Vacuum techniques and thin-film deposition

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October 8, 2013

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

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

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

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

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

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

where $V$ is the volume of the chamber.
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.

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 1600s. 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 turbo-molecular 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 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 pumps 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 across the turbo pump for these oils is considerably higher than $10^7$, and the turbo pump effectively blocks any backstreaming from the roughing pump.

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 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.
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 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 gauge for measuring pressure between 2 torr and 10 mtorr, and an ion gauge in the molecular-flow regime. A thermocouple gauge consists of a filament and thermocouple in contact with each other. There is a range of pressures, approximately 10 mtorr to 2 torr, where the thermal conductivity of a gas depends on the pressure. If we dissipate a known amount of heat in the fil-

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.
Figure 5: A Bayard-Alpert type ion gauge.

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.

Very low pressures can be measured using an ion gauge. An ion gauge 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 burst of crud that comes off of our source when it first melts.

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7 Apparatus

The apparatus for this lab consists of a glass bell jar, approximately 30" tall and 17" in diameter, inner dimensions. This chamber is connected to an 80 liter per second turbo pump by a gate valve with an inner diameter of roughly 3.5" and an effective length of roughly one inch. 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 the source metal, usually silver in this lab, but you can evaporate
anything you like within reason. A large current, supplied by a transformer and a variac, is passed through this boat, heating it up and melting the source. There is a sheet-metal shield surrounding the evaporation hardware to keep the inside of the bell jar from getting coated when you make a film. When you set up to grow a film, you will use aluminum foil to cover any gaps in this shield, and you will want to use a glass microscope slide to cover one of these gaps so that you can watch the source as it melts and evaporates.

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

8 Prelab exercises

1. Read Some Fundamentals of Optical Thin Film Growth, by Norbert Kaiser, to get a feel for what’s going on as a film grows. There are copies in the lab. If you’re in a hurry, you may read just Sections 3 and 5.

2. The distance between the source and the substrate in the thin-film deposition system you will be using is approximately one foot. What pressure do you expect to be an upper limit for successful deposition?

3. The pressure you calculated in the previous problem should be fairly low, i.e. ten-to-the-minus-something torr. How many gas molecules are there, approximately, in a one cubic centimeter volume at this pressure at room temperature?

4. The vacuum chamber you will be using is enclosed in a glass bell jar approximately 17 inches in diameter and 30 inches tall, and it has a “gate” valve approximately 3.5 inches in diameter leading directly (i.e. over a distance of no more than an inch or two) to the turbo pump (all inside dimensions). At approximately what pressure is the transition between viscous and molecular flow? If the pump has a speed of 77 liters per second, how long will it take to pump from this transition pressure down to the upper limit for successful deposition?

5. If the outgassing rate for our apparatus is $5 \times 10^{-7}$ torr liters per second per square centimeter, what is its base pressure?
6. (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 gloves to handle all vacuum-compatible surfaces and parts._

9.1 Assembly

9.1.1 Orientation

Identify and sketch in your lab notebook the following parts. Ask your TA if you need help identifying or locating anything. You can substitute photographs for sketches, if that is easier to do and just as clear.

- Bell jar
- Baseplate
- Main seal
- Turbo pump
- Gate valve
- Roughing pump
- Foreline
- Roughing-pump on/off switch
• Turbo-pump control panel
• Vent valve, including how to open and close it

For complicated or elaborate systems, it is often useful to make separate records of different *subsystems*. You have recorded the basic vacuum envelope and pumping apparatus. In a separate diagram, identify and sketch (or photograph) the pressure-measurement hardware.

• Ion gauge
• Thermocouple or Pirani gauge, if present
• Mechanical (high-pressure) gauge, if present - Note what type of gauge this is. Is it a Bourdon gauge? A capacitance manometer? Don’t forget to record make and model as well.

• Controllers for all installed gauges and how to read them

Now go through and identify the thin-film-deposition system. For this you will need to vent the system, raise the bell jar, and remove the evaporation shield. See your TA for help with this stage.

• Evaporation boat
• Shutter
• Evaporation high-current leads
• Main transformer (supplies current to the evaporation boat through the high-current leads)
• Evaporation-control variac (connected to the main transformer)
• Rate-monitor head (crystal housing)
• Rate-monitor controller
• Rate-monitor cooling lines, and how to turn the cooling water on and off
9.1.2 Cleanliness

Check the interior surfaces of the vacuum system and verify that they are all clean. A good way to do this is by wiping them down with a kimwipe and isopropanol. If the wipe comes off clean, the system is probably clean enough to achieve an adequate vacuum. The subject of cleanliness in a vacuum system is both vast and subtle, and if you want your base pressure to get below $10^{-7}$ Torr this is something you have to put some serious study into. For our purposes, a simple wipedown is sufficient. Don’t forget to wear gloves.

