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# Surface Passivation of Silicon Using HfO<sub>2</sub> Thin Films Deposited by Remote Plasma Atomic Layer Deposition System

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## Abstract

Hafnium oxide (HfO<sub>2</sub>) thin films have attracted much attention owing to their usefulness in equivalent oxide thickness scaling in microelectronics, which arises from their high dielectric constant and thermodynamic stability with silicon. However, the surface passivation properties of such films, particularly on crystalline silicon (c-Si), have rarely been reported upon. In this study, the HfO<sub>2</sub> thin films were deposited on c-Si substrates with and without oxygen plasma pretreatments, using a remote plasma atomic layer deposition system. Post-annealing was performed using a rapid thermal processing system at different temperatures in N<sub>2</sub> ambient for 10 min. The effects of oxygen plasma pretreatment and post-annealing on the properties of the HfO<sub>2</sub> thin films were investigated. They indicate that the in situ remote plasma pretreatment of Si substrate can result in the formation of better SiO<sub>2</sub>, resulting in a better chemical passivation. The deposited HfO<sub>2</sub> thin films with oxygen plasma pretreatment and post-annealing at 500 °C for 10 min were effective in improving the lifetime of c-Si (original lifetime of 1 μs) to up to 67 μs.

**Keywords:** HfO<sub>2</sub> thin films, Atomic layer deposition, O<sub>2</sub> plasma pretreatment, Surface passivation

## Background

High-quality surface passivation is very important for a range of crystalline silicon (c-Si)-based electronic devices, and especially for high-efficiency c-Si solar cells. As the need for lower-cost silicon solar cells increases, since Si material has a rather high cost, thinner Si substrates are required. Therefore, their surface/volume ratio of such substrates and the contribution of their surfaces to the overall performance are increasing. Traditional surface passivation for Si involves the formation of a thin silicon dioxide (SiO<sub>2</sub>) layer. However, this process requires a high thermal budget process, which involves long period at high temperature. Owing to these process-related issues, considerable efforts have been made in the past to develop low-temperature surface passivation methods for both heavily doped and moderately doped c-Si surfaces. Besides SiO<sub>2</sub>, other layers such as SiC, a-Si:H and Si<sub>3</sub>N<sub>4</sub> have been used for surface

passivation [1]. Recently, Al<sub>2</sub>O<sub>3</sub> films that are grown by atomic layer deposition (ALD) have been demonstrated to provide good surface passivation on c-Si [2–4]. ALD technique is a powerful method. It provides a high-level degree of precise control over the properties of the material, and especially the morphology and thickness of dielectric layers.

In the advanced semiconductor industry, hafnium dioxide (HfO<sub>2</sub>) thin films are used to replace SiO<sub>2</sub> as the gate dielectric in field-effect transistors because they have better functionality and performance at lower cost [5, 6]. Additionally, the high refractive index of HfO<sub>2</sub> makes it a potential candidate for anti-reflection coatings [7] and interference filters [8]. However, its surface passivation properties, particularly on c-Si, have scantily been studied. For example, Jun Wang et al. [9] presented the surface passivation properties of a Si surface using a thin HfO<sub>2</sub> layer grown by ALD without further annealing. In another study Huijuan Geng et al. [10] reported advanced passivation using simple materials (Al<sub>2</sub>O<sub>3</sub>, HfO<sub>2</sub>) and their compounds H<sub>(Hf)</sub>A<sub>(Al)</sub>O deposited by ALD. All of the previous attempts were performed to

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deposit  $\text{HfO}_2$  on c-Si substrates without any pre-treatments.

In this work, the surface passivation properties of the  $\text{HfO}_2$  films deposited by a remote plasma atomic layer deposition system (RP-ALD) on p-type c-Si with and without in situ oxygen plasma pretreatment were investigated. Samples were annealed at different temperatures by rapid thermal annealing (RTA) system. The structural changes and the electrical properties of the thin films induced by RTA were characterized by field-emission transmission electron microscope (FE-TEM), X-ray photoelectron spectroscopy (XPS) and capacitance-voltage (C-V) measurements. The passivation mechanism of  $\text{HfO}_2$  films on Si is also investigated.

