

4th International Conference on Silicon Photovoltaics, SiliconPV 2014

Simple cleaning and conditioning of silicon surfaces with UV/ozone sources

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Abstract

Within this paper the applicability of three methods to clean and condition the surface of silicon wafers by an ultra-thin oxide layer were tested: two UV/O₃ sources (an Hg vapor lamp and a high efficiency excimer module) as well as a wet chemical oxidation in HNO₃.

It has been demonstrated that organic residues resulting from wafering and masking the surface during alkaline texturing can be removed by UV/O₃ (Hg vapor lamp) exposure. Moreover the use of UV/O₃ (excimer) and HNO₃ oxides result in an improvement of passivation quality and emitter saturation current in combination with Al₂O₃/SiN_x or AlN/SiN_x passivation stacks. For n-type silicon wafers with a 70 Ω/sq. boron emitter and Al₂O₃/SiN_x passivation very low j_{oe} values of 49 fA/cm² are achieved by conditioning with UV/O₃ or HNO₃.

Another important application of ultra-thin SiO_x layers is as tunnel oxides for the rear side of the TOPCon (Tunnel Oxide Passivated Contact) cell concept [1,2]. High iV_{OC} values exceeding 720 mV on planar samples and 710 mV on textured samples are obtained with UV/O₃ grown tunnel oxides after annealing. The structural properties of the SiO_x layers that were analyzed by XPS and compared to the iV_{OC} data, indicate that a minimum oxide layer thickness of approx. 1.4 nm and a high amount of oxygen rich sub oxides species (Si₂O₃, close to the structure of stoichiometric SiO₂) are required to obtain a good interface passivation stable up to annealing temperatures of 900°C.

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Peer-review under responsibility of the scientific committee of the SiliconPV 2014 conference

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Keywords: UV/ozone, surface cleaning, pre-passivation conditioning, tunnel oxide

1. Introduction

With introduction of high efficiency silicon solar cell concepts into industrial production the cleaning and conditioning of the surface prior to passivation gains increasing importance. The quality of the interface between silicon substrate and passivation layer plays a significant role and is very sensitive to various contaminations, defects, surface termination and charging.

As cost optimized processes are a major goal for the PV industry, simple, inline capable cleaning is desirable to substitute cost and time intensive wet-chemical cleaning approaches like RCA clean [3] derived from microelectronics applications. An alternative to wet-chemical processes for the removal of organic residues is given by UV/ozone based light sources which are already used for the cleaning of glass or plastic components [4] and offer a simple integration, low amount of process consumables and are further known for the generation of high-quality ultra-thin silicon oxide layers [5].

To ensure equal starting conditions without organic residues as glue or handling traces for all wafer material used for cell fabrication cleaning prior to alkaline texturing is required. For this process low cost SC1 (standard clean 1 mixture within which NH_4OH is replaced by KOH) approaches are utilized so far. In this paper the removal efficiency concerning organic residues by an Hg vapor lamp (UV/O_3) is tested and compared to that of SC1 cleaning.

Focusing on the development of new passivation layer concepts for (n-type) silicon solar cells pre-passivation conditioning with ultra-thin SiO_x layers can play a key role. Ultra-thin SiO_x layers can thereby be used to improve the interface defect density or to change the surface charging between the silicon substrate and the passivation layer.

In addition, they are also applied as passivating tunnel oxide layers for the high efficiency TOPCon [1] cell concept that enables high iV_{OC} and FF values due to an optimized full-area passivated rear side contact.

In the further course of this paper UV/O_3 generated and wet chemically in HNO_3 [6,7] grown ultra-thin oxides will be tested and compared for their suitability to improve the interface passivation of (highly doped) silicon surfaces passivated with $\text{Al}_2\text{O}_3/\text{SiN}_x$ (both PECVD) or AlN/SiN_x (Sputter/PECVD) [8] stacks and tunnel oxides for the TOPCon approach. Furthermore the results of the oxide properties (layer thickness, stoichiometry) analysis by spectral ellipsometry (SE) and X-ray photoelectron spectroscopy (XPS) are presented.

