Plasma Prep III™ Solid State Low Temperature Asher/Etcher Operation Manual

SPI # 11050

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For further information regarding any of the other products designed and manufactured by SPI Supplies, contact your local representative or directly to SPI Supplies at the address above, or visit **www.2spi.com**

- Carbon and Sputter Coaters
- Plasma Reactor for ashing and etching
- High Vacuum Bench Top Evaporators
- Critical Point Dryers
- Electron Microscopy Supplies, Consumables and Accessories

Warranty

The SPI Supplies unit you have purchased is guaranteed to be free of defects in workmanship on the day of shipment. This warranty covers parts and labor for a period of one year, excluding shipping charges or consumables. Breakage of glassware is specifically excluded from this warranty.

Proper use of your unit, according to the operation manual, should result in trouble-free operation. Any improper use of the SPI Supplies unit through modifications or unreasonable operating procedures will void this warranty.

DISCLAIMER

SPI Supplies Plasma-Prep III is designed for simplicity of installation and operation. This manual provides full and complete information in both of these areas. SPI Supplies therefore assumes no liability or responsibility of any kind for damage or injury resulting from incorrect installation or operation of the machine.

If any questions arise, call SPI Supplies from the USA/Canada 1-800-2424-SPI or 1-610-436-5400 for assistance. For all other countries, contact our nearest agent or SPI Supplies directly. A listing of our agents may be found on our website at:

http://www.2spi.com/info/agents/

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SECTION 1

PLEASE - TAKE A MOMENT TO READ SECTION ONE [1] BEFORE PROCEEDING TO INSTALLATION

GENERAL DESCRIPTION - SPI PLASMA-PREP III

1-1 GENERAL COMMENTS

The SPI Supplies Plasma-Prep III is a table-top plasma chemistry reactor designed to provide plasma technology at a moderate cost. This simple-to-operate instrument can perform repeatable plasma chemical reactions with a minimum of automation. All controls are manual; however, where necessary, automatic monitors and controls take over to protect the equipment and the samples in the reactor.

The Plasma-Prep III comes equipped with an internally housed solid state RF generator. RF power is transferred from the power amplifier via a matching network to the reaction chamber. A variable capacitor provides a tuning control. Metering of the forward and reverse power and enables tuning of the instrument and is displayed on an LED selectable push button switch.

1-2 FUNCTIONAL DESCRIPTION

The Plasma-Prep III is a small reactor that weighs under 31 pounds fully assembled, less vacuum pump. With the exception of the external vacuum pump, it is a fully self-contained machine. It consists of an RF generator and associated tuning circuits, a vacuum system with solenoid control valves, a constant feed gas supply system and a reaction chamber system, which includes two Pyrex® glass or quartz chamber elements.

1-2.1 RF Generator - The RF power source includes a solid state oscillator operating at 13.56 MHz, the FCC-authorized industrial frequency. A solid state linear driver and amplifier supplies a continuous wave power to maximum of 100 watts. Power transfer to the reaction chamber is accomplished via an impedance matching network.

1-2.2 Vacuum System - The vacuum system includes the vacuum

pump (not supplied as part of the Plasma-Prep III), the vacuum hose, the vacuum valves and the control circuitry. The vacuum valve switch is interlocked with the RF generator switch to prevent the RF power from coming on unless the vacuum valve is energized. An internal vacuum meter is used to monitor chamber pressure.

1-2.3 Gas Supply System - The gas supply system for the Plasma-Prep III consists of the gas delivery system inside the reaction chamber. This delivery system is a glass tube sealed on the inner end and perforated along its bottom surface. Connections to the delivery tube are made by sliding the tubing over the barbed fittings on the glass chamber.

1-2.4 Reaction Chamber - The reaction chamber sub-system consists of an upper and lower electrode and a two element Pyrex glass reaction chamber (See Fig. 2-1). (For CF_4 operation, a quartz chamber should be used). These are open ended cylinders designed to fit into each other to form a closed chamber. The chamber is sealed with a flat gasket which seats against a raised lip on the inner chamber portion. The gas delivery tube feeds through the back of the outer chamber section. This chamber section also provides connection to the vacuum hose by a glass tube joined at the front of the chamber. The inner chamber is perforated by a series of slots located on the bottom surface of the chamber. Four small glass "feet" on the bottom of the inside chamber raise it off the inside surface of the outer chamber to provide a space between the two chamber sections. This arrangement provides for the best gas conduction flow and results in repeatable, dependable processing.

