Plasma Cleaning and Its Applications for Electron Microscopy

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Abstract: The effectiveness of applying a high-frequency, low-energy, reactive gas plasma for the removal of hydrocarbon contamination from specimens and components for electron microscopy has been investigated with a variety of analytical techniques. Transmission electron microscopy (TEM) analysis of specimens that have been plasma cleaned shows an elimination of the carbonaceous contamination from the specimen. With extended cleaning times the removal of existing carbon contamination debris due to previously conducted microanalysis is shown. Following plasma cleaning, specimens may be examined in the electron microscope for several hours without exhibiting evidence of recontamination. The effectiveness of plasma cleaning is not limited to applications for TEM specimens. Scanning electron microscopy (SEM) specimens that have been plasma cleaned likewise show an elimination of carbonaceous contamination. Furthermore, other electron microscopy parts and accessories, such as aperture strips, specimen clamping rings, and Wehnelts, among others, can benefit from plasma cleaning.

Key **words:** plasma cleaning, electron energy-loss spectrometry (EELS), energy dispersive X-ray spectrometry (EDS), specimen preparation, inductive plasma

INTRODUCTION

To achieve optimum results using electron microscopy, a sample that is both representative of the bulk and free of artifacts is imperative. There are several preparation methods used to produce high-quality specimens, with large, representative electron transparent areas for microanalysis (Schoone and Fischione, 1966; Goodhew, 1985; Benedict et al., 1987; Newcombe et al., 1988; Fischione et al., 1991; McCaffery, 1992; Hsu et al., 1997). However, current specimen preparation techniques (mechanical polishing, electrolytic thinning, ion beam milling) often introduce amorphous damage layers, residual surface layers, and specimen contamination (Bahnck and Hull, 1992). Specimen-borne contamination can severely limit the ability to perform accurate electron microscope analysis of materials, especially as specimen areas of interest decrease in size. To analyze smaller areas of interest, electron probe sizes have decreased, while currents in those probes have increased. The combination of these two factors results in an enhanced

migration of hydrocarbon contamination on the specimen surface to the impingement point of the electron probe. This enhancement is due to an increase in the local thermal and electronic gradients from the area of the specimen under the electron probe to the areas not being probed.

The amount of hydrocarbon contamination formed upon the sample is dependent upon five factors: the specimen preparation history of the sample, the sticking coefficient for hydrocarbons upon the specimen, the vacuum level of the microscope, the existing contamination within the microscope vacuum system, and the electron probe current density. Hydrocarbon contamination during specimen preparation may come from several sources: inadvertent touching of the specimen or specimen holder, backstreaming of oil from a diffusion-pumped ion-milling system, chemicals used during electrolytic thinning, or adhesives and/or solvents. Even when great care is taken to clean the specimen, standard methods are not fully successful in removing contamination from the sample (Hren, 1979).

Recently, it has been shown that processing of transmission electron microscopy (TEM) specimens and specimen holders in a non-equilibrium, cold gas plasma mitigates and even prevents this hydrocarbon contamination (Thomas, 1974, Zandbergen et al., 1994; Zaluzec, 1996; Fischione, 1997; Grant et al., 1997; Roberts et al., 1997; Zaluzec et al., 1997; Isabell and Fischione, 1998). In many reported instances, the plasma processing times are on the order of a minute or less (Dickey et al., 1997; Fischione et al., 1997; Isabell and Fischione, 1998). In this work, it is shown that plasma cleaning of specimens and specimen holders for short periods of time allows fine-probe TEM microanalysis for extended periods without carbonaceous buildup under the electron beam. In addition to the prevention of contamination, the removal of existing carbon contamination on the specimen by extending the plasma cleaning time is demonstrated.

The benefits from plasma cleaning are not solely limited to TEM specimens. In systems with appropriate chamber configurations, bulk specimens are readily introduced into the plasma for cleaning. These specimens can then be used for surface science or scanning electron microscopy (SEM) analysis without concern over carbon contamination of the specimen. This is especially important for low-voltage SEM in microscopes with field emission sources, whereby carbon contamination of the specimen often hampers high-resolution imaging and accurate energy dispersive X-ray spectrometry (EDS) analysis. Finally, by virtue of the ability to remove existing carbonaceous debris, plasma processing

is readily extended to the cleaning of specimen holders, apertures, specimen clamping rings, tweezers, and any other component where carbon contamination becomes an issue.

