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Infrared spectroscopy study of initial stages of oxidation of hydrogen-terminated Si surfaces stored in air

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We have studied the initial stages of oxidation of the hydrogen-terminated Si(111) and (100) surfaces stored in air, using infrared spectroscopy in the multiple internal reflection geometry. We investigate the effect of surface roughness and humidity of air on the oxidation of the hydrogen-terminated Si surfaces. We suggest that surface roughness on a microscopic scale does not significantly affect the oxidation of the hydrogen-terminated Si surface and the oxidation occurs on the entire surface. It is demonstrated that water is predominantly involved in the oxidation of the surface Si—H bond, and that the surface Si—H bond is quite inert to the oxygen molecule.

I. INTRODUCTION

It is well established that treatment in dilute hydrofluoric acid (HF) solution produces a hydrogen-terminated, clean Si surface.[1–4] This treatment is widely used in the fabrication of semiconductor devices such as metal-oxide-semiconductor (MOS) field-effect transistors. The hydrogen-terminated Si surface is chemically inert but when exposed to air for a long time, is oxidized by oxidants present in air to generate the so-called native oxide film. Since native oxide most probably degrades the quality of Si epitaxial film or thin gate oxide film which may be subsequently grown on the Si surface, the undesirable formation of native oxide film must be suppressed. Therefore, in order to fabricate high-reliability ultralarge-scale integration (ULSI) devices, it is important to investigate the chemical stability of the HF-treated surface under the atmospheric environment and to elucidate the mechanism of the oxidation at the atomic level.

A number of studies have so far been carried out to investigate the chemical nature of HF-treated Si surfaces.[5,6] Among them, x-ray photoemission spectroscopy (XPS) studies[7,8] revealed that oxidation proceeds layer-by-layer on HF-treated surfaces exposed to room air. However, little is known about the early stages of the oxidation process taking place on the topmost layer of the hydrogen-terminated Si surface. In investigating the oxidation process on the hydrogen-terminated surface at the atomic level, it is essential to observe changes in the concentration of the hydrogen-associated intermediate oxidation states for which both hydrogen and oxygen atoms are bonded to a Si atom. As previously reported,[3,9,10] these oxidation species can be clearly observed in infrared absorption spectra in the Si—H stretching vibration region. In this study, we therefore use infrared spectroscopy in the multiple internal reflection geometry to investigate the initial stages of oxidation of hydrogen-terminated Si(111) and (100) surfaces which are stored in air.

The issues we address in this study include whether the oxidation at defects sites such as steps and kinks is favored, and which oxidant, oxygen molecule, or water, is predominantly involved in the oxidation of the topmost layer of hydrogen-terminated Si surfaces. In this study, we focus on the effect of the microscopic roughness of the surface and the humidity of air on the oxidation in air. We therefore present and discuss the experimental results on the oxidation of Si surfaces with different microscopic roughnesses and those exposed to air with different humidities.

II. EXPERIMENT

Samples used here were obtained from n-type P-doped Si(100) and (111) wafers. The samples were treated in dilute hydrofluoric acid (HF) or 40% NH_4F solution after the traditional RCA cleaning. After surface treatment, the samples were stored in air. In order to examine the effect of surface roughness on the oxidation, sample wafers cut vicinal to the (100) plane were also used with the thought that a high density of defects such as steps and kinks might occur after HF treatment influencing the oxidation. The study of Si(111) surfaces with HF and NH_4F treatments was undertaken to examine the effect of surface morphology on the oxidation of the hydrogen-terminated Si(111) surface. It is well established that treatment in a NH_4F solution produces an atomically flat, hydrogen-terminated Si(111) surface.[11,12] This should be compared to the HF treatment in which a microscopically rough Si(111) surface is created.[13]

The relative humidity of room air was about 40%. To examine the role of water present in air in the oxidation of the Si surfaces during storage in air, the samples were also stored in a desiccator in which the relative humidity was approximately 10%, and in a dry O_2 ambient. Oxidation experiments in the O_2 ambient were carried out using a vacuum vessel which was first evacuated to below 10^{-2} Torr and then filled with O_2 gas to 1 atm. The purity of the oxygen gas used was 99.99%. The concentration of water in the gas was below 0.25 ppm, as estimated from the measured value of dew point.

