MODEL 1070 PLASMA CLEANER
INSTRUCTION MANUAL

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PN 011-1071 Revision 0

1. Safety Instructions

WARNING! Before proceeding with the installation, please read and understand this manual in its entirety. Failure to perform activities in accordance with this manual could result in physical harm, improper instrument performance, and invalidation of the warranty.

WARNING! When servicing any component of the Model 1070 it is imperative that the mains power is turned off. Following a one-minute period to ensure that any charged electronic component will discharge, disconnect the mains cord from the power entry module located on the back of the instrument. Due to the high voltages associated with plasma processing, failure to follow this procedure could result in a harmful or lethal electrical shock.

WARNING! When performing service that involves energizing either the vacuum system or the high-frequency (HF) power supply, it is imperative that safety glasses be worn at all times. Should a fault condition exist within the quartz tube, there is a possibility of implosion when the vacuum system is energized.

In addition, ultraviolet (UV) light may be emitted from the area of the quartz tube. Prolonged exposure to UV light could result in possible eye damage. Therefore, never look directly at or into the plasma without proper eye protection or without the plasma chamber enclosure secured in place.

WARNING! Should the plasma become energized at any time during the maintenance procedure, extreme care must be taken to avoid contacting the HF antenna. After de-energizing the plasma do not touch the quartz tube. Due to thermal heating during plasma processing, contact with the quartz tube could result in a skin burn.

CAUTION! If the pumping system has pumped harmful substances, determine the nature of the hazard and take appropriate safety precautions before performing any vacuum system service.

WARNING! Use extreme caution when working with, connecting, disconnecting and opening compressed gas cylinders. Serious injury or death can occur. If you are unfamiliar with safe handling procedures seek the assistance of a qualified individual.

2. Introduction

The Model 1070 Plasma Cleaner is an indispensable tool for eliminating contamination from electron microscopy specimens. It applies a low-energy, radio-frequency (RF) plasma that effectively removes hydrocarbon contamination from all materials research specimens. The specimen's surface is effectively cleaned *without* changing its elemental composition or structural characteristics. An ultra-clean, oil-free vacuum system ensures optimal specimen cleaning. The Model 1070 readily accepts side-entry specimen holders for all currently available commercial transmission electron microscopes (TEMs), as well as SEM specimens, specimen clamping rings, and other electron microscope accessories. User controllable gas flow, gas mixtures, power settings and automated RF matching provide excellent cleaning with very short cycle times.

2.1 Background

For optimal imaging and microanalysis in electron microscopy, it is imperative to have a clean, well-prepared specimen. This is especially true for many modern electron microscopes, which employ high brightness electron sources, such as LaB_6 filaments and field emission gun (FEG) sources. Such high brightness systems combine a small electron probe for microanalysis with increased beam current density, giving high resolution specimen imaging as well as enhanced analytical data. However, specimen contamination rates tend to increase as probe size decreases and beam current density increases. This places greater importance on the quality of the specimen and the cleanliness of both the specimen and the specimen holder. The Model 1070 Plasma Cleaner effectively removes hydrocarbon contamination from materials research specimens and specimen holders.

To effectively analyze TEM/SEM specimens, it is essential to prepare specimens without significantly altering their microstructure or composition. Current specimen preparation techniques often introduce amorphous damage, specimen contamination, or both. Contamination may also occur in several other ways, including: inadvertent touching of specimens or specimen holders, back-streaming of oil from an oil diffusion pumped ion milling system, EM column contamination and application of adhesives and/or solvents in the preparation process. Even when great care is taken to clean the specimen, standard cleaning methods are often not 100% successful.

In contrast, the Model 1070 enables specimens to be cleaned immediately before insertion into the electron microscope. It applies a low-energy, RF plasma that effectively cleans a specimen's surface without changing its elemental composition or structural characteristics. The Model 1070 readily accepts side-entry specimen holders for all commercial TEM's and can accept bulk specimens for cleaning before SEM imaging and analysis.

3. Cleaning Theory

In a non-equilibrium, high-frequency plasma, free electrons are accelerated to high velocities by an oscillating electromagnetic field that excites gas atoms and creates the plasma. This slightly increases the temperature of the material being processed, typically by a few degrees above ambient temperature. To optimize cleaning, a mixture of 25% oxygen and 75% argon is generally recommended, but other gases and mixtures are possible. Oxygen plasma is highly effective in removing hydrocarbon contamination. The plasma process creates disassociated Oxygen which combines chemically with the carbonaceous material on the specimen and specimen holder. This reaction chemically reduces the organic contamination to H_2O, CO, and CO_2 that are evacuated by the vacuum system. Complete cleaning of highly-contaminated specimens can occur in 2 minutes or less.

The impinging plasma ions impact the surface with energies of less than 15eV. This energy level is below the sputtering threshold for materials research specimens; therefore, organic contamination is successfully removed without altering the specimen's properties.

3.1 TEM Specimen Preparation

Prior to plasma processing it is necessary to ensure the integrity of the specimen. Proper specimen preparation procedures always need to be applied. When conducting any form of specimen preparation it is essential to utilize optimal preparation techniques. Different materials research specimens require the utilization of differing types of preparation procedures in order to obtain optimum results. For most metals electropolishing is a time-proven technique for producing high quality, TEM-ready specimens. Ion milling can assist electropolishing by further thinning the specimen. For composites, ceramics, and semiconductors, which are not easily prepared by electropolishing, a combination of mechanical pre-thinning and ion milling is an excellent means to obtain a TEM specimen.

Prior to commencing any procedure, it is important to select which means of preparation is most suitable. It is also important to consider what type of TEM analysis is required. For example, when analyzing a metal specimen for stress related dislocations, it is imperative that the preparation procedure does not contribute to the dislocations. In this case, extra care must be given when mechanically thinning the specimen to avoid the introduction of artifacts in the specimen.

It is important to remember that no single technique is suitable for all types of materials. Ion milling is an excellent final specimen preparation technique. However, because the energetic ion beam may induce artifacts in the specimen, it is advantageous to pre-thin the specimen to minimize the duration of ion milling.

Plasma processing is a final operation that can be applied to most types of materials research specimens. Plasma processing will not, however, significantly improve the results of an ill-prepared specimen.

3.2 SEM Specimen Preparation

Preparation of specimens for imaging and analysis by SEM follow protocols which depend on the particular material, its mechanical and chemical properties, and application. Plan view or cross-sectional (XSEM) specimens of bulk materials are produced by first cleaving or mechanical sectioning. Mechanical grinding, chemical–mechanical polishing (CMP), or electropolishing are often used to enhance the surface to be studied. Remaining physical and chemical artifacts can then be removed by low incident angle, low energy argon ion milling. Representative topography can be raised by applying a second gaseous etch using a reactive gas (RIE, RIBE) rather than argon.

Whether an “as received” or prepared surface is examined, plasma cleaning to remove residual contamination is highly recommended. The size, shape, porosity, and configuration (embedded in epoxy or mounted on carbon adhesive) of specimens promote retention of absorbed water, chemical residues and physical debris. Plasma cleaning promotes enhanced imaging, especially at low accelerating voltages whereby surface contamination significantly diminishes both image quality and the accuracy of analytical data. Electron back scatter diffraction (EBSD) or standard chemical analysis (EDX, WDX) of a surface is optimal once carbonaceous contaminants are removed.

4. Installation and Setup

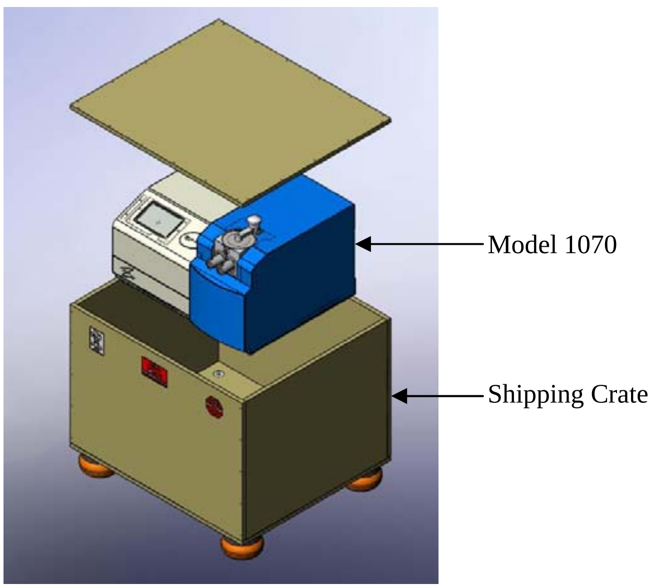
Before installing the Model 1070 Plasma Cleaner ensure that the location chosen has been suitably prepared to fulfill the pre-installation site requirements.

Immediately upon receipt, carefully inspect the shipping container for signs of damage. If evidence of damage exists notify both the shipper and the supplier immediately.

NOTE: The weight of the system is approximately 160 lb (73kg). Under no circumstances should removal from the shipping crate, lifting, or positioning of the instrument be attempted by one person. To conduct any movement, firmly grasp the instrument from the bottom base plate only.

4.1 Uncrating

If the shipment has been received in good condition, proceed with unpacking.



Shipping and Packing Assembly

- Remove the top of the crate.
- Remove the accessory carton.
- Remove the top layer of Foam-In-Place material
- The instrument can now be removed by lifting from the bottom base plate. This should be done by two individuals that are accustomed to lifting heavy objects.
- Place the instrument on a firm surface, where it can be accessed from front and back.
- Remove the instrument from the plastic shipping bag.
- Replace the Foam-In-Place material, accessory carton, and plastic bag in the crate, and close it.
- Place the instrument on a firm surface where it is to be operated.

The shipping crate should be saved for re-use in the event that the instrument needs to be returned to the factory for repair or maintenance.

