

Ex situ wafer surface cleaning by HF dipping for low temperature silicon epitaxy

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Received 22 October 1996; accepted 26 February 1997

Abstract

Ex situ silicon wafer cleaning by HF dipping has been studied in terms of cleaning efficiency. Silicon epitaxial layers were deposited in the MC-CVD (Multi-Chamber Chemical Vapor Deposition) reactor, which had a Load Lock Chamber. In situ cleaning was performed with an ECR (Electron Cyclotron Resonance) hydrogen plasma exposure. The XTEM (Cross-sectional Transmission Electron Microscopy) was performed to investigate the structural qualities of the epitaxial layer and the epilayer/substrate interface. The SIMS (Secondary Ion mass Spectroscopy) was performed to investigate their interfacial carbon and oxygen concentrations. HF dipping without water rinsing, followed by thermal heating up to 660°C provided the best results in terms of contamination. Rinsing seemed to help the surface natural oxide grow, but it improved the surface smoothness of the epitaxial layer. Blow-drying by nitrogen was important in reducing the interfacial oxygen concentration of the deposited epitaxial layer. Spin-drying turned out to be efficient in removing hydrocarbons or organic species. The ex situ cleaning played a major role in reducing the amounts of surface contaminants. © 1997 Elsevier Science S.A.

Keywords: Epitaxy; Silicon; Surface and interface states; Transmission electron microscopy

1. Introduction

In the new ULSI (Ultra Large Scale Integration) era, the ultra-clean wafer surfaces represent particle free, metallic and organic contaminant free, and native oxide free surfaces. In addition, wafer surfaces need to be smooth and hydrogen-terminated [1]. The silicon bare surface is highly reactive and susceptible to impurity adsorption.

Before silicon epitaxial growth, two precleaning strategies were widely used. The first one used an oxidizing chemical clean to form a thin hydrophilic carbon-free oxide [2] which should then be removed in situ to expose the crystalline silicon surface for epitaxy. In the second strategy, a final strong acid dipping or exposure was used to create a hydrophobic hydrogen-terminated surface [3–5]. In our experiments, the second approach was chosen because the CVD Chamber in our system was not exposed to the atmosphere, and thus the protecting oxide was not needed, and because the protecting oxide could contaminate the CVD chamber. Thermal desorption could not be attained in this low temperature cleaning process; it was

more important to get a contamination-free, damage-free substrate with undestroyed hydrogen passivation.

HF cleaning is a method used to achieve clean interfaces, and it is most efficient in getting rid of residual natural oxide. Dangling bonds of silicon surface are terminated by hydrogen atoms. Fluorine atoms remain as a minor species after HF treatment. This hydrogen passivation prevents the surface from being exposed to air and to oxidation [6], but the effect of HF dipping on the carbon contamination on the substrate surface is not clear.

The last steps in wafer cleaning are rinsing and drying. Both are extremely critical steps because clean surfaces become recontaminated easily if not processed properly [7]. Rinsing after wet cleaning is done with flowing high purity and ultrafiltered high-resistivity deionized (DI) water, usually at room temperature [8]. Rinsing in DI water is important at all stages of the preparation; it removes species that are weakly bound to the surface (physisorbed or hydrogen bonded) and can even etch the surface [9]. Not only is deionized water rinsing applied during RCA cleaning steps to remove the residual chemicals, it is also employed especially after HF dipping to remove fluorine-species and to bring about hydrogen passivation.

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In this study, the cleaning efficiencies of the rinsing, the spin-drying and the blow-drying were evaluated. The effect of the air-exposure time before loading into the chamber was also considered. The ex situ cleaning procedure to grow device quality epitaxial films without applying the in situ cleaning process was explored.

2. Experimental

Substrates were 4 inch, CZ-grown, *p*-type $\langle 100 \rangle$ Silicon with a resistivity of 0.5–20 Ω cm. All the experimental processes were done in the TRL (Technology Research Laboratory), which is a class 100 cleanroom. The wafer was treated with RCA cleaning, and went through the 10% diluted HF dipping for 20 to 30 seconds to remove native oxide from its surface. The HF solution used was a ULSI grade 49% aqueous hydrofluoric acid. Deionized water was added to prepare a 10:1 solution by volume. Then some wafers were rinsed with deionized water for about three minutes and the other wafers were not. At the end of the water rinsing, the wafers were dried by blowing nitrogen or spin-dried. The spin/dry sequence was comprised of a 160 second DI water spin-rinse at 1000 rpm (revolution per minute) and a 240 second spin-dry in nitrogen at 2000 rpm. RCA-cleaned Teflon tweezers were used in handling the wafer, to prevent contamination.