MATERIALS AND METHODS

Microscopy and Analysis

All specimens for electron microscopy were prepared using standard methods (bulk grinding, dimple grinding, ion milling, electropolishing, etc.). No extraordinary steps were taken to keep the specimen free of contamination during the preparation process, nor were any steps taken to purposely contaminate the specimen. TEM of 304 stainless steel and $SrTiO_3$ specimens was conducted with a Hitachi HF-2000 cFEG-TEM operated at 200 kV with a probe size on the order of 5 nm, probe current of 0.5 nA at the specimen, a chamber vacuum near the specimen of 1×10^{-8} torr, and a cooled LN_2 cold trap. Electron energy-loss analysis for the determination of specimen contamination layer thickness was done on the same microscope with a Gatan Model 666 PEELS and EMiSPEC analysis software. Microscopy and microanalysis of silicon was performed on a Philips CM200 FEG-TEM operated at 200 kV with a probe size on the order of 1 nm, probe current of 0.7 nA at the specimen, and a chamber vacuum on the order of 5×10^{-8} torr near the specimen. This microscope is equipped with an EDAX energy-dispersive spectrometry (EDS) system. Scanning electron imaging and microanalysis were conducted on a Hitachi S-4500-II FEG-SEM with a probe size on the order of 10 nm, probe current of 0.3 nA, chamber vacuum of 1×10^7 torr, and cooled LN_2 cold trap, and equipped with a NORAN Voyager EDS system. Other SEM analysis was performed on a Philips XL30 S-FEG with a probe size on the order of 10 nm, probe current of 0.3 nA, chamber vacuum of 1×10^7 torr, and an EDAX EDS system. Auger spectroscopy was performed on a Physical Electronics scanning auger microscope with a probe size on the order of 1 µm, probe current of 1.6 µA, and a chamber vacuum of 1×10^{-9} torr. All plasma cleaning was done in an E.A. Fischione Instruments, Inc. Model 1400 Plasma Cleaner.

Plasma Cleaning Mechanism

At sufficiently high temperatures, the molecules in a gas decompose to form a gas of atoms that move freely in random directions, except for collisions between atoms. If the temperature is further increased, these atoms then ionize into charged particles (electrons, neutrals, negative and

positive ions), and the substance is termed a plasma (Lieberman and Lichtenberg, 1994). In a non-equilibrium, high-frequency plasma, free electrons are accelerated to high velocities by an oscillating electromagnetic field. The mobile electrons are preferentially heated while the heavier ions remain at about the neutral gas temperature (Melliar-Smith and Mogab, 1978; Hasegawa et al., 1992; Lieberman and Lichtenberg, 1994). This occurs due to electron-neutral collisions within the plasma. In the presence of the plasma-generating electric field, the electrons with their smaller mass will be accelerated more than the other charged particles, and thus gain substantial energy between collisions. Assuming these collisions to be elastic, the electrons lose very little energy during collisions, but there is a large momentum change. Eventually, a steady state is reached in which the energy gained by the electrons between collisions is equal to the energy lost during the collision. Since the energy lost is a small fraction of the total energy gained, the electrons will heat up to a sufficiently high energy to establish the energy balance (Cecchi, 1990).

When suitable plasma processing parameters (chamber configuration, plasma chamber pressure, electrode configuration, and input power) are established, the ions accelerate through low-voltage sheaths toward the object being processed and possess an energy of between 10 and 15 eV. These sheaths are thin, positively charged layers that join the charged plasma to the grounded chamber walls and to the specimen and specimen holder.

In plasma processing, various process gases are used to induce chemical reactions with different materials on a specimen surface. An oxygen plasma is highly effective in removing hydrocarbons. The disassociated oxygen created by the plasma chemically reacts with the hydrocarbon material present on the surface, converting it into CO, CO_2, and H_2O which are evacuated by the vacuum system through an exhaust system (Tuszewski et al., 1995). Alternative gases can be substituted to assist in either removing other types of contamination or in selectively etching individual elements within a specimen.

For the removal of hydrocarbon contamination, a mixture of 25% oxygen and 75% argon was found to be suitable (Fischione et al., 1997; Isabell and Fischione, 1998). It was determined that 25% oxygen would be sufficient for the chemical removal of the hydrocarbons, while remaining less of a problem for laboratory safety as compared to pure oxygen. Following the selection of the 25% oxygen concentration, the balance gas was chosen. The requirements were for an inert gas that is easily ionized. Argon was selected
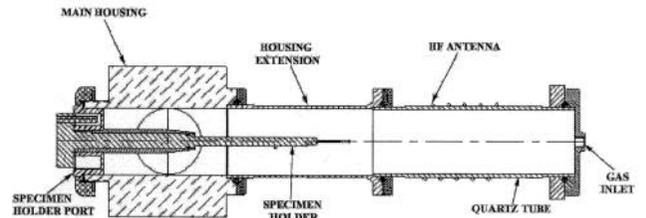

Figure 1. Plasma chamber assembly showing high-frequency (HF) antenna located external to the chamber.

since it does not chemically react with the specimen and is commonly used for the conventional ion milling of TEM specimens. Furthermore, compared to other inert carrier gases, argon has a smaller ionization energy and greater ionization efficiency, enhancing plasma creation (Wasa and Hayakawa, 1992). For the sake of convenience a blended oxygen/argon process gas, contained in a single cylinder, was used for general cleaning.

