Laboratory Cautions

1. The energy from the RF amplifier can cause burns or even be lethal. Be aware of all electrical connections to the output of this device. Make sure all connections are correct before turning on the amplifier.

2. Prolonged exposure to RF radiation may be harmful. Make sure the quartz end flange is screened.

3. The bottom of the diffusion pump is hot and can cause serious burns.

4. Do not touch the magnet coil conductors while they are operating.

5. Watch water condensation and electricity. Make sure all electrical cords and devices are kept dry.

6. While the magnets are in operation, all magnetic materials should be clear of the machine. The strong magnetic field might throw metal objects.

7. Do not touch the RF antenna/plasma source. This is a delicate device and should not be tampered with.

8. **Do not apply excessive force on the controlled leak valve!** This is a very delicate, precision instrument. Fingertip force is sufficient to open or close the valve.

9. Do not leave the controlled leak valve open when the vessel is not being evacuated. There is no over-pressure release valve on the vessel. If the pressure is increased too much by "leaked" in gas the ion gauge can fly off and hurt you (not to mention destroy the gauge).

10. Before leaving, make sure that the water supply and return for the magnet coils are both off. This prevents problems with leaks and condensation.
Theory

Purpose: The purpose of this experiment is to illustrate basic plasma diagnostics. In particular how to determine the density and electron temperature of a typical plasma by using the method of Langmuir probes. Sidebar: (Irving Langmuir originally coined the term "Plasma" in the 1920's to describe the excited state of gas molecules in vacuum tubes during his 50 year tenure at the G.E. Research Laboratories. Dr. Langmuir received the Nobel Prize in chemistry in 1932 for the theory of covalent bonding, invented the diffusion pump, pioneered advances in the development of radio, invented the technique of cloud seeding, published hundreds of papers in Physics, Chemistry, and Biological science journals, and held over 60 patents.)

A plasma is a gas of charged and neutral particles. But not all ionized gases are plasma. In order for an ionized gas to be a plasma the gas commonly satisfies three criteria. The plasma must have an overall charge close to zero at all times. If there are any charge concentrations in the ionized gas or external potentials imposed on the gas, their effects are shielded by the plasma in a distance short compared to the size of the experiment.

For example, if we put a positively charged sphere in a plasma, electrons will be attracted to the sphere and ions will be repelled. This will give rise to a cloud of negative charge around the sphere. This "sheath" will shield the rest of the plasma from the positive charge on the sphere. If the experiment size (dimension of the plasma) was only as large as the "sheath" (the Debye length) then there would be no plasma, just a cloud of negative charge in the vessel. Second, in order for an ionized gas to be a plasma it must also exhibit collective behavior. All elements of charge and current in a plasma experience forces from all other elements of charge and current both close and far. This gives rise to many of the interesting properties of plasma. A third criterion for a plasma is that the motions of the particles must be dominated by electromagnetic forces. This is different from a gas at room temperature and atmospheric pressure for example, where collisions dominate the motions of the particles. For an ionized gas to be considered a plasma then it must exhibit "group behavior" and be dominated by electromagnetic forces.
Three important properties of a plasma in experimental physics are the density of the plasma, the ion temperature, and the electron temperature. These properties can be determined by the use of a Langmuir probe. The Langmuir probe in this experiment (see fig A) consists of a 1mm diameter molybdenum disc welded to a tungsten wire. Molybdenum is used for the tip because it has a large work function. This reduces emission of electrons from the tip of the probe. The wire is insulated from the plasma and the vessel so that only the molybdenum disc is exposed to the plasma.
By applying a potential between the disc and the vessel wall and monitoring the current through the probe, the electron temperature and plasma density can be determined. The graph of current through the probe vs. potential applied across the probe is shown in figure B. For large positive potentials (the probe positive with respect to the plasma), all ions are repelled from the probe, and a saturation electron current is reached (portion I). A negative "sheath" forms around the probe. All electrons that strike the probe are due to the thermal motion of the electrons passing into this "sheath." As the potential on the probe is made less positive, the more energetic ions (in the tail end of the Maxwellian distribution) are able to overcome the sheath. In the first knee region (region II on the graph), more and more ions are over-coming the sheath, while some electrons are now repelled. At zero volts on the probe, there is still an electron current (negative conventional current) present. This occurs because the thermal velocities of the electrons and ions vary by the ratio of the square root of their masses. Many more electrons strike the probe tip than do ions in the same time.

