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P1 Glove Box- Standard Operating Procedure

Badger Name: P1 Exfoliation Glove Box
Model: Labconco Protector Controlled Atmosphere Glove Box
Location: PAN-Bay 1

Revision: 2
Revisionist: Tony Whipple
Date: 3/27/2020

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1. Scope

1.1 This document provides detailed instructions on how to properly operate the PAN Cleanroom Glovebox.

2. Tool description

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2.1 This is a very low oxygen glove box to processing of samples that are semiconductor related such as 2D materials. The Labconco Protector Controlled Atmosphere Glove Box allows materials to be manipulated while isolated from oxygen and moisture. Starting with ambient air, the glove box uses a vacuum pump to purge its volume, and then fills the glove box with dry nitrogen gas form a cylinder. This process is repeated automatically for a programmed number of cycles. A level of <100 ppm of O2 can be reached after about 75 purge-fill cycles.

3. Reference Documents

3.1 See the section at the end of this Sop for more information on process hints & tips.

3.2 Safety Data Sheets (SDS) are available at www.mnc.umn.edu

4. Safety

4.1 Some materials that could be placed in the chamber could have reactions if exposed to room atmosphere. Be aware of any possible dangers.

4.2 The spinner and hot plate and heated chucks could be a source of dangers if not used correctly.

4.3 The use of a glovebox has lower and higher pressures, avoid using system if it not working correctly.

5. Equipment and Substrate restrictions

5.1 Tools and equipment

5.1.1 Wafer or glass substrates or transfer material

5.1.2 Air tight containers

5.1.3 Razor blades, scoring pad, and other cleaning tools.

5.1.4 PDMS

5.1.5 Other items

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Figure 1 *Glove box with equipment inside*

5.2 Substrate restrictions

5.2.1 The system can have any material as long as it will fit in the chamber and not cause danger to the user or others around it.

5.2.2 No outgassing material allowed in the chamber that could produce extra oxygen or other material that could damage the low oxygen chamber.

6. Procedure Loading the Glove Box.

6.1 loading chamber

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6.1.1 Check **Badger** for any reservations before starting. *Making a reservation is recommended* because of unpredictable run times. Clean substrate thoroughly if needed.

To preserve the atmosphere inside the glove box, the transfer chamber is used to load items from the outside environment.

a. Check to ensure that the inner transfer chamber door is closed and sealed. If not, close it and lock it with the locking handle.

b. Check the position of the four manual valves on the front of the transfer chamber. Move all valves to the “open” position if not already open.

c. Toggle the front panel display to bring up the transfer chamber pressure screen. The chamber should be at or near atmospheric pressure, given by a value of 0 +/- 3 mbar. If the chamber is under-pressured, use the “fill” key on the transfer chamber purge/fill menu to bring the pressure to ambient (i.e., a value of “0”).

d. Open the outer transfer chamber door and load your tools/materials, then close and lock the outer transfer chamber door.

e. Use the front selector panel to bring up the transfer chamber purge/fill menu. Input a number of purge fill cycles to replace the air in the chamber with the dry N2 atmosphere; 2-3 purge/fill cycles should be sufficient. Press “Start” to begin purging.

f. After these cycles are complete, the inner transfer chamber door may be opened by reaching into the glove box. Retrieve and position your equipment and materials and close the inner transfer door.

g. Close and lock the inner transfer door.

h. Check the oxygen and water meters. The act of loading may have raised both oxygen and water vapor levels. Depending on your requirements, a pump down sequence for the main chamber may be necessary to reduce these gases.

6.1.2 Main Chamber purge/fill (pump down) sequence. To decrease the oxygen or water vapor levels in the box, do the following steps.

a. Check the position of the four manual valves on the front of the transfer chamber. These valves are shown in Figure 3. Move all valves to the “open” position if not already open.

b. Toggle the front panel display to bring up the glove box purge/fill screen. During a pump down, the glove box will automatically pump the box down to a pressure of 12.5 millibar

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(mbar) below ambient, then fill the chamber with dry nitrogen up to a pressure of 12.5 mb above ambient. This process is repeated for as many cycles as the user selects in this screen (1 to 199 cycles

General Atmospheric Conditions

O2 209,460 20.946% https://en.wikipedia.org/wiki/Atmosphere_of_Earth From *(2016–2017), CRC Handbook of Chemistry and Physics (97th ed.), CRC Press*

Gas	Ppm in atmosphere	Ppm in glovebox: Typical (possible)
O2	209,500* (20.95%)	1,000 [O2 monitor (blue) has units of % and has lower limit of 0.1%, or 210 ppm; actual should be lower] (1**)
H2O	25,000* Varies wildly list no value	60 (1**)

**When AtmosPure gas purifier is on.

6.2 Computer

Password : PANcleanroom

This is a touchscreen. A keyboard can also be used and is stored on top of the transfer chamber. Make sure to turn off the Bluetooth keyboard when done.