An ex situ cleaned wafer was loaded into the Load Lock Chamber which was then pumped down to about 1×10^{-7} Torr. After the wafer was loaded onto the heater stage, the base pressure was measured to be around $1 - 2 \times 10^{-8}$ Torr. The wafer was then heated up to 600°C or 660°C in hydrogen flow with a pressure of 1 mTorr and a flow rate of 20 cm³/s. It took about 7 minutes to heat the wafer from 25°C to 600°C and took another minute to heat it from 600°C to 660°C. In some cases, in situ predeposition cleaning was done by using an ECR hydrogen plasma. Depositions were done by flowing SiH₄, immediately after the plasma was extinguished. In other cases, silane gas, without carrier gases, was introduced with a pressure of 1 mTorr and a flow rate of 10 cm³/s. As the in situ cleaning, 300 W of microwave power, 150 A/120 A of currents for the top and the bottom magnets, 20 cm³/s of hydrogen flow in 1 mTorr was applied. Cleaning conditions are listed in Table 1. The effect of the substrate temperature was not discussed here, but low temperature seems to provide the higher chance of the generation of stacking faults by its lower surface mobility. The growth rate turned out to be about 15 Å/min at 600°C and 31 Å/min at 660°C.

The evaluations of the ex situ/in situ cleaning processes were done by depositing epitaxial films on the substrate surface and performing material characterizations; XTEM (Cross-sectional Transmission Electron Microscopy) and SIMS (Secondary Ion Mass Spectroscopy). XTEM was used to observe the epitaxial layer and the

Table 1
Ex situ cleaning characteristics

	Rinse/dry	In situ cleaning	Deposition	Characteristic
Sample A	rinse/blow	none	600°C	–
Sample B	/blow	none	600°C	–
Sample C	rinse/blow	done at 600°C	600°C	–
Sample D	/blow	done at 600°C	600°C	–
Sample E	/blow	done at 600°C	600°C	HF/methanol
Sample F	rinse/spin	done at 600°C	600°C	–
Sample G	/spin	done at 600°C	600°C	–
Sample H	/blow	none	660°C	–
Sample I	–	none	660°C	–
Sample J	–	none	660°C	exposure to air
Sample K	rinse/spin	done at 660°C	660°C	–
Sample L	/spin	done at 660°C	660°C	–
Sample M	/blow	done at 660°C	660°C	–

epilayer/substrate interface. The TEM used here was the model of JEOL 200CX with a LaB₆ filament and a line-to-line resolution of about 2.7 Å. The SIMS measurement was done in Evans East, New Jersey and Cs⁺ ion was used as the ion source for sputtering. The detected elements included carbon and oxygen.

3. Results and discussions

When no in situ cleaning was applied at 600°C, the water rinsing step in the ex situ wafer cleaning process increased the thickness of the resulting epilayer/substrate interface. Samples A and B were RCA cleaned and dipped into 10:1 DI water:HF solutions. Sample A was rinsed in deionized water and dried by blowing nitrogen while Sample B was dried by blowing nitrogen without water rinsing. Both wafers were directly loaded into the Load Lock Chamber of our CVD system. Deposition was performed at 600°C. Fig. 1(a) shows the XTEM micrographs of Sample A. Stacking faults and dislocations were nucleated in the epilayer/substrate interfaces and the thickness of the interface was measured to be 100 to 150 Å. The SIMS analysis was done for Sample A and its data are in Table 1. The interfacial oxygen concentration was 1.0×10^{15} atoms/cm². The interfacial carbon concentration was 1.1×10^{14} atoms/cm². There was no ion bombardment, no chance of substrate mechanical damage and the stacking faults seemed to be oxygen-induced. The growth of the silicon film around the patches of oxide occurred as a result of the generation of stacking faults at the oxide-silicon boundary. Fig. 1(b) shows the XTEM micrographs of Sample B. The epitaxial layer on the substrate was almost defect-free and the thickness of the epilayer/substrate interface was measured to be 50 Å. The structural quality of the epilayer/substrate interface in Sample B was higher than that of Sample A. In Sample A, additional water rinsing assisted the growth of surface natural oxide on the silicon surface. In addition, the oxide could not be

