Protochips
Quantifiably Better™

Atmosphere

USER MANUAL

Protochips, Inc.

Protochips, Inc. provides solutions to enable development and commercialization of revolutionary new materials and applications. Utilizing technology developed around miniaturized semiconductor devices, Protochips transforms one of the most widely used tools in nanotechnology – the electron microscope – into a complete nano-scale laboratory. *In situ* study of materials for the life and physical sciences in the electron microscope is now Quantifiably Better.

Your satisfaction and success with our products is very important to us.

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I. Atmosphere 200 System Description

Atmosphere 200 is a complete *in situ* solution designed to convert nearly any TEM into an environmental TEM on an as needed basis. It is centered on a workflow-based software package, automated gas manifold and advanced semiconductor-based sample supports called E-chips (environmental chips).

Atmosphere supplies experiment gases to samples at pressures between 3 Torr to 760 Torr (1 atm) in a static or flowing gas environment. It controls the temperature of the E-chip heating membrane between room temperature and 1000 °C at any pressure within its operating range. During each experiment, the software continuously records the system state automatically for later review and analysis of the experiment, as well as actively monitoring for gas leaks. Additionally, the system self-purges gas from the tanks and lines, and can be heated to drive off potential contaminants from internal surfaces of the manifold.

- ***The Atmosphere Gas Handling Manifold is designed to keep experiment gases pure and safe to handle. It is not designed to mix gases.***

FIGURE 1. ATMOSPHERE 200 SYSTEM.

1.1. Atmosphere Software

Atmosphere 200 is operated using a custom workflow-based software package, which features a powerful yet easy-to-use interface and flexible design. The software provides at-a-glance information so the TEM operator can quickly see the current system status yet maintain focus on the TEM and experiment. The "System Status" clearly displays holder pressure and temperature, valve positions and system alarms. The "Workflow" guides users from step by step from setup, through the experiment and system shutdown. As the user steps through the workflow, options including automated pump and purge cycles, to clean residual gas out of the system, and the leak check ensure that the system begins in optimal conditions. The temperature and pressure is controlled independently during the experiment while the system constantly maintains a stable environment by monitoring and logging pressures and temperatures for safety.

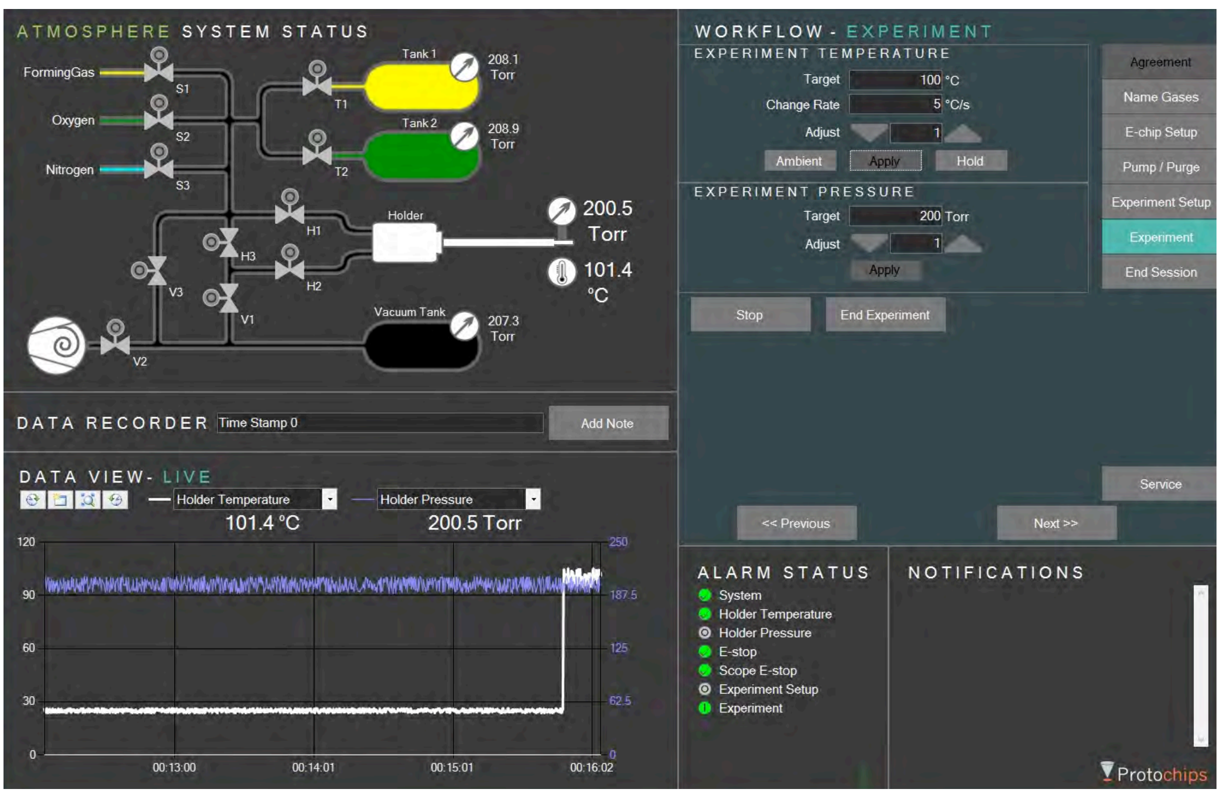


FIGURE 2. SCREENSHOT OF THE ATMOSPHERE SOFTWARE. PANES INCLUDE SYSTEM STATUS, WORKFLOW, DATA RECORDER, DATA VIEW, ALARM STATUS, AND NOTIFICATIONS.

1.2. E-chips

Each experiment requires a pair of Atmosphere E-chips, which when loaded into the holder form the sealed gas cell chamber. They are consumable devices and serve as the sample support and heating membrane. E-chips integrate a thin, durable amorphous silicon nitride window and a silicon carbide based ceramic thin film heating membrane to provide a strong barrier for containing gases with minimal electron beam scatter. Each E-chip is calibrated for temperature at the Protochips factory, and a unique calibration file is generated. The calibration files are shipped with every pack of E-chips, and are used with the Atmosphere software to ensure accurate and precise temperature control. The SmartTemp™ closed loop temperature control also uses the calibration file when actively adjusting the temperature in different gas environments. The ceramic membrane is polycrystalline, and not suitably electron transparent for imaging. To maximize electron transparency in the TEM, an array of 9 holes, 9 µm in diameter, is patterned in each membrane, see Figure 3 and 4. The continuous amorphous SiN membrane covers the ceramic membrane, including the holes, to provide the vacuum barrier. Samples must be placed over one or more of these holes before loading the E-chip into the TEM to ensure maximum electron transparency and unobstructed imaging. The heating area is limited to this array of holes, and is approximately 90 microns square.

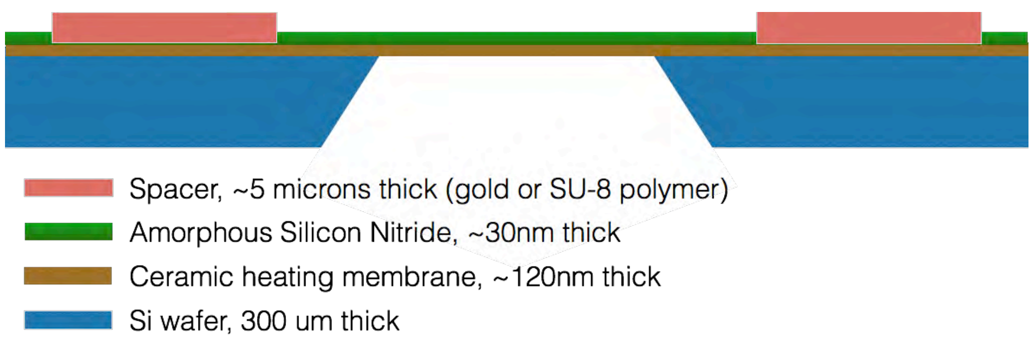


FIGURE 3. A CROSS SECTION SCHEMATIC OF AN ATMOPSHERE THERMAL E-CHIP.

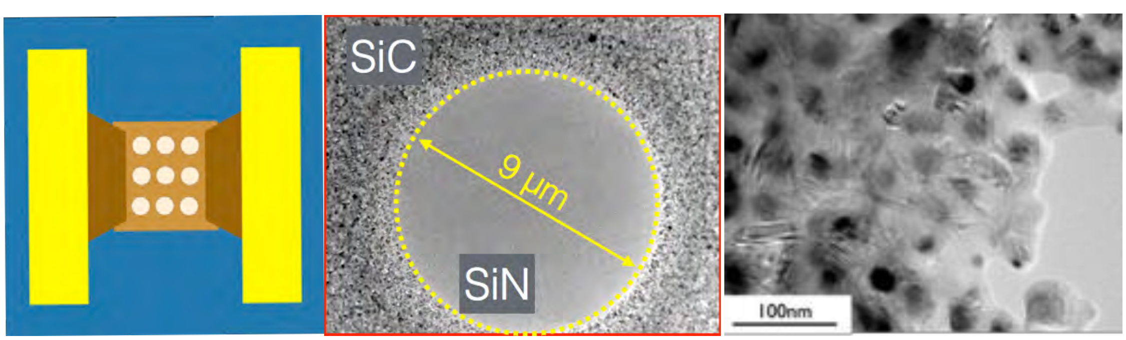


FIGURE 4. LEFT FIGURE IS A SCHEMATIC OF THE THERMAL MEMBANE. CENTER FIGURE IS A BF-TEM IMAGE OF A HOLE IN THE SIC CERAMIC MEMBRANE. THE HOLE IS COVERED WITH THE CONTINUOUS SIN MEMBRANE. IMAGING IS DONE IN THIS AREA. THE RIGHT IMAGE IS A BF-TEM IMAGE OF THE CERAMIC. IT IS POLYCRYSTALLINE AND SHOULD NOT BE CONFUSED WITH THE SAMPLE.

Gold electrical leads patterned on the chip surface interface with the electrical contacts (flexible circuit) on the holder to provide the electrical current required for heating. Proper cleaning and handling techniques for the E-chips are discussed in the following section. Please consult the Atmosphere E-chip ordering guide for specifications and ordering information.

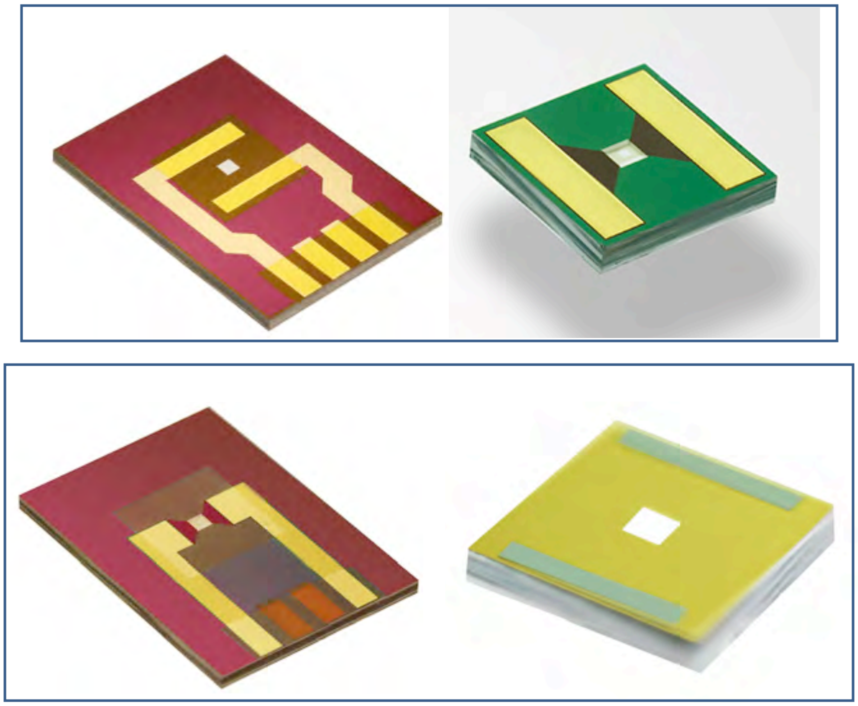


FIGURE 5. ATMOSPHERE E-CHIPS. A PAIR OF E-CHIP DEVICES IS REQUIRED TO FOR THE CLOSED CELL. A LARGE E-CHIP WINDOW (TOP LEFT) IS PAIRED WITH A SMALL E-CHIP THERMAL DEVICE (TOP RIGHT), OR A LARGE E-CHIP THERMAL DEVICE (LOWER LEFT) IS PAIRED WITH A SMALL E-CHIP WINDOW (LOWER RIGHT)

1.3. Holder

The Atmosphere holder contains the E-chips, provides a gas and electrical path to the E-chips, and hermetically seals the closed cell in the TEM. The sample is contained between a pair of E-chips, both with electron transparent windows, within the holder tip. They are secured in the tip by a lid and three screws, with two O-rings, or gasket type O-ring, pressing against each E-chip to provide a vacuum tight seal. The electrical contacts of the holder are not exposed to the experiment gases and are outside of the large O-ring. The holder has three connections to the manifold: 1) a gas inlet connection, 2) a gas exhaust connection, and 3) an electrical cable with a circular Hirose type connector. The unions in the back of the holder are PEEK to electrically isolate the TEM goniometer voltage bias from the gas manifold.

