a few meters are covered with icy.

B. Pressures with 4.5 psi would be enough after initial transfer.

3.4. One-way leak of the pre-cool line.

In the dilution unit, there are dilution unit plates to prepare the DR cooling at the mixing chamber. Each plate is thermally isolated and has different base temperature to cool the mixture below than the triple point temperature of the mixture. The mixing chamber and the (de)still needs to be cooled than 20 K to begin the DR process. These are already mentioned in the section 3.2.

The dilution unit plates cannot exchange a heat from the helium bath because they are thermally isolated. Exchange gas is ordinarily used to make a thermally connected state between the plates and the helium bath. $^4$He gas is used as exchange gas and injected into the STM vacuum chamber. The exchange gas is evacuated after the (de)still and the mixing chamber are cooled.
than 20 K and the mixture begins to circulate in the closed cycle circuit for the DR cooling.

**Figure 25.** A description of the mixture line$^{15}$. (a) The DR line (green) and the pre-cool line (red). (b) A leak-check filling with the $^4$He gas in the STM vacuum chamber and leak detecting at the pre-cool line. (c) A leak-check with throughput test. If there are any leak, the mixture is detected at the STM vacuum chamber.

In our DR, the exchange gas is replaced with a pre-cool line to make the thermally connected state. The pre-cool line is attached with each dilution unit plates. After filling the helium bath with
liquid $^4$He, the mixture circulates the pre-cool line and cools the dilution unit plates. The pre-cool line is thermally connected to all plates of the DR and the mixture directly cools the dilution unit plates which provides a faster cooling than using the exchange gas$^{15}$. There is no gas injection to the STM vacuum chamber if the DR has the pre-cool line so it is easy to make UHV using the pre-cool line than the exchange gas.

Both the DR line and the pre-cool line should be leak-checked carefully. If there is any leak, the mixture can be lost from the closed cycle circuit and inhibits to make the UHV. The contaminants can enter into the closed cycle circuit during the mixture circulates in the closed cycle circuit. It may lower purity of the mixture.

There are two ways to leak-check the DR line and the pre-cool line. One way is throughput test of the DR line and the pre-cool line which are already described in the section 3.3. A leak detector is connected to the STM vacuum chamber. If there is any leak at the DR line or pre-cool line, the small amount of mixture will flow to the vacuum chamber during the throughput test and the leak detector recognizes it. It is depicted in the figure 25-(c).
Other way is filling the helium gas in the STM vacuum chamber. First, the DR line and the pre-cool line should be disconnected from the closed cycle circuit. Next, a leak detector is directly connected to the DR line or the pre-cool line of the insert. If there is any leak at the DR line or pre-cool line, the small amount of the $^4$He gas will flow to the DR line or pre-cool line and the leak detector recognizes it. It is depicted in the figure 25-(b).

The leak-check should be done in both ways to confirm that there is no leak in bi-direction. In an actual leak-detecting process, there is a one-way leak between the STM vacuum chamber and the pre-cool line. The leak is only detected for the throughput test as the figure 25-(c), but not detected when the leak-check is done with filling the $^4$He gas in the vacuum chamber of the STM as the figure 25-(b).

It is expected that the mixture would be lost and contaminates the UHV of the STM vacuum chamber during the DR cooling is in progress. For this reason, the pre-cool line should be replaced with a leak-tight condition. It is regrettable that an actual experiment with the DR cannot be performed.
Chapter 4. Vibration Isolation

4.1. Brief explanation of vibration Isolation

It is important to isolate the STM head from an external vibration because the tunneling current is sensitive to the tip to sample distance. A method of the vibration isolation is same as the mechanism of the seismometer. The heavily weighted insert is isolated from the external vibration by air springs. The laboratory has double vibration isolation system\textsuperscript{17}. The primary system isolates the vibration between the building and the experimental acoustic room. The secondary system isolates the vibration between the STM frame and the insert in the experimental acoustic room. The STM experiment is accomplished under the ultra low vibration with the double vibration isolation system The vibration isolation system is depicted in the figure 26.

For the primary vibration isolation, the ground of a room is dug below the floor level. Then the base is packed with the concrete. Up to the base, a heavy concrete block about 30ton is put on the base up to the floor level. On the concrete base, six air springs are installed between the concrete base and the concrete block which
isolate the vibration between the concrete block and the concrete base as the figure 26. An acoustic room is installed on the concrete block as an experimental room. The primary vibration isolation allows that the experimental room is isolated from the entire building\textsuperscript{17}. There is a pit hole at the center of the concrete block for a maintenance. The dewar is stored in the concrete base when the experiment is not on running. The pit hole makes the maintenance experimental equipment easy and saves the space of the limited experimental acoustic room.