6.3 Microscope

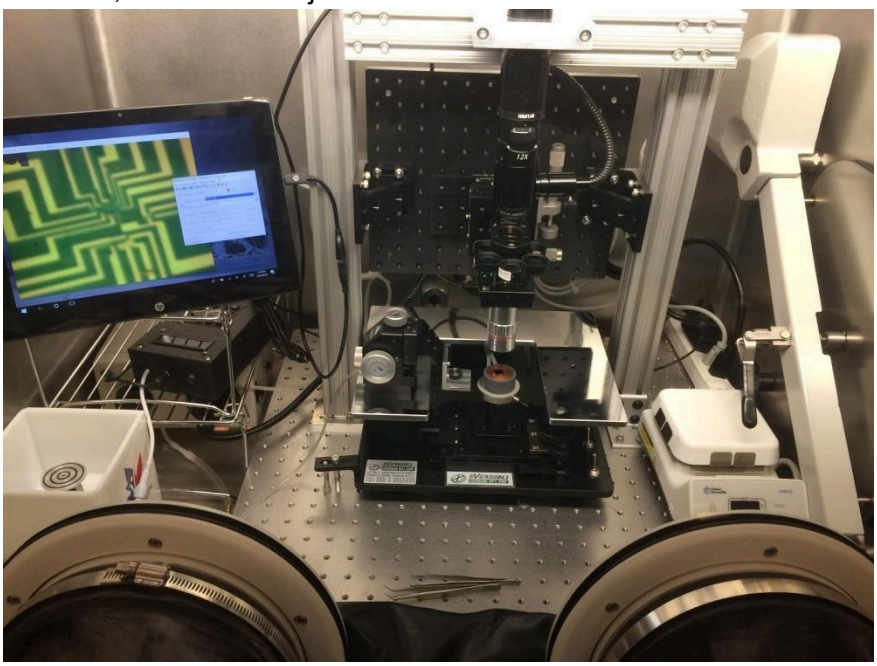
The microscope provides magnification power of 5x to 60x (double check this) adjustable via a rotating tuning knob. A number of optical filters are provided including a linear polarizer and color filters. Microscoped images can be viewed and saved using the attached ccd camera.

Using the microscope:

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1. Open the “Infinity Capture” program via its desktop icon. A live shot of the sample should show on the screen.
2. Turn on microscope illumination by powering on the light source in the back left corner of the glovebox under the shelf and adjusting the tuning knob to the desired brightness. (Note: Do not power on light source and hot plate at the same time. This could blow a fuse.)
3. Adjust focus using knob above the sample stage.
4. X-y adjustments can be made using the control knobs below the stage.
5. To capture an image, click “File” then “Capture Image.” Save the image (via “File” → “save image as”) in the user’s folder. Current folders are under “Documents.”
6. Close out of the frozen image, then reopen “Infinity Capture.” If you find a slicker way of doing the above, feel free to adjust the directions.



6.4 Transfer Stage

The transfer stage section explains how to use the sample vacuum chuck and the micromanipulator to align materials.

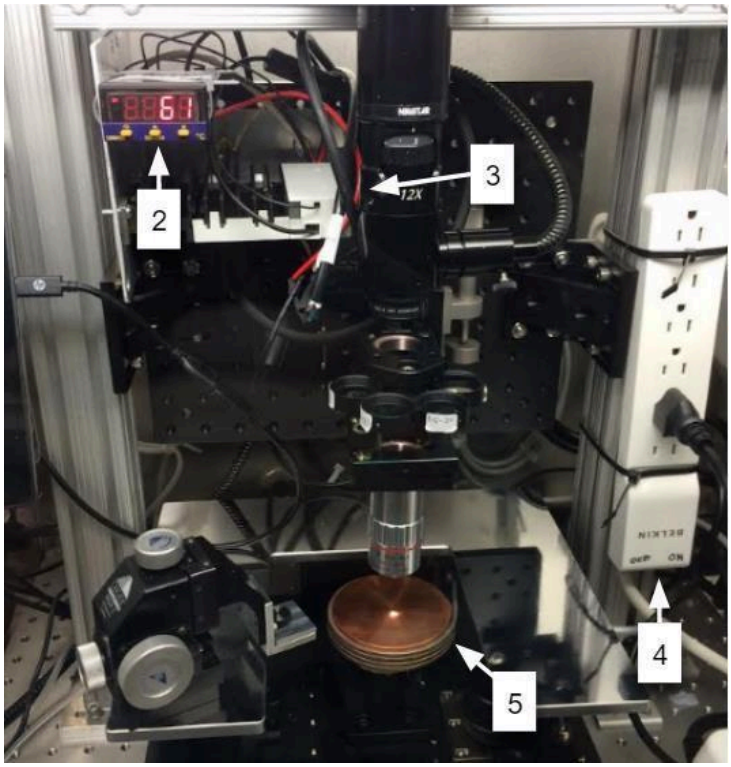
1. Mount sample onto the vacuum chuck ensuring it fully covers the center hole and that the chuck is centered on the probe station stage.
2. **IMPORTANT: Before enabling vacuum, be sure that all vacuum lines are sealed. This means a sample must be on the sample chuck, the valve to the spin-coater must be shut, and the manipulator must be sitting upright on its metal stage. Also**

