Influence of microwave annealing on optical and electrical properties of plasma-induced defect structures in Si substrate

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The authors investigate the effects of microwave annealing (MWA) on the recovery of plasma process-induced ion-bombardment damage in Si substrates. For damage creation, Ar discharges by a capacitively coupled plasma etcher are used. For damage repairing, the MWA or a rapid thermal annealing (RTA) are conducted. The authors employ spectroscopic ellipsometry (SE), photoreflectance spectroscopy (PRS), and capacitance–voltage (C – V) measurement to analyze the damaged structures. Recovery of the damage is discussed by comparing the obtained parameters before and after the annealing processes. In the SE analysis, the change in the real part of dielectric constant ($\Delta\epsilon_r$) is investigated. The $\Delta\epsilon_r$ spectral peak around 3.4 eV decreases with an increase in the annealing temperature (T_a) for both MWA and RTA, indicating the decrease in the density of defects created in the Si substrate. In the PRS analysis, the spectral peak intensity is found to decrease by the plasma exposure, and then to increase with T_a , which implies the recovery of the Si crystalline structure by MWA as well as by RTA. In the C – V measurement, the voltage shift (ΔV_b) in the respective C – V curves is used as a measure of the number of defects present in the surface and interface damaged regions. The ΔV_b in the negative bias direction is observed after the plasma exposure for all damaged structures, while after annealing the ΔV_b in the positive bias direction is confirmed, suggesting the recovery of the damage in the surface and interface regions. Moreover, it is experimentally found that MWA induces larger $|\Delta V_b|$ than RTA. These findings indicate the decrease in carrier trap sites in the surface and interface regions of damaged structures, and that MWA is more effective in repairing such damage than RTA. The authors propose a model explaining these mechanisms where defect-induced dipole moments that interact with the incident microwave are considered. The present results draw a new damage-recovery picture by MWA, in particular, for plasma-induced damage in surface and interface regions of Si substrates. © 2015 American Vacuum Society.

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I. INTRODUCTION

Increasing demands for higher performance large-scale integration circuits have required aggressive scale down of device feature sizes.¹ Shallow diffusion length and stringent control on dopant activation in the junction of metal–oxide–semiconductor field-effect transistor (MOSFET) is indispensable. To control the diffusion, low thermal budget using rapid thermal annealing (RTA)² and laser annealing^{3–5} is requisite. A microwave annealing (MWA) technique has been proposed as an alternative candidate for lower thermal budget, which may replace the conventional annealing methods. It was reported^{6–8} that the dopant activation by an MWA technique could be done at a lower temperature than

an RTA, which can potentially enable shallower junction and a higher amount of activated dopant. Another thermal diffusion process applied to silicidation by MWA was also studied.^{9,10} Yamaguchi *et al.*¹¹ reported that performance of complementary-MOS devices was improved by MWA. They also pointed out that the created defect structures were reconstructed by MWA. In general, incident microwave is considered to interact with defects such as dislocation and vacancy, because those defect structures have the induced dipoles.¹² A model of resonant coupling with microwave in dipoles has been proposed.¹³

Plasma etching is widely employed in the fabrication of microelectronics. The critical dimension control of a few nanometers in 300 mm wafers is required for advanced devices such as Fin-FETs, where surface reaction within several atomic layers is precisely designed. In general, in

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such regimes, the precise control is inhibited by the damage induced by plasma etching. The damage is classified as three categories from its mechanisms—charging damage, radiation damage, and ion bombardment damage. The ion bombardment damage is induced by high-energy ions impacting on the surface of materials. In this study, we focus on this ion bombardment damage to Si substrates, which is denoted as plasma-induced physical damage (PPD) hereafter. The PPD structure is consisted of an amorphous-like damage layer on the surface and local point defects in the substrate. Since it is widely believed that the PPD degrades device performance,^{14,15} repairing of the PPD by thermal annealing techniques has been extensively studied for realizing high performance MOSFETs. Comprehensive guidelines for the recovery of PPD with an RTA process were presented, addressing that the reconstruction process requires higher activation energy than the conventional dopant activation in shallow junctions.^{16,17} However, such high temperatures for the PPD recovery are usually unacceptable in the modern MOSFET manufacturing. From the previous studies of dopant activation and silicidation by MWA techniques, one can expect the resonant coupling between microwave and the plasma-induced defect under the temperature lower than conventional thermal annealing techniques such as RTA. However, there have been few reports discussing the microwave effects on PPD recovery. In this paper, we investigate in detail the influence of MWA on the structural change in plasma-damaged samples by using optical and electrical analysis techniques.

