GENERAL PROCESS AND OPERATION SPECIFICATION

Oxford System Plasmalab 100 ICP-RIE

I. SCOPE

a. The purpose of this document is to describe requirements and basic operating instructions for the Oxford Instruments Plasmalab 100 ICP-RIE. This tool is intended for general purpose use to perform Reactive Ion Etching on a variety of substrates and films.

II. SAFETY

a. You must be trained and signed off to use this equipment.
b. Keep all doors and protective shields in place before operating this equipment.
c. This tool can produce high voltage, RF energy, high temperatures, and low temperatures. Use caution when interacting with the various components of this tool.
d. This equipment uses liquid nitrogen (LN2) to cool the substrate holder in the chamber. Use caution and wear the cryo gloves when moving the LN2 valve.
e. Minimum operating temperature of substrate table/holder: DO NOT operate the substrate chiller below -100°C. This places the O-rings and seals at risk and reduces the life of the equipment.
f. If you are unsure about any procedure or indication while operating this equipment, contact a staff member or trainer for assistance.

III. APPLICABLE DOCUMENTS, MATERIALS, AND REQUIREMENTS

a. For more information about the hardware, software operating system, and general system specifications see the factory manual (sk AggieFab staff for a copy).
b. Process gases are available to the system: O2, Ar, SF6, CHF3. He is used for backside cooling.
c. Appendix A: Recipe Parameter Information
d. Appendix B: Common Substrate Chemistry Recipes
e. Appendix C: Cryogenic Deep RIE

IV. OPERATION

1. Log in:
   a. Open the liquid nitrogen (LN_2) valve using the cryogenic gloves provided.
      i. This valve is on to the large dewar to the left of the tool.
   b. Log in by selecting “System,” then “Password.”
      i. Optionally, you can click where it says “View_Only” in the top center of the screen.
2. Open the chamber:
   a. Select “System,” then “Pumping” to access the “PUMP CONTROL” page.
   b. Press “STOP” in the loadlock menu on the lower left side.
      i. A message saying, “Wafer process complete, ready to vent” may pop up, if so, select “OK.”
   c. Press “VENT” in the loadlock menu on the lower left side.
      i. Wait until “Vent Time Left” is 0 sec.
   d. Open the loadlock door and remove the carrier wafer.

3. Ready sample and close the chamber:
    a. If your sample is not a standard 4-inch wafer, use Kapton tape to mount it to the carrier wafer.
        i. Ensure the sample is away from the outside edge of the carrier wafer. The clamp may hit your sample if it is mounted too close to the edge.
    b. Place the wafer into the loadlock.
        i. Ensure the wafer is sitting flat and that the round edge is touching the two circular knobs.
        ii. If the carrier wafer has a major flat, face it to the right (towards the process chamber).
    c. Close the loadlock door.
    d. Press “STOP” on the pump menu.
4. Load, edit, and run your recipe:
    a. Select “PROCESS” in the top left of the screen.
    b. Select “Recipes”
    c. Select “Load”
    d. Select “YES” to overwrite the current recipe. This does NOT delete the recipe; it loads a new one.
        i. The prompts for overwriting a recipe and loading a new one are similar and can be easily confused. Look at which action is highlighted (load or save) before proceeding.
    e. Select desired recipe from the list.
    f. To edit a recipe, right-click a recipe step and select “Edit Recipe”.
        i. Time is in “hh:mm:ss”.
        ii. See “Appendix A - Recipe Parameter Information” for recipe constraints.
    g. Select “RUN” when the recipe is ready.
        i. **ALWAYS ENTER A WAFER NAME**. Not naming a wafer can cause software errors.
        ii. The system will automatically evacuate the loadlock, move the sample into the process chamber, and begin the recipe.
5. While running the recipe:
    a. Notice that at the top left of the “Process” => “Chamber 1” screen, there is a box that shows the current running recipe name, recipe step, and step time left in hh:mm:ss.
    b. Fill out the logbook as your recipe is running.
6. Unload your sample:
    a. Before venting, visually confirm the sample is in the loadlock, and that the tool is finished moving.
    b. Follow the instructions above to vent the loadlock and unload the sample.
    c. Place the carrier wafer back into the loadlock.
7. Run a chamber clean:
    a. Load and run the “OPT-CHAMBER O2 CLEAN” recipe.
    b. Ensure the LN2 valve is open to allow cooling of the chamber substrate before beginning this recipe.
        i. You may log out of the tool as the clean is running.
    c. Close the LN2 when the recipe is finished.
8. Log out:
    a. Click “System” then Password”.
    b. Click “Verify” then “OK”.