4.2 Visual Inspection

A complete inspection of the Model 1070 should begin by examining the outside of the instrument for any damage. Should evidence of damage exist, contact both the freight carrier and the supplier immediately.

WARNING! The Model 1070 contains a high voltage power supply which provides electrical potentials that could result in a harmful or lethal electric shock.

DO NOT CONNECT THE MAINS CORD TO THE INSTRUMENT DURING THE INSPECTION PROCEDURE.

Only connect the mains cord when the entire instrument enclosure is closed and fully secured.

4.3 Making the Gas and Exhaust Connections

4.3.1 Making the Process Gas Connection

The M1070 uses 3 independent gas paths. A variety of gases can be connected to fulfill the needs of the customer. Typically, for the purposes of plasma processing it is recommended to utilize a 25% oxygen/ 75% argon mixture (± 2%) with a grade in excess of 99.998% purity. Individual Argon and Oxygen supplies can be connected to two of the 3 gas inputs on the back panel of the instrument. These should be 99.998% pure or better. Alternatively, a pre-mixed bottle of 25% Oxygen – balance Argon can be purchased locally and is contained in a single cylinder. A third input is available for customer requirements.

For each of the Process Gas Inputs:

- Position the process gas supply so that it is located within five feet of the Model 1070.
- Mount a suitable pressure regulator (Victor HPT 270 or equivalent) onto the process gas supply.
- Remove the cap covering the gas inlet to the M1070. This cap should be saved for re-use should the instrument need to be shipped again.
- Connect the 0.25 inch tubing (supplied) between the pressure regulator on the process gas supply and the Gas Inlet connection on the rear of the Model 1070. Tighten the connection 1¼ turns after finger tight.
- Observing proper safety precautions, open both the cylinder and the process gas supply valves. Adjust the cylinder pressure regulator to a pressure of 15 psig (103kPa).
- Loosen the fitting on the back of the Model 1070 until gas can be heard escaping through the fitting. Wait for several seconds, then retighten the fitting 1¼ turns after finger tight. This will purge the delivery line of atmospheric gas and fill the line with the process gas.
- Close the process gas cylinder valve and observe the cylinder pressure regulator gauge. The indicated pressure should remain constant for a minimum of fifteen minutes. This action ensures that the process gas connection is properly sealed.
- Open the process gas cylinder valve and it is ready for service.

4.3.2 Making the Control Gas Connection

The M1070 uses a control gas to operate the gate valve. A variety of compressed gas sources can be used for this purpose. High purity gas is not a requirement for this input. A clean dry source of Nitrogen, Argon, Air, or any other available, safe gas can be used for this application.

For the Control Gas Input:

- Position the control gas supply so that it is located within five feet of the Model 1070.
- Mount a suitable pressure regulator (Victor HPT 270 or equivalent) onto the control gas supply.
- Remove the cap covering the gas inlet to the M1070. This cap should be saved for re-use should the instrument need to be shipped again.
- Connect the 0.25 inch tubing (supplied) between the pressure regulator on the control gas supply and the Gas Inlet connection on the rear of the Model 1070. Tighten the connection 1¼ turns after finger tight.
- Observing proper safety precautions open both the cylinder and the process gas supply valves. Adjust the cylinder pressure regulator to a pressure of 60 psig (414kPa).
- Close the control gas cylinder valve and observe the cylinder pressure regulator gauge. The indicated pressure should remain constant for a minimum of five minutes. This action ensures that the control gas connection is properly sealed.
- Open the control gas cylinder valve.

WARNING! If hazardous materials will be inserted into the Model 1070 for processing it is imperative that the unit be connected directly to an exhaust system capable of accommodating the hazardous material. Use of gas tubing and fittings other than those specified is not recommended and will void the warranty.

4.3.3 Making the Exhaust Gas Connection

Connect the Model 1070's Exhaust connection located on the rear of the unit to a suitable exhaust system. Any fittings required should be obtained locally. It is recommended to utilize at least a 0.5inch (12mm) diameter exhaust duct to avoid restricting the flow of exhaust gas. It is also recommended to keep the length of 0.25 inch diameter tubing that connects the Model 1070 to the external exhaust system as short as possible.

4.4 Unbolting the Diaphragm Pump

In order to minimize instrument vibration during operation, the diaphragm pump is supported by a vibration isolation mechanism. To avoid damage to this mechanism during shipment, the diaphragm pump is secured to the base plate with a single “wing nut” bolt.

- Using wooden blocks (not provided) raise the left side of the unit approximately 10 cm above the tabletop.
- Reach under the front of the instrument and completely unthread the wing nut. Keep the wing nut with the system in the event the system will be shipped again.
- Remove the wooden blocks and ensure that the unit sits firmly on the tabletop.

4.5 Mains Connection

The power entry module of the Model 1070 has been configured to operate based upon the operating voltage of the country from the original purchase order. If the Model 1070 is to be used under other power conditions, please consult the factory.
For US Applications: Use the supplied line cord.

For Non-US Applications: Attach a supplied line cord to a suitable mains connector that matches the local receptacles. The color code of the wires in the supplied mains line cord are:

Blue	=	Neutral	(0V)
Brown	=	Phase	(Line)
Green	=	Ground	(earth)

Once the proper operating voltage has been verified, connect the mains cord between the power entry module and a suitable receptacle. The Model 1070 is now ready for operation. Power up and operation will be discussed later in the manual.

5. System Overview

Refer to the Model 1070 internal view and chamber cross-section view on the following page.

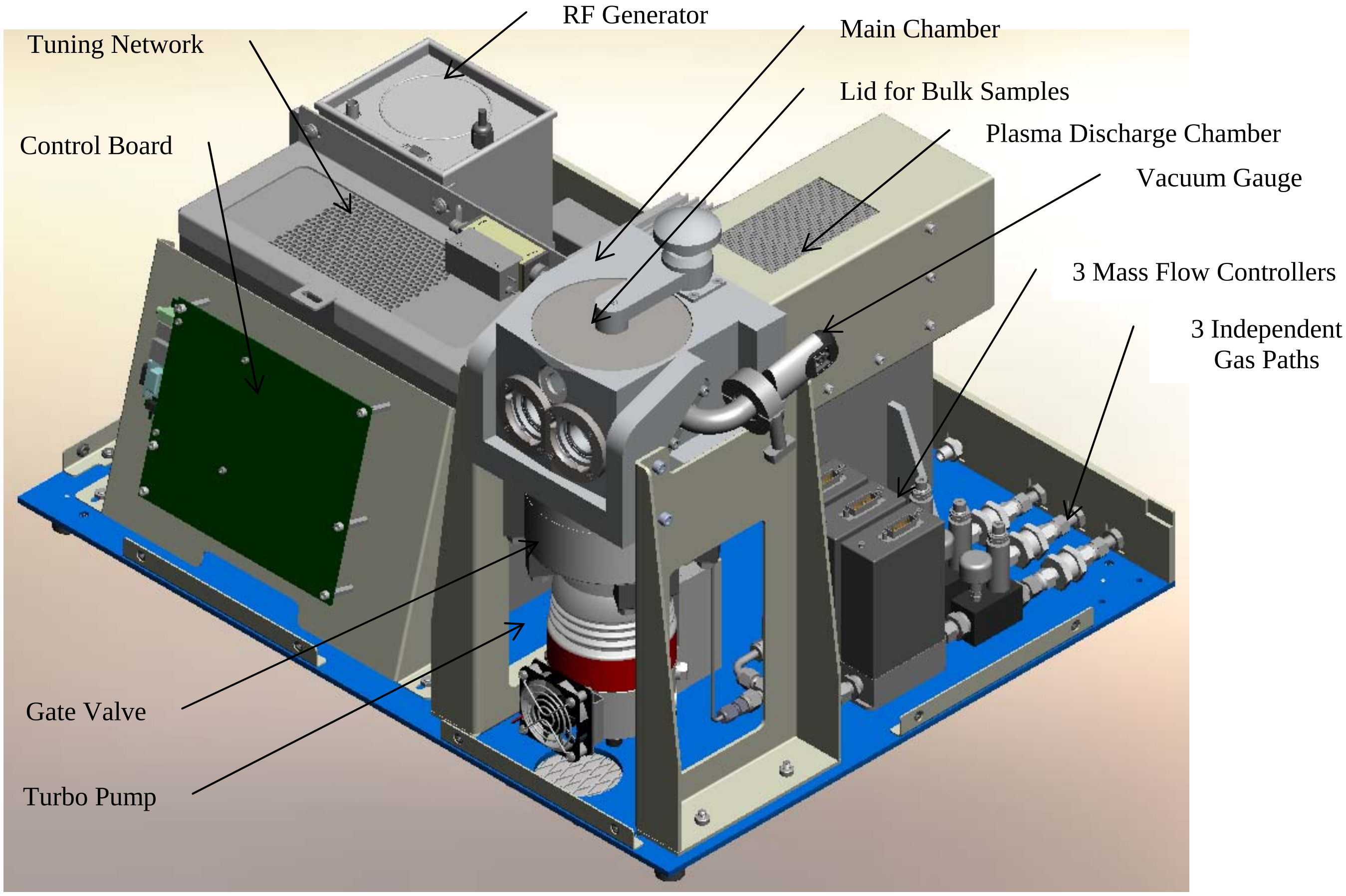
5.1 Plasma (Specimen) Chamber

The plasma chamber has two main areas. A plasma discharge is created in the area enclosed by the quartz tube (toward the rear of the instrument). The specimen and specimen holder are downstream from the plasma in the main chamber. Sophisticated gas dynamics ensure an even distribution of plasma within the chamber, enabling effective cleaning with negligible heating. A high-frequency (HF) antenna, located outside the chamber, inductively couples the oscillating field to the process gas and permits plasma processing. No components are located within the plasma chamber, other than the specimen and specimen holder being cleaned.