9.1.3 Rate monitor

To check the rate monitor, first turn on the water cooling to the rate-monitor head. You don’t need more than a trickle here, so don’t overdo it, and check for obvious leaks. Turn on the rate-monitor controller, and check that it is programmed with the correct density and Z-ratio values for the material you are about to deposit. Start the rate monitor, and when it reaches full power, use the LIFE button to check the crystal. Consult the manual to see what is an acceptable value. If the crystal is good, record its LIFE value in your notebook, and press STOP to turn the power off to the head. Shut the cooling water off as well. You will turn it back on again once you are ready to deposit.

9.1.4 Evaporation system

Put a pellet of silver into the evaporation boat. The boat may have some silver left over from the last deposition. If this is the case (and the boat is more than about half full), you may skip this step.

Mount your substrate above the source, as nearly the same distance from it as the rate-monitor head as you can get. Be sure to position it so that the shutter will work, and note the positions of the knob when the shutter is closed and when it is open.

Make sure that the high-current leads are not shorted, and install the evaporation shield. Use aluminum foil to cover any gaps in the shield to keep from depositing a film on the inside of the bell jar.

You may want to make a window using a microscope slide so that you can see your source melt. If you have trouble getting this to work, ask your TA for help.
9.1.5 Closing up

Wipe down the main seal and the baseplate, and close the bell jar.

9.2 Pumpdown

Close the vent valve. Open the main gate valve, and turn on the roughing pump. When the pressure drops below 100 mTorr, turn on the turbo pump. The turbo pump should spin up to “normal operation” in under two minutes. If it does not, shut it down, and contact your TA.

Monitor the pressure as you pump down, and keep a record of pressure vs. time. Plot this on a log-log scale, and compare it with your expectations.

9.2.1 Bake

When outgassing begins to dominate the pumpdown, turn on the current to the evaporation boat, and set the variac to about 30 Volts. This will gently warm the evaporation boat and all the surfaces near it, accelerating the outgassing process. This is a simplified form of “baking” the vacuum chamber, which is standard procedure for attaining a decent vacuum in any system. Most bakeouts are more involved and/or elaborate than this, but this procedure is sufficient for our purposes. Record the pressure vs. time. What you should observe is a rise in pressure until the adsorbed gasses begin to get depleted, after which the pressure will begin to fall again. You may grow a film pretty much any time after this, but the lower the pressure is when you do grow your film, the better your results will be.

9.3 Deposition

9.3.1 Rate monitor

Start the cooling water to the rate monitor. As in your pre-pumpdown test, a trickle is all you need. Watch the pressure. If it rises suddenly, this could be an indication of a leak, in which case you won’t be able to grow a film. Notify your TA immediately if this happens.

If you didn’t check the density and Z-factor before pumpdown, now is the time to do it. The inficon manual has a list of values for both for most commonly-deposited materials.
Start the rate monitor, and when the controller shows MAX PWR, you are ready to start.

9.3.2 Melting the source

With the shutter closed and the evaporation current on, increase the variac’s setting to 70 Volts. Watch both the pellet and the deposition rate displayed on the rate-monitor controller. The pellet and boat should begin to glow, and soon the pellet should melt, forming a bead of liquid. If it does not melt within a couple of minutes, increase the voltage on the variac slowly until it does.

9.3.3 Growing a film

Once the pellet is liquid and most of the dross has cleared from its surface, increase the current further until you get a high rate of deposition as measured by the rate monitor. Two angstroms per second is about right if you can get it. Three is even better. Record this rate in your lab notebook.

1. Open the shutter, and press the ZERO button on the rate monitor.

2. Wait until the thickness reaches your desired goal. If you are growing a film for the weak-localization experiment, between two and three hundred angstroms is about right. If you are making a mirror, the thicker the film is, the better your mirror will be.

3. When your film reaches your desired thickness, close the shutter, press STOP on the rate monitor, and turn the variac back down to zero.

The ideal thickness of your film depends on the level of microscopic disorder in the film, and your actual goal is to grow the thinnest film you can that will still uniformly conduct electricity. In the language of Kaiser (the background reading) you want to grow a film to just over the percolation thickness and then stop. Typically percolation thickness depends on the individual parameters of the deposition system in which a film is grown, and without growing a number of films and doing a thorough study of their microscopic structure and conductivity vs. thickness, you have no way of knowing what it is for this system. Fortunately, weak localization benefits from a lot of disorder in a film, and the particulars of the film are not important for that experiment as long as it conducts.
9.3.4 Venting

Once the evaporation current is off,

1. Turn off the variac and the rate monitor,

2. Shut off the cooling water to the rate-monitor head.

3. Close the main gate valve.

4. Open the vent valve.

You may also press STOP on the turbo-pump controller. It will take some time to spin down, but as long as you have closed the gate valve and left the roughing pump on, that’s OK.

Once the system is vented, raise the bell jar, remove your substrate, and have a look. If you have grown a sample to use in the weak-localization experiment, ask your TA about proper storage of your film until you are able to start that project.

References
