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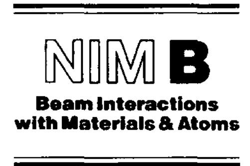

Hydrogen passivation of Si(100) wafers as templates for low temperature ($T < 600°C$) epitaxy [1]

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Abstract

Epitaxial growth requires an initial surface that is ordered and as free as possible of contaminants such as C, O, or metallic impurities. Wet chemical etching of Si(111) wafers by a solution of HF in alcohol after a modified RCA clean, has been shown to produce (1 × 1) H-terminated hydrophobic Si surfaces that are ordered at room temperature and can be desorbed at 200°C in UHV. Less is known about a similar treatment on Si(100) wafers, more commonly used in semiconductor technology. However, high temperature ($T > 800°C$) thermal desorption of the native oxide on Si(100) is known to induce detrimental effects such as surface roughness, precipitation and dopant segregation. Therefore, this study is motivated by the development of a low-temperature ($T < 600°C$) surface cleaning method for Si(100). RBS combined with ion channeling and nuclear reaction analysis is conducted to measure the coverage of C, O, and H as well as the residual disorder at the surface at different steps of wet chemical cleaning prior to low temperature desorption. Hydrogen is detected by the forward elastic recoil of H by $^4He^{2+}$ at 2.8 MeV. O and C are detected by nuclear reaction analysis (NRA) at 3.05 and 4.265 MeV, respectively, in combination with ion channeling along the Si⟨111⟩ direction to increase the detection sensitivity for C and O as well as to measure the Si surface peak to correlate it to surface disorder. Atomic force microscopy of these surfaces has shown different degrees of roughness in addition to defect formation and is correlated to the ion beam analysis results. Our results indicate a strong dependence of final H-passivation on the pretreatment of the Si surfaces before the final dip in the HF/alcohol solution.

1. Introduction

Surface preparation of Si wafers by wet cleaning prior to processing is crucial for the removal of particulates and chemical impurities from the semiconductor surface without damaging or contaminating the surface [1]. In recent years, device performance and reliability requirements have increased. Surface preparation techniques that avoid surface contamination and generate very clean wafer surfaces have become of critical importance [2]. Trace surface impurities, such as sodium ions, metals and particles, are especially detrimental if present on semiconductor surfaces. This is particularly true for high temperature processes such as thermal oxidation, diffusion, and epitaxial growth [2]. A clean surface is critical for successful epitaxy with minimum defects in the epitaxial layer [3,4]. The motivation for low temperature processing is to minimize the thermal budget and maintain the temperature below 800°C in order to preserve shallow dopant profiles and abrupt hetero-interfaces [5,6]. Silicon dioxide can only be desorbed from the Si surfaces at temperatures greater than 800°C. For temperatures lower than 800°C, the freshly cleaned Si surface is very reactive and will quickly oxidize, even under UHV conditions [3]. In recent years, interest in passivating Si surfaces with hydrogen has grown as H-passivation prevents or reduces re-adsorption of oxygen by saturating Si dangling bonds on the surface at low temperatures [7–9]. These surfaces are relatively stable at room temperatures in air, and hydrogen can be desorbed in UHV between 200 and 600°C [10]. Hydrogen passivation can be obtained either by in situ annealing in H_2 atmosphere at temperatures greater than 800°C [11,12], or by ex situ wet chemical processing in HF solutions at room temperature [13,14]. The haze formation seen during aqueous HF processing of Si wafers can be avoided by processing in HF/alcohol solutions. Also hydrogen passivation obtained via HF/alcohol solutions can be desorbed at temperatures below 600°C [15,16]. Since the temperature is maintained below 600°C, no carbide formation can occur. Studies of hydrogen passivation of Si(111) surfaces with HF/alcohol solutions report that these surfaces are

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stable in air [7,8]. To achieve hydrogen termination of surfaces, the Si surfaces are first subjected to an "RCA-type cleaning" [2,17]. In a typical "RCA cleaning", the Si surface undergoes treatment in a high pH alkaline solution with hydrogen peroxide for removing organics and metallic contaminants, followed by an oxide-stripping in an aqueous HF solution and oxide re-growth in a hydrogen peroxide based acidic solution [18].