Plasma Chamber Design

For plasma processing, the gas plasma is created within a cylindrical chamber containing the object being plasma cleaned, as shown in Figure 1. A high-frequency (HF) antenna, located external to the chamber, inductively couples the oscillating electromagnetic field through the quartz chamber walls and generates the plasma. A critical aspect of this inductively coupled, low-energy form of plasma creation is that no components (i.e., electrodes) are located within the plasma, other than the objects that are being cleaned. The quartz tube chamber wall serves as a dielectric window and is coupled to the plasma through the thin, positively charged sheath. In this form of plasma generation, sheath voltages are sufficiently low whereby the plasma ions and electrons are accelerated to yield enough energy to catalyze the chemical removal of the hydrocarbon species without sputtering. This enables effective cleaning with negligible specimen heating and eliminates the sputtering of chamber material onto the specimen and specimen holder.

Sheaths are thin, positively charged layers that join the plasma to the chamber wall and specimen and specimen holder surfaces. Existing within the sheath regions are both electrons and ions. The electrons possess a thermal velocity 100 times greater than that of the ions. These fast-moving electrons within the sheath are lost to the specimen holder and chamber walls that are maintained at ground potential. Therefore, a positively charged layer is created near these
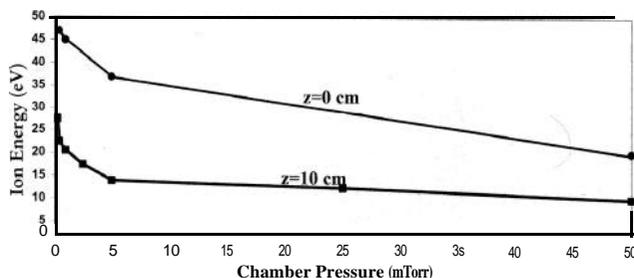
Figure 2. Ion energy as a function of chamber pressure for specimen positions corresponding to the center of the HF antenna coil (z = 0) and the plasma-cleaning position (z = 10). Typical plasma process gas pressure is 5-50 mTorr during plasma cleaning.

Figure 3. Ion density as a function of chamber pressure.

surfaces. In the inductive mode of operation, the heavier, slow-moving ions are accelerated toward electrically grounded surfaces at typical energies between 10 and 40 electron volts as the system proceeds toward thermal and electrical equilibrium.

When processing with an inductive plasma, lower ion energies result from avoidance of stray capacitive coupling from the HF antenna. Capacitive coupling yields the generation of higher-energy ions. Low-voltage ion creation in an inductively coupled plasma results in plasma densities roughly 5-10 times higher than in a capacitive discharge (Lieberman and Lichtenberg, 1994).

Plasma Parameter Settings

To optimize the plasma processing of TEM specimens and specimen holders, instrumentation parameters were developed to ensure the creation of appropriate ion energies, plasma density, and electron temperature. When it is required to remove carbonaceous contamination from the specimen, inductively coupled ion energies corresponding to sheath voltages of between 10 and 15 eV are needed. At this ion energy, only the carbonaceous contamination residing on the specimen and specimen holder is chemically removed. Sputtering of the specimen, specimen holder, or plasma chamber does not take place under these conditions.

Factors contributing to the experimental value ion energies are the gas pressure within the plasma chamber, the position of the specimen in proximity to the HF antenna, and the setting of the HF power supply output. With the specimen and specimen holder at the plasma cleaning po-
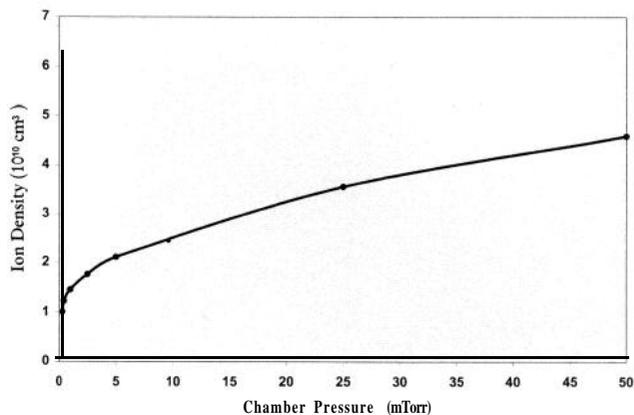
sition, and with typical process gas pressures of 5-50 mTorr, low ion energies in the range of 10-15 eV are attained. Figure 2 indicates the correlation of ion energy at the specimen with respect to plasma chamber pressure, where the typical specimen position for plasma cleaning is 10 cm (z = 10 on the figure) from the center of the antenna coil. These ion energies were determined by use of a Langmuir probe (Lieberman and Lichtenberg, 1994). Locating the specimen in the center of the HF antenna (z = 0) results in ion energies of 30-45 eV (see Fig. 2).