Stainless steel (316) tubing is integrated into the shaft of the holder. Additional tubing is connected to the input and output ports via compression fittings and nut and ferrule adapters (all PEEK). The PEEK unions can be replaced with 316 SS if necessary. However, a non-conductive tube is required in the tube lines to break the electrical path between the gas manifold and TEM.

- ***PEEK (polyether ether keytone) is stable to temperatures of 260 °C, and has a high chemical resistivity. However, it should not be used with the following chemicals: bromine, nitric acid >90% and sulfuric acid >95%. Solvents such as dimethylsulfoxide (DMSO) and tetrahydrofuran (THF) may cause PEEK to swell, and should be avoided.***

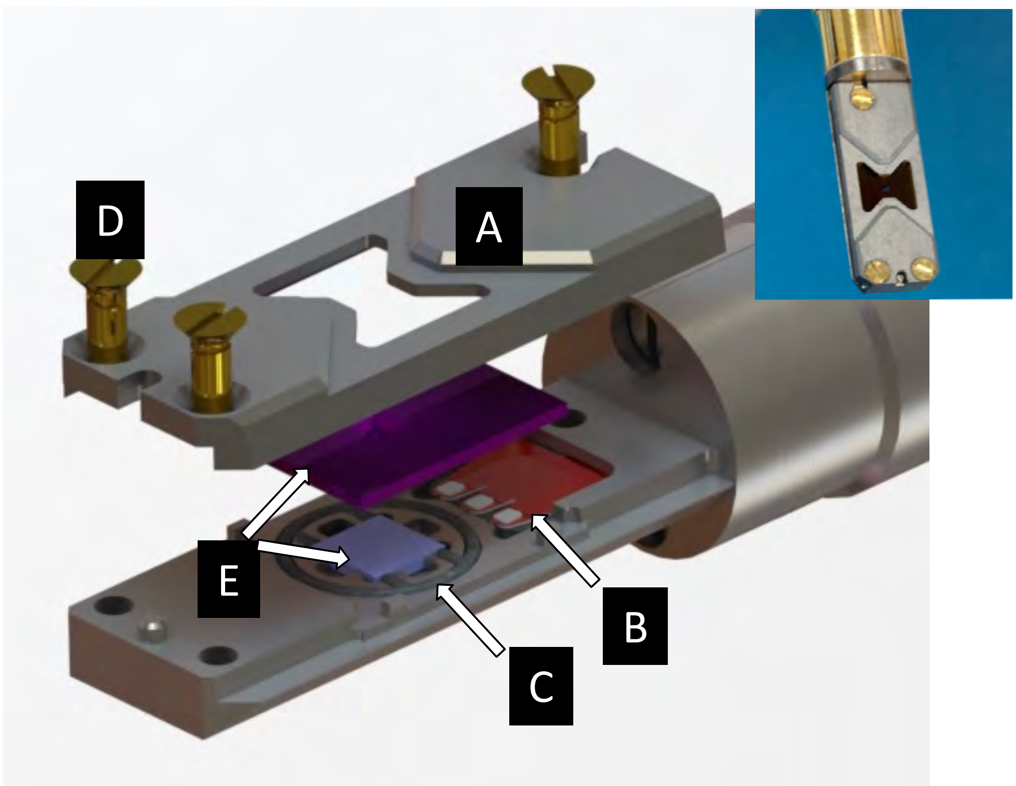


FIGURE 6. ATMOSPHERE HOLDER TIP (EDS COMPATIBLE MODEL). A) LID. B) FLEXIBLE CIRCUIT. C) O-RINGS. D) BRASS SCREWS. E) SMALL/LARGE E-CHIP PAIR.

1.4. Gas Handling Manifold

The Manifold consists of 7 subsystems:

1. Solenoid Driven Pneumatic Valves (11)
2. Experiment Tanks (2)
3. Vacuum Tank (1)
4. Gas Lines
5. Pressure Sensors (4)
6. Manifold Heater and Thermocouples (bake out)

7. ECU with RJ45 (Ethernet) connection

And 6 gas connections:

1. Gas Cell holder connections (2)
2. Experiment/Purge Gas Connections (3)
3. Air Pressure Connection
4. Electrical Power Connection
5. Ground Connection
6. Vacuum Pump Connections

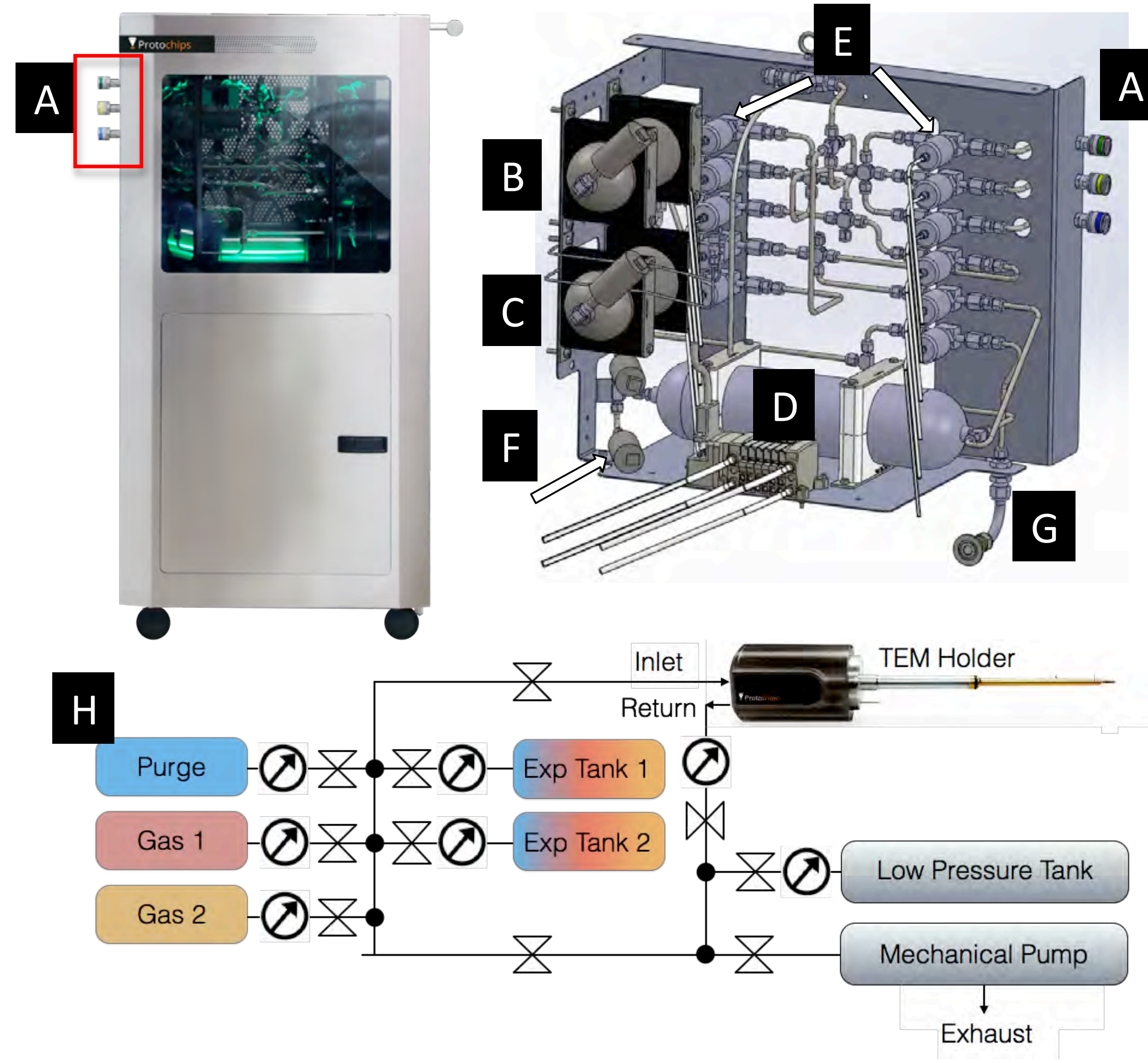


FIGURE 7. A) 3 GAS INPUT PORTS. B,C) EXPERIMENT TANK 1 AND EXPERIMENT TANK 2. D) VACUUM TANK. E) PRESSURE CONTROL VALVES (11). F) PRESSURE SENSOR (4). G) GAS ENTRY MODULE FOR CONTROL VALVES. H) VACUUM DIAGRAM OF THE GAS HANDLING MANIFOLD.

The pneumatic valves control the entry and exhaust of experiment and purge gases, and are used to fill either of two experiment tanks for storing and delivering experiment or purge gases. The vacuum tank provides a pressure differential to control and set pressure in the experiment tanks and holder. The manifold has a pressure relief valve set to 22 psig (1.5 bar) to protect the system if it becomes over pressurized, which would result in a gas experiment error. A roughing pump removes gases from the manifold and the holder when in operation. All subsystems are controlled with a Windows-based desktop computer using the Atmosphere software through the electronics control unit (ECU).

- ***The system runs at or below atmospheric pressure, so the potential for gas escaping the manifold and holder lines is low.***

- ***The pressure sensors should be calibrated once per year to ensure they are reading the correct pressure. Please contact Protochips for assistance.***

1.5. ECU

The ECU (Electronics Control Unit) controls the valves, reads the pressure sensors, monitors the E-stop, controls the temperature of the heating chip, and communicates with the Atmosphere software.

The ECU has electrical connections to the holder, a heater used to bake out the system, manual E-stop, pressure sensors, valves, thermocouples, ground line, computer, and vacuum pump. It is located at the bottom of the gas manifold and can be accessed via the door on the front of the unit. The power button is located at the top of the ECU box.

1.6. Manifold Bake Out System

- ***The manifold bake out process should only run when the Atmosphere system is not being used for experiments. You should allow the system to run overnight to complete the full cycle.***

The bake out system heats two zones in parallel: the manifold lines and the experiment tanks. The vacuum pump will also run during the bake out cycle in order to help remove manifold contaminants. During the cycle, the system will heat to 50 °C while pumping, and return control to the user after the manifold returns to below 35 °C. The full cycle should run overnight.

II. Atmosphere E-chip and Holder Setup

2.1 E-chip Preparation

Each Atmosphere E-chip has a thin silicon nitride membrane (window) or ceramic heating membrane. ***All E-chips should be handled very carefully using carbon-tipped tweezers to avoid damage.*** E-chips are shipped in "gel-paks" with the membrane (flat) side facing up. They should be removed from the gel-pak by gripping the sides of the chip with the carbon fiber tweezers provided, as shown in Figure 9. Metal tweezers can chip the edges of the E-chips and should not be used. All E-chips are thoroughly inspected before shipment, but inspection is also recommended upon receipt using a stereoscope or other light microscope.

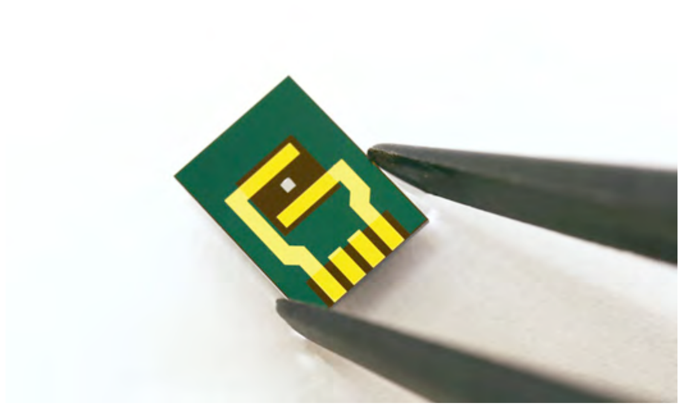


FIGURE 8. ATMOSPHERE E-CHIP HELD CORRECTLY WITH CARBON-TIPPED TWEEZERS.

New silicon nitride (SiN) window E-chips have a protective photoresist coating to prevent damage to the membrane during fabrication and must be removed prior to use. Only the top surface receives the protective coating. The bottom of the E-chip may appear to be a different color prior to removal of the coating. After cleaning, both surfaces should appear the same color.

- ***Thermal E-chips do not have a photoresist protective coating and thus do not require this cleaning procedure.***

Photoresist Removal Procedure:

1. Acetone rinse: To dissolve the protective coating, immerse the E-chip(s) in a clean beaker filled with high purity acetone (0.2 µm filtered), and swirl gently for about 2 minutes. **Do not**

allow the E-chip(s) to dry during the transfer from acetone into methanol. The E-chip surface must remain wet to prevent debris from resettling on the surface prior to the methanol rinse. **Do not** use ultrasonic cleaning as this will break the membrane.

2. Methanol rinse: Immediately transfer the E-chip(s) into a new beaker filled with high purity methanol (0.2 µm filtered) and swirl gently for about 2 minutes. Ethanol can also be used.

3. Dry: Remove the E-chip(s) and quickly blot it (flat side up) on a lint-free paper towel, filter paper or lens paper to remove excess methanol. Dry the surface of the E-chip by directing a flow of air across (parallel to) the flat surface of the E-chip. Nitrogen, compressed or canned air may be used.

4. After the E-chip(s) are dry, use a stereoscope or other light microscope to check that the membrane has remained intact and the surface appears smooth with minimal debris.

If the clean E-chips are not used immediately, they can be stored in the gel-pak for later use.

