

Pulsed Lasers Deposition Procedure

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Cleaning Procedures

We should always wear gloves, lab coat, and mask when working with the targets and samples. Also always wear eye protection when using the laser.

0.1 Wet Sanding Procedure

1. We start by folded up four paper towels and placing them in the bottom of the sink. Place a piece of glass over the paper towels so that each paper towels are located at a corner of the glass.
2. Place the desired grit sand paper face up on the glass.
3. Turn on the deionized water so that it drips on the sand paper at a rate of about one drip every 3 seconds.
4. Place the material that you are sanding face down on the sand paper. Fold up a paper towel and place it over the material. Sand the material by pressing down on the paper towel with three fingers and rotating the material in a circular pattern.

0.2 Dry Sanding Procedure

1. Dry Sanding is done in the hood by the door. Clean the sanding stone by squirting it with methanal and then wiping it clean with a paper towel.
2. Place the desired grit sand paper face up on the grinding stone.
3. Place the material that you are sanding face down on the sand paper. Sand the material by pressing down with three fingers and rotate the material by 60 degrees while moving the target in a circular pattern.

0.3 Ultrasonic Cleaner

1. Place a cleaning solution, methanal or deionized water, in a small glass beaker.
2. Place the material in the solution and put the glass beaker in the card board holder.
3. Turn on the ultrasonic cleaner and set the timer for the amount of time you would like to clean the material.

1 PLD Preparation

1.1 Substrate Preparation

1. First we should scribe the samples name on be back of the substrate with a glass scribe.
2. We will clean the substrate by placing the it in a bath of methanal and setting the ultrasonic cleaner to run for 15 minutes.
3. Third we will clean the substrate by placing the it in a bath of deionized water and setting the ultrasonic cleaner to run for 15 minutes.
4. Dry with the compressed nitrogen and set face up on weight paper to dry for 5 minutes.

1.2 Substrate Holder Preparation

1. Wet sand the removable disk at the center of the substrate holder. There should be no deposited materials visible on the top side of the disk when done sanding.
2. Dry with the compressed nitrogen and set face up on weight paper to dry for 5 minutes.
3. Select a mask and wet sand it so there is no deposited material visible on either side.
4. Dry the mask with the compressed nitrogen and set face up on weight paper to dry for 5 minutes.
5. (*optional*) If the rest of the substrate holder needs to be cleaned, then prepare a solution of HCl mixed with water and soak the substrate holder for 10 minutes. Place the screws and holding arms in a HCl bath and run in the ultrasonic cleaner for 10 minutes.

1.3 Target Preparation

1. Dry sand the surface of the target making sure to rotate the target by about 60 degrees with each sanding.
2. Keep dry sanding until the track is no longer visible on the surface of the target.
3. Make sure to use a single piece of sand paper for each target. When done dry sanding, put the sanded material in the proper plastic container in the hood.
4. While under the hood, spray compressed nitrogen over the surface of the target to remove any loose material.
5. Remove the target from the hood and place it face down on a piece of weight paper.

2 Substrate and Target Assembly

2.1 Target Assembly

1. Loosen the three screws on the target holder to allow clearance of the target.
2. Place target holder over the face down target.

3. Tighten the screws on the target holder until the target is tightly held in the holder. Do not over tighten the screws or the target will break.
4. Spray the surface of the target with compressed nitrogen and check to make sure the target is flush with the top of the target holder. If the target is not flush, then repeat the above process.

2.2 Substrate Assembly

1. Place the disk back on the center of the substrate holder. Reassemble the substrate holder if it was cleaned.
2. Loosen the four screws of the substrate holder clamps.
3. Place the substrate on the disk. If the substrate is transparent, check if optical fringes can be seen. It is necessary to keep everything very clean so the substrate is flush with the disk. Repeat cleaning of the disk and substrate if no fringes are seen.
4. Position mask over the substrate and place two clamps on opposite sides of the substrate over the mask. Make sure the clamps will press down on the substrate and not only the mask or you will bend the mask.
5. Press down with a finger on each clamp and evenly distribute the force on each clamp. While pressing down, screw down the two clamps until tight.
6. Place the other two clamps over the mask making sure the clamps will press down on the substrate. While pressing down as before, screw down the two clamps until tight.

