Structural and electrical characterization of HBr/O_2 plasma damage to Si substrate[a]

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Silicon substrate damage caused by HBr/O_2 plasma exposure was investigated by spectroscopic ellipsometry (SE), high-resolution Rutherford backscattering spectroscopy, and transmission electron microscopy. The damage caused by H_2, Ar, and O_2 plasma exposure was also compared to clarify the ion-species dependence. Although the damage basically consists of a surface oxidized layer and underlying dislocated Si, the damage structure strongly depends on the incident ion species, ion energy, and oxidation during air and plasma exposure. In the case of HBr/O_2 plasma exposure, hydrogen generated the deep damaged layer ($\sim$10 nm), whereas ion-enhanced diffusion of oxygen, supplied simultaneously by the plasma, caused the thick surface oxidation. In-line monitoring of damage thicknesses by SE, developed with an optimized optical model, showed that the SE can be used to precisely monitor damage thicknesses in mass production. Capacitance–voltage (C–V) characteristics of a damaged layer were studied before and after diluted-HF (DHF) treatment. Results showed that a positive charge is generated at the surface oxide–dislocated Si interface and/or in the bulk oxide after plasma exposure. After DHF treatment, most of the positive charges were removed, while the thickness of the "Si recess" was increased by removing the thick surface oxidized layer. As both the Si recess and remaining dislocated Si, including positive charges, cause the degradation of electrical performance, precise monitoring of the surface structure and understanding its effect on device performance is indispensable for creating advanced devices. © *2011 American Vacuum Society*. [DOI: 10.1116/1.3596606]

I. INTRODUCTION

As the dimensions of ultralarge-scale integrated devices continue to shrink, it is necessary to suppress both the fluctuation of the critical dimension of the gate electrode and damage to the Si substrate to within several atomic layers during etching. One of the most critical issues in advanced devices is the reduction of Si substrate damage caused by energetic ion bombardment, which causes the "Si recess" structure during gate electrode etching.[1–4] HBr/O_2 plasma is generally used for the overetching steps of gate electrode etching due to the high etching selectivity of poly-Si over gate SiO_2. Hydrogen ions in the HBr/O_2 plasma generate a deep damaged layer in the Si substrate through the thin gate oxide. Oxygen, which is exposed to the samples simultaneously, is diffused through the damaged $SiO_x(H_y)$ and oxidizes the underlying dislocated Si, resulting in the generation of a thick oxidized layer on the surface. After etching, a wet treatment is performed to remove the oxidized layer, resulting in the Si recess structure (Fig. 1).[1] Hydrogen ions are reported to generate the deep damaged layer and cause the degradation of electrical properties in the mixture of fluorocarbon chemistry to etch the dielectric film.[5–11] However, a detailed analysis of hydrogen-induced damage in HBr/O_2 plasma and the impact of this damage on electrical performance has not yet been performed.

In this work, the damage generation by HBr/O_2 plasma exposure and the removal of damage by wet treatment was investigated. As a variety of ion species are exposed to the Si substrate in HBr/O_2 plasma, we initially investigated the damage caused by H_2 and O_2 plasma exposure to simplify the complex damage generation processes. We also investigated Ar plasma-induced damage as a reference. Ion energy, flux, and doses were fixed for all plasma conditions. Next, we studied the HBr/O_2 plasma-induced damages and the impact of this damage on electrical performance.

Destructive analytical methods such as high-resolution Rutherford backscattering spectroscopy (HRBS), SIMS, and TEM have been widely used for analyzing the damaged layer. Thus, we developed a simplified and nondestructive method of monitoring Si substrate damage by using conventional spectroscopic ellipsometry (SE) with our optical model for

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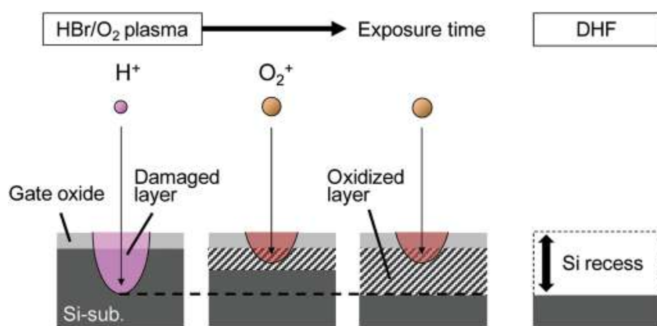

FIG. 1. (Color online) Model of enhanced surface oxidation and Si recess generation.