1-3 SAFETY INFORMATION

Interlocks - There are two microswitch interlocks engineered into the machine to prevent injury to operating personnel. These are:

a. Front door interlock - cuts off AC to the RF power supply
b. Right side, center - shuts off all primary power

*****WARNING*****

SINCE THE GAS MOST USUALLY USED IN THE PLASMA-PREP III IS OXYGEN, NO SMOKING SIGNS SHOULD BE POSTED NEAR THE MACHINE AND THE NO SMOKING RULE OBSERVED

Even if a different gas is going to be used, we still recommend the enforcement of the NO SMOKING rule because, quite frequently, the user has an unanticipated need for oxygen and the NO SMOKING rule would be appropriate.

1-4 TYPICAL PLASMA PROCESS

The "Plasma Process" is accomplished through the use of a low pressure, RF induced gaseous discharge. The material or specimen is loaded into the reaction chamber. The chamber is evacuated to a mild vacuum (approximately 250 microns) by a mechanical vacuum pump. A carrier gas is drawn through the chamber over the specimen. Radio frequency power is applied around the chamber (at 13.56MHz). This excites the carrier gas molecules and changes some of them into chemically active species.

The mechanism employed in this process is one of oxidation. Electrons, produced by ionization of gas, gain energy in the electric field. Subsequent collisions between these energetic electrons and neutral gas molecules result in an energy transfer to the molecules producing chemically active atoms, free radicals, ions and free electrons. The combustion products, which are completely dissociated and harmless, are carried away in the gas stream. The unique property of this process is that it occurs near ambient temperatures without employing toxic chemicals.

TABLE OF SPECIFICATIONS

PHYSICAL DIMENSIONS

Height	10.5 inches (26.7cm)
Width	11.8 inches (30.0cm)
Length	14.8 inches (37.6cm)

WEIGHT

Assembled	31 pounds (14kg)

EFFECTIVE CHAMBER SIZE

Inside length	5.9 inches (15.0cm)
Inside diameter	4.15 inches (10.54cm)

RF POWER	0 to 100 Watts
RF FREQUENCY	13.56MHz crystal controlled
AC POWER	100 to 240 VAC, 50/60Hz (not including vacuum pump)

NOTE:

This equipment complies with Title 47, Part 18 of the Federal Communications Commission Rules when operated as set forth in the accompanying instruction book. The fundamental (+/- 7 kHz) falls within the unlimited radiation ISM bands defined in FCC 18.301. Field strength levels of emissions outside the fundamental are below the limits of FCC 18.305.

This ISM device complies with Canadian ICES-001.
Cet appareil ISM est conforme à la norme NMB-001 du Canada.

SECTION 2

INSTALLATION AND PREPARATION FOR USE

2-1 FACILITY REQUIREMENTS

The Plasma-Prep III is designed for table-top use. The facility requirements to operate the Plasma-Prep III are:

a. Vacuum pump capable of 50-100 liters per minute. We recommend the SPI#10405-AB (with Fomblin® oil) for use with O_2 or CF_4 process gas.

b. Gas supply - oxygen (O_2) or other reactive gas, industrial grade with regulator capable of supplying 5 PSIG, and shut off valve.

c. 100 to 240 VAC 50/60 Hz, 15 amp service.

2-2 SITE REQUIREMENTS

There are very few constraints on the location of the Plasma-Prep III. The machine should be operated in a well ventilated area away from any corrosive fumes. The supply from the cooling fan should not be obstructed. The machine's exhaust (the vacuum pump exhaust) should be vented away from operating personnel. Finally, the machine should be far enough away from high voltage equipment to prevent possible high voltage interference.

2-3 ASSEMBLY

The only assembly required when installing a Plasma-Prep III consists of putting the outer chamber into the electrodes, and connections to gas, vacuum and electrical power.

2-3.1 INSTALLING THE OUTER CHAMBER (Fig. 2-1)

With the machine resting on a convenient work surface, remove
the four screws (two on each side) at the bottom of the sheet
metal top cover. Remove the top cover only.

A. Open the door and remove the two nylon screws and spacing bushings on the left side and loosen the two nylon screws on the right side.

B. Remove the outer chamber from its shipping container. Make sure no packing material is in the chamber.

C. Lift the upper shell (cover will "hinge" on the two loosened screws). Slide chamber to the rear of the shell and engage the pre-fitted silicon tubes. The upper tube will attach over the barbed fitting on the gas inlet valve. The tube from the middle of the chamber is not used as part of the standard operation. A cap is placed over this fitting.

D. Locate the exhaust manifold at the front-center floor of the cabinet. Loosen the brass compression nut to accept the extension of the outer chamber for fitting into vacuum manifold.

E. Following insertion of the glass extension inside the manifold, tighten the knurled compression nut, <u>finger</u> <u>tight</u>. **DO NOT OVERTIGHTEN** as this can affect the vacuum. Care must be used to ensure that the silicon tubes are firmly in place and the glass neck fits into the vacuum manifold without it being cocked. If not properly aligned, the glass neck can break upon use.