II. EXPERIMENT

A. Sample treatments by plasma etcher and MWA system

Figure 1 illustrates process flow of the experiments in this study. A chemical SiO₂ (2 nm) layer was formed on n-type (100) Si substrates. They were exposed to Ar plasma in a dual-frequency (60/13.56 MHz) capacitively coupled plasma (CCP) etcher. Figure 2(a) shows a schematic diagram of the CCP etcher. Incident ion energy during the exposure was controlled by an RF power (P_{rf})

FIG. 1. Process flow of sample treatment performed in this study. Schematic illustration of samples structure is shown below.

applied to a bottom electrode. The RF power ranged from 30 to 500 W. Ar flow rate and the operation pressure were kept at 300 sccm and 3.0×10^{-2} Torr, respectively. The processing time was 60 s. After the plasma exposure, the samples were annealed by MWA or RTA systems. Figure 2(b) schematically illustrates the MWA system equipped with a 5.8 GHz magnetron source and a pyrometer.¹⁸ Incident microwave was controlled by the magnetron source, and the power was varied from 450 to 1000 W. Figure 3 shows the profiles of the Si wafer temperature in the course of annealing time. The temperature was monitored with the pyrometer from the backside. The annealing temperature (T_a) was defined by the maximum temperature, giving the T_a ranging from 400 to 600 °C. The annealing time (t_a) was defined by a period of microwave irradiation of 300 s. We annealed the samples also by the RTA system for comparison. T_a and t_a in the RTA process were set to be the same as those in the MWA process. After annealing, we removed a surface layer by diluted hydrofluoric acid solution. For the analysis of photoreflectance spectroscopy, a chemical oxide layer was formed again by hydrochloric/peroxide mix¹⁹ solution. Table I shows the plasma exposure and MWA/RTA conditions employed in this study.

FIG. 2. (a) Dual-frequency capacitively coupled plasma etcher used for damage creation. (b) Microwave annealing system used in this study.

FIG. 3. Time evolutions of the temperature monitored from the backside by samples by a pyrometer under various microwave powers. The annealing temperature is defined as the maximum value.

B. Damage evaluation techniques

Energetic ion bombardment during plasma exposure forms both the surface and interfacial damaged layers and the localized defects beneath the Si substrate surface.^{20–22} The surface and interfacial regions are denoted as a surface SiO₂ layer ("SL") and an interfacial layer ("IL"), respectively. Figure 4 provides cross sectional transmission electron microscope (TEM) images of the damage structures with and without plasma exposures and MWA. Although the growth of the SiO₂ layer was clearly observed by the plasma exposure, defects ascribed to the PPD were undetectable in the TEM image. In order to clarify the damage, we employed spectroscopic ellipsometry (SE) and photoreflectance spectroscopy (PRS) as the optical analyses.

The SE technique is widely used for studying structural and material properties^{23–25} and as in-line monitoring for PPD measurement.^{22,25–27} In the present experiments, the SE spectra were obtained in the photon energy range from 1.5 to 5.5 eV. We determined the optical thickness of SL and IL. The SL was modeled as SiO₂ phase and the IL, as a mixture

FIG. 4. TEM images of samples (a) before plasma exposure, (b) after the exposure, and (c) after MWA.

of crystalline-Si and SiO₂ phase using Bruggeman's effective medium approximation.²⁷ Figure 5 shows the optical thicknesses of SL and IL in the damaged samples for P_{rf} from 30 to 500 W. Figure 5 also shows the unbiased estimator χ^2 for model fitting of the SE spectra under various plasma conditions. As seen, the increase in χ^2 is confirmed for the case of $P_{rf} = 500$ W, indicating that the optical model used in the SE analysis is not applicable due to the heavy damage under that condition. In this study, we focus on the differences in the real part of dielectric constants ($\Delta\epsilon_r$) before and after the plasma exposure, and before and after annealing processes.

