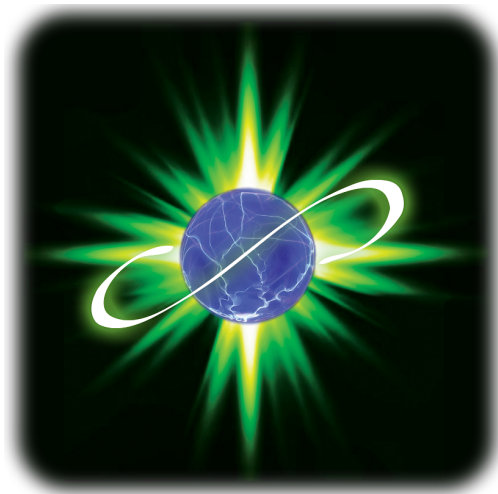


Plasma Etching: Safety Operating Procedures

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1 Introduction

The purpose of this document is to provide a comprehensive guide to properly and safely plasma etch polystyrene samples with an oxygen gas in vacuum. It will be structured in a hopefully reasonable format. First, we will discuss the setup and terminology in Section 2 such that a reader can become familiar with the equipment and feel more comfortable around it. This discussion will also make the procedures more understandable, so we encourage looking at this section carefully. We will follow this discussion with detailed procedures in Section 3 for effectively and safely completing a plasma etch. Finally, we will briefly recap the important safety hazards to be aware of in Section 4.

If at any time you feel uncomfortable with the setup or any of the procedures, do not operate the equipment until you have sorted it out. Do not hesitate to contact a knowledgeable individual for help in understanding any part of this document, or for assistance in running the etching itself. See Section 5 for details on people available for contact.

1.1 Goal of Etching

Before jumping into the first section of this SOP, it may be useful to a reader to know why we are using a plasma etching setup at all. As of November 2015, the Donnelly Research Group is attempting to develop a method of delivering individual polystyrene spheres to the focus of a high-intensity laser. The current method involves depositing these spheres onto a silicon substrate in a deposition process. Unfortunately, through this process, the spheres wind up bonding to each other. This is not acceptable because they appear to remain bonded after being ejected from the substrate via laser ablation. That would cause the spheres to enter the focus of a high-intensity laser in clusters, not as isolated individuals.

The details of our experiment are not important to understand, however, to be able to carry out a plasma etch. The key point is that we want the spheres to not be bonded on the substrate after our depositions. So, with this in mind, the purpose of the plasma etching system is to break the bonds between polystyrene spheres in order to create isolated spherical targets. When a sample (a silicon substrate with polystyrene spheres deposited on it) is in

the presence of an oxygen plasma, the polystyrene will be etched out, which will dissolve the bonds between spheres. It will also reduce the sphere sizes because the plasma will etch into every area of contact with polystyrene at equal rates, presumably. This is another important factor to consider when using plasma etching.

2 Setup and Terminology

In this section, we will describe the setup for plasma etching and define some of the terms that will be used frequently in the procedures section. We will first begin with the overall setup of the vacuum pumps' interactions with the etching chamber.

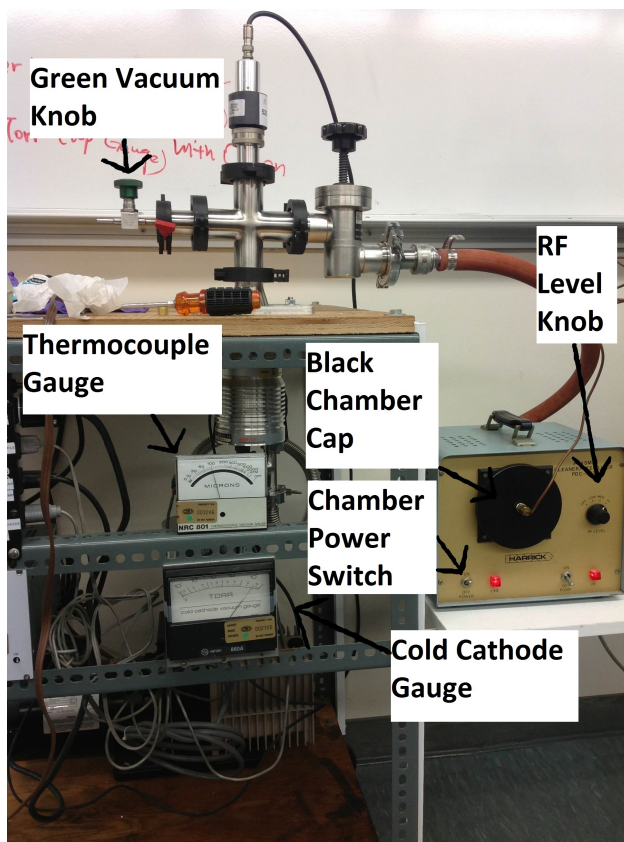


Figure 2.1: View of the vacuum pump system and etching chamber setup, with pertinent labels.

