Appendix E: Vacuum System and Sample Preparation for the Hall Effect Experiment

I. Introduction

With proper care, most vacuum systems can operate trouble-free for long periods of time: not only does continuous operation not harm them but it in fact cleanses them of some contaminants. It should be understood that the ultimate vacuum (within the capability of the system) and the time it takes to reach that vacuum are determined by the cleanliness of the system. It takes only one "lunkhead" opening a valve at the wrong time to contaminate a vacuum system, slowing the pumping speed and ruining its vacuum capability. Students operating vacuum systems should read and understand the operating instructions before starting the experiment, and ask for assistance if the instructions are not understood.

II. Vacuum Systems

Vacuum systems in the Advanced Laboratory consist of a mechanical rotary pump for low vacuum, an air-cooled oil diffusion pump for high vacuum, and the necessary valves and traps to interconnect them. Low vacuum is measured by thermocouple gauges and high vacuum by cold- cathode ion gauges.

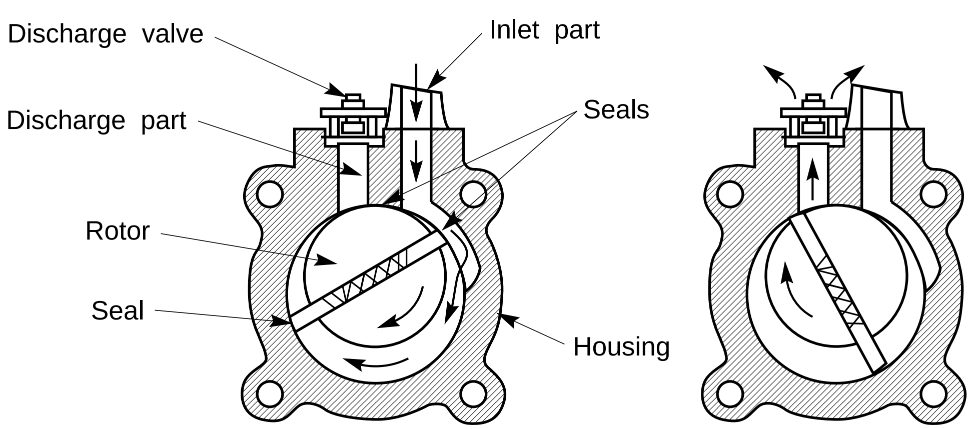

Figure E-1

A mechanical rotary pump is used for both foreline and roughing pumping. (See Fig. E-1). A solid steel cylindrical rotor is mounted eccentrically in a larger steel cylinder. Contact between the two is affected by two spring-loaded vanes in diametrical slots in the rotor. As the rotor turns, it pulls air particles through the intake port and expels them through the outlet valve. Vacuum sealing is supplied by the precise machining of the parts and special mechanical pump vacuum oil.

High vacuum is supplied by a three-stage oil diffusion pump. A special silicone oil is boiled by an electric heater. As the oil turns into a gas it naturally increases in volume. The expanding gas jets up the concentric tubes and is deflected downward pulling air particles from above. When the gas hits the cooled walls of the pump, it condenses and returns to the bottom of the pump for reheating, while the air particles are pulled from the diffusion pump through a "foreline" by the mechanical pump. A diffusion pump cannot operate without this foreline pumping.

Since there are gaseous oil particles in the lower part of the diffusion pump and a high vacuum above, some method must be used to prevent the oil from "backstreaming" into the high vacuum area. One such method is the use of baffle plates which, being at a lower temperature, condense the oil. The method we use is a liquid nitrogen cold trap which not only condenses the oil but also freezes out water vapor, air, and other contaminants.

Thermocouple (TC) gauges are used to measure low vacuum pressures. A thermocouple is a junction of two metals which generates a small amount of electric current depending on the temperature of the junction. In a vacuum thermocouple gauge the junction is heated by a calibrated amount of current fed to a heater adjacent to the junction. The current generated by the junction is then measured by a sensitive ammeter. As the vacuum increases and the number of air particles decreases, there is less transfer of heat to the junction, causing less current to be generated, thereby giving an indication of the vacuum.

In cold cathode ion gauges (used to measure high vacuums) a high voltage anode ionizes the gases in the gauge, thereby causing a current to flow as an indication of vacuum pressure. The higher the vacuum, there are fewer gas particles, less ionization, smaller current. Cold cathode gauges should never be operated above 25 microns as the ionization of organic compounds at that pressure contaminates the gauge.