We performed PRS, a highly sensitive optical technique for surface property analysis of the Si substrate.²⁸ The PRS is applied also to quantitative analysis of PPD.^{29–32} The photoreflectance (PR) spectrum is described as³³

$$\Delta R/R = \Re[Ce^{i\theta}(E - E_g + \Delta\Gamma)^{-n}], \quad (1)$$

where C is the amplitude, θ the phase factor, and the exponent n depends on dimensionality of the critical points corresponding to the structure of conduction and valence bands of

TABLE I. Plasma exposure and MWA/RTA conditions performed in this study.

ID	Plasma exposure P_{rf} (W)	Anneal		
		Process	T_a (°C)	P_{MW}^a (W)
1	30	—	—	—
2	100	—	—	—
3	200	—	—	—
4	500	—	—	—
5	30	MWA	600	1000
6	30	MWA	500	600
7	30	MWA	400	450
8	30	RTA	600	—
9	30	RTA	500	—
10	30	RTA	400	—

^aMicrowave power.

FIG. 5. Change in the optical thicknesses of SL and IL in the damaged samples determined by SE.

Si. A peak of ideal PR signal in a Si substrate appears around 3.4 eV, which reflects the direct band gap of E_1 and/or E_0 transitions. The two transitions E_1 (L point transition) and E_0 (Γ point transition) are corresponding to the two-dimensional³⁴ ($n=3$)³³ and the three-dimensional critical point³⁴ ($n=2.5$)³³, respectively. The present PRS system^{29–31} is equipped with two light sources. A Xe discharge lamp is used for the probe light, and a solid-state laser (527 nm), for a modulation beam chopped at 500 Hz by a mechanical chopper. The probe beam reflected on a Si surface is detected as the reflectance (R) by a photomultiplier. While the modulation beam is irradiated on the surface, the reflectance change ($\Delta R/R$) is obtained as the signal that matches the frequency of the chopper. We measured the $\Delta R/R$ signal as an indicator of the PPD created in the samples by plasma exposure and the recovery by MWA and RTA.

Capacitance–voltage (C – V) characteristics were obtained using a mercury (Hg) probe system.³⁵ We constructed a pseudo-MOS structure consisting of the Hg electrode, the chemical SiO_2 , and the Si substrate, with a contact area of 0.02 cm^2 . In the C – V measurement, the voltage was swept from the negative (inversion) to the positive biases (accumulation). The frequency of voltage modulation superimposed on the DC bias was 100 kHz. We define a characteristic V_b as a voltage at the measured capacitance of $0.5 \text{ } \mu\text{F}/\text{cm}^2$ and compare the shifts among the control, plasma damaged, and annealed samples. Note that the V_b shift corresponds to the presence of trapped charges³⁶ as

$$\Delta V_b = -\frac{1}{C_i} \left[\frac{1}{d_{\text{SL}}} \int_0^{d_{\text{SL}}} x \rho(x) dx \right], \quad (2)$$

where $\rho(x)$ and d_{SL} are the charge density per unit volume and the SL thickness, respectively. Since $|\Delta V_b|$ is in proportion to the charge trap site in the SL and the SiO_2 –Si interface state, the $|\Delta V_b|$ can be used as a measure of the defect density.