As the probe is made negative, some electrons are repelled from the tip. At the voltage (point III on the graph) called the "floating potential" no current flows through the probe. At this point enough electrons are being repelled from the tip so that the ion and electron currents are equal. Note that this voltage is displaced from zero volts because of the mass disparity of ions and electrons. If the mass and temperature of the positive and negative charges were the same then the floating potential would be zero.

As the voltage is brought below the "floating potential", more electrons are repelled from the tip. As the higher energy electrons in the Maxwellian distribution of velocities are repelled a knee region (region IV) is formed. At large negative voltages (region V), all electrons are repelled, and ion current is at saturation. A positive "sheath" forms around the tip and all current is due to the random thermal motion of the ions passing into the "sheath." Notice how the ion portion of the curve is smaller in magnitude than the electron portion. This again arises from the disparity of masses. Because the electrons are moving at higher velocities than ions their current flux is greater (qnv). Now we will see how by measuring this curve we can measure plasma density and electron temperature for a plasma.

By fitting region IV of the current-voltage graph to a Maxwellian distribution of velocities the temperature for electrons can be determined. Ion saturation current must be subtracted before the curve can be fitted. This can be done by drawing a straight line through the ion saturation current on the graph. All measurements are then made with respect to this ion saturation current. Once the electron temperature has been calculated from the data, the density can be determined. For any cross sectional area in the plasma, the flux of particles through a given area is the density of particles times the drift velocity of the particles. The current flux is then given approximately by: J=qnv/2, the factor of 1/2 arises from averaging the cosine function over a hemisphere around the collection disc (see Plasma Diagnostics, Chen). It is important to note that in Chen's derivation he is only assuming that particles are being collected on one side of a disc. For this reason his current density has an extra factor of two in the denominator.
To determine the current flowing into the probe we multiply the current density $J$ by the area of the probe tip. Solving

$$I = JA = qnvA/2$$

for the plasma density yields

$$n = 2I/qvA$$

where $A$ is the area of one side of the disc. In this experiment, the argon ions are singly ionized so $q = e$. For the determination of $n$, find the beginning of the ion current saturation (left of region IV) and move three electron temperatures (in Volts) to the left on the graph. Take the current from this point on the graph for the density calculation. The drift velocity of the ions is not used in this calculation however. Ions typically move to the probe with speeds of

$$v = (KTe / Mi)^{1/2}$$

where

- $Te$ = the electron temperature,
- $K$ = Boltzmann's constant,
- $Mi$ = the mass of the ion.

Some assumptions are made in the calculations. One assumption that is made is that the "sheath" remains small and constant for all bias voltages put on the probe. However, this does not happen in reality. The sheath size actually increases with increasing voltage. This will show up in the saturation currents on the experimental graphs as a slope in the saturation regions.
Fig. C Source Configuration

Fig. D Circuit Diagram

Figure E. Pumping System
Experimental Outline

**Ion gauge**: The ion gauge is a device used to measure low pressures (less than one mtorr). It consists of a glass tube (located above the vessel) with a filament and two other electrodes. A voltage is placed on the tungsten filament which heats the filament and causes electrons to be boiled off. The electrons fill the glass tube and ionize neutral atoms. A negative potential is placed on the straight wire to collect ions. Ions are then collected as a current in the gauge. This current is proportional to the density of neutral atoms in the gauge.

**Ion gauge controller**: The ion gauge controller converts the current measured from the ion gauge into a pressure. This gauge must be calibrated for different gases. In this lab the gauge is calibrated to argon. Also on the ion gauge controller there are gauges labeled "TC1" and "TC2." This stands for "thermocouple 1" and "thermocouple 2." These gauges give the pressure in the foreline and the vessel respectively (see pumping down procedure for details on the pump system). This is done with the use of a heater and a temperature sensor in direct contact with each other. Since heat transfer in a gas is proportional to the concentrations of atoms, the rate of cooling of the thermocouples depends on the amount of gas present (pressure). The thermocouples are usually used for higher pressures (higher than 1 mtorr).

**Roughing pump**: The roughing pump is a conventional 2-stage rotary pump which is used to get the vessel and diffusion pump down to pressures on the order of one mtorr.