3 Loading Substrate and Target

3.1 Placing Substrate and Target in Load Lock

1. Close the PLD/Load lock valve, labeled 8 in figure 2 on page 10.
2. Turn off load lock low pressure pump on control panel, labeled 9 in figure 4 on page 12. Make sure to turn off the load lock low pressure gauge on control panel, labeled 1 in figure 4 on page 12. Take off the load lock lock, labeled 4 in figure 2 on page 10.
3. Close the load lock rough pump valve on control panel, labeled 8 in figure 4 on page 12. Open the nitrogen gas valve to bring up the pressure in the load lock, labeled 20 in figure 2 on page 10. You should hear a hissing sound when you open the valve and the load lock door should open on its own. Close the nitrogen valve when load lock's door opens.
4. Insert the substrate holder with the substrate facing down into the substrate loader arm. We need to align the three hooks on the substrate loader with three notches on the substrate holder. We can chose any three of the six notches on the substrate holder as they are all the same. Rotate the substrate loader at the end of the arm, labeled 1 in figure 2 on page 10. The hooks should engage the notches and hold the substrate holder in place.
5. Place the target holder target up on the substrate loaders fork below the substrate holder.
6. Close the load lock's door and lock the load lock, labeled 4 and 5 in figure 2 on page 10.

7. Open the load lock rough pump valve, labeled 8 in figure 4 on page 12, and wait 5 minutes of pumping before turning on the low pressure gauge.
8. When the pressure reaches 10^{-5} torr turn on the load lock's low pressure pump on the control panel, labeled 9 in figure 4 on page 12.
9. Wait until the load lock pump down to the pressure of the PLD chamber before proceeding.

3.2 Loading Substrate and Target in to PLD chamber

1. When the pressure in the load lock and PLD chamber are the same open the PLD/Load lock valve, labeled 8 in figure 2 on page 10.
2. Using the y-axis loading arm adjustment, labeled 2 in figure 2 on page 10, angle the loading arm at about 10 degree angle as shown in figure 1.

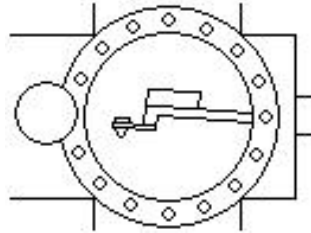


Figure 1: The load arm should be positioned at an angle with respect to horizontal while loading the target and substrate into the PLD chamber.

3. Turn on the gas to the substrate shutter. The nitrogen gas cylinder is located on the wall next to the PLD control panel tower.
4. Open the substrate shutter by twisting the shutter control located on the end of the PLD pump, labeled 17 in figure 2 on page 10.
5. Insert the substrate loader into the PLD chamber being careful to not to bump the side or bottom of the chamber's walls.
6. Position the substrate loader so the target is right above the target holder. Raise the target holder from the y-axis adjustment of target holder, labeled 9 in figure 2 on page 10, until the target is seated in the target holder. Make sure the target is seated, then remove the fork by sliding it out along the z axis.
7. Rotate the target to a covered position with the rotational adjustment for target holder, labeled 10 in figure 2 on page 10. Lower the target holder as far as you can.
8. Lower the sample holder to the position of 3.5 on the y-axis adjustment scale, labeled 11 in figure 2 on page 10. Move the substrate holder so that it will fit inside of the sample ring. Raise the substrate holder until it is just below the sample holder.
9. Rotate the sample holder so we can see two hooks through the viewing ports, labeled 13 in figure 2 on page 10. Rotate the substrate holder, labeled 1 in figure 2 on page 10, until two of the notches line up with the two hooks on the sample holder.

10. Raise the substrate holder with the y adjustment, labeled 2 in figure 2 on page 10, until the hooks go into the notches. This may take some wiggling and adjustment to get the hooks into the notches.
11. When you get the hooks in the notches rotate the substrate with the rotational adjustment, labeled 1 in figure 2 on page 10, holder to make sure all three hooks are in the three notches.
12. With quick movements rotate the substrate holder with the rotational adjustment, labeled 1 in figure 2 on page 10, until the substrate is connected to the sample holder.
13. Lower the substrate loader with the y adjustment, labeled 2 in figure 2 on page 10, making sure it is not still connected to the substrate holder.
14. Pull the substrate loader into the load lock with the z adjustment, labeled 1 in figure 2 on page 10, and close the PLD/Load lock valve with, labeled 8 in figure 2 on page 10.
15. Raise the sample holder to about 3 on the y axis scale, labeled 11 in figure 2 on page 10, and close the substrate shutter by twisting the shutter control located on the end of the PLD pump, labeled 17 in figure 2 on page 10.