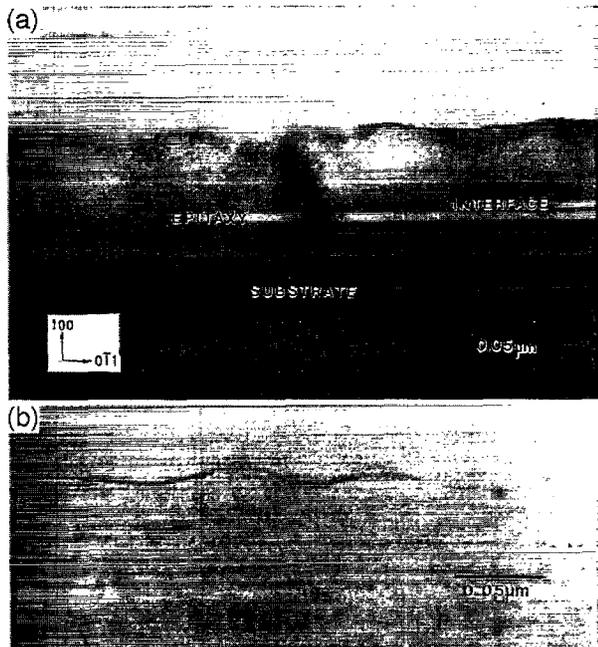


Fig. 1. (a) XTEM micrograph of epitaxial film, without in situ cleaning (Rinsed and blow-dried). (b) XTEM micrograph of epitaxial film, without in situ cleaning (blow-dried).

removed during heating to 600°C prior to epitaxial growth. However, interfacial carbon concentration was not affected by the water rinse. Sanganeria et al. reported that the Si–OH bonds on the silicon substrate were responsible for the increased oxygen at the epilayer/substrate interface in a water rinsed substrate.

The degree of surface smoothness of the epitaxial layer was observed to be improved by application of the water rinsing (Fig. 2). The surface roughness was defined as the average difference of the most thick and the most thin part in the 10,000 Å-interval of the deposited film. Rinsing in DI water has resulted in the planarization of the surface of epitaxial layers. HF dipping removes the surface native oxide, but the resulting silicon surface is prone to contamination. If the wafer is not rinsed after HF dipping, HF

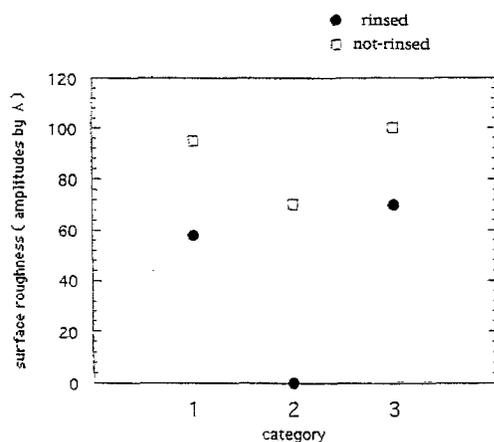


Fig. 2. Effect of water rinse on surface roughness.

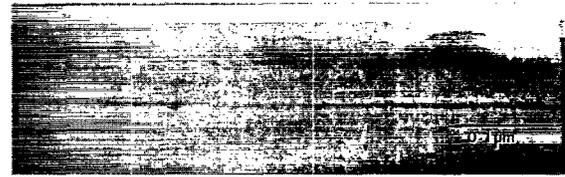


Fig. 3. XTEM micrograph of epitaxial film, cleaned in situ with standard condition at 600°C (rinsed and spin-dried).

solutions may stay on the silicon surface, producing Si–H and Si–F species [10]. The solution can be locally present on the surface. HF acid etching begins on a fluorine terminated surface. These F-terminated surfaces are unstable and are subject to further HF attack [11]. Surface silicon atoms can be removed locally as a form of SiF₄ and minute surface irregularities will be generated. Therefore, subsequently grown epitaxial layer may have rough surface. However, DI water rinsing may result in the elimination of HF solutions from the silicon surface and local etching will not proceed. The surface roughness could be induced by the growth process. However, in our set of experiments, an almost completely flat surface was found. The growth process itself seems not to induce the major surface roughness.