When the protective coating is present, the surface has a lacquered finish, as shown in Figure 10, and the edges of the windows are not clearly defined. After cleaning, the surface should appear smooth, and the edges of the windows (where the window meets the silicon substrate) should appear sharp. Small drying residues do not generally interfere with use and can be minimized by drying the E-chip under a flow of air after removal from the methanol rinse.

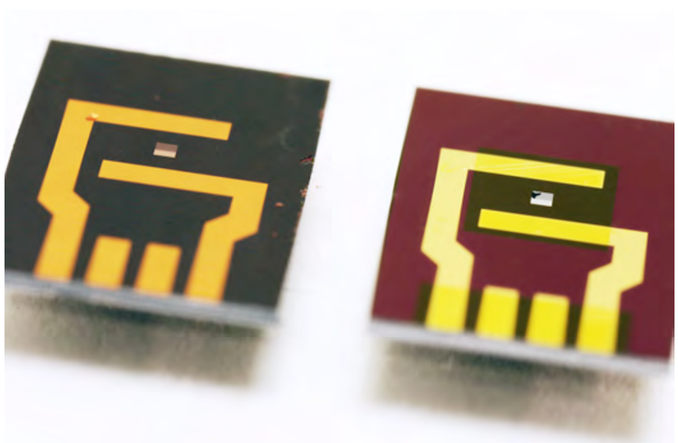


FIGURE 9. ATMOSPHERE E-CHIP WITH PHOTORESIST (LEFT) AND WITHOUT (RIGHT)

Do not use devices that have drying residue or debris directly over the SiN window. Also, avoid using devices if large particles of debris are located on the spacer, which can inhibit electrical contact between both devices. Remove debris from the gold contacts by very gently brushing away the debris using a small wooden or bamboo stick. See the "Sample Preparation" section in the Appendix for more information. This should be done under the stereoscope and only when using a small E-chip thermal device.

- ***E-chips should be plasma cleaned before use to remove contamination from the E-chip surface. Plasma clean each device for about 5 min in an oxygen containing plasma (lab air is also sufficient). Oxygen is key in removing carbon containing compounds***

2.2 Holder Tip Inspection

Figure 10 shows the inside of the holder tip, without E-chips and O-rings. Before use, carefully check the inside of the tip under a stereoscope and remove any lint or debris with carbon tweezers (metal tweezers may scratch the sealing surfaces) that may interfere with the seating of the E-chips or the sealing of the O-rings. It is particularly important to remove any lint or particles in the O-ring pockets, as the presence of debris may cause the holder to fail a leak check.

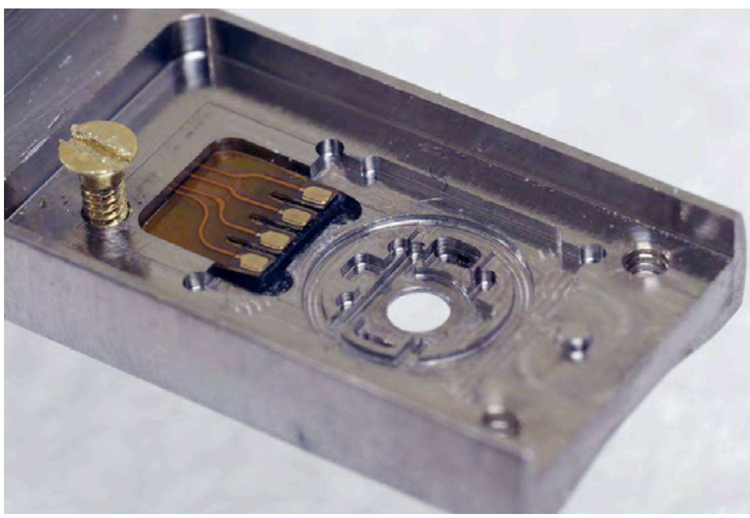


FIGURE 10. ATMOSPHERE HOLDER TIP (JEOL UHR DESIGN) SHOWING O-RING POCKETS, FLEXIBLE CIRCUIT, GAS PORTS AND SCREWS

Do not touch the holder tip without clean gloves. Touching the holder in this area could cause contamination in the microscope and reduce the performance of the holder and microscope.

Plasma Cleaning the Holder

Plasma clean the Atmosphere holder to remove residual organic material from the tip. Typical plasma cleaning procedures, such as 5-10 minutes in argon and/or oxygen plasma, should be followed. It is also recommended that the holder be stored under vacuum when not in use. To plasma clean the holder:

1. Remove the gas capillaries, ferrules and screws, and insert the PEEK plugs on the input/output ports located on the holder handle. Screw in the plugs until they are finger tight.
2. Remove the lid and O–rings from the holder tip. Insert the holder into the plasma cleaner and clean it.
3. Replace with the existing O-rings or with new ones (recommended).
4. The lid can be cleaned using the same procedure with plasma cleaners that contain a shelf for cleaning smaller parts.

Replacing O-rings

The O-rings in the tip play an essential role as they provide the vacuum seal around the holder and E-chips in the microscope. Over time the O-rings and O-ring pockets can collect debris. If the dry pump (see the "Leak Check" section below), or TEM pumping system take longer than normal to achieve the proper vacuum level (~ 1E-6 Torr/mbar range) after about 15 minutes it may indicate that the O-rings need to be cleaned or replaced. To clean and/or replace holder O-rings:

1. Remove existing O-rings from the holder and check for debris in tip and O-ring pocket under a stereoscope.
2. Gently remove debris from the O-rings and O-ring pocket in the holder tip with tweezers or small pick tool. ***Take care not to scratch the O-ring pocket surface or other tip surface.***
3. The O-rings used in the Atmosphere holder are specifically designed for this application. ***Only use O-rings supplied by Protochips.*** Consult the gas compatibility guide for the type of O-ring that fits your experiment needs. Viton, EPDM and FFKM (Kalrez®) O-rings are available.
4. Inspect the O-rings under a stereoscope to check for cracks, lint or debris before placing them in the holder.

5. Proceed with tip assembly and E-chip loading as detailed in the following section.

If the vacuum is not satisfactory when leak checking the holder in the dry pump after loading E-chips, the O-rings and holder tip may need further inspection.

1. If further cleaning is required, remove the O-rings from the tip and wipe them gently on a clean nitrile or powder-free latex glove to remove particles and lint fibers. If cleaning the O-ring does not satisfactorily remove debris, or if the O-ring has cracked or flattened, it should be replaced.
2. Remove any debris left in the tip that could interfere with proper sealing of the O-ring. Residue-free, compressed air can be used to clear the tip surface of dust and other small particles. Do not use compressed air on the flexible circuit, as it may damage the electrical connections or the spring pad underneath the flexible circuit.
   - ***Silicon shards can break off of E-chips, are often difficult to see and can affect sealing. Rotating the tip back and forth under the stereoscope helps to identify shards.***
3. If vacuum is still unsatisfactory, check the O-ring(s) on the holder shaft under a stereoscope and remove any lint or other debris. Replace if necessary with OEM (FEI, JEOL, and Hitachi) approved O-rings.

2.3 Tip Assembly

Two E-chips – one small and large – are required for each experiment. One E-chip is a thermal device with the ceramic heating membrane and the other a SiN window device. The small E-chip has a spacer that sets the gas layer thickness. Consult the E-chip ordering guide for information on the spacer thicknesses available.

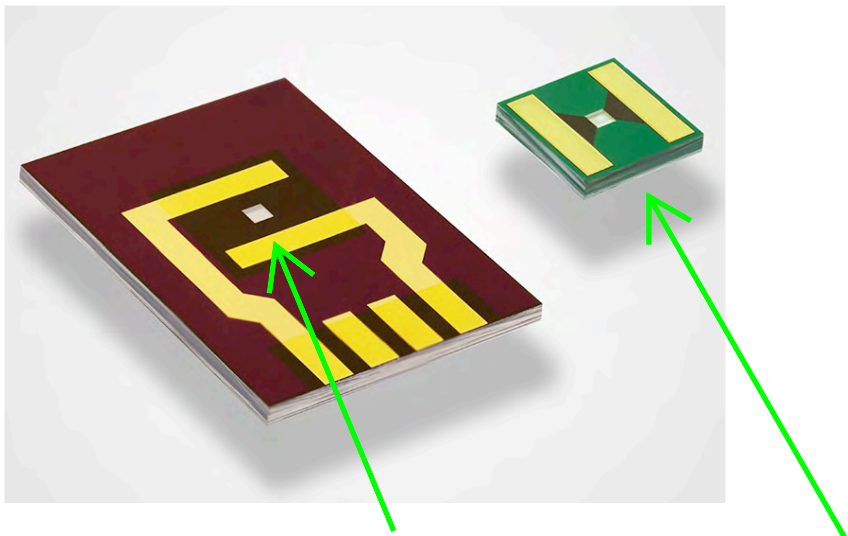
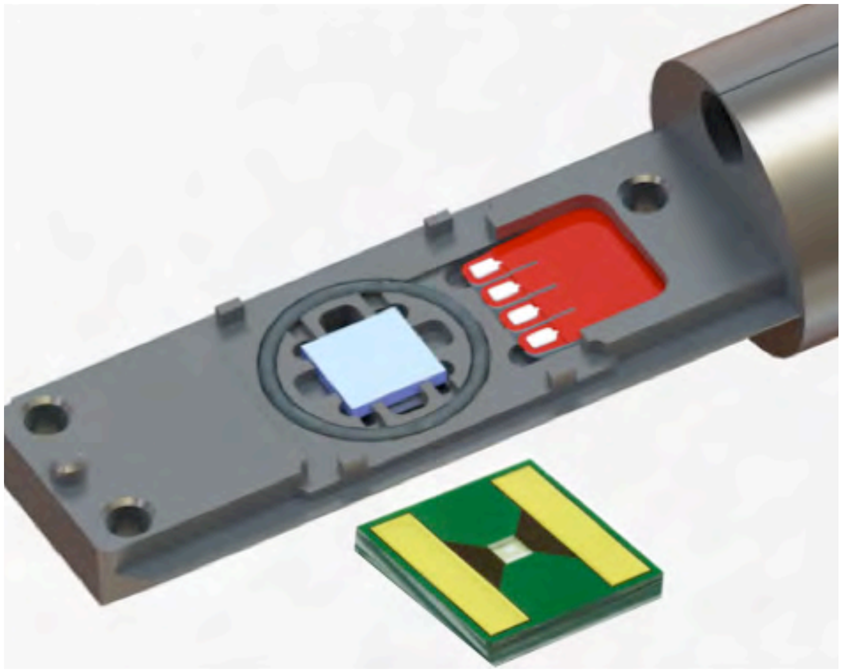


FIGURE 11. ATMOSPHERE E-CHIP PAIR. LARGE E-CHIP WINDOW DEVICE (LEFT), SMALL E-CHIP THERMAL DEVICE (RIGHT). BOTH E-CHIP ARE MEMBRANE SIDE UP IN THIS PHOTO. NOTE THAT THE SMALL E-CHIP IS ROTATED 90 DEGREES IN THIS IMAGE. THE GOLD CONTACTS SHOULD ALIGN WITH THE HORIZONTAL GOLD CONTACTS ON THE WINDOW DEVICE.

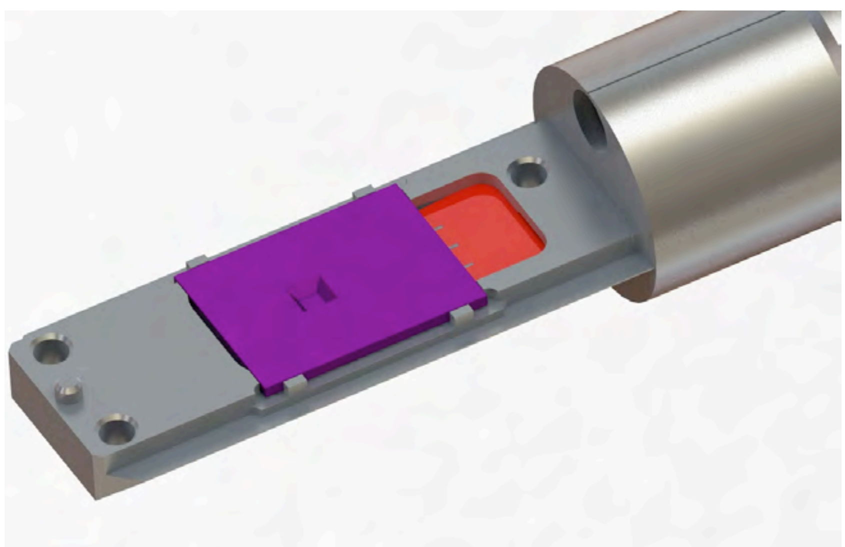
- ***Sample preparation depends on the sample and application. See the Appendix or consult the Protochips sample prep guides for help and tips. The sample should already be prepared on the E-chip before loading it into the holder.***

E-chip Loading:

1. After the tip and O-rings are thoroughly inspected, insert the small E-chip into the pocket with the membrane (flat) side ***UP***. The cavity side should face **DOWN**. If using a small E-chip thermal device, ensure the gold contacts/polymer spacer are in the proper orientation. The contacts should be perpendicular (rotated 90°) relative to the holder shaft. The E-chip should sit flat on top of the small O-ring.



2. Insert the large E-chip into the pocket. This E-chip should be inserted with the membrane (flat) side ***DOWN***. The cavity side should face **UP**.



