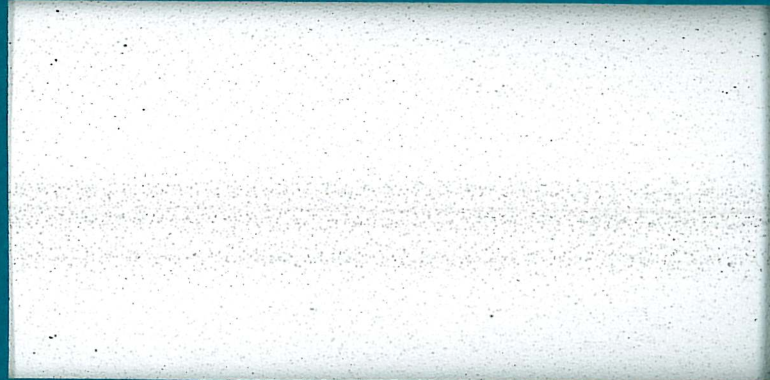

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by

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Sub Monolayer Silicon Oxide Growth Rate versus Oxygen Concentration in UPW

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Abstract

The growth rate of ultra-thin sub-monolayer "native oxide" on a HF cleaned silicon (100) surface during a ultra-pure water (UPW) rinse was studied at room temperature. The study was accomplished by stripping the oxide layer and analyzing it for Si using Inductively Coupled Plasma Optical Emission Spectroscopy (ICP-OES). It was demonstrated that the rapid acid etching method developed in our laboratory coupled with ICP-OES is capable of detecting and clearly distinguishing the difference in native oxide growth within a monolayer range with a possible 0.2 Å resolution. The initial results obtained indicate that the native oxide growth is significantly affected by both the exposure time of a silicon surface to UPW and the dissolved oxygen concentration in UPW. The significance of control of the dissolved oxygen concentration in UPW for advanced ULSI processes was demonstrated in a post-UPW rinse polycrystalline silicon deposition process.

Introduction

With advances in ULSI miniaturization and high-level integration, "native oxide" formation on a silicon surface and interface control are becoming critical to device performance[1-3]. Native oxide not only degrades thickness and quality of thin gate oxides but also provides an unfavorable increase in contact resistance for metal/Si contact formation in via holes[4-5]. Existence of native oxide on silicon surfaces also greatly affects the quality and deposition processes of polycrystalline silicon and dielectric thin films[4-6]. Selective chemical vapor deposition (CVD) and atomic layer epitaxy are especially susceptible to surface heterogeneity caused by "native oxide" growth[7]. In addition, electrical characteristics of MOS field-effect transistors are also found to be adversely affected by disorder of the oxide-semiconductor interface[4-7]. In order to improve device performance and reliability, it is becoming ever more crucial to prepare a well-defined, native oxide-free, and atomically flat surface prior to various advanced ULSI processes including ultra-thin gate oxide growth[1-7].

Preparation of a hydrogen-terminated silicon (Si-H) surface by HF acid etching followed by a UPW rinse to remove the chemically combined fluorine is an example of use of wet chemical pretreatment to prepare such a clean surface[8]. This pretreatment terminates the surface with a monolayer of hydrogen and passivates the chemically cleaned reactive surface to suppress the native oxide growth. It will be shown that there is an urgent need to have an analytical technique capable of quantitatively monitoring ultra-thin (e.g. < 1 nm) "native oxide" growth during various wet processes.

The native oxide has been studied by X-ray photoelectron spectroscopy (XPS, also known as ESCA)[9-10], Auger electron spectroscopy (AES)[11], Rutherford backscattering (RBS)[12], secondary ion mass spectrometry (SIMS)[13], spectroscopic ellipsometry (SE)[14], high resolution cross-section transmission electron microscopy (TEM) [15], and Fourier transform infrared reflection absorption spectroscopy - attenuated total reflection (FTIR-ATR)[16]. Among these techniques, TEM, SE, and XPS were found to be able to measure native oxide thickness less than 3 nm[17]. TEM offers the only true measure of oxide thickness because it makes no assumption about density. However, TEM is not able to measure ultra-thin native oxide with a film thickness less than 1 nm. SE is only suitable for homogenous smooth reflective surfaces and oxide thickness greater than 2 nm[18]. XPS was found to be sensitive enough to measure differences in native oxide growth on films less than 1 nm. Unfortunately, there has been considerable disagreement in XPS measurements among different laboratories[19]. The constants used to calculate the oxide thickness in XPS measurements were found to be sample-dependent. The calibration methods used were inconsistent. In addition, the "native oxide" may continue growing during the wafer delivering and analysis processes. Shive and his coworkers[20] have developed a method of native oxide thickness measurement using Colorimetry. They used an acid drop to extract the oxide layer to stop the growth of native oxide and then analyze HF strippable silicon from a wafer surface colorimetrically. The molybdenum-blue method they used was found to be extremely sensitive for silicon measurement down to sub ppm level and can also be standardized against a Si standard. However the molybdate complexation was found to be affected by solution pH and the presence of phosphate (PO_4^{3-}) and fluoride (F^-) in the sample solution[20].