The spin-drying technique was compared to the blow-drying technique. Spin-drying was accomplished by the physical removal of the water rather than by allowing it to evaporate. Samples C, F, D and G were dipped into aqueous HF solution. In Sample C, the wafer was rinsed for 3 minutes and dried by blowing nitrogen on it. In Sample F, the wafer was rinsed for 3 minutes and spin-dried (160 seconds rinse/240 seconds dry). In Sample D, the wafer received a blow drying and in Sample H, the wafer received a spin drying, immediately after an HF dipping. The in situ cleaning process was applied at 600°C. Fig. 3 shows the XTEM micrographs of Sample F. The thickness of the epilayer/substrate interface was about 50 to 100 Å. In the SIMS analysis, the interfacial oxygen and carbon concentration of Sample F was 7.5×10^{14} atoms/cm² and 2.9×10^{14} atoms/cm², respectively. The carbon contamination was as much as that of Sample C and the oxygen concentration was higher than that of Sample C. The XTEM micrographs of Sample G (not shown here) showed the broad and contaminated interface (50 to 100 Å). In the SIMS analysis, the interfacial oxygen and carbon concentration was 1.5×10^{15} atoms/cm² and 1.7×10^{13} atoms/cm², respectively. By comparing Sample G with Sample D, the interfacial oxygen concentration of Sample G was higher than that of Sample D. For both cases, in comparison to blow-drying, spin drying did not reduce the surface contamination in our experiments and even degraded the quality of the epitaxial layer and its interface by providing more chances for oxygen adsorption. Hydrophobic surfaces after an HF dipping are susceptible to particle deposition [12]. Drying can be an important source of

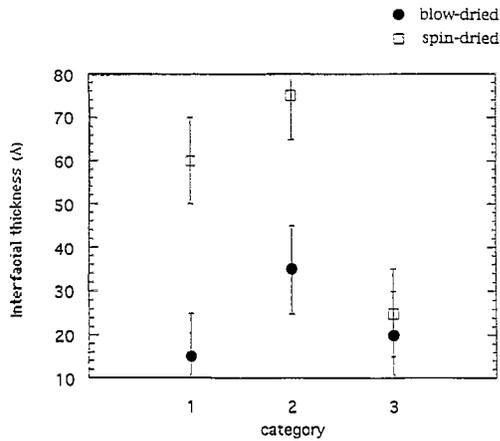


Fig. 4. Effect of drying technique on interfacial thickness.

particle deposition. Verhaverbeke et al. found that particles and metallic contamination depended on drying techniques [13]. The spin dry-rinse technique was more detrimental than the blow-drying technique in terms of contamination, in our experiments.

When the in situ cleaning was applied at 600°C and 660°C, broad and contaminated interfaces were observed in samples which were spin-dried (Fig. 4). A spin-drying step in the ex situ cleaning process helped to increase the oxygen concentration at the epilayer/substrate interface (Fig. 5(a)). Spin-drying process is comprised of spin-rinsing and spin-drying step. By applying spin-drying, 160

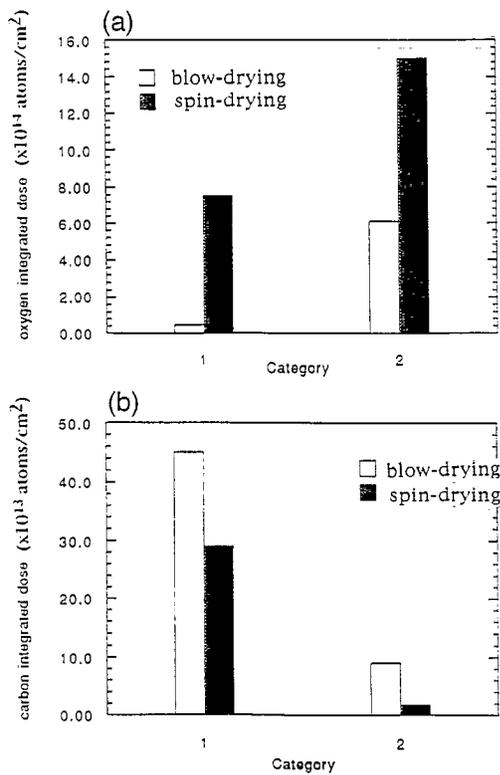


Fig. 5. (a) Effect of drying technique on interfacial oxygen concentration. (b) Effect of drying technique on interfacial carbon concentration.

