Vacuum Techniques and Thin Film Deposition

Experiment 3

(October 2001)

1   Introduction

Much of modern experimental physics is done under vacuum. Design and construction of vacuum apparatus is one of the most useful "bread and butter" skills an experimentalist in condensed matter, atomic, or optical physics can have, and the subject of vacuum engineering is a vast one. This lab serves as an introduction to basic vacuum techniques and thin film growth, another often essential skill for condensed matter physicists. This lab is an optional prerequisite for Experiment 10, Condensed Matter Physics at Cryogenic Temperatures, for which you can grow your own samples for Weak Localization measurements if you choose.

2   Pressure and gas flow

In vacuum work, pressures are almost always measured in millimeters of mercury, or *torr*. One torr is just the pressure necessary to support a column of mercury with a height of one millimeter. The conversion to units more familiar to readers of physics textbooks is

$$1 \text{atmosphere} = 101 \text{kPa} = 760 \text{torr}$$

There are two pressure regimes of interest to the scientist working with vacuum systems, and gases behave differently in each regime. The first, the *viscous flow regime*, describes the case where gas flows as a fluid, where the mean free path of the gas molecules is much smaller than the dimensions of the apparatus. The second, the *molecular flow regime*, describes the high-vacuum case, where the mean free path is much longer than the characteristic dimensions of the apparatus. In this regime, gas molecules interact almost entirely with the walls of the chamber, acting independent of each other.

Gas flow in either regime is measured in *torr liters per second*, which is equivalent to mass per second. The *conductance* of a tube describes how much gas flows through the tube for a given pressure differential between the ends. If Q is the mass flow, P_1 is the pressure at the input of the tube, and P_2 is the pressure at the output, then the mass flow is given by

$$Q = (P_1 - P_2)C$$

where C is the *conductance* of the tube. Conductance in the viscous flow regime is proportional to the average pressure in the tube and is quite high, compared to the molecular-flow regime, because the gas molecules push each other along. In the molecular-flow regime, conductance through a tube is independent of pressure and is given by

$$C = 12\frac{\text{liters}}{\text{second}}\left(\frac{D}{1cm}\right)^3\left(\frac{1cm}{L}\right)$$

where D is the diameter of the tube in centimeters, and L is its length, also in centimeters.

Pumping speed is expressed in liters per second. The amount of mass going through the pump is given by

$$Q = PS_p$$

where P is the pressure at the inlet of the pump, and S_p is the pump speed. It is not hard to show that the net speed of a pump connected to a vacuum chamber by a tube is

$$\frac{1}{S} = \frac{1}{S_p} + \frac{1}{C} \tag{1}$$

and that the time required to pump the system from an initial pressure of P_0 down to P is

$$t = 2.3\frac{V}{S}\ln\frac{P_0}{P} \tag{2}$$

where V is the volume of the chamber.

3 Vacuum Pumps

A large number of clever designs for vacuum pumps have been implemented over the years, dating back to the first leather-and-grease sealed, hand-operated pumps of the 1600's. These first pumps were modified ships' water pumps, used for pulling water out of the holds of the sailing ships of the day, and they operated by a simple valve-and-piston mechanism. The valve-and-piston principle is still the most widely used way of extracting air in the viscous-flow regime, though today our implementation is considerable more efficient! Modern mechanical pumps feature multiple stages, specialized low-vapor-pressure oil sealants, and electric motors. Good, modern mechanical pumps can often attain base pressures of a few millitorr or a few tens of millitor, though below about 100 mtorr the oil used in them will often leak back into the chamber being pumped on. This is called *backstreaming* and is usually undesireable. Backstreaming can be eliminated by placing a trap or high-vacuum pump between the mechanical pump and the chamber.

Mechanical pumps are seldom operated below 100mtorr, and for this reason they are often referred to as *roughing pumps*. To achieve even a moderate vacuum of 10^{-2} torr or better, a different pump design must be employed. The most common and reliable *high-vacuum pumps* in use today are *turbomolecular pumps*, or turbo pumps for short. These are basically just very high-speed fans, whose blades are moving at speeds comparable to the speeds of
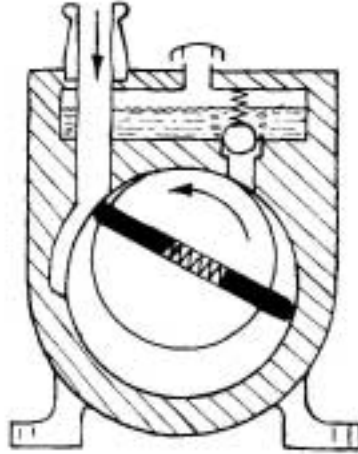
Figure 1: Cross section of a single-stage, rotary-vane mechanical roughing pump. Gas is pulled in the inlet (arrow pointing down), circulated counterclockwise and compressed, then blown out through a ball valve on the outlet. The theoretical ultimate base pressure is the pressure at the outlet (approximately atmospheric) divided by the compression ratio.