This paper focuses on the use of inductively coupled plasma optical emission spectrometry (ICP-OES) for characterization of ultra-thin native oxide growth. Our approach is to use an acid drop etching procedure developed in our laboratory to rapidly extract the ultra-thin oxide layer and then measure the silicon concentration in the resultant solution directly without any chemical pre-treatment. The initial work obtained on 200 mm p-type Si (100) wafers shows that this method is able to quantitatively detect the difference in native oxide growth rate on films less than 5 Å (< approximately a monolayer) with a possible 0.2 Å resolution. The method can also be used for characterizing both smooth and rough silicon surfaces and both ultra-thin and thick oxide films.

Experimental

Analytical Instrumentation. All analyses were performed using a Model IRIS ICP-OES (Thermo-Jarrell Ash, Franklin, MA), equipped with a 27 Hz Ar ICP source and a charge-injection device (CID) solid detector. The operating conditions for the ICP-OES include a forward RF power of 1.15 kW, a reflected power < 5 W, an argon coolant flow rate of 16

L/min, auxiliary flow rate of 5 L/min and nebulizer flow rate of 0.9 L/min. The silicon concentration in the sample solution was determined at Si (I) 251.6 and 288.2 nm lines. The amount of dissolved oxygen in the UPW used was determined colorimetrically using a R-7501 (0-1.0 PPM) and a R-7512 (1-12 PPM) CHEMets self-filling ampoules (CHEMetrics, Inc., Calverton, VA).

Reagents, Standards, and Wafers. Hydrofluoric acid (HF) used through this work was of MB electronics grade (Ashland Chemicals, Columbus, OH). The UPW used was in-house high purity deionized water with 18.2 MΩ resistivity. A 10000 PPM (µg/ml) single-element standard of Silicon, (National Institute of Standards and Technology, Gaithersburg, MD) was used as the stock standard solution. Calibration standards were prepared by serial dilution of the stock standard and by adding the appropriate amount of HF so that the final concentration of HF matches with the sample solution (4%). The silicon wafers used in this work were polished 200 mm p-type wafers with > 5 Ωcm resistivity. The crystal orientation of the wafers was <100>. Prior to the UPW rinse, the wafers were chemically cleaned by immersing in 4% aqueous HF solution for 5 min to remove the thermal oxide on the surface. The oxygen saturated UPW used in this work, unless otherwise noted, contains approximately 6 ppm dissolved oxygen.

Sample Preparation. Experiments were designed and performed to assess the ultra-thin native oxide growth on a HF treated silicon surface during a very short UPW rinse. In practice, the samples were prepared in the following manner: Before each experiment, a wafer was always cleaned by immersion in 4% aqueous HF solution for 10 min. The wafer was taken out the 4%HF bath, immersed in a UPW bath for couple of seconds to remove residue HF on the surface, then quickly transferred to another UPW bath for rinsing experiment at various times. With a 4% HF, the oxide layer formed during the UPW rinse was stripped off of the wafer being converted into an aqueous solution. The resultant solution was then directly introduced, via a conventional nebulizer without any chemical pre-treatment, into the ICP for silicon determination.

Results and Discussion

Effect of UPW Rinsing Time. The thermal oxidation of the silicon surface at elevated temperatures in either dry oxygen (> 20 nm) or steam can be characterized by the Deal and Grove relationship[21]. However, the formation of native oxide on silicon surface at room temperature has not been well understood. Ohmi[1-2] has studied the growth of native oxide during a UPW rinse within a relative long period of time (e.g. 1 hour and 60 days) using XPS. His work indicated that native oxide was formed on silicon surface during a UPW rinse process due to the presence of dissolved oxygen. He also found that the thickness of native oxide on a n-type Si (100) surface increased with increasing the concentration of dissolved oxygen and the exposure time of a wafer surface to UPW. The surface roughness was also gradually increased[1-2].