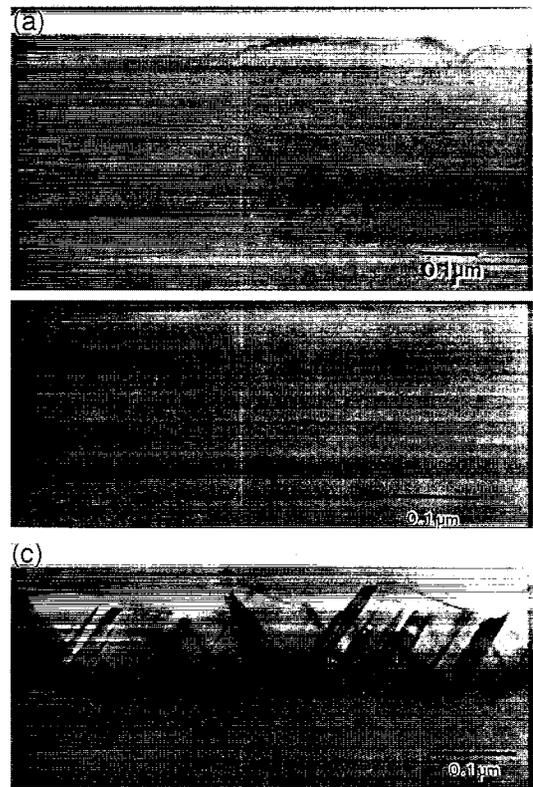


Fig. 6. (a) XTEM micrograph of epitaxial film, deposited at 660°C without in situ cleaning (blow-dried without rinsing). (b) XTEM micrograph of epitaxial film, deposited at 660°C without in situ cleaning (without blow-drying and rinsing). (c) XTEM micrograph of epitaxial film, deposited at 660°C without in situ cleaning (without blow-drying and rinsing). It was kept for 5 hours in cleanroom.

minutes of rinsing is added compared to blow-drying. In addition, rinsing water at high pressure will help increase surface native oxide on the wafer surface. However, it was shown that interfacial carbon concentration was reduced by the additional spin-rinsing (Fig. 5(b)), spin-rinsing turned out to be efficient in removing hydrocarbons or organic species. Hydrocarbons on the water surface may be removed by the physical force of rinsing water molecules or by dissolution in spin-rinsing step. Cleaning effects of running deionized water was reported on GaAs substrate in removing surface oxides [14].

Samples H, I and J were dipped into aqueous HF solution. Sample H was then N₂ blow-dried without water rinsing. In Samples I and J, the wafer was delivered directly into the Load Lock Chamber without blow-drying or water rinsing. In Samples H and I, the wafers were directly loaded into the Load Lock Chamber of our CVD system within 10 seconds. In Sample J, the wafer had been kept for about 5 hours in a cleanroom atmosphere before being loaded into the Chamber. Samples H, I and J were heated up to 660°C.

Fig. 6(a) shows the XTEM micrographs of Sample H. The base pressure was around 1×10^{-8} Torr and 1 mTorr and 20 cm³/s of hydrogen was kept flowing during heat-

ing. It took about 8 minutes to heat up to 660°C. At this temperature, the deposition was started. The XTEM micrograph showed a defect-free epitaxial layer with an almost invisible interface. SIMS data are listed in Table 2, the oxygen concentration of the epitaxial film was $1 - 2 \times 10^{18}$ atoms/cm², which was comparable to that of czochralski-grown substrates. Interfacial oxygen concentration at highest point was about 9×10^{18} atoms/cm². The interfacial concentration was one order of magnitude higher than those of the substrate and the film. The integrated dose of oxygen at interface was 1.4×10^{13} atoms/cm².

Fig. 6(b) shows the XTEM micrographs of Sample I. Defect-free epitaxial layers were observed and the thickness of the epilayer/interface was measured to be about 80–90 Å. The interfacial oxygen concentration of Sample I was 5.2×10^{14} atoms/cm² and higher than that of Sample H. Sample I was exposed to the atmosphere for a shorter exposure compared to Sample H. Interfacial carbon concentration of Sample I was a little bit higher than that of Sample H. From the previous studies, a large amount of organic contaminants were on the surface after HF treatment because the purity of the aqueous HF solution was not sufficient [15].