In this paper, the surface coverage of C, O, and unregistered Si atoms on a Si(100) surface is measured after each step of the wet chemical cleaning used to achieve final H-passivation of Si(100). Nuclear reaction analysis (NRA) with ion channeling are combined to detect small amounts of C and O on the Si(100) surfaces. Elastic recoil detection is used to detect H. Both techniques are demonstrated to provide a sensitive measure of surface composition. Si surface disorder and oxygen coverage are correlated using the current understanding of the chemistry and structure of $Si(100)/SiO_2$ interface.

2. Experimental procedure

Experiments are performed in a chemical laminar flow hood, constructed of polypropylene, located in a class 100 clean room. The process steps used in this study are given in the flow chart of Fig. 1. A modified RCA type pre-clean is used in this work. 30% hydrogen peroxide is used to make SC1 and SC2 solutions. The SC1 solution is used to de-grease the silicon wafers. The composition of the SC1 solution is 4 : 1 : 1 parts of $H_2O : H_2O_2 : NH_4OH$. The SC2

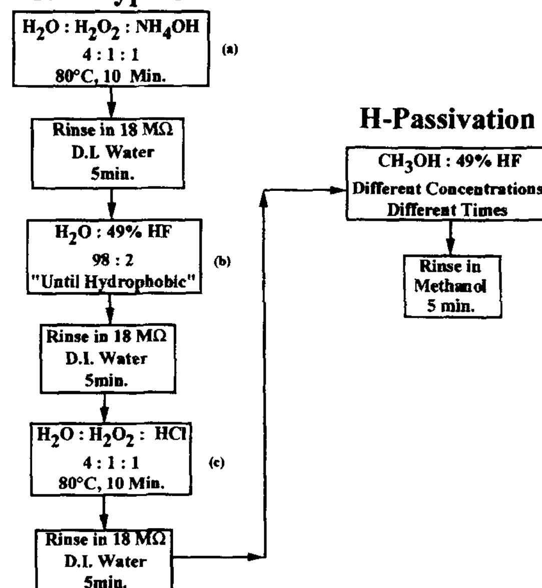

Fig. 1. Flow chart of the processing steps used in the cleaning and passivation sequence: (a) de-greasing solution, (b) oxide etchant, and (c) oxidizing solution.

solution is used for growing a chemical oxide. The composition of the SC2 solution is 4 : 1 : 1 parts of $H_2O : H_2O_2 : HCl$. The SC1 and SC2 solutions are heated

Table 1
Summary of ion beam analysis data for O, C, H and unregistered Si coverages as a function of wet chemical treatment and corresponding root mean square roughness as measured by tapping mode AFM

Sample #	Sample preparation	Atoms/cm^2 ($\times 10^{15}$)			Hydrogen (arbitrary units) [c] rel. error (%) ±1.3	rms values [Å]
		Silicon error [a] ±0.43	Carbon error [a] ±1.1	Oxygen error [a] ±0.09		
VB1-C1	As-received from Motorola Inc.	15.76	7.89	5.44	0.66	0.23
VB1-C2	After SC1 at 80°C for 10 min + 5 min D.I. water rinse	16.10	3.47	4.89	0.50	0.19
VB1-C3	After HF/H_2O (2 : 98) until hydrophobic (2 min) + 5 min D.I. water rinse	12.65	4.13	2.25	0.63	0.14
VB1-C4	After SC2 at 80°C for 10 min + 5 min D.I. water rinse	13.96	6.39	4.42	0.37	0.16
VB1-R1	After HF/CH_3OH (1 : 99) for 1 min + 5 min CH_3OH rinse	16.96	6.89	3.49	0.40	0.39
VB1-R2	After HF/CH_3OH (1 : 99) for 5 min + 5 min CH_3OH rinse	12.51	7.32	3.16	0.70	0.49
VB1-R3	After HF/CH_3OH (1 : 9) for 1 min + 5 min CH_3OH rinse	13.41	4.15	1.95	1.12	0.76
SDW6 [b]	After HF/CH_3OH (1 : 9) for 1 min + 5 min CH_3OH rinse	13.47	1.00	2.72	0.61	0.70

[a] Error units: 10^{15} at/cm^2.
[b] Without prior "RCA type" pre-clean.
[c] As a fraction of the hydrogen detected in the polystyrene standard.

to 80°C in pyrex glass containers placed in 18.3 MΩ de-ionized water (D.I.) filled tanks fabricated in flame retardant PVDF material and equipped with 4 kW teflon coated immersion heaters. The tanks are monitored by Modutek Controllers model #1215a. The treatments in HF/H_2O and HF/Alcohol solutions are carried out at room temperature in teflon tanks also located inside the hood. The samples are rinsed in a 18.3 MΩ D.I. water tank purged with nitrogen after each step, except for the final rinse after HF/alcohol which is done in an alcohol solution. The rinses last for 5 min. A HF/methanol solution held at room temperature is used for the final hydrogen passivation step. Different compositions of the HF/methanol solution and dipping durations are used in this study. All chemicals used in this study are of ppb grade and are ranked as class 10 grade by the vendor [19].