F. Replace the two nylon screws that were removed from the left side and retighten them. **DO NOT OVERTIGHTEN** as this can damage the threads.

<u>WARNING</u>

Never use metallic screws in place of the nylon screws. Metal, even this small amount, will short circuit the chamber's electrodes.

Figure 2-1. Connecting Outer Chamber to Vacuum Manifold

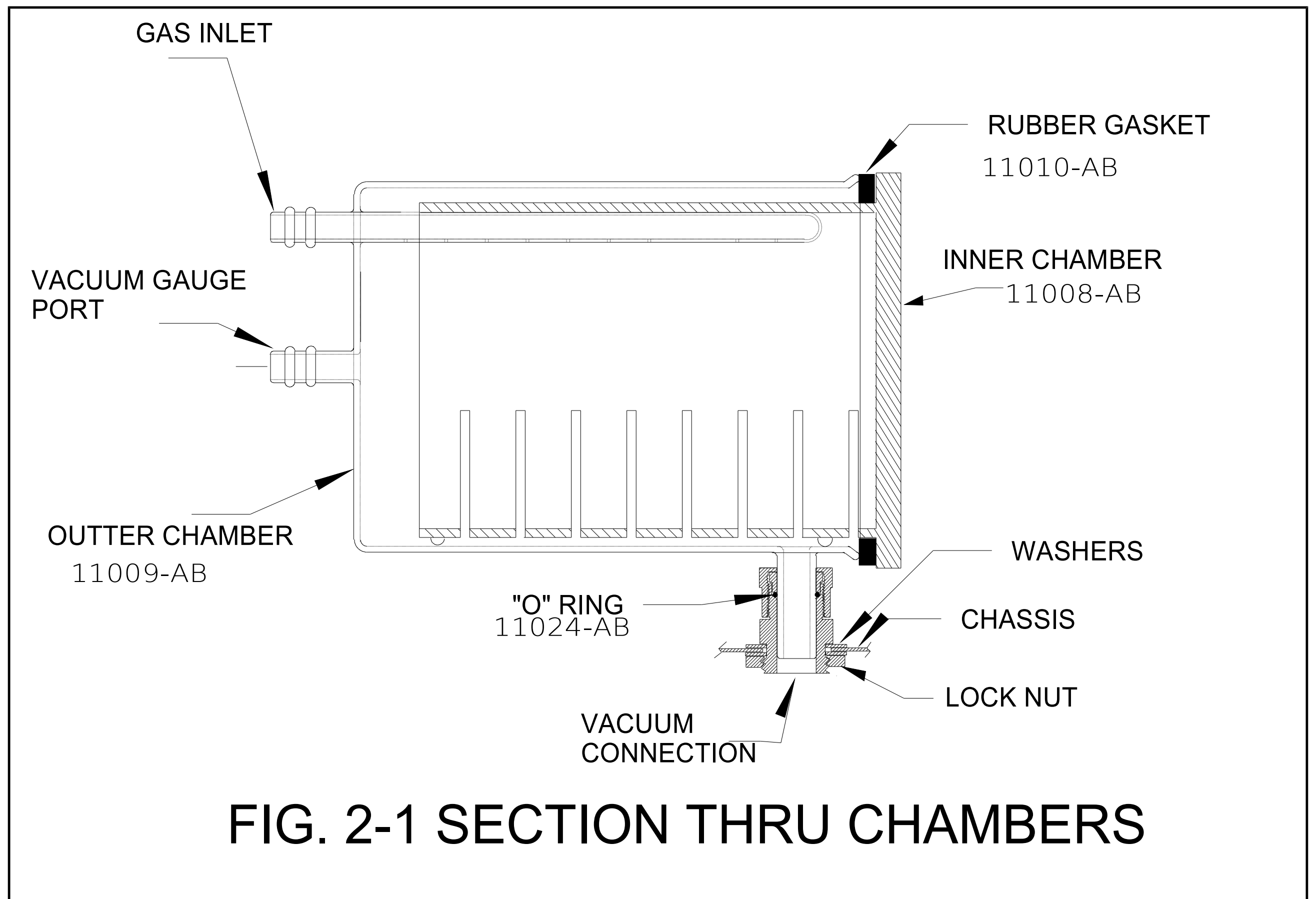


FIG. 2-1 SECTION THRU CHAMBERS

2-3.2 CONNECTING VACUUM PUMP

A. Connect the flexible metal vacuum pipe to the NW16 flange connections on the rear of PPIII and to the vacuum pump using the “O” ring and clamp provided.