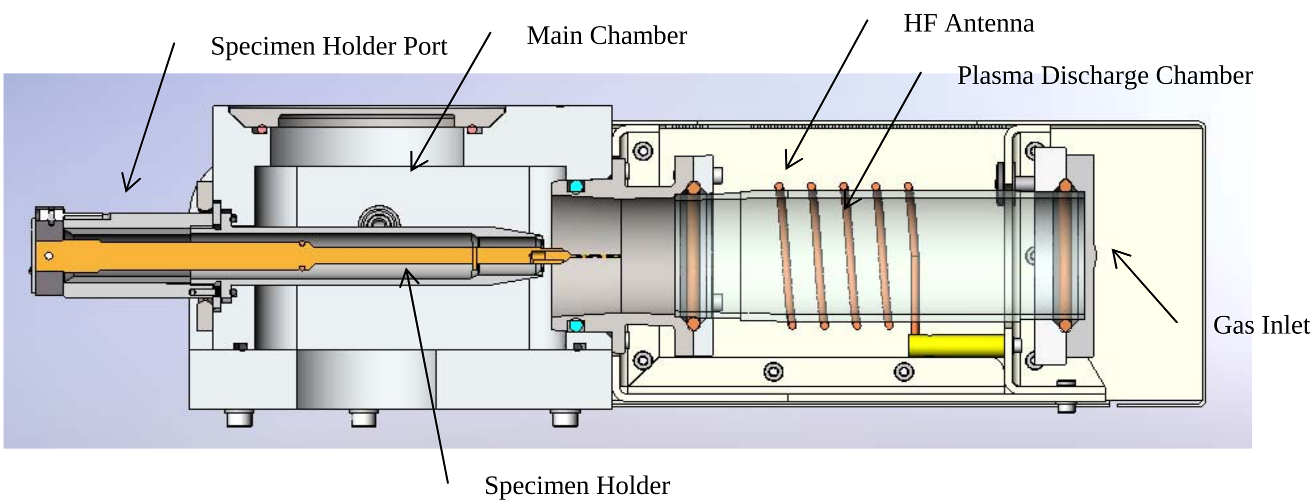
The plasma chamber's configuration makes it possible to clean specimen holders used with any commercially-available TEM, as well as SEM specimens, and other electron microscopy accessories.

O-ring seals ensure the vacuum integrity of the various chamber components. Located to the right of the main chamber is a KF16 flange that serves as the port for the vacuum gauge. Beneath the chamber is a gate valve that is used to isolate the turbo-molecular drag pump from the chamber. When the gate valve is closed the chamber is isolated from the pumping system. When the gate valve is open, the pumping system will work to evacuate the plasma chamber. The front surface of the main housing contains two bores which serve as receptacles for the various Specimen Holder ports.

As depicted in the cross-section view, the Specimen Holder port accepts standard side entry TEM specimen holders. The O-ring contained on the specimen holder is used to maintain the vacuum seal. In this configuration, the specimen and the specimen holder are simultaneously cleaned. The plasma process treats all surfaces, in front of the O-ring seal, which are exposed to the vacuum in the microscope.



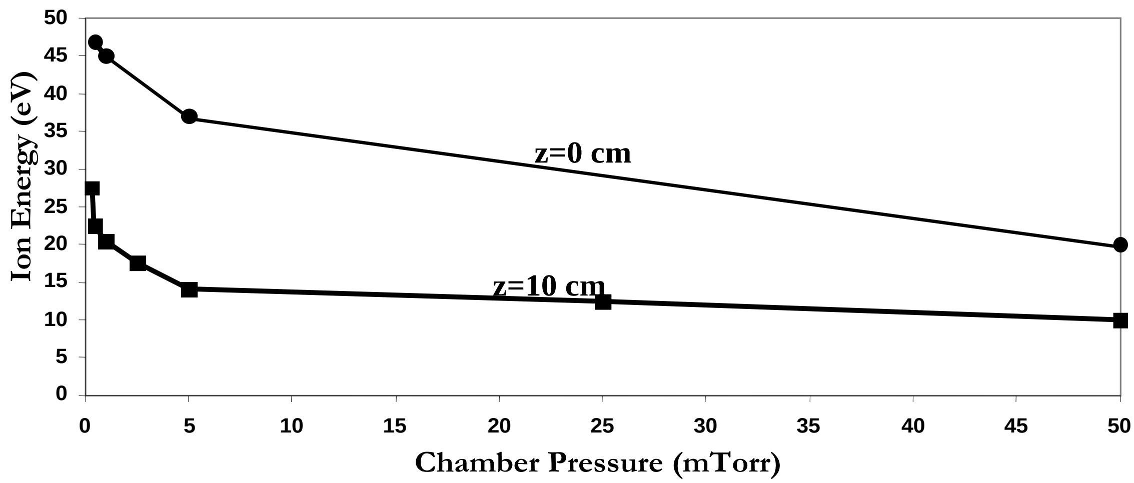
Model 1070 Internal Components



Cross-section of Plasma Chamber

5.2 RF System

The RF power system is composed of several items. It is based on a 50 Watt solid-state RF generator running at 13.56 MHz. Output from the generator is monitored by an RF sensor and the resulting forward and reflected power readings are available on the user interface. A tuning network is used to match the RF generator to the HF antenna and gas system. The tuning network will adjust as the plasma is lit and the load changes. It will adjust to achieve the lowest possible reflected power for the current operating conditions.



Ion Energies as a function of chamber pressure and specimen position

Factors contributing to the ion energies are the gas pressure within the plasma chamber, the position of the specimen in proximity to the HF antenna, as well as the delivered power from the RF generator. With the specimen and specimen holder at the plasma cleaning position, and with typical process gas pressures of 5-50mtorr, low ion energies in the range of 10-15eV are attained. The above figure indicates the correlation of ion energy at the specimen with respect to plasma chamber pressure, where the typical specimen position for plasma cleaning is 10 cm (z=10 on the figure) from the center of the antenna coil. As a function of the inductively coupled plasma creation, the ion energies in the center of the HF antenna (z=0) are 30-45eV. The ion energy graph shown above is based on the RF generator running at 100%. Power can be adjusted by the user to further reduce ion energies if that is desired.

5.3 Vacuum System

An oil-free vacuum system is essential to ensure optimal cleaning. The Model 1070 incorporates an oil-free vacuum system consisting of a turbo-molecular drag pump backed by a diaphragm pump. The pumping system provides appropriate vacuum levels to ensure optimum performance. Since the plasma chamber is isolated via a gate valve, the turbomolecular pump will remain at operational speed and specimens can be exchanged very quickly. Typically, it takes 5 seconds to activate the gate valve and vent the plasma chamber. Once the sample is exchanged, it takes less than 45 seconds to pump down the plasma chamber and open the gate valve. Plasma chamber vacuum is sensed by a Pirani gauge.

5.3.1 Turbo-Molecular Drag Pump (TMP)

The Model 1070 is equipped with a 67 liters/second turbomolecular drag pump. With no gas flow, the ultimate plasma chamber vacuum is $\sim 10^{-6}$torr. Under normal plasma processing conditions, the system vacuum operates at in the range of 20 to 100 mTorr.

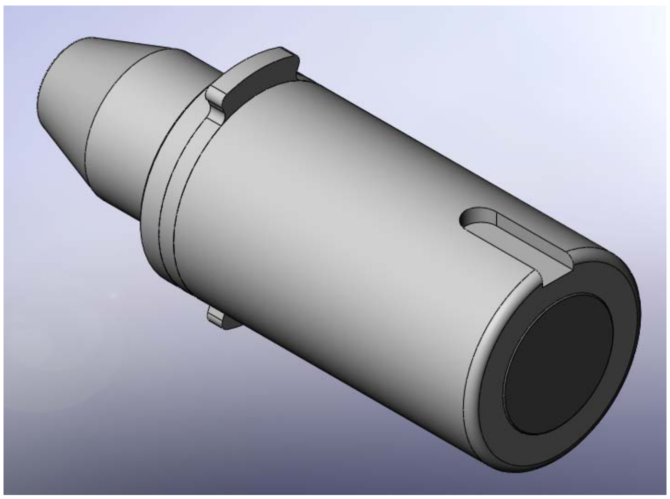
The TMP is equipped with an inlet protection screen to prevent any foreign objects from entering the gate valve or pump and damaging the turbine blades.

5.3.2 Diaphragm Pump

The Model 1070 utilizes a two-stage, oil-free diaphragm pump with a pumping speed of 20 liters/minute (at atmosphere). The diaphragm pump is supported by four vibration- isolated mounts.

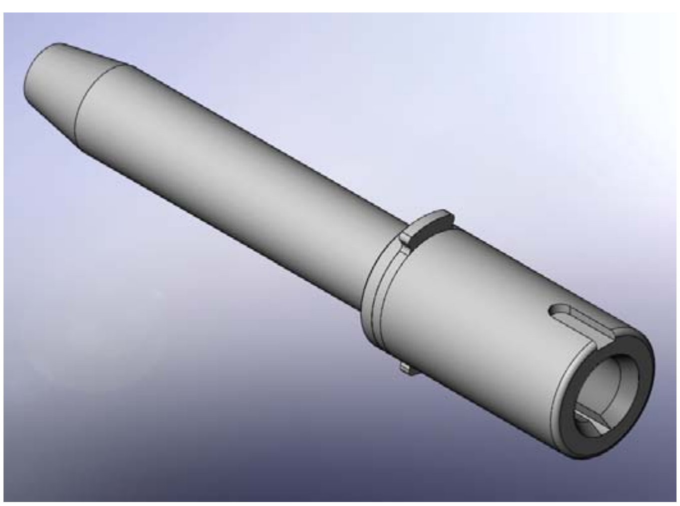
5.4 TEM Holder Ports, Pumping Ports and Plugs

Various Specimen Holder ports are available to support different manufacturers and models. Each Specimen Holder port has a corresponding plug that can be used when the TEM holder is not in the M1070. Port plugs should be used to seal the specimen chamber when the M1070 is not being used, or to seal the chamber when bulk specimens are inserted for plasma cleaning. No tools are required to insert or remove the Specimen Holder ports and plugs. Specimen Holder ports are secured to the chamber with a bayonet style mechanism. To insert a holder port into the chamber, align the tabs on the port with the slots on the face of the chamber and rotate clockwise 30°. The notch shown in the figure below should be on top once the port adapter is secure in place. An external or "face" seal is created between the chamber port and O-ring contained on the M1070's main housing. The Port Plug is pushed into the opening of the Specimen Holder port. The O-ring on the front of the plug will make seal against the inside surface of the port adapter.



