

In-situ low-temperature (600 °C) wafer surface cleaning by electron cyclotron resonance hydrogen plasma for silicon homoepitaxial growth

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Abstract

Electron cyclotron resonance (ECR) hydrogen plasma was applied at 600 °C to in-situ clean the silicon surface of contaminants, such as oxygen and carbon species for subsequent low-temperature epitaxial growth. The standard condition with in-situ hydrogen plasma cleaning at 600 °C has been optimized and process variables such as d.c. bias, cleaning time, microwave power, and cleaning gas pressure were varied with respect to the standard condition. The epitaxial films, which were deposited in our chemical vapor deposition systems, immediately after these in-situ cleaning processes were characterized by cross-sectional transmission electron microscopy, etc. The role of a hydrogen ion in in-situ cleaning was clarified by investigating cleaning efficiencies for a variety of conditions. The hydrogen ion, which was produced by ECR plasma excitation, played a crucial role in removing surface contaminants in low-temperature (600 °C) in-situ ECR plasma cleaning processes.

Keywords: Chemical vapour deposition; Hydrogen; Plasma processing and deposition; Transmission electron microscopy

1. Introduction

As device dimensions are reduced into the sub-micron regime in pursuit of higher integration density and better circuit performance, low-temperature epitaxy has been a major issue in microelectronics and will be more critical in the future. Low-temperature processing, which includes low-temperature cleaning and low-temperature epitaxial growth, is very important for future silicon ultra large scale integration (ULSI) technology. Thermal exposure at high temperatures needs to be minimized to reduce the dopant diffusion and interface broadening [1] to obtain an abrupt, well-controlled dopant transition profile.

Thermal heating can achieve the surface cleanliness, especially at high temperature. With in-situ plasma cleaning, plasma energy can replace a considerable amount of thermal energy; it can be applied physically or chemically immediately before the deposition. In-situ Ar or He ECR plasma sputtering was applied in our system [2]. However, many defects were generated in the epilayer/substrate interfaces, where the substrates were submitted to ECR argon plasma bombardment prior to growth. A hydrogen plasma is reported to remove the native silicon oxide at high temperatures such as 800 °C [3] and even at lower temperatures [4] without causing much damage, due to its light mass and because hydrogen reacts chemically and removes the surface oxide

and hydrocarbons [5]. This issue is particularly important for our low-temperature epitaxial growth, because once the stacking faults were generated, defect-free epitaxial film could not be obtained.

The ECR plasma system was selected because it has some advantages compared to conventional radio frequency (r.f.) plasma systems. The electron cyclotron resonance (ECR) plasma system, which operates at a microwave frequency of 2.45 GHz, is able to deliver a higher density of low energy ions to the wafer, because both ionization (about 10% of the gas is ionized in our system) and the saturated ion current are much higher than in the r.f. plasma system. Particularly, the ECR plasma can be operated with low energy (without sacrificing cleaning efficiency) and ion flux and ion energy can be controlled easily [6]. Possible substrate damage may be reduced because of reduced plasma exposure time and less contamination may be expected due to electrodeless discharge [7]. ECR is compatible with ultra-high vacuum (UHV) chemical vapor deposition (CVD) systems.

It was analyzed and proved by measuring the O/Si ratio with X-ray photoelectron spectroscopy that exposure to ECR hydrogen plasma for a few minutes without applying heat or bias to the substrate removed the hydrocarbons and the native silicon oxide [5]. It is thought that this resulted in a hydrogen-terminated surface that was resistant to reoxidation. In our

studies, epitaxial layers were deposited at the cleaning temperatures (mostly 600 °C) and their structural qualities were investigated to evaluate the efficiency of the ECR hydrogen plasma cleaning processes.

2. Experimental

A multichamber single-wafer CVD reactor, located in a class 100 cleanroom in the TRL (Technology Research Laboratory) was used. The reactor was composed of four chambers: Load Lock, Analysis, CVD and ECR chambers. The main pumping unit was under the analysis chamber and consisted of a chemical resistive turbomolecular pump (Leybold Turbovac, $1\,500\text{ l s}^{-1}$) and a backing mechanical pump. The load lock chamber had its own turbomolecular pump (Alcatel CFF100, 100 l s^{-1}) and backing mechanical pump.