## Methods

In this study, (100) oriented boron-doped p-type crystalline Czochralski (Cz) Si wafers that were polished on both sides and had a resistivity of  $30 \Omega \cdot \text{cm}$ , original lifetime of  $1 \mu\text{s}$  and a thickness of  $250 \mu\text{m}$  were used. Prior to the deposition of the  $\text{HfO}_2$  film, all wafers were cleaned through a standard Radio Corporation of America (RCA) cleaning process followed by a dip in diluted hydrofluoric acid (HF) solution (5%) for 2 min to remove the native oxide and dried in nitrogen.

The  $\text{HfO}_2$  thin films were grown in an RP-ALD reactor (Model: Picosun, Finland) using tetrakis (ethylmethylamino) hafnium (TEMAH) and remote  $\text{O}_2$  plasma as the precursors for hafnium and oxygen respectively with  $\text{N}_2$  as the carrier gas. In the ALD process, one deposition cycle consisted of two half cycles, one TEMAH pulse (for 1.6 s) and one  $\text{O}_2$  plasma pulse (for 10 s). The nitrogen purge times for TEMAH and  $\text{O}_2$  were 10.0 and 12.0 s, respectively. The samples were divided into two groups. For group one,  $\text{HfO}_2$  thin films were deposited directly on the cleaned Si wafers. For group two, before deposition of  $\text{HfO}_2$  thin films, Si wafers were additionally treated by remote  $\text{O}_2$  plasma for 1 min. The  $\text{O}_2$  plasma power for the pretreatment and for the ALD deposition process was 2500 W. The  $\text{HfO}_2$  films for all of the samples were deposited at  $250 \text{ }^\circ\text{C}$ . Different  $\text{HfO}_2$  thickness (5, 15, and 25 nm) were prepared on as-cleaned Si wafers followed by annealing at  $500 \text{ }^\circ\text{C}$ , and the corresponding minority carrier lifetimes of the passivated wafer were 9.98, 66.8, and  $4.2 \mu\text{s}$ , respectively, at the injection level of  $3 \times 10^{14} \text{ cm}^{-3}$ . Therefore, the thickness of 15 nm (corresponding to 168 ALD cycles) was used. The substrate pre-treatment could affect nucleation, leading to different film thickness. The thicknesses of the deposited  $\text{HfO}_2$  are  $15 \text{ nm} \pm 0.5 \text{ nm}$  and  $13 \text{ nm} \pm 0.7 \text{ nm}$  for the samples with and without the oxygen plasma pretreatment, respectively. The wafer was flat on a platen. The double side coated samples processed twice, with a break in vacuum to flip the wafer in the chamber.

The  $\text{HfO}_2$  thin films were deposited on 2-in wafers. As the substrate holder was about 8 in, four samples were placed on the holder and processed at a time. The samples in the two groups are referred hereafter as SD (direct depositing samples) and SO ( $\text{O}_2$  plasma pretreatment samples), respectively. Annealing process was performed using a RTA system at  $400\text{--}650 \text{ }^\circ\text{C}$  in  $\text{N}_2$  ambient for 10 min. Samples were identified with suffixes A400 to A650 that represent the annealing temperatures. Table 1 lists the samples.