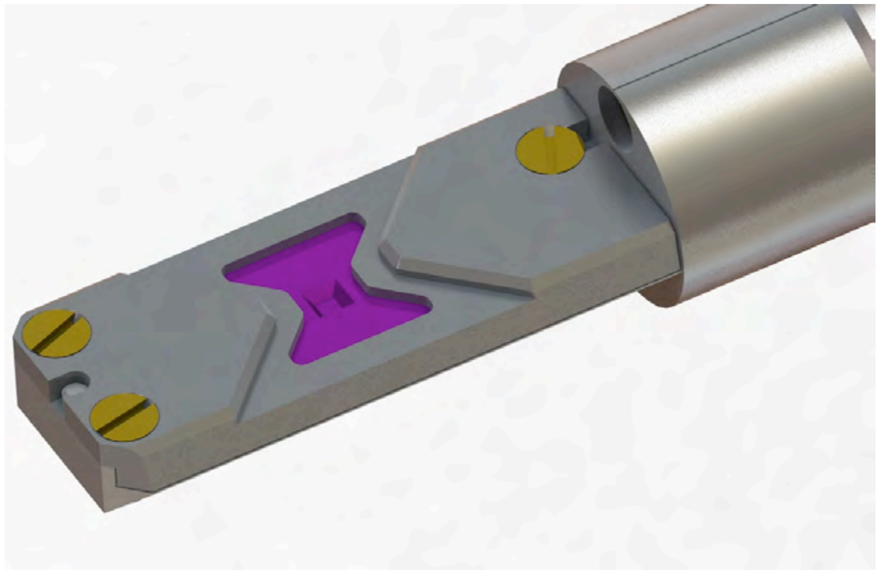
3. Each membrane should now face inwards (facing each other), containing the sample. Verify that the E-chips are seated properly in the pocket, lying flat with respect to the tip. The electrical pads on the E-chips will self align with the contacts on the flexible circuit of the holder.

4. Under the stereoscope check that the membrane regions of each E-chips align. The thermal membrane should be clearly visible and near the center of the window on the opposite E-chip window device.
5. If there is an electrical connection issue, this will be identified when the device check is performed, which is described in a later section of the manual.

Attaching the Lid

When the holder is fully assembled, the O-rings form a vacuum-tight seal. The lid is secured to the tip using three screws, which provide the compression necessary to seal the E-chips and holder tip.

1. Verify that the E-chips are aligned within the tip of the holder using a stereoscope.
2. There are three screws in the holder tip. The back screw, closest to the holder shaft, should only be removed when the screw needs replacing. It should be left in place to serve as a pivot point for positioning the lid. Carefully slide the lid on the tip at a ~45 degree angle around the back screw. Be careful not to displace the E-chips. (If the old screw is stripped and a new one is required, insert the new back screw and tighten it until it begins to thread into the hole. Once it begins to thread, turn it about 2 revolutions until it is secure – do not fully tighten it.)
3. Tighten the back screw until it just catches the lid. This will prevent the lid from slipping while screwing in the front two screws.
4. Insert the two remaining screws into the tip. Gradually tighten each screw, moving from screw to screw so that pressure is applied evenly to the lid. Finish sealing the tip using the torque screwdriver supplied with the system to prevent over-tightening. When the set torque value of *0.1-0.14 lbf-in* is reached, the driver will slip, preventing further tightening. See the Appendix for instructions on setting the torque value.



5. Inspect the top and underside of the Atmosphere tip under a stereoscope. The lid should sit flat against the tip.
6. Rotate the holder very slightly so that the light is reflected off the top window to verify that the window is intact. Rotate the holder 180 degrees and do the same for the bottom window.
7. Ensure the heating membrane is visible through the window E-chip. Holes in the ceramic membrane on the heating device should be readily visible under a stereoscope.
8. To remove the lid, loosen the back screw, remove the front screws and slide the lid up and forward.

- ***The brass screws will strip after some use and need to be replaced periodically. Only screws supplied by Protochips can be used.***

- ***Plasma clean the holder at this point for at least 5 minutes. This helps to remove contamination from the holder tip and E-chip windows. Use an oxygen containing plasma (lab air is also sufficient). Oxygen is key in removing carbon containing compounds***

2.4 Gas lines

The stainless steel capillary lines are connected to the holder through PEEK unions in the holder handle. The lines are sealed with PEEK ferrule compression fittings secured with PEEK screws. PEEK is primarily used to break the electrical connection between the holder and TEM. It is recommended that the capillaries lines remain sealed in the PEEK unions to avoid contamination. The lines are connected to the manifold via quick connect fittings at the end of the boom.

- ***Disconnecting the holder at the quick connects is recommended when possible; they are self-sealing and a reliable method of connection designed to maintain a hermetic seal.***

PEEK connections

First, insert the capillary end through the PEEK screw and ferrule compression fitting. Insert the line into the PEEK union until it bottoms out and cannot be pressed farther. While pushing/bottoming out the lines into the PEEK fitting, finger-tighten the PEEK screws with enough torque to grip the lines to the holder. The gas lines should not be easily pulled out. If the gas lines pull easily out of the holder PEEK fittings, reinsert the tube until it bottoms out in the union and turn the PEEK screws with more torque.

- ***Use clean gloves when inserting the lines into the holder to avoid contamination.***

Holder quick disconnect fittings

The quick connects can be inserted partially with no force, but to properly seat the fitting for a tight seal, the sleeve on the female end of the fitting must be slid back and away from the quick disconnect joint while the male end of the quick disconnect is inserted into the female part.

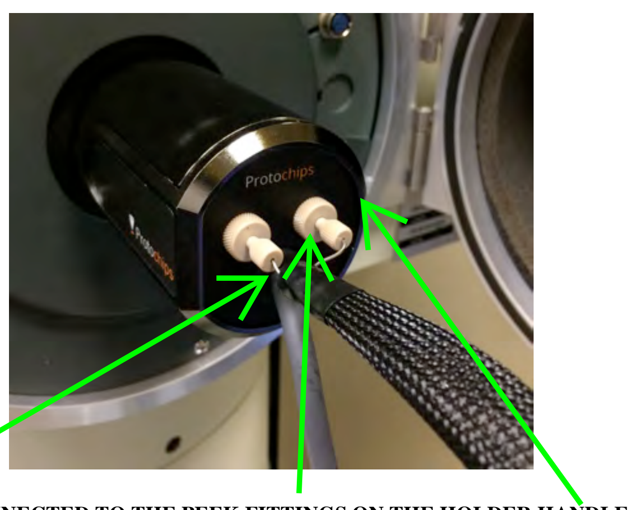


FIGURE 12. GAS CAPILARIES CONNECTED TO THE PEEK FITTINGS ON THE HOLDER HANDLE.

2.5 Leak Check

A thorough leak check is required prior to loading the Atmosphere holder into the TEM. A dry pumping station is necessary to verify that the holder maintains the proper vacuum. To perform the leak check:

1. Insert the holder into the pumping station and allow the holder to pump down for 10-15 minutes. The time required for pump down will depend on the dry pump used. Carefully observe the vacuum gauge for the first few minutes. On an analog gauge, look for the needle to decrease at a steady rate, without jumping back and forth. Jumping back and forth may indicate a leak or moisture on the tip. On a digital gauge, fast pressure fluctuations may not be noticeable, but the pressure should decrease at a steady rate.
2. Once vacuum has equilibrated, **typically in low 10^{-6} to high 10^{-7} Torr/mbar range**, remove the holder and check the windows under the stereoscope as described in step 1.

   ❖ **Do not pull the holder out of the leak check station without first fully venting the chamber. Abruptly pulling the holder out shocks the windows causing a rupture.**

3. If the lines are sealed in the PEEK fittings and connected with the quick connects attached, small leaks, such as those caused by debris on the internal O-rings, may not be identified. **The tip of the quick connect should be depressed with a gloved finger to allow air to enter the holder tip.**

Once the leak check is complete, the holder is safe to insert into the TEM.

III. Atmosphere Gas Handling Manifold Setup and Operation

Before starting an experiment, ensure the desired experiment and purge gases (typically nitrogen or argon) are properly connected to the input lines of the gas manifold. Each source should have a gas regulator set to the recommended pressure as outlined in the system requirements (see Appendix).

Each gas source must be pure, and grade 5.5 (99.9995%) or purer is highly recommended. Electro-polished stainless steel gas lines are also highly recommended between the gas source and manifold input connections to minimize contamination.

- ***The Atmosphere Gas Manifold is designed to keep experiment gases pure and safe to handle. It is not designed to mix gases.***

To begin an experiment, power on the computer, monitor, and manifold. The manifold power is on the beveled edge of the controller and will light up blue when pressed (Figure 13). Connect the holder to the capillary lines via the PEEK unions or the quick connect fittings, if they are not already connected. Connect the electrical cable with the Hirose barrel connectors. The Hirose connectors are keyed and can only be inserted in one orientation. Ensure that you hear a "click" when pushing the connectors together, which verifies a proper connection. These connections can be made either while the holder is on the bench directly next to the TEM, or while the holder is in the vacuum exchange position in the TEM goniometer.

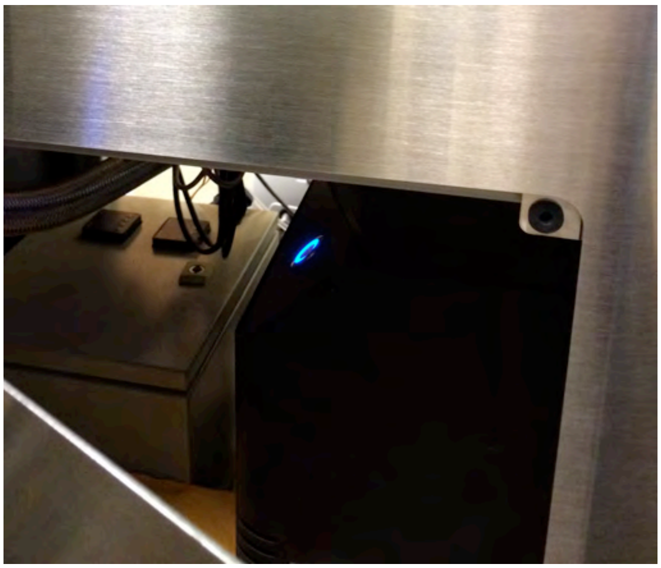


FIGURE 13. THE POWER BUTTON (ROUND BLUE LED)

- ***Before connecting the holder to the manifold, verify that the holder lines and system are at a minimum of 700 Torr. If the pressure in the manifold holder lines is very low, the connection of a holder to the manifold can rapidly decrease the gas pressure in the holder and cause the E-chip windows to break.***
- ***The in-gap objective aperture should be removed prior to inserting the Atmosphere holder and remain out during use in TEMs with the JEOL UHR and FHP type pole piece.***

3.1 Atmosphere Software

The Atmosphere software is divided into 6 panels. Each panel is designed to provide specific information or functionality, such as an easy-to-use workflow-based experiment process and an easy-to-read snapshot of the system status and data logging.

- ***The pressure and temperature units are selectable. The default is Torr, but units such as mbar and kPa can also be used.***

1. System Status

The System Status panel schematic shows the status of the all of the valves, pressure sensors, pump, device temperature, gas locations, and locations of errors in the manifold.

FIGURE 14. VALVE STATES. THE LEFT IMAGE INDICATES AN OPEN VALVE, AND THE RIGHT IMAGE INDCATES A CLOSED VALVE.

The color of the manifold shows the location of a particular gas inside the manifold. This color is user-defined. A ***black color*** represents a “vacuum” or base pressure state. When the user input for a low pressure is reached, the system will consider this pressure as the base pressure and change the color to black. For example, if the base pressure is set to 5 Torr, the color will turn black upon reaching this pressure. Alternatively, if the base pressure is set to 20 Torr, the color will turn black upon reaching this target pressure. A ***tan color*** represents an unknown gas (typically upon software startup, power outage, or when a leak has occurred). For example, in the left figure below, tan is shown in each area of the manifold, indicating an unknown gas. In the right figure, yellow in Tank 1 indicates forming gas (Gas 1), and green in Tank 2 indicates oxygen (Gas 2). The black color in the manifold lines and vacuum tank indicates these areas are at the specified base pressure or vacuum state.

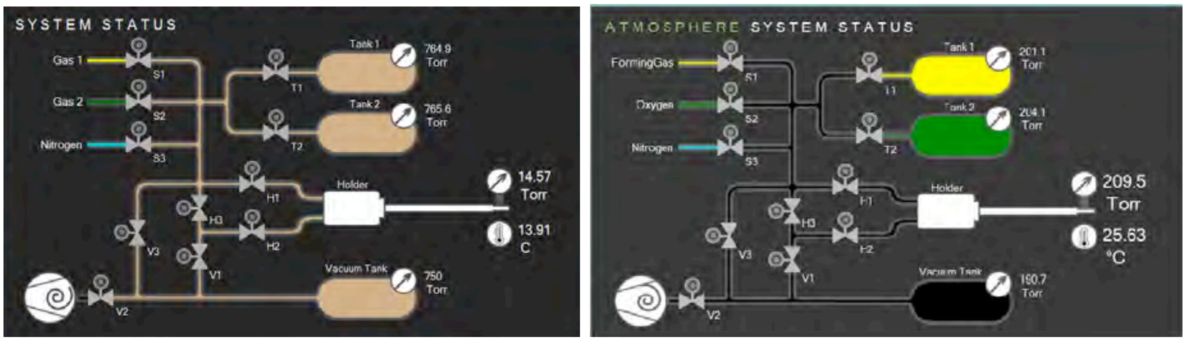


FIGURE 15. THE LEFT IMAGE SHOWS THE STATE OF THE MANIFOLD WHEN AN UNKNOWN GAS OCCUPIES EACH COMPONENT OF THE MANIFOLD, AS INDICATED BY THE TAN COLOR. THE RIGHT IMAGE SHOWS 3 STATES. THE YELLOW INDICATES TANK 1 IS FILLED WITH FORMING GAS (GAS 1), AND GREEN INDICATES TANK 2 IS FILLED WITH OXYGEN (GAS 2). THE BLACK INDICATES A VACUUM STATE.