2. Experimental procedures and results

2.1. Set-up

For the occurring photochemical reaction with oxygen the wavelengths 185 and 254 nm of the UV light are important and can be produced by low pressure Hg vapor lamps (Fig. 1). UV light at a wavelength of 185 nm is absorbed by molecular oxygen (O_2) whereby ozone (O_3) is generated. The UV light at 254 nm on the other hand is absorbed by both hydrocarbon compounds and O_3 whereby hydrocarbon contaminations are cleaved and O_3 dissociates again in O_2 and oxygen radicals. Thus a dynamic equilibrium arises. An alternative to the previous described Hg vapor lamps are excimer systems which emit monochromatic UV radiation. In analogy to the Hg-vapor lamp the dissociation of molecular oxygen is induced and ozone is generated. The narrow spectral line and the monochromatic spectra allow increasing the focus on photo processes and targeted application of UV radiation.

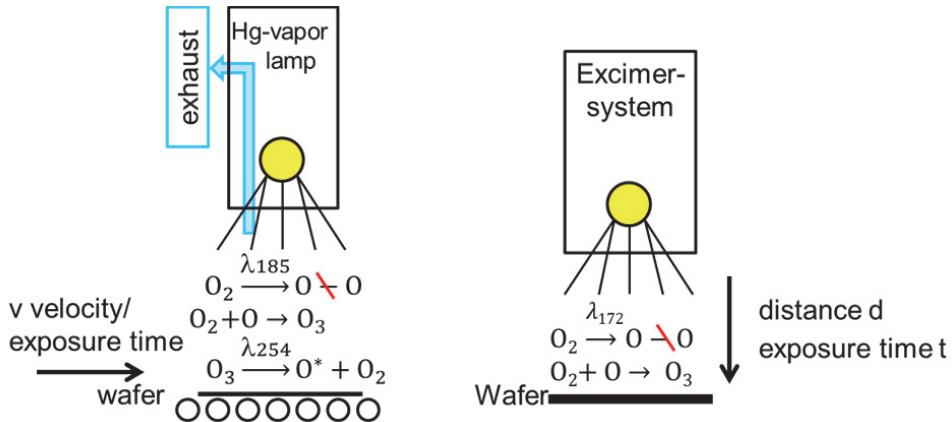


Fig. 1. Schematic picture of Hg vapor lamps and reaction mechanism (left), schematic picture of excimer system and reaction mechanism (right)

2.2. Removal of organic residues

Alkaline texturing or saw damage removal is one of the first steps of monocrystalline solar cell fabrication after wafering. The initiation of the pyramids is highly sensitive to process conditions and organic residues as glue or handling traces which can mask the wafer surface [9]. To test the removal of organic residues by the Hg vapor lamp UV/O₃ source ($\lambda = 185$ and 254 nm) Cz-Si as cut wafers are first contaminated with glue used during the wafering process dissolved in acetic acid (which is pipetted on the wafer surface and dried in N₂-atmosphere) and fingerprints simulating handling traces (Fig. 2). Then the samples are exposed to the Hg vapor lamp whereby the parameters exposure time and intensity of the exhaust system (0% or 75%) are varied. An intensity of 0% results in a higher and an intensity of 75% in a lower O₃ concentration. The distance from the UV/O₃ source was kept constant at 0.5 cm. After UV/O₃ cleaning the samples are alkaline textured and scanned with a flatbed scanner to identify the masked surface areas where contaminations are not completely removed. For a long exposure time and high O₃ concentration (low intensity of exhaust system) the fingerprint is completely and the glue residues mostly removed (Fig. 2, F).

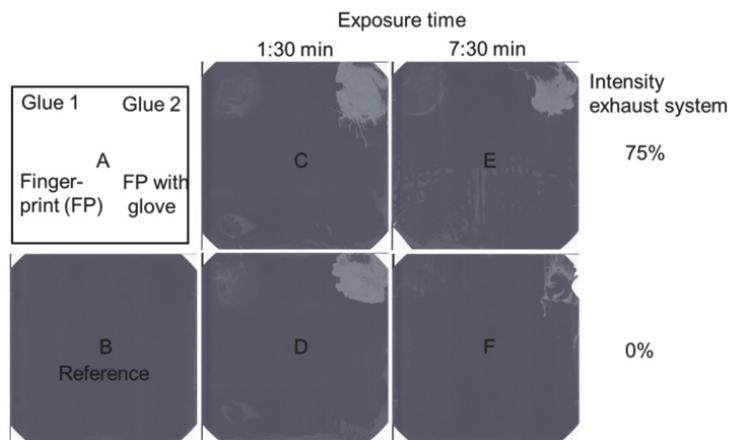


Fig. 2. Schematic picture of a contaminated sample (A), flatbed scans of cleaned textured reference sample without contamination (B) and contaminated and UV/O₃ cleaned samples with parameter variation (C-F).