4 PLD Prep

4.1 Heating Up the Substrate

1. Start the heating process by turning on the cooling water pump, located behind the PLD chamber. Check the flow meters to make sure the water is circulating.
2. On the Heat lamp control, labeled 6 in figure 4 on page 12, go to SP menu and set the following variables:
 - (a) SP 1 - 20
 - (b) SP 2 - desired temperature, note the scale on the control panel is not one to one with temperature scale.
 - (c) SPrr - 30
3. Now we need to set the heater lamp to reset, turn on the lamp, and set the lamp to a minimum value of about two on the dial. do not turn on the lamp if the OP1 light is on. It will take about 3 to 5 minutes for it to go away.
4. Allow the lamp to warm up to until it reaches 75 on the heat lamp control, labeled 6 in figure 4 on page 12, then change the heater lamp to run and allow the substrate to warm up to the desired temperature.

4.2 Transmission Measurements

Put on your safety goggles!

1. Turn on the excimer laser by switching both switches at the back. The laser will require about 10 minutes to warm up. Remove any attenuators from the laser.
2. When the laser is warm, set the following variables on the laser control panel:

- (a) Select Mode to Hv
 - (b) Set Hv to 25 kV
 - (c) select Trig to Int
3. Turn on the power meter by pressing the power button for a few seconds. Remove the cap that is covering the power meter.
 4. Position the power meter's detector in front of the transmission entrance port, labeled 21 in figure 2 on page 10. Turn on the Laser in use sign by the light switches and open the shutter on the transmission exit port, labeled 16 in figure 2 on page 10.
 5. Open the safety cover of the laser, hit the run button on the laser control panel, and then hit the Execute button.
 6. Record the value of the power in J/cm^2 and hit the stop button on the laser control panel to turn off the laser.
 7. Position the power meter's detector in front of the transmission exit port, labeled 16 in figure 2 on page 10. Hit the run button on the laser control panel, hit the Execute button, and record the value of the power. To turn off the laser by hitting the stop button on the laser control panel.
 8. Put the power values into the excel worksheet and this will tell you what energy to set the laser to to achieve the power you want on the target.
 9. Set the following variables on the laser control panel:
 - (a) Select Mode to Egy NGR
 - (b) Set Egy NGR to the desired energy from the excel worksheet
 - (c) select Trig to Ext

4.3 Pre PLD Target Ablation

1. Rotate the target to a non covered position with the rotational adjustment for target holder, labeled 10 in figure 2 on page 10.
2. Turn on the rotation of the target with the rotation control, pushing the button away from you, on top of the PLD chamber, labeled 12 in figure 2 on page 10.
3. Close all shutters on the viewing ports and on the transmission exit port, labeled 16 and 22 in figure 2 on page 10. Be very careful on the large viewing port's shutter. If you close it too hard or too far it will become wedged in the viewing port and you will need to take the PLD chamber apart to free it.
4. Raise the target holder to the position of 0.5 on the y axis scale, labeled 9 in figure 2 on page 10. There are two scales on the y axis. The large one with the needle and a finer scale on the dial. Make sure the large scale is set to 0.5 and the finer scale is set to 0.
5. Set the computer which controls the laser to emit 500 pulse at a rate of 1 Hz. Then hit run on the program.

6. Look through the view ports to see where the laser is hitting on the target. Adjust the position of the laser with the adjusting lens at the top of the optics tower. Be careful to move only the x axis knob and not the z axis knob. If you do move the z axis knob make sure to return it to the original location.
7. When we have the proper position on the laser we will hit stop on the laser program.
8. Start the pre ablation by setting the computer which controls the laser to emit 1000 pulse at the desired rate for deposition. Then hit run on the program. This should take about 5 to 10 minutes.

5 PLD

1. Open the sample shutter by twisting the shutter control located on the end of the PLD pump, labeled 17 in figure 2 on page 10.
2. Turn on the sample rotation switch, labeled 12 in figure 2 on page 10. Make sure to follow the directions on the switch in terms of witch way to push the switch.
3. Move the substrate to the desired height and x axis position with respect to the target.
4. Set the computer which controls the laser to emit the desired number of pulses at the desired rate for deposition. Then hit run on the program. This will start the PLD process.