A STM frame is installed in the experimental room. The frame has three legs in triangular position. The STM plate is placed on the three legs. Between the legs and the plate, there are three air springs on the top of each STM frame leg which isolate the vibration between the STM frame and the STM plate as the figure 26. The plate and the legs contains the lead shots to increase mass for the inertia. The heavy frame and the acoustic room can be considered as a single rigid. The insert is fixed to the heavy STM plate, and these two components can be considered as a single rigid. The secondary vibration isolation allows that the insert is isolated from the acoustic room\textsuperscript{17}. For the cool down, the dewar engages the insert by lifting up the dewar.
Figure 26. Schematics of Vibration isolation system. Only two legs of the STM frame are drawn for more clearance. The dewar is lifted from the pit hole by a winch.
Figure 27. Secondary vibration isolation components

In the warm up state, the dewar is put into the pit hole of the concrete block. For the safety, the pit hole is closed with a pit cover. During the warm up state, the experimental equipment can be in maintenance including the STM head and the insert. When the experiment begins, the dewar is lifted up and fixed below the STM plate. The pit hole is closed with a heavy pit cover to isolate the external noise. By the “dewar lifting”, the dewar and the insert can be joined together. Comparing with the lowering the insert to the dewar, the experiment can be proceeded without disconnecting the electrical and the vacuum lines of the insert. The insert is
stationary without any movement and less likely to be damaged.

Inside the acoustic room there must be no vibration source, so all the pumps and gas bottles are placed at the pump room. There are conduits in the concrete block and the concrete base to connect between the experimental acoustic room and the back room as the green conduit line in the figure 26. The both conduits are connected with the bellows. The all pumps and the gas bottles are connected to the experimental room via the conduits of the concrete base and the concrete block. Also, most of the vacuum components in the acoustic room are connected to the conduits of the concrete block with bellows to isolate the vibrations of the pump and gas bottles in the back room via a gas panel. The gas panel facilitates evacuation control of the experiment.

![Image](image_url)

(a) Gas panel  
(b) Bottom panel

**Figure 28.** Gas panel
Figure 29. Success of dewar lifting by a red winch and a pair of cables. A pair of white slings are tied to the dewar for the safety.
4.2. Dewar Lifting

![Blueprints of dewar lifting. Left: Components of the dewar lifting. Right: Verifying the dewar lifting with a 3D modeling. A winch plate and the pulley plate is machined after checking actual distance measurements.](image)

The dewar should be lifted up from the pit hole to proceed the cool down. For lifting up the dewar, two cables at both sides of the dewar are connected to a winch. The winch is wound up the two cables with the same rate. Dewar has a treadmill-like shaped attachment for lifting the dewar. On both sides of the lower end of the treadmill, a hoist-ring is engaged for each side laterally. If it is
engaged in the upward direction, the two cables interfere with an upper part of the dewar. The hoist ring has a degree of freedom of a half sphere in steradian to adjust its orientation. If there is no degree of freedom in the hoist-ring, the dewar would rotate to adjust its stable position during the dewar lifting which makes the entire dewar lifting process unstable. A cable connects between the hoist-ring and a pulley. Since the pulley cannot be connected to the STM plate directly, each pulley is joined to the STM plate with a pulley plate.

![Image](image.png)

(a) hoist-ring  (b) Fixed pulley  (c) Assembled pulley plate

**Figure 31.** Parts for the dewar lifting

The off-center may occur while combining the insert and the dewar. The off-center issue can be solved by the gravity of the insert if the insert is lowered to the dewar. However, the dewar “lifting” method cannot use the gravity to adjust the off-center
issue. If there is an off-center issue, the dewar and the insert cannot be joined together. Even they managed to join together, an excessive force may apply to the insert and the insert may be damaged.