HBr/O_2 plasma-induced damages. We also compared the damage thicknesses measured by SE, HRBS, and TEM.

II. EXPERIMENT

We used a dual frequency capacitively coupled plasma (CCP) reactor to compare the damages generated by H_2, Ar, and O_2 plasma in an *n*-type (100) Si substrate. A 60 MHz rf power was applied to the upper electrode and a 2 MHz power to the bottom electrode. The high energy peaks of the simulated ion energy distribution function (IEDF) for all gas chemistries were nearly equal to the peak-to-peak voltage (V_{pp}), measured by a high-voltage probe. The plasma density, which is required for IEDF calculation, was measured with a plasma absorption probe.[12] The high energy peaks of IEDF were kept constant at 500 ± 50 V by adjusting the source power while the bottom power was fixed. The estimated V_{dc} was approximately 260 V. The incident ion energies and ion fluxes to the samples were almost constant for all conditions. The calculated ion flux and total dose were 1.44×10^{16} cm^{-2} s^{-1} and 8.64×10^{17} cm^{-3}, respectively. The pressure was fixed at 40 mTorr.

A dual-frequency CCP reactor (60/13.56 MHz) was used to study the Si recess. A 60 MHz rf power was applied to the upper electrode and a 13.56 MHz power to the lower electrode. An SiO_2 layer (1.7 nm) was grown on the Si substrate by using an *in situ* steam grown (ISSG) process. The ISSG process is a wet oxide process in which steam is generated in close proximity to the wafer surface in contrast to the conventional furnace wet oxidation.[13,14] We assume that the projected ranges of incident ions in the ISSG oxide are the same as that in conventional thermal oxide. The samples were exposed to HBr/O_2 and H_2 plasmas. The V_{pp} was kept constant at 400 V. The pressures were 60 mTorr for the HBr/O_2 plasma and 30 mTorr for the H_2 plasma. The plasma exposure time was varied from 3 to 600 s. The temperature of the bottom electrode was 60 °C. Diluted-HF (DHF; 1:100) was used to perform a wet treatment (2 min).

The Si substrate damage was analyzed by spectroscopic ellipsometry (SE), HRBS, and TEM. In the SE analysis, data were fitted using a three-layer model (SiO_2/dislocated Si/substrate)[15] wherein dislocated Si was modeled as a mixture of SiO_2 and polysilicon by using the Bruggeman effective medium approximation.[16,17] Current–voltage (C–V) characteristics were measured with a mercury probe system without introducing any additional damage by forming electrodes. The samples were assumed to be MOS [metal (Hg)/oxide (damaged layer)/semiconductor (Si substrate)] structures with *n*-type (100) Si (0.01–0.02 Ω cm) used as a substrate and the area of the metal was 2.1×10^{-2} cm^2.