Our work intended to focus on the effect of short UPW rinse (e.g. ≤ 10 minutes) on the growth rate of native oxide so that the initial stage of the native oxide formation can be studied. A plot of the silicon concentration found in the 4% HF stripping solution versus oxygen saturated UPW rinse time, ranging from 0.0 to 10.0 minutes, is given in Figure 1. The silicon concentration was typically measured by ICP-OES at Si (I) 251.6 nm line. The silicon wafers used in this study were 200 mm p-type with > 5 Ωcm resistivity. The crystal orientation of the wafers was <100>. The amount of dissolved oxygen in the UPW used was measured to be 6 ppm.

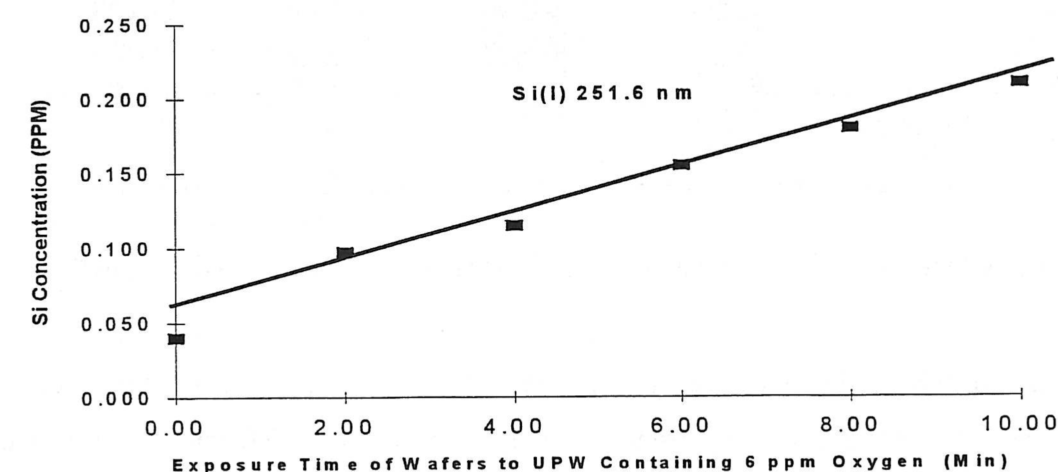


Figure 1. The effect of UPW rinsing time on the native oxide growth at room temperature.

As seen in Figure 1, the amount of silicon found in the HF stripping solution increases almost linearly with increasing exposure time of wafers to oxygen saturated UPW. This rate behavior is different from the parabolic relationship obtained by Ohmi and his coworkers[1-2, 22] in their relatively long time rinsing studies, suggesting that the oxidation mechanism may be different during short time frame of an UPW rinse that is typical for the wafer cleaning processing today.

Figure 2 shows the behavior of silicon concentration obtained by ICP-OES as a function of even shorter UPW rinse time within 1.5 minutes. The similar rate behavior was observed with the silicon concentration being increased as the exposure time of the wafer to oxygen saturated UPW is increased. The reagent blank was found to be low compared to the silicon concentrations in the etching solution and does not show evidence of any interference. However, it was found that the silicon concentration obtained at 0.0 minute rinse time was unexpectedly high.

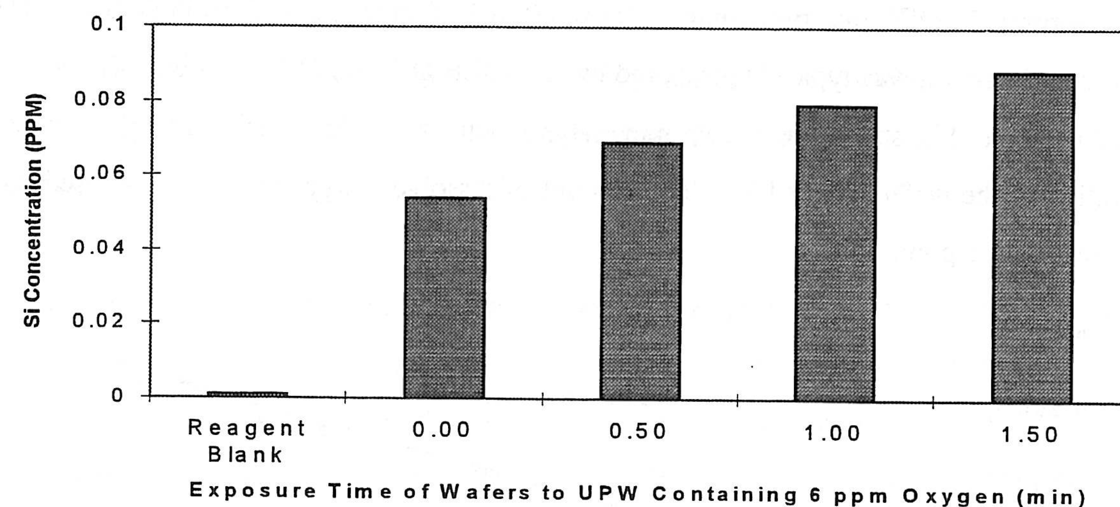


Figure 2. Early time study showing the effect of UPW rinse on the native oxide growth.