Interconnecting the various parts of a vacuum system are special vacuum valves. See Figure E-2. Since pumping speed is determined in part by the size of the pumping line, vacuum valves are usually operated either fully opened or fully closed. The largest valve is the HIGH VACUUM valve connecting the diffusion pump with the experimental Bell Jar area. A "FORELINE" valve connects the lower part of the diffusion pump with the mechanical pump. A "ROUGHING" valve connects the experimental Ball Jar area to the mechanical pump so that the bulk of the air or atmosphere in the Bell Jar can be removed before connection to the diffusion pump. A small "AIR INLET" valve is installed to permit the Bell Jar to be brought up to atmospheric pressure. Most removable parts of a vacuum system are vacuum-sealed to the system by "O-Ring" seals, exceptions being the mechanical pump which is connected to rough rubber vacuum tubing, and the bell jar which uses an “L” shaped gasket. A light coating of vacuum grease is used on these seals to prevent vacuum leaks, but gobs of grease are not necessary or desirable.

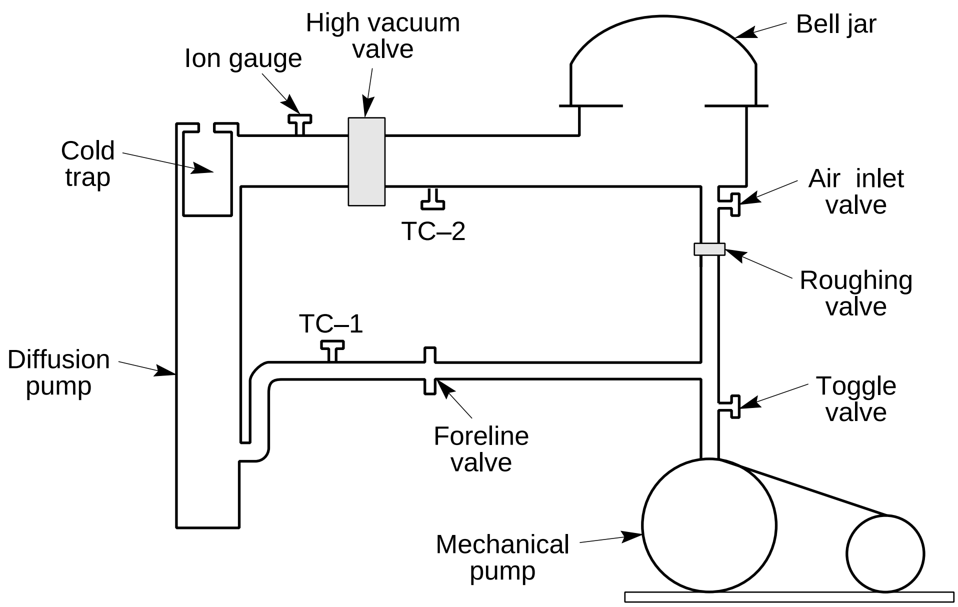

Figure E-2

III. Operating procedure to achieve high vacuum

A. Warming up the system

1. Make sure all of the valves are closed (turned clockwise). Little force is needed for these valves to seal, and over-tightening can destroy them.
2. Turn on the mechanical pump. (The left-most switch located on the pump station control panel)
3. **OPEN** the foreline valve. (Turn the valve knob counter-clockwise 3 or 4 complete turns)
4. Turn the knob on the thermocouple gauge controller to TC-1. (This measures low vacuum in the diffusion pump.)
5. When the thermocouple gauge controller reads 35 microns or less, turn on the diffusion pump. (The switch is located on the control panel.) It takes approximately 30 minutes for the diffusion pump to reach operating temperature. If the system has been contaminated, the diffusion pump may need to run for several hours before use.
6. Turn on the ion discharge gauge to the lowest scale (25×10^{-3}mm).

B. Roughing out the Bell Jar

1. Before proceeding, DOUBLE CHECK that:
   a. the mechanical pump is running
   b. the foreline valve is the only valve that is open
   c. the Bell Jar is installed properly with the metal screen guard in place.
2. **CLOSE** the foreline valve. **(***NOTE -NEVER leave the foreline valve closed for more than 5 minutes when the diffusion pump is operating***)**
3. **OPEN** the roughing valve. (as the mechanical pump is removing air from the Bell Jar, it will make a gurgling noise)
4. Turn the thermocouple gauge controller to TC-2. (This measures low vacuum in the bell jar.)
5. When the thermocouple gauge reaches 50 microns or less close the roughing valve.
6. Open the foreline valve. (Remember this valve should not be closed for more than 5 minutes.)
7. **OPEN** the High Vacuum Valve

C. Pumping with the complete system to achieve high vacuum

1. Before proceeding, DOUBLE CHECK:
   a. the upper chamber has roughed out (TC-2 meter reads 10^{-2} or less)
   b. the foreline valve is open
   c. the roughing valve is closed
   d. the high vacuum valve is open.
2. Obtain liquid nitrogen to put into the cold trap. (the technician will do this for you; please notify technician when you are ready to have the liquid nitrogen added)
3. Adjust the scale on the ion gauge controller to read the high vacuum pressure.