gas molecules. Turbo pumps are capable of sustaining very high *compression ratios*, the ratio of the gas pressure at the output to that at the input. Typical compression ratios are on the order of 10^7 for air, for an outlet pressure of 100mtorr. This low outlet pressure is maintained by a mechanical pump, which acts as both a roughing pump for the system and a backing pump for the turbo. One advantage of using a turbo pump in conjunction with a mechanical pump is that the turbo pump's compression ratio depends strongly on the molecular weight of the gas being pumped. Specifically, the log of the compression ratio is proportional to the square root of the molecular weight of the gas. Because the oils used in mechanical pumps typically have very high molecular weights, the compression ratio accross the turbo pump for these oils is considerably higher than 10^7, and the turbo pump effectively blocks any backstreaming from the roughing pump.

Speeds for turbo pumps are usually independent of the type of gas being pumped. Turbo pumps are specified by their speed, and the small turbo pump used in this lab has a speed of 80 l/s.

4   Chambers and Seals

Two things that limit the level of vacuum in any experiment are leaks and outgassing. (Both are mass flows and are expressed in torr liters per second.) Leaks are just poor seals that allow air to enter the chamber from the outside atmosphere. Outgassing refers to sources of gas "stored up" inside the vacuum chamber and released slowly into the vacuum. Typical sources of outgassing are trapped pockets of air in blind screw holes, rough surfaces, and
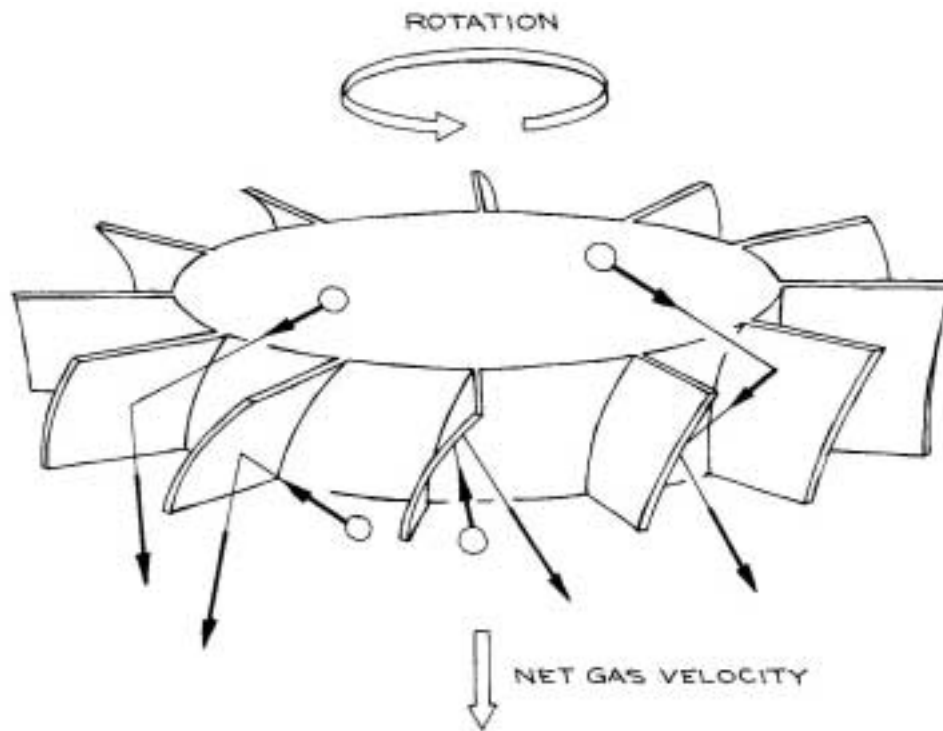
Figure 2: How a turbo pump works. The rotor spins fast enough to impart a significant downward component to the velocity of the gas molecules, creating a pressure differential between the region above the rotor and the region below it. Turbo pumps are only effective in the molecular-flow regime.