Root Cause of High Silicon Signal at Zero minute UPW Rinse. Although it can be subtracted as a method blank, the substantial Si signal observed at 0.0 minute rinse would somewhat restrict the detection limit of silicon for native oxide thickness measurement and may also lead to an overestimation of native oxide thickness. The ICP-OES spectral

interference was first investigated by simultaneously monitoring the Si (I) 212.4 nm, 251.6 nm, and 288.1 nm lines. The indifferent results obtained suggest that the high silicon results were not caused by spectral interference. The reagent blank obtained, as shown in Figure 5, was extremely low indicating that there was no noticeable silicon contamination from the reagents, the centrifuge tubes, the container used for HF stripping, and laboratory environment. Suspecting that some "native oxide" may be quickly formed during the initial 2 seconds UPW rinse for the purpose of removing residue HF and any dissolved silicon on the surface after the HF dip (See Sample Preparation in Experimental Section), a true zero minute UPW rinse was studied. A wafer taken out of the HF bath was immersed in another clean 4% HF bath for 5 seconds then quickly stripped and analyzed. Figure 3 shows a comparison of the result obtained with 5 seconds UPW rinse. It can be seen that the silicon concentration obtained with 5 seconds HF rinse (true zero minute UPW rinse) is still substantially high and comparable with that obtained with 5 seconds UPW rinse, indicating that the amount of native oxide formed during the initial 5 seconds UPW rinse is minimal. The high silicon result was likely due to the dissolution of silicon substrate when the HF was used to strip oxide layer for sample collection.

A possible explanation of the dissolution of silicon substrate is that a slow oxidation of hydrogen-terminated and passivative surface followed by a fast removal of surface oxide by HF during the stripping. The oxidation-dissolution processes could occur during and after ultra-thin native oxide is removed. Higashi and coworkers[24] used dilute HF solution to prepare a hydrogen terminated silicon surface and found that the diluted HF solution induced microscopic roughness on both Si (111) and Si (100) surfaces while concentrated HF solutions do not alter the surface morphology. Their results suggested that the microscopic surface roughness was due to the dissolution of silicon surface in diluted HF solution. Since HF acid itself does not etch the silicon surface, the H_2O and/or dissolved oxygen in diluted HF solution would have to be responsible for the oxidation[23]. This is further supported by the silicon dissolution studies done by Ogawa and coworkers[24] on both Si (100) and Si (111) surfaces using FT-IR-ATR. Their results seem to indicate that the dissolved oxygen in HF solution is responsible for the oxidation of silicon surface.

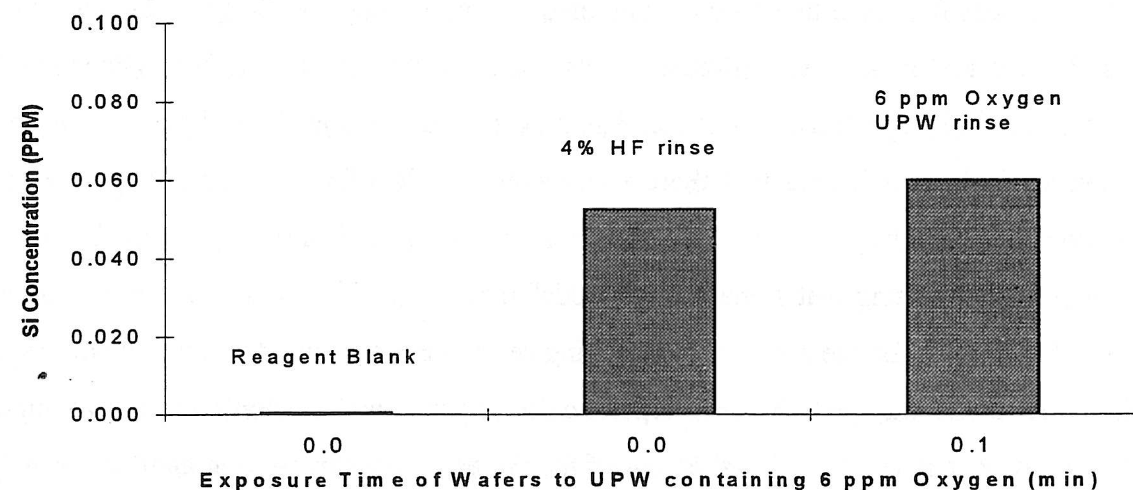


Figure 3. Investigation of root cause of high Si blank.