Decreasing the process gas pressure has the effect of decreasing the plasma ion density, as shown in Figure 3. A higher-density plasma not only provides a greater population of gas atoms to be ionized, but more importantly, provides a greater likelihood of an ion colliding with either another ion or a gas atom. As this occurs, the energy of the colliding ion is reduced, particularly in the high-density, low-energy inductive sheath regions that develop near the chamber wall, specimen, and specimen holder (Lieberman and Lichtenberg, 1994).

For the proper inductive plasma processing of TEM specimens, it is important that appropriate ion energies be established. With capacitive forms of plasma creation, it is possible for ion energies on the order of 100 eV to be created; however, these can significantly alter specimen properties. Since these higher ion energies are sufficiently high to break molecular bonds, sputtering will occur (Wasa and Hayakawa, 1992; Lieberman and Lichtenberg, 1994). In such high-energy plasmas, the various components in contact with the plasma, including the specimen, specimen holder, chamber walls, and plasma electrodes (in the case of an immersed electrode configuration), will have material
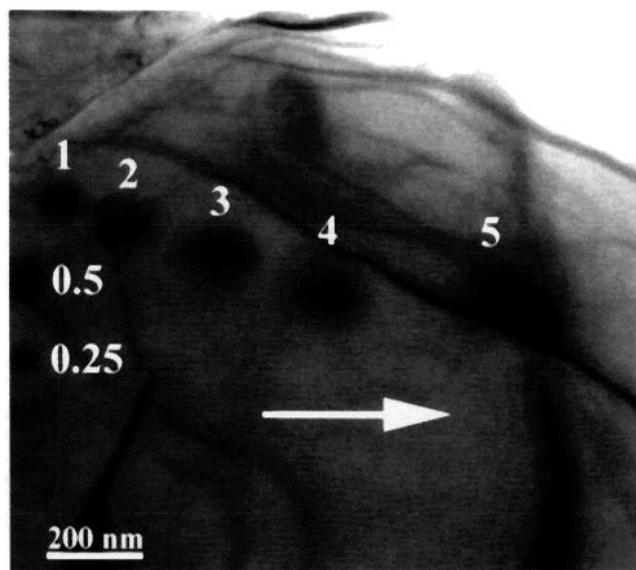
Figure 4. TEM micrograph of electropolished 304 stainless steel. The black spots are carbonaceous contamination formed prior to plasma cleaning by converging the electron probe on the specimen and holding it in place for the number of minutes indicated by the number by each spot. After **1** min of plasma cleaning, the probe was converged on the specimen and held for 5 min at the position indicated by the arrow.

Figure 5. EDS spectra of silicon, collected before (a) and after (b) 1 min of plasma cleaning.

removed from them, which may be redeposited onto the specimen (Roberts et al., 1997).

RESULTS AND DISCUSSION

Plasma Cleaning for TEM

To study the full effectiveness of plasma cleaning, experiments were conducted on a variety of specimens with various specimen processing histories. Different materials will have different sticking coefficients for hydrocarbons and thus will contaminate at different rates. Likewise, different specimen preparation techniques will affect the amount of latent hydrocarbon contamination resident on the specimen. For example, from an electropolished specimen, contamination could come from either the electrolyte used for thinning or the solvents used for cleaning the specimen after thinning. For ion-milled materials, contamination could result from the back-streaming of oil from an oil-pumped vacuum system. For our experiments we chose to study 304 stainless steel, silicon, and $SrTiO_3$ to encompass the analysis of metals, semiconductors, and oxides. The stainless steel was electropolished, whereas the silicon and
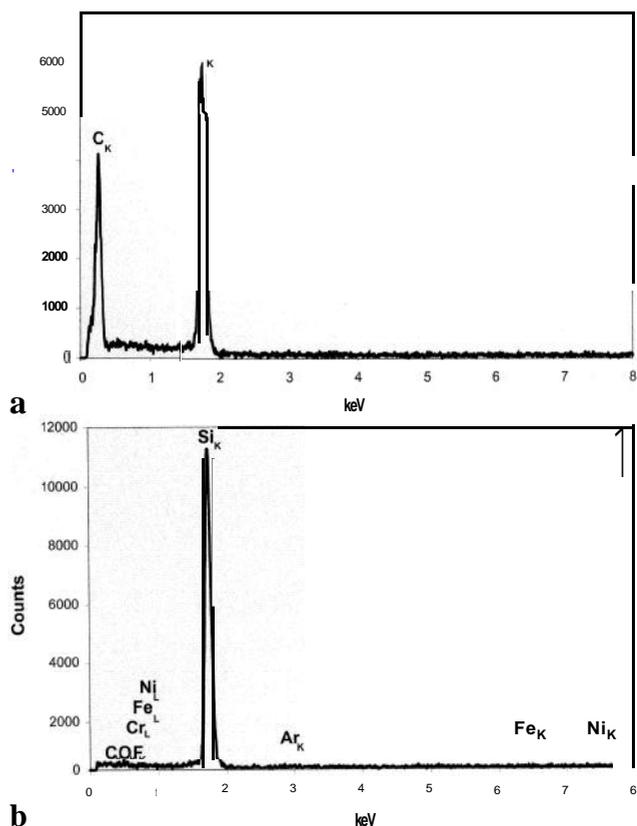
$SrTiO_3$ were mechanically polished, dimpled, and ion milled.