Figure 3: Exploded view of a gasket seal.

contaminants. Blind screw holes are often dealt with by using screws with a hole drilled through the center, so that the screw hole communicates to the rest of the chamber and gets pumped out along with the rest of the apparatus. Look for these *vented screws* in the apparatus when you perform this experiment!

Outgassing by contaminants can be eliminated by keeping the system clean. Always wear gloves when handling anything that goes inside a vacuum system, and never use ordinary lubricants on these parts. The preferred modern method for lubricating threads is to silver plate them. Silver does not stick to stainless steel well, and a silver-plated screw will turn in a threaded, steel hole almost as easily as one that is lubricated. Look for silver-plated screws inside the vacuum chamber, as well as vented ones!

Rough surfaces outgas simply because air, and especially water in the air, sticks to them, coming off at a low but regular rate when the system is under vacuum. Clean stainless steel typically outgasses at a rate of 10^{-7} torr liters per second per square centimeter of surface area. Dirty stainless steel outgasses more.

The ultimate pressure of a system with leaks or outgassing is determined by the mass-flow equation $Q = PS$.

5 Pressure measurement

Just as different pumping schemes must be used in the viscous and molecular flow regimes, different methods of measuring the pressure must be used in different ranges as well. In this lab, we will use a thermocouple guage for measuring pressure between 2torr and 10mtorr, and an ion guage in the molecular-flow regime. A thermocouple guage consists of a filament and thermocouple in contact with each other. There is a range of pressures, approximately 10mtorr to 2torr, where the thermal conductivity of a gas depends on the pressure. If we dissipate a known amount of heat in the filament, then its temperature, as measured by the thermocouple, will depend on the rate of heat lost to the surrounding gas. The
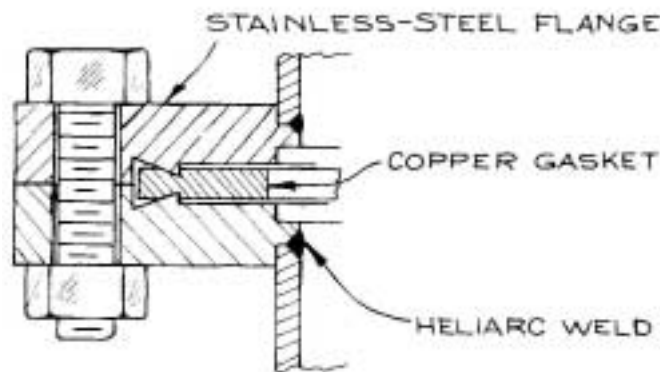
Figure 4: Cross section of a copper gasket seal. The knife edges on either side of the flange bite into the copper gasket and form a bakeable, high-vacuum seal.

guage itself, the filament and thermocouple, are enclosed in a plug attached to the chamber. The controller supplies power to the filament and performs the necessary conversion of thermocouple temperature to pressure, displaying the result on its front panel. This type of guage ceases to be useful below a few milli-torr, where the thermal conductivity of the gas is negligible.

Very low pressures can be measured using an ion guage. An ion guage consists of a filament (cathode), a positively charged grid (anode), and a negatively charged collection wire. Electrons boil off the filament by thermionic emission and are accelerated towards the grid. On the way to the grid, they collide with atoms in the surrounding gas, producing ions. These positively-charged ions then go to the collection wire, and the resulting current in the collection-wire circuit is proportional to the gas pressure. *This proportionality constant is different for different gases,* because different gases have different ionization potentials.

6   Thin-film growth

It is no understatement to say that thin-film growth techniques have, in the past three decades, fundamentally changed both condensed matter physics and everyday life. Well established thin-film technologies are used to grow the integrated circuits in our computers, cell phones, and palm pilots, while novel effects in thin films continue to be discovered and explored by both solid-state physicists and optical physicists. Many of the techniques used to grow thin films are related, and many involve physics and technology of marvelous subtlety. In this lab we will practice an elementary thin-film growth technique, evaporative deposition, as an introduction to this field. We will grow a thin film of silver on a glass substrate.