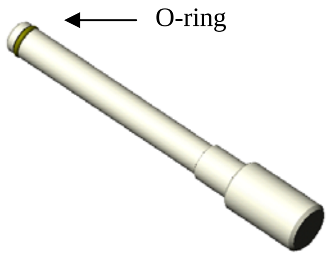
Specimen Holder Port

A single side-entry TEM specimen holder or aperture rod is inserted through a Specimen Holder port into the specimen chamber. The M1070 will accept any combination of two Specimen Holder ports or chamber plugs. Specimen Holder ports are available for side-entry specimen holders used with FEI, JEOL, Hitachi, and Zeiss microscopes. These Specimen Holder ports are easily interchangeable.



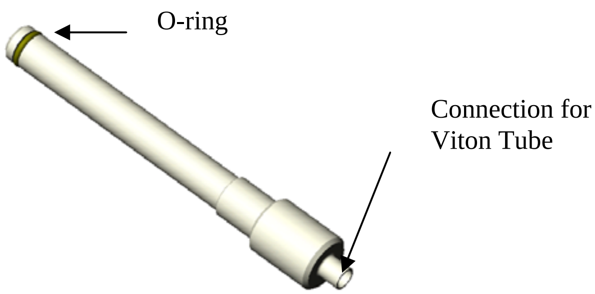
Specimen Holder Port

A port plug is available to seal the Specimen Holder port to isolate the vacuum chamber from atmosphere when a side-entry TEM holder is not inserted.



Port Plug

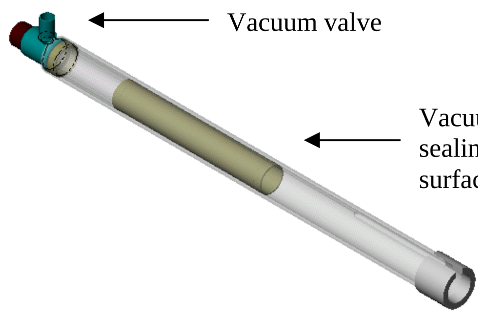
A vacuum pumping port is available to connect a Model 9010 Vacuum Storage Container or Model 9020 Vacuum Pumping Station to the M1070. See the section on Optional Vacuum Storage Accessories for a description of the Model 9010 and Model 9020. One end of a Viton tube is fitted onto the pumping port while the other mates with the vacuum storage container's valve. Once the connection exists and with the valve stem pulled in the outward direction, a particular TEM holder is inserted into either the Model 9010 or Model 9020. The vacuum system of the Model 1070 is then used to evacuate the storage container. By closing the valve, the specimen holder remains under vacuum during storage.



Pumping Port

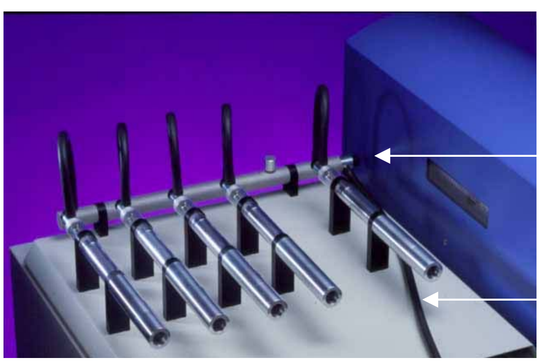
5.5 Optional Vacuum Storage Accessories

After plasma cleaning, TEM specimen holders (and specimens) can be inserted into optional containers (Model 9010 Vacuum Storage Container or Model 9020 Vacuum Pumping Station) for storage in a clean, vacuum environment. A sight glass allows observation of the holder tip in the Model 9010 Vacuum Storage Container. The Model 9010 facilitates either storage or transportation of TEM specimen holders. A vacuum is created within the storage container once a temporary connection is made to the Model 1070's specimen chamber. Contact Fischione Instruments for assistance in selecting the appropriate accessories to match the corresponding Model 9010 with given Specimen Holders ports.



Model 9010 Vacuum Storage Container

For the simultaneous storage of multiple specimen holders, the Model 9020 Vacuum Pumping Station is utilized. It includes a heavy duty metal base with non-skid feet, five vacuum storage containers, a vacuum pumping manifold, and all necessary fittings and valves for connection to the Model 1070. The Vacuum Storage Containers utilized on the Model 9020 employs individual valves so that single specimen holders can be inserted and removed from the Vacuum Pumping Station without affecting the vacuum in the remaining containers.



Model 9020 Vacuum Pumping Station - installed on top surface of Model 1070

5.6 Power Sources

5.6.1 Electrical Power

Under normal operating conditions the power consumption of the Model 1070 is approximately 660 watts. Fuses located within the power entry module provide system protection. The system can be configured for mains voltages of 100 – 120 VAC or 220 - 240 VAC.

5.6.2 Electronic Circuitry

The Model 1070 is controlled by one printed circuit board (PCB). All control and monitoring is done from this board. An embedded computer is connected to the control board and a touch screen display is used for the user interface. All user interaction is with the touch screen.

5.6.3 RF Power Supply

The Model 1070 contains a high-frequency oscillating power supply that initiates and sustains a low-energy plasma. An auto-tuning auto-matching network ensures effective coupling of the high-frequency field to the plasma chamber. This also ensures compatibility with specimen holders produced for diverse EM applications.

The instrument is shielded to prevent the emission of high-frequency interference.

5.7 Plasma Gas System

The Model 1070 has 3 independent gas paths. A variety of pure or pre-mixed gases can be connected to the instrument. To optimize cleaning a mixture of Oxygen and Argon is generally recommended. Additionally, Hydrogen and Oxygen can be used if the user prefers. Oxygen plasma is highly effective in removing hydrocarbon contamination. This reaction process yields H_2O, CO, and CO_2 that are evacuated by the vacuum system. Complete cleaning of highly-contaminated specimens can occur in 2 minutes or less. Users can select the mixture to suit their application by connecting pure Argon and pure Oxygen to the system and entering the desired mix. Additionally, pre-mixed 75% Argon - 25% Oxygen can be utilized.

5.7.1 Process Gas

The Model 1070 contains a microprocessor controlled process gas system. Process gas is connected to a bulkhead fittings located on the rear of the instrument. The purity of gas should be ≥99.998% and supplied at a pressure of ~15psig (103kPa).

5.7.2 Gas Flow

Internal pressure regulators are preset to provide the proper supply pressure to the mass flow controllers. The mass flow controllers will provide the correct proportion of each gas into the mixing manifold. The mixed gas is then delivered to the plasma chamber.

5.8 Air Flow

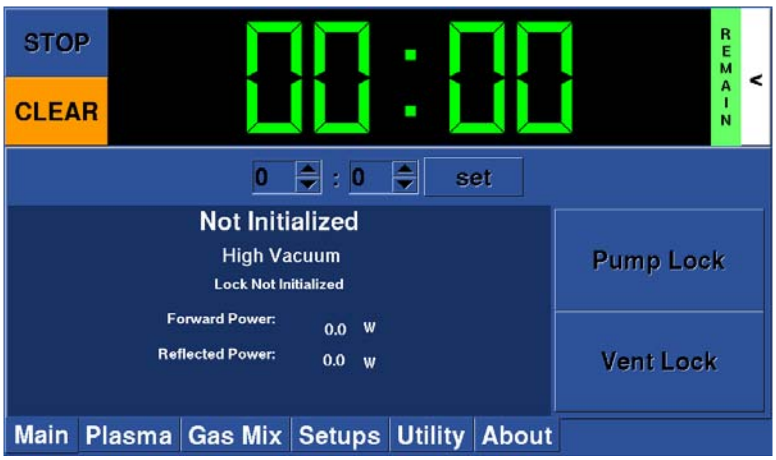
Air inlets are located on the base plate of the Model 1070. Cooling fans mounted on the instrument’s rear panel ensure proper air flow throughout the enclosure to reduce any heat build-up.

6. Operating Interface, Operations and Procedures

6.1 Power Up

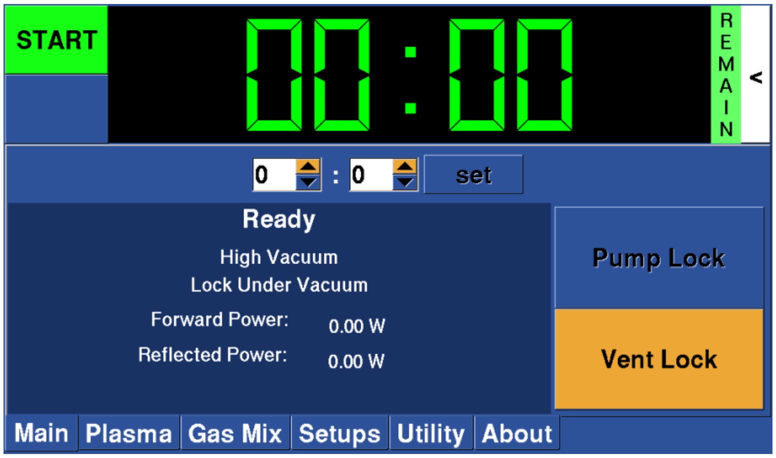
Verify the control gas and process gas connections have been made in accordance with local regulation and as described earlier in the manual. Verify the line cord is connected to the appropriate voltage source as described earlier in the manual.