2. Workflow

The Workflow panel has 7 main screens. During an experiment, users should follow the buttons on the right side of the panel from top to bottom, which guides the user from setting up the experiment to performing to experiment and ending the experiment session.

A. Agreement

The Agreement screen shows the user agreement information, and requires a user name to be entered.

- ***Clicking the "E-Stop" button at the bottom of the Workflow screen will immediately shut all valves in the manifold and bring the temperature back to room temperature. This button should only be clicked when necessary, and serves the same function as the mechanical E-Stop button hardwired to the manifold.***

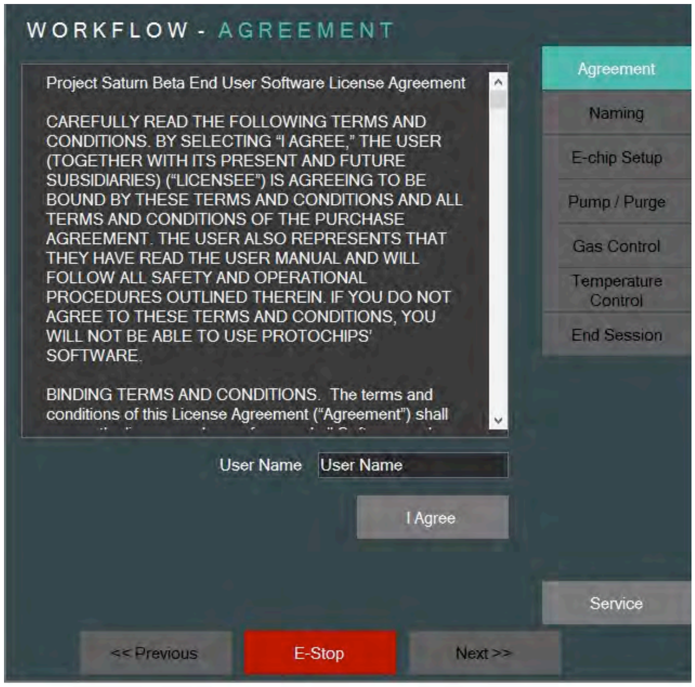


FIGURE 16. THE AGREEMENT SCREEN, WHICH IS SHOWN UPON SOFTWARESTARTUP.

B. Naming

Gases

Enter the names of the gases and change the color code of the gas if desired on the "Name Gases" screen. Any name and color entered for the gas will change the name and color of the gas on "System Status". If the color of the gas is changed, the new color will be used for the gas in the manifold and on the "Data View" when that gas enters the manifold. Note that all names and colors entered will remain until changed or a new version of software is installed. It is highly recommended that the gas name and the actual gas connected to the manifold are the same to avoid confusion.

- ***The gas color code default is "Gas 1" = Yellow, "Gas 2" = Green, "Gas 3/Purge" = Light Blue. These colors match the color bands on the input lines on the side of the manifold. Changing the colors on the software is not recommended; it may cause confusion.***

FIGURE 17. THE NAME GASES SCREEN, WHERE EACH INPUT GAS CAN BE NAMED AND COLOR CODED.

Log File

The Log File automatically records the pressure of each sensor, including Experiment Tank 1 and 2, Vacuum tank and the holder pressure, and the holder temperature. The Log File Name and saved location can be changed by pressing the "…" button. The log file name should reflect the appropriate information, such as the date and name of the sample, etc. If a "File Exists" dialog box appears, press "Cancel" and change the location or name of the log file, or press the OK button to overwrite the log file (not recommended). Press the "Next" button when both the user name and log file have been entered.

FIGURE 18. "FILE EXISTS" DIALOG BOX INDICATES THATA LOG FILE WITH THE SAME NAME ALREADY EXISTS.

C. E-chip Setup

The E-chip Setup screen is used to select the corresponding calibration file for the E-chip used for the experiment. The software will not allow manifold operation until a valid calibration file is loaded.

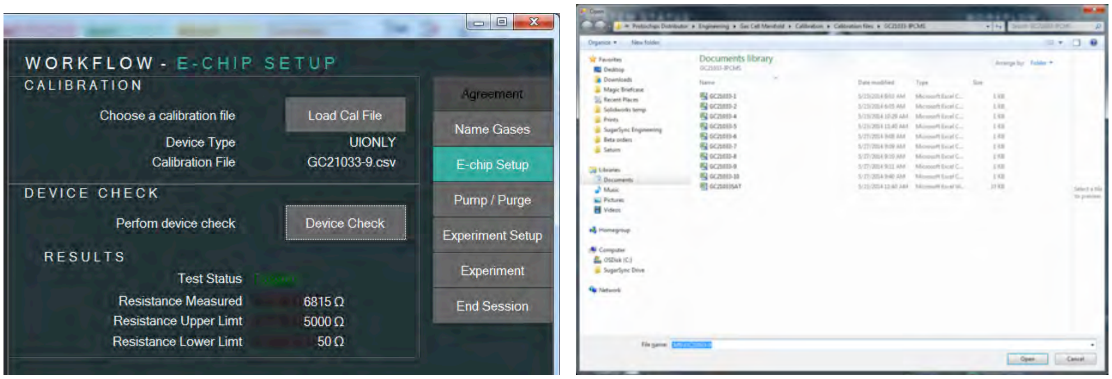


FIGURE 19. LOADING THE CALIBRATION FILE UNDER E-CHIP SETUP.

Press "Load Cal File" and select the calibration file that matches the E-chip used for the experiment. The calibration files are supplied on CD with every box of E-chips. Each E-chip has a number next to it in the box, and every box has a unique serial number. For example, E-chip GC21033-9, as shown above, is number 9 in the box with serial number G21033 (Figure 20).

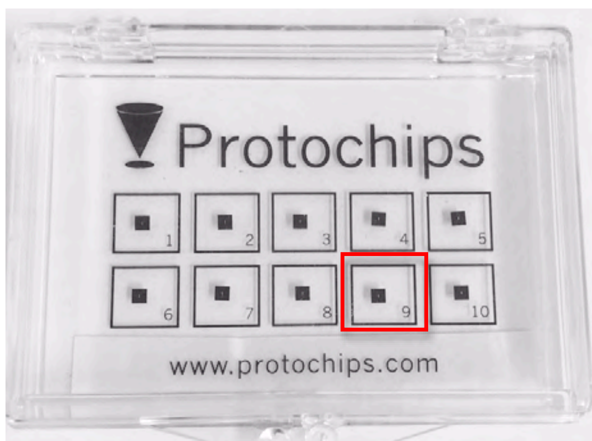


FIGURE 20. A BOX OF ATMOSPHERE (SMALL) E-CHIP DEVICES.

- ***Each calibration file is unique for each E-chip. Take care not to lose track of the calibration file and its corresponding E-chip.***

- ***Be sure that the holder is properly loaded with E-chips, inspected under the stereoscope and leak checked. The electrical cable should also be connected to the holder dongle before running a device check.***

When running a device check, the manifold will verify whether the thermal E-chip is in good electrical contact and has the expected resistance based on information in the calibration file.

Some errors may occur when running the device check.

1. Very high resistance, in the MΩ, usually resulting from an open circuit.
   a. Causes: dirty contacts, improperly loaded or missing E-chip, missing small O-ring, or an unconnected holder.
   b. Solution: Check holder connection to the manifold, clean and/or reload the E-chips.
2. High resistance in the kΩ range.
   a. Causes: A thermal E-chip with a broken window will still conduct some current, but the resistance is above what is expected based on the calibration file.
   b. Solution: Verify that the heating membrane is still intact using a stereoscope and replace the E-chip if it is cracked or broken.
3. Resistance value that passes the device check, but does not fall directly between the upper and lower limits.
   a. Cause: The wrong calibration file may have been loaded, but the calibration file happens to be close to the E-chip currently being used.
   b. Solution: Verify that the correct calibration file has been selected for the E-chip installed in the holder.
4. The resistance falls below the lower limit.
   a. Causes: Either the sample has created a low resistance path between the electrodes, or the wrong calibration file was selected.
   b. Solution: (1) Verify that the correct calibration file is selected. If the error persists, ensure the contacts are free of sample or debris. A new sample may have to be prepared. (2) If using a small E-chip thermal device, ensure the gold contacts are in the proper orientation. The contacts should be perpendicular (rotated 90°) relative to the holder shaft.

D. Pump/Purge

The "Pump/Purge" screen cleans the manifold and holder by displacing and removing residual gases. It is the first step in preparing the system for an experiment. The number of pump/purge cycles can be selected, which determines gas purity. A low vacuum experiment can also be prepared using this menu. Additionally, a system leak check and system bake out is performed at this point if desired.

1. Backfill With Purge Gas

When selected, the manifold backfills (purges) the selected volume, after pumping down to the specified pressure. Clicking this box activates the "Backfill Gas", "Backfill Pressure", and "Backfill Cycles" selections. The "Backfill With Purge Gas" selection defaults to OFF, so it must be selected each time to perform this function.

Target

The "Target" is the intended volume of the manifold to be vacuumed and purged with gas. There are 8 selectable volumes:

1. System: Pump/purge gases from all volumes inside the manifold, but **not** the lines from the gas source tanks to the manifold.
2. Sample Holder: Pump/purge gases from the sample holder and the vacuum tank/manifold.
3. Experiment Tank 1: Pump/purge gases from the experiment tank 1 and the vacuum tank/manifold.
4. Experiment Tank 2: Pump/purge gases from the experiment tank 1 and the vacuum tank/manifold.
5. Manifold: Pump/purge gases from the central lines in the manifold but does not open experiment tanks or the sample holder.
6. S1 Input Line: Pumps the input lines when the tank regulator valve is closed (an error will occur if the valve is not closed). These lines can be pumped down, but not purged with gas, to approximately 1 Torr and should be leak checked anytime a new gas source is connected or when a line may have been disconnected. The user should leak check for at least 5 minutes. If there is a steady increase in pressure in the lines during a leak check, verify all connections to the tank and manifold are leak free.
7. S2 Input Line: See "S1 Input Line" above
8. S3 Input Line: See "S1 Input Line" above

❖ ***Pumping gas from the S1-3 input lines is very important when working with gases that are dangerous when coming into contact with air.***

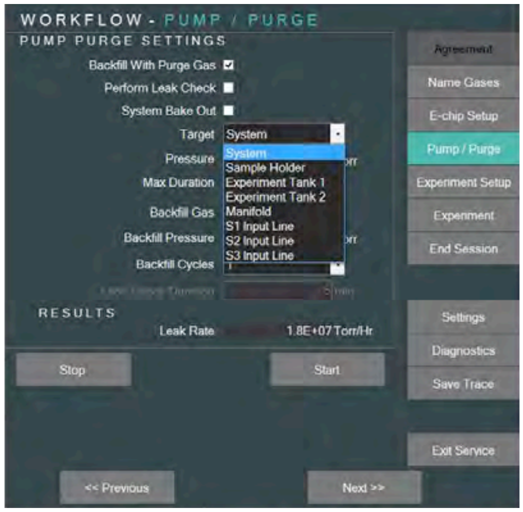


FIGURE 21. PUMP/PURGE SCREEN. SETTING UP THE PUMP/PURGE TARGET.

Pressure
This is the target pump pressure all active sensors must achieve before the valves close and the pump process ends or the next purge cycle begins. The experiment tank and holder pressure sensors have a minimum pressure reading of about 1-2 Torr, and pumping those volumes below 2 Torr is not recommended. 50 Torr is the maximum pressure, and any higher value entered will automatically revert to 50 Torr.

Max Duration
A maximum time for a pump down process to control by time instead of pressure.

Backfill Gas
The purge gas selected for purging the system during the pump/purge cycle. The “Backfill Gas” selection can be the intended experiment gas, or very pure non-reactive gas such as argon or nitrogen.

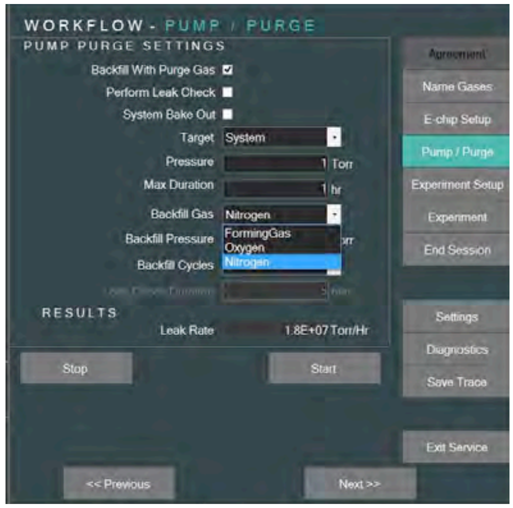


FIGURE 22. PUMP/PURGE SCREEN. SETTING THE BACKFILL GAS TYPE.