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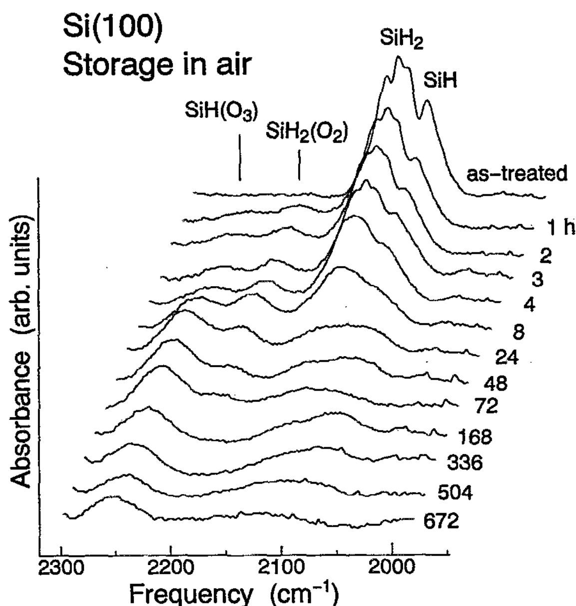

FIG. 1. Si—H stretching vibration spectra of hydrogen-terminated Si(100) surfaces exposed to air for different durations. The exposure time, in units of hours is indicated for each spectrum.

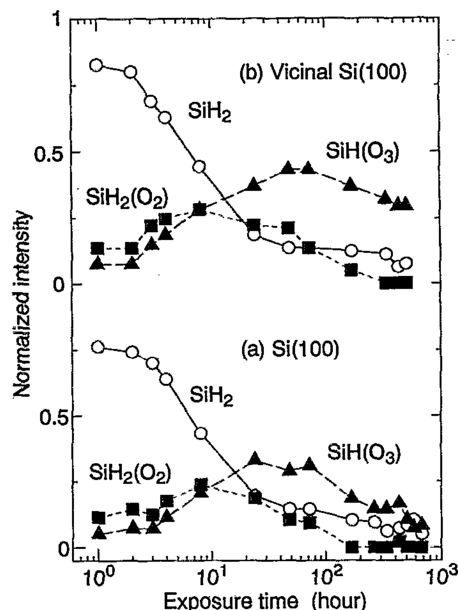

FIG. 2. Normalized intensities of Si—H_2, $SiH_2(O_2)$, and $SiH(O_3)$ peaks as a function of exposure time, for (a) Si(100) and (b) vicinal Si(100) surfaces.

The oxidation of hydrogen-terminated Si surfaces caused by exposure to air or O_2, was monitored by infrared spectroscopy in the multiple internal reflection geometry.[14] The sample used for this measurement was $0.38\times10\times50$ mm^3 with 45° bevels on each of the short edges. The infrared radiation exiting an interferometer (BOMEM MB-100) was focused at normal incidence onto one of the two bevels of the sample, and propagates through the wafer, internally reflecting about 100 times. The radiation that exits the sample through the other bevel, is focused onto a liquid-N_2-cooled HgCdTe detector. The reflection spectra measured for chemically oxidized Si surfaces were used as the background reference spectra. The chemical oxidation was performed by immersing the samples into a 1:1 boiled solution of H_2SO_4:H_2O_2 for 5 min.

III. RESULTS AND DISCUSSION

A. Effect of surface morphology

First we will present and discuss the results of the oxidation of the hydrogen-terminated Si(100) surface. Figure 1 shows Si—H stretching vibration spectra of a series of HF-treated Si(100) surfaces exposed to room air for different durations. As is shown in the top spectrum of the figure, the surface immediately after HF treatment exhibits two peaks at 2080 and 2110 cm^{-1} which are due to the monohydride Si (Si—H) and dihydride Si (Si—H_2), respectively.[13] The surface is predominantly terminated with the dihydride Si. When the surface is exposed to air for several hours, however, new peaks appear at 2200 and 2250 cm^{-1}. Upon further exposure to air, additional broad peaks appear at 2080 and 2130 cm^{-1}. We attribute these peaks to surface silicon atoms with different atomic bonding configurations. Lucovsky[15,16] has calculated the Si—H vibration frequency for a Si atom with different numbers of oxygen and hydrogen atoms bonded to it. From comparison of the observed peak positions with the calculation,[15–17] we assign the peaks at 2080, 2130, 2200, and 2250 cm^{-1} to bonding configurations $SiH(Si_2O)$, $SiH_2(SiO)$, $SiH_2(O_2)$, and $SiH(O_3)$, respectively, and determine that configuration $SiH(SiO_2)$ also contributes to the peak at 2200 cm^{-1}.[9,10]