**Diffusion pump**: The diffusion pump is used to get down to pressures below $10^{-3}$ torr. Oil at the bottom of the pump is heated and caused to vaporize. The oil then rises rapidly through a central cylinder and is sprayed onto cooled walls which causes the oil to condense and run down the walls back to the bottom of the pump. By having the oil spray in a downward direction, the oil molecules collide with gas molecules that randomly "walk" into the pump and force them into the bottom of the diffusion pump. Molecules trying to get out of the diffusion pump will be continually knocked back down into the bottom of the diffusion pump by the oil molecules. Eventually these trapped gas molecules will wander out to the roughing pump and are expelled. The diffusion pump must always be run at pressures lower than 100 mtorr. If the pump is allowed to run at too high of a pressure in the presence of oxygen, the silicon based oil will oxidize. (Definitely not good!) What’s another word for Silicon Oxide? (Hint: It’s at the beach.)

SEE THE SECTION ON PUMPING DOWN FOR THE CORRECT OPERATING PROCEDURES OF THE PUMP.
RF source: Keithley 3390. The Arbitrary Waveform Generator produces radio frequency (RF) waves in the 0 – 50MHz range. The default frequency is 1kHz. Both the frequency and level of the RF can be controlled from here. The RF power level is the power that the indicated voltage would give rise to when driving a 50 ohm load. The amplifier (see below) has a maximum power input of 0.8mW, so do your math for how much indicated voltage you need to output in order to avoid damaging the amplifier.

Pulse generator: HP8012B. The pulse generator outputs a square wave to be mixed with the RF signal from the RF source (See figure C). Both pulse width and delay are controlled here.

Mixer/Switch: The mixer combines the square wave from the signal generator with the RF output to give a pulsed RF signal. It acts like a switch letting the signal through on the positive edge of the square wave and cutting it off on the negative edge of the square wave.

Amplifier: Amplifier Research Model 200L. This unit amplifies the RF signal which comes out of the pulse generator. The maximum input power is 0.8mW — any power higher than that could damage the amplifier. This unit is capable of delivering 200W of power, so care must be taken when it is used not to put too much power into the RF antenna. This unit also has a high voltage output and care should be taken not to touch the output cable connector while it is in operation. USE "STAND-BY" BEFORE TOUCHING ANY WIRING. USE EXTREME CAUTION!!!! RF POWER CAN BURN THE SKIN AND CAN EVEN BE LETHAL!!!!

Magnet power supply: KEYSIGHT N8755A. The power supply is capable of delivering 5,100W (30V/170A) of DC power, and is used to supply current to the magnet coils on the apparatus. It can be operated in either constant voltage (CV) or constant current (CC) mode. For this experiment, the CC mode is to be used. The power supply should never be running unless the chilled water is running to cool the magnets. When operating with large currents, be sure to turn the current down before shutting down. Repetitive start-ups may cause a current surge and produce forces which can damage current carrying elements in the apparatus.

Controlled leak valve: The calibrated silver knob on the gas pressure regulator is used to allow controlled amounts of gas to be leaked into the system. This valve should always be closed completely when the pumps are off or the vessel is at atmosphere since there are no automatic relief valves to let out high pressures in the vessel. NOTE: Excessive positive pressures will launch the Ionization Gauge tube into the ceiling! This can occur even while the pumps are operating and the Hi-Vac valve is open. If the chamber becomes over pressurized, make sure the Hi-Vac valve is closed before using the vent valve above the diffusion pump to relieve the pressure.

The leak valve is a sensitive instrument. DO NOT USE EXCESSIVE FORCE TO OPEN OR CLOSE THE VALVE! Doing so will damage it beyond repair. Using two fingers provides enough force to actuate the valve.
The valve above the silver controlled leak valve (brass knob, closer to the tank) should always be left open when the leak valve is closed. This keeps the line at positive pressure with respect to atmosphere and prevents air from contaminating the gas between the tank and control valve. Do not apply excessive force when closing any of the valves as this will permanently damage the valve seals. Also be aware that there is one additional valve on the gas line where the line enters the chamber. This valve should usually be kept open except when changing gas cylinders.

**Oscilloscope: (Tektronix digital phosphor scope)** A memory storage oscilloscope is used to measure the currents drawn in the probe. (See procedure and the oscilloscope book for usage.)