The p-type silicon wafers of 100 mm diameter with a resistivity ranging between 10 and 14 Ω cm and doped with B are used [20]. A single wafer is manually cut by a diamond scribe into several 1 in. × 1 in. square pieces, in a class 100 laminar hood. The pieces are transferred between chemical treatments in a teflon carrier. At each step of the cleaning procedure, samples are taken for characterization. Table 1 outlines the type of chemical treatment different pieces underwent. The pieces are stored in 2 in. diameter teflon sample holders prior to further analysis.

The samples are analyzed for surface contaminants, disordered Si, carbon, oxygen, and hydrogen by ion beam analysis using $^4He^{2+}$ ions. A 1.7 MV accelerator located in the ion beam analysis of Materials Facility at Arizona State University, Tempe, Arizona is used in this study. IBA is conducted in a chamber maintained at 10^{-7} Torr vacuum by an ion pump [21]. This pump generates less hydrocarbon contamination than turbo pumps. Disordered Si, carbon, and oxygen coverages are measured by gathering spectra on a sample aligned in a ⟨111⟩ direction as well as in the rotating random mode [22]. Ion channeling at 2 MeV is used to reduce the background noise by a factor of 100 and also to enable the measurement of the Si surface peak [23]. The area of the Si surface peak provides a measure of the number of disordered Si atoms at the surface [22]. Nuclear reaction analysis of oxygen at 3.05 MeV and of carbon at 4.265 MeV is also used in combination of Si ⟨111⟩ channeling. The $^{16}O(\alpha,\alpha)^{16}O$ resonance enhances the oxygen signal by a factor of 10 and the $^{12}C(\alpha,\alpha)^{12}C$ resonance increases the carbon signal by a factor of 128 [24–28]. Hydrogen coverage on the sample is measured by forward elastic recoil detection (ERD) and