ensure all tubing is connected and there are no open lines. Failure to do this may result in rapid depressurization of the glove box and severe damage to the system.

3. After ensuring all vacuum lines are sealed, the vacuum can be enabled by turning the knob on the left (outside) of the glove box counter-clockwise (viewed from top).
4. **IMPORTANT: While vacuum is enabled, monitor the pressure in the glove box and compensate for any pressure decreases caused by slight vacuum leaks increasing the pressure with the foot pedal.** In the case of a sudden decrease in pressure, use the foot pedal to increase the pressure and disable the vacuum by closing the valve on the left side of the glovebox.
5. Next, if desired, glass slides can be mounted to the micromanipulator using the clamping mechanism.
6. The micromanipulator may be moved by pressing the button on the backside (bottom) disabling the vacuum and releasing the button when the manipulator is in the desired location.
7. Once, finished with the manipulator and sample stage the vacuum should be disabled before removing sample.

6.5 Heated Chuck

1. See instruction guide, below (next section).



Air tight containers

2. PID control: leave this on. This tells the temperature of the stage, important for subsequent users. Always toggle the screen to the present temperature value when leaving the glovebox.
3. LED light (green): can be viewed obliquely from the right side of the glovebox with the global glovebox light off. If the LED light is on, the PID is sending a signal to the SSR to allow the chuck to heat. This light should be on when the temperature is below the set point value. If the temperature of the chuck is below the setpoint and the green light is off, check the circuits. Reconnecting wiring may be necessary.
4. Power strip for heater. Turn this off when heating the stage is *not* desired. Turning this power strip on when the LED light is on sends current into the coil, heating the chuck.

Always turn off when leaving the glovebox.

5. Heated stage.

a. Try to keep this centered on the support for vacuum purposes. When at room temperature, go ahead and lift up the chuck to see its underside and how it fits into the vacuum system on the support.

Be aware of the heating coil and its slipping downward. In particular, be aware of its proximity to the vacuum tubing. Brett believe the tubing is silicon and should be rated to 200C+. Care should still be taken. Similar precautions should be given for the small rubber rotation adjuster on the front of the stage.

Appendix A

Heated chuck instruction guide

Author: Mathieu De Kruijf (5/13/2019), Pribiag Lab

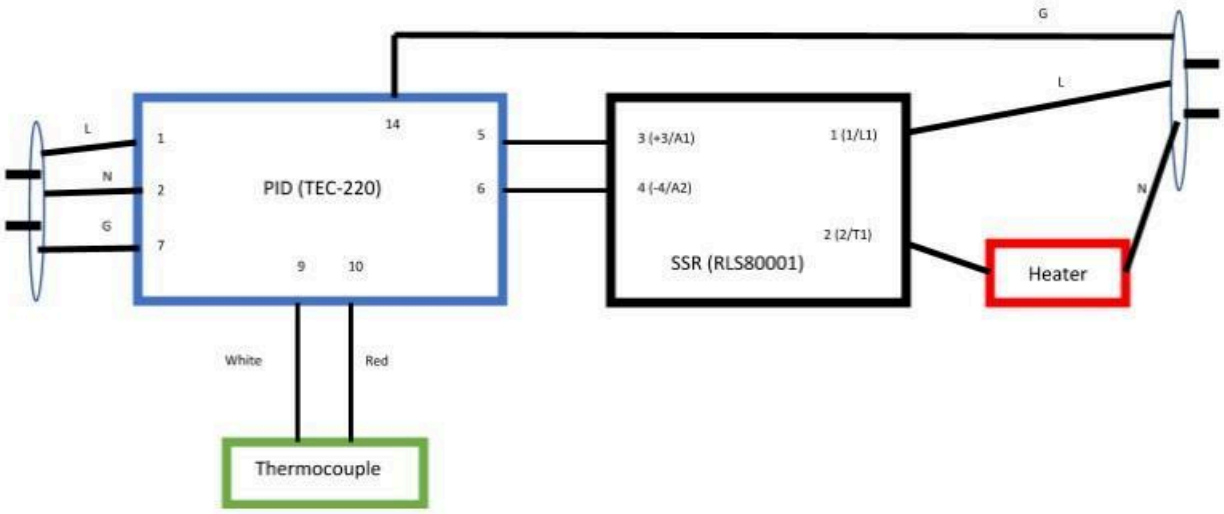
The heated chuck is generally installed in the glovebox in the PAN cleanroom. The heated chuck consists of a heater coil wrapped around a metal plate. The heater is controlled by a proportional–integral–derivative (PID) controller and a Solid State Relay (SSR), these basically check what the temperature of the chuck is and determine if the heater should heat or not.

