SOUTH BAY TECHNOLOGY

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PC-2000
PE-2000

PLASMA CLEANER / PLASMA ETCHER

MATERIALS PROCESSING SOLUTIONS

The **PC-2000 Plasma Cleaner** allows simultaneous cleaning of a specimen and a specimen stage which minimizes and, in some cases, eliminates contamination of inorganic specimens analyzed via SEM, TEM, STEM and/or AEM. The technology involves subjecting the specimen and the specimen stage to a reactive gas plasma which efficiently removes a wide range of contaminants from critical surfaces. The procedure may be carried out prior to inserting the specimen and specimen stage into the EM by either mounting the specimen holder in the supplied port or by inserting the entire sample or sample assembly through the hinged upper port and into the 8" diameter x 4" high chamber. The system can be used with any side entry TEM holders, top entry TEM holders or with any sample that will fit inside the large, easily accessible chamber.

- Suitable for all side entry and top entry TEM holders. Large chamber also accommodates specimens up to 8" diameter and 4" high for cleaning tweezers, specimen mounts, etc.
- Digital LCD displays, indicate forward power in watts, reflected power, DC bias and vacuum level.
- System comes standard with 3 ports which allow simultaneous cleaning of multiple holders or insertion of analytical tools into the plasma chamber.
- Compatible with Argon, Oxygen, CF4, CI2, CCI4, O2, CF4 and other gas mixtures.
- Power level can be optimized for each specimen type and gas species to maximize cleaning rate without risk of etching the specimen stage or the chamber.
- Viewport allows easy monitoring of the process.
- Safety interlocked controls simplify operation - ideal for multi-user environments.

The **PE 2000 RF Plasma Etcher** is specifically designed for reactive gas plasma etching and surface treatments. The unit is capable of 150 watts RF forward power at 13.56 MHz and up to four gas processing. The system is ideal for R&D applications where single sample processing is needed and total control of each process parameter is necessary. Processes such as photoresist strip, BPSG removal, oxide and nitride layer etch, surface treatment of plastics and plasma cleaning are typical applications. Samples up to 6" diameter as well as irregular shaped substrates can be accommodated in the 200mm diameter vacuum chamber. A fully manual control system coupled with digital readouts and integral matching network with switching type power generator offer a wide range of experimental etch parameters.

- Manual controls, with digital readouts, make it possible to process in a wide range of vacuum pressure levels using an unlimited combination of reactive species including oxygen, fluorine and chlorine.
- A stainless steel gas system with three-position feed provides maximum etch uniformity and the best possible utilization of the reactive species. Includes a separate vent to atmosphere line.
- Safety interlocks prevent mis-operation thereby protecting the user and the system.
- High etch rates at moderate power levels of 100 watts forward power can be achieved - greater than 200 Å/minute for oxide and 500Å/minute for nitride.
- Gas lines, fittings and stage assembly are all stainless steel construction and designed for corrosive applications. A Fomblin charged corrosive series rotary vane pump is included.
- System is supplied with two manual gas channels, but up to four mass flow channels can be added.

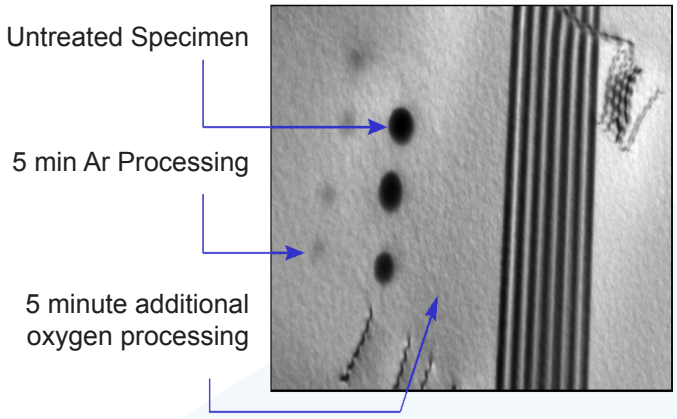
Plasma Cleaner Built under license from Argonne National Laboratory pursuant to US Patent No. 5,510,624. Inventor Dr. Nestor J. Zaluzec.