From this figure, there are several things to note. First is that the labeled entities will

be referenced throughout the procedural sections of this document. They are important to be familiar with. In the picture itself, the plasma etching chamber is the brown-faced box with a gray cover and a leather handle on top to the right of the image. Connected to this chamber is a hose connecting it to the vacuum system, which is contained in the cart that occupies the remainder of the image.

Notice as well in Figure 2.1 that the black chamber cap has a copper tube connected to it and directed out of the frame to the right of the image. This tube connects to the oxygen canister that is used to produce the oxygen plasma in vacuum.

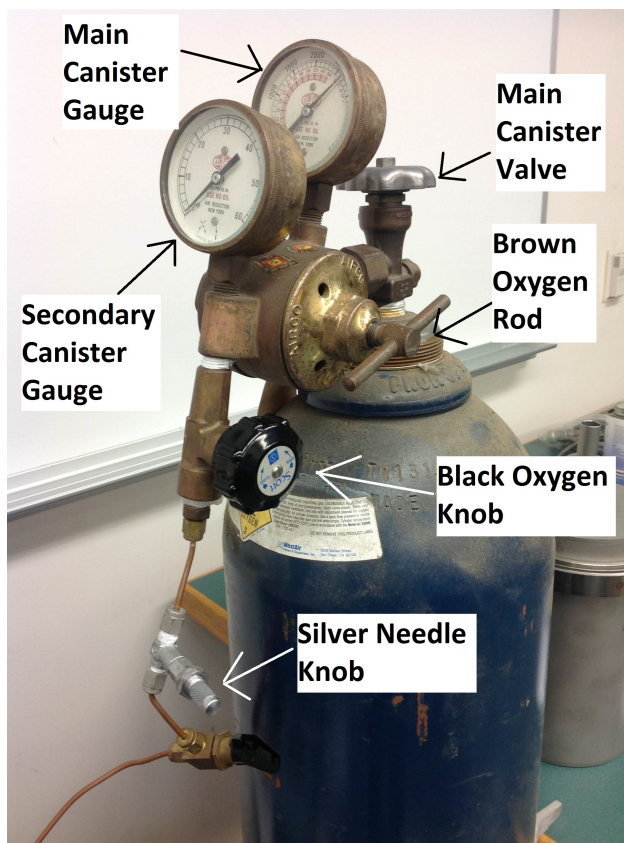


Figure 2.2: View of the canister containing the oxygen gas used for etching, with pertinent labels.

This figure shows the various components of the regulator on the oxygen gas canister. Note that the lowermost black knob visible in the picture (just below the Silver Needle Knob) is another way to control the oxygen flow through the tubing. This may be important when

fiddling with the knobs and rods to get the flow rate and pressure to the desired values, but mostly you will just have that open and adjust with the other components.

The final figure, Figure 2.3, below, shows the switchboard for turning on the Rough Pump and the Molecular Drag Pump, as well as their respective gauges, the Thermocouple Gauge and the Cold Cathode Gauge. The LED switches light up when in the On position, as shown in the image.

