

Operating Procedure for the UNC Pulsed Laser Deposition (PLD) System

1. You must take the UNC online laser safety training. The gases should be on (in the chase) so only the valves at the tool base need to be opened. The only gases to turn on are Nitrogen (always used) , Oxygen (only if needed for your films) , Argon (for sputtering)
2. Note: Before turning tanks on, back out the regulator diaphragm (ccw; not doing so can damage the regulator), open the tank, set regulator, open the valve. Because there is no flow, you cannot set the pressure lower than the previous user since gas is trapped in the regulator. If the chamber does not vent, check the regulator pressure, once flow has begun the regulator pressure may be lower than you think. Set pressure to 20-40psi.
3. Open the PLD software and open tab **MAIN**.
4. Note that dark green on a tab (“pushbutton”) means closed/inactive status; light green means open/active status on the screen.
5. Select **pump sequence** → **chamber vent** in the Pump Control screen. Note, if the chamber does not vent, go check the nitrogen regulator and make sure the previous pressure reading of 20-40 psi was not an erroneous one due to trapped gas in the regulator diaphragm. Most likely, chamber inability to vent is due to that and the regulator diaphragm will have to be set by turning clockwise and/or the bleed valve needs to be opened.
6. Turn on the laser: first, turn the large red **MAINS** knob to on position, a red light will come on; second, turn the key switch to on position, the white light will turn on.
7. Once the **chamber vent** process is complete and the chamber is at atmosphere, open the chamber door. Carefully turn the screw to release the door clasp mechanism.
8. Make sure that all substrate and target rotations are off (dark green). Load the appropriate target material. The target material will appear in the slot below the substrate holder. If the desired target is in the chamber but not in the correct place then change the targets by selecting **F5** → **load target #__**; this puts the target in the correct location for deposition. If the desired target is not in the chamber then load it into one of the target slots. Do this by first unscrewing one screw on the target shield (the flat rectangular piece of metal with rounded corners inside the deposition chamber above the targets). Then select **F6** → **load target #__**. Remove the undesired target (lift out with tweezers), replace with the desired target, and re-attach target shield. This is very important so there is no cross-contamination of targets.
9. Note: Select **F5** is used to move targets into place that are already loaded.
10. Note: The **F5** and **F6** selections must both be on the same target number so that the correct target is below the sample.
11. The software will ask if target change is complete; check to make sure this is true, the select **okay**.
12. Load sample(s) onto the metal substrate holder and secure with the fastening screw. Check for secure fastening (turn upside down). Load sample into the PLD chamber with the sample surface facing down. The groove of the holder will be toward the back of the chamber and a hole in the substrate holder will be at the front when the holder is lined up. Make sure that the stage is locked into place via the groove fitting into the stage holder.
13. Move stage into position: select the **MOTION CONTROL** tab and select ‘**move up**’ twice (Z stage movement). Wait for the adjustment—look for the bellows of the stage to move.
14. Before closing the chamber: **Confirm that the sample has moved above the shutter level.**
15. Close the door to the chamber. Do not over-tighten; the door just needs to be slightly tightened. Go back to the **MAIN** tab; select **F7** → **chamber pump** in the Pump Control area.
16. Turn on sample **rotate** and target **rotate** and set the desired rotation rates. Typical values are target: 40 rpm, substrate: 20 rpm.
17. Close the substrate shutter on the computer screen (dark green means it is closed).
18. Remember that there are 3 shutters: the substrate shutter, the laser shutter, and the magnetron shutter. The magnetron shutter is only used in sputtering mode.
19. Make sure that the ion gauge reads below 10^{-4} Torr. The ion gauge turns on automatically. When base pressure on the ion gauge is $<5e-7$, the alignment can be checked. Note, the ion gauge pressure can be read directly off the gauge or on the MAIN screen.
20. Remember: for the laser setup, **internal** means the user starts and stops the laser; **external** tells the laser to emit the specified number of pulses and then stop.
21. Perform laser alignment test: select **laser internal trigger mode**, this allows you to turn the laser on and off manually); select okay;
22. The alignment check laser conditions are 200mJ, 40 Hz. This step checks the laser on the Iw photodetector, with readout on the tabletop instrument LCD screen.

23. Turn on or open:
 - a. stage rotation
 - b. target rotation
 - c. laser on (on the computer screen)
 - d. Iw (record in the logbook)
 - e. laser shutter (in pump control)
24. If the Iw is not approximately 0.66W, the laser needs to be re-aligned or there is not enough gas in the laser. Contact the lab manager if either of these occurs. Do not proceed any further. Note: No raster on this step.
25. Turn off Iw.
26. Now check for the plume: (substrate shutter should be closed-dark green)
Turn on (or leave on) the following:
 - a. raster
 - b. laser (on the computer screen),
 - c. laser shutter,
 - d. laser should still be on internal trigger
 - e. check for plume; turn off laser.
27. To fill in gases: select tab **PUMPS**, turn on '*throttle valve*'. In tab **MAIN** go to Mass Flow Controllers and select desired gas(es); typical flow rates are 3 sccm (O₂) and 8 sccm (Ar) select the desired mTorr in VAT control (typical value is 50 MT); select the desired setpoint and wait for tool to reach the specified pressure (this value is read from the display below the optical housing on the PLD system).
28. Set Deposition Condition:
 - a. For a typical deposition, use the **External** setting so that the pulse number is controlled as entered.
 - b. Set **External** laser setting and desired number of pulses, pulse energy, pulse frequency. Record these values in the log book.
29. Confirm the following:
 - a. Deposition parameters in laser section of tab **MAIN**.
 - b. substrate rotate on
 - c. target rotate on
 - d. laser on
 - e. **raster on**
 - f. open substrate shutter
 - g. open laser shutter
 - h. trigger on to begin the deposition
Note: For external style deposition, select laser then select trigger; for internal style deposition select laser you will start the laser manually.
30. Monitor deposition (plume confirmation).
31. Once the deposition is finished:
 - a. turn off the laser (**on the computer screen**);
 - b. stop substrate and target rotations;
 - c. stop rastering
 - d. close the laser shutter;
 - e. **make sure the substrate shutter is open since the sample holder will be lowered.**
32. If gases were used for the deposition: VAT control → close; select off gas. In PUMPS tab stop throttle valve; close laser shutter
33. In tab MAIN select pump control → chamber vent. Open chamber; tab MOTION CONTROL select F2 down twice. Remove substrate.
34. Close chamber. Select **pump control** → **chamber pump**. Make sure laser shutter is off. Turn off laser (first turn off key switch, then turn off laser mains).
35. Turn off the gases at the tool.
36. Record all necessary information in the PLD log book and on the log sheet. Log into the CHANL website and record equipment usage and parameters. Tidy up the work space. Make sure the chamber has pumped down sufficiently.
37. End.

NOTE: The KrF gas requires refilling when the laser status box has a voltage of 26kV. When this occurs, notify the lab manager to refill the gas. Do not change any parameters not discussed in this document.