Figure 4 is a TEM micrograph of an electropolished specimen of 304 stainless steel. The black spots are carbonaceous contamination formed on the specimen prior to plasma cleaning by converging the electron beam at that position. The numbers adjacent to each spot indicate the length of time in minutes that the probe was converged and maintained at that position. After plasma cleaning for 1 min using a process gas of 25% oxygen in argon, the probe was converged and held for 5 min at the position indicated by the arrow in the figure. Following this probe exposure the micrograph was captured. No further contamination is evident.

In preventing the formation of contamination debris on the specimen surface, plasma cleaning also improves microanalysis. Figures 5a and b are EDS spectra (100 sec live time) from the silicon substrate of an ion-milled cross-section specimen of a silicon oxide film deposited on silicon, taken before and after plasma cleaning, respectively. The plasma cleaning duration was again 1 min, utilizing the

25% oxygen mixture. As determined from the spectra, no carbon is detected by EDS after plasma cleaning. Carbon contamination and buildup on the specimen have been mitigated. Furthermore, no oxygen or argon are detected, indicating no implantation of the process gas species. In addition, no iron, nickel, chromium, fluorine, or other elements used in the construction of the plasma chamber and specimen holder are detected within the limits of EDS microanalysis. This indicates the absence of sputtering and subsequent redeposition of the chamber and holder constituents onto the specimen.

To further analyze the surface composition of the specimen surface after plasma cleaning, Auger electron spectroscopy (AES) analysis was performed. The silicon/silicon oxide specimen was plasma cleaned for 10 min, removed from the cleaner, affixed to the holder for Auger spectroscopy, and analyzed at 3 kV with a probe size on the order of 1 μm and probe current of 1.6 μA Figure 6 shows the Auger spectrum, with silicon, carbon, and oxygen as the only species present on the specimen surface. The silicon and oxygen are specimen constituents whereas the slight amount of carbon detected is most likely due to the exposure of the specimen to handling in ambient conditions during transfer from the plasma cleaner to the Auger spectrometer (Grant et al., 1997). This small amount of carbon is not detectable with EDS or EELS in the electron microscope and therefore would not affect analysis.

Electron energy-loss spectroscopy (EELS) analysis is likewise affected by carbonaceous contamination of the specimen and specimen holder. EELS can be used to quantify the effectiveness of plasma cleaning. Specimen thickness at any time can be determined from an EELS spectrum through use of the following relation (Egerton, 1996):

$$t/\lambda = \ln(I_T/I_0) \tag{1}$$

where t if the specimen thickness, λ is the mean free path for energy loss, I_T is the total intensity under the EELS spectrum, and I_0 is the intensity under the zero-loss peak. As the electron probe is converged and held in place on a specimen, the only increases in specimen thickness will be due to an increase in the carbonaceous contamination under the beam. Thus, the relation in Eq. (1) becomes

$$t_s/\lambda_s + t_c/\lambda_c = \ln(I_T/I_0) \tag{2}$$

where the subscript s refers to the specimen thickness and mean free path, while c refers to the thickness and mean free
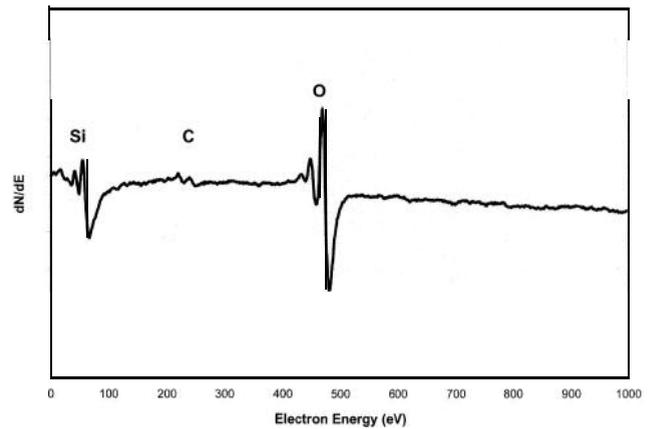