PLASMA CLEANER PROCEDURE

It has been well documented that low energy plasmas can be used to reactively etch/ash organic materials found on the surface of inorganic materials. This has been used by the industrial community to clean semiconductor wafers and other bulk materials for many years. In the PC-2000 procedure, a related technique is employed for the cleaning of EM specimens. Instead of subjecting only the material of interest (i.e. specimen) to the plasma, the entire stage and sample is plasma cleaned. The nature of the gas selected is based upon the desired effect. Although nominally pure argon followed by nominally pure oxygen is generally used, specific gases (BCI3, CF4,...) may be used to tailor a reaction. Cleaning time is typically 10 - 30 minutes.

Specimens that previously contaminated in minutes can, after treatment, be studied for several hours.

Fig. 1: TEM micrograph of 304 stainless steel specimen



Background

Microcharacterization of materials by electron microscopy is ultimately limited by the ability to observe, detect and analyze the constituent materials present in microvolumes of specimen.

In the environment of the electron microscope, the interaction of surface borne contaminants with the high energy incident electron probe can create deposits on surfaces of a sample which can prohibit these analyses. The contaminants may be introduced directly by the specimen, deposited on the specimen via the specimen stage or deposited on the specimen via the microscope system. Electron microscope manufacturers attempt to minimize the last of these by judicious design. However, the first two sources of contaminant introduction are out of their direct control.

Surface borne contamination is introduced in the preparation of inorganic specimens (metals, ceramics, semiconductors, etc.) for EM work. The materials to be studied are frequently subjected to chemical or electrochemical polishing followed by solvent rinsing and air drying. This typically leaves residual organic material on the specimen surface.

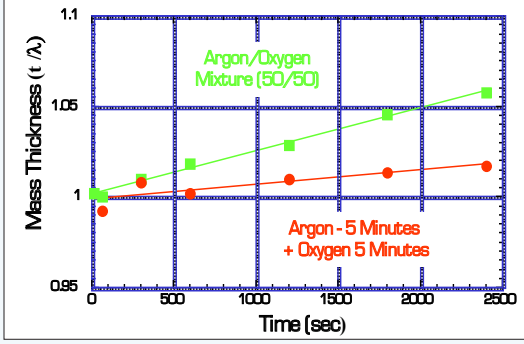
Attempts to clean the specimen stage include rinsing the stage with various solvents. Improper or poor techniques used to store both specimens and / or stages prior to insertion in the microscope can introduce additional organic residue. Finally, contamination derived within the column instrument can mitigate analysis.

While the majority of the organics involved in these processes dissipate, a small amount generally remains on the surface and is sufficient to cause problems when the specimen is subsequently examined in modern analytical microscopes. Although these organic residues are widely distributed and generally at low concentrations on the various surfaces, they can become mobile in the microscope environment and are “attracted” to the periphery of any focused electron probe, forming deposits. Since these contaminants can travel large distances over the surface of the specimen, it is important to remove or immobilize them as much as possible prior to an analysis without disturbing the microstructure of the specimen. It is important to note that once the deposits are formed, they are not easily removed.

Process Gas

Mixing of the process gases can be done directly from the unit itself as the PC-2000 is supplied with two independently controlled gas inlets. This allows a wide range of flexibility with both process parameters and process gases. Less thoughtful designs, with only a single gas inlet, use a pre-mixed gas supply for single step processes or switch gas supplies when implementing a two step process. In this experiment, two different methods of gas plasma processing were used. In one case, a 50/50 mixture of Ar/O2 gas was mixed at the unit and then used to treat an electropolished specimen of silicon for 10 minutes. Following processing the specimen was inserted into the TEM and the contamination rate was then measured. In the other case, a two step plasma process was used. First, a 5 minute treatment with nominally pure argon was carried out, followed by a 5 minute treatment with nominally pure oxygen. Again, the specimen was inserted into the TEM and the contamination rate was measured. In all cases, the contamination was cleansed most effectively using the two step process as opposed to the single process step for mixed gas processing.