In evaporative deposition, our source and substrate will be placed inside a vacuum chamber, and the source will be heated until it melts and begins to evaporate. The resulting vapor will then condense on all surfaces inside the vacuum chamber, including our substrate. We will use a shutter to control the growth of our sample and to shield it from the initial
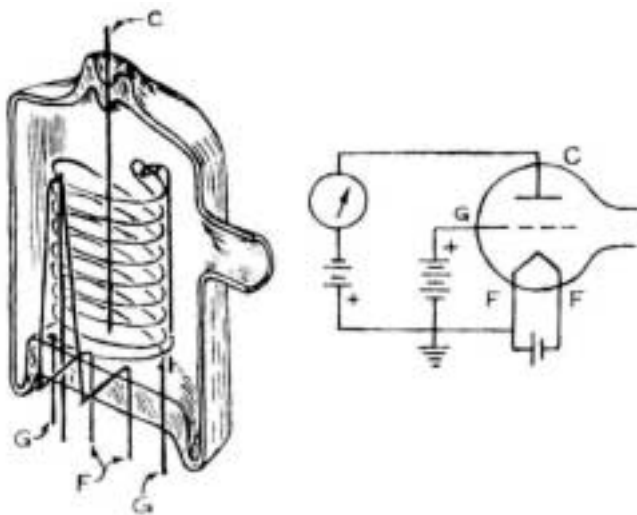
Figure 5: A Bayard-Alpert type ion gauge.

burst of "crud" that comes off of our source when it first melts.

The principal requirement for successful thin-film growth in this experiment is that the *mean-free path of the silver atoms must be greater than the distance between the source and substrate.* The mean free path of a molecule in a gas is

$$\ell = \frac{k_B T}{\pi d^2 P}$$

where d is the diameter of the gas molecules, and P is the pressure of the gas.

7   Apparatus

The apparatus for this lab consists of a cylindrical, stainless steel vacuum chamber, approximately 24" tall and 8" in diameter. This chamber is connected to an 80 liter per second turbo pump by a flexible steel hose, approximately 36" long and 2" in (inside) diameter. There is a sample stage at the top of the chamber for holding substrates, including a shutter for controlling film growth, and there is an evaporation boat at the bottom of the chamber. The evaporation boat is a tungsten filament that holds a lump of silver. A large current, supplied by a transformer and a variac, is passed through this boat, heating it up and melting the silver. There is a small observation window near the boat that allows you to monitor the source as it melts and evaporates.

Pressure is measured by both a Huntington 1518 thermocouple guage, and a Bayard-Alpert type ion guage. The Bayard-Alpert type guage is nice because it is entirely contained in a glass tube, and you can see its inner workings.

8   Prelab exercises

1. Estimate the pumping speed of the vacuum system used in this lab in the molecular-flow regime. At approximately what pressure is the transition between viscous and molecular flow?

2. If the outgassing rate for our apparatus is 5×10^{-7} torr liters per second per square centimeter, what is its base pressure?

3. What pressure do you expect to be an upper limit for successful deposition?

4. How many ideal gas molecules are there in a one cubic centimeter volume at room temperature, at a pressure of 10^{-5} torr?

5. (Optional) Consider an ideal gas inside a vacuum chamber. Derive or look up a formula for the number of gas molecules that strike the walls per unit area per unit time. This formula will also describe the flux of gas molecules against your sample. Now consider a reactive thin film under a hard vacuum. If the pressure in the vacuum chamber is 10^{-8} torr, and 20% of the residual gas is oxygen, how long will it take for the surface of the film to completely oxidize? Assume that "complete oxidation" just means forming a monolayer of oxygen, with each oxygen atom occupying an area of one square angstrom, and that the probability that an oxygen molecule striking the surface of your sample will stick (sticking coefficient) is 50%.

9   Experimental tasks

Caution: Do not touch any surface that will go under vacuum with your bare hands. Wear latex gloves to handle all vacuum-compatible surfaces and parts.

1. With the pump turned off, vent the system by opening the main valve, and remove the 24 bolts securing the top flange to the vacuum chamber. Remove the top flange, and place it upside down on the wooden box on the bench. Cover the top of the chamber with aluminum foil to keep dust out of the chamber. Place a microscope slide on the top flange under the shadow mask, and secure the mask and slide with the vacuum-compatible (cleaned, vented, and silver-plated) cap screws provided. This microscope slide will be the substrate on which you will grow a thin film of silver.

2. Remove the aluminum foil from the chamber, and use it to cover the flange to protect your "sample." Check to see if the evaporation boat is loaded; it should contain a pea-sized bead of silver. If it does not, use the insertion tool to load a new pellet of silver into the evaporation boat.