III. RESULTS AND DISCUSSION

A. Structural characterization of plasma damage to the Si substrate (H_2 versus Ar versus O_2 plasma)

Figure 2 shows the TEM images and depth profiles of HRBS spectra for the Si substrates exposed to H_2, Ar, and O_2 plasma. Dislocated Si means that the Si has been moved from the Si crystal structure and a dislocated Si layer exists in all samples under the surface oxidized layer. The thicknesses of both the oxidized and dislocated Si layers are quite consistent with previously reported results measured by spectroscopic ellipsometry.[15] The depth of H-induced damage is more than 20 nm, which is much deeper than those of the Ar- and O-induced damages. The depth profile of the damaged layer is strongly related to the penetration depth of incident ions. The H (small mass number) can penetrate deeper than Ar and O with the same incident energy (Fig. 3). The ion penetration depth was calculated using a Monte Carlo simulation (SRIM)[18] which is based on the assumption that the stopping medium is disordered, i.e., amorphous. Thus, the SRIM calculation does not account for channeling effects. However, the calculated projected range (R_p) is supposed to be almost the same regardless of an amorphous or crystal substrate in these experimental conditions. Only the ions, whose trajectory is aligned along the crystal structure, affect the maximum penetration depth of ions in the Si substrate. The surface oxidation of H-induced damage is suppressed despite the fact that the analyses were performed after air exposure. In the case of Ar-induced damages, however, surface oxidation (SiO_x layer formation) occurred. The number of incident atoms existing in a unit volume around the R_p is much higher for Ar plasma than for H_2 plasma (Fig. 3), causing the different types of damage and consequently resulting in different surface oxidations. As the dominant stopping power of H in this energy range is electronic, while that of Ar is nuclear,[19] the generated defects in the damaged layer are different, which can lead to different types of oxidation. Any Si–H bond, which is formed by the termination of an Si dangling bond, is also thought to suppress the oxidation at room temperature.[20] The highest atomic percentage of oxygen, except for the surface of the Si substrate exposed to Ar plasma, is located on the R_p of incident Ar. This means that a higher degree of damage causes a higher degree of oxidation in the damaged layer during air exposure. Not only the quality but also the degree of damage affects the oxidation of the damaged layer. In the case of O_2 plasma, the SiO_2 was formed during the plasma exposure. In short, the surface structure strongly depends on the incident ion species, ion energy, and oxidation during air and plasma exposure.

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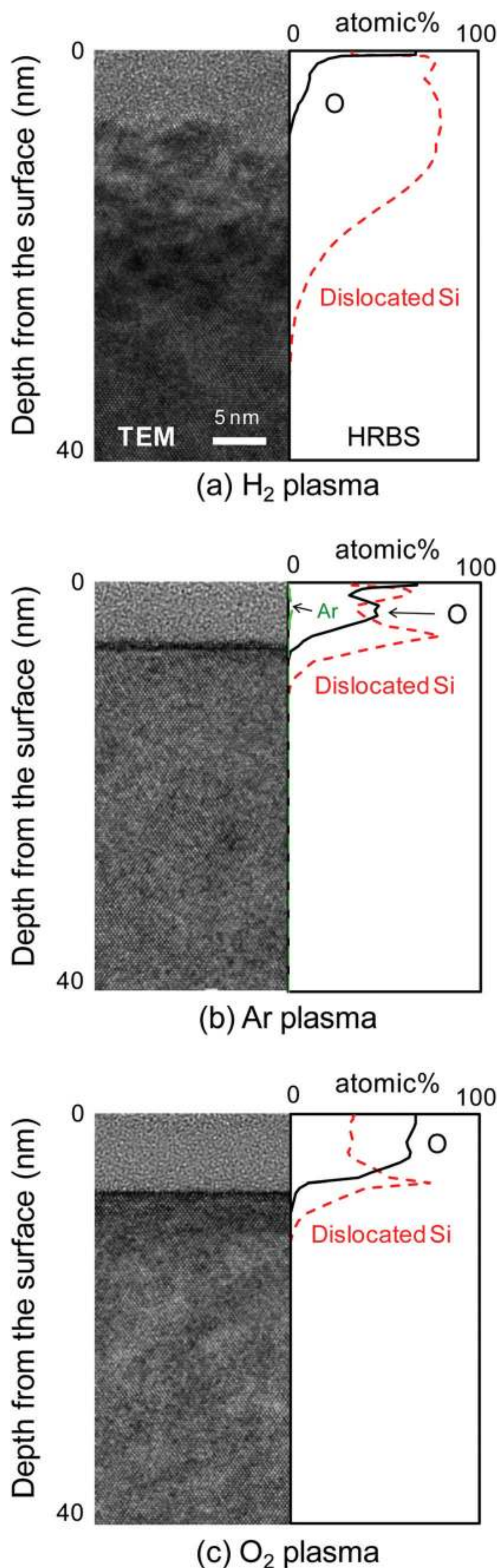

FIG. 2. (Color online) TEM images and depth profiles of HRBS spectra of Si substrate with 1.7 nm gate oxide after (a) H_2 plasma exposure, (b) Ar plasma exposure, and (c) O_2 plasma exposure. The V_{pp}, ion flux, and ion dose are fixed at 500 V, 1.44×10^{16} cm^{-2} s^{-1}, and 8.64×10^{17} cm^{-3}. The scales of y axes are the same for all graphs.