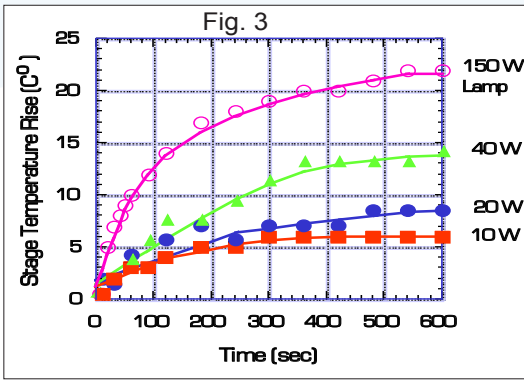
Fig. 2 Normalized Contamination Rate on Silicon



Heating Effects

The generation of a plasma can produce a large amount of heat at high power outputs, typically around 100 watts. However, temperature fluctuations of large degrees can be detrimental to the specimen, especially when dealing with materials that are beam sensitive or those that can alter their microstructure at relatively low temperatures. To characterize any heating effects which may be generated during plasma cleaning, a standard thermocouple gauge was used to measure any change in specimen or specimen holder temperature. The temperature was measured as a function of input power to the plasma, and was plotted over several seconds.

Fig. 3



At standard cleaning conditions, it was found that the temperature rise is nearly insignificant. In fact, a 150W lamp produces a much greater rise in temperature (~20C°) as compared to the plasma itself (~5-6°).

Process Analysis

Although plasma cleaning processes use relatively low power (<20 Watts) there were some concerns over possible sputtering or deposition of the plasma chamber onto the specimen surface. To verify whether or not deposition occurred, analytical methods such as XEDS, EEELs, and XPS were employed to analyze the specimen composition both before and after plasma processing.

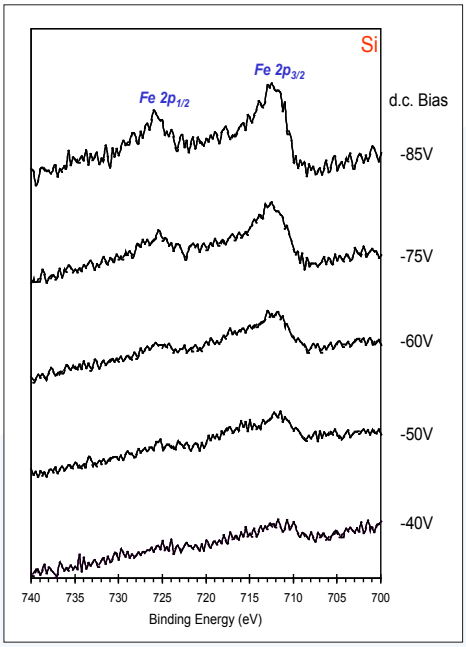


Fig. 4: XPS data acquired from Si specimens following plasma processing for different dc bias voltage settings.

X-ray Photoelectron Spectroscopy (XPS) is a highly surface sensitive characterization technique capable of detecting elements present in the top 10-20 atomic layers with a sensitivity of about 0.1%, and can observe chemical changes of the surface, such as oxidation. Therefore, XPS was used to detect any material sputtered from the plasma cleaner onto the specimen surface. If the dc bias of the system is above the threshold voltage for sputtering of the antenna material in the plasma cleaner, then Fe, Cr, and Ni will be sputtered onto the specimen. The extent of sputtering is shown in (Figure 4) where multiple scans were used to collect the spectra in order to improve detectability. The XPS spectra shown in (Figure 4) are the Fe and Cr 2p peaks from plasma cleaned silicon specimens with different dc bias values obtained by adjusting the power level. In this experiment, the pressure was held constant at 205 mTorr using a gas mixture of 75% argon 25% oxygen. As the dc bias is decreased from -85 to -40V, the Fe and Cr peaks decrease to the point where they are barely detectable. The concentrations of the contaminants are given in (Figure 5) which shows that for Fe and Cr they are less than 1%, even at the highest dc bias used (-85 V). The fluorine on the surface may originate from the fluorinated vacuum oil that was used in the mechanical pump. However, similar fluorine peaks have also been found in systems using an "oil free" pump [1]. Proper use of an oil filled mechanical pump will minimize this effect [Figure 5]. A recent analysis of the expected TEM sample composition for a given surface composition of flourine and chromium shows that the quantities given in [Figure 5] will not be detected by AEM techniques [1]. This was verified experimentally by XEDS and EELS for a silicon specimen after plasma cleaning using 10 W, -30 V dc bias, 150- 200 mTorr, and Ar and O2 processing, as shown in [Figure 6]. Since the suggested operating conditions of the PC-2000 plasma cleaner are 10 watts with a dc bias voltage between -20 and -30 V sputtering from the antenna and oil backstreaming is not a problem with respect to TEM microanalysis or imaging.