Given that silicon substrate is continuously dissolved in diluted HF solution used to strip oxide layer, this phenomenon subsequently leads to a overestimated silicon reading for the native oxide measurements. Since the concentration of HF stripping solution used and stripping time are fixed, the amount of stripped silicon from silicon substrate is expected to be constant. Therefore, the silicon concentration observed at 0.0 minute UPW rinse can be subtracted as a method blank from all other obtained results. To reduce the silicon dissolution in the HF stripping solution, the concentration of dissolved oxygen in HF solution should be decreased. It may also be helpful if the etching time is shortened.

Conversion of Silicon Concentration to Oxide Thickness. The conversion of stripped Si concentration to oxide thickness was found to be difficult due to the lack of the enough information on the structure and density of the ultra-thin native oxide formed. The literature[25-27] has indicated that ultra-thin native oxide (10-100 Å) formed on silicon surface is essentially a heterogeneous mixture of "SiO" and "SiO_2". At the interface of Si-SiO_2 structure, oxides thinner then 20 Å are not fully stoichiometric, and there is a transition region in Si-SiO_2 structure. The XPS studies of the 2p electron spectral line of silicon atoms showed oxide has the form SiOx with x increasing from 0 to 2 as the distance from the Silicon surface increases up to 20 Å[27]. One way of estimating the thickness of the native oxide is to use surface

concentrations (atoms/cm^2) by assuming that a monolayer (~ 4 Å) of oxide has an approximately 1.0×10^{15} atoms/cm^2.

The surface concentration of silicon in atoms/cm^2 was calculated based on solution concentrations of stripped silicon, volume of the stripping solution, atomic mass of silicon, Avogadro constant (6.023×10^{23}), and surface area of the wafers (314 cm^2 for 200 mm wafers). However, the surface concentrations of SiO and SiO_2 were calculated based on the theoretical mole ratios. Suppose the form of "native oxide" on silicon surface is SiO and one mole of silicon is bonded with one mole of oxygen. The total number of the atoms (Si + O) in monoxide form should be twice the number of oxidized surface silicon atoms. For example, if the calculated silicon surface concentration is 2.0×10^{14} atoms/cm^2, the SiO surface concentration will be 4.0×10^{14} atoms/cm^2. If the form of the native oxide is completely SiO_2 and one mole of silicon is bonded with two moles of oxygen (mole ratio is 1:2), the SiO_2 surface concentration will be 6.0×10^{14} atoms/cm^2.

Figure 4 shows the surface concentrations of extractable silicon and reported as SiO, and SiO_2 as a function of exposure time of a wafer to oxygen saturated UPW. Although the form of native oxide is unknown, it is clearly evident that the analytical method described in this work is able to monitor and distinguish the difference in ultra-thin native oxide growth. Since SiO_2 is the extreme, the results elucidated in Figure 4 suggested that the thickness of the native oxide formed on a p-type Si (100) surface resulted from a $\leq$ 10 minutes of oxygenated UPW rinse was less than a monolayer. By assuming, for purpose of calculation, the native oxide to be fused quartz with a density of 2.20 gram/cm^3 as other researchers have done[20], the silicon concentration can also be converted to oxide (SiO_2) film thickness in Å (See Table I).

However, the film thickness calculated in Å do not match the thickness the surface concentrations calculated in atoms/cm^2 imply. For example, the SiO_2 surface concentration resulted from the 10 minute oxygenated UPW rinse indicated an approximately one monolayer (~ 4Å) oxide formed on silicon surface while the calculated thickness based on the fused quartz (SiO_2) was only 1.32 Å. This discrepancy indicated that the assumption of fused quartz was wrong. The density of the native oxide with a thickness less than a monolayer must be

less than 2.2 gm/cm^3. Furthermore at the interface, the ultra-thin native oxide may have large quantities of =Si=H3 and ≡Si-H bonds[23] and may have an average form of SiOx with x between 0 and 1, e.g. Si_2O, without considering the hydration. This seems to be in close agreement with the results described by Deal and Kao using a pulsed laser atomic probe[3].