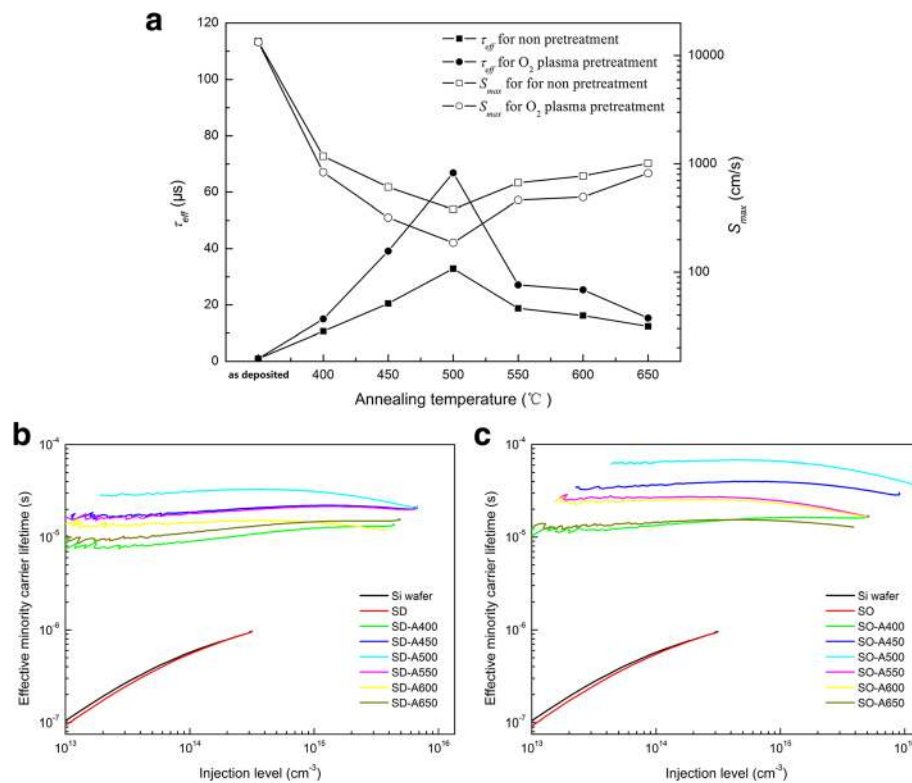
The minority carrier lifetimes ( $\tau_{\text{eff}}$ ) of the samples were assessed by photo-conductance decay method (Model: WCT-120, Sinton lifetime tester) in the quasi-steady state mode. Metal-insulator-semiconductor (MIS) structures were prepared by depositing Al electrodes with diameters of  $500 \mu\text{m}$  onto the passivation layer using a sputter system and a shadow mask. The C-V characteristics were measured with a HP4284A semiconductor characterization system to extract the electrical parameters. The chemical composition and states of elements in the  $\text{HfO}_2/\text{Si}$  were analyzed by XPS (Thermo Fisher K-Alpha). The ion energy used for the depth profile was  $3000 \text{ eV}$ . The physical thicknesses, microstructure and interface properties of the  $\text{HfO}_2$  thin films were determined by FE-TEM (JEM-2100 F).

## Results and Discussion

Generally, the quality of passivation is assessed in terms of  $\tau_{\text{eff}}$  or surface recombination velocity ( $\text{SRV} = S_{\text{max}}$ ). The  $\tau_{\text{eff}}$  refers to the recombination at surface defects. Figure 1(a) plots  $\tau_{\text{eff}}$  and  $S_{\text{max}}$  for all samples at the injection level of  $3 \times 10^{14} \text{ cm}^{-3}$ . The  $\tau_{\text{eff}}$  measurements were performed three times for each sample in the

**Table 1** Details of the  $\text{HfO}_2$  thin films

Sample	$\text{O}_2$ plasma pretreatment	Annealing temperature( $^\circ\text{C}$ )
SD	N/A	N/A
SD-A400	N/A	400
SD-A450	N/A	450
SD-A500	N/A	500
SD-A550	N/A	550
SD-A600	N/A	600
SD-A650	N/A	650
SO	Yes	N/A
SO-A400	Yes	400
SO-A450	Yes	450
SO-A500	Yes	500
SO-A550	Yes	550
SO-A600	Yes	600
SO-A650	Yes	650



**Fig. 1** a  $\tau_{eff}$  and  $S_{max}$  of the samples at the injection level of  $3 \times 10^{14} \text{ cm}^{-3}$ . Injection level-dependent effective minority carrier lifetime of the (b) SD and (c) SO samples