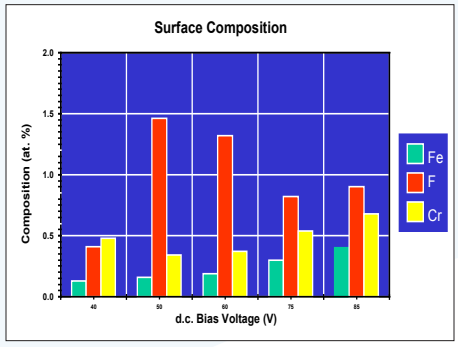
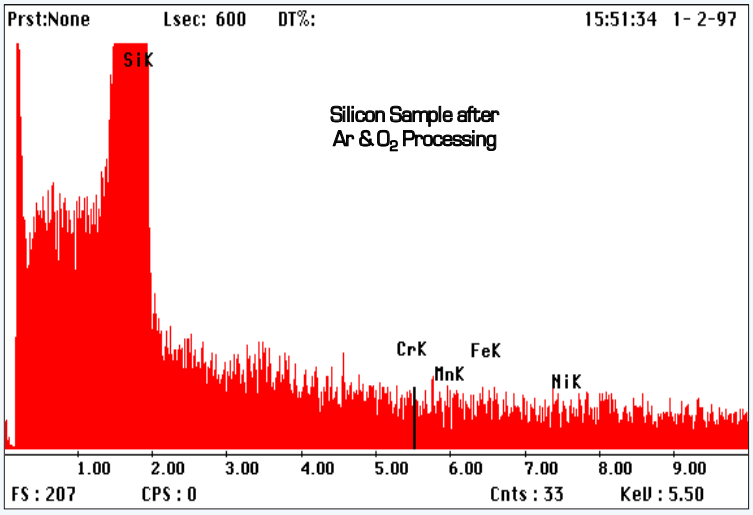


Fig. 5: Graph illustrating the composition of contaminants at various dc bias voltage values.



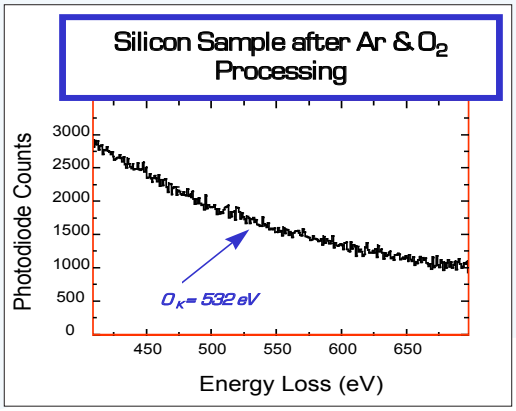


Fig. 6: XEDS and EELS spectra collected to show the lack of sputtering contamination present in the specimen examined.

Specimen Storage and Transfer

Once the samples have been cleaned, they can be inserted into an evacuated storage container for transport to the microscope. The specimen holders can also be stored in these optional containers for an indefinite period preventing further contamination. These aluminum containers will not outgas nor further contaminate the sample as may happen with polycarbonate containers.