Figure 6. Auger spectrum of silicon collected after 10 min of plasma cleaning. No elemental constituents of the plasma chamber, vacuum system, or specimen holder are detected.
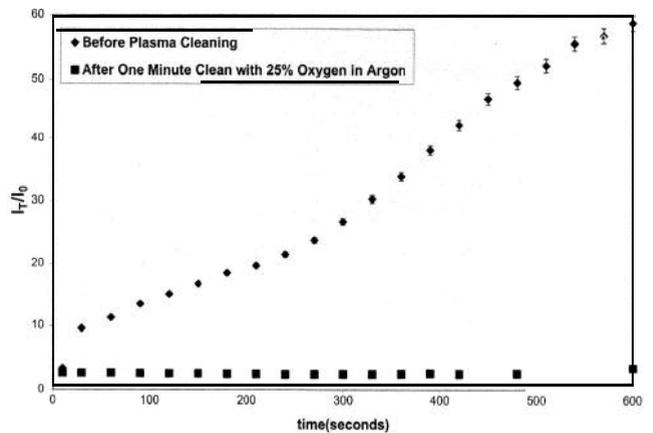

Figure 7. Ratio of integrated intensity of the whole spectrum to zero-loss intensity (I_T/I_0) for 304 stainless steel as a function of time before and after 1 min of plasma cleaning.

path of the carbonaceous contamination formed. Therefore, a measure of the increase in specimen thickness due to contamination buildup can be determined by studying the change in the ratio I_T/I_0 with time.

Figure 7 shows plots of the ratio I_T/I_0 as a function of time for electropolished stainless steel. The upper plot is that for the specimen prior to plasma cleaning, the bottom for the specimen after 1 min of plasma cleaning in the 25% oxygen-in-argon mixture. The ratio, and thus the specimen thickness, does not vary, even after the probe is held on the specimen for 10 min. No contamination is detected. Similar results were obtained for ion-milled $SrTiO_3$ and silicon.

Further investigations were conducted with the 304 stainless steel specimen to determine the effect of using the 25% oxygen-in-argon process gas versus a two-step opera-
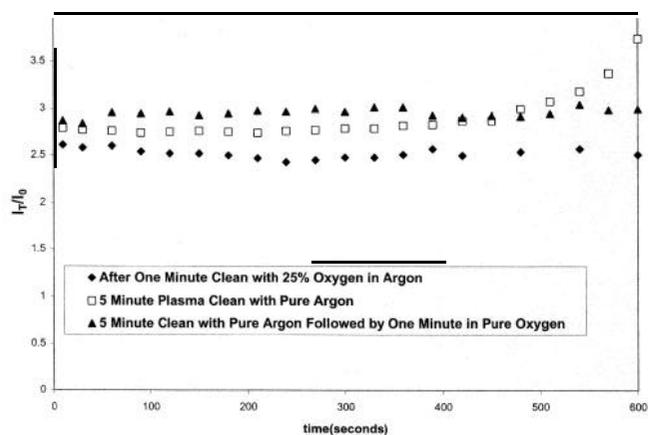
Figure 8. Ratio of integrated intensity of the whole spectrum to zero-loss intensity I_T/I_0 for **304** stainless steel as a function of time for different plasma-processing gases.

Figure 9. TEM micrographs of $SrTiO_3$ taken after 1 min **(a)** and 5 min (b) of plasma cleaning, showing the removal of existing carbonaceous contamination by extending plasma cleaning times.

tion employing pure argon and pure oxygen (Roberts et al., 1997). Figure 8 shows plots of the ratio I_T/I_0 versus time for the 304 stainless steel specimen for various plasma process gases. A 5-min plasma cleaning with pure argon initially slows specimen contamination, but with time the specimen will become significantly contaminated. Following 5 min of argon cleaning with 1 min of cleaning with pure oxygen eliminates contamination forming on the specimen. Likewise, as before, a single-step plasma cleaning process of 1 min in 25% oxygen in argon prevents carbonaceous formation completely. No advantage is seen to using a two-step process of pure argon followed by pure oxygen when compared to using a gas mixture of the two.