different locations, and the errors of the minority carrier lifetime were within  $\pm 5\%$ . As the annealing temperature was increased from 400 to 500 °C, the  $\tau_{eff}$  of the annealed  $HfO_2$  sample with  $O_2$  plasma pretreatment at the injection level of  $3 \times 10^{14} \text{ cm}^{-3}$  increased significantly. The increase of the annealed  $HfO_2$  samples without  $O_2$  plasma pretreatment at the same injection level was much less than that of the annealed  $HfO_2$  samples with  $O_2$  plasma pretreatment. At lower temperatures ( $T < 500$  °C), the annealed  $HfO_2$  samples without  $O_2$  plasma pretreatment had lower  $\tau_{eff}$  than those with  $O_2$  plasma pretreatment. The annealing process provides energy to the  $HfO_2$  layer to active the passivation. When the annealing temperature higher than 500 °C, the minority carrier lifetime decreases, which might be due to the defects generated by the increased microcrystalline fraction and grain boundaries in the  $HfO_2$  layer. The  $O_2$  plasma pretreatment sample that had been annealed at the temperature of 500 °C had the highest  $\tau_{eff}$  of 67  $\mu s$ , corresponding to an  $S_{max}$  value of 187 cm/s. This calculation was based on the quasi steady-state photo conductance (QSSPC)  $\tau_{eff}$  data for the injection level of  $3 \times 10^{14} \text{ cm}^{-3}$ .  $S_{max}$  represents the upper limit of SRV, and is estimated from the measured lifetime values using the following relation [11],

$$S_{max} = \frac{W}{2\tau_{eff}}, \tag{1}$$

where  $W$  ( $=250 \mu m$ ) is the thickness of the silicon substrate. The lower value of  $S_{max}$  can be attributed to a lower density of interface traps. It also can be seen from Fig. 1a that the  $O_2$  plasma pretreatment samples exhibited better passivation than the directly deposited samples, so they had a lower interface recombination velocity. This difference is attributable to the diffusion of O from the  $O_2$  plasma to the interfacial region to form a  $SiO_2$  thin film, which provides better chemical passivation of the dangling bonds.

Figure 1b, c shows the injection level-dependent effective minority carrier lifetime of the samples without and with  $O_2$  plasma pretreatment. For the SO samples, the minority carrier lifetime increases with the annealing temperature between 400 and 500 °C. All of the SO samples without annealing exhibited almost no passivation, and their  $\tau_{eff}$  values were similar to that of the bare Si wafer. However,  $\tau_{eff}$  of the annealed samples increased significantly and then decreased as the injection level increased from  $4 \times 10^{13} \text{ cm}^{-3}$  to  $5 \times 10^{15} \text{ cm}^{-3}$ . The drop in  $\tau_{eff}$  with increasing injection levels is caused by Auger recombination in the bulk of the c-Si substrate. The  $\tau_{eff}$

of the as-deposited samples depends very strongly on injection level, decreasing by approximately one order of magnitude as the injection level is decreased from  $3 \times 10^{14}$  to  $10^{13} \text{ cm}^{-3}$ . This dependence in injection levels is much weaker for the annealed samples. The  $\tau_{\text{eff}}$  values of the annealed samples decrease only slightly as the injection level is reduced [12].