**Bipolar power supply: (Kepco Model BOP 100-1M)** This power supply is used to sweep positive and negative Bias voltages on the probe.

**Pump down procedure**

Read this section several times until you understand it completely, before operating the equipment. Refer to figure E. for a diagram of the pumping system.

1. The Hi-Vac valve should be closed, and the foreline valve should be switched to the foreline position (toward the diffusion pump).

2. Turn on the chilled water to the diffusion pump. Open the return valve first, then the supply valve. Leave the yellow valves closed until you run the magnet.

3. Turn on the roughing pump and pressure gauge controller. At this stage you are only pumping down the diffusion pump with the roughing pump.

4. Monitor the pressure on thermocouple one. When the foreline pressure (thermocouple one) falls below 100 mtorr then turn on the heater of the diffusion pump. There might be an initial increase in pressure due to the out-gassing of the oil as the diffusion pump heats up. Continue to pump on the diffusion pump until the pressure is as low as it will go (~20 mtorr).

   **DO NOT OPEN THE HI-VAC VALVE AT THIS TIME!**

5. Switch the foreline valve over to the roughing valve position. While monitoring the pressure in the foreline, pump the vacuum vessel below 100 mtorr as indicated on thermocouple two. If at any time the foreline pressure goes above 100 mtorr, switch back to pumping on the diffusion pump by closing the roughing valve and then reopening the foreline valve (switch the valve from roughing position to foreline position). Pump the diffusion pump back down to as low as it will go, then start step 5 again. Repeat this if necessary until the vacuum vessel will stay below 100 mtorr.
6. Once the vessel pressure stays below 100 mtorr, switch the valve to the diffusion pump, and very slowly open the Hi-Vac valve. This will cause a temporary rise in foreline pressure. Do not allow the foreline pressure to exceed 200 mtorr for more than brief periods.

7. Give the vacuum vessel about fifteen minutes to pump down. Turn on the ion gauge using the ion gauge controller. The pressure should display on the order of $10^{-4}$ torr or lower. Wait until the chamber is pumped down into the $10^{-5}$ range before proceeding.

**Shut down procedure**

1. Turn off the ion gauge.

2. Close the variable leak valve.

3. Close the Hi-Vac valve and turn off the heater on the diffusion pump (keep the chilled water running).

4. When the diffusion pump reaches room temperature, close the foreline valve (middle position) and turn off the chilled water and the roughing pump.

5. If you intend to change the probe position, or need to break vacuum for some other reason then carefully bleed air into the vacuum vessel using the leak valve or the vent valve above the diffusion pump to bring the vacuum vessel slowly to atmospheric pressure. Make sure the vent valve is closed before continuing.

6. You may allow the mechanical pump to run continuously for the duration of your experiment in order to maintain a vacuum on the apparatus and reduce pump down time.
Data Acquisition

Set up the circuit in figure D. The oscilloscope should be triggered from the output of the pulse generator. Choose R from 100k ohms to 400k ohms for best results. There should be a blue box with a series resistor already on your bench. Check to see if the ion gauge is on by looking at the filament. If it is not refer to the pumping down procedure to get the gauge running.

Once the gauge is running make sure that the pressure is on the order of $4 \times 10^{-5}$ torr or lower. If it is not, then check to see if controlled leak valve is closed to ensure that this is not the source of the higher pressure. If the pressure is higher than this with the valve closed then talk to Ferdinand Badescu regarding the possibility of a leak.

Turn the RF source, pulse generator, and amplifier on to warm up. The pulse length should be initially set to $\sim 0.65$ms with a 50% duty cycle.

Open up the controlled leak valve (about 10 full turns) until a pressure of $6 \times 10^{-4}$ torr is reached. There will be fluctuations in pressure for a short time after the valve has been opened. Be sure to check this pressure about five minutes later and readjust it back to this pressure. (This can be done after the other startup procedures have been completed.)

Turn the chilled water on to partial flow by first opening the return valve completely and then opening the supply line just enough to move the flowmeter. Make sure the magnet coils do not get hot. Or, if you detect an excessive amount of condensation, particularly on the return line, then close the supply valve a little further. Make sure that when you are turning on the water that the return valve (R) is opened first. When turning the chilled water off, always turn the supply (S) off first, and then the return.