B. Connect the regulated gas supply hose to the quarter-inch fitting GAS IN, Item 12 of Figure 3-2, located at rear of the machine.

2-3.3 INSTALLING THE INNER CHAMBER

A. On the Inner Chamber Section, make sure that the sealing "O" ring is not twisted and will seat against the inner lip of the chamber section.

B. Carefully insert the Inner Chamber into the Outer Chamber, orienting the slot perforations to the bottom of the chamber. There are four small glass knobs, which function as "feet" to provide some space at the bottom between the Inner and Outer Chamber sections.

C. Replace the top cover and secure with the four screws.

NOTE OF CAUTION: There is an interlock switch mounted on the right side (as viewed from the front) at chassis height. This is a lever type. When replacing the top cover, be sure to pull it slightly up, to clear the switch lever, to avoid damaging the interlock switch.

SECTION 3

OPERATION

3-1 GENERAL

Operation of the Plasma-Prep III consists of loading the object to be processed, evacuating the reaction chamber, applying the process gas and applying the energizing RF power. With the power level control about midway the tuning control should be rotated to obtain a plasma in the chamber and for a minimum reading of the reflected power or a maximum forward power reading. The desired power level can then be set. Upon completion of the process, the RF power switch is turned off, and then the vacuum valve switch is turned off. The primary power must remain on for the chamber to bleed back to atmospheric pressure. After the vacuum valve switch is turned off, the VENT valve switch must be energized. The main power can only be turned off after the vent switch is eneregized.

3-2 CONTROLS AND INDICATORS

Table 3-1 and Figures 3-1, 3-2 are a table of controls and indicators and illustrations of their location on the machine respectively.

3-3 OPERATION

3-3.1 PUMP DOWN AND OPERATION OF THE PLASMA PREP III

A. Power switch should be off (the red LED will be unlit). The POWER LEVEL control should be fully counter clockwise.

B. Start vacuum pump. Turn on AC power by depressing the POWER switch (No. 1, Fig. 3-1).

C. Open the chamber access door. Make sure the inner chamber is fully seated into the outer chamber, and the gasket is sealed between inner and outer chambers.***** Turn on vacuum switch (No. 2, Fig. 3-1), allow approximately 1 minute for pump-down or until the chamber pressure reads approximately 250 mTorr. Close access door.

***CAUTION**: Do not put excessive pressure on the inner chamber. This can cause stress on the glass extension to the vacuum connection and possible failure by implosion of the chamber.

D. Turn on the RF power switch (No. 3, Fig. 3-1). Advance (clockwise) the power level control (No. 4, Fig. 3-1) until there is an indication on the LED POWER (No. 6, Fig. 3-6) of approximately 50 watts. Turn the tuning control (No. 5, Fig. 3-11) counterclockwise and clockwise until the reading is maximized. Pressing the meter button will change the reading from forward (F) to reverse (r). Rock the tuning control back and forth until a minimum reading is achieved. Advance the power level control until the meter is indicating 100 watts and again rock the tuning control until a maximum reading is achieved with the forward measurement and a minimum is achieved in reverse. Operating at lower levels of power is achieved by reducing the power level to the desired watts and maintaining a minimum reverse power with the tuning control.

NOTE: After the Plasma-Prep III is tuned on is initial use, allow the unit to run for approximately one hour before using it. This will eliminate any contamination, which might be present in the chamber. Either air or process gas (O^2) can be used for this purpose.

3-3.2 TURNING OFF THE PLASMA PREP III

A. Turn LEVEL control fully counterclockwise.

A

B. Turn RF switch off.

C. Turn VACUUM switch off.

D. Turn VENT switch on. The chamber will vent in approximately one minute.

E. The machine can now be loaded and the process performed.

F. If this is the last cycle of the day, precautions must be taken to avoid contaminating the exhaust line with pump oil. [NOTE: Make sure tank pressure is shut off or else gas will continue to bleed and empty the tank]. If the main power switch is turned off before the chamber returns to atmosphere, it may take a long time to vent. The main power should remain on and the vacuum switch off to vent the chamber.

1. Remove the sample from the chamber.

Remove the inner chamber.

2. With AC on and VACUUM switch OFF, shut off the vacuum pump.

3. Leave the pump off for about a minute.

4. Throw the VACUUM on, then turn the pump back on again for another minute, and then shut it off [NOTE: When using system for asbestos filter preparation, place inner chamber back before proceeding with Step 4].

3-4 OTHER USEFUL INFORMATION

Each gas produces a slightly different color for the resulting plasma:

a. oxygen blue/white
b. argon fuchsia
c. carbon tetrafluoride blue/white
d. nitrogen violet

If the color of the plasma is red to violet, and the etch rate seems low, then air may be leaking into the chamber (the color is due to the presence of nitrogen).