Apply power to the system by turning on the switch on the back-left side of the instrument next to where the line cord is attached. The front panel will light up and begin to display information. You may hear movement inside the instrument as the tuning network is adjusted and initialized. Once the following screen is displayed, the system is ready for user input. At this point, the vacuum system is not running. Pressing the “Clear” button will initialize the vacuum system, pump the main chamber, open the gate valve and start the turbo pump.



6.2 Instrument Interface

The instrument is operated via the touch screen located on the front panel.



Common Display

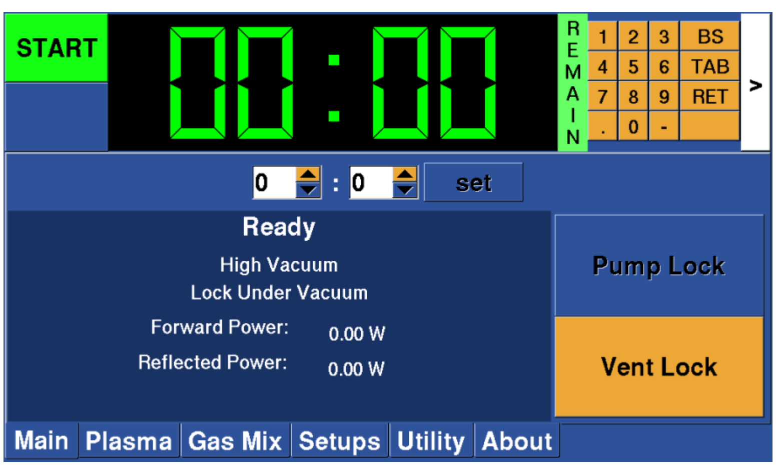
Tab Specific Display

Main Tab

The User Interface is via a 7” touch screen computer that is mounted on the front cover of the instrument. The screen is divided into two portions, with the common part (the top) of the display visible at all times, and the tab specific part (the bottom) of the display changes depending on the tab that is being displayed.

6.3 Common Display

The common part of the screen consists of the start, stop, and clear buttons in the upper left corner, followed by the time display (in minutes and seconds). Next to the time display is a vertical bar displaying either “Remain” or “Elasped”. If “Remain” is displayed, the time represents how much time is remaining in the current cleaning process. If “Elapsed” is displayed, the time represents how much time has elapsed in this cleaning process. To toggle between the two displays, just touch the vertical bar displaying the word. The last item on the common screen is the “<” sign in the upper right corner of the touch screen. Touching this symbol will reconfigure the common screen to include a numeric keypad. This keypad will allow numeric entry into any field on the screen that has the input focus. To hide the keypad, touch the “>” sign.



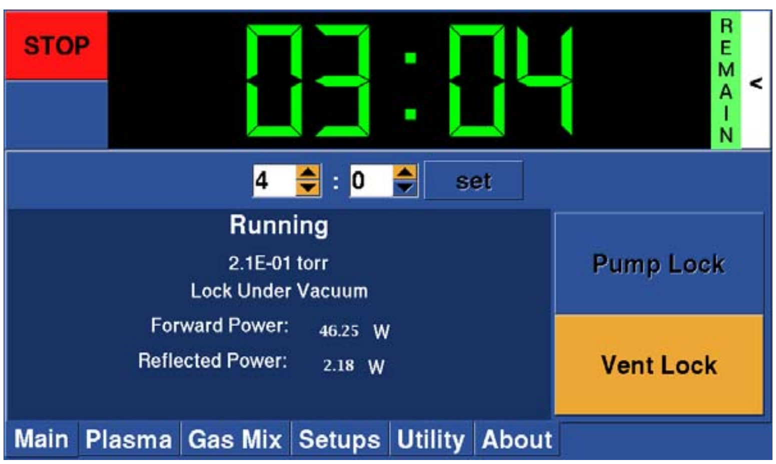
Main Tab with Keypad Displayed

6.4 Main Tab

All items below the common display are dependent on the tab that is being displayed. For the “Main” tab, the time entry is show as two input fields with up and down arrows. A maximum time of 99 minutes 59 seconds can be entered. The up down arrows can be used to change the numbers or the numeric keypad can be used. If the numeric keypad is used, double tap the minute input and the number will be highlighted. Enter the number on the keypad and press the “TAB” button on the keypad to move to the seconds entry. Enter the seconds and press the “TAB” a second time. Now, touch the “Set” button to set the time and the time will appear in the large display. If the time setting does not appear, verify the word “Remain” is shown next to the time. If “Elapsed” is displayed, the time will not appear since no time has yet elapsed on this process.

The pump and vent load lock are large buttons in the lower right of the Main display. Usually, only one of the two buttons is active. In an error condition, both buttons are available. Verify the chamber is completely plugged (holder ports are installed and holders or plugs are in the holder ports). Pressing the “Pump” button will begin to pump the load lock area of the system. Pressing the “Vent” button will vent the load lock so the sample and holder can be removed from the system.

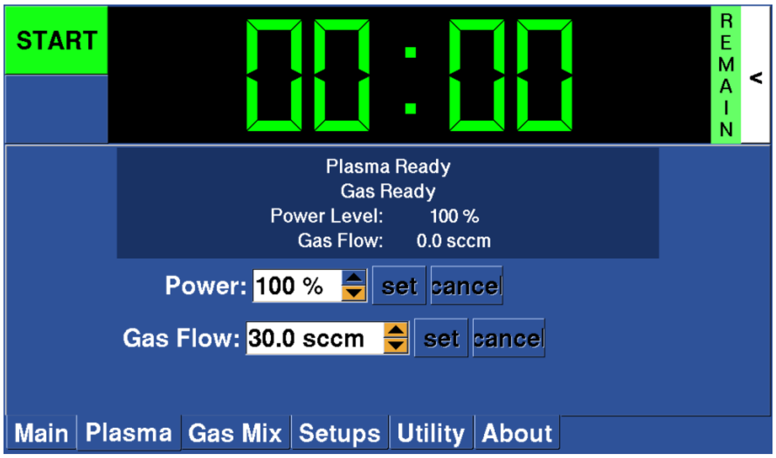
Additionally, there is a status panel that displays the state of the plasma cleaning system, the vacuum system, the load lock, and the delivered power. The top line displays the state of the system. It will display Ready, Preparation, or Running. The 2nd line of the display is the vacuum system. It will display the chamber pressure in Torr (or High Vacuum if the pressure is below the working range of the sensor). The 3rd line is the Load Lock Vacuum. It is either, Atmosphere, Evacuating, Venting, or Under Vacuum. The last 2 lines display the forward and reflected power in watts when the RF system is running.



Main Tab during cleaning process

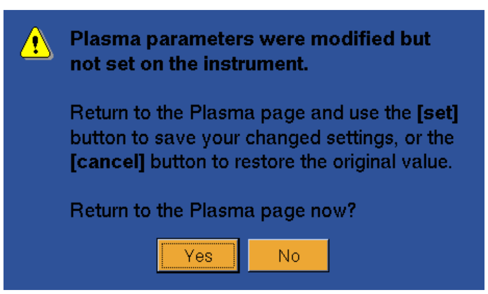
6.5 Plasma Tab

The Plasma tab will display the state of the plasma system, the gas system, the power level and the gas flow. Below the status display are the input fields to set the Power level and the Gas Flow. To set the power level, use the up-down arrows associated with the field, or use the numeric key pad in a similar fashion as described in the time entry on the main tab. When the power level is at the correct level, press the “Set” button. Power can be set in the range of 15 – 100%. Gas flow can be set in the range of 20 – 40 SCCM. The gas flow will have a direct impact on the operating pressure of the plasma. With 75%Ar - 25% O2 and 30 SCCM, the chamber will operate in the range of 20 to 30 mTorr during cleaning. The factory default for this screen is 100% power and 30 SCCM. If changes are made to the power or flow inputs, the corresponding “Set” button must be clicked to register the change.



Plasma Tab

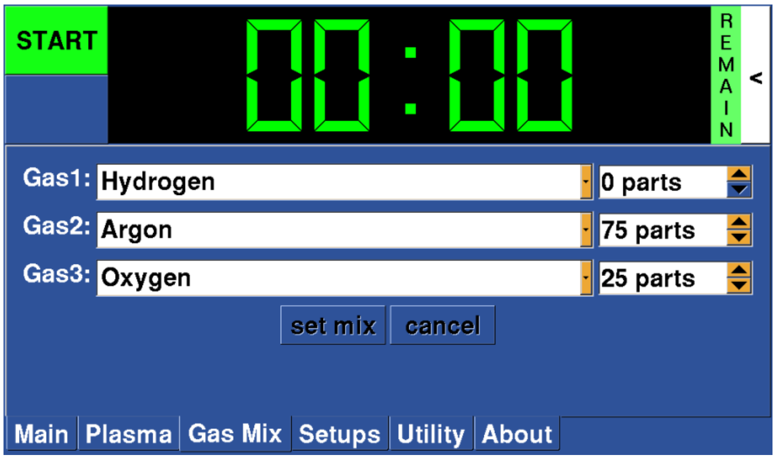
If the user selects another tab with either of the “Set” buttons highlighted, the following warning will appear. It is best to select the “Yes” button and return to the Plasma tab to finalize the settings before moving on.