3. Remove the old copper gasket from the top of the vacuum chamber. Clean the conflat knife-edges on both the flange and the chamber with isopropanol and a kimwipe, and

clean a new copper gasket. Install the new gasket, and replace the top flange, aligning the slot in the flange with the slot in the top of the vacuum chamber. Make sure that your substrate is on the opposite side of the rate monitor, so that the rate monitor does not block deposition!

4. Bolt the top flange down. It is important at this stage to tighten the bolts evenly, and the following is the usual procedure for making a conflat seal. First, insert all 24 bolts and tighten them by hand. Second, tighten the four bolts at the 12 o'clock, 6 o'clock, 3 o'clock, and 9 o'clock positions *in that order.* Do this gently, turning each of the bolts by no more than one-eighth of a turn after you first feel resistance. Now tighten the four bolts just next to the first four in the same fashion, again turning each bolt by no more than one-eighth of a turn after you first feel resistance. These next bolts will occupy the 12:02:30, 6:02:30, 3:02:30, and 9:02:30 positions, and *they must be tightened in that order.* Now tighten the next four bolts, in the 12:05, 6:05, 3:05, and 9:05 positions, in that order. If you have ever rebuilt an engine, this is the same kind of bolt-tightening pattern used to assemble the block. This gentle, symmetric procedure is used whenever you wish to make a large, gas-tight gasket seal, from automobiles to vacuum systems to spacecraft. Keep going around the flange in this pattern, being gentle with each bolt, until the two sides of the conflat flange meet all the way around. You should not be able to see daylight between the sides of the flange. A seasoned experimentalist can form this seal in ten or fifteen minutes without hurrying. First-timers can expect to take between twenty and thirty minutes. If you find yourself taking more than thirty minutes to form the seal there may be problems with the hardware, so notify the instructor.

5. Making sure the roughing pump's exhaust is vented out the window, switch the pumping system on, and open the main pumping valve. The pumping system's power switch is on the power strip on the pumping cart.

6. Connect the thermocouple gauge controller to the chamber's gauge, and plug it in. The pressure should immediate read between atmosphere and 2,000 milli-torr.

7. Switch the turbo-pump controller on, and activate its start switch. Within just a few minutes, you should see the thermocouple gauge bottom out.

8. When the turbopump gets up to speed (indicated by a green light on the front of the controller), turn on the ion pump. To do this, you will need to turn on the power to the ion-gauge controller (main switch is in the back) and press the EMIS button on the front panel. Record the pressure vs. time at regular intervals until the system gets to the low 10^{-6} torr range. Plot the pressure vs. time curve in your lab book. How does it compare with your predictions?

9. When the pressure gets below about 5×10^{-6} torr, turn on the evaporation boat current by switching the variac to 120V. Increase the variac setting slowly to 60%. After a few seconds the silver pellet will melt, and you should be able to observe this through the

viewport. After the pellet has melted, open the shutter for one minute, then close it again. Dial the variac back down to zero, and switch it off.

10. Turn off the ion gauge by pressing the EMIS button, then turn the ion-gauge controller's main power switch off. Close the main valve and shut down the turbo pump by activating the STOP switch. The turbo pump will take a few minutes to spin down, and after that the vent valve will automatically open. When this happens, and the hose vents, it is safe to open the main valve and vent the chamber. Do so, then remove the top flange. (Remember to use gloves for handling vacuum-compatible parts.) Remove your substrate, and examine your film. Record your observations, and tape your sample into your lab book. Replace the top flange to protect the chamber. You do not need to use more than two bolts this time, and you do not need to form a new seal. *Leave the old copper gasket in place to protect the conflat knife edges.*

10 Questions and exercises

All questions should be reproduced in your notebook. Please do not include just the reference to the question number.

1. Why do you think the mean free path has to be greater than the distance between the source and substrate for evaporative deposition to work?

2. Suppose you wanted to grow a two-layer film, for example a gold film on top of a titanium film. How would you modify the apparatus used in this lab to do that? (Titanium is often used as an adherant to help gold films stick to substrates. Pure gold films tend to peel off most substrates.)

References

[1] John H. Moore, Christopher C. Davis, and Michael A. Coplan, *Building Scientific Apparatus: A Practical Guide to Design and Construction*, (Perseus Books, 1991).