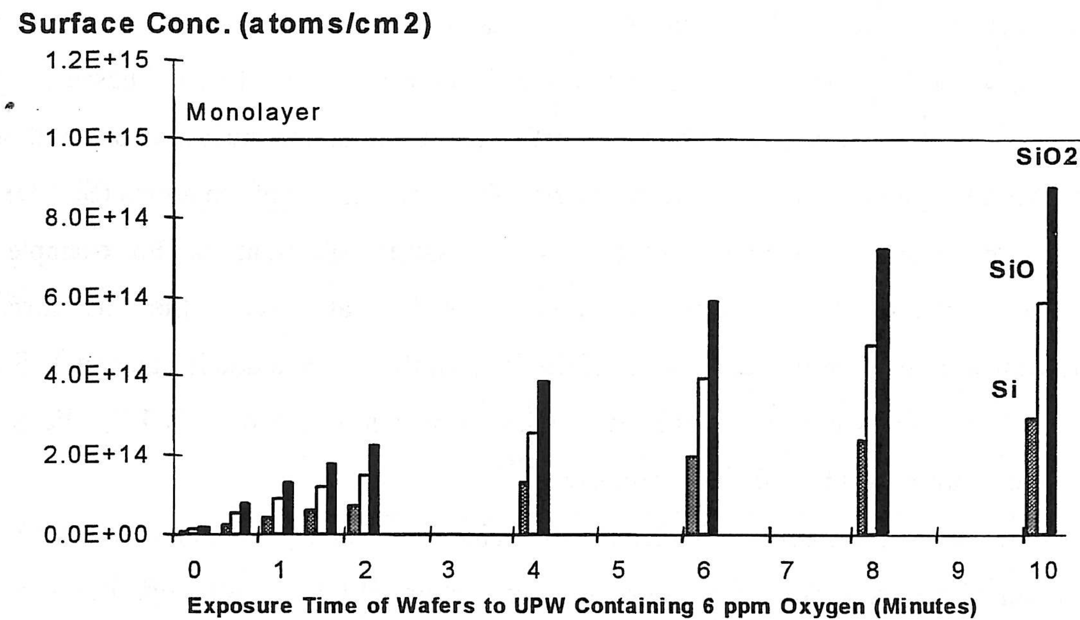


Figure 4. Surface concentrations of Si, SiO, and SiO_2 versus UPW rinsing time.

Table I. Native oxide thickness as a function of oxygenated UPW rinsing time

UPW Rinse (min)	Solution (ppm)	Si Surface (atoms/cm^2)	SiO Surface (atoms/cm^2)	SiO_2 Surface (atoms/cm^2)	SiO_2 (angstromÅ)
0.0	0.001	1.71.E+12	3.41.E+12	5.12.E+12	0.01
0.5	0.015	2.56.E+13	5.12.E+13	7.68.E+13	0.12
1.0	0.026	4.35.E+13	8.70.E+13	1.31.E+14	0.20
1.5	0.035	5.89.E+13	1.18.E+14	1.77.E+14	0.27
2.0	0.044	7.42.E+13	1.48.E+14	2.23.E+14	0.34
4.0	0.075	1.28.E+14	2.56.E+14	3.84.E+14	0.58
6.0	0.115	1.96.E+14	3.92.E+14	5.89.E+14	0.89
8.0	0.140	2.39.E+14	4.78.E+14	7.17.E+14	1.09
10	0.170	2.90.E+14	5.80.E+14	8.70.E+14	1.32

* The thickness were calculated based on the assumption that the native oxide formed on the surface were completely SiO_2 and the density of the oxide were 2.2 gram/cm^3. The extraction volume was 25 ml and the surface area of the 200 mm wafer was 314 cm^2.

A Case Study. In a fab, engineers were depositing polycrystalline silicon on a bare silicon surface in two different locations. Prior to the deposition, the wafers were treated with dilute HF, rinsed with UPW and dried. The equipment used to clean the wafers and to deposit the poly was exactly the same in both locations. However, the end result of the poly deposition was quite different with one location producing useless product. The size of the grain grown at that site were found to be unfavorably big and not uniform. The grain distribution was also uneven. Surface contamination was suspected and a study was undertaken to identify any unwanted metallic or organic surface contamination. However, no contaminants were found by XPS, SIMS, and GC-MS. Knowing that poly will not deposit well if an oxide is formed on the surface, a measurement of the amount of native oxide on wafer surface was made using the method described in this paper. It was found that the wafers cleaned for 10 minutes at the two sites contained different amount of oxide on surface (See Figure 5).