2.3. Pre-passivation Conditioning

Shiny etched and textured n-type 1 Ωcm FZ-Si samples with 70 Ohm/sq. boron emitter are RCA [3] cleaned with an HF-dip (1%HF in DI water, 1min) as last process step. One part of the samples is immediately passivated with $\text{Al}_2\text{O}_3/\text{SiN}_x$ (both PECVD) or AlN/SiN_x (Sputter/PECVD) [8] stacks (Fig. 3). The other part of the samples are oxidized either wet chemically in HNO_3 or by exposure to an UV/O_3 source (exposure time 1 min, excimer module $\lambda = 172$ nm) prior passivation (Fig 3). The emitter saturation currents (j_{0e}) are calculated from the minority carrier lifetime data measured by QSSPC. The lowest j_{0e} values of 49 fA/cm^2 can be achieved by conditioning with ultra-thin UV/O_3 or HNO_3 grown oxide and the $\text{Al}_2\text{O}_3/\text{SiN}_x$ passivation stack. The obtained mean value for the Al_2O_3 based passivation stack without conditioning is 63 fA/cm^2 . Moreover mean values of 75 fA/cm^2 are reached by conditioning with the AlN/SiN_x stack. Without conditioning a mean value of 146 fA/cm^2 is achieved for the AlN based stack. This shows that the conditioning with ultra-thin oxides can significantly increase the passivation quality of (highly doped) silicon surfaces. The improvement in passivation is expected to result from a lower interface defect density due to the interfacial oxide layer [10].

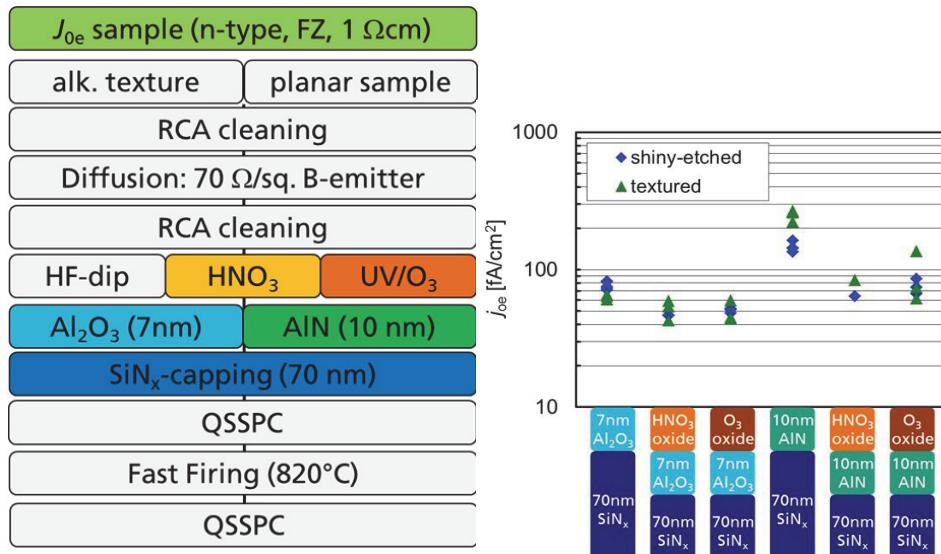


Fig. 3 Schematic picture of j_{0e} sample preparation (left); Emitter saturation current density (j_{0e}) for samples with/without ultra-thin SiO_x -layer as pre-passivation conditioning (right)

2.4. Ultra-thin tunnel oxide layers for the TOPCon cell concept

Within the TOPCon (Tunnel Oxide Passivated Contact) cell concept [1,2] the rear side of the solar cell consists of an ultra-thin tunnel oxide and a phosphorus-doped silicon layer, whereas the front side exhibits a diffused boron-doped emitter ($140 \Omega/\text{sq.}$) which is passivated by atomic layer deposited (ALD) aluminum oxide Al_2O_3 and a plasma enhanced chemical vapor deposited (PECVD) silicon nitride SiN_x layer (Fig. 3). It has been shown that the TOPCon approach enables a highly efficient surface passivation and an excellent performance regarding V_{oc} and FF. Thereby the tunnel oxide plays a major role due to its excellent interface properties that secures the excellent contact passivation. In the first approach the tunnel oxide is grown wet chemically in HNO_3 [6,7], a proven method from