Warning Prompt

6.6 Gas Mix Tab

The Gas Mix tab will configure how the system is connected to the process gas bottles and also how the gas will be mixed during operation. There are 3 gas fittings on the rear panel of the instrument. Click the drop-down menu for Gas 1 and select the correct gas. Possible choices for each gas connection are Argon, Oxygen, Hydrogen, 75% Ar – 25% O2, or No Selection. Set all 3 gas inputs to match the connections on the rear of the instrument, Contributions of each gas can be changed as needed for differing applications. A general approach to entering contributions for each gas has been used in the M1070 system. Contribution by parts is a more general approach than entering the contribution by percent. Percent can be used if the user chooses to, but that is not the only option. Consider the screen show below.



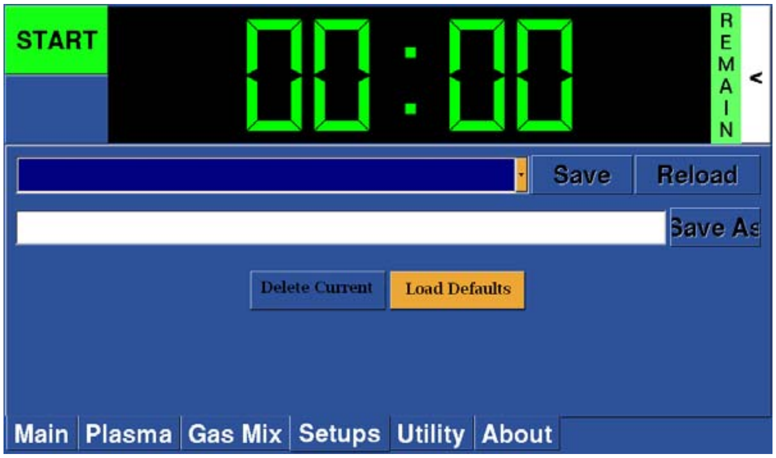
Gas Mix Tab

In the example shown, Hydrogen is connected to the Gas 1 input, but is not being used for the current process. Gas 2 is set to Argon and 75 parts (or 75%) of the mix is based on this input. Gas 3 is set to Oxygen and 25 parts (or 25%) of the mix is based on this input. Another way to get the same mix would be to set Argon to 3 parts and Oxygen to 1 part. Parts and Percent are interchangeable if the total of the numbers equals 100.

Any time a change is made to this screen, the “Set Mix” button will be highlighted. The “Set Mix” button must be clicked for the changes to take effect. If you attempt to move to a different tab before accepting the changes, the system will remind you that there are unsaved changes and ask you if you want to return to the page to make the changes permanent. As with the Plasma tab, it is best to return to this tab and make the changes permanent to avoid later confusion.

6.7 Setups Tab

The Setups tab will allow the user to save and recall configurations. All parameters are saved with the exception of the Cleaning Time that is entered on the main tab. All parameters on the Plasma tab and the Gas Mix tab can be saved and recalled via the Setups.



Setups Tab

To save the current configuration, enter the name in the “Save As” box and click the “Save As button. The numeric keypad is used to enter the name of the setup.

To recall a previously used setup, click the arrow on the drop-down box next to the Save and Reload buttons. All previously stored setups are listed here. Select the desired setup by clicking the correct entry, and then click the “Reload” button. The configuration stored in the setup will become active.

To change a saved configuration, load the configuration as described above. Make the desired changes on the Plasma and Gas Mix tabs. Return to the Setups tab. Verify the setup that was recalled is still the current name next to the “Save” and “Reload” buttons. Click “Save” to make the changes persistent.

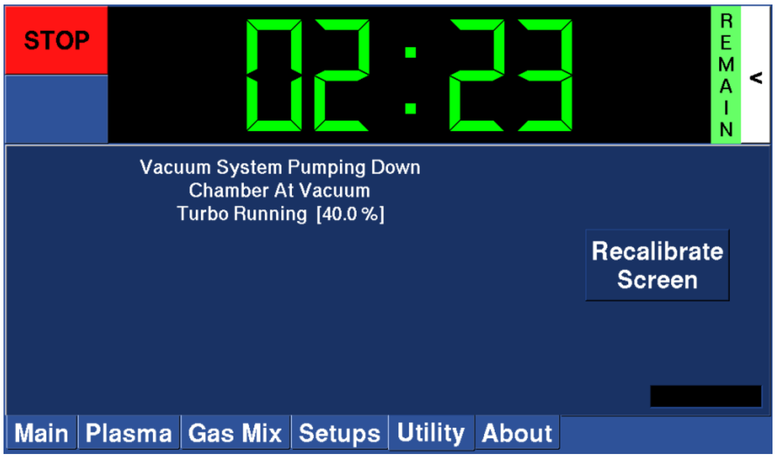
If a setup is no longer needed, you can select the setup from the drop down as described above. Click the “Delete Current” button. The setup will be removed from the persistent storage. The settings on the Plasma and Gas Mix tabs will not be changed, but the setup will be removed.

The factory default configuration can always be restored by clicking on “Load Defaults”.

6.8 Utility Tab

The Utility tab will allow the user to recalibrate the touch screen if that becomes necessary. Additionally, it provides information about the Vacuum System, the vacuum level of the chamber, and the speed of the turbo pump. Normal operating speed of the turbo pump is 40%.

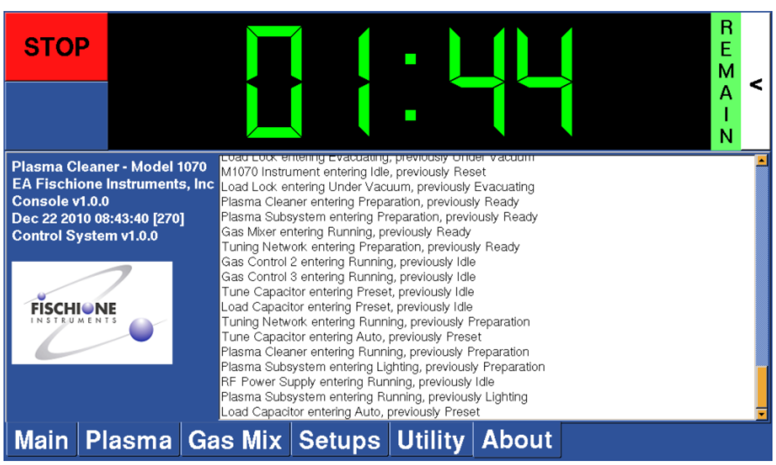
If you are having trouble selecting the item you are touching on the touch screen, it may be necessary to recalibrate the screen. To recalibrate, click on the button. You will be presented with a confirmation screen. Upon acknowledging the confirmation screen, the recalibration will occur the next time the unit is turned on.



Utility Tab

6.9 About Tab

The About tab will provide the user with information specific to the software revision and build dates. There is also a window showing messages based on recent activity.



7. Basic System Operations

Basic system operations will be detailed in the following sections.

7.1 Venting the Plasma Chamber

With the system under vacuum, the chamber can be vented by pressing the “Vent Lock” button on the Main tab. If the system is running a plasma the vent button will be disabled. To vent from this state, the plasma must be shut off by pressing the “Stop” button or waiting until the process completes. Once the vent button is pressed, the gate valve in the system will close and the chamber will be vented. The main display will show the load lock at atmosphere and the pressure displayed will be close to atmosphere (750 Torr).

7.2 Removing a Specimen Holder or Chamber Plug

Removing a specimen holder or chamber plug can only be done when the chamber is vented. If the chamber is not vented, follow the description of how to safely vent the chamber. Once the chamber is vented, the Specimen Holder or Chamber Plug can be removed by gently pulling straight out on the holder or plug. Continue pulling straight out until the holder or plug is completely clear of the Specimen Holder port adapter.

7.3 Removing and Replacing the Bulk Specimen Chamber Lid

Removing the bulk specimen lid can only be done when the chamber is vented. If the chamber is not vented, follow the description of how to safely vent the chamber. Once the chamber is vented, the bulk specimen lid can be removed by gently pulling straight up on the handle directly above the lid. After lifting about 30 mm, a lever will come out and hold the lid up. With the lid held in place, the entire lid mechanism can be rotated to provide access to the bulk specimen chamber.

Replacing the lid can be done by rotating the lid to align with the chamber. While supporting the lid by holding onto the handle, push the lever in to the post. When the lever is completely in the post, the lid will be allowed to lower. Gently lower the lid into place. Do not allow the lid to drop as this may cause scratches on the O-Ring surface of the lid.

7.4 Evacuate the Plasma Chamber

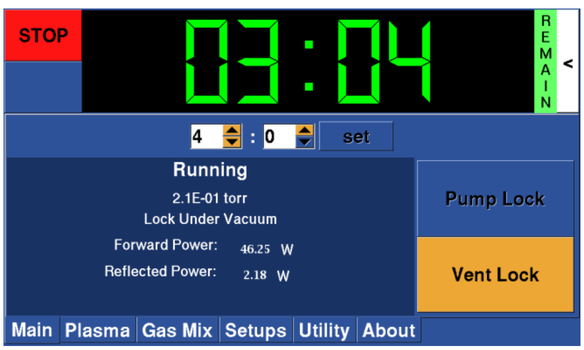
Before attempting to evacuate the Plasma Chamber, ensure both Specimen Port adapters have either a Specimen Holder or a plug installed. Additionally, verify the Bulk Specimen lid is properly installed on the top of the chamber. Once the chamber is properly sealed, press the “Pump Lock” button on the main tab. The mechanical rough pump will evacuate the plasma chamber. When a sufficient base pressure is reached the gate valve will open and the turbo pump will finish the pump down process.