Once the chilled water is running, switch the magnet power supply to "standby" and turn it on. Give it a minute to warm up, then flip the switch to "operating". Dial the current to produce the magnetic field you wish to use. Look on the calibration curve to find out what current should be used to produce the desired magnetic field. 100 amperes is a good current to use (B=842 Gauss). Verify the presence of a magnetic field either by using a gaussmeter or a small steel screwdriver near the magnet coils.

Dial in a frequency of $\sim 25$MHZ on the RF source and adjust the RF level to .35 Vp-p (peak-to-peak). Monitor the SWR meter for forward and reflected power. Try to use a frequency that minimizes reflected power.

NEVER RUN THE RF SOURCE HIGHER THAN 350 millivolts peak-to-peak!
When the "Standby" light comes on the amplifier is warmed up. Push the "Operate" button. Briefly look down the end of the vessel to see that a plasma has been ignited. If there is no plasma then check to make sure all procedures were followed correctly. If your parameters (RF level, pulse period or rep. rate, gas pressure, etc) are different than the suggested values, they may be too far out of range to ignite a plasma.

Turn on the Bipolar power supply and oscilloscope. The trace on the oscilloscope should show an RF burst followed by a decay. Adjust the pulse generator so that the pulse width is ~0.65ms on the scope with a 0.75ms (or longer) delay between pulses.

Note that the circuit in Fig. D does not directly measure the quantities that are graphed in Fig. B. Measure the resistances in your circuit and use Ohm's law to relate the desired quantities (I and V of the probe) to the measured quantities. In particular, measure resistor R to a reasonable precision (use a 5½ digit multimeter or better) and measure the input resistance of the scope using the circuit of Fig. D without a plasma. To do this, set the bias voltage to some manageable value, measure the voltage drop across R and compute the current $I_s$. Then use $R_{\text{Scope}} = V_{\text{Bias}} / I_s$. Current through the scope will of course vary with probe voltage. Remember, this current must be subtracted from the total current through R in order to determine probe current for each data point.

Re-attach the circuit to the plasma probe and put the RF Amplifier in the operating mode. Avoid touching the center conductor of any of the coax cables connected to either the RF amplifier, the Langmuir probe, or any parts which are directly connected to the center conductors.

Turn the Bias voltage to zero volts and put the oscilloscope in the average mode. Position the (highlighted) vertical cursor on the scope at a high point of the plasma decay curve greater than 100usec after the RF has turned off. The DC voltage at this point on the curve will be displayed numerically near the top of the oscilloscope display next to the "@" sign. Record this as Probe voltage $V_p$. Also record the Bias voltage $V_B$ for many positive and negative Bias voltages. Calculate total current $I_T = (V_P - V_B)/R$; then Scope current $I_S = V_P / R_S$; and then Probe Current $I_P = I_T - I_S$. Plot $V_P$ vs. $I_P$ as in Fig. B.

When shutting down the equipment, put the RF amplifier on "standby" and let it cool down for about five minutes.
Turn off the bipolar power supply and the oscilloscope.

Dial down the magnet current to about 50 amperes and switch it to "standby".

Turn off the RF source, and the pulse generator.

When the air coming out of the magnet power supply is cool turn the magnet power supply off.

Turn the chilled water to the magnets off after the magnet power supply is completely off. First turn off the supply, and then turn off the return (opposite of start-up).

Close the controlled leak valve all the way (remember not too tight). The ion gauge controller should read four times ten to the minus five torr or less again. When the air coming out of the back of the RF amplifier is cool, then it can be shut off too. Make sure all equipment is off when you are done. (Leave the diffusion pump and ion gauge running until you are completely done with the experiment.)

After you plot the probe current vs. the probe voltage, identify the various regions such as “ion saturation” and “floating potential” on your graph. Subtract the ion saturation current from I_P, take the logarithm, and calculate the electron temperature from your data.

Calculate the plasma density from your data.

Measure the ion saturation current vs. time in the plasma “afterglow” (after the RF is turned off). (You must have done the I-V trace of the experiment first before you can do this part.) Use the calibration factor you found in step 22 to convert these data to an approximate graph of density vs. time. Assume that T_e does not change appreciably after the first 100 uS of the afterglow.

If you have time, move the probe to the other port to explore the plasma characteristics at a different position in the plasma or measure the electron temperature at a different time. Alternatively, you could explore the properties of other inert gasses such as neon or helium.
References