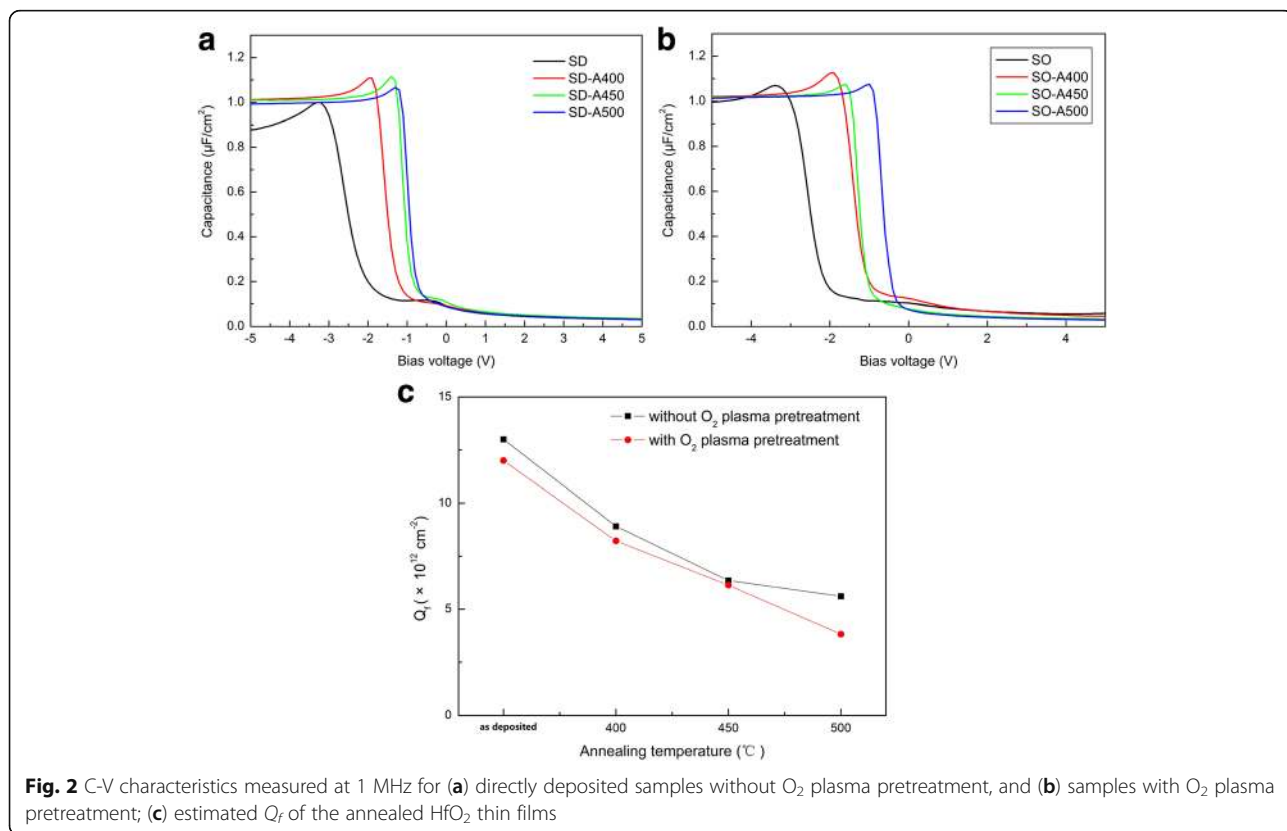
C-V measurements are commonly used to characterize the quality of dielectric layers and their interface with the substrates. C-V measurements were performed herein at room temperature in the dark conditions at 1 MHz on a standard MIS (Al/HfO<sub>2</sub>/p-Si) structure. Figure 2a, b shows the C-V curves of the HfO<sub>2</sub> thin films without and with O<sub>2</sub> plasma pretreatment, respectively. The voltage ( $V_A$ ) that was applied across the MIS device was varied ( $-5 \text{ V} < V_A < 5 \text{ V}$ ) with a sweep step length of 100 mV and signal amplitude of 50 mA, shifting from accumulation to inversion. The shift of C-V curves toward negative voltages demonstrates the presence of effective oxide charges of positive polarity in the as-deposited HfO<sub>2</sub> thin films. The effective oxide charge represents the sum of mobile ionic charges ( $Q_m$ ), oxide trapped charges ( $Q_{OT}$ ) and oxide fixed charges ( $Q_f$ ).  $Q_f$  significantly affects the flat band voltage ( $V_{FB}$ ), as it is located at the oxide-semiconductor interface. In Fig. 2a, the C-V curves are shifted in the positive direction by the  $V_{FB}$  shift because  $Q_f$  decreases as the annealing temperature increases. The slope of the C-V curve

increases with the annealing temperature increases, indicating that the interface trap density decreases as the annealing temperature increases. The HfO<sub>2</sub> thin films with O<sub>2</sub> plasma pretreatment exhibited a similar relationship, as shown in Fig. 2b. The presence of fixed charges arose from the charged oxygen vacancies in the films [13]. The fixed charge density is estimated using Eq. (2), assuming a negligible effect of the interface traps [14],

$$V_{FB} = \phi_{ms} - \frac{qQ_f}{C_{ox}}, \tag{2}$$

where  $\phi_{ms}$  ( $=0.32 \text{ eV}$ ),  $q$  ( $=1.602 \times 10^{-19} \text{ C}$ ),  $C_{ox}$ , and  $V_{FB}$  are the difference between the work functions of metal and the semiconductor, the electronic charge, the capacitance of the dielectric per unit area and the flat band voltage, respectively.