The most likely sources of air leaks would be:

a. vacuum line to back of Plasma-Prep to mechanical pump.

b. "O" ring seal between the chambers (check to make sure no extraneous particles are present that would interfere with the vacuum).

c. vacuum line from back of Plasma-Prep to the glass etching chamber.

Sometimes mixtures of gases are required in order to accomplish specific ends. One very important case is the removal of epoxy or silicone thermosetting materials (such as the plastic package of an electronic device). Although basically "organic", they are oftentimes filled with various inorganic (most common SiO_2) fillers. The gas composition usually used is 10% CF_4, 90% O_2, with the CF_4 removing the filler and the O_2 taking away the polymer.

Typical etch times are as follows:

a. Removal of 1µm SiO_2: 25-30 minutes.

b. Removal of 1µm Si_3N_4: 30-35 minutes.

c. Cleaning of hybrid device: several minutes.

d. Package decapsulation of I.C.s with several mils of polymer remaining: In this case, the top portion of the package is ground or milled away so that what remains is only several mils. This can take place within a few hours or may take a day or more.

e. "Etching" or "ashing" away of a "Nuclepore" membrane filter: 45 minutes in an oxygen environment.

f. Etching of a "thick section" of OsO_4 stained biological material for SEM observation: 3 minutes or less.

g. Gentle etching to reveal inorganic material in an organic polymer matrix, such as magnetic storage media, pigment plastics, etc.: several minutes.

h. Cleaning of metallographically prepared samples prior to electron probe microanalysis for carbon: several minutes.

Figure 3-1 Front View of Plasma-Prep II Controls and Indicators

Figure 3-2 Rear View of Plasma-Prep III

TABLE 3-1 CONTROLS AND INDICATORS

ITEM	CONTROL & INDICATOR	POSITION	TYPE	FUNCTION
1	AC POWER	TOGGLES	PUSHBUTTON / LED	STARTS MAIN POWER
2	VACUUM	TOGGLES	PUSHBUTTON / LED	ENERGIZES SOLENOID TO EVACUATE CHAMBER
3	VENT	TOGGLES	PUSHBUTTON / LED	VENTS CHAMBER TO ATM
4	RF	TOGGLES	PUSHBUTTON / LED	ENERGIZES RF POWER
5	RF LEVEL	0 – 100 CCW-CW	KNOB	SETS THE POWER LEVEL
6	FWD-REV	TOGGLES	PUSHBUTTON	TOGGLES DISPLAY BETWEEN FORWARD & REVERSE POWER
7	TUNING	ROTATES	360 DEG.	SETS MATCH TO CHAMBER FOR MAXIMUM FORWARD OR LOWEST REVERSE POWER AT THE CURRENT LEVEL SETTING
8	KF16 FLANGE	N/A	N/A	FLANGE CONNECTION FOR VACUUM PUMP
9	POWER INLET	N/A	N/A	ACCEPTS MAIN POWER CORD
10	GAS INLET BARB	N/A	N/A	REACTION GAS INLET

SECTION 4

USER MAINTENANCE

4-1 GENERAL

Maintenance on the Plasma-Prep III consists of cleaning and replacing such parts as "O" rings. Any other repairs should be performed by SPI Supplies.

4-2 CLEANING

Cleaning consists of a routine procedure, which should be performed periodically. This schedule will depend on frequency of use, type of materials processed and the environment in which the machine has to operate.

Routine Procedure: The inner chamber will require more frequent cleaning than the outer chamber as it is more likely to "see" contamination. For light contamination, running an oxygen plasma for a few minutes should be enough to clean the chamber. For more difficult contaminants, (inorganic films and residues), use standard laboratory cleaning procedures. Remove the inner chamber and wash the chamber with a test tube brush and a good laboratory detergent. If this is not effective, it will be necessary to use an oxidizing agent. Refer to the "Handbook of Chemistry and Physics" for the proper agent.

The outer chamber will also require a similar procedure, but requires cleaning about half as frequently as the inner chamber.

4-3 VACUUM SYSTEM ADJUSTMENTS

The process gas flow is adjusted by the internal needle valve located at the rear top of the instrument just after the connector for the gas line. The needle valve is set so that the pressure in the chamber should read approximately 250 mTorr with the chamber empty and without any gas input. The needle valve typically does not need adjustment and if an significant difference in pressure is observed, checking for possible leaks at the o-rings and tubing should be done before adjusting the needle valve setting. The needle valve adjustment is done by turning the needle clockwise or counterclockwise. Keep in mind when making such an adjustment that the needle is sensitive and a slight adjustment cam significantly change the pressure in the chamber. For this reason we suggest only changing the setting if clearly needed.