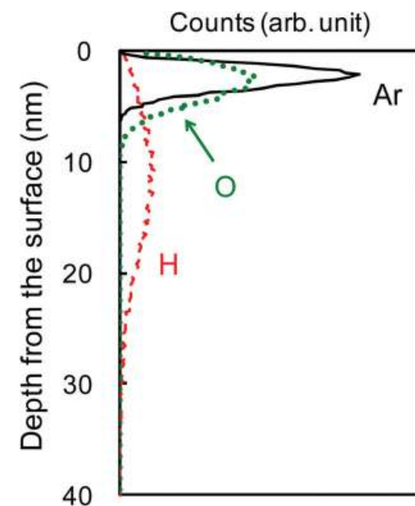

FIG. 3. (Color online) Simulated depth profiles of H, Ar, and O in Si substrate by SRIM. The incident ion energy is 500 eV.

B. Damage removal by DHF treatment (H_2, Ar, or O_2 plasma-induced damages)

Different surface structures cause the different types of damage removal by wet treatment. Figure 4 shows the depth profiles of HRBS spectra for Si substrates after DHF treatment. Most of the H-induced damage remains even after DHF treatment due to less surface oxidation. In the case of Ar and O_2 plasma exposure, the dislocated Si layer still remains, while the surface oxidized layer and upper part of the dislocated Si are removed. The remaining dislocated Si is reported to be a carrier trap site, which causes the degradation of device performance.[15,21,22]

C. Structural characterization and in-line monitoring of HBr/O_2 plasma damage to the Si substrate

Precise in-line monitoring of surface oxide and underlying dislocated Si without cleaving the commercial wafers is required because these damaged layers cause the fluctuation and/or degradation of device performance.[21,22] Thus, we developed monitoring methods for HBr/O_2 plasma-induced damages by using SE.[15] Figure 5 shows a comparison of these damages measured by SE, HRBS, and TEM. The thicknesses of the surface oxidized layer (T_{ox}) and the underlying dislocated Si layer (T_d) are summarized in Table I. The results were quite consistent across all analyses, demonstrating that the in-line monitoring of damage thickness by SE can be applied to mass production. The measured T_{ox} is thicker than the oxygen ion penetration depth in this experimental condition.[1] The thick oxidation is caused by the ion-enhanced diffusion of oxygen during HBr/O_2 plasma exposure.

D. Ion-enhanced diffusion of oxygen through the surface oxidized layer

Some earlier studies report that the ion-enhanced diffusion of oxygen through the SiO_2 in H- and O-containing plasma causes the excess oxidation under the thin gate oxide.[1,4] Ohchi *et al.*[1] proposed that the deep damage layer

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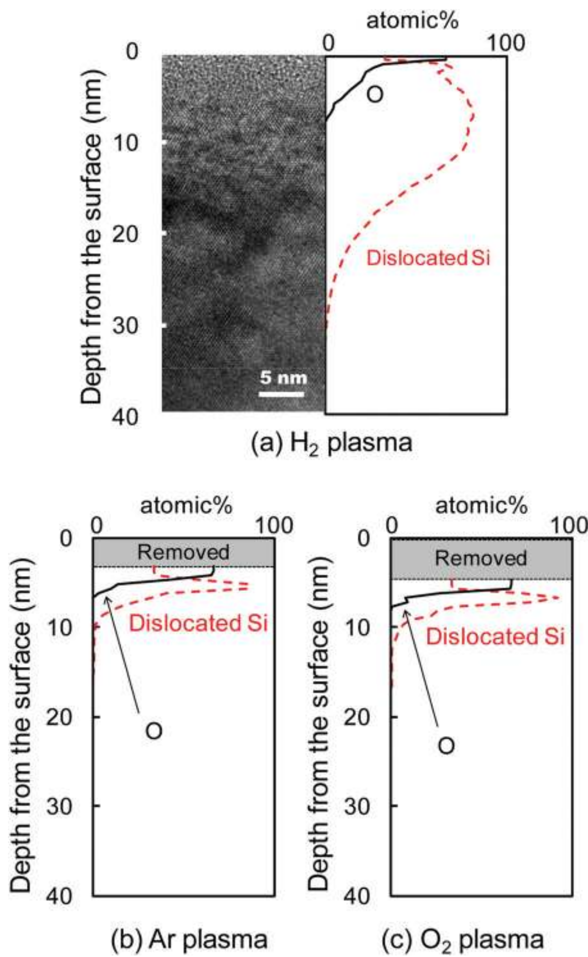