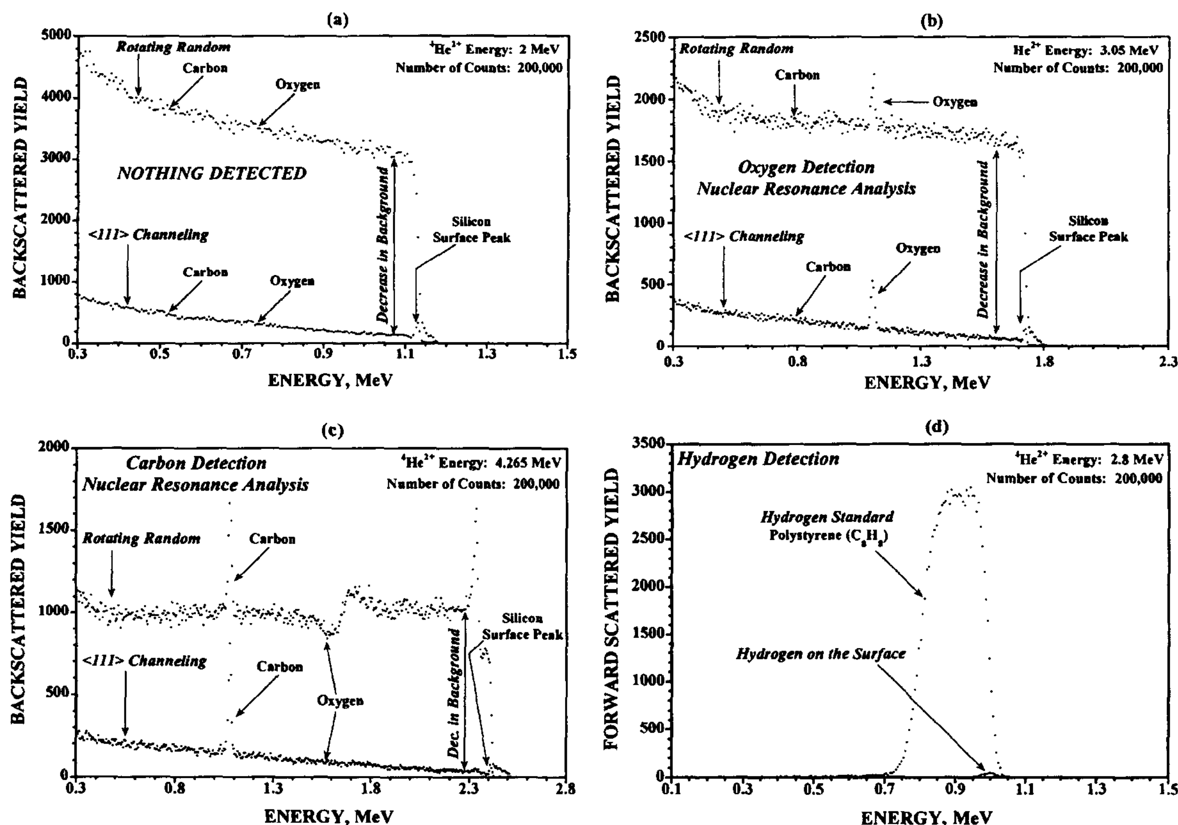

Fig. 2. Ion beam analysis of an as-received Si(100) wafer prior to chemical treatment. (a) RBS combined with ion channeling at 2 MeV, (b), (c) NRA combined with ion channeling at 3.05 and 4.265 MeV, and (d) ERD at 2.8 MeV.

compared to the hydrogen coverage on a standard [29–31]. In this study, polystyrene (C_8H_8) is used as a standard, and hydrogen at the sample surface is given as a fraction of the hydrogen coverage in the standard [31]. Several hydrogen standards, including thin films with low H content, for evaluating the surface hydrogen on the sample have been investigated in this project. More details are given in another paper [32]. In the ERD experimental setup, a thick stopper foil (10 μm Mylar) is placed in front of the detector and the incident beam energy is adjusted to 2.8 MeV to allow the hydrogen atom to be detected by penetrating to higher depths [21]. All elements with $Z > 2$ are stopped in the foil. The result is a depth profile of the H distribution in the sample.

3. Results and discussion

Three effects are investigated: (1) the effect of a modified RCA type pre-cleaning on the final hydrogen passivation, (2) the effect of the composition of final HF/alcohol, and (3) the duration of immersion in HF/alcohol solutions on hydrogen passivation. Table 1 gives the summary of the measured surface coverage of C, O, unregistered Si atoms and H coverage obtained by ion beam techniques. The values for unregistered Si, C, and O are given in atoms/cm^2 and hydrogen is given as a fraction of the hydrogen detected in the polystyrene standard. The root mean square (rms) roughness values on the sample surface are measured by an atomic force microscope (AFM) on a 20 μm × 20 μm area. In this study a Dimension 3000 model tapping mode atomic force microscope (TMAFM) is used [33]. The data clearly demonstrate that significant variations in the absolute amount of surface coverage are detected for each step of the cleaning when compared to the absolute error listed on top. The evolution of C and O amounts during cleaning, and H- coverage is consistent with the expected modification brought about by the chemical treatment.

One key result from the ion channeling analysis in Fig. 2a, however, is that the amount of unregistered surface Si atoms can be measured sensitively. According to Table 1, this amount varies from 1.696×10^{16} at/cm^2 to 1.251×10^{16} at/cm^2 with a relative error of 0.043×10^{16} at/cm^2 as a function of the cleaning steps. These data demonstrate that surface peak measurements can be used as a tool to measure surface disorder stemming from both oxide formation and variation in roughness, as discussed below. By associating the number of disordered Si atoms with the oxygen coverage by NRA, the contribution of Si in amorphous SiO_2 and SiO can be separated from the amount of unregistered surface Si atoms. The latter amount can be correlated to roughness measurements.