Substrates were 4 inch, czochralski-grown, p-type <100> silicon with $0.5\text{--}20\ \Omega\text{ cm}$ resistivity. The wafer was RCA cleaned and HF dipped for 20 to 30 s in 10:1 aqueous solutions. It was then rinsed in deionized (DI) water and dried by blowing nitrogen with the nitrogen gun inside the station. All the processes were performed inside the class 100 cleanroom and it took approximately 10 s to load the wafer into the load lock chamber, after the wafer was blow-dried. After the load lock chamber was pumped down to about 1×10^{-7} Torr, the wafer could be transferred to the analysis chamber by opening the gate valve between the chambers. Before the gate valve was opened, hydrogen gas was introduced and kept flowing through the CVD chamber, to avoid cross contamination. A magnetically coupled rod with a pincher held a wafer and it was transferred linearly to the CVD chamber, where it was unloaded to the heater stage. After the wafers were loaded onto the heater stage, the CVD Chamber was pumped down and ultimately $1\text{--}2 \times 10^{-8}$ Torr could be attained. It took about 7 min to heat the wafer to 600 °C, with the flow of the hydrogen gas.

In-situ predeposition cleaning was performed by using ECR hydrogen plasma at 600 °C. The ECR chamber was at the side of the CVD chamber. The ECR source was commercial ASTEX mirror magnets and the magnetic field was generated by the electromagnets around the ECR Chamber. The ECR system was operated at the 2.45 GHz S-band microwave frequency and resonance occurred in the region of 875 Gauss, where a high density of electrons and hydrogen ions could be produced. The window magnet was centered on the top flange of the ECR chamber and the bottom magnet was centered on the lower flange. Flux density was computed as a function of linear distance along the axis of the ECR chamber, starting at the face of the top flange (Fig. 1). Depositions were performed by flowing 10 sccm SiH_4 without carrier gases, immediately after the plasma was extinguished. Pure silane and house hydrogen were used for our experiments and these gases were passed through the Nanochem purifiers, which were known to be effective in reducing trace impurities [8].

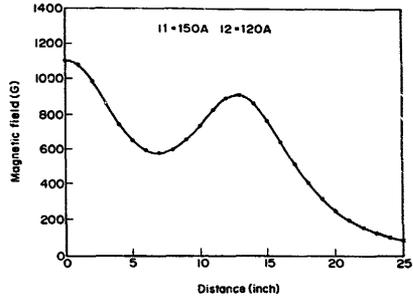


Fig. 1. Magnetic field profile in our two-magnet system. The currents were set at 150 A for the top magnet and 120 A for the bottom magnet.

Rutherford backscattering spectroscopy (RBS) was used to measure and determine the structural quality of the epitaxial films. The backscattering yield was obtained, when the 2 MeV He^+ beam was aligned along a channel direction. The structural quality of the epitaxial films could be determined by the ratio of the channeling yield of the film to the random yield (i.e. χ_{rma}). Cross-sectional transmission electron microscopy (XTEM) was used to observe the epitaxial layer and the epilayer/substrate interface. The TEM used was a model of JEOL 200CX with a LaB_6 filament and a line-to-line resolution of about 2.7 Å. The secondary ion mass spectrometry (SIMS) measurement was done in Evans East, New Jersey and a Perkin Elmer 6600 secondary ion mass spectrometer was used. A Cs^+ ion was used as the ion source for sputtering, the primary ion energy was 5 keV, and beam current was 200 nA. The detected elements included carbon and oxygen.

3. Results

The in-situ cleaning condition was optimized by the structural characterization of the deposited films. The XTEM micrographs were used to observe the images of the substrate and epitaxial layer and their interfaces. Among the process variables, the effects of microwave power, d.c. bias voltage, cleaning time, gas pressure and cleaning temperature were the main points to be investigated. Table 1 shows the summary of in-situ cleaning conditions in our experiments.

3.1. Condition

The in-situ cleaning process was optimized and the process was evaluated. Samples A and A⁺ were RCA cleaned and dipped for 20–30 s into 10:1 DI water:HF solutions, to remove the surface natural oxide. The wafers were rinsed in DI water for about 3 min, and were dried by blowing nitrogen. Sample A received the standard (optimized) in-situ cleaning, while Sample A⁺ did not. The standard condition corresponded to