Even all parts of the dewar lifting are made symmetrically, there was an off-center issue as the figure 32-(a). The off-center should be adjusted by breaking the symmetry of the pulley plates, because the pulley plates are only parts can be machined asymmetrically as the figure 32-(b). It solved the off-center issue in some extent. For a complete solution of the off-center issue, some guides are required between the dewar and the insert during the dewar lifting. The insert has a sliding shell as the guide by contacting with an inner rim hole of the dewar. The sliding shell is on the figure 32-(c) The off-center issue still presented after machining the pulley plates asymmetrically because the sliding shell was not exactly perpendicular to the STM plate. It could be solved by a fine-tuning of the sliding shell coupling components. The off-center was adjusted by the pulley plate until the top of the dewar reaches the sliding shell. After the dewar reaches the sliding shell, the off-center was finally adjusted with the sliding shell.
Figure 32. The off-center issue. (a) The dewar is biased to the northeast relative to the insert. (b) The pulley plates are machined in asymmetrically. (c) The sliding shell should be perpendicular with the STM frame.

After the cable reaches to the pulley, the cable is connected to the winch plate. However, the cable between the pulley plate and the winch plate interferes with the top of the treadmill attachment of the dewar. The pulley plate is moved forward to the winch plate so the cable between the pulley plate and the winch plate does not
interfere with the dewar. The dewar is lifted up with slightly off-centered which can be adjusted with the sliding shell. The winch plate has a pair of pulleys at the top which adjust the direction of each cable to edges of a winch drum. A winch drum is machined in double tied for each side, so each cable can be bound on both sides of the winch drum. It is depicted in the figure 33-(b) The winch drum is designed as both cables are wound exactly same rate at once. The winch drum and the cable are fixed with a bolt. The double tie method with a bolt makes it easy to adjust the length of both cables. If the total length of both cables are not matched, the dewar is lifted up with tilted due to the tension difference of the two cables. In the worst case, the dewar can lurch abruptly. The diameter of the winch drum slightly enlarged. Every time the stratum of wound cable changes, there is an abrupt shaking at the dewar. The enlarged drum needs less than one stratum for the dewar lifting to avoid the abrupt shaking.
Figure 33. Winch for dewar lifting

After the dewar lifting, the STM plate and the dewar should be joined together. Six full threads bolts are used for fixing the dewar with the inner rim. Before the dewar lifting, three bolts are fixed at the dewar and the other three bolts are fixed at the plate. After the dewar lifting, six full threads bolts are tightened with nuts. The outer rim of the dewar needs eight bolts to fix the dewar. After fixing all fourteen bolts, the winch drum should be unwound and the cables must not be in tension. Sometimes, full threads bolts are not separated from the dewar after the dewar is lowered from the STM plate. After tightening two nuts to the bolts, loosen only a below nut, and the bolts are separated from the dewar.
4.3. Gas panel

![Gas panel diagram]

**Figure 34. Gas panel description**

The gas panel is parted into two areas. One is UHV part, and other is Non-UHV part. The main chamber is a major UHV part which is referred as the transfer passage in the chapter 2. The end of the main chamber is connected with the turbo pump. The turbo pump needs a backing pump with a scroll pump connected via the gas panel. For a leak check of the UHV, the backing pump is interchanged between a leak checker and the scroll pump via the gas panel. There is a pump rack for the DR in the back room. The pump rack also requires a backing pump for the evacuation of the
closed cycle circuit of the DR. The scroll pump can be the backing pump of the DR via the gas panel.

Figure 35. Interchanging backing pump

The dewar bath is a major Non-UHV part. There are several dewar bath operations. First, the dewar bath needs to be evacuated before the cryogenic state which can be done by a rotary pump. Second, helium gas injection is needed to the dewar bath for a leak check. Third, a nitrogen venting line is needed for an explosive nitrogen gas release during the cryogenics state with liquid nitrogen. Lastly, a helium recovery line is needed during the cryogenics state with liquid helium. The helium recovery line can be evacuated via the rotary pump of the gas panel. The 4-way-cross or called as a differential stage for the sample transfer is needed to be evacuated via the rotary pump of the gas panel.
Figure 36. Evacuation, and venting line

Helium gas can be injected into the UHV part and the Non-UHV part. For the dewar bath, Helium gas is injected for the leak check between the IVC and the helium bath. For the main chamber, Helium gas can be injected for the venting to prevent damages.

There is a separated line in the gas panel to inject nitrogen gas for the table air spring. In addition, the nitrogen gas may be used to pressurize the experimental dewar for the back transfer of liquid nitrogen to the storage dewar.
The $^3\text{He}$ gas flows through the still gas line. If the still gas line is connected to the pump rack via the gas panel, there would be one more channel between the pump rack and the still line. It may lower the purity of mixture or cause the mixture loss. So the still gas line is directly connected to the pump rack by the bellows without passing through the gas panel.
Chapter 5. Summary

In this thesis, operations of the experimental equipment are described to prepare the STM at the ultra low vibration and the ultra low temperature conditions.