Figs. 2b and 2c show NRA spectra taken on the same Si(100) wafer measured at 2 MeV in Fig. 2a, but this time at the resonant energy for O and C, respectively. The $^{16}O(\alpha,\alpha)^{16}O$ nuclear resonance occurs at a $^4He^{2+}$ energy of 3.045 MeV and FWHM is 10 keV wide, which is less than the energy resolution of the RBS system (14 keV). Oxygen which could not be detected at 2 MeV is now detected since the O cross section is enhanced by a factor 10 [26,27]. Furthermore combination of NRA with ion channeling along the ⟨111⟩ axis lowers the signal to noise ratio by a factor 100, thereby yielding an accuracy better than 0.01×10^{16} at/cm^2 (see Table 1). The absolute error on O atomic coverage is thus of the order of 10% of an atomic layer. It is a factor 5 lower than the absolute error on the amount of unregistered surface Si atoms measured in the 2 MeV data of Fig. 2a and listed in Table 1. Both HF/H_2O (2:98) and HF/CH_3OH (1:99) solutions removed less oxygen compared to HF/CH_3OH (1:9) solution. The residual oxygen amount detected decreases from 4.42×10^{15} at/cm^2 to $3.46 \pm 0.09 \times 10^{15}$ at/cm^2 for a 1 min etch in HF/CH_3OH (1:99) (sample VB1-R1). It decreases a little bit further to $3.16 \pm 0.09 \times 10^{15}$ at/cm^2 after 5 min etching (sample VB1-R2). However, when a stronger solution is used, i.e. HF/CH_3OH (1:9) for 1 min, oxygen decreases to as little as $1.95 \pm 0.09 \times 10^{15}$

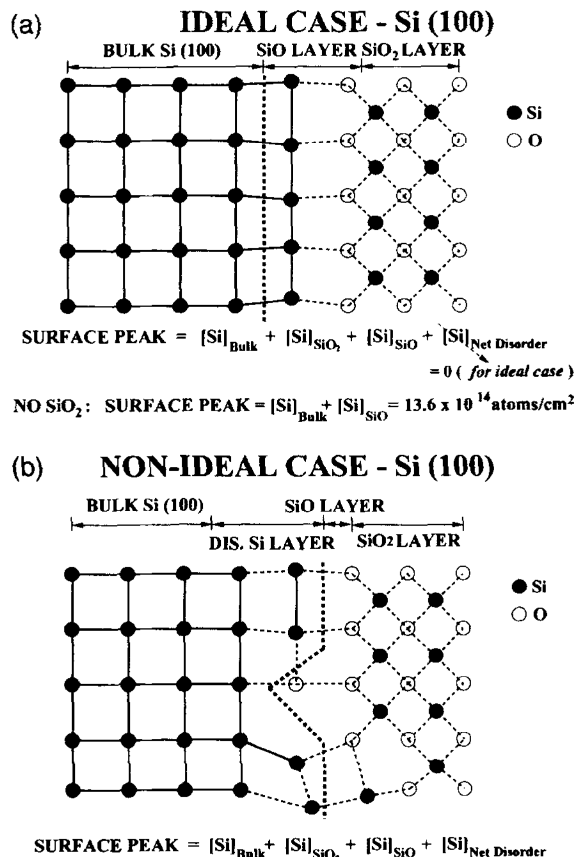

Fig. 3. Illustration of the theoretical model of Si/SiO_2 interface for an (a) ideal case and (b) realistic case.

at/cm^2, which is significantly less than what is detected after etching in aqueous HF/H_2O (2:98): $2.25 \pm 0.09 \times 10^{15}$ at/cm^2.

The $^{12}C(\alpha,\alpha)^{12}C$ nuclear resonance occurs at a $^4He^{2+}$ energy of 4.265 MeV and FWHM is 55 keV wide [24,25]. C is now detected too, since the cross section is enhanced by a factor 128 [28]. As expected, there is a significant decrease in carbon coverage after immersion in SC1 solution. HF/H_2O (2:98) and HF/CH_3OH (1:9) showed similar effects on carbon coverage. The HF/CH_3OH (1:99) did not have much effect on carbon even after immersion for longer durations.