Table 1
Summary of in-situ cleaning conditions

	Power (W)	D.c. bias (V)	Temp. (°C)	Time (min)	Pressure (mTorr)
Sample A	300	10	600	5	1
Sample B	300	0	600	5	1
Sample C	300	-50	600	5	1
Sample D	300	30	600	5	1
Sample E	300	10	600	2	1
Sample F	300	10	600	20	1
Sample G	150	10	600	5	1
Sample H	5-0	10	600	5	1
Sample I	300	10	600	5	8
Sample J	300	10	600	5	0.5

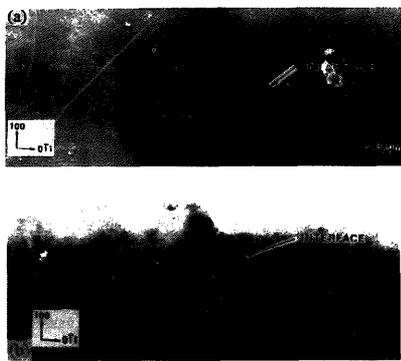


Fig. 2. (a) HRXTEM micrograph of an epitaxial film, cleaned in-situ with standard conditions at 600 °C. (b) XTEM micrograph of an epitaxial film, without in-situ cleaning (rinsed and blow dried).

Table 2
Summary of SIMS data on interfacial oxygen

	Oxygen (cm ⁻²)
Sample A ⁺	1.0 × 10 ¹⁵
Sample A	4.8 × 10 ¹³
Sample B	8.1 × 10 ¹⁴
Sample F	5.9 × 10 ¹⁴
Sample I	1.0 × 10 ¹⁵

the microwave power of 300 W, magnet currents of 150 A and 120 A for the top and bottom magnets, respectively. A positive 10 V d.c. bias was applied: the positive ions were repelled rather than accelerated across the sheath region, which means that the hydrogen ion energy was reduced. The cleaning was performed by flowing 20 sccm of H₂ at 1 mTorr, at 600 °C for 5 min. Deposition was done by introducing 10 sccm of SiH₄ at 1 mTorr and at 600 °C, immediately after the plasma was extinguished. Fig. 2(a) shows the high-resolution cross-sectional transmission electron microscopy (HRXTEM) micrograph of sample A. A defect-free epitaxial

layer and almost invisible epilayer/substrate interface were observed.

Fig. 2(b) shows the micrographs of Sample A⁺. Stacking faults were initiated in the epilayer/substrate interface. The thickness of the epilayer/substrate interface was about 100 to 150 Å. The interfacial oxygen concentration of Sample A⁺ was much higher than that of Sample A (Table 2). Therefore, the standard in-situ cleaning was effective in removing surface natural oxide, which was present on the wafer surface after ex-situ cleaning. Since Sample A⁺ did not receive an in-situ cleaning, i.e. surface contaminants, surface oxygen, should have caused the stacking faults.

In RBS channeling experiments, the X_{\min} of Sample A and Sample A⁺ was about 3% and 7%, respectively. Therefore, the structural quality of the epitaxial layer in Sample A was comparable to that of the substrate. Also in RBS channeling experiments, a detector with a high glancing angle (about 75°) was used to maximize the sensitivity in detecting heavy metal elements. For Sample A, no trace of such elements were found by this technique. This result is compared to an ECR Ar (argon) plasma cleaned sample [9] in our system, in which a considerable amount of heavy elements (Ar, Mo, Ta) were found in the epilayer/substrate interfaces.

3.2. Effect of d.c. bias

The substrate was either floated or d.c. biased in our experiments. $V_p - V_i$ is the difference between the plasma potential and the floating potential, and $e(V_p - V_i) - e(V_{d.c.})$ thus represents the ion energy in the presence of the substrate d.c. bias. Fig. 2(a), Fig. 3(a) and Fig. 3(b) are XTEM micrographs of Samples A, B and C and in these cases +10 V, 0 V (floated) and -50 V of d.c. bias was applied, respectively. The other process variables were the same. When increasing the hydrogen ion energy by the amount of 10 eV from the standard condition, the substrate was floated and the interface became clear and thick (about 30 Å) enough to be observed, while the defect-free epitaxial layer was deposited (Fig. 3(a)). It is interesting to observe that the surface of the 0 V cleaned epitaxial layer is a little bit curved compared to the +10 V cleaned one, in which case, the surface of the epitaxial film is quite smooth. SIMS data tell us that Sample