SPECIFICATIONS PC-2000:

Power

RF: 13,56 MHz frequency, 150 watts forward power
Readouts: Digital LCD displays for forward power in watts
Power Controls: 1-150 watts forward power control
Indicator: LED On/Off indicator
Reflected Power Control: Manually adjustable, +/- 180 degree potentiometer
DC Bias: Digital LCD Display
Electrode: Immersed Stainless Steel 200mm diameter
Electronics: Solid State, crystal controlled, 100khz switchmode
DC Power Converter to RF generator
Termination: Digital interlocked timer or manual termination
Cooling: Water cooled
Interlocks: RF, Gas and vacuum fully interlocked for user & lab safety
Power Requirements: 110/220 VAC Single phase, 5 amps FCC Verified

Chamber

Size: 8" OD x 4" high stainless steel electropolished cylinder with viewport. Easily removed for cleaning
Vacuum Sensor/Readout: Capacitance manometer and digital LCD readout
Time to Reach Base Pressure: 30 seconds
Specimen Interface: "No Tools" quick connect for all side and top entry TEM holders. Top entry holders mounted through hinged top port
Gas Delivery and Control: Dual independent needle valves with safety interlock solenoid
Operating Pressure: User variable between 20-200 millitorr
System Vacuum Vent: Independent solenoid interlocked with connection for dry nitrogen

Enclosure

Construction: Heavy duty steel with baked hard coat paint
System Weight: less than 50 Lbs.
Dimension: 20.25" W x 13" D x 8.75" H

Pumps

Mechanical Pump: Corrosive series 2-stage rotary vane pump
Base Pressure: 2x10-2 torr
Pump Access: Fully accessible, remote mount, KF-25 connection, all hoses & filling provided
Activation: Front Panel LED "ON" indicator
WARRANTY: [2] Year Limited Warranty

SPECIFICATIONS PE-2000:

Vacuum Pump: 43l/min two stage direct drive corrosive series pump. System base pressure 20 millitorr; Typical operating pressure 50-200 millitorr
Vacuum Readout: Capacitance manometer with digital front panel display in millitorr
Vacuum Chamber: Quartz, 8" OD x 4" high
RF Power: User variable 0-150 watt; 13.56 MHz frequency; manual tune; air cooled; solid state design
Process Timer: Auto-termination of etch up to 99:99:59
Input Power: 115/230 VA, 50/60 Hz, 10/5 amps
Dimensions: 20.25" W x 16" D x 15" H
System Vent: Independent interlocked solenoid with connection for dry nitrogen
Gas Delivery: Two channels independently controlled with precision needle valves and positive off solenoid valves. Normally closed solenoids eliminate any gas flow in the event of power loss
Gas Control: Dual independent needle valves with safety interlocked solenoid
Sample Table: 6" diameter stainless steel
RF Readouts: Digital front panel LCD meters for Forward and reflected power
DC Bias: Digital/front panel LCD meter
System Weight: 60 pounds (without rotary pump)
Warranty: 1 year on parts and labor
Options: Stainless steel chamber with viewports; Mass Flow Controller System up to four channels; Cold Cathode Gauge.

PC-2000 References:

1. Surface Science Aspects of Contamination in TEM Sample Preparation, J.T. Grant, S.D. Walck, F.J. Scheltons, A.A. Voevodin, Materials Research Society Vol. 480 1997.
2. Application of Reactive Gas Plasma Cleaning in Mitigating Contamination of Specimens During Transmission and Analytic Electron Microscopy, Shane P. Roberts, Scott D. Walck, John T. Grant, Nestor J. Zaluzec, Materials Research Society Vol. 480 1997.
3. Reactive Gas Plasma Specimen Processing for use in Microanalysis and Imaging in Analytical Electron Microscopy, Nestor J. Zaluzec, Bernard J. Kestel, David Henriks, Microscopy & Microanalysis 1997.
4. Simultaneous Specimen and Stage Cleaning for Analytical Electron Microscopy, David Henriks, Microscopy Today issue 96-8 October 1996 pgs. 16-17.

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