As can be seen from Fig. 1, as the exposure time is increased, the hydride modes, Si—H and Si—H_2, decrease in intensity, suggesting that surface Si—H bonds are attacked by oxidants present in air to be oxidized. On the other hand, Si—H stretching modes corresponding to the intermediate oxidation species, $SiH_2(O_2)$ and $SiH(O_3)$, initially increase and then drop. In addition, the generation of $SiH(O_3)$ follows that of $SiH_2(O_2)$. These trends are clearly illustrated in Fig. 2(a), in which the peak intensities of Si—H_2, $SiH_2(O_2)$, and $SiH(O_3)$, normalized to the initial peak intensity of Si—H_2, are plotted as a function of exposure time. We can interpret the observed evolution in concentration of the hydrides and intermediate oxidation states as follows: Figure 3 schematically illustrates the oxidation process on the Si(100) surface. In this figure, the configurations which are enclosed with dotted circles exhibit S—H vibration modes. The initial surface is characterized by two Si—Si back bonds and two Si—H bonds per Si atom. At the first stage of oxidation, oxygen attacks the Si—H bond or the back bond of the surface Si atom. This should lead to a decrease in the concentration of Si—H_2. At this stage of oxidation, $SiH_2(SiO)$ is produced when one of the two back bonds is converted into the Si—O bond with the two surface Si—H bonds left unbroken. We observed that the SiH_2 peak is slightly shifted to higher frequencies with increasing the exposure time. Since, as mentioned above, the $SiH_2(SiO)$ peak is expected to be positioned slightly higher than the SiH_2 one, this peak shift can be interpreted as a result of generation of the $SiH_2(SiO)$

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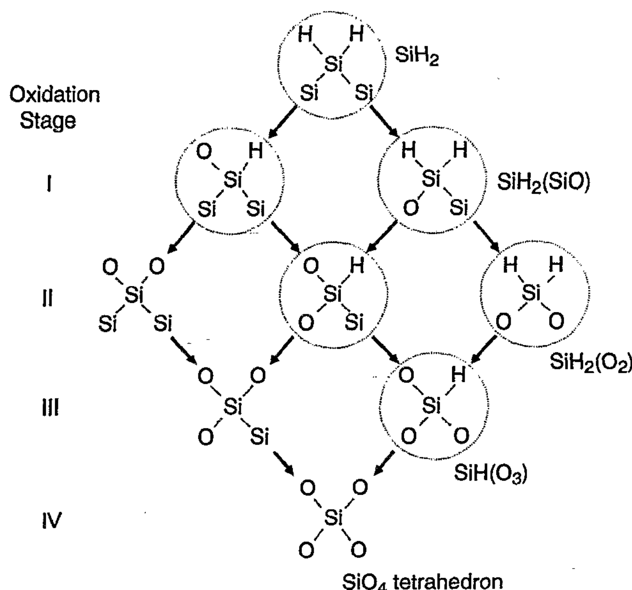

FIG. 3. Schematic illustration of the oxidation process.

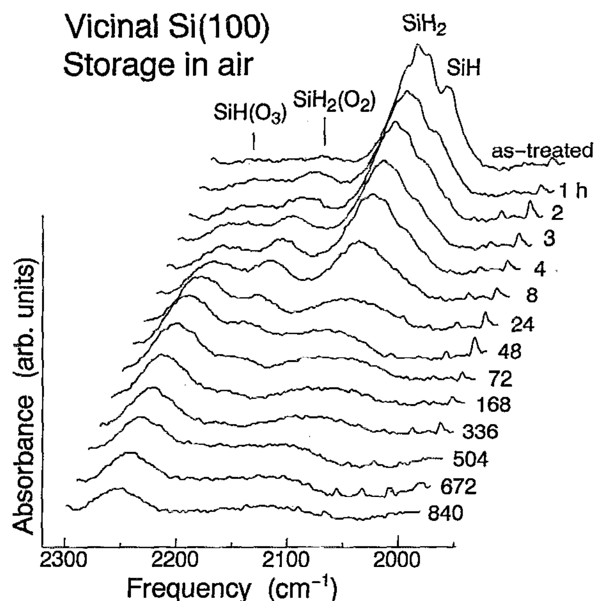

FIG. 4. Si—H stretching vibration spectra of hydrogen-terminated vicinal Si(100) surfaces exposed to air for different durations. The exposure time, in units of hours, is indicated for each spectrum.