FIG. 4. (Color online) Depth profiles of HRBS spectra of Si substrate after DHF treatment. The removed thicknesses were calculated by comparing the HRBS spectra from pre-and post-DHF treatment. A TEM image is also shown for H_2 plasma-exposed sample.

caused by hydrogen penetration enhances the oxygen diffusion through the damaged $SiO_x(H_y)$ layer. Ito *et al.* studied these phenomena by using a beam system with an oxygen radical source and reported that the oxidation of a Si surface

TABLE I. Thickness of T_{ox} and T_d.

	SE	HRBS[a]	TEM
T_{ox} (nm)	4.1	4.0	4.2
T_d (nm)	1.4	1.2	1.6

[a]Thicknesses were defined by the positions at half-peak intensities.

exposed to oxygen radicals is enhanced when it is simultaneously subjected to energetic hydrogen ion bombardment.[23,24] Figure 6 shows a comparison of the depth profiles of HRBS spectra of Si substrates exposed to HBr/O_2 and H_2 plasma. Oxygen is diffused deeper than 10 nm along with dislocated Si generated by the hydrogen penetration. To investigate the ion-enhanced diffusion in detail we measured the T_{ox} as a function of exposure time by SE (Fig. 7). The T_{ox} was almost constant at 2 nm below 10 s [region (I)]. In region (I), the T_{ox} thickness is dominated by the oxygen ion penetration depth where sufficient oxygen ion flux to the substrate causes immediate ion-induced oxidation. In region (II), the T_{ox} was found to depend on $t^{1/2}$, thus demonstrating a so-called parabolic relationship (diffusion-controlled oxidation) in the classical Deal–Grove model.[25] The parabolic oxidation law is described as

$$T_{ox}^2 = B(t+\tau), \qquad (1)$$

where B is the parabolic rate constant, t is the processing time, and τ is a lag time associated with the thickness of ion-induced oxidation. The fitted B for oxidation by HBr/O_2 plasma in region (II) is 6.87×10^{-4} μm^2/h. This value is higher than that for the oxidation of silicon in dry oxygen at 700 °C ($\sim 4 \times 10^{-4}$ μm^2/h for 760 Torr).[25] The maximum depth of enhanced diffusion is limited by the hydrogen ion penetration depth. If the diffusion depth of oxygen exceeds the maximum penetration depth of hydrogen, the value of B could decrease drastically. Vitale and Smith have proposed a depth-dependent reaction rate constant to include the ion-enhancement effect of oxygen diffusion by modifying the Deal–Grove model.[4] In our study, however, B does not

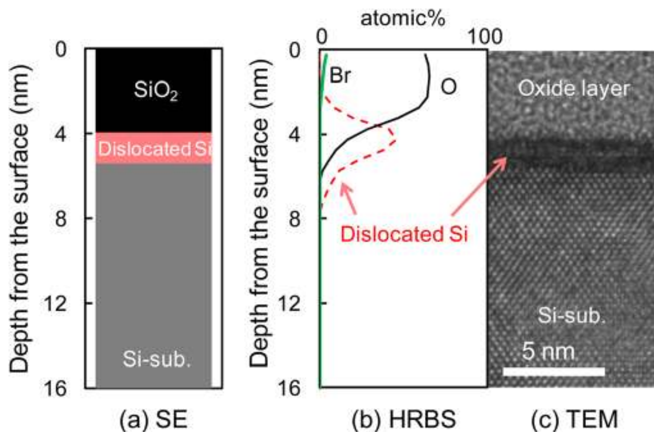

FIG. 5. (Color online) Comparison of surface structure after HBr/O_2 plasma exposure measured by SE, HRBS, and TEM. Plasma exposure time was 60 s. The V_{pp} of the bottom electrode was 400 V. Dual frequency CCP (60/13.56 MHz) was used.