6 Sample and Target Removal

6.1 Cooling down the Substrate

1. Turn off the sample and target rotation switches, labeled 12 and 13 in figure 2 on page 10
2. Close the sample shutter by twisting the shutter control located on the end of the PLD pump, labeled 17 in figure 2 on page 10.
3. Rotate the target to a covered position with the rotational adjustment for target holder, labeled 10 in figure 2 on page 10.
4. On the Heat lamp control, labeled 6 in figure 4 on page 12, go to SP menu and set the following variables:
 - (a) SP 1 - Current temperature
 - (b) SP 2 - 25
 - (c) SPrr - 10
5. Now we need to set the heater lamp to reset and then change the heater lamp to run. do not turn on the lamp if the OP1 light is on. It will take about 3 to 5 minutes for it to go away.
6. Allow the lamp to cool before proceeding.

6.2 Removing the Sample and Target

1. Open the PLD/Load lock valve with, labeled 8 in figure 2 on page 10, and pull the substrate loader into the load lock with the z adjustment, labeled 1 in figure 2 on page 10.
2. Lower the sample holder to the position of 3.5 on the y-axis adjustment scale, labeled 11 in figure 2 on page 10. Move the substrate holder so that it will fit inside of the sample ring. Raise the substrate holder until it is just below the sample holder.
3. Rotate the sample holder so we can see two hooks through the viewing ports, labeled 13 in figure 2 on page 10. Rotate the substrate holder, labeled 1 in figure 2 on page 10, until two of the notches line up with the two hooks on the substrate loader.
4. Raise the substrate loader with the y adjustment, labeled 2 in figure 2 on page 10, until the hooks go into the notches. This may take some wiggling and adjustment to get the hooks into the notches.
5. When you get the hooks in the notches rotate the substrate with the rotational adjustment, labeled 1 in figure 2 on page 10, holder to make sure all three hooks are in the three notches.
6. With quick movements rotate the substrate holder with the rotational adjustment, labeled 1 in figure 2 on page 10, until the substrate is connected to the substrate loader.
7. Rotate the substrate holder to make sure the substrate holder is lock in the substrate loader. Lower the substrate loader with the y adjustment, labeled 2 in figure 2 on page 10, making sure it is in connected to the substrate holder.
8. Rotate the target to a non covered position with the rotational adjustment for target holder, labeled 10 in figure 2 on page 10.
9. Slide the substrate loader along the z axis, labeled 1 in figure 2 on page 10, so that the target fork goes in to the target holder so the prongs are on either side of the target.
10. Lower the target holder from the y-axis adjustment of target holder, labeled 9 in figure 2 on page 10, until the target is seated in the target fork. Make sure the target is seated, then lower the target holder all the way so the target is free from the target holder.
11. Using the y-axis loading arm adjustment, labeled 2 in figure 2 on page 10, angle the loading arm at about 10 degree angle as shown in figure 1.
12. Remove the substrate loader out of the PLD chamber being careful to not to bump the side or bottom of the chamber's walls. If you do bump the side or bottom it is very easy for the target to fall off of the fork. If the target falls off of the fork you will need to take the PLD chamber apart to retrieve it.
13. Close the PLD/Load lock valve, labeled 8 in figure 2 on page 10.
14. Turn off load lock low pressure pump on control panel, labeled 9 in figure 4 on page 12. Make sure to turn off the load lock low pressure gauge on control panel, labeled 1 in figure 4 on page 12. Take off the load lock lock, labeled 4 in figure 2 on page 10.

15. Close the load lock rough pump valve on control panel, labeled 8 in figure 4 on page 12. Open the nitrogen gas valve to bring up the pressure in the load lock, labeled 20 in figure 2 on page 10. You should hear a hissing sound when you open the valve and the load lock door should open on its own. Close the nitrogen valve when load lock's door opens.
16. Remove the target and substrate holder from the load lock.
17. Close the load lock's door and lock the load lock, labeled 4 and 5 in figure 2 on page 10.
18. Open the load lock rough pump valve, labeled 8 in figure 4 on page 12, and wait 5 minutes of pumping before turning on the low pressure gauge.