Backfill Pressure

The "Backfill Pressure" is the target purge gas pressure. It is recommended to purge to 760 Torr (1 atm) if possible, unless a lower pressure is necessary.

Backfill Cycles

The number of "Backfill Cycles" determines how many cycles the system will pump down to the low pressure target and purge to the backfill pressure target. A typical vacuum and backfill cycle with the experiment or purge gas is three cycles from 760 Torr to 5 Torr.

FIGURE 23. PUMP/PURGE SCREEN. SETTING THE NUMBER OF BACKFILL CYCLES.

- ***Three purge cycles from 760 Torr to 5 Torr will normally dilute any gas contaminants below the level of contaminants of***

the experiment gas. However, more cycles can be done if desired.

Purge cycle number	Gas	Pstart (Torr)	Pfinish (Torr)	Percent of contaminants remaining
1	Unknown gas	760	5	0.7%
2	Experiment gas	760	5	0.004%
3	Experiment gas	760	5	0.00003%
4	Experiment gas	760	5	0.0000002%
5	Experiment gas	760	5	0.000000001%

TABLE 1. THE NUMBER OF PUMP/PURGE CYCLES WILL AFFECT THE AMOUNT OF CONTAMINATES LEFT IN THE SYSTEM. RUNNING 3 OR MORE CYCLES WILL DISPLACE ENOUGH RESIDUAL GAS SO AS TO NOT AFFECT THE PURITY OF THE SOURCE GAS.

Stop and Start

The "Start" and "Stop" buttons begin and stop the Pump/Purge process. When the "Start" button is pressed, all settings will be applied and the system will run until the process completes. The "Stop" button can be pressed anytime during the process, which stops the pump and closes all valves.

2. Perform Leak Check

When "Perform Leak Check" is selected the system will monitor for any leaks in the manifold, holder and lines. The "Leak Check Duration" can be changed to allow user-defined leak check durations, and to minimize the effect of pressure waves from the initial valve closings, which can result in an artificially high leak rate. The default time is 5 minutes, but **a longer leak check time of at least 1 hour for more accurate results is recommended**.

Leak Rate

The software calculates a leak rate when a leak check is performed. This number should be recorded and used as a baseline for verifying the system is leak free and compared to future values to verify system performance. Note that the longer the leak check duration, the more accurate the leak rate calculation will be. Leak rates in the mTorr/Hr range can be expected during normal operation.

FIGURE 24. PUMP/PURGE SCREEN. PERFORMING A LEAK CHECK AND SETTING THE LEAK CHECK DURATION.

3. Bake Out Manifold
   During bake out, the experiment tanks and manifold lines are heated to 50 °C, while the vacuum pump runs, to remove volatiles and contaminants. This process typically takes several hours, and the manifold cannot be used until the temperature of the manifold returns below 35 °C. A bake out cycle should be performed periodically to ensure the manifold remains free from contaminates. This process can be done overnight, as the bake out system automatically turns off when the cycle completes.

FIGURE 25. PUMP/PURGE SCREEN. SETTING UP THE SYSTEM BAKE OUT.

E. Gas Control

After the pump/purging cycle is complete, the system can be set up with the desired gas species and pressure. Each Experiment Tank is set up individually with different gas types.

Type

The experiment Type is "Static" or "Flow".

When "Static" is selected, the manifold opens the experiment tank and the lines to the holder (H1, H2 and H3 valves), which allows the pressure to equalize between the experiment tank and the holder. The gas travels into the holder and the pressure reading in the experiment tank will be the pressure in the cell. The pressure equalizes quickly – usually less than 10 seconds.

When "Flow" is selected, the manifold opens the experiment tank and holder line valves and allows the pressure to equalize just as in the "Static" configuration. During the experiment, the experiment and vacuum tank valves and the holder valves H1 and H2 will remain open to allow gas to flow from the experiment tank through the holder into the vacuum tank. The flow rate depends on the differential pressure between the experiment and vacuum tank as well as the size and length of the capillary configuration.

Gas

The experiment gas is selected in the "Gas" dropdown menu. Only one gas can be selected for each tank. **Gas mixing is not allowed in the Atmosphere manifold**. **Experiment Tank 1 and 2 are identical in function.**

Pressure

An initial target pressure for the holder is entered here. The pressure can be increased or decreased during the experiment.

- ***After setting the system to "Static" or "Flow" and the target pressure is set, press the "Start" button. To change the pressure or Type, make the appropriate selections and press "Update". Note that changing the holder pressure requires the valves to temporarily close in the manifold. This is a safety precaution to avoid shocking the cell with pressure.***

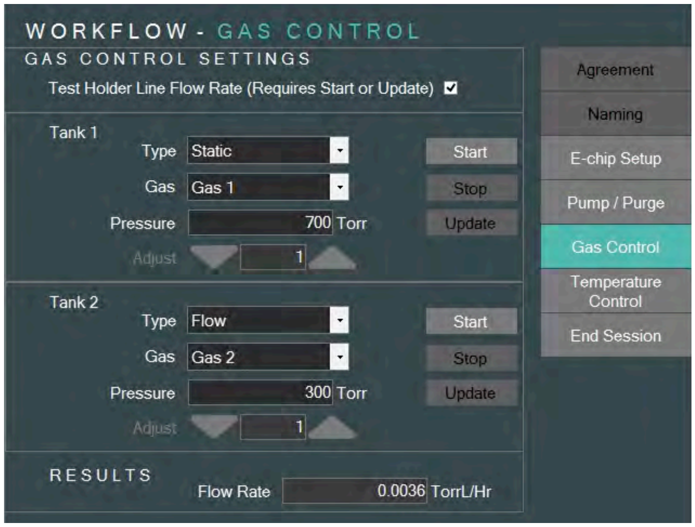


FIGURE 26. EXPERIMENT SETUP SCREEN. THE EXPERIMENT TYPE (STATIC, FLOW OR VACUUM), SOURCE GAS, EXPERIMENT TANK AND PRESSURE ARE ALL SET HERE.

- ***To switch from Experiment Tank 1 to Experiment Tank 2, or vice versa, the gas in the holder lines must be pumped out and purged. First press "Stop" on the Experiment Tank currently in use. Go to the "Pump/Purge" tab and complete one or more pump/purge cycles on the "Sample Holder" by selecting it in the Target drop down menu. Once the pump/purge cycle is complete, the new gas can be introduced into the sample holder.***

F. Temperature Control

Once the Gas Control setup is complete, temperature can be applied to the sample. On the "Temperature Control" screen the temperature, between room temperature (~35 °C) and 1000 °C, is set.

1. Experiment Temperature

   Change rate

   The fastest change rate is 5 °C/s, and the slowest is 0.1 °C/hr.

   Target

   The target temperature is entered into "Target". After entering the target temperature, click "Start" and the system will begin to increase or decrease the temperature at the specified change rate. To change the temperature input the target temperature and click "Update".

   Adjust

   In addition to entering a "Target" temperature, the temperature can be incremented and decremented at a user-defined step size. Enter the step size in "Adjust" and click the "Up" or "Down" arrows to increment or decrement, respectively, by the specified step. There is no need to click "Update", and clicking on an arrow will begin the temperature change.

Ambient and Hold Buttons

Clicking "Ambient" will adjust the temperature to room temperature at the specified change rate. When the "Hold" button is clicked while the temperature is changing, the system will hold at the current temperature. To begin changing the temperature again, specify a target temperature in "Target" and click "Update". Click "Stop" to end the experiment and bring the sample back to room temperature.

❖ ***The temperature does not change in a continuous ramp, but instead changes in a stepwise fashion as a result of the closed loop temperature control algorithm. The average change is equal to the specified change rate.***

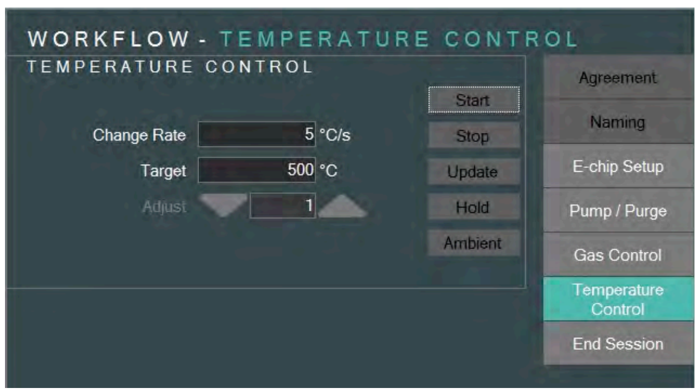


FIGURE 27. EXPERIMENT SCREEN. THE TEMPERATURE AND PRESSURE IS ADJUSTED HERE DURING EXPERIMENTS.

G. End Session

Before removing the Atmosphere holder from the TEM, the experiment gas must be removed and inert gas backfilled into the holder to at or near 1 atm. This process is the same as the "Backfill With Purge Gas" in the "Pump/Purge" screen. Before the End Session can start, the Temperature and Gas Control processes must be stopped. Go to each screen and click the "Stop" button to ensure each process has stopped running.

❖ ***When the Atmosphere holder is removed from the TEM, it should be at or near atmospheric pressure. This avoids shocking the windows and causing the membranes to come into contact due to the inverse pressure differential.***

Backfill Settings

Pump Down Pressure

The manifold first pumps the system to a specified pressure. The target pressure is entered in "Pump Down Pressure".

Max Duration

A maximum time can be entered for a pump down process to control by time instead of pressure.

Backfill Gas

The manifold purges the system with a specified gas after reaching the target pump down pressure. Normally an inert gas such as nitrogen or argon is used. The gas type is chosen in the "Backfill Gas" dropdown menu.

Backfill Cycles

The number of pump/purge cycles can be specified in "Backfill Cycles", and determines the number of pump/purge cycles. A typical vacuum and backfill cycle with the experiment or purge gas is three cycles from 760 Torr to 5 Torr.

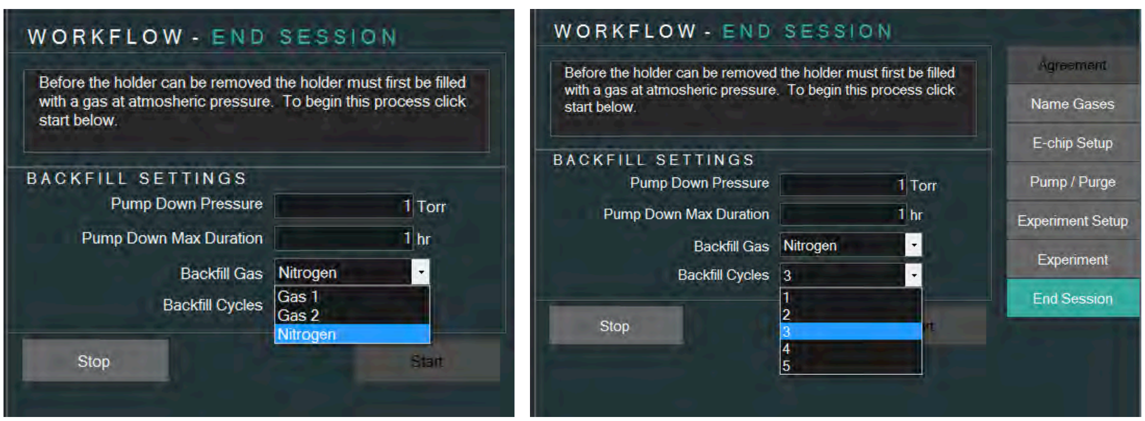


FIGURE 28. END SESSION SCREEN. AFTER COMPLETING THE EXPERIMENT, THE SYSTEM IS SHUT DOWN HERE. THE ENTIRE SYSTEM IS PUMPED OUT AND BACKFILLED WITH INERT GAS (NITROGEN OR ARGON) TO 1 ATM PRESSURE.

When the pump/purge cycle completes, a dialog box will appear. At this point the holder can be removed from the TEM. The system will stop logging data and the log file can be accessed. It is saved in ".csv" format; a format readable by many data analysis programs such as MS Excel, Matlab, Origin, etc. The log file is saved in the location specified in the "Agreement" screen.

- ***The gas manifold should be filled with nitrogen or argon to 1 atm when not in use.***

To turn off the power to the gas manifold, open the front door panel and press the blue LED button on the top of the ECU.

H. Data View

The "Data View" screen shows a graphical readout of the pressure sensors and temperature. Large text readouts provide an at a glance view of the selected parameter. The pressure sensor or holder temperature can be selected using the drop down menus. The system logs the holder temperature and each pressure sensor 4 times per second, starting when the "Agree" button is clicked. If a different pressure sensor is selected in the drop down menu, data will be shown from the beginning of the experiment.