III. RESULTS AND DISCUSSION

A. Optical analysis by SE and PRS

Figure 6(a) shows the real parts of the dielectric constant (ϵ_r) for the control and the samples after the plasma exposures. The ϵ_r for a sample before damage has a maximum peak at 3.3 eV and a minimum peak at 4.5 eV. Note that this spectral shape of Si has been widely discussed in many literatures.^{23–25,33} The peak intensities decrease with an increase in P_{rf} . The change in the dielectric constant ($\Delta\epsilon_r$) spectra is determined from the difference between two SE spectra before/after plasma exposure. It was reported²⁴ that the origin of $\Delta\epsilon_r$ can be classified into three mechanisms: (1) residual-overlayer effect, (2) surface-local-field (SLF) effect, and (3) energy-derivative effect. They are described by

$$\Delta\epsilon'_{\text{overlayer}} = \frac{4\pi d}{E} \epsilon_s^{3/2}, \quad (3)$$

FIG. 6. (a) Real part of pseudo-dielectric function spectra ϵ_r of plasma damaged samples obtained by SE. Spectrum of the control sample is also shown. (b) Representative differences in ϵ_r ($\Delta\epsilon_r$) between the control sample and damaged samples.

$$\Delta\epsilon'_{\text{SLF}} = \frac{4\pi d_g \Delta g}{E} \epsilon_s^{5/2}, \quad (4)$$

$$\Delta\epsilon'_{\Delta E_g \text{ or } \Delta\Gamma} = -\Delta E_g \frac{d\epsilon_s}{dE} + i\Delta\Gamma \frac{d\epsilon_s}{dE}, \quad (5)$$

where $\epsilon'_{\text{overlayer}}$, ϵ'_{SLF} , and $\epsilon'_{\Delta E_g \text{ or } \Delta\Gamma}$ are the real part of dielectric constants for the overlayer effect, the SLF effect, and the energy-derivative effects, respectively. d is the thickness of overlayer, $d_g \Delta g$ is the SLF effective thickness, E is the photon energy, and ϵ_s is the ideal bulk dielectric function of the crystalline–Si substrate.³⁷ These effects originate from the difference in an SL thickness, a modification between the SiO_2 layer and Si substrate associated with an IL region, and derivative-like features in the Si substrate which corresponds to the shift of critical point energies (ΔE_g) and that of broadening parameters ($\Delta\Gamma$), respectively. The residual-overlayer and SLF effects are investigated from the optical thickness as seen in Fig. 5. Now, we focus on the $\Delta\epsilon_r$ applied for the

energy-derivative effects interpreted as the change in the Si band structure around the surface. Figure 6(b) shows the $\Delta\epsilon_r$ spectra for the samples for various P_{rf} . As seen in these spectra, two peaks are clearly presented at around 3.4 and 4.2 eV in the $\Delta\epsilon_r$ spectra. Hence, the dominant factor of the spectrum is considered to be the energy-derivative effect as described in Eq. (5). The 3.4 and 4.2 eV peaks mean the signals of the E_1 and/or E_0 transitions and that with the E_2 transition in the band structure, respectively. The peak intensity at 3.4 eV increases with an increase in P_{rf} . This result indicates the change in the band structure induced by an increase in the number of defects in the Si substrate. Figure 7 plots the peak intensity at (a) 3.4 eV and (b) 4.2 eV as a function of P_{rf} . As seen, the intensity at 3.4 eV monotonically increases with an increase in the P_{rf} , implying heavier damage, although the intensity at 4.2 eV is maintained constant at the P_{rf} between 50 and 500 W. Repairing PPD in IL is classified in terms of the peak intensity at 3.4 eV in the $\Delta\epsilon_r$. Figure 8 shows the $\Delta\epsilon_r$ spectra of the samples by (a) RTA and (b) MWA at the temperature of 400 and 600 °C. The solid lines are the spectra of as-damaged samples for $P_{rf}=30$ W. Figure 9 shows the annealing temperature dependence of the peak intensity for damaged samples for the case of MWA and RTA processes. The $\Delta\epsilon_r$ is found to

FIG. 7. $\Delta\epsilon_r$ at the photon energy of (a) 3.4 eV and (b) 4.2 eV as a function of P_{rf} (see Fig. 6). The solid lines are to guide the reader's eye.

FIG. 8. Change in $\Delta\epsilon_r$ spectra after (a) RTA and (b) MWA processes. (Samples were damaged under the P_{rf} of 30 W.)