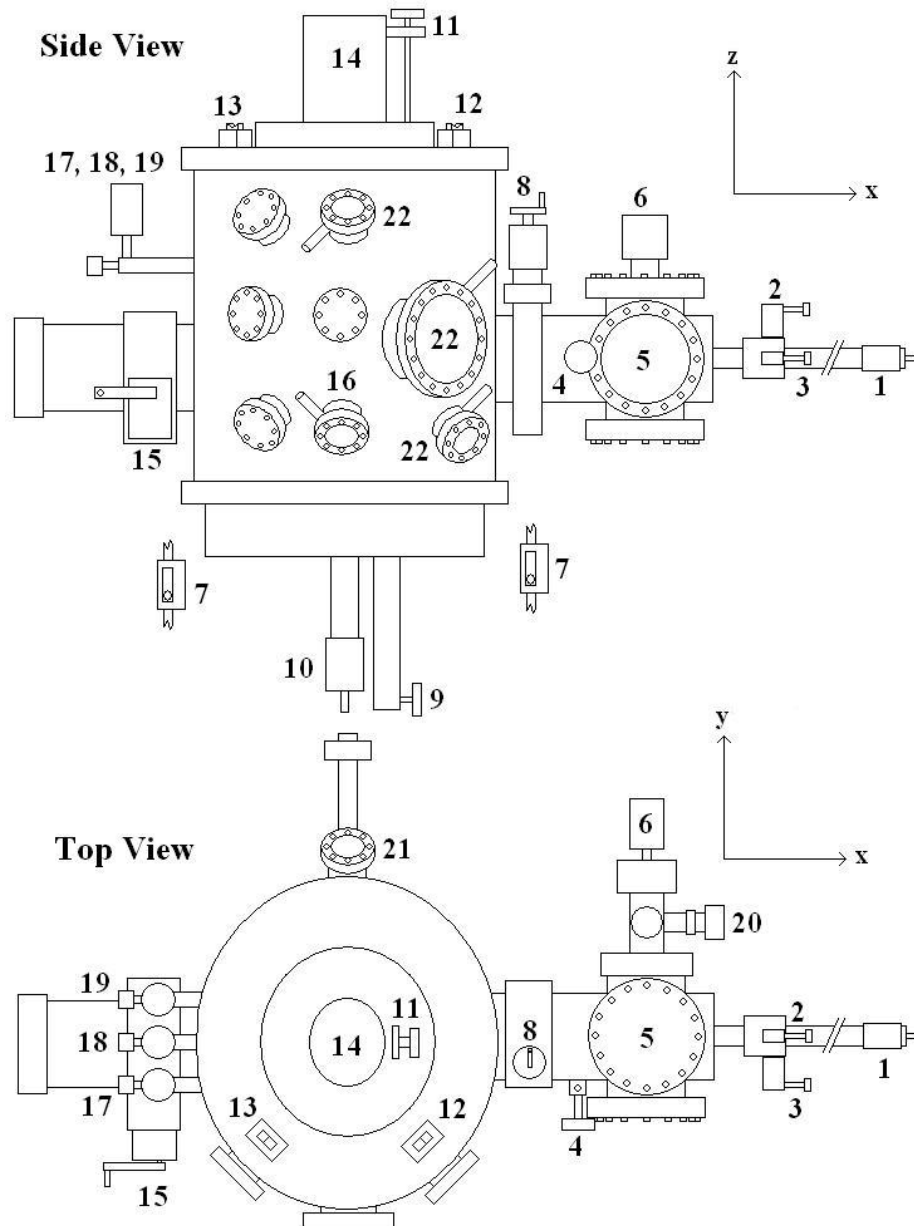


Figure 2: 1. Rotational and z-axis adjustment of substrate loader 2. y-axis adjustment of substrate loader 3. x-axis adjustment of substrate loader 4. Load lock lock 5. Load lock 6. Load lock low pressure pump 7. Flow meters 8. PLD/Load lock valve 9. y-axis adjustment of target holder 10. Rotational adjustment of target holder 11. y-axis adjustment of sample holder 12. Target rotation on/off switch 13. Sample rotation on/off switch 14. Heating Lamp 15. PLD pump valve 16. Transmission exit port 17. High pressure Bartron pump 18. MKS gauge 19. Pump gauges 20. Load lock nitrogen valve 21. Transmission entrance port 22. Viewing ports