V. SIGNATURES AND REVISION HISTORY

a. Author of this document: Sandra Malhotra
b. Author Title or Role: Technical Manager
c. Date: 26 June 2023
d. Revision: D
e. Revision notes: Removed approved materials TiW and GaN from Appendix B

Approvals:

Technical Manager Signature: ___Sandra G Malhotra___________________________

Date: ____6/26/2023_____________________________________

Revision History:

Revision	Author	Date
Original Issue	L. Rehn	03 April 2014
Rev A	E. Morse	21 October 2019
Rev B	E. Morse	15 January 2020
Rev C	Elijah Colter	10 January 2022
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Rev E		

Appendix A: Recipe Parameter Information

- Step time [hh:mm:ss]: Amount of time the step should run.
- Ignore tolerance: Certain parameters have certain tolerances. For example, if the table temperature setpoint is 20 °C and the actual value is 30 °C, then it is out of tolerance. By checking this box, the recipe will continue to run if the tolerances are not satisfied.
- Hold: When "Hold" is on, the plasma is continuous between steps. If NOT selected, the plasma will turn off between steps while the APC stabilizes.
- Automatic Pressure Controller (APC): The APC controls the chamber pressure automatically based on the gas flow rates and pressure setpoint.
    - Chamber Pressure [mTorr]: Pressure in the process chamber.
    - Strike Pressure [mTorr]: Value at which the RF should turn on and strike the plasma. If a zero is entered, the feature is disabled, and the RF will turn on once the pressure has stabilized at the requested process pressure
    - DC Bias Minimum [V]: Enter a positive number for the minimum DC bias value expected once the plasma has struck. Enter zero if DC bias cannot be read because the substrate (and any wafer clamp) completely covers the electrode, or if the electrode has an insulating coating. A non-zero value is used by the software to detect if the plasma has been properly established. If a zero is entered, then the software assumes the plasma has struck once the RF reflected power goes low.
    - Ramp Rate [s]: Sets the rate at which the pressure is reduced from the strike value to the set point. The higher the number entered, the faster the transition to process conditions will be. Too high a value can cause the plasma to go out.
- RF Generator:
    - Forward Power [W]: Value of the RF power applied to the plasma.
        - **MAXIMUM = 200 W**
        - Increasing RF power increases etch rate, increases anisotropy/directionality, and decreases selectivity
        - DC Bias [V] and Reflected Power [W] are functions of the forward power and other parameters. If DC Bias is larger than 500 V, an error will occur, and the tool will abort the process.
- ICP (Inductively Coupled Plasma):
    - Forward Power [W]: Value of ICP power applied to the plasma.
        - **MAXIMUM = 2000 W**
        - Increasing RF power increases etch rate, decreases anisotropy/directionality, and increases selectivity.
- Helium Backing: Used to cool wafers.
    - Pressure controller [Torr]: Pressure of helium on the wafer's backside.
    - Sccm [SCCM]: Helium flow rate.
- Cryo [°C]: Temperature of the process chamber.
    - **MAXIMUM RANGE = -100 °C to 30 °C**
- Gases [SCCM]: Flow rates of the provided gases (SF_6, O_2, Ar, CHF_3). Larger etch gas flow rates (SF_6, CHF_3) increase etch rate.
    - **MAXIMUM = 100 SCCM**