The XTEM micrographs of Sample J are shown in Fig. 6(c) and a highly defective epitaxial layer was observed. SIMS data for Sample J was shown in Table 2. Sample J had a much higher oxygen level (one order of magnitude higher) in the interfaces. The oxygen concentration in the film was also higher. It was shown from the XTEM micrographs that there are major differences in the structural quality of the epitaxial layer as a result of keeping it in the cleanroom environment for long periods. Fig. 6(c) represents the well-grown oxygen-induced stacking faults. This can be confirmed by the very high interfacial oxygen concentration of Sample J. So, natural oxide seems to be grown further in the cleanroom atmosphere. Furthermore, these oxygen species could not be desorbed easily under the vacuum condition (1×10^{-8} Torr) inside the CVD chambers [16]. The growing and thicknesses of the native oxide in clean room air at room temperature is reported

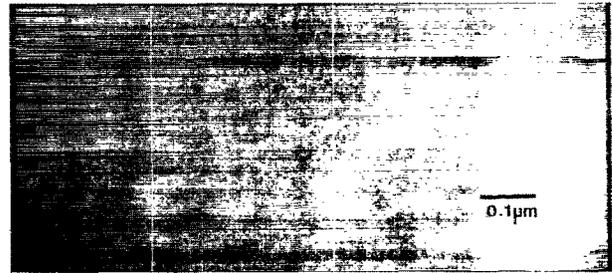


Fig. 7. XTEM micrograph of epitaxial film, cleaned in situ with standard condition at 600°C (methanol/HF solution was used, blow-dried without rinsing).

[17]. It was about 2 Å up to 200 minutes; it increased to 4 Å in 5 hours.

When no standard in situ cleaning was applied at 660°C, omitting both the blow-drying and water rinsing steps was not an effective cleaning technique for subsequent epitaxial growth. It means that blow-drying is one of the essential steps in reducing surface contaminants after HF dipping. Usually, drying should be done after rinsing to physically remove the water on the wafer surface. In our case, wafers did not receive the water rinsing, however, HF aqueous solution already contains water and additional water can be generated by the reaction: $\text{SiO}_2 + 6\text{HF} \rightarrow \text{H}_2\text{SiF}_6 + \text{H}_2\text{O}$ as long as the etching continues. From SIMS data (Table 2), the wafer surface which did not receive the blow-drying contained higher carbon contaminants, although the difference is small compared to oxygen contaminants. When no standard in situ cleaning was applied at 660°C, blow-drying (by nitrogen) without a water rinsing step turned out to be an effective process in reducing the interfacial oxygen concentration and getting a discrete epilayer/substrate interface (Fig. 6(a)).

An alcohol/HF combination was reportedly more efficient in surface cleaning than the water/HF combination [18]. In Sample E, a 10:1 methanol:HF solution was used to remove the surface natural oxide. The wafer was then dried by blowing nitrogen on it and the standard in situ wafer cleaning process was applied at 600°C. Fig. 7 shows the XTEM micrograph of Sample E, where the thickness of the epilayer/substrate interface was about 15–20 Å. Sample E can be compared to Sample D; the surface smoothness of the epilayer became higher, by replacing DI water with methanol. Rinsing in DI water has resulted in the planarization of the surface of the epitaxial layer. In Sample D, by omitting the rinsing step, the surface of the epitaxial layer became roughened. However, we found that a smooth surface was obtained by replacing water with methanol, without applying the water rinsing. Because the addition of alcohol is surmised to lower the surface energy of the HF solution [18], a droplet of the solution has a smaller contacting angle on the silicon surface. Therefore, the HF solution with methanol would have a higher ability to spread onto the wafer surface and, as a result, would attack the surface oxide evenly.