7.5 Cleaning a Sample

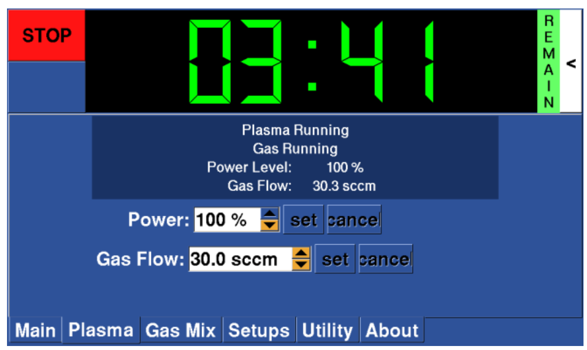
Follow the steps outlined below to prepare the system to clean a sample. These steps have been described in detail earlier.

- Vent the chamber
- Load the desired sample in either the specimen holder port or the bulk chamber
- Evacuate the plasma chamber
- Configure the gas system using the Gas Mix tab
- Select the desired gas ratio using the as Gas Mix tab
- Select the cleaning power using the Plasma tab
- Select the gas flow using the Plasma tab
- Set the cleaning time using the Main tab

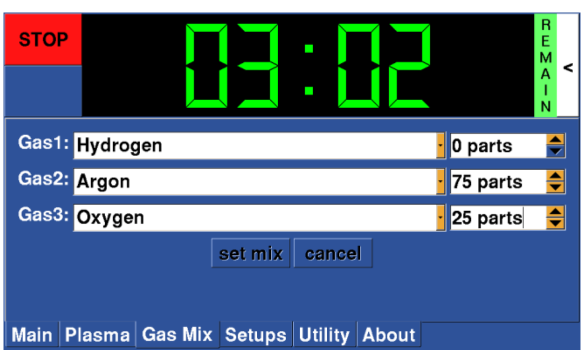
At this point, the system has a sample loaded and the system is ready to start a plasma cleaning cycle. To start the plasma cleaning process, press “Start” button. The system will enter the preparation phase. During this time, gas is flowing into the chamber to raise the chamber pressure, the tuning network is moved to the lighting position, and when conditions are correct, the RF power will be applied. After the power is applied, the plasma will light. Further tuning will occur and the system will then enter the running phase. While running, the system time will either count down or count up depending on whether the time displayed is remaining or elapsed. The tabs shown below show the system during a cleaning operation.



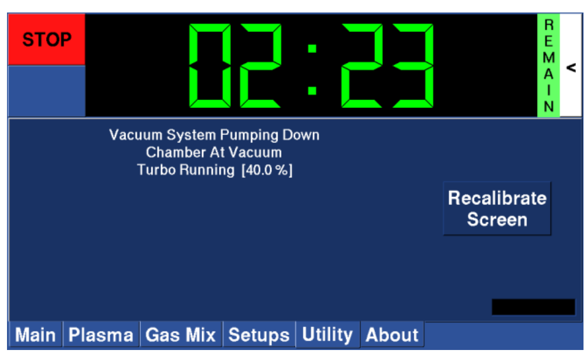
Main Tab



Plasma Tab



Gas Mix Tab



Utility Tab

7.6 Working with Vacuum Storage Containers

TEM holders can be stored under vacuum with or without samples in the appropriate Vacuum Storage Container. Storing holders and samples under vacuum prevents contamination from occurring between the time the sample is cleaned and the time the holder is placed in the TEM for analysis. To store a TEM holder in the appropriate Vacuum Storage Container, follow the steps outlined below. Each of these steps has been described in detail earlier.

- Vent the chamber
- Place the correct Specimen Holder port into either of the chamber openings
- Insert the correct vacuum Pumping Port into the Specimen Holder port
- Connect one end of the supplied Viton tube to the pumping port
- Connect the other end of the tube to the Vacuum Storage container
- Insert the TEM holder into the storage container
- Open the valve on the storage container by pulling out on the black valve knob
- Evacuate the plasma chamber – this will also evacuate the storage container
- Wait for the system to reach high vacuum (indicated on Main tab)
- Close the valve on the storage container by pushing in on the black valve knob
- Vent by clicking on “Vent Lock” button
- Wait for system to reach atmosphere (less than 5 seconds)
- Remove the tube from the end of the storage container
- Remove the Pumping Port from the Specimen Port holder
- Place the appropriate plug in the Specimen Port holder

Caution must be exercised when working with a Vacuum Storage Container. If the procedure outlined above is not followed, atmospheric pressure could be introduced to the plasma chamber and the turbo pump will not be able to operate. Be sure to vent the system before removing the tube from the pumping port or the storage container. Failure to do so will result in problems with the vacuum system and could create long term damage to the turbo pump.

7.7 Working with Vacuum Pumping Station

TEM holders can be stored under vacuum with or without samples in the appropriate fixture in a Vacuum Pumping Station. Storing holders and samples under vacuum prevents contamination from occurring between the time the sample is cleaned and the time the holder is placed in the TEM for analysis. The pumping station also provides a clean and convenient location to store TEM holders that are not currently in use. To store a TEM holder in the appropriate fixture on the Vacuum Pumping Station, follow the steps outlined below. Each of these steps has been described in detail earlier.

- Vent the chamber
- Place a Specimen Holder port into either of the chamber openings
- Insert the correct vacuum pumping port into the Specimen Holder port
- Connect one end of the supplied Viton tube to the pumping port
- Connect the other end of the tube to the Vacuum Pumping Station
- Insert the TEM holder into the correct fixture on the pumping station
- Open the valve on the fixture holding the TEM holder
- Open the main valve on the pumping station by pulling out on the black valve knob
- Verify the valves on all unoccupied locations on the pumping station are closed
- Evacuate the plasma chamber – this will also evacuate the pumping station
- All fixtures that have their valves open will be evacuated
- Wait for the system to reach high vacuum (indicated on Main tab)
- Close the main valve on the Vacuum Pumping Station by pushing in on the black valve knob
- Close the valves on all TEM holders in the fixture
- All holders that were evacuated will now be held under vacuum until they are removed
- Vent by clicking on “Vent Lock” button
- Wait for system to reach atmosphere (less than 5 seconds)
- Remove the Pumping Port from the Specimen Holder port
- Place the appropriate plug in the Specimen Holder port

Caution must be exercised when working with a Vacuum pumping station. If the procedure outlined above is not followed, atmospheric pressure could be introduced to the plasma chamber and the turbo pump will not be able to operate. Be sure to vent the system before removing the tube from the pumping port or the storage container. Failure to do so will result in problems with the vacuum system and could create long term damage to the turbo pump.

8. Advanced System Operations

Advanced operations are for users who would like to experiment with different power and gas combinations to create a cleaning process that is unique to their specific application

8.1 Power Adjustment

The M1070 uses a solid state RF Power generator to create the plasma. The system is calibrated to 50 watts at the factory. Users have the flexibility to vary the power delivered to the plasma chamber by modifying the power setting on the Plasma tab. If less power is desired, the user can enter a number between 20 and 100% on the Plasma tab. This number will vary the output of the 50 Watt generator. The system will always light at 80% power, but once the plasma is generated, the output will be changed to what the user requested. Some combinations of gas flow, power and gas mix may not light. If you are using a new combination of parameters it is best to observe the plasma thru the sight glass on the front of the instrument to verify the plasma lit.

8.2 Gas Flow Adjustment

The M1070 uses three independent Mass Flow Controllers (MFC) to mix the gas that is delivered to the plasma chamber. The gas flow that the user sets on the Plasma tab is the total flow (the sum of all 3 flow controllers) of gas going into the chamber. This flow can be set from 20 to 40 SCCM. The factory default is 30 SCCM. The flow rate has a direct impact on the plasma chamber pressure. A lower flow will result in a lower chamber pressure. Conversely, a higher flow will result in a high chamber pressure. The chamber pressure will have a slight impact on the ion energy in the plasma, but over the range of 20 – 40 SCCM, the variation is very small. Some combinations of gas flow, power and gas mix may not light. If you are using a new combination of parameters it is best to observe the plasma thru the sight glass on the front of the instrument to verify the plasma lit.

8.3 Gas Mix Adjustment

Each MFC has a range of 0 to 50 SCCM. The flow of each MFC is dependent on the total flow and the gas ratio set on the Gas Mix tab. As an example, consider a total flow of 30 SCCM with 75 % Argon and 25 % Oxygen specified. For this arrangement, the Argon MFC will set a flow of 22.5 SCCM and the Oxygen MFC will set a flow of 7.5 SCCM. The total flow will be the sum of the individual gases for a total of 30 SCCM. There is a practical limit on the low end of the range for the MFC. The MFC will reliably control down to 1 SCCM. Below that the flow is not guaranteed. This lower limit must be considered when setting the gas mix. A mix of 99% Argon and 1% Oxygen would result in the Oxygen MFC getting set to 0.3 SCCM which is below its range. Some combinations of gas flow, power and gas mix may not light. If you are using a new combination of parameters it is best to observe the plasma thru the sight glass on the front of the instrument to verify the plasma lit.

9. Applications

9.1 Literature References

The Model 1020 is applicable to plasma cleaning a variety of surfaces prior to FE SEM or TEM analyses. References include:

Fischione PE (1997) Plasma Processing System For Transmission Electron Microscopy Specimens and Specimen Holders. U.S. Patent Number 5,633,502: E.A. Fischione Instruments, Inc.

Fischione PE, Ringnalda J, Feng Y, Krekels T, Hayles M, Colijn JO, Mills MJ, Wiezorek JM (1997) The Use of a Cold Gas Plasma For the Final Processing of Contamination Free TEM Specimens. In: *Specimen Preparation for Transmission Electron Microscopy of Materials – IV,* Anderson RM and Walck SD (eds). Mater Res Soc Symp Proc 480:225-234.