microelectronics applications which is used to improve the Si/SiO₂ interface characteristics. An excellent interface passivation and high implied V_{OC} values > 720 mV have been reached for annealing temperatures (T_{anneal}) up to 900°C for planar samples. When considering the textured samples a strong decrease in the interface passivation is observed for T_{anneal}=900°C. This effect can be attributed to the local disruption of the tunnel oxide in oxygen-free ambient and indicates that a non-defective tunnel oxide layer is required to obtain a high passivation quality. For T_{anneal} > 900°C the contact resistance is reduced similar to that of a poly-silicon contact [11] and can be advantageous for further optimizations of the TOPCon approach. It is therefore important to understand which parameters influence the properties of the tunnel oxide and test potential alternatives as UV/ozone grown oxide layers for the investigated application.

As described by Fink et al. [5] ozone-grown oxides are found to exhibit improved interface (less strain, transition layer is reduced [12]) and electrical characteristics over dioxygen-grown oxides due to the occurring damage-free oxidation of the silicon surface. In addition, the more reactive oxygen radical species are supposed to be more efficient in saturating silicon dangling bonds resulting in a lower defect density. Further the oxidation of silicon with O₃ by an UV/ozone generator is a fast and simple applicable process.

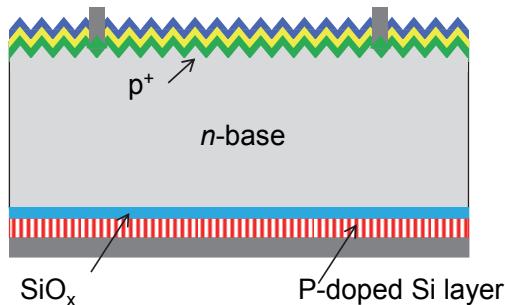


Fig. 4. Schematic cross section of the TOPCon cell structure [1].

2.4.1. Interface passivation quality

The interface passivation quality is determined on symmetrical lifetime samples by QSSPC. As a measure for interface passivation the implied open-circuit voltage iV_{OC} at 1 sun is calculated.

$$iV_{OC} = \frac{kT}{q} \ln \left(\frac{\Delta n(\Delta n + N_D)}{n_i^2} \right) \quad (1)$$

For the symmetrical lifetime samples n-type 1 Ωcm (100)-oriented FZ silicon wafers with a thickness of 200 μm with shiny-etched and alkaline textured (111) surfaces have been cleaned by a complete RCA cleaning sequence finishing with a HF-dip. Directly after cleaning an ultra-thin oxide layer of a thickness of approx. 1.4 nm (determined by spectroscopic ellipsometry) is grown either wet chemically in HNO₃ or by exposure to an UV/O₃ source (excimer module, λ = 172 nm) whereby the exposure time and the distance of the sample to the irradiation source have been varied. The investigated testing conditions of the first and second experiment are given in Table 1 and 2. Subsequently, a 20 nm thick phosphorus-doped Si layer is deposited on both sides and the passivation is activated by thermal treatment (furnace anneal and hydrogen passivation RPHP) [13] at either 800°C or 900°C.

Table 1. Testing conditions of first experiment.

Oxidation	Distance d (cm)	Exposure time t (min)
1 UV/O ₃	0.2	1
2 UV/O ₃	0.5	1
3 UV/O ₃	0.5	3
4 UV/O ₃	1.5	1
5 HNO ₃ Ref.	-	-

Table 2. Testing conditions of second experiment

oxidation	Distance d (cm)	Exposure time t (min)
1 UV/O ₃	0.5	1
2 UV/O ₃	0.5	3
3 UV/O ₃	0.5	5
4 UV/O ₃	1.5	3

High iV_{OC} values exceeding 710 mV are obtained for all planar samples independent of the oxidation process applied to grow the tunnel oxide layer for $T_{anneal}=800^{\circ}\text{C}$. Further for the UV/O₃ oxides the distance between the sample and the irradiation source has only a minor impact on the iV_{OC} . Concerning the textured samples at the same T_{anneal} the resulting iV_{OC} values of HNO₃ (Ref.) and UV/O₃ oxidized samples for all oxidation conditions except for a distance of 