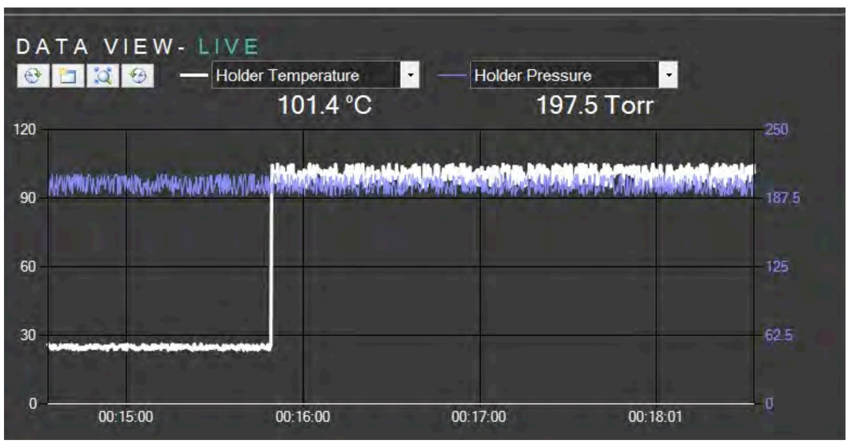


FIGURE 29. THE DATA VIEW SCREEN. DATA VIEW SHOWS A GRAPHICAL REPRESENTATION OF THE DATA AND LARGE TEXT SHOWING THE CURRENT STATUS OF THE SYSTEM PRESSURE AND/OR HOLDER TEMPERATURE.

"Data View" has four buttons at the top left side of the pane. The first button is the "Live" view, which shows the most up to date view of the data. The second button opens a new window, which can be maximized for easier visualization of the data. The third button is "Zoom". After clicking this button, a zoom box can be created over a region of interest using the left mouse button, and clicking, holding and dragging for a zoomed in view. The fourth is "History". The graph in the "Data View" only shows the past 4 minutes. By clicking "History" the entire data log is shown.

FIGURE 30. DATA VIEW BUTTONS. LEFT TO RIGHT: LIVE VIEW, NEW WINDOW, ZOOM AND HISTORY.

FIGURE 31. DATA VIEW SCREEN. AFTER CLICKING ON "NEW WINDOW", A SECOND DATA VIEW SCREEN APPEARS.

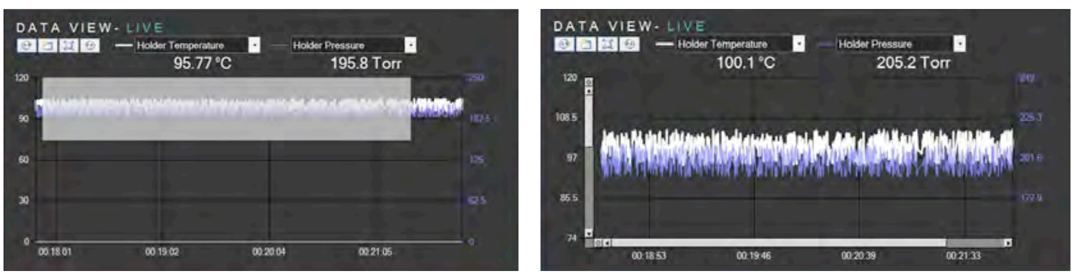


FIGURE 32. DATA VIEW SCREEN. CLICKING ON "ZOOM" AND USING THE LEFT MOUSE BUTTON (CLICK AND DRAG) TO SELECT A SPECIFIC AREA OF THE DATA WILL ZOOM TO THAT AREA (LEFT IMAGE). THE RIGHT IMAGE SHOWS THE ZOOMED IN VIEW OF THE DATA.

I. Data Recorder

During a session, notes can be added to the log file at specific times during the experiment. For example, if a change in the material happened 15 minutes after a temperature change, a note can be added to the log file with the "Data Recorder" for future reference. To add a note, type in the dialog box and click "Add Note". This note will be added to the log file at that specific time.

FIGURE 33. THE DATA RECORDER SCREEN. NOTES ENTERED INTO THE DIALOG BOX WILL BE RECORDED DIRECTLY TO THE LOG FILE.

J. Service

The Service screen allows manual control of the manifold, including independent control of each valve, temperature control and the vacuum pump.

- ***The "Service" screen is password protected. Only users that are certified by Protochips can access this menu.***

Diag.

The "Diag." tab, short for diagnostics, opens the manual control of the manifold. Each valve can be opened and closed independently by clicking on the small silver disk next each valve name. The vacuum pump can be switched on and off as well. This function is designed for service and can be used to diagnose a problem, such as to pinpoint a leak in the system.

Save Trace

When clicking "Save Trace", an output file is generated that records the state machine's data log. It saves the recent status of every subsystem, and is used for diagnostic purposes.

Reset Errors

When diagnosing an issue with the Atmosphere system, errors often occur. The "Reset Errors" button clears any current error.

Settings

Some system settings can be changed in the "Settings" menu. They should only be changed by Protochips Service, or by users trained and certified by Protochips.

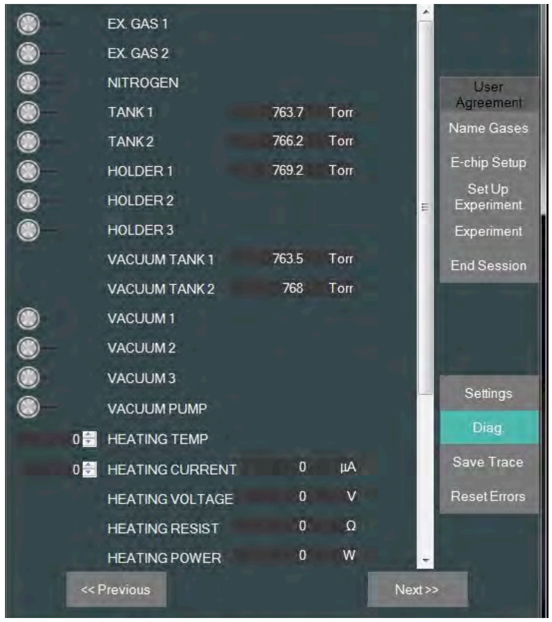


FIGURE 34. THE DIAGNOSTICS SCREEN. THE TEMPERATURE, VACUUM PUMP AND EACH VALVE CAN BE INDEPENDENTLY CONTROLLED HERE.

FIGURE 35. THE DIAGNOSTICS SCREEN. HOLDER VALVES 1 AND 2 ARE OPENED USING THE SWITCHES IN THE DIAGNOSTICS SCREEN. THE VALVE STATUS IS OPEN IN THE SYSTEM STATUS PANE AS WELL.

3.2 Initial Imaging

Inserting the Holder

Reduce magnification (400-1500X is recommended) and spread the beam so that the phosphor screen or camera is fully illuminated prior to inserting the holder. Allot adequate pumping time in the load lock position to allow any outgassing prior to insertion into the microscope column. Longer leak checks using a dry pump will decrease pump time in the load lock, but outgassing may still occur in the microscope column. It is recommended to allow 5-10 minutes in the load lock position prior to inserting the holder into the column.

Window Location and Focusing

Use low magnification mode (low mag) to search for the window region. It should look like an illuminated square on the screen. Once the window is located in low mag mode, reduce the beam intensity (either by spreading the beam or decreasing the spot size) to reduce the possibility of contamination and increase the magnification to >2000x. Search for an area of interest and set the Z-height accordingly. If possible, continue to **adjust the beam illumination to maintain a low electron dose**. It is best to optimize imaging conditions within a small region to prevent sample damage and then move to the area of interest. Contamination will be minimized if the temperature is above 250 °C.

- ***The SiC heating membrane is polycrystalline, and is often confused with the sample. Be sure to find one of 9 holes in the SiC membrane to optimize imaging conditions and beam transparency.***

3.3 Post-Experiment

When the experiment is complete, the holder should be disassembled and the E-chips removed. Although it is recommended that each E-chip device be used for one experiment, they can be reused depending on the experiment. They can also be saved for further analysis in the TEM or SEM.

Holder and Gas Capillary Storage

The holder should be stored in vacuum if possible, the E-chips and O-rings removed, the lid reattached to the tip, and the gas capillaries attached to the holder. The quick connects self seal, so the holder will be vacuum tight. If the gas capillaries are removed, the PEEK plugs can also be screwed into the fittings on the holder handle. The plugs will maintain a vacuum seal. If the gas capillaries are not attached to the holder during vacuum storage, they should be protected from contamination. Clean aluminum foil can be wrapped around the end of the capillaries, or they can be stored in a clean environment, such as a nitrogen dry box.

APPENDIX

SETTING THE TORQUE SCREWDRIVER

A torque screwdriver is supplied with each Atmosphere system to ensure the proper torque is applied to each screw on the holder tip. This results in a more accurate seal and prevents the E-chip windows from rippling or breaking due to uneven pressure across their surface. A torque setting of 0.1-0.14 lbf-in is recommended, which is set before the system ships from Protochips. In case the torque requires adjustment, the follow the steps below to change the torque settings.

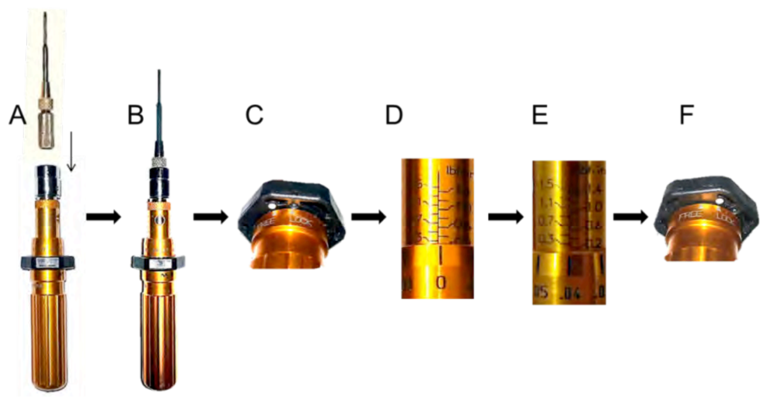


FIGURE 36. SETTING THE TORQUE ON THE TORQUE SCREWDRIVER.

1. Assemble the torque driver by pressing the flathead tip into the driver until it clicks and remains locked in position (Figure 32A-B). This requires strong pressure.
2. To input the torque limit, twist the black plastic ring counter-clockwise so that it is in the "FREE" position, as shown in Figure 32C.
3. Because "0.1" is not listed on the torque scale, first set the torque value to 0.20 lbf-in, as in Figure 32D.
4. Turn the handle counter clockwise six steps to give a final recommended range of 0.1 – 0.14 lbf-in Figure 32E.
5. Finally, set the torque value by rotating the plastic disc clockwise from "FREE" to "LOCK" as in Figure 32F.

6. When leak checking the holder, if the O-rings do not seal reliably for more than one loading, test a slightly higher torque (0.12 to 0.14 lbf-in).

SYSTEM SPECIFICATIONS

Atmosphere 200 Specifications	
Pressure range	3 to 760 Torr (1 atm)
Temperature range	Room temperature to 1000 °C
Manifold/holder tubing and tanks	Electropolished 316 stainless steel (flow restrictor line on holder exit is nickel)
E-chip dimensions	Small E-chip: 2 x 2 mm Large E-chip: 4.5 x 6 mm
E-chip membrane dimensions	Overall membrane size: 300 x 300 μm Heating area: 90 x 90 μm Viewable heating area: 452 μm^2 (9 holes, 9 μm in diameter)
Manifold gas input connections	Three 0.25 inch Swagelok color coded and keyed quick connect fittings (self-sealing)
Internal Manifold O-ring material	Perfluoro-elastomer (FFKM, Kalrez), Viton on the exit ports
Tank volume	Experiment tanks (2): 1 L Vacuum tank: 2.2 L
Manifold line sizes	1/8 inch (3.175 mm) OD, .069 inch (1.75 mm) ID
Line sizes to/from holder	795 um (1/32 inch) OD, 125 um (0.005 inch) ID inlet and outlet, short 50 um ID nickel restrictor on the outlet. All lines are large enough to allow laminar flow.
Fittings and connections from the manifold to the holder	Holder – PEEK unions and ferrules. Manifold – Swagelok quick disconnects with perfluoro-elastomer (FFKM, Kalrez) O-rings
Manifold pressure sensors	4 total pressure sensors. Experiment Tanks and Holder outlet line: 3 gas independent gauges with a full range of 1000 Torr (1.3 bar) and minimum pressure reading of 0.2 Torr (0.27 mbar) Vacuum Tank: A combination of two gas independent gauges in series for a full range of 1000 Torr (1.3 bar) and minimum pressure reading of 2 mTorr (0.0027 mbar)
Holder tubing	795 um (1/32 inch) OD, 125 um (0.005 inch) ID inlet and outlet
Holder tip material	Grade 5 titanium (beryllium lid option for lower background EDS analysis)
Holder connections	2 PEEK unions with ferrule compression fittings and PEEK screws. 1 electrical connection, Mogami cable with 10 pin male Hirose barrel connector
Holder tip O-ring material	Viton, EPDM or perfluoro-elastomer (FFKM, Kalrez)
Control valves	11 total pneumatically controlled solenoid valves
Manifold to holder electrical connection	10-pin Mogami cable with 10 pin female Hirose barrel connector
Flow rate	0.00012 Torr * Liters / second (0.00948 sccm) for nitrogen with standard capillary configuration.
Holder tip volume	1.192 μL
Heating/cooling rate	Up to 5 °C per second
Valve fittings	SC-11

Gas manifold weight	130 kilograms
Gas manifold footprint	0.714 x 0.605 x 1.205 meters (W x L x H) not including boom

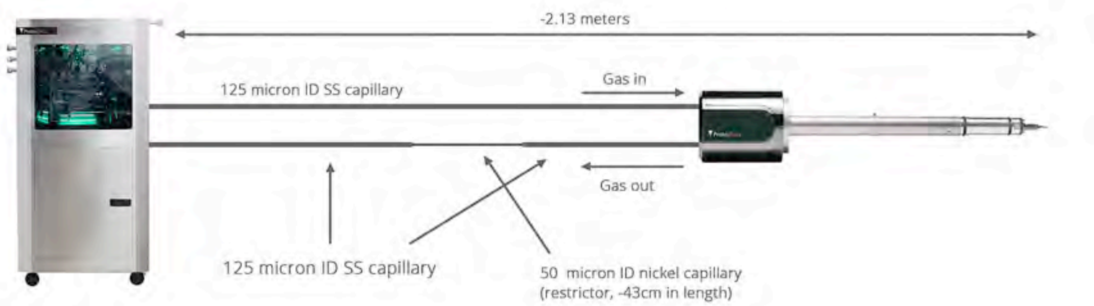


FIGURE 37. MANIFOLD TO HOLDER CAPILLARY CONFIGURATION. THE TOTAL LENGTH FROM THE MANIFOLD TO THE HOLDER TIP IS 2.13 METERS. THE INPUT LINES ARE 125 MICRON ID THE ENTIRE LENGTH. ON THE EXIT PORT THERE IS A 43 CM LONG, 50 MICRON ID NICKEL CAPILLARY THAT ACTS AS A RESTRICTOR. THIS IS THE STANDARD CAPILLARY CONFIGUATION FOR THE ATMOSPHERE SYSTEM.