The values of  $Q_f$  for the as-deposited and annealed HfO<sub>2</sub> thin films are shown in Fig. 2c.  $Q_f$  decreases as the annealing temperature increases. The annealing process appears to reduce the density of oxygen vacancies that are responsible for the presence of positive fixed charges, which may be related to the reconstruction of the oxide film near the interface [15]. Furthermore, the  $Q_f$  of SO samples are lower than that of SD samples at the same annealing temperature. The interfacial defect density ( $D_{it}$ ) is determined using an approximation method



**Fig. 2** C-V characteristics measured at 1 MHz for (a) directly deposited samples without O<sub>2</sub> plasma pretreatment, and (b) samples with O<sub>2</sub> plasma pretreatment; (c) estimated  $Q_f$  of the annealed HfO<sub>2</sub> thin films

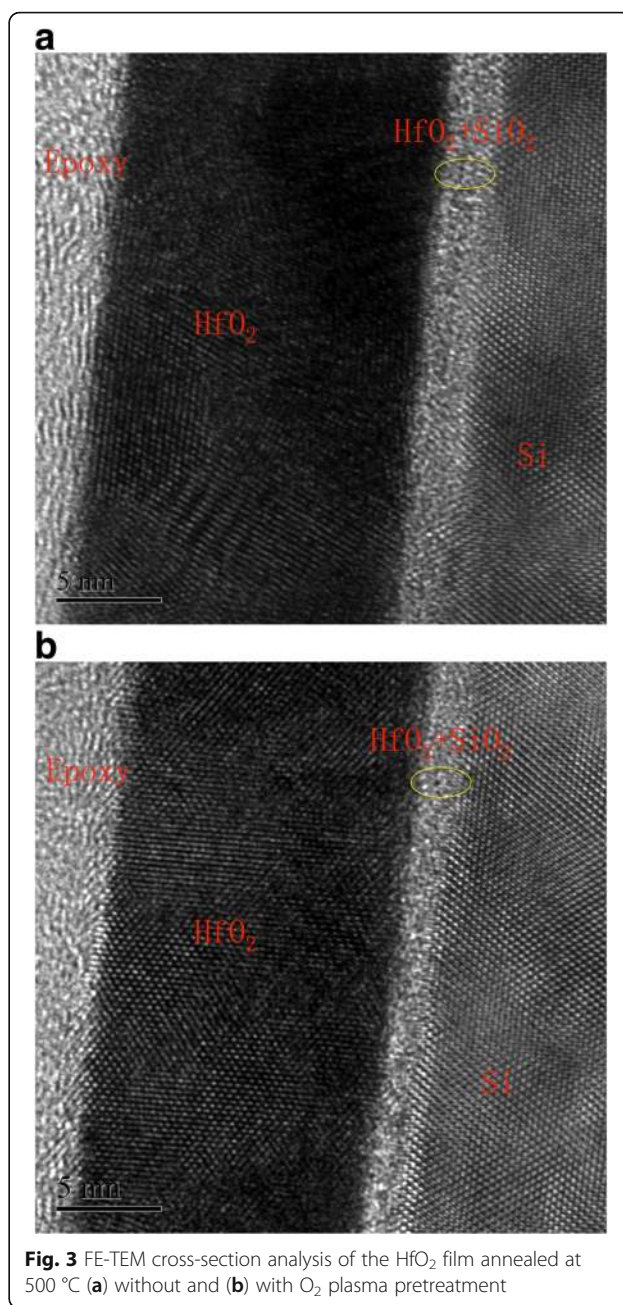
given by W. A. Hill and C. C. Coleman [16]. The  $Q_f$  and  $D_{it}$  values are listed in Table 2.