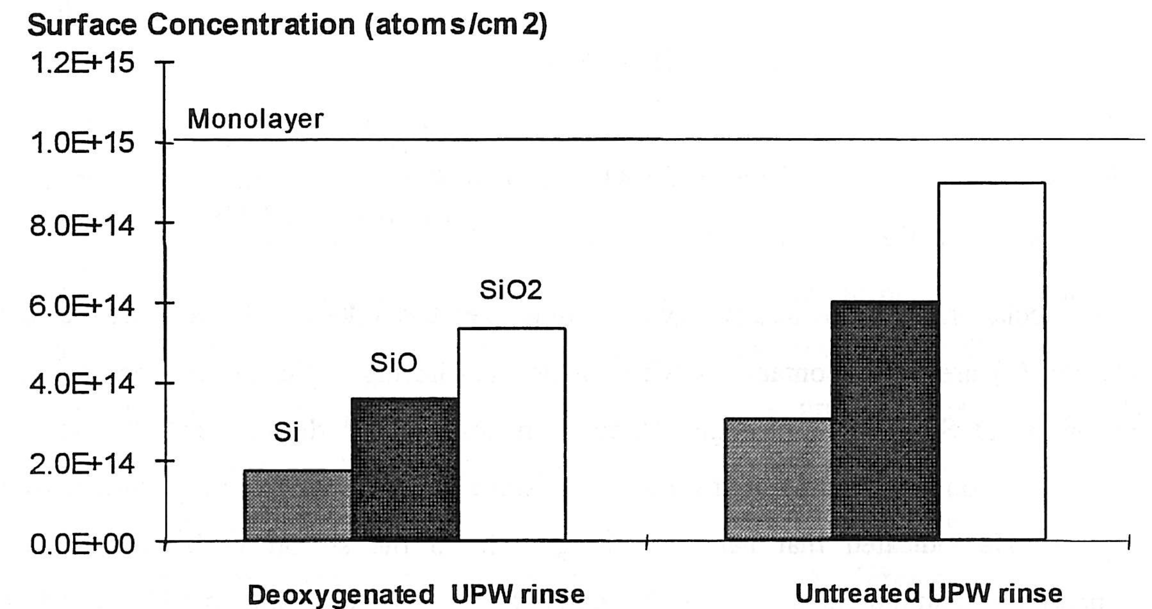
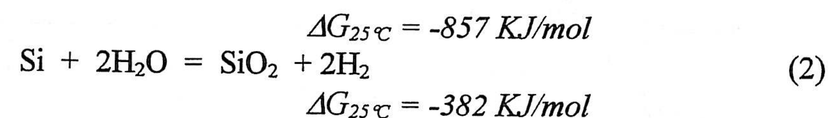


Figure 5. Effect of oxygen containing and deoxygenated UPW rinse on the native oxide growth.

Further studies were then done to determine the exact nature of the difference between the two sites and how that difference affected the wafer, the amount of oxide on it and the relationship between the oxide thickness and poly deposition problem. It was found that the UPW systems used at the two sites were slightly different. The dissolved oxygen measurements showed that one UPW system contained 50 ppb O_2 while the other contained 6 ppm. It was the later system that produced more oxide on the silicon surface and caused the poly deposition failures. Due to the existence of more native oxide on the silicon surface, which formed a heterogeneous and rough silicon surface, it was difficult to grow high quality and uniform polycrystalline silicon. This study showed that it si important to control oxide growth even in the sub-monolayer concentrations to produce quality poly. This may also be true for other semiconductor processes[1-2].

An important question naturally arises. *Is dissolved O_2 in UPW solely responsible for the native oxide formation on silicon surface during a UPW rinse?* Based on the thermodynamics theory, O_2 and H_2O can both oxidize silicon to form SiO_2. The oxidation reactions may be represented by following equations[28]:

$$Si + O_2 = SiO_2 \qquad (1)$$



$$\Delta G_{25℃} = -857\ KJ/mol$$

$$Si + 2H_2O = SiO_2 + 2H_2 \qquad (2)$$

$$\Delta G_{25℃} = -382\ KJ/mol$$

The calculations of Gibbs free energy at room temperature ($\Delta G_{25℃}$) showed that the reactions (1) and (2) are both spontaneous with reaction (1) having a greater tendency towards the formation of SiO_2[29]. Interestingly, Ohmi's kinetics work[27] showed that the native oxide hardly grew on silicon surfaces at room temperature during a seven-day exposure of wafers to dry air. He indicated that native oxide growth on the silicon wafer surface at room temperature required coexistence of O_2 and H_2O[22, 30-32]. It is possible that Si-H bonds on the hydrogen-terminated surface are easily converted into Si-O-Si or Si-OH in the presence of H_2O[22]. Another possibility would be that the dissolved O_2 or OH^- attacks the interior Si-Si bonds without breaking the Si-H bonds. Although the growing mechanism of native oxide has

not been understood, it seems that the native oxide formation is largely determined by reaction kinetics rather than thermodynamics alone.