This result is contrary to that published in Roberts et al., who compared the two-step process of pure argon followed by pure oxygen to a one-step process using a 50/50 oxygen-argon mixture. Their results showed that the two-step process was more efficient at preventing contamination than the single step using the gas mixture. An explanation for this discrepancy lies in the creation of the plasma used for cleaning. Roberts et al. used a capacitively coupled, parallel plate geometry to form their plasma, whereas we created an inductively coupled plasma. These two forms of plasma creation differ most in their fractional ionization of the plasma, with inductively coupled plasmas being on average two orders of magnitude more ionized than capacitive discharges (Lieberman and Lichtenberg, 1994). This is important because the charged particles within the plasma play central roles in sustaining the discharge and in specimen processing. It is the electrons that dissociate the feedstock gas to create the free radicals and etchant atoms required for
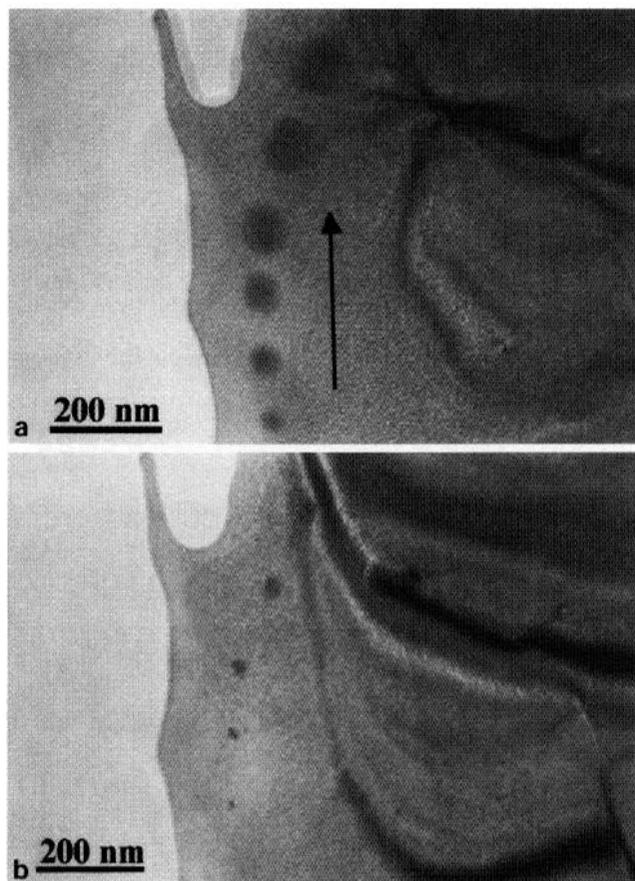
the chemical reaction at the specimen and specimen holder. Therefore, a higher fractional ionization results in more free radicals and etchant atoms, and thus a more efficient chemical reaction at the specimen surface.

In addition to preventing carbonaceous contamination from occurring, extending the plasma processing time can remove existing contamination debris. Figures 9a and b are TEM micrographs of $SrTiO_3$ taken after 1 and 5 min of plasma cleaning, respectively. The process gas used was again the 25% oxygen-in-argon mixture. As in Figure 4 for the stainless steel, the black spots in Figure 9a are from converging the probe at that position in a prior analysis. After 1 min of plasma cleaning, the probe was again converged for 5 min at the position indicated by the arrow in Figure 9a. No further contamination occurred, and the existing spots remained unaffected. After plasma cleaning for 4 more min (Fig. 9b), the existing contamination spots are reduced, or in some cases, removed. Additionally, in Figure 9a, an edge of carbonaceous contamination can be seen
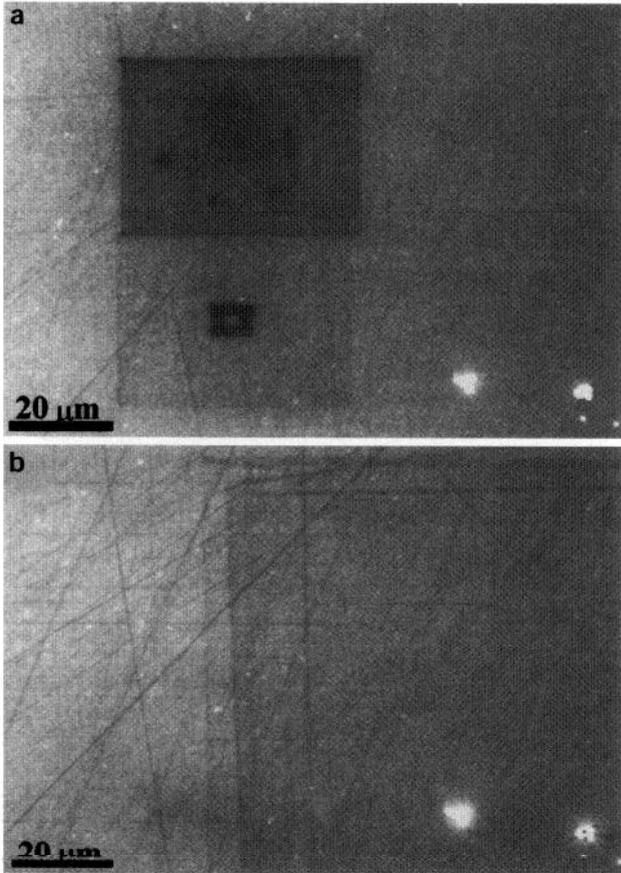
Figure 10. SEM images of silicon taken before (a) and after (b) plasma cleaning.

Figure 11. EDS spectra gathered by SEM of a silicon specimen before **(a)** and after **(b)** 2 min of plasma cleaning.

around the hole in the upper left portion of the image. After 5 min of cleaning, the edge is no longer present in Figure 9b.