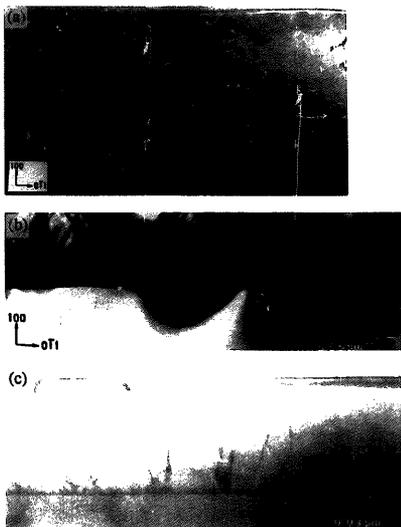


Fig. 3. XTEM micrographs of epitaxial films, cleaned in-situ at 600 °C with the d.c. bias set at (a) 0 V, (b) -50 V, (c) 30 V.

B has a higher oxygen concentration than Sample A in the epilayer/substrate interface (Table 2). An oxygen species may be present at the epilayer/substrate interface of Sample B. As the epitaxial layer grows to some thickness, the silicon around the second phase coalesces and forms some surface structures. This may happen without generating dislocations or stacking faults if the sizes and quantities of the second phases are small. Hydrogen ion bombardment may have generated damage to the substrate, and the hydrogen ion energy is transferred to the silicon substrate, rather than used in removing oxygen.

In Sample C, where -50 V of d.c. bias was applied to the substrate, the ion energy was increased by 60 eV with respect to the standard condition (Sample A). About a 300 Å thick layer of substrate was highly damaged and the substrate below this region was observed to be highly stressed (Fig. 3(b)). Dark/bright contrasted regions were observed in the substrate, and high densities of stacking faults and dislocations were present in the film. This observation can be related to the hydrogen ion bombardment, which was energized by applying a negative substrate d.c. bias.

Fig. 3(c) is a XTEM micrograph of Sample D and in this case, +30 V of d.c. bias was applied in its in-situ plasma cleaning and the other process variables were the same as the standard condition. The film was an epitaxial layer with a smooth surface, but it had a considerable amount of stacking faults which were initiated from the interface. Threading dis-

locations were generated in the epilayer/substrate interface and they existed throughout the film. It was surmised that oxygen or other contaminants could not be removed easily from the surface, because the ion bombardment energy was reduced by supplying a positive 30 V of d.c. bias. The substrate d.c. bias played a significant role in obtaining a damage-free, chemically clean interface [10].

3.3. Effect of cleaning time

Fig. 4(a) and 4(b) show the XTEM micrographs of Sample E and Sample F, which were cleaned for 2 min and for 20 min, respectively and the other process variables were the same as the standard condition. Fig. 4(a) showed broad (about 200 Å) interfaces, but a well-grown epitaxial layer. In some positions (not shown here), however, defects were present near the epilayer/substrate interface, which were regarded as oxygen-induced stacking faults or dislocations. Fig. 4(b) shows the HRXTEM micrographs of Sample F, which represent the well-grown epitaxial layer. This sample had a higher interfacial oxygen concentration than Sample A (Table 2). The interface looked discrete at some points but compared to Fig. 2(b), the interface was thicker (20–30 Å) and more distinct.

Cleaning for 20 min, did not improve the quality of the epilayer/interface that much. Clearly, the oxygen species accumulated in the epilayer/substrate interface, during the long-time cleaning for some reason; this could be proved by SIMS oxygen analysis (Table 2). Small quantities of process-generated oxygen containing species may have chemisorbed on the surface during exposure to the hydrogen plasma [11], and the bare silicon surface may have been reoxidized during exposure to the plasma. However, longer cleaning at

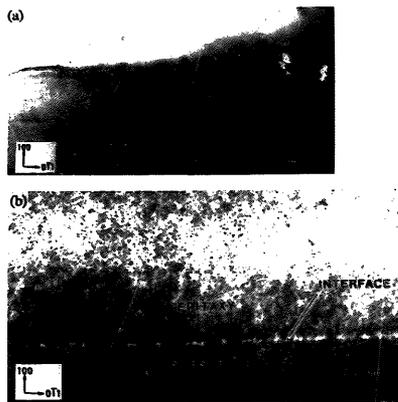


Fig. 4. (a) XTEM micrograph of an epitaxial film, cleaned in-situ at 600 °C for 2 min. (b) HRXTEM micrograph of an epitaxial film, cleaned in-situ at 600 °C for 20 min.