An experimental acoustic room has vibration isolation systems. A dewar is placed down into a pit hole of a concrete block to take full utilization of a limited experimental acoustic room. The dewar is lifted up from the pit hole to make the cryogenic and UHV. Even a single person can sufficiently lift up the dewar by winding up a winch. A gas panel makes the evacuation control of the experimental equipment easier. A vacuum chamber with the STM can be evacuated to the UHV via the gas panel. A helium bath of the dewar can be evacuated, and filled with the $^4$He gas to prepare the cryogenic condition via the gas panel.

A sample is loaded on the STM by a sample transfer rod. The sample transfer rod is engaged with an insert by a 4-way-cross not to directly expose the vacuum chamber to an ambient pressure condition. An *in-situ* cleaver is used for preparing a clean and flat sample surface at UHV and cryogenics conditions. The sample is
finally loaded on a STM by a key-hole mechanism.

A DR can make the ultra low temperature condition. A DR cooling lowers the temperature to 7 mK by the enthalpy difference of two mixture phases. Due to the mixture is valuable, a closed cycle circuit is used for the DR which liquefies the mixture gas and returns the liquid mixture to the DR to prevent loss or contamination of the mixture. The DR is evacuated before the cryogenic state to prevent blockage and damage to the circuit and the DR, and leak-checked with a pressurized mixture which confirms the throughput of the mixture in the DR for the stable DR cooling.

With the series of the equipment operations, the STM is carried out in the ultra low temperature and UHV conditions. The spectroscopic-imaging scanning tunneling spectroscopy would be the first goal which can be only accomplished under the ultra low vibration. It must be preceded that a proper operation of the in-situ cleaver to prepare the sample with a good condition. The DR cooling operation and the tip preparation should be done proficiently so the SI-STM can be performed before the liquid helium is depleted from the dewar. I expect and I hope, once the
one-way leak problem is addressed, that DR-STM system will be an extremely crucial tool in the investigation of a physical properties of the correlated electron systems at milli-kelvin temperatures.
Bibliography


2. G. Binnig and H. Rohrer, Surface Science, 126, 236-244 (1983)


Appendix

Lifting components

https://www.grainger.com/

Vacuum components

https://www.mdcvacuum.com/

https://www.n-c.com/

https://www.lesker.com/
초록

고체의 표면을 시각화 할 수 있고, 차동 진도도 측정을 통한 분광 영상
을 수행할 수 있는 주사 터널링 현미경은 응집물질물리학에서 널리 사용됩
니다. 본 주사 터널링 현미경에는, 초저온에서 새로운 현상들을 탐구하기
위해서 희석 냉각기가 통합되어 있습니다.

희석냉각기를 사용하면, 초저온에서 주사 터널링 현미경 검사를 수행할
수 있습니다. 희석냉각기는 3He 와 4He의 헬륨 혼합물이 사용되고, 단단한
순환 회로에 의해 연속적으로 헬륨 혼합물이 순환됩니다. 희석냉각기는 몇
거지 구성요소가 있고, 복잡한 작동 방법이 동반됩니다. 희석냉각기의 기
본적인 작동법을 설립하기 위한 희석냉각기에 대한 설명과 작동 방법이 서
술되어 있습니다.

주사 터널링 현미경의 표적 시료는 일반적인 환경에서 준비되어서 극저
온, 초고진공으로 전달됩니다. 특수한 시료 전달 막대를 통하여 시료는 위
에서 투입하는 방식으로 주사 터널 현미경으로 전달됩니다. 시료는 시료
전달 통로의 최상단에서 주사 터널링 현미경의 헤드가 존재하는 바닥으로
전달됩니다. 시료 전달 통로 중간에, 깨끗하고 평평한 표적 시료를 준비하
기 위해서 시료 가르키 단계가 위치해있습니다. 시료 전달 작업을 능숙하
게 수행하기 위해서 전체 시료 전달 절차 및 시료 전달 구성요소들의 미세
조정 목록이 설명됩니다.

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본 논문에서는 의학생활과 진동 절연 설계를 이용하여 쇄처리 및 혈액 진동에서 주사 터널링 현미경을 수행하기위한 전반적인 장비 동작 및 설명을 기술합니다.