species. At the second stage of oxidation, if the two back-bonds are converted to the Si—O bond, $SiH_2(O_2)$ is generated. After the back bond oxidation is completed, the remaining Si—H bonds of the surface Si atom should be replaced with the Si—O bond. This results in generation of $SiH(O_3)$, following that of $SiH_2(O_2)$. When all the bonds around the surface Si atom are replaced with the Si—O bond, the SiO_4 tetrahedron is generated and all the S—H stretching vibration modes disappear. This is the reason why the $SiH(O_3)$ peak vanishes after a long-term exposure to air.

In order to find out whether microscopic roughness has some influence on the oxidation of the outermost layer of the hydrogen-terminated Si surfaces, we have investigated the oxidation of a vicinal Si(100) surface which is inclined at 4° with respect to the (100) plane toward the [001] direction. We anticipated that upon HF treatment a larger number of steps and kinks are generated on the vicinal Si(100) than on the Si(100) surface. If oxidation at steps and kinks were preferred, the oxidation rate on the outermost layer would be enhanced on a microscopically rough surface.

Figure 4 shows Si—H stretching vibration spectra of a series of the HF-treated, vicinal Si(100) surfaces exposed to air for different durations. A comparison of Figs. 1 and 4 shows that the spectrum of the vicinal Si(100) surface immediately after HF treatment is similar in spectral shape to that of the Si(100) surface. This indicates that a small difference in the surface orientation of the Si substrate does not affect significantly the hydrogen termination, which is consistent with the results obtained by Chabal *et al.*[13] However, it should be noticed that the spectrum of the vicinal Si(100) surface is slightly broadened as compared to that of the Si(100) surface, indicating that the vicinal Si(100) surface immediately following HF treatment may have more steps and facets than the Si(100) surface. Similar to the Si(100), the hydride modes decrease and the intermediate oxidation species increase with increasing the exposure time. We plot in Fig. 2(b) the normalized intensity of the hydrides and intermediate oxidation species as a function of exposure time. We see the time evolution of peak intensities is almost the same as observed on the Si(100) surface. In particular, the oxidation rate of the surface Si—H_2 species is very much the same on the two surfaces. This indicates that oxidation on the topmost layer proceeds in the same manner on the two surfaces. We thus suggest that microscopic roughness does not affect significantly the oxidation of the topmost layer of the hydrogen-terminated Si surfaces and the oxidation process takes place on the entire surface.

Next we will present and discuss the results on the oxidation of Si(111) surfaces during storage in air. Figure 5 shows Si—H stretching vibration spectra of a series of HF-treated Si(111) surfaces exposed to air for different durations. As is shown in the top spectrum of Fig. 5, the surface immediately following HF treatment exhibits two intense peaks at 2080 and 2130 cm^{-1} which are due to the monohydride Si and trihydride Si (Si—H_3), respectively.[13] The structure at 2110 cm^{-1} can be attributed to the dihydride Si. Since all the hydride modes appear, the surface should be rough on a microscopic scale, which is consistent with the previous results.[13,18] When the surface is exposed to air for a few hours, peaks due to the intermediate oxidation species, $SiH_2(O_2)$ and $SiH(O_3)$, appear. Figure 6(a) shows the intensities of Si—H, $SiH_2(O_2)$, and $SiH(O_3)$, normalized to the initial intensity of Si—H, as a function of exposure time. As the exposure time is increased, Si—H decreases in intensity, and $SiH(O_3)$ initially increases and subsequently drops. However, we observe that the $SiH_2(O_2)$ peak is quite weak, which is in contrast to the behavior observed on the Si(100) surface. We can attribute this observation to the difference in the bond topology of the outermost layer of the surface: On the hydrogen-terminated Si(111) surface, a Si atom on the