Summary

ICP-OES has proved to be a suitable and sensitive analytical technique for the characterization of ultra-thin native oxide formed on silicon surface. By coupling with the rapid acid etching method developed in our laboratory, ICP-OES is capable of detecting and clearly distinguishing the difference in native oxide growth within a monolayer range with a possible 0.2 Å resolution. Since the native oxide layer can be chemically stripped and converted into an aqueous solution within 30 seconds, it is possible to obtain real-time surface information with an off-line measurement. The formation of native oxide on silicon surface resulting from various wet surface processes could then be representatively evaluated without experiencing the continuous oxidation problem. The method was simple, reproducible, and standardizable to NIST standard. The analysis was also found to be free of both spectral and matrix interference.

Evidence is presented in this work that the silicon signals due to the dissolution of silicon substrate at the HF stripping step become crucial since the overall silicon concentrations being detected by ICP-OES were very low. To suppress the dissolution of silicon substrate and improve the signal-to-background ratio, the stripping time may need to be shortened and the dissolved oxygen concentration in HF solution be reduced. The conversion of silicon solution concentrations obtained by ICP-OES to oxide thickness is greatly dependent on the understanding of structure and growing mechanism of the ultra-thin native oxide on silicon surface. Since it is the most stable form, SiO_2 with a density of 2.2 gm/cm^3 was used as the form of the native oxide to calculate oxide thickness (Å) in order for different laboratories to make a direct comparison.

The initial results show that ultra-thin native oxide is grown immediately at room temperature on a HF pre-cleaned p-type Si (100) surface during an oxygenated UPW rinse. The initial growth rate was found to increase linearly with increasing the exposure time of the silicon surface to UPW during the first 10 min oxygen saturated UPW rinse. The results obtained with deoxygenated UPW (containing ~50 ppb dissolved oxygen) rinse indicated that the native oxide formation on silicon surface can be reduced by decreasing the dissolved oxygen concentration in UPW.

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Biographies

Fuhe Li is a research chemist at Balazs Analytical Laboratory, working with Marjorie K. Balazs to tackle the semiconductor industrial problems. He received his Ph.D. degree in Analytical Chemistry from University of Vermont and did his postdoctoral study in University of California at Berkeley. He was also a visiting scientist at McGill University in Canada. His research interests have been in the field of the trace elemental analysis of semiconductor materials using ICP optical emission spectroscopy, ICP mass spectrometry, flame and graphite furnace atomic absorption spectroscopy, and laser techniques. He is a member of the American Chemical Society and the SEMI Global Process Chemical Committee.

Marjorie K. Balazs is the founder and CEO of Balazs Analytical Laboratory in Sunnyvale, CA and Austin, TX. She has worked in the Semiconductor Industry since 1968. Prior to her work in the semiconductor industry, she spent ten years at Stanford Research Institute and taught for six years at the University of San Francisco. She received her M.S. Degree in chemistry from the University of San Francisco and her M.A. Degree in chemical education at Stanford University. She is a member of ECS, ASTM, and ACS. Outstanding honors include: Award from President Reagan in 1986 as an outstanding women entrepreneur in the United States; 1992 City of San Jose Small Business Award; and the 1993 SEMI Lifetime Achievement

Award for North America, recognizing her contribution in yield enhancement to the semiconductor industry over the past twenty-five years.

Bruce E. Deal is currently a Consulting Professor in the department of electrical engineering at Stanford University, an Adjunct Professor at Santa Clara University, and consults for several semiconductor-related companies. He received an A.B. in chemistry from Nebraska Wesleyan University in 1950, and a Ph.D. degree in physical chemistry from Iowa State University in 1955. He has been associated with the semiconductor industry in Silicon Valley since 1959. After four years at Rheem Semiconductor, Dr. Deal conducted research at Fairchild's R&D laboratory for twenty five years in areas of silicon passivation and oxidation. He helped to establish stable MOS technology, developed the Deal-Grove relationship for silicon thermal oxidation (with Andy Grove), and characterized oxide charges in the Si-SiO_2 system. Dr. Deal has published nearly one hundred technical papers, and has been active in various technical societies, such as IEEE, MRS, and ECS (President in 1988-89). He has received several industrial awards including 1998 SEMI Lifetime Achievement Award for North America.

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