FIG. 9. Temperature dependence of $\Delta\epsilon_r$ at the photon energy of 3.4 eV for the samples treated with MWA and RTA.

Fig. 10. PR spectra for damaged samples for various RF powers.

decrease with an increase in T_a , and moreover, Arrhenius behavior for the damage recovery is observable for both MWA and RTA.

Figure 10 shows PR spectra for the damaged samples for the P_{rf} from 30 to 500 W. The peak intensity at the photon energy of 3.4 eV corresponds to the amplitude C in Eq. (1), which is related to the surface potential of a Si substrate. The decrease in the peak intensity with an increase in the P_{rf} means the presence of defects (in the IL and/or the Si substrate) playing a role in capturing photon-generated carriers. The enhanced carrier trapping process suppresses the perturbed electrostatic potential that affects the spectral peak under irradiation of the modulation beam. Therefore, the PPD is evaluated by the amplitude C as discussed previously.³² For the purpose of the PPD evaluation with a high signal-to-noise ratio, we annealed the samples damaged by the plasma exposure with P_{rf} of 30 W as shown in Fig. 11. The PR spectra for as-damaged and annealed samples by RTA and MWA (400 and 600 °C) are shown in the upper half and the lower half of Fig. 11(a), respectively. We obtained the minimal peak intensity around 3.4 eV for various T_a cases. Figure 11(b) shows the temperature dependence of the $\Delta R/R$ peak intensities for MWA and RTA, and it is shown that the intensity increases with an increase in T_a for both MWA and RTA processes, whose dependence seems approximately identical. Therefore, carrier trapping sites created by the PPD are annihilated by annealing.

From the experimental data by the SE and the PRS analyses, which identify the defects present in the IL and/or the Si substrate, the recovery mechanism of the damage by MWA is considered to be similar to that by RTA, which is governed by a macroscopic parameter T_a .

B. Electrical analysis by C–V measurement

C–V curves for various damaged samples are shown in Fig. 12. Large $|\Delta V_b|$ in the negative bias direction is seen in

Fig. 11. (a) PR spectra for as-damaged and annealed samples by RTA (upper half) and MWA (lower half) processes. (b) Temperature dependence of $\Delta R/R$ peak intensity around 3.4 eV for the samples treated with MWA and RTA.

the damaged samples, which indicates that the samples experience positive charge trapping by the PPD in the SL or IL-Si substrate interface. We speculate that the defects in the regions are hole-trapping sites or trapped electrons. The shift of a C–V curve can be used as an indicator of the PPD because it reflects the density of defects in the SL or IL-Si substrate interface. Figure 13(a) provides typical C–V curves obtained for the samples with and without the plasma exposure. This figure also shows the curves for the samples treated by MWA and RTA. The $|\Delta V_b|$ in the positive bias direction is seen after MWA and RTA. This implies that the charge trapping sites by the PPD is annihilated by annealing, i.e., the PPD is recovered. Temperature dependence of V_b is compared in Fig. 13(b) for MWA and RTA cases. The

FIG. 12. C - V curves of the control and damaged samples.

“recovery” of V_b —the decrease in the $|\Delta V_b|$ —is confirmed by annealing from the as-damaged value. Moreover, the larger recovery of V_b is found for MWA for all T_a . This is the main result of this study, namely, that MWA is effective to reconstruct the defect sites in the SL or IL-Si substrate interface, compared to conventional RTA, while there are no apparent difference in repairing the defects created in the IL and the Si substrate as clarified by the SE and the PRS. In Sec. III C, a possible mechanism for these observations is presented.