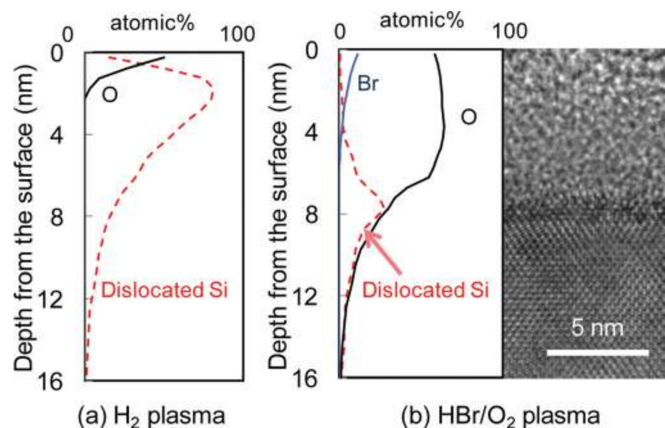

FIG. 6. (Color online) HRBS depth profile and TEM image of Si substrate after (a) H_2 and (b) HBr/O_2 plasma exposure. Plasma exposure time was 600 s. The V_{pp} of the bottom electrode was 400 V. Dual frequency CCP (60/13.56 MHz) was used.

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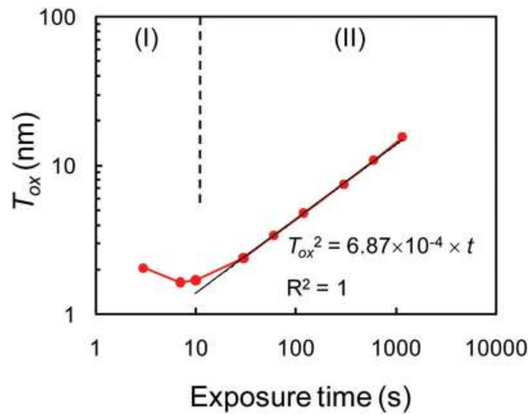

FIG. 7. (Color online) T_{ox} as a function of plasma exposure time measured by SE. To investigate the oxygen diffusion from the plasma precisely, only the Si substrate without gate SiO_2 (1.7 nm) was used. Native oxide only remains on the Si due to the difficulty of its complete removal.

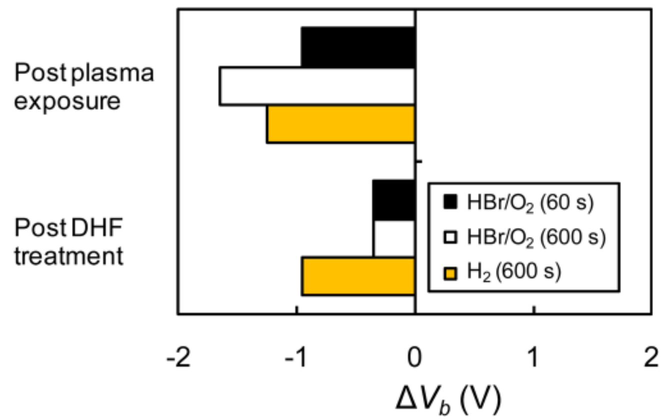

FIG. 8. (Color online) ΔV_b of C–V characteristics. "Post plasma exposure" denotes the ΔV_b between the plasma-exposed and the untreated samples. "Post DHF treatment" denotes the ΔV_b between the subsequent DHF-treated and the untreated samples.

have depth dependence because the oxidation process is diffusion-limited due to sufficient ion flux and ion penetration depth. If the ion flux is relatively low compared to the neutral oxygen flux and/or the ion penetration depth is shallower, the oxygen diffusion could be an ion flux or ion penetration depth-limited process. A systematic study is required for further understanding. Energetic hydrogen ion bombardments break Si–Si and Si–O bonds and generate the highly concentrated defects on the surface, and this excess concentration of defects is expected to lead to an enhancement of oxygen diffusion. However, Ito *et al.* reported that the Si and SiO_2 damage does not assist faster diffusion of oxygen when a sequential application of H_2 plasma and O_2 plasma is applied. Enhanced oxygen diffusion was only observed in the case of simultaneous injections of hydrogen and oxygen.[23,24] Localized heating by ion bombardment might be the origin of accelerated diffusion of oxygen atoms and molecules. It is also possible that the ultraviolet (UV)/vacuum ultraviolet (VUV) radiation from the plasma or the synergy of impinging ions and UV/VUV radiation assists with oxygen diffusion.[26]