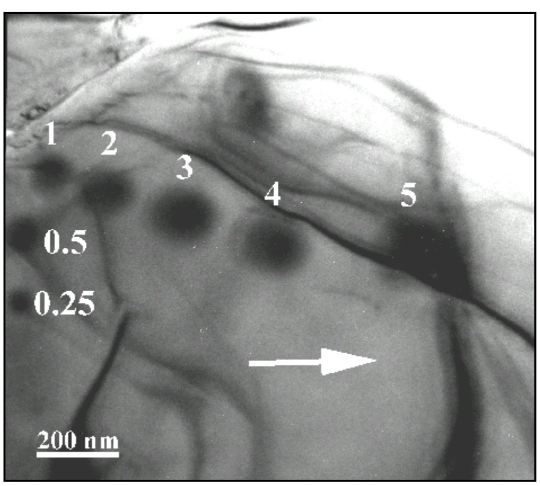
Isabell TC and Fischione PE (1998) Applications of Plasma Cleaning For Electron Microscopy of Semiconducting Materials. In*: Electron Microscopy of Semiconducting Materials and ULSI Devices*. Hayzelden C, Hetherington C and Ross F (eds). Mater Res Soc Symp Proc 523:31-38.

Isabell TC, Fischione PE, O'Keefe C, Guruz MU and Dravid VP (1999) Plasma Cleaning and Its Applications for Electron Microscopy , Microsc Microanal , 5:126-135.

Zaluzec NJ (1996) Simultaneous Specimen and Stage Cleaning Device for Analytical Electron Microscopy . U.S. Patent Number 5,510,624, - Argonne National Laboratory and the University of Chicago.

9.2 Application of Plasma Cleaning for TEM

Proper processing of TEM specimens results in improved imaging and chemical analysis. As demonstrated and explained in the literature, existing carbonaceous contamination spots are removed with the Model 1070. However, the best practice is to plasma clean every specimen and holder prior to insertion into the TEM. Processing times of ten seconds to 2 minutes are usually sufficient to remove organic contamination. Slightly longer times may be required for heavily contaminated surfaces.
An example of plasma cleaning and its ability to prevent contamination is presented below.



304 Stainless Steel (200 kV, 5 nm – electron probe live time)
1) Before Plasma Cleaning
- 1 – Minute
- 2 – Minutes
- 3 – Minutes
- 4 – Minutes
- 5 – Minutes

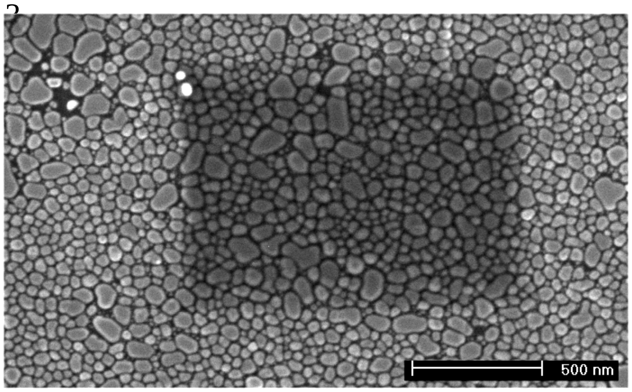
2) After Plasma Cleaning (1 Minute)
- 5 – Minutes at location indicated by arrow

Condensing the electron probe on the specimen's surface polymerizes hydrocarbons and results in the creation of contamination "spots". Once plasma cleaned, the specimen shows no build-up of contamination, even when subjected to extended working times under a focused probe.

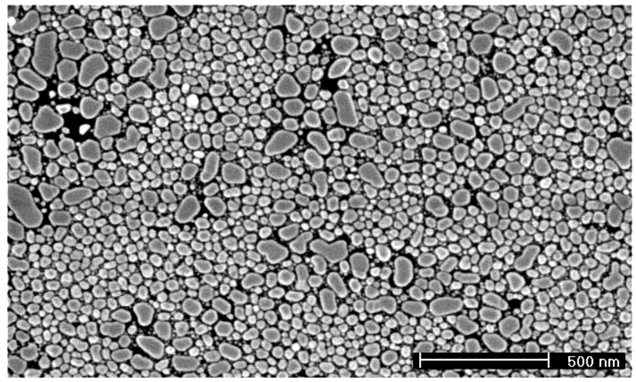
9.3 Application of Plasma Cleaning for SEM

Proper processing of SEM specimens results in improved imaging and chemical analysis. As demonstrated and explained in the literature, existing carbonaceous contamination spots may be removed with the Model 1070. However, the best practice is to plasma clean every specimen and holder prior to insertion into the SEM. Processing times of ten seconds to 2 minutes are usually sufficient to remove organic contamination. Slightly longer times may be required for heavily contaminated surfaces.

A typical example of improved imaging at low kV is presented below.



Before Plasma Cleaning



After Plasma Cleaning

In this case, a “box” of contamination is built up by rastering the electron probe over an unclean surface. The specimen used is a resolution standard of gold islands deposited on a carbon substrate. Following 30 seconds of plasma cleaning, existing contamination is removed and re-contamination is prevented.

10. Maintainence

WARNING! When servicing any component of the Model 1070 it is imperative that the mains power is turned off. Following a one-minute period to ensure that any charged electronic component will discharge, disconnect the mains cord from the power entry module located on the back of the instrument. Due to the high voltages associated with plasma processing, failure to follow this procedure could result in a harmful or lethal electrical shock.

10.1 Cleaning the Air Filters

Due to the high flow of air through the enclosure it is recommended to clean the air inlet filters annually. These filters are beneath the instrument at various locations on the base plate of the system.

To remove the filters, carefully position the instrument near the edge of a bench. Using wooden blocks (not provided) raise the unit approximately 10cm above the table top. While one person can perform this operation, two people are recommended. One should stabilize the instrument and one should remove the filters. Under no circumstances should the instrument project over the edge of the bench to access the filters.

NOTE: Do not place the wooden blocks under the air filters themselves. Doing so will make the filters inaccessible for removal and may damage the filters.

The filters are removed from their mounts by removing the four retaining screws. Once removed the filter elements should be washed in warm, soapy water and then allowed to dry in a clean environment.

The filters are comprised of a metallic weave, which prevents the emission of EMI/RFI from the Model 1070. The filter should be evaluated for both integrity and cleanliness. When completed, reinstall the filters by screwing them onto their respective mounts.

Finally, remove the wooden blocks and ensure that the unit sits firmly on the table top.

10.2 Replacing the Fuses

Should the instrument not power-up, it can be an indication of a blown fuse. To change a fuse, switch off the main power, and disconnect the mains cord from the instrument. Using a small blade screwdriver, gently remove the plastic cover of the power entry module to expose the fuse cartridge. The power entry module requires 2 fuses of the same rating. Replace only with approved fuses, Fischione Part Number 500-2024.

11. Troubleshooting

Symptom	Possible Cause	Possible Remedy
No activity (sounds or lights) when main power switch is in the "on" position.	Power supply interrupted	→Check fuses in the power entry module →Check power connection →Measure voltage coming from supply and verify it is correct
Erratic control behavior.	Configured for incorrect AC mains voltage.	→Measure voltage coming from supply and verify it is correct
System will not enter High Vacuum state	Air leak into vacuum system. Vacuum gauge malfunction Vacuum pump malfunction	→Check vacuum for leaks →Verify port plugs are installed and O-Rings are good →Check chamber lid and O-Ring
Plasma is dim or invisible.	Gas supply is shut off. Plasma tuning is incorrect RF Power Supply malfunction	→Check gas supply. →Check Plasma tuning
Plasma is unstable (intensity fluctuates continuously).	Gas Supply not sufficient Plasma tuning is incorrect	→Adjust for 15psig (103kPa) with no gas flow →Check Plasma tuning →Modify plasma parameters (gas flow, gas mix, power level).
Plasma color is pink.	Air leak into vacuum system. Incorrect type of gas supply.	→Check vacuum for leaks →Check process-gas source →Purge Process Gas lines

12. Service

In the event that repairs are necessary, the following options are available:

- Return the Plasma Cleaner to Fischione Instruments for repair.
- Request on site repair by an authorized Fischione service engineer.

Before returning the instrument to Fischione:

- Please contact Fischione Instruments for a Return Material Authorization Number (RMA).
- Please request packaging material if not available.

13. SPECIFICATIONS

Plasma System

- A high-frequency (HF) oscillating field system inductively coupled to a quartz plasma chamber
- Ion energies of less than 15eV
- Compatible with side-entry specimen holders for FEI, JEOL, Hitachi and Zeiss Transmission Electron Microscopes (TEM)

Vacuum System

- Turbo-molecular drag pump backed by an oil-free diaphragm pump
- Vacuum sensing with Pirani gauge
- Sample exchange accomplished in less than 40 seconds

Process Gas

- Argon – UHP 99.998% pure
- Oxygen – UHP 99.998% pure
- Hydrogen – UHP 99.998 % pure
- Pre-mixed 25%Oxygen – Balance Argon. UHP – 99.998 pure
- All gases should be set to nominal delivery pressure of 15 PSIG (103kPA).

Control Gas

- Nitrogen – HP 99.99% pure
- Clean-Dry-Air (CDA)
- Argon – HP 99.99% pure
- Nominal delivery pressure of 60 PSIG (414kPA).

Enclosure

- Self-contained - 27” (686 mm) Wide x 22.3” (566mm) Deep x 22” (559mm) high
- All components fully accessible via removable covers
- Weight: 160 lbs. (73 kg)

User Interface

- Single 7” touch-screen operator panel to control pumping, timing, initiation of plasma, and venting
- Process timer allows automatic termination

Power Requirements

- 100/120/230/240 VAC single- phase
  660 Watts

Warranty

- One Year