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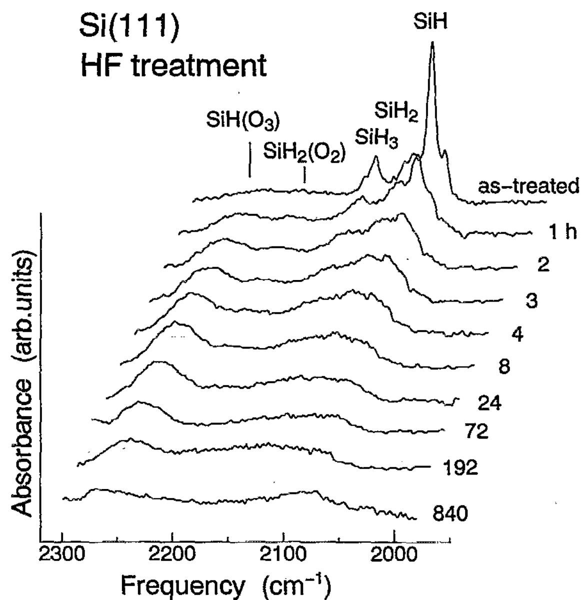

FIG. 5. Si—H stretching vibration spectra of HF-treated Si(111) surfaces exposed to air for different durations. The exposure time, in units of hours, is indicated for each spectrum.

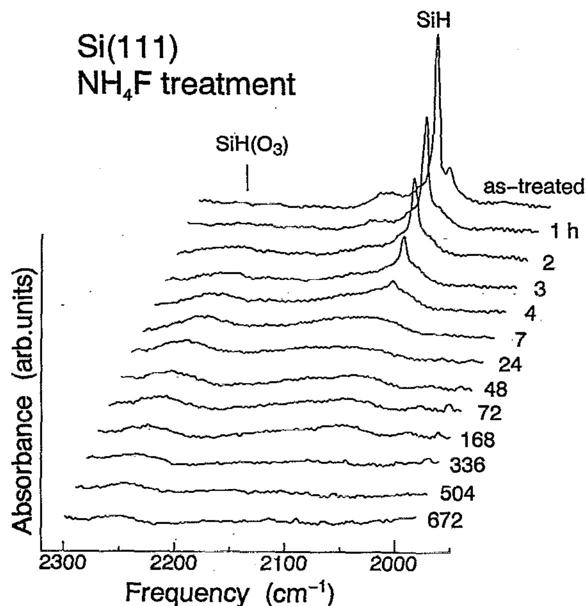

FIG. 7. Si—H stretching vibration spectra of NH_4F-treated Si(111) surfaces exposed to air for different durations. The exposure time, in units of hours, indicated for each spectrum.

outermost layer has the Si—H bond protruding from the surface as well as three Si—Si back bonds. We therefore tentatively interpret that $SiH(O_3)$ may be generated through the attack of the three back bonds by oxidants. However, it should be noted that if the back bonds were successively attacked by oxidants, $SiH_2(O_2)$ should be generated. This point will be discussed briefly below.

As mentioned previously, it is reported that treatment in basic solutions such as a saturated solution of NH_4F (pH~8) produces a hydrogen-terminated, atomically flat Si(111) surface with no detectable impurities such as oxygen, fluorine, or hydrocarbons.[11,12,19,20] In Fig. 7 we plot Si—H stretching vibration spectra of a series of NH_4F-treated Si(111) surfaces exposed to air for different durations. As is shown in the top spectrum of Fig. 7, the surface immediately following NH_4F treatment exhibits an intense peak at 2080 cm^{-1} which is due to the monohydride Si. Other hydride modes are not clearly observed. We therefore confirm that the surface is microscopically flat, and is predominantly covered with the Si—H bond oriented perpendicular to the substrate surface. When the surface is exposed to air for a few hours, the peak due to the intermediate oxidation, $SiH(O_3)$, appears, although it is quite weak. We plot in Fig. 6(b) the peak intensities of Si—H and $SiH(O_3)$, normalized to the initial peak intensity of Si—H, as a function of exposure time. With increase in exposure time, Si—H decreases in intensity, and $SiH(O_3)$ initially increases, and then decreases. This is the same trend as observed on the HF-treated Si(111) surface. However, it should be noticed that the $SiH_2(O_2)$ peak cannot be identified in the spectra of Fig. 7. This suggests that all the back bonds around the topmost Si atom are oxidized almost simultaneously on the atomically flat hydrogenated Si(111) surface. In other words, we may say that the intermediate oxidation species such as $SiH(Si_2O)$ and $SiH(SiO_2)$ is quite unstable on the flat Si(111) surface, therefore leading to a rapid conversion of these oxidation species to $SiH(O_3)$. As mentioned above, on the HF-treated surface, the generation of $SiH_2(O_2)$ was observed, although its peak intensity was quite small. This is most probably due to the back bond oxidation of the dihydride Si which exists at defects sites on the surface. Also noteworthy about Fig. 6 is that the oxidation rate of the surface Si—H bond on the NH_4F-treated surface is almost the same as on the HF-treated surface; the Si—H mode almost completely vanishes for exposures around 7 h on the two