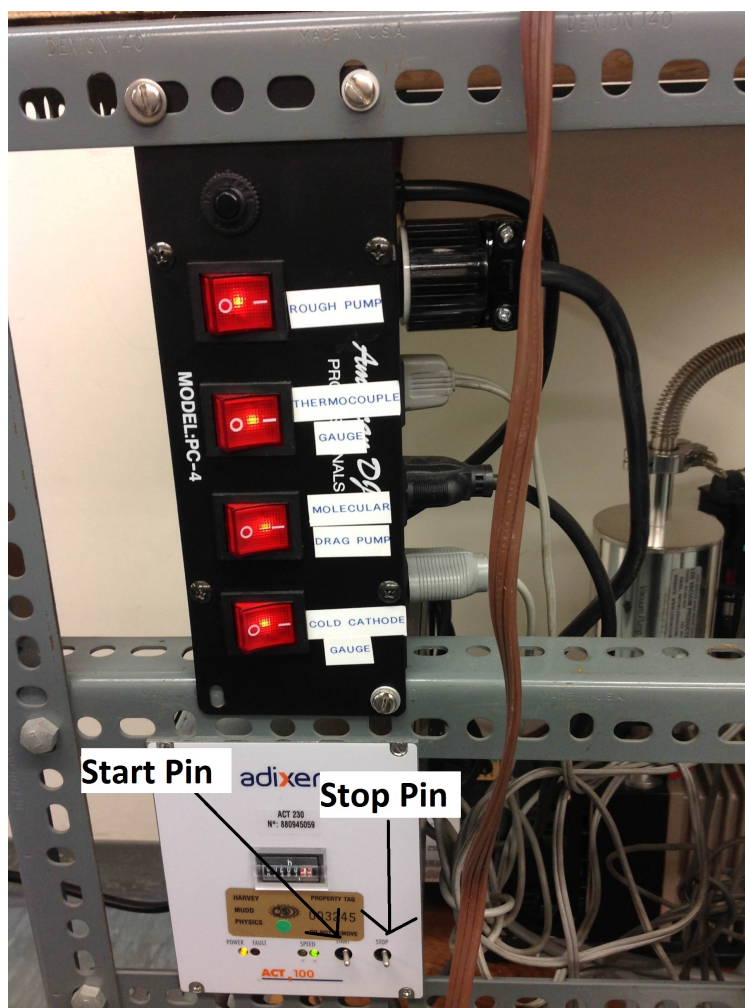


Figure 2.3: Photo of the switches which control the pumps and gauges.

3 Operating Procedures

The procedures in this section will detail steps to safely complete all plasma etching in our current setup with our current equipment.

3.1 Procedure: Pumping Down

Follow this procedure for starting a pumpdown cycle for an etching trial.

- (1) Before pumping down, ensure the following:
 - (i) Have the Black Oxygen Knob completely tightened such that oxygen is not flowing from the canister.
 - (ii) Have the Green Vacuum Knob completely tightened as well such that air is not getting into the vacuum system. NOTE: This means to have the knob finger-tight. Too much tightening could cause damage.
 - (iii) Put in the sample on a clean microscope slide and center the slide with sample in the plasma etching chamber so that the sample is between the two coil rings.
- (2) Press the Black Chamber Cap on the etching chamber opening, flip the Rough Pump and Thermocouple Gauge switches. You can cease pressing the Black Chamber Cap with your hand when the pressure is sufficient to hold it in place.
- (3) Once the Thermocouple Gauge reads about 10 microns, flip the Molecular Drag Pump switch on. Then flick the Start Pin to the left and wait until the green LED is visible. NOTE: The light takes a while to turn on after flicking the Start Pin.
- (4) Flick the Cold Cathode Gauge switch.
- (5) Once the Cold Cathode Gauge reads about 2×10^{-4} Torr, oxygen can be introduced. Continue to the secondary procedures as appropriate.

NOTE: In steps 3-4, both the Thermocouple Gauge and the Cold Cathode Gauge could potentially increase slightly, but this is fine. These pressures will decrease again once the Molecular Drag Pump is warmed up.

3.2 Procedure: First-Time Oxygen Settings

Follow this procedure for introducing oxygen to the system if previous knob settings have not been established or if the Silver Needle Knob had to be tightened for the Rough Pump.

- (1) Make sure the main canister valve is open (the Main Canister Valve should read a large PSI number; in November 2015, it reads about 2500 PSI when opened). Also have the Black Oxygen Knob and Silver Needle Knob completely tightened.
- (2) Make sure the Brown Oxygen Rod is untightened and feels loose (the Secondary Canister Gauge should read zero and the needle should not be raised at all). Slowly tighten the rod until the needle just pops above zero.
- (3) Open the Black Oxygen Knob slowly while observing the Cold Cathode Gauge to make sure the pressure does not increase too drastically. It should open all the way and still have the pressure under 2 mTorr.
- (4) Open the Silver Needle Knob VERY carefully because it is extremely responsive. You do not want the pressure to jump too quickly or too largely here as it could damage the Rough Pump. The goal is to get the system to a set pressure with the oxygen flowing in at a given pressure. As of 11/20/2015, our target pressure is 5 mTorr on the Cold Cathode Gauge.
- (5) Once the reading on the Cold Cathode Gauge is steady at the target pressure, turn the plasma etching chamber power on. Turn the RF Level Knob on the chamber to the “Hi” setting and make sure there is a light purple-pink color in the chamber indicating a plasma. Simultaneously, if a plasma is observed, start a stopwatch. Otherwise, increase the pressure slightly with the oxygen controls and try to initiate the plasma again.