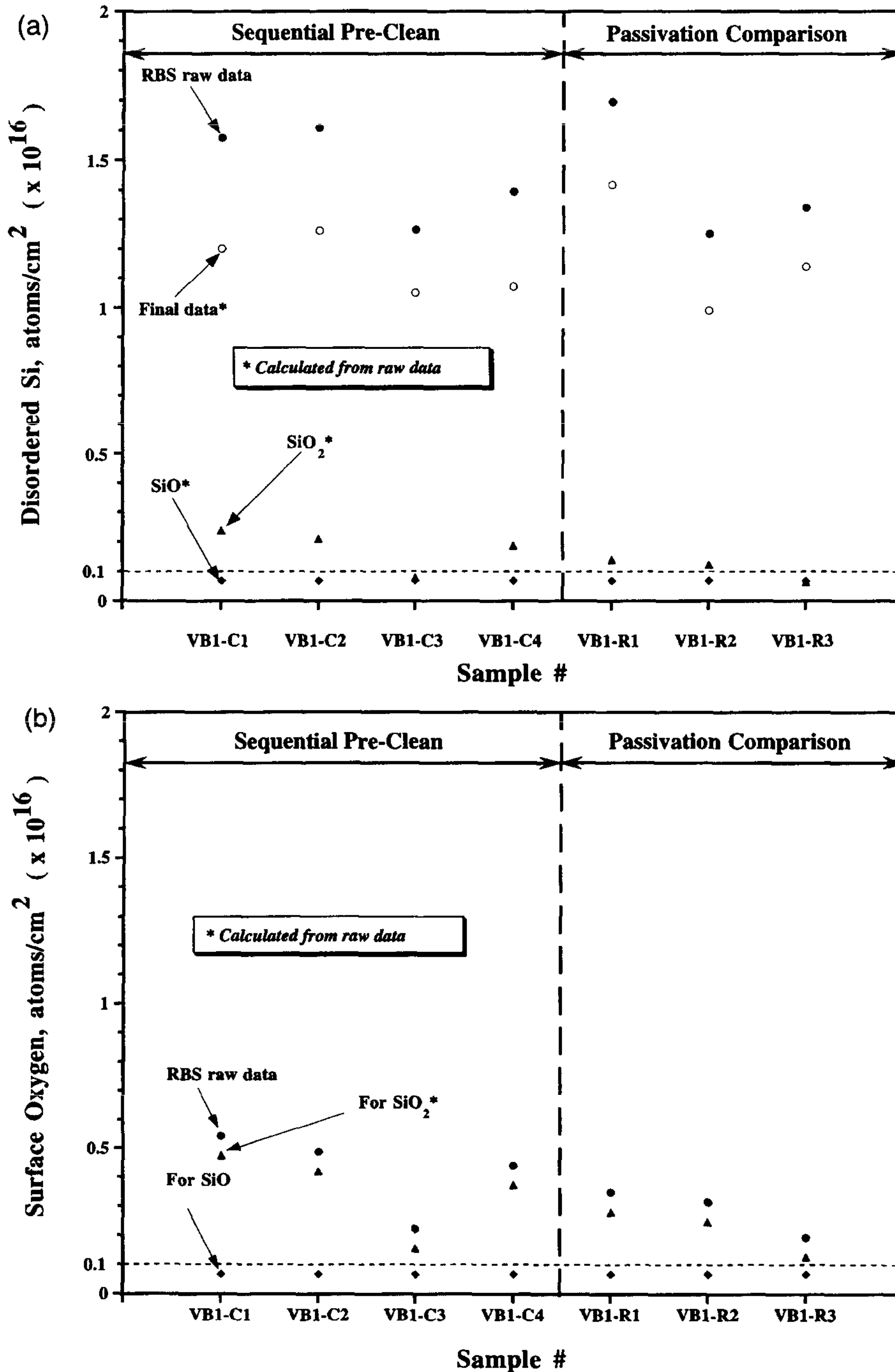

Fig. 4. Contribution for SiO and SiO_2 layers of (a) disordered Si and (b) oxygen.

As can be seen in Fig. 2d, the surface coverage of hydrogen is well resolved and confined to the surface, confirming the capability of ERD to measure quantitatively the hydrogen amounts typically found on Si(100) surfaces before wet chemical processing and as a function of cleaning. However, the depth resolution is significantly worse than in RBS because the beam energy width is broadened as it travels through the stopper foil. The biggest drawback of ERD is that it requires a standard with known H concentration to evaluate the amount of H in the sample [15,31,32]. Fig. 2d gives the ERD spectra for virgin Si(100) and a polystyrene standard. The samples exhibit an increase in H-coverage matching closely the observed decrease in oxygen coverage (Table 1). When samples VB1-C3, etched in aqueous HF, i.e. in HF/H_2O (2:98), and the four last samples in Table 1 are compared, the sample VB1-R3, etched for 1 min in HF/CH_3OH (1:9) shows the best hydrogen passivation characteristics, with the largest amount of H detected on the surface by ERD. The same etching conditions without prior "RCA type" pre-clean (sample SDW6) shows less hydrogen coverage and a higher oxygen coverage. All the samples, with and without prior "RCA type" pre-clean, demonstrate a decrease in oxygen coverage and an increase in hydrogen coverage after etching in an HF solution. The surface coverage of Si(100) with hydrogen is thus found to increase when HF in methanol is used rather than HF in D.I. water. Increase in oxygen coverage with a decrease hydrogen coverage in SC1 and SC2 solutions is understandable due to the chemistry of SC1 and SC2 solutions. SC1 solution removes the organic and metallic contaminants as well as grows an oxide layer. SC2 is an oxidizing solution.