C. Repairing mechanism by MWA

Microwave power absorption of solid materials is governed by their magnetic, dielectric, and conductive properties. In the case of nonferromagnetic materials, microwave power absorption is directly related to the imaginary part of the dielectric constant as generally described by¹⁰

$$P_{\text{abs}} = \sum_k P_k = \sum_k \omega \varepsilon_0 \varepsilon_k'' |E|^2 \quad (k = \text{SL, IL, c-Si}), \quad (6)$$

where P_{abs} is the microwave power absorbed per unit volume, ω is the microwave frequency, ε_0 is the permittivity in vacuum, E is the root mean square internal electric field, and ε_k'' is the effective loss factor of the respective region. The ε_k'' is determined by integration of the dipole momentums in the regions [SL, IL, and Si substrate (c-Si)]. The defect structures created by plasma exposure are interstitials, vacancies, and dangling or strained bonds. These damaged structures are considered to have larger dipoles due to the asymmetric electrostatic field around the defects, compared with undamaged c-Si and SiO₂ structures. Moreover, under the external electric field, the dipoles eventually align in the direction of the electric field and are reoriented (oscillated) at the same frequency. The lag of aligning the dipole

FIG. 13. (a) C - V curves for the control, damaged ($P_{\text{rf}}=30$ W), and the annealed samples by RTA and MWA. The annealing temperatures were 400 and 600 °C. As seen, after annealing, the C - V curves shift in the positive bias direction. (b) The shift of C - V curves defined at a capacitance of 0.5 $\mu\text{F}/\text{cm}^2$ for various T_a .

leads to dielectric loss, and a certain amount of microwave power is irreversibly transferred to the materials (in the present case, to local defect structures). From the results by the optical analyses and the C - V measurement performed in this study, it is considered that the PPD creates defects (carrier trapping sites) in the SL, the IL, and the Si substrate. The SE and PRS clarified equivalent impacts on the T_a -dependent PPD repairing between MWA and RTA, while the C - V measurement identified the difference in the recovery mechanism. Considering the fact that the SE and the PRS assign the damage structures primarily in the IL and the Si substrate, the PPD is difficult to recover completely in the regions at lower temperatures below 600 °C, which may be

suggested previously.^{16,17} However, the C - V measurement signifies that the PPD in the SL and/or at SL-IL interface is significantly repaired in the case of MWA, which is attributed to the enhanced interaction between the induced-dipole and the incident microwave as mentioned above and in the literatures.^{11,13} Figures 14(a) and 14(b) illustrate the PPD recovery mechanism by MWA. As shown in Fig. 14(a), induced dipoles in the defect structure and the SL are larger than that in the substrate. Thus, all the defects in the damaged sample can potentially interact with incident microwave. Figure 14(b) shows in detail the dipoles by PPD in the SL and the IL regions. Since the SL (heavily damaged region) is easily oxidized by air exposure, it has a larger dipole moment, which can be confirmed from the nature of Si-O bond in the SL. Under the microwave irradiation, those dipoles in the SL can be free to change their direction, i.e., to reconstruct the network. On the other hand, even if the dipoles in the IL and the Si can be coupled with the microwave radiation, it may be more difficult to reconstruct the original network because the defects in the regions are more localized under structural constraints. Therefore, it is concluded that MWA can repair the PPD in the surface and interface regions effectively under low temperatures.

Fig. 14. (Color online) (a) Comparison of induced dipoles that determine the effective loss factors for the damaged and control samples. (b) Schematic illustration of the dipoles due to the presence of defects in the SL and IL of a damaged structure.

IV. CONCLUSIONS

We investigated mechanisms of repairing PPD in a Si substrate under MWA by using optical and electrical analysis techniques. From the optical techniques, the recovery of PPD in the interfacial layer (IL) and the Si substrate is found to obey an Arrhenius relationship with respect to the annealing temperature, irrespective of annealing techniques such as MWA and RTA. Moreover, the electrical C - V measurement identified that MWA effectively repairs the PPD in the surface SiO_2 layer (SL), compared to RTA. We propose a model accounting for the coupling between induced dipoles in the damaged region and incident microwave to explain the findings. Since the dielectric loss factor of the defect structure in the SL is considered to be larger than that in the IL and the Si substrate, the induced-dipoles in the SL are free to change their direction (reconstruct the network) under the microwave irradiation. Thus, reconstruction of the surface PPD can be promoted by MWA at lower temperatures than for the RTA process. Therefore, microwave annealing is a promising technology for resolving critical issues such as plasma-induced surface and interface states in future scaled devices.

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