D. Opening the Bell Jar

1. Close the high vacuum valve
2. Turn the thermocouple gauge controller to TC-1
3. Open the air inlet valve
4. When the hissing stops, close the air inlet valve
5. Remove the protective screen and Bell Jar

E. After you have finished

Vacuum systems should not just be turned off. The diffusion pump needs time to cool down before the mechanical pump can be turned off. This is accomplished by:

1. Close the high vacuum valve (Leave the foreline valve open)
2. Turn off the Thermocouple Gauge Controller
3. Turn off the Ionization Gauge Tube Controller

4. Turn off the diffusion pump
5. The mechanical pump should run for 45 minutes as the diffusion pump cools down. You do not have to wait for the diffusion pump to cool; simply inform the lab technician that you have completed the experiment.

IV. Sample Preparation:

A. Introduction

The evaporator for making the films consists of a large brass base plate connected by a 2" line to a vacuum system. There are two electrodes connecting through the base plate to provide mechanical support for, and electric power to, a filament "boat" or heater. The boat is a dimpled strip of molybdenum and is heated by high-current, low-voltage electric power controlled by a variable autotransformer. There is also a movable shutter between the filament and substrate holder.

The substrate holder is a removable brass cylinder surrounding the electrodes and shutter. The end of the cylinder places the substrates the correct distance from the filament for good film thickness and also acts as a mask to shape the film to the correct pattern. There is a hole in the cylinder to observe the evaporation.

The base plate and cylinder are enclosed in a removable glass bell jar, which has a rubber gasket for vacuum sealing and a metal screen safety guard. The metal screen safety guard should be in place at all times when the bell jar is under vacuum.

B. Preparing the Sample

1. Start the vacuum system so it will be at operating pressure when needed. The instructions are given above. (This takes approximately 45 minutes time.)
2. Bring the bell jar up to atmospheric pressure by opening the air inlet valve. Remove the screen guard and then the bell jar, placing the bell jar in the wooden cradle to prevent it from rolling off the table and to keep dirt from the gasket. If the bell jar adheres to the base plate after being brought up to atmospheric pressure, twist or rotate it to help loosen the bond of the seal. (See Lab staff for assistance) Remove the mask and substrate holder cylinder.
3. Clean all the parts of the evaporator with Kimwipes and Methanol, including the bell jar and cylinder. DO NOT USE METHANOL ON RUBBER GASKET AT BASE OF BELL JAR! Cleaning the seal is unnecessary.
4. Get 2 or 3 small coils of silver wire from the white envelope and place them in the dimple of the filament boat. Center the shutter over the boat and set the cylinder and mask in place, checking to see that the hole in the side of the cylinder is positioned so that the evaporation can be observed.

5. Clean one microscope slide. Cleanliness is essential for the production of pure thin films. To clean the slide, wipe with a clean Kimwipe which has been dampened with alcohol.

6. Place the slide in the mask, or substrate holder. Then carefully set the bell jar in place by lowering it over the cylinder so that the entire bottom edge makes contact at the same moment with the base plate. Seat the bell jar on the base plate by giving it a slight twist. Place the metal guard over the bell jar.

7. **Be sure the air inlet valve is closed**. Evacuate the bell jar following the vacuum system instructions.

C. Evaporation of thin film

1. Make sure the vacuum system is warmed up and at acceptable operating pressure. (At this point you should have the High Vacuum valve open, diffusion pump has been on for at least 45 minutes and liquid Nitrogen is in the cold trap)

2. Turn on the filament power switch. Slowly rotate the filament control knob in a clockwise direction while observing the filament boat. It may be difficult to see at first, but as it begins to glow red you will be able to observe the silver metal. Slowly increase the current until the silver wire just melts, then reduce it to allow the silver to re-solidify. (You can tell when this happens by observing the convection currents on the surface of the molten silver bead.) The pressure in the vacuum system should rise as the heat outgases the silver and the metal components of the evaporator. The shutter protects the substrate from the contaminants and from the radiant heat.

3. When the pressure returns to normal, move the shutter to one side and turn the filament control knob clockwise again until the temperature is high enough to cause the silver to boil away, or evaporate. Do not raise the temperature once the evaporation starts, as too much current will burn out the filament. The silver vapor condenses as a mirror-finish thin film on all cool surfaces, i.e. the substrates, cylinder, bell jar, etc.

4. On completion of the evaporation, reduce the electrical power to zero. Wait a few minutes for the substrate to cool to deter oxidation when air is admitted, then bring the bell jar up to atmospheric pressure by closing the high vacuum valve and then slowly opening the air inlet valve. Air should be admitted slowly to prevent damage to the substrate.

5. After the slide have been taken out of the mask, remove the excess film that has been evaporated on the base plate, cylinder, bell jar and other parts of the evaporator using Kimwipes, methanol and "elbow grease." Replace the cylinder, bell jar and guard and evacuate the bell jar so it will be clean for the next evaporation. Shut down the vacuum system following the instructions provided as it will no longer be needed.