System Requirements

Gas Handing Manifold Pressure and Electrical Requirements	
Solenoid valves	Air or nitrogen source required, set between 60 and 100 psi (4.2 to 6.9 bar). House air/nitrogen or gas cylinder recommended. Gas entry module has 0.25 inch or 6 mm port for elastomer tube.
Source gas pressure setting	1 to 6 psi (0.07 to 0.4 bar)
Manifold electrical	100-240 V, 50/60 Hz **dedicated** electrical outlet At least 3.6 Amps current (steady state) At least 5.8 Amps with scroll pump on At least 15.1 Amps (inrush required for pump) Note: The vacuum pump must be physically switched to the appropriate source voltage, which is either 100-120 or 220-250 V.
Ground	Shared with the TEM. Less than 1 Ohm and 0 V measured with digital multimeter
MS Windows computer	Electrical outlet for computer and monitor
Gas supply lines from tank to manifold	Electropolished 316 stainless steel lines highly recommended

Gas Handing Manifold Pressure and Electrical Requirements	
Experiment and purge gas purity	Grade 5.5 (99.9995%) or purer highly recommended

- ***Proper grounding is critical for the Atmosphere system. The controller and heater must share the same electrical ground with the TEM. The ground connection between the gas manifold and TEM must be less than 1 Ω of resistance and 0 V as measured with a digital multimeter. If an existing dedicated outlet that meets these requirements is not available, a grounding kit must be used. This consists of a power cable without a ground connection and a separate grounding line that is connected from the gas manifold to a ground point that meets the above requirements. Protochips will supply a grounding kit for the installation if necessary.***

Gas Compatibility

Care should be taken to avoid gases that could react with the materials used to manufacture the Atmosphere system. These materials include titanium holder tip, silicon, silicon nitride, SU-8, gold (E-chips), PEEK compression fittings in the holder handle, Viton/EPDM/FFKM(Kalrez) O-rings and 316 stainless steel tanks and tubing. As of the publication of this document, the gases compatible with Atmosphere are H_2, N_2, O_2, Ar, NH_3 (< 10% concentration), CH_4, COx, Air and mixtures of these gases. Contact Protochips to discuss other gases. Protochips has worked with the electron microscope original equipment manufacturers to develop and approved gas list. That approved gas list is updated from time to time and will be made available to all current customers when it is amended or expanded.

SAMPLE PREPARATION

Suspended Solutions

Samples suspended in solution, such as nanoparticles, nanotubes and other nanomaterials can be loaded using a small volume pipet (0.5-10 µL). Common liquids used to suspend samples are ethanol, methanol, IPA and water. Other liquids can be used, but liquids that leave a residue behind upon drying (such as acetone) are not recommended.

1. Choose the thermal E-chip(s) to be used for the experiment. Note the serial number of the E-chip(s), so the proper calibration file is used during the experiment.
2. Place the E-chip on the sticky surface of the gel-pak to keep it in place. Make sure the E-chip is membrane side up – do not allow the membranes to contact the sticky gel surface.
3. Dispense 0.5-1 microliter of the sample suspension onto the surface of the E-chips and let the droplet sit for 5 minutes, or until the liquid has evaporated.
4. Excess liquid can be removed from the E-chip either by wicking it off with filter or lens paper or by removing it with the pipet.

   - ***If the sample is a powder deposited from a solution or deposited dry and placed on the small thermal E-chip, be sure to clean the gold contacts. If the sample collects on the contact, it could result in an open circuit, and result in a failed device check. Use a small bamboo or wooden stick to gently scrape away excess sample under a stereoscope, as shown in the image below.***

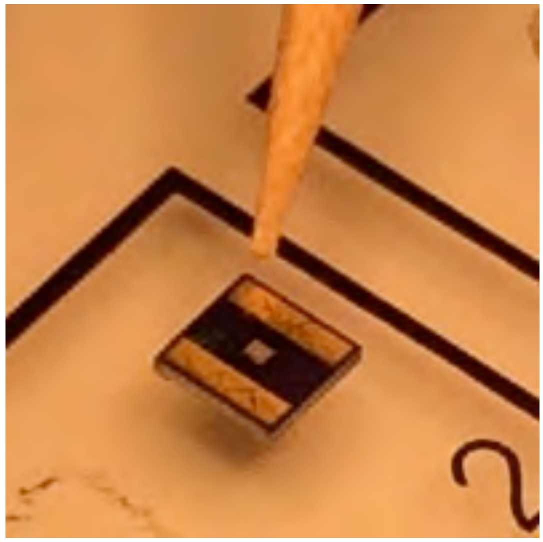


FIGURE 38. A BAMBOO STICK CAN BE USED TO GENTLY SCRAPE AWAY EXCESS SAMPLE FROM THE GOLD CONTACTS.

Other Sample Preparation Techniques

Sample preparation using a focused ion beam (FIB) tool, thin film deposition, and other growth techniques are used. Consult the sample preparation notes for help and tips on these techniques.

FREQUENTLY ASKED QUESTIONS

Q: What gases are safe to use with Atmosphere?
A: A chemical compatibility guide is available, but please note this is not an exhaustive list. Please contact Protochips if you are interested in a gas type not on this list.

Q: What type of samples can I image with Atmosphere?
A: Many types of samples and processes can be imaged using Atmosphere. Examples include: nanoparticles, nanowires, FIB prepared samples, nano-carbons, catalytic reactions, particle coalescence, growth and corrosion.

Q: How large of a sample can I image?
A: Samples must be electron transparent, so sample thicknesses must be less than around 200-300 nm. Samples should be less than around 5 microns in length and width. Keep in mind that the cell thickness is 5 microns, so samples must be thinner than this. Thicker samples may result in punctured windows.

Q: What is the gas volume in the holder tip?
A: 1.192 µL is the volume in the holder tip. The volume in between the chips is determined by the spacer, between 5 and 11 µm, dependent on the pressure and temperature of the gas and E-chips.

Q: What is the thickness of the membranes?
A: The window E-chip membrane is SiN and 50 nm thick. The thermal E-chip has a SiN membrane that is 30 nm thick on top of the ceramic heating material, which is about 120 nm thick.

Q: Can I sonicate the E-chips to clean them?
A: No. Sonicating the E-chips will result in rupture of the thin membranes.

Q: Can I reuse the E-chips?
A: In order to ensure reliable performance and prevent cross-contamination of samples, E-chips should not be reused. However, used E-chips can be saved to further analyze a sample, in the SEM for example, or to demonstrate/practice loading the holder.

Q: What is the gas flow rate?

A: When using the system in Flow mode, the gas flow rate is approximately 1.2e-4 Torr*L/sec. This is based on the standard configuration.

Q: What type of O-ring grease is compatible with Atmosphere?
A: Braycote Micronic lubricant is recommended.

Q: Where can I get replacement screws and O-rings?
A: Contact us at contact@protochips.com for replacement parts.

TROUBLESHOOTING

Sample Loading

Stripped screw heads or brass debris in holder tip area

(1) The brass screws will wear over time. This process will occur more rapidly when using excessive torque. Use the supplied torque screwdriver to avoid over-tightening. 0.11 in-lb/in is the recommended initial value for torque. Replacing worn screws is part of the routine maintenance of the platform.

Leak Check

Manifold has a leak reported by the software

(1) There are two recommended leak checks: one on the holder with E-chips installed, and the other a leak check for the entire system with the holder in a leak check station/goniometer and holder lines connected to the manifold boom. Refer to sections 2.5 and 3.1 for the two leak checks. In addition, verify that the holder lines are fully seated in the PEEK bulkhead connections in the holder and the PEEK nuts are very tight by hand and cannot be turned easily by turning with fingers.

Holder does not maintain vacuum and E-chips are verified intact.

(1) The lid or O-rings are not sealing due to debris such as fibers, debris, excessive grease, or brass shavings from screws. Before loading E-chips, do a thorough inspection and cleaning of tip area and O-rings. Stereoscope and fine tweezers need to be available to allow proper cleaning. Test for leaks in a dry pump station prior to insertion into the microscope. If problems persist, disassemble the tip, clean and replace O-rings.

(2) E-chips are not seated properly in the tip. Make sure the small internal O-ring is seated properly. Load the holder using a stereo microscope and tighten the screws evenly to prevent pressure points from forming during sealing.

(3) Degradation of torr seal. Check torr seal for signs of degradation and if found, return to the factory for repair.

Vacuum

Vacuum crashes when inserting Atmosphere holder into microscope.

(1) Incomplete drying or outgassing of solvent/moisture on exterior of holder. Dry any visible moisture from exterior of holder and plasma clean prior to insertion into microscope.

(2) Insufficient time in the load-lock. Increase the time the holder remains in the load lock position to facilitate complete outgassing of the holder tip.

- JEOL: Wait for the vacuum to fall below 35, or the recommended vacuum level, prior to inserting the holder into the column
- FEI: The load lock time can be adjusted/increased prior to inserting the holder into the column.

❖ **Do not pull the holder out of the load lock then reinsert it to reset the timer. This could cause a vacuum shock to the windows and cause a rupture.**

(3) Not properly sealed prior to insertion. Pump the holder in a pump down station/leak checker until ~1E-6 Torr/mbar vacuum level is achieved.

Poor vacuum recovery between holder insertions

(1) Vacuum recovery can be slowed due to repeated TEM insertion/removal during a short period. If the microscope is configured for cryo, running a 1 hour cryo-cycle may restore vacuum faster. For non-cryo microscopes, remove the Atmosphere holder and allow the vacuum to recover with a standard holder in place.

Membrane rupture

(1) If one of the silicon nitride windows cracks or breaks, the gas in the holder tip will be pulled into the column vacuum. The small volume of gas will not normally cause any damage to the TEM. Typically, the manifold safety system will see a drop in pressure and close the valves. The user can also press "Stop" or the illuminated E-stop button to close the valves. In either case, it is recommended for the user to remove the holder from the column and/or disconnect the quick connects on the boom to minimize the gas pulled into the column. In the event that a window breaks, it is possible that the column pressure could rise

slightly, but a large increase in pressure or a crash is not likely, and this has not been observed during several years of customer and collaborator experience. The holder should be carefully inspected with a stereoscope before further use.

Causes of membrane rupture

(1) Imaging at high intensity for long periods of time with a focused TEM beam, or a stationary STEM probe, can lead to perforation of the membrane. Avoid imaging at high intensity when possible. Use the smallest spot size required to maintain image quality to reduce the overall electron dose.

(2) Membrane rupture could result due to perforation of the membrane by sample (specifically when imaging growth of materials with a whisker structure). Test growth experiments ex situ, followed by a leak check to ascertain if particle growth may compromise the integrity of the membranes.

Imaging

Unable to locate the transparent membrane

(1) Stage is offset such that translation does not reveal transparent area or the illumination is too low. Go to low mag and make sure the beam is defocused (spread out). Be sure that the objective, SAED, and other apertures are out. The condenser aperture should remain in. Track the stage location history with software, and systematically move the stage to locate the window.

(2) Z-height is too high/low making the window out-of-focus. Translate the stage at several different Z-heights. Digital cameras are typically more sensitive than a phosphor screen, so use the camera to locate the window, and then adjust the Z-height until it comes into focus.

(3) Sample is too thick

(4) Film has formed on the outside of the E-chips

Locating and focusing on the sample

(1) Holder is not at eucentric height and window is out of focus. After locating the window, bring the edge of the window into focus. Once the window edge is in focus, increase magnification, adjust the Z-height and scan for samples. Apply fiducials to the membranes to aid in focusing.

(2) Magnification is too high. Reduce magnification and look for window using low magnification mode.

(3) Illumination is too low. Adjust TEM imaging conditions to provide more beam intensity.

Gas is not flowing through the tubing

(4) The tubing may be clogged with sample or kinked so that gas cannot flow through. If a clog occurs prior to an imaging session, insert the holder with no E-chips or O-rings installed into a leak check station with the holder lines attached. Pump down the holder and when the holder is at a low pressure, press the tip of the quick connect. If no pressure increase is seen, the holder inlet line is kinked or clogged. Inspect the tip of the holder for debris in the sample ports in the tip. Inspect the holder lines for kinks and contact Protochips if lines are damaged.