Plasma Cleaning for SEM

The benefits of plasma cleaning are not solely limited to TEM. From Figure 1, it can be seen that bulk specimens can be introduced into the plasma for cleaning. Thus, specimens for surface science and SEM analysis can be cleaned as well as SEM specimen holders, aperture strips (Isabell and Fischione, 1998), tweezers, specimen clamping rings, and anything else that can be placed into the 52-mm inside diameter of the plasma chamber.

The effects of plasma cleaning on improving an SEM image are varied. Obviously, if the specimen being investigated is nonconductive, plasma cleaning will only enhance specimen charging through the removal of the conducting carbon contamination layer. Figure 10a is a SEM micrograph of silicon, showing black carbonaceous areas formed
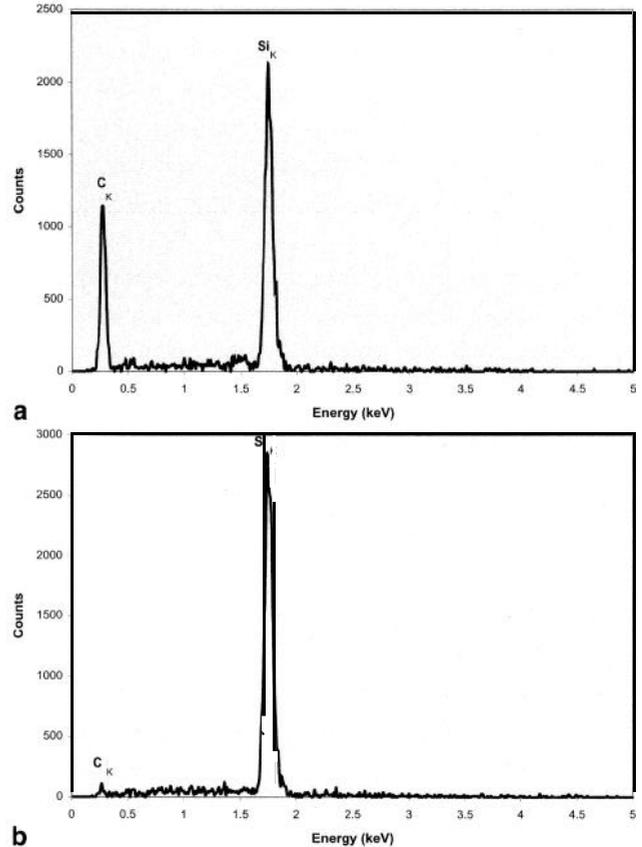
by converging the electron beam at that position for EDS analysis. The black box was formed by rastering the probe at that position while imaging the specimen at a higher magnification. Figure 10b is a SEM micrograph of the same specimen region, showing the elimination of all black spots and the black box after plasma cleaning.

Figures 11a and b are SEM EDS spectra of a bulk specimen of silicon before and after plasma cleaning. This experiment was conducted with the SEM operating in spot mode at an accelerating voltage of 5 kV with an accumulated live time of 300 sec. In Figure 11a, a large carbon peak is seen on the spectrum. After plasma-cleaning the bulk specimen for 2 min in the gas mixture of 25% oxygen in argon, the carbon peak is greatly reduced, but not eliminated (Fig. 11b).

CONCLUSIONS

Plasma cleaning is becoming an essential final step in the preparation of specimens for electron microscopy. The mi-

croscopist can conduct fine-probe analysis for extended periods of time without concern over carbonaceous contamination precluding imaging or microanalysis. A wide variety of materials, prepared by a number of specimen preparation conditions, can be plasma cleaned and yield identical results, with no contamination of the sample under the electron beam.

Simultaneous plasma cleaning of TEM specimens and specimen holders for times on the order of a minute prevents the formation of contamination debris under the electron beam. Furthermore, EDS analysis of plasma-cleaned specimens detects no carbon on the specimen, nor any elemental components of the plasma chamber or specimen holder as would be found if the process induced sputtering. Ion-energy analysis by Langmuir probe methods indicates that when utilizing processing parameters for cleaning, ion energies within the plasma are between 10 and 15 eV. This ion energy is below the sputtering threshold of most materials. Through the use of EELS, a better quantification of the effects of plasma cleaning can be determined by studying the effective specimen thickness as a function of time. On plasma-cleaned specimens, this thickness remains constant under the high-current density electron probe, indicating a complete mitigation of contamination.

Extended plasma cleaning times remove existing carbonaceous contamination. This expands the usefulness of plasma cleaning to surface science and SEM specimens. Plasma processing may also be used to clean aperture strips, specimen clamping rings, tweezers, and other microscopy accessories. SEM applications of plasma cleaning extend beyond cleaning carbonaceous debris. As in TEM, EDS analysis of plasma-cleaned specimens detects no carbon on the specimen.

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