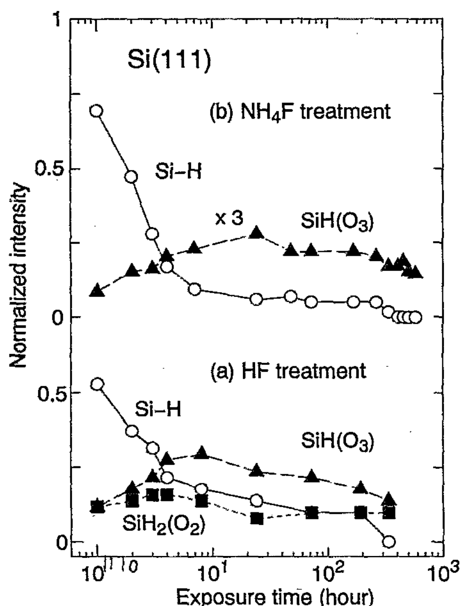

FIG. 6. Normalized intensities of Si—H, $SiH_2(O_2)$, and $SiH(O_3)$ peaks as a function of exposure time, for (a) HF- and (b) NH_4F-treated Si(111) surfaces.

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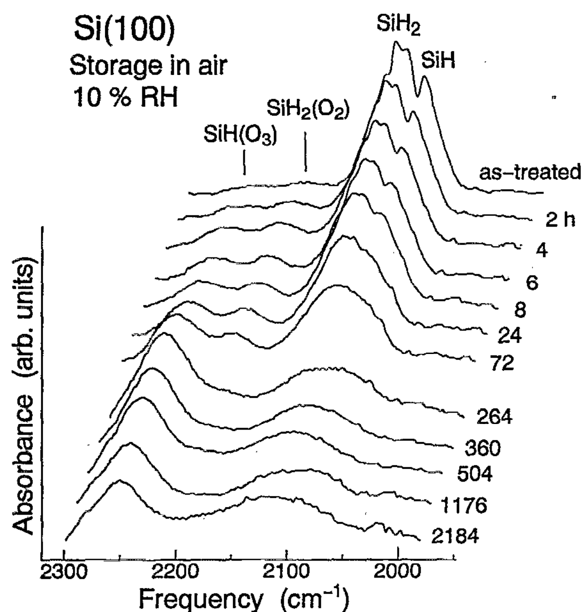

FIG. 8. Si—H stretching vibration spectra of HF-treated Si(100) surfaces exposed to air with 10% humidity for different durations. The exposure time, in units of hours, is indicated for each spectrum.

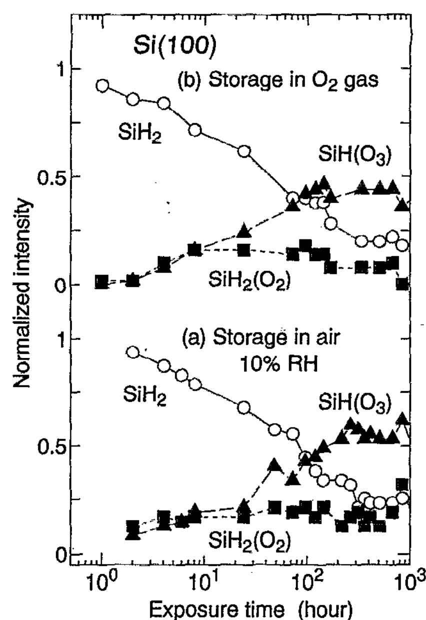

FIG. 9. Normalized intensities of SiH_2, $SiH_2(O_2)$, and $SiH(O_3)$ peaks as a function of exposure time, for HF-treated Si(100) surfaces stored in (a) low-humidity air and (b) dry O_2.

surfaces. This also suggests that oxidation of the topmost layer takes place on the entire surface.