Cross-sections of the annealed thin films were evaluated by a FE-TEM for assessing the film microstructure and HfO<sub>2</sub>/Si interface. The FE-TEM cross-section analysis of the HfO<sub>2</sub> thin film annealed at 500 °C (a) without and (b) with O<sub>2</sub> plasma pretreatment is shown in Fig. 3. From the FE-TEM images, the annealed HfO<sub>2</sub> thin films consist of three regions, which are the HfO<sub>2</sub> layer, an interfacial oxide and the Si substrate. The atoms in the HfO<sub>2</sub> layer are orderly arranged in some areas, indicating that the HfO<sub>2</sub> layer is microcrystalline structure. A very thin interfacial oxide layer is formed between the high *k* film and the substrate in the as deposited and annealed samples [17]. The HfO<sub>2</sub> layer and the interfacial layer of the sample with oxygen plasma treatment are 15.3 and 2.7 nm, respectively. Whereas, the HfO<sub>2</sub> layer and the interfacial layer of the sample without the pretreatment are 13.9 and 2.2 nm, respectively. This thickness difference should not cause the significant lifetime variation (35 and 67 μs for the samples without and with the pretreatment). Therefore, the significant lifetime improvement could be attributed to the different interface layers with the oxygen plasma pretreatment.

Figure 4 shows the elemental depth profiles of the HfO<sub>2</sub> films annealed at 500°C without and with O<sub>2</sub> plasma pretreatment obtained by XPS. Three regions are observed. In Region A, when the etching time was below 100 s, the relatively uniform atomic percentages of Hf and O corresponded to the RP-ALD μc-HfO<sub>2</sub> layer. In Region B, the O and Hf atomic percentages decreased as the etching time increased from 130 to 175 s, indicating that the O elements diffused into the c-Si substrate, forming an interfacial layer [18, 19]. In Region C, when the etching time increased above 175 s, the Si signal drastically increased up to more than 60%, corresponding to the surface of the c-Si substrate. The oxygen atomic percentage and Hf atomic percentage in the c-Si substrates are due to the Ar ion sputtering effect. During the sputter process of the XPS measurement, some of the Hf or O atoms may reside on the silicon substrate surface and then be detected. Notice that in Region B, in addition to lower Hf and O with a corresponding increase in Si signal in the interface region,

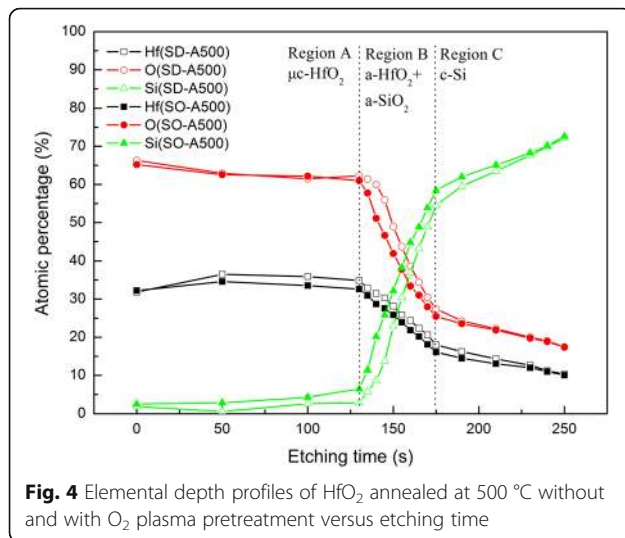
**Table 2** Calculated fixed charge density ( $Q_f$ ) and interface defect density ( $D_{it}$ ) from C-V measurement of the HfO<sub>2</sub> thin films

Sample	Fixed charge density, $Q_f$ ( $\times 10^{12}$ cm <sup>-2</sup> )	Interface defect density, $D_{it}$ ( $\times 10^{13}$ eV <sup>-1</sup> cm <sup>-2</sup> )
SO	12.0	5.21
SO-A400	8.22	4.73
SO-A450	6.13	4.42
SO-A500	3.82	3.82



**Fig. 3** FE-TEM cross-section analysis of the HfO<sub>2</sub> film annealed at 500 °C (a) without and (b) with O<sub>2</sub> plasma pretreatment

the sample with the oxygen pretreatment has also a larger Si signal in the bulk of the HfO<sub>2</sub> film that may account for the percentage differences. The similar results can be obtained at the other investigated annealing temperatures. A possible reason might be that the O<sub>2</sub> pretreatment leads to the growth of a very thin SiO<sub>2</sub> layer reducing the Hf and O diffusion coming from the subsequently deposited HfO<sub>2</sub>. Fewer atomic vacancies are formed by diffusion in the HfO<sub>2</sub> on the sample with the O<sub>2</sub> pretreatment. Thus, the O<sub>2</sub> pretreatment can be expected to yield fewer interface traps and exhibited higher chemical passivation quality.