E. Electrical characterization of HBr/O_2 plasma damage

Understanding what effect the damage has on device performance is indispensable in the fabrication of advanced devices. Eriguchi and co-workers reported that both the Si recess and remaining damage after DHF treatment cause the degradation of device performance,[21,22] and therefore the C–V characteristics of MOS structures for H_2 plasma- and HBr/O_2 plasma-exposed samples were analyzed.[21] We evaluated the bias-voltage shift (ΔV_b) in C–V curves, which is defined as the difference of the applied voltages at 14 nF (Hg-contact area of 2.1×10^{-2} cm^2) between the damaged sample and the untreated sample. Further details of the C–V measurements are presented elsewhere,[21] but briefly, the ΔV_b is considered to be equivalent to the flatband voltage (V_{fb}) shift widely used for the conventional metal-oxide-semiconductor (MOS) characterization. Figure 8 shows the ΔV_b due to the plasma exposure and subsequent removal by DHF treatment. A negative bias voltage shift was observed after plasma exposure, which suggests that positive charge (hole) trapping was generated.[27] Most of the positive charges generated by H_2 plasma exposure remained, even after DHF treatment. This result is consistent with the structural analysis results shown in Fig. 4, namely that less oxidation of the dislocated Si layer suppresses the removal of damaged Si. In the case of HBr/O_2 plasma, longer exposure time generates a larger amount of positive charges. It is possible that the generated positive charges exist at the surface oxide/dislocated Si interface and/or in the bulk oxide. After DHF, most of the positive charges are eliminated for both HBr/O_2 plasma exposed samples. Figure 9 shows the HBr/O_2 plasma-induced damage after DHF treatment as measured by SE and TEM. The result shows that the dislocated Si still remains on the surface while the surface oxidized layer is completely removed. The remaining dislocated Si causes the slight

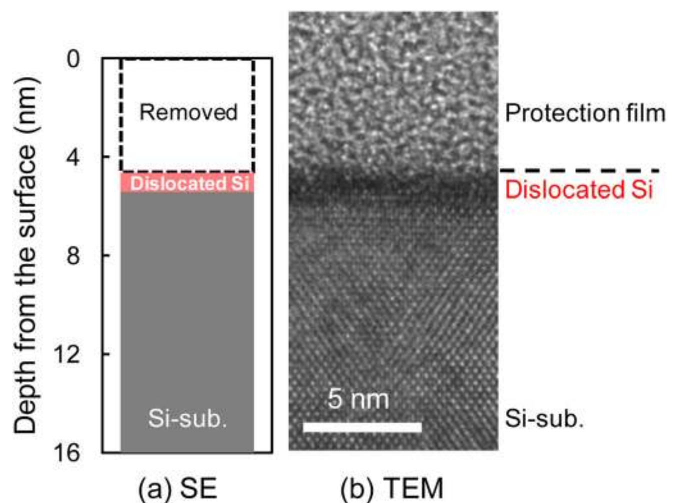

FIG. 9. (Color online) HRBS depth profile and TEM image of Si substrate caused by HBr/O_2 plasma exposure after subsequent DHF treatment. Plasma exposure time is 60 s. The surface oxidized layer is completely removed. The measured thickness of dislocated Si is 1.3 nm by TEM. The removed thicknesses were calculated by comparing the SE and TEM results of pre- and post-DHF treatment.