Fig. 5. XTEM micrographs of epitaxial films, cleaned in-situ at 600 °C with the microwave power set at (a) 150 W and (b) 540 W.

the standard condition did not induce significant damage to the substrate. During the in-situ cleaning process, there should be a competition between the silicon dioxide etching and the reoxidation of the silicon surface. The source of reoxidation is not clear but the internal surfaces of the stainless steel chamber were probably covered with layers of water molecules. These could be excited and desorbed by ECR plasma energy during the in-situ cleaning process and may have adsorbed on the silicon surface.

3.4. Effect of microwave power

Current literature reports that in an ECR-type plasma system, an increase of microwave power causes the growth of the level of microwave power absorption, which results in further ionization and an increase of plasma density and ion saturation current [12]. In an electron cyclotron resonance hydrogen plasma, an increase of net microwave power (0–1000 W range) causes an increase in the plasma density and ion saturation current. Delfino et al. [5] verified that in their ECR hydrogen plasma cleaning experiments, the oxygen removal rate turned out to be proportional to the microwave power. The hydrogen atom density was proportional to the net microwave power (i.e. applied microwave power – reflected microwave power).

In our studies, the net microwave powers varied from the standard condition; XTEM micrographs are shown in Fig. 5(a) and 5(b). Fig. 5(a) shows the micrograph of Sample G, where the net microwave power was 150 W. The micrograph shows a highly defective epitaxial layer. Stacking faults were formed in the epilayer/substrate interface, probably due to surface oxygen species. Sample H, where the net microwave power was about 540 W, shows a continuous interface and a defect-free epitaxial layer. Although high-microwave-powered in-situ cleaning proved to be effective in cleaning the substrate, it did not generate many defects in spite of its higher plasma density. Instead, the cleaning effi-

ciency became saturated as the net microwave power increased to 300 W and above. Delfino et al.'s cleaning experiments were performed at 2.5 mTorr and at room temperature, for 60 s. Two distinct regimes of oxygen removal were observed and there was a discontinuous transition at around 600 W. Our experiments were performed at 1 mTorr and at 600 °C, for 300 s, a condition which is favored in efficient cleaning compared to the Delfino et al in-situ cleaning conditions. In our case, a threshold power to obtain a defect-free epitaxial layer seems to have moved to a lower value.

3.5. Effect of hydrogen pressure

The mean free path of an ion, defined by collisions with neutral particles, is inversely proportional to the gas pressure [13]. Microwave plasma etching tends to be performed at lower gas pressure, because the mean free path of an ion becomes longer than the width of the sheath and the accelerated ions along electric fields can impinge on the substrate perpendicularly, without being scattered by other particles. This argument can be applied to our microwave plasma in-situ cleaning processes. In the r.f. plasma system, at a lower pressure of 10–100 mTorr, the energy-driven damage mechanism tends to be more important than the chemical etching and finally, at a very low pressure, below 10 mTorr, physical sputtering dominates [14]. In an ECR plasma system, ions are accelerated by low potential energy and both surface damage and contamination can be reduced. Therefore, in the ECR plasma system, the regime of chemical etching will expand compared to r.f. plasma systems, lower pressure is needed to turn the in-situ cleaning process into the physical sputtering regime. Delfino et al. observed the decrease of the O/Si ratio with the decrease of hydrogen pressure over the 0–25 mTorr range in their ECR plasma cleaning system. However, the oxygen removal rate was relatively insensitive to pressure up to 14 mTorr.

In our experiments, standard in-situ cleaning was carried out at the hydrogen pressure of 1 mTorr. Fig. 6(a) shows the XTEM micrograph of Sample I. In this experiment, in-situ cleaning was performed at 8 mTorr instead of 1 mTorr. Stacking faults were shown to be initiated in the epilayer/substrate interface and the thickness of the interface is around 100 Å. Table 2 shows the SIMS data of Sample I. High concentrations of oxygen were observed in the epilayer/substrate interfaces. Probably oxygen contaminants could not be removed easily in this case. Fig. 6(b) shows the XTEM micrographs of Sample J. In-situ cleaning at lower pressure (0.5 mTorr) was proved to be effective as well and its XTEM micrograph shows the continuous but relatively thin interface (around 20 Å) and defect-free epitaxial layer. It is reasonable to assume that the ECR hydrogen plasma cleaning in our experiments is not purely chemical in nature, because mainly ions, not neutrals, were involved in the cleaning processes.