- (6) Turn the RF Level Knob back to the “Off” setting once the desired etching time has elapsed, as determined by the stopwatch. Continue to the venting procedure.

3.3 Procedure: Using Previous Oxygen Settings

Follow this procedure for introducing oxygen if the Brown Oxygen Rod and Silver Needle Knob have already been set to the proper positions for the appropriate oxygen concentration in the etching chamber.

- (1) Make sure the Main Canister Valve is open (the Main Canister Valve should read a large PSI number; in November 2015, it reads about 2500 PSI when opened).
- (2) Open the Black Oxygen Knob slowly while observing the Cold Cathode Gauge to make sure the pressure does not increase too drastically. Theoretically, if the Brown Oxygen Rod and Silver Needle Knob are set from a previous trial at a previous target, opening this Black Oxygen Knob should bring the pressure to that target. As of 11/20/2015, our target pressure is 5 mTorr on the Cold Cathode Gauge.
- (3) If the target pressure is not reached just from opening the Black Oxygen Knob, see steps 2-4 of the first time oxygen settings procedure and apply as appropriate to reach the target pressure.
- (4) Once the reading on the Cold Cathode Gauge is steady at the target pressure, turn the plasma etching chamber power on. Turn the RF Level Knob on the chamber to the “Hi” setting and make sure there is a light purple-pink color in the chamber indicating a plasma. Simultaneously, if a plasma is observed, start a stopwatch.
- (5) Turn the RF Level Knob back to the “Off” setting once the desired etching time has elapsed, as determined by the stopwatch. Continue to the venting procedure.

3.4 Procedure: Venting

Follow this procedure to vent the system back to air.

- (1) Tighten the Black Oxygen Knob to stop the flow of oxygen. NOTE: the Silver Needle Knob may also need to be tightened to completely stop the flow of oxygen. If this is the case, then for the next run, the first time settings procedure will have to be followed again.
- (2) Flick the Stop Pin to the left on the Molecular Drag Pump and wait for the pump to spin down. Then, flick the Cold Cathode Gauge and Molecular Drag Pump switches to the off position.
- (3) Flick the Rough Pump switch to the off position.
- (4) Slowly loosen the Green Vacuum Knob to allow air back into the system.
- (5) Once the hissing noise ceases (or the Black Chamber Cap to the etching chamber falls off), you know the system is at air. At this point, venting is complete. NOTE: When venting, one should not simply allow the Black Chamber Cap to fall. Rather, hold it lightly with your hand until there is no longer a vacuum pressure holding it on, at which point you can gently place it to the side wherever is appropriate.
- (6) If you are completely done with all etching trials for the day, close the Main Canister Valve on the oxygen tank. Otherwise, return to the pumping down procedure and continue with more trials.

4 Safety Hazards

Most of the things to watch out for have been noted in Section 3 above, so we encourage the reader to look through these sections in some detail: they will provide the best sense of what can go wrong and at what times. During the etching process, there are really two main hazards that one must take precaution against and seek to minimize, mostly through acting carefully, thoughtfully and deliberately at every step.

The two main hazards are: (i) Flooding the Rough Pump and Molecular Drag Pump when introducing oxygen to the system, and (ii) Damaging the main oxygen canister.

For (i), care should be taken when introducing oxygen to the system so that the system pressure does not increase too rapidly. If such a pressure change did occur, both the Drag Pump and the Molecular Gauge Pump could potentially fail, which in the worst case could induce an explosion.

For (ii), the main oxygen canister is pretty hefty and well-built already, but it is important to keep in mind that it could easily become a dangerous projectile if damaged. Therefore, specific care should be taken to not damage the cap of the canister where the Main Canister Valve is located. This includes being extra careful when removing and fixing any of the components in Figure 2.2 as these could potentially affect the Main Canister Valve.

5 Contacts

For more information on this document or plasma etching with our equipment in general, feel free to contact any of the following individuals.

Chemistry Faculty	
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