Appendix B: Common Substrate Chemistry Recipes

Table 1. Etchant-etch gas combinations

Material Being Etched	Etch Gas
Si	SF_6
SiO_2	CHF_3, SF_6
Si_3N_4, SiN_x	CHF_3, SF_6
SiC	SF_6
Poly-Si	SF_6
Graphene (C)	O_2
W	SF_6
Photoresist	O_2 (Ashing)

Appendix C: Cryogenic Deep Reactive-Ion Etching

Managing Heat

If the sample is smaller than a 4" wafer, some type of thermal grease must be used between the sample and carrier wafer to promote thermal conduction. If the sample gets too hot, anisotropy will decrease significantly. So, there are three ways to promote sample cooling:

- Thermal conductor: COOL-GREASE ZXM is a thermal grease used for heatsinks. It is ZnO-filled and has a thermal conductivity of approximately 20 W/m-°C. This goes between the sample and the carrier wafer.
- Backside helium cooling: Helium is used for cooling the wafer. Set the pressure to 10 mTorr and flow rate to 38 SCCM.
- Chamber temperature: -100 °C is the lowest temperature the tool can safely handle.

To apply the grease, gently push some out of the syringe and dab it on the sample's backside. Once there is a large-enough dab, place the sample onto the carrier wafer. Press across the sample to spread the grease, then move the sample a bit in all four directions (up, down, left, right) to further spread the grease. Do not go so far that grease escapes from underneath the sample. Applying a vacuum will also spread the grease. When complete apply Kapton tape as normal and run the DRIE recipe.

To remove the sample, remove the Kapton tape and slide the sample to the edge of the wafer or push around in circles to remove the grease's adhesion. To clean up the residual grease, a cotton swap covered with a cleanroom wipe works well. Ensure all the grease is removed from the carrier wafer's surface.

Recipe Guidelines

It has been determined that cryogenic recipes work best when two things occur:

- The process chamber is cleaned beforehand with an O2-based plasma.
- The etching gas (SF6, CHF3) is gradually introduced into the plasma.

Begin the process with a 15:00+ min O2 clean, then decrease the chamber temperature to your desired value.

To gradually introduce the etching gas, first strike a plasma using the supplementary gas (O2, Ar) if you are using one at a pressure of X mTorr, Y W ICP power, and Z W RF power. Hold this plasma for 10-20 s, then add 1 SCCM of the etching gas and decrease both the supplementary gas flow and RF power slightly. Hold for 5 s, then repeat: increase etching gas flow and decrease supplementary gas flow and RF power slightly. See the example recipe below (Table 2), where ICP power, temperature, and helium backside cooling remain constant throughout (holding ICP power constant is optional, but cooling parameters should remain constant). The O2 environment is stabilized in step 1, an O2 plasma is formed in step 2, then SF6 is slowly introduced in steps 3-8, with RF power, pressure, and O2 flow rate also decreasing. The "Ignore tolerance" (second bullet point of Appendix A) and "Hold" options (last bullet point of Appendix A) must be selected for this to work properly.

Table 2. Example Recipe Steps

Step	Pressure (mTorr)	RF Power (W)	SF_6 Flow (SCCM)	O_2 Flow Rate (SCCM)
1	30	0	0	60
2	30	100	0	60
3	27	85	1	55
4	24	70	5	50
5	21	55	10	45
6	18	40	20	40
7	15	30	40	35
8	15	20	90	30

Table 3. Optimal DRIE recipe parameters and values

Parameter	Value
Pressure	15 mTorr
Temperature	-100 °C
Helium Backing (Pressure, Flow)	10 Torr, 38 SCCM
SF_6 Flow	90 SCCM
O_2 Flow	20 SCCM
ICP Power	1000 W
RF Power	10 W

Table 4. Optimal DRIE recipe resulting values

Parameter	Value
Average Vertical Etch Rate	3.06 µm/min
Average Horizontal Etch Rate	0.085 µm/min
Selectivity (Cr, SiO_2, AZ 5214 E-IR)	1700, 100, 20