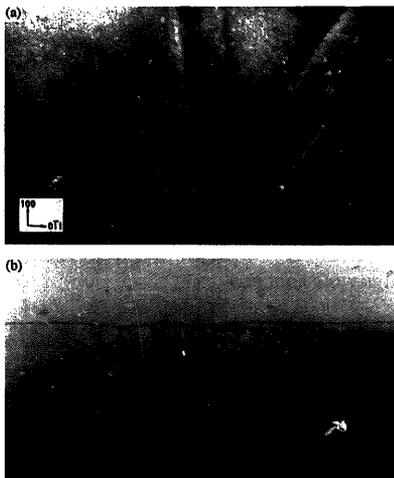


Fig. 6. XTEM micrographs of epitaxial films, cleaned in-situ at 600 °C with the pressure set at (a) 8 mTorr and (b) 0.5 mTorr.

4. Discussions

In-situ ECR plasma cleaning may proceed by physical sputtering or chemical reaction (etching) or ion-assisted mechanisms. Excessive hydrogen ion bombardment produced a highly damaged silicon surface [15] and in our experiments, applying -50 V of d.c. bias during in-situ cleaning damaged the substrate. A positive bias was needed to reduce the hydrogen ion energy [16]. In our experiments, reducing the hydrogen ion energy by supplying a positive d.c. bias resulted in growing a defect-free silicon epitaxial layer. It is surmised that hydrogen ions played a major role in surface cleaning because the cleaning efficiency was affected significantly by varying a substrate d.c. bias. It was also observed that in-situ cleaning at higher hydrogen pressure generated many defects in the epitaxial layer and a defect-free epitaxial film was deposited with the in-situ cleaning at pressures of 1 mTorr or less. In this low pressure, the probability of collisions with other particles are lower and the impinging ions may not have lost their kinetic energy. Therefore the ECR hydrogen plasma cleaning is more like an ion-assisted or physical sputtering process, rather than a pure chemical etching process. The effect of microwave power on the in-situ cleaning efficiency was not clear but ion density is reported to scale with microwave power [17]. Delfino et al. reported that more efficient oxygen removal occurred at a higher microwave power regime [5] and this trend has been proven in our experiments by real epitaxial depositions and structural analysis of the epitaxial films. The ECR hydrogen

plasma cleaning process is closely related to the behavior of a hydrogen ion, whether that hydrogen ion leads to reactions with some Si–O bonding configuration or whether it directly strikes the bonding configurations and breaks them by ion bombardment.

The kinetic energy of the ions can be obtained by summing the plasma potential: $e(V_p - V_f)$ and a negative d.c. bias energy: $e(-V_{d.c.})$. If the collision-cascade theory of physical sputtering holds, the threshold energy for physical sputtering is proportional to surface binding energy. Relative threshold energy (threshold energy/surface bonding energy) is a function of the mass ratio: the mass of incident ion to that of target atom. In case of SiO₂ physical sputtering with hydrogen ion, the relative threshold energy is approximately 4 [18]. The surface binding energy of SiO₂ can be derived from the sublimation energy (8.4 eV), it is 33.6 eV and in good agreement with the measured value (34 eV) in our system. Therefore, the threshold energy for physical sputtering is calculated to be about 134 eV (magnitude) in our case and if it is assumed that $e(V_p - V_f)$ is about 30 ± 5 eV for ECR hydrogen plasma [19], it is surmised that at least approximately -100 V of d.c. bias is needed for physical sputtering to be effective in our ECR hydrogen plasma cleaning system. In our experiments, ion energy was reduced by supplying a positive d.c. bias in optimizing the in-situ cleaning processes. So the physical sputtering cannot be a dominant process and since the hydrogen ion is doing a crucial role in surface cleaning, ion-assisted etching will be a dominant process. In this case an impinging ion damages the surface and increases its reactivity. Hydrogen neutrals or ions react with some Si–O bonding configuration and volatile species can be desorbed from the surface.

Reoxidation of a silicon surface may occur because the plasma may transform a portion of the residual water vapor into OH radicals which could adsorb on the silicon bare surface. In our experiments, longer (20 min) exposure to plasma did not improve the interface quality that much and oxygen clusters could be shown in HRXTEM micrographs (Fig. 4(b)). This observation may be explained by the reoxidation of a silicon surface during over-exposure to plasma. Water vapor, the most abundant source of oxygen, may exist in the form of residual gas inside the chamber and also may be present on the inner surfaces of the chamber wall.

Acknowledgements

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