A key finding is that only Si(100) surfaces treated with HF in alcohol are found to be (1×1) reconstructed at room temperature when subjected to RHEED in UHV. Only such surfaces can be then cleaned and (2×1) reconstructed in UHV by thermal desorption below 600°C, while wafers treated with aqueous HF are not found to be reconstructed but are amorphous at room temperature. They can only be reconstructed after thermal desorption in UHV at $T \geq 800$°C.

A measurement of surface disorder can be obtained from the Si surface peak measurements in Table 1 based on the fairly well-established understanding of the chemistry and structure of the Si(100)/amorphous SiO_2 interface. Grovernor and Cerezo have clearly demonstrated back in the eighties than an intermediate layer of Si monoxide is present between SiO_2 and Si(100) to bridge the cubic diamond structure of Si(100) with that of the fourfold coordinated aperiodic network of silica, using atomic probe microscopy [34]. These results were in agreement with other work in the literature. In depth work on the formation of oxides on Si substrate by the use of XPS were reported in the literature during the last decade [35]. These studies indicate formation of Si suboxides prior to the complete growth of stoichiometric SiO_2 on top of the Si substrate. More recently, Ouzmard was able to image on very flat, almost ideal surfaces, the Si–O dimers constituting the single monoxide silicon layer [36]. The resulting model enables to construct a model for the idealized interface/SiO_2 layer (Fig. 3a) and for a realistic interface in Fig. 3b where disorder and roughness contribute additional displaced atoms. From this model one can thus deduce the net number of displaced Si atoms once the contribution to the SiO_2 and SiO interfacial layer is substracted using the following equation

$$\text{Surface Peak} = [\text{Si}]_{\text{SiO}_2} + [\text{Si}]_{\text{SiO}} + [\text{Si}]_{\text{Bulk}} + [\text{Si}]_{\text{Net Disorder}}$$

$[\text{Si}]_{\text{Net Disorder}} = 0$ in the ideal case

$[\text{Si}]_{\text{Bulk}} = [\text{Si}]_{\text{SiO}} = 6.8 \times 10^{14}$ atoms/cm^2 without SiO_2

where $[\text{Si}]_{\text{Bulk}}$ is the number of atoms/cm^2 terminating the atomic row on the cubic diamond lattice [37].

The results from these calculations based on the data in Table 1 are shown in Figs. 4a and 4b for oxygen and silicon respectively. It can be clearly seen that a significant fraction of the displaced Si atoms is not due to oxide or monoxide formation. These data demonstrate that one can deduce surface disorder based on this model.

4. Conclusions

In summary, the present characterization by IBA of Si(100) at different stages of surface cleaning and for different etching conditions demonstrates that: (1) disordered Si, O, C, and H coverages and their evolution during cleaning can be accurately measured by combining nuclear reaction analysis and ion channeling to a precision of 0.43×10^{15} for disordered Si, 1.1×10^{15} for C, 0.09×10^{15} for O, and 1.3% for H, (2) the evaluation of O, C, and H coverage as measured by IBA reflects the expected modifications of surface composition and is able to track quantitatively the H and O coverage as a function of surface treatment. In general, with the increase in H coverage, there is a decrease in the amount of surface oxygen. This trend is more obvious during the passivation phase. Combining NRA with IC and ERD can thus be used as a quantitative tool for optimization of Si(100) surface preparation and H-passivation. According to these preliminary results, after SC1, the amount of unregistered Si is seen to increase with the presence of oxygen, as expected from an oxidizing solution. HF/H_2O, SC2 and HF/CH_3OH are all found to lead to a decrease of unregistered Si as expected from the SiO_2 etching. The amount of unregistered Si atoms is seen to decrease with increasing HF concentration and/or etching duration.

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