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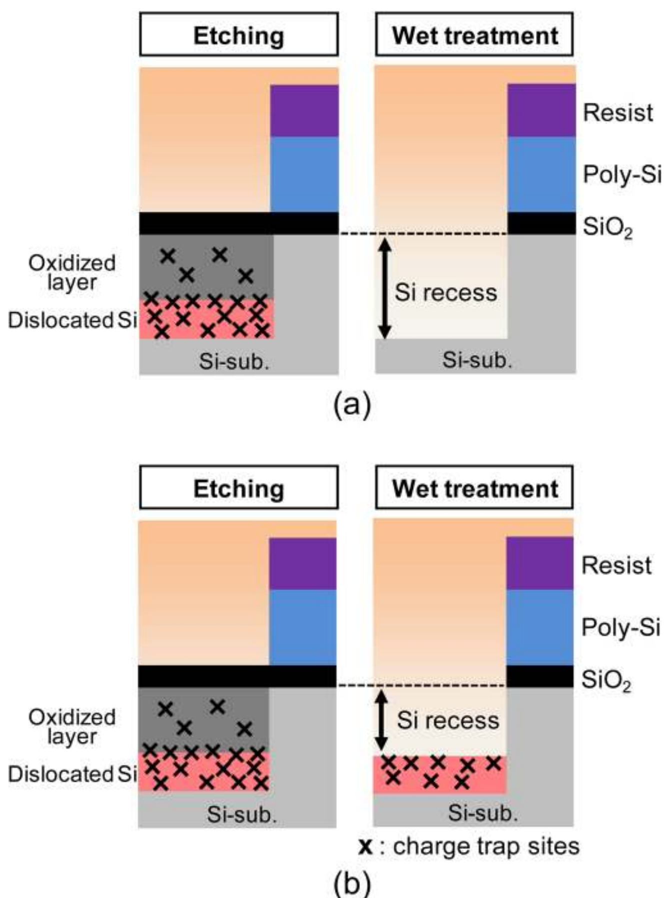

FIG. 10. (Color online) Schematic illustration of the trade-off relationship of thicknesses between the Si recess and dislocated Si with (a) "excess" wet treatment, which removes the oxidized layer and dislocated Si completely, and (b) "soft" wet treatment,which removes the oxidized layer to minimize the Si recess.

negative shift of ΔV_b. Further analysis of the electrical characteristics is required to understand the effect of the remaining dislocated Si, since the analytical model by Eriguchi and co-workers predicted that the effect of damage on device performance increases as the device size is shrunk. In addition, annealing is generally performed in an attempt to restore the device performance and/or to annihilate generated defects. Therefore, an additional study on the restoration of generated charges and dislocated Si by annealing is the subject of a future study.

Figure 10 shows a schematic of the generation and removal of the Si recess and dislocated Si. The excess wet treatment, which can completely remove the dislocated Si, causes an increase of recess depth [Fig. 10(a)]. To reduce the Si recess, one can use the relatively "soft" wet treatment, which causes an increase of the dislocated Si layer [Fig. 10(b)]. Annealing has possibilities to remove the generated charges and related defects completely. However, earlier studies report that the damage is not completely removed by annealing in certain etching and annealing conditions.[28,29] In the case that the annealing completely annihilates generated defects, it is of primary importance to reduce the physical Si recess because the recess structure is reported to induce the threshold voltage (V_{th}) shift [leading to an off-state leakage current (I_{off}) increase] and V_{th} instability of MOSFETs.[22] In the case in which generated defects still remain even after annealing processes, the Si recess and residual damage have a trade-off relationship.[30] Thus, we need to apply an optimum wet treatment as well as a sufficient annealing process after understanding the effect the Si recess and dislocated Si layer have on advanced device performance.

IV. SUMMARY

Si substrate damage caused by HBr/O_2 plasma exposure and damage generated by H_2, Ar, and O_2 plasma exposure were investigated by SE, HRBS, and TEM. The impact of plasma-induced damage on electrical properties was also investigated by studying the *C*–*V* characteristics of an MOS capacitor. Results demonstrated the following:

(1) The depth of dislocated Si caused by H_2 plasma is much deeper than those caused by Ar or O_2 plasma-induced damage. This greater depth is strongly related to the penetration depth of the ion species.
(2) In the case of HBr/O_2 plasma-induced damage, hydrogen generates the deep damaged layer (~10 nm) and ion-enhanced diffusion of oxygen causes the thick surface oxidation.
(3) The accurate in-line monitoring of damage thickness can be applied to mass production by using SE with the developed three-layer optical mode (SiO_2/dislocated Si/substrate).
(4) Positive charge is generated at the surface oxide–dislocated Si interface and/or in the bulk oxide after HBr/O_2 plasma etching.
(5) Most of the generated positive charges were removed by subsequent DHF treatment, however, the thickness of the Si recess increased. The thickness of the Si recess and the amount of residual charges in the dislocated Si have a trade-off relationship in the case that the dislocated Si still remains even after annealing processes.

Therefore, precise monitoring of surface structure and understanding the effects of damage on device performance is indispensable for the development and fabrication of future scaled devices.

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