The device can be turned on and off by flipping the switch on the power cord. The temperature of the chuck is generally set at 150 C. This temperature can be changed by pressing the ↻ until the display shows “SP1”, then press the up or down buttons to adjust the set point temperature. It might take a while for the heater to adjust the temperature after a new set point is set.

The wiring scheme for the heated chuck is displayed below.

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On the right an on the left we have two standard AC power-cord cables (120V). When rewiring it is important to wire these to the proper ports as indicated in the sketch, otherwise you might blow up either the PID controller or the SSR. Ports 7 and 14 are don't have a direct function on the PID controller so we use them to ground the device. We have included two 5 amp fuses on the live wires of these power cables, these are there for your own safety.

While wiring the thermocouple (the small wirings coming from the heater) note that the red line goes in to port 10 and the white one in port 9 on the PID controller. If these are flipped the temperature read-out will be incorrect, causing erratic behavior of the heater.
Ports 5 and 6 on the PID connect up to ports 3 and 4 respectively on the SSR (take note of the + and – on both sides of the wires, if not sure check the instruction manual for the PID). These wires are sending signals from the PID to the SSR, with these signals the SSR controls if the heater is heating up or in its off state.

Appendix B



Light source - push center small button – adjust brightness turn left knob.

Appendix C

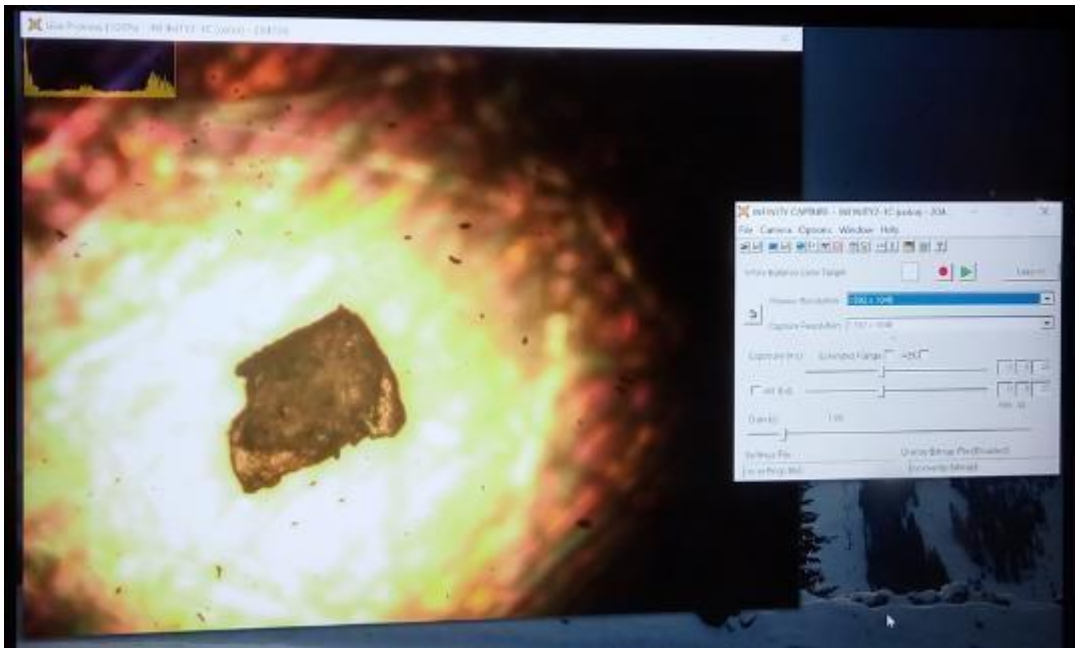


Image capture software displaying a small flake sample.

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Appendix D



Foot control to adjust pressure in chamber.
Push on left side is less pressure in chamber and right side increase.