Table 2
SIMS data for interfacial contamination

	Oxygen (cm ⁻²)	Oxygen (cm ⁻³)	Carbon (cm ⁻²)	Carbon (cm ⁻³)
Sample A	1.0×10^{15}	7×10^{20}	1.1×10^{14}	1×10^{20}
Sample C	4.8×10^{13}	7×10^{19}	4.5×10^{14}	5×10^{20}
Sample D	6.1×10^{14}	1×10^{20}	9.1×10^{13}	3×10^{19}
Sample F	7.5×10^{14}	6×10^{20}	2.9×10^{14}	3×10^{20}
Sample G	1.5×10^{15}	8×10^{20}	1.7×10^{13}	1×10^{19}
Sample H	1.4×10^{13}	9×10^{18}	6.7×10^{13}	1×10^{19}
Sample I	5.2×10^{14}	2×10^{20}	9.0×10^{13}	3×10^{19}
Sample J	1.8×10^{15}	2×10^{21}	1.4×10^{13}	1×10^{19}
Sample K	1.8×10^{14}	5×10^{19}	9.6×10^{14}	2.5×10^{20}
Sample L	1.0×10^{14}	3×10^{19}	1.7×10^{14}	7×10^{19}

Surface native oxide has been known to be removed by hydrogen pre-bake at high temperatures. Kobayashi [19] studied on thermal cleaning at 760°C and 900°C without hydrogen flowing, the reaction product was identified as SiO. Racanelli et al. revealed that hydrogen bake aided the removal of surface oxygen and that higher temperature bake (795°C) was more efficient in their UHVCVD system. Meyerson also used high temperature bakes to remove surface native oxide in an hydrogen ambient [20]. They suggested that a dominant etching reaction should be: $\text{Si(s)} + \text{SiO}_2 \rightarrow 2\text{SiO(g)}$. It can be assumed that other reduction reactions by hydrogen may occur and in addition to that, hydrogen was necessary for the first reaction to proceed because silicon can be etched in the presence of oxidizing species. The possible thermal etching reactions in hydrogen ambient at relatively low temperatures ($\leq 660^\circ\text{C}$) in our system were listed and their free energies of reactions were presented [21]. By considering free energy changes, in reaction 1: $\text{Si(s)} + \text{SiO}_2 \rightarrow 2\text{SiO(g)}$, $\Delta G_{660^\circ\text{C}} = 319,433 \text{ J}$ and $P_{\text{SiO(eq)}} \cong 10^{-6} \text{ Torr}$. In reaction 2: $\text{SiO}_2(\text{s}) + \text{H}_2 \rightarrow \text{SiO(g)} + \text{H}_2\text{O(g)}$, $\Delta G_{660^\circ\text{C}} = 305,059 \text{ J}$. If it is assumed that $P_{\text{H}_2} = 1 \text{ mTorr}$ and $P_{\text{H}_2\text{O}} = 10^{-8} \text{ Torr}$, $P_{\text{SiO(eq)}} \cong 6.3 \times 10^{-10} \text{ Torr}$. This reaction may occur slightly. However, Reaction 1 occurs in the same system and thus Reaction 2 can be neglected. It was shown that in our system, Reaction 1 is a dominant etching reaction and by comparing Sample H with Sample B, interfacial oxygen concentration was reduced by heating from 600°C to 660°C in hydrogen ambient (1 mTorr). Reaction 1 occurred and SiO(g) was generated and desorbed at 660°C. Thomas et al. found that SiO(g) was desorbed at 700°C [22]. Ishizaka et al. [23] suggested that the thermal etching process will be conducted in two steps, the reaction and the migration of SiO through the oxide film. They found that the migration of SiO through the oxide film was not a rate-determining step when the oxide film was very thin.

4. Summary

Ex situ wafer surface cleaning for low temperature silicon epitaxial growth has been investigated and characterized. In particular, the effects of HF dipping, water rinsing, blow drying and spin drying were studied in terms of the interfacial quality (interface thickness) and the carbon and oxygen contamination. HF dipping was critical in removing the surface natural oxide. The subsequent water rinsing helped the natural oxide to grow, but it seemed to prevent the surface irregularities and helped to generate the smoother surface of the deposited film. Replacing the water with the methanol in HF dipping was also effective in preventing the surface irregularities.

Blow-drying by nitrogen was efficient in reducing the interfacial oxygen contaminants and in reducing the interfacial thickness. Spin-drying, which includes the additional rinsing, was efficient in reducing the interfacial carbon concentration. By simple calculation, $\text{Si(s)} + \text{SiO}_2 \rightarrow 2\text{SiO(g)}$ could be a dominant cleaning reaction, when no in situ cleaning was applied.

Acknowledgements

This work was funded by SEMATECH.

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