B. Effect of humidity of air

Oxidants such as oxygen and water molecules which are present in air, are most probably involved in the oxidation of hydrogen-terminated Si surfaces stored in air. Furthermore, it is most likely that these oxidants react with the hydrogen-terminated surfaces in different ways. In order to determine which oxidant is mainly involved in the oxidation of the hydrogen-terminated Si surfaces, we have investigated the oxidation of Si surfaces stored in a desiccator in which the relative humidity was 10%, and stored in a dry O_2 ambient.

Figure 8 shows Si—H stretching vibration spectra of a series of HF-treated Si(100) surfaces exposed to air with 10% humidity for different durations. We show in Fig. 9(a) the normalized intensities of Si—H, $SiH_2(O_2)$, and $SiH(O_3)$ as a function of exposure time. As the exposure time is increased, the hydride mode SiH_2 decreases in intensity, and $SiH_2(O_2)$ and $SiH(O_3)$ initially increase and subsequently drop, which is the same trend as observed on the surface exposed to air with higher humidity. However, we notice that the oxidation of the surface Si—H_2 bond is significantly retarded and the generation of $SiH(O_3)$ is enhanced as compared to the oxidation in high-humidity air. From these observations we conclude that water present in air is predominantly involved in the oxidation of surface Si—H bonds.

Similar results were obtained on the Si(111) surfaces. Figure 10 shows Si—H stretching vibration spectra of a series of the NH_4F-treated Si(111) surfaces exposed to air with 10% humidity for different durations. In Fig. 11(a) the exposure-time dependence of the normalized intensities of Si—H and $SiH(O_3)$ is shown. For comparison, we also plot in Fig. 11(b) the results obtained for higher-humidity air. We can see from Fig. 11 that oxidation of the surface Si—H bond is significantly retarded in the 10% humidity case. Additionally, the $SiH(O_3)$ component is significantly enhanced. These findings give further evidence to the conclusion that water present in air is predominantly involved in the oxidation of surface Si—H bonds.

In order to investigate the role of the oxygen molecule in the oxidation of hydrogen-terminated Si surfaces, the oxidation process in the O_2 ambient has been investigated. Figure

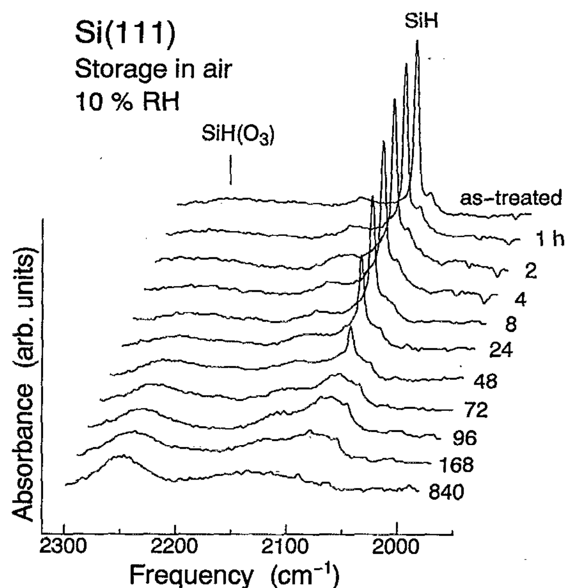

FIG. 10. Si—H stretching vibration spectra of NH_4F-treated Si(111) surfaces exposed to air with 10% humidity for different durations. The exposure time, in units of hours is indicated for each spectrum.

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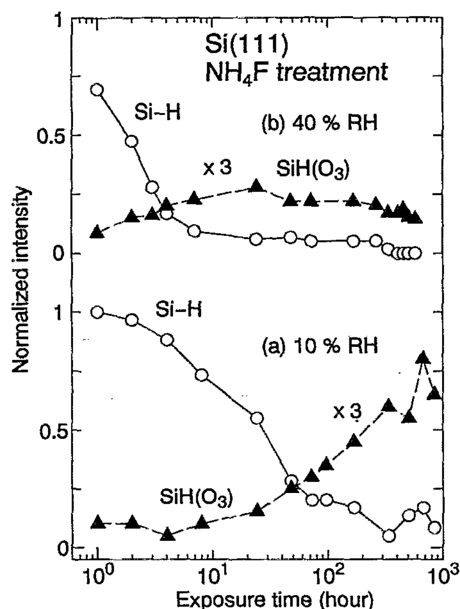


FIG. 11. Normalized intensities of Si—H and $SiH(O_3)$ peaks as a function of exposure time, for NH_4F-treated Si(111) surfaces stored in (a) 10% and (b) 40% humidity air.