Thermionics PLD Diagram

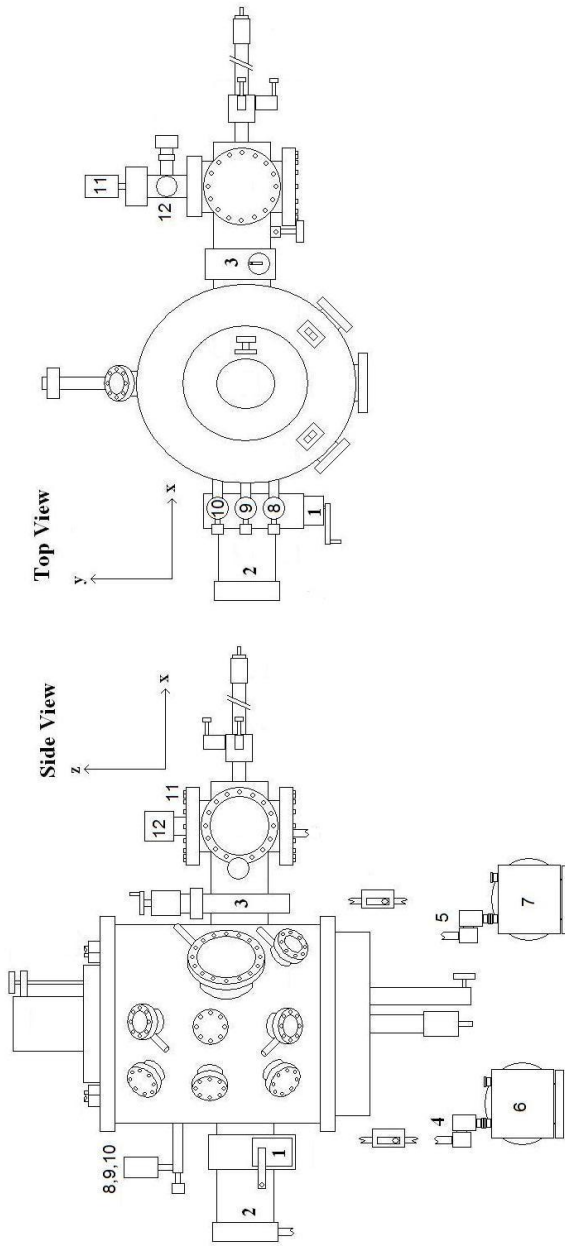


Figure 3: 1. Main Chamber Gate Valve 2. Main Chamber Turbo Pump 3. Load Lock Gate Valve 4. Main Chamber Isolation Valve 5. Load Lock Isolation Valve 6. Main Chamber Rough Pump 7. Load Lock Rough Pump 8. 628B Capacitance Monometer Pressure Gauge and Valve 9. 627B Capacitance Monometer Pressure Gauge and Valve 10. Ion Pressure Gauge and Valve 11. Load Lock Turbo Pump 12. 943 Cold Cathode Pressure Gauge

Thermionics PLD Control Tower

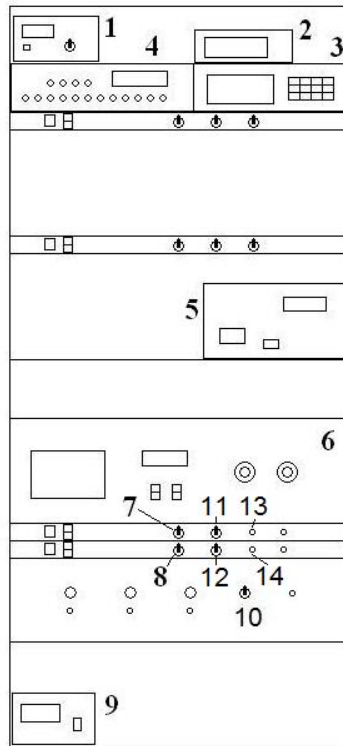


Figure 4: 1. Load lock cold cathode pressure gauge reader 2. 627B capacitance monometer pressure gauge reader 3. 628B capacitance monometer pressure gauge and ion pressure gauge reader 4. 5. Main Chamber turbo pump 6. Heat lamp control 7. Main Chamber isolation valve control 8. Load Lock isolation valve control 9. Load lock turbo pump 10. Main Power Reset Switch