The chamber bleed to atmosphere is controlled by a bleed valve solenoid located on the left side of the chassis. The adjustment is a setscrew. Turning it clockwise closes the valve and increases the time to bleed to ATM.

4-4 SUCCESSFUL FUNCTIONING OF YOUR EQUIPMENT

Successful functioning of your equipment depends upon a proper vacuum. Your vacuum pump should be serviced on a frequent and regular schedule. Pump fluid changes should be done on a regular schedule per the pump manufacturer recommendation.

4-5 SPARE PARTS LIST

SPI#	PART NUMBER DESCRIPTION
11008	4" ID Inner Pyrex Chamber
11009	4" ID Outer Pyrex Chamber
11010	4" ID "O" Ring
11013	Solenoid Valve, Straight thru
11014	Solenoid Valve, Metering
11024	"O" Ring for Outer Chamber
11026	Quartz Inner Chamber
11027	Quartz Outer Chamber

For other components not listed contact SPI Supplies

4-6 CUSTOMER RETURN INFORMATION

If a Plasma Prep III is to be returned to SPI Supplies, for whatever reason, the following procedure should be followed:

1. From the USA or Canada, call SPI Supplies Customer Service Department, 1-800-2424-SPI or 1-610-436-5400 for a Return Authorization Number (RA#). If unit is not received with this RA number on the outside of the box, it will be rejected by the Receiving Department. For other countries, contact either your closest SPI Supplies agent or SPI Supplies in the USA by phone 1-610-436-5400, FAX: 1-610-436-5755 or e-mail spi3spi@2spi.com following the same instructions.

2. Repack the machine in its original shipping container. If this is no longer available, take special precautions to avoid damage to the glass chamber sections and other fragile components. **DO NOT SHIP ANY GLASSWARE ASSEMBLED INSIDE THE UNIT.**

3. If the machine is still under warranty, the only charges are shipping costs. If the machine is out of warranty, a purchase order will be required for approved accounts and customer can expect to be billed for all parts and service. All others will require some form of guaranteed prepayment such as a letter of credit.

4-7

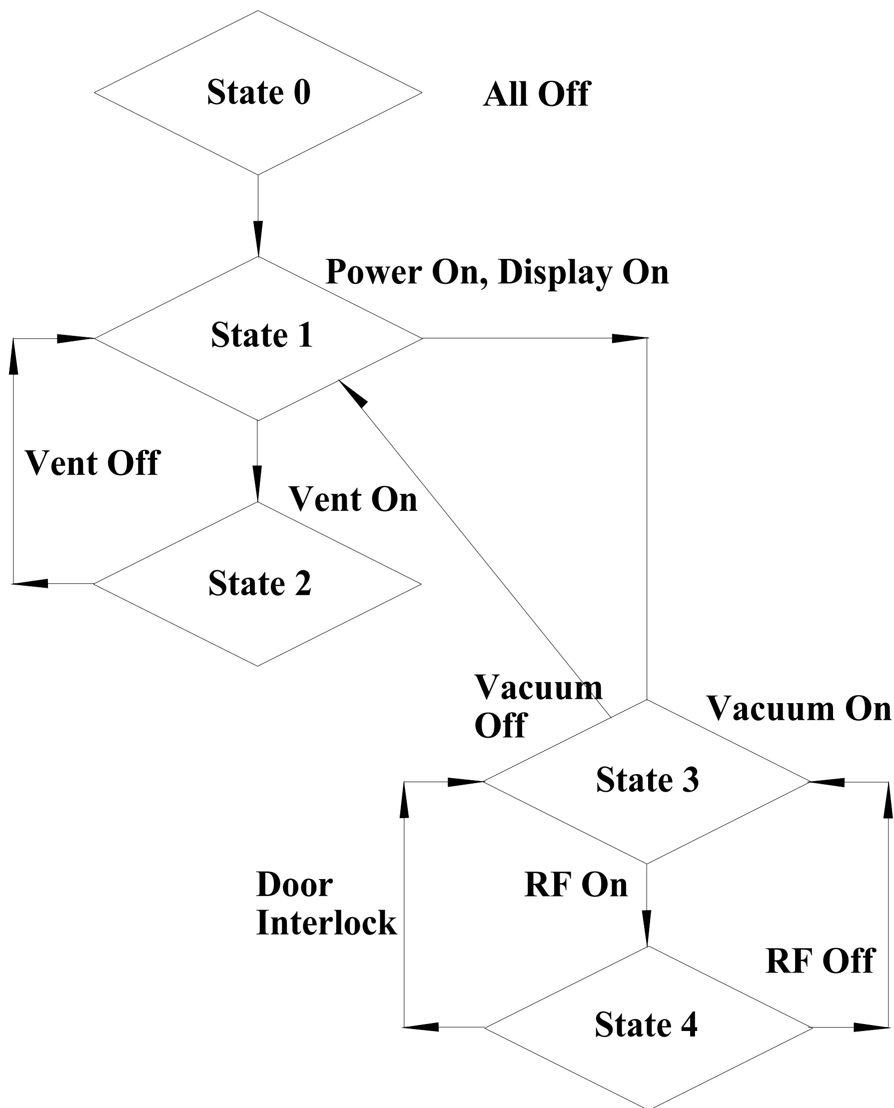
SUBSTRATE TO BE ETCHED	**REACTIVE GAS OR GAS MIXTURE**
Stainless Steel	CF_4 CF_4/O_2 mixture (experiment for correct proportions) CCl_4-necessary if chromium present
Hydrocarbons, all organics, polymers, etc.	O_2
Metal oxides	Ar
SiO_2; Si_3N_4 (passivation layers)	CF_4 or other reactive fluorine gases
Plastic packages	Combination of O_2 and CF_4, O_2 to etch plastic, CF_4 to etch glass filler. Typical formula: % 10 O_2/% 90 CF_4
Aluminum and aluminum alloys	Cl_2; CCl_4; or mixtures of Cl_2 + CCl_4
Al_2O_3; all forms of anodized aluminum; oxide layers on electronic circuits	**NOTE**: Initially use small amount of Br_2 in mixture to remove surface skin of Al_2O_3. Also useful are CF_4 and CHF_3 for this purpose