12 shows Si—H stretching vibration spectra of a series of HF-treated Si(100) surfaces exposed to O_2 for different durations. In Fig. 9(b) the normalized intensities of Si—H, $SiH_2(O_2)$, and $SiH(O_3)$ are shown as a function of exposure time. We can see that the time evolution in intensity of Si—H, $SiH_2(O_2)$, and $SiH(O_3)$ is the same as observed on the surface exposed to air with low humidity. This strongly suggests that oxygen molecules are involved in the oxidation on the topmost layer when the humidity is reduced to approximately 10%.

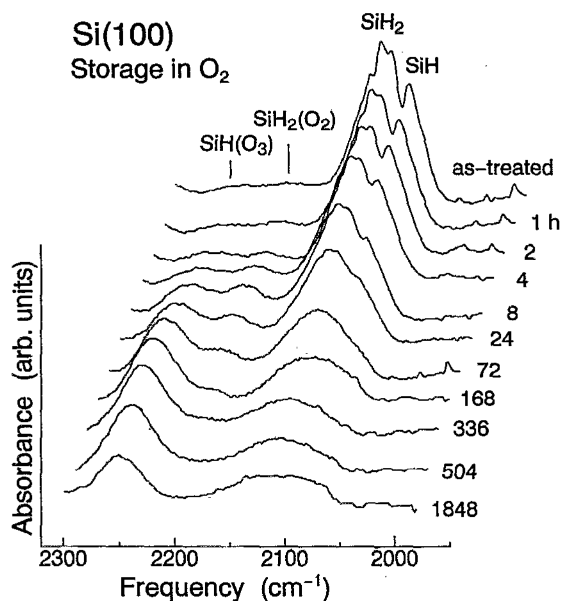
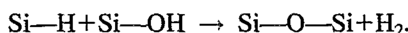


FIG. 12. Si—H stretching vibration spectra of HF-treated Si(100) surfaces stored in dry O_2 ambient for different durations. The exposure time, in units of hours, is indicated for each spectrum.

The experimental results mentioned above demonstrate that the oxidation of the surface exposed to high humidity air is quite different from that exposed to O_2, and that water present in air is crucial to the initial stages of oxidation of hydrogen-terminated Si surfaces during storage in air. It is most probable that water readily reacts with the surface Si—H bond. One plausible reaction process would be the conversion of the surface Si—H bond into the Si—OH bond and the Si—O—Si bridge bond:

$$\text{Si—H} + H_2O \rightarrow \text{Si—OH} + H_2,$$



$$\text{Si—H} + \text{Si—OH} \rightarrow \text{Si—O—Si} + H_2.$$

As a consequence of these reactions, the surface which initially was hydrophobic, becomes hydrophilic. Once the surface turns hydrophilic, it would react readily with oxidants such as water or the oxygen molecule, resulting in an enhancement of the oxidation rate of the outermost Si atom. In fact, comparison of the present IR results with the XPS results of Morita *et al.*[7] shows that when the Si—H_2 component vanishes, the thickness of native oxide film starts to increase. On the other hand, we observed that the oxidation rate of Si—H is much smaller for the oxygen molecule than for water. We thus conclude that the Si—H bond on the outermost layer of the hydrogen-terminated Si surface is quite inert to the oxygen molecule. From the fact that $SiH(O_3)$ is enhanced on the Si(111) surface, we speculate that oxygen molecules preferentially attack the Si—Si back bonds of the surface Si atom. In order to give further evidence to this speculation, we need more elaborate studies including *ab initio* molecular-orbital calculations.

IV. SUMMARY

We have investigated the initial stages of oxidation of hydrogen-terminated Si(111) and (100) surfaces which are stored in air, using infrared spectroscopy in the multiple internal reflection geometry. This spectroscopic technique is quite surface sensitive and therefore is beneficial to the investigation of the changes in the chemical state of Si surfaces. Spectral features characteristic of the intermediate oxidation states were clearly observed. We suggested that oxidation of the outermost layer of hydrogen-terminated Si surfaces exposed to air, occurs on the entire surface. We also demonstrated that water present in air is crucial to the initial stages of oxidation of hydrogen-terminated Si surfaces stored in air, and that the surface Si—H bond is quite inert to the oxygen molecule.

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