**Fig. 4** Elemental depth profiles of HfO<sub>2</sub> annealed at 500 °C without and with O<sub>2</sub> plasma pretreatment versus etching time

Growth of a thin oxide film on a clean but unpassivated Si surface leads to the formation of new covalent bonds (chemical passivation) and termination of the dangling bonds [9]. Si/oxide interfaces often carry some fixed charges. These charges can induce an electric field at the surface of Si and can potentially reduce the recombination rate at the Si/oxide interface (field effect passivation). It has been reported by Hoex et al [20] that when preparing Al<sub>2</sub>O<sub>3</sub> thin films by plasma ALD, they found a very thin (~1.5 nm) SiO<sub>x</sub> interfacial oxide layer was formed, which provides good passivation to c-Si surface. They attributed this to the exposure of the substrate to the oxygen plasma in the very first ALD cycles. Although in this study the HfO<sub>2</sub> thin films are prepared, the oxygen plasma pretreatment is found to result in a similar interfacial oxide layer (a-HfO<sub>2</sub> + a-SiO<sub>2</sub>). The oxygen plasma pretreatment could improve the surface passivation of Si wafers.

## Conclusions

In this work, HfO<sub>2</sub> thin films with a thickness of 15 nm were deposited on p-type crystalline silicon wafers using a remote plasma atomic layer deposition system. In situ remote O<sub>2</sub> plasma pretreatment of the Si substrate before the deposition of HfO<sub>2</sub> thin films and post-annealing at 500 °C for 10 min effectively reduced the trap density at the HfO<sub>2</sub>/Si interface, yielding a highest lifetime of 67 μs. The HfO<sub>2</sub> thin films deposited by RP-ALD with O<sub>2</sub> plasma pretreatment have potential as passivation layers in high-quality Si solar cells.

## Abbreviation

ALD: Atomic layer deposition; C<sub>ox</sub>: The capacitance of the dielectric per unit area; c-Si: Crystalline silicon; C-V: Capacitance-voltage; Cz: Czochralski; D<sub>i</sub>: Interfacial defect density; FE-TEM: Field-emission transmission electron microscope; HF: Hydrofluoric acid; HfO<sub>2</sub>: Hafnium oxide; MIS: Metal-insulator-semiconductor; q: The electronic charge; Q<sub>f</sub>: Oxide fixed charges; Q<sub>m</sub>: Mobile ionic charges; Q<sub>OT</sub>: Oxide trapped charges; QSSPC: Quasi steady-state photo

conductance; RCA: Radio Corporation of America; RP-ALD: Remote plasma atomic layer deposition; RTA: Rapid thermal annealing; SD: Direct depositing samples; SiO<sub>2</sub>: Silicon dioxide; SO: O<sub>2</sub> plasma pretreatment samples; SRV: Surface recombination velocity; TEMA: Tetrakis (ethylmethylamino) hafnium; V<sub>f</sub>: Voltage; V<sub>FB</sub>: Flat band voltage; XPS: X-ray photoelectron spectroscopy; τ<sub>eff</sub>: Lifetimes; φ<sub>m</sub>: The difference between the work functions of metal and the semiconductor

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## Authors' contributions

XYZ carried out the characterization of the HfO<sub>2</sub> thin films deposited by ALD and drafted the manuscript. CHH and SYL led the experimental and analytical effort on the passivation of the HfO<sub>2</sub> on Silicon. SYC and WH contributed to deposit the HfO<sub>2</sub> thin films. CHY and CYK assisted in design and analysis of the experiments for the HfO<sub>2</sub> thin films. WZZ, FBX, and XGM contributed to the valuable discussion on experimental and theoretical results, respectively. All authors read and approved the final manuscript.

## Competing interests

The authors declare that they have no competing interests.

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