Section 5

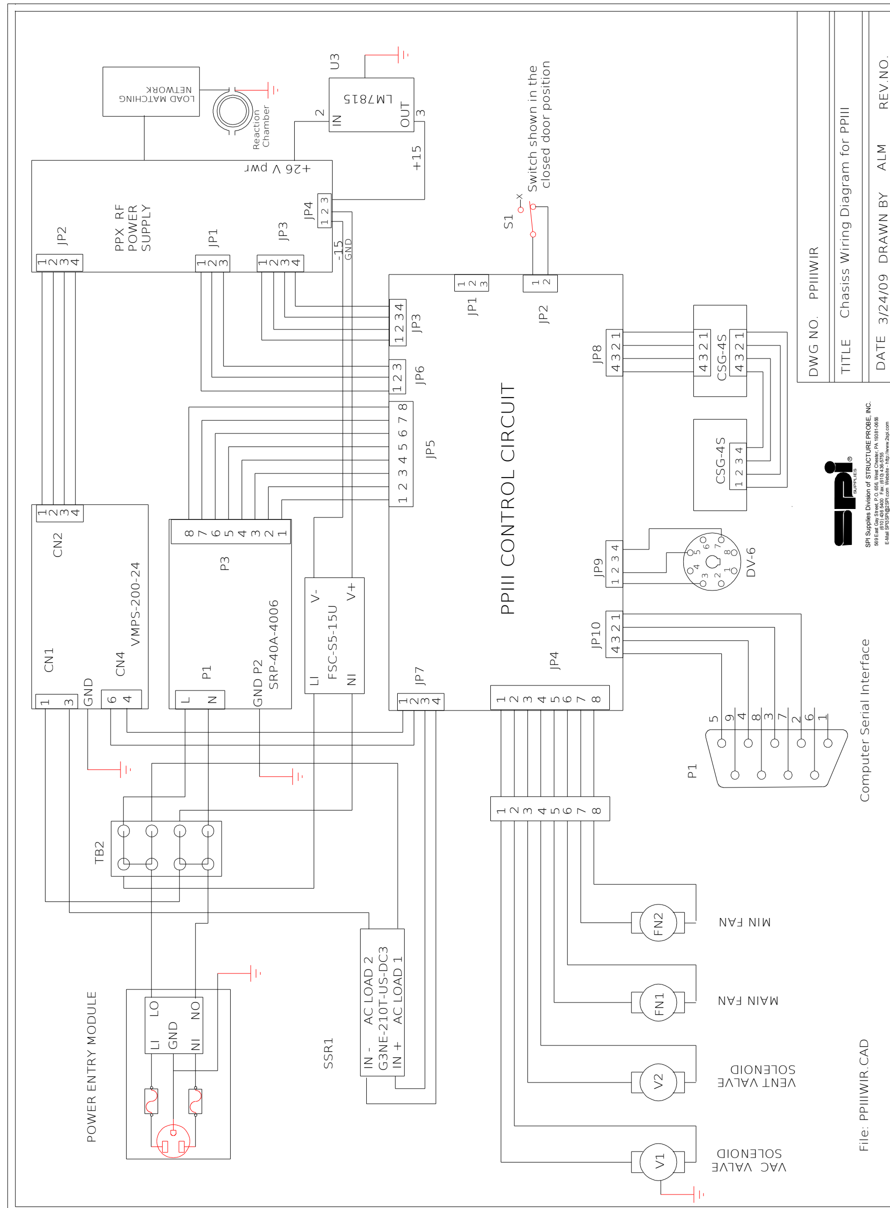
APPENDIX

5.1 PLASMA PREP III LOGIC FLOW CHART

Plasma Prep III Logic Flow Chart



5-2 CURCUIT DIAGRAMS

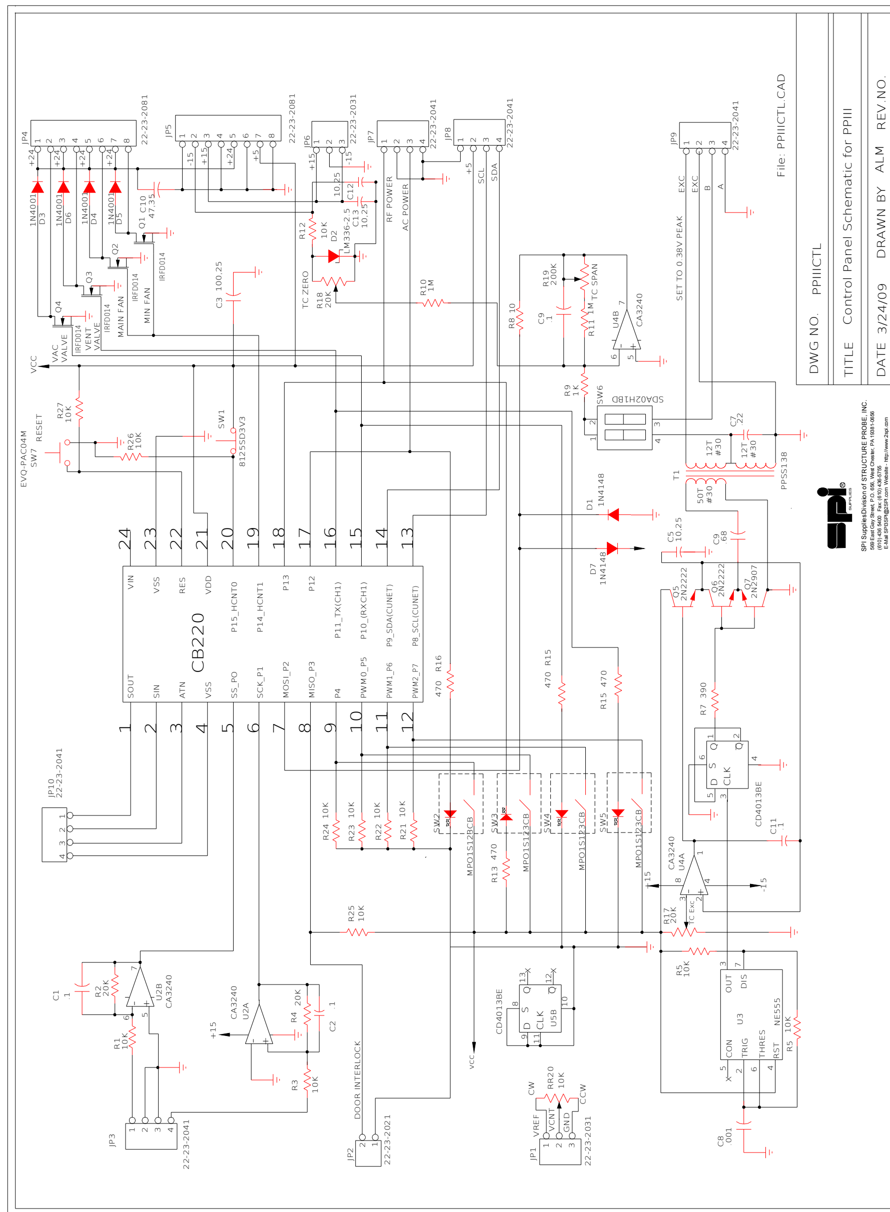


DWG NO. PPIIIRFD

TITLE RF Control Board Schematic

DATE 3/24/09 DRAWN BY ALM REV.NO.

Note: For closed loop control, close 1, 2 & 4 with 1 open for open loop control close 2. Open loop used only for initial calibration



DWG NO. PPIIICTL
TITLE Control Panel Schematic for PPIII
DATE 3/24/09
DRAWN BY ALM
REV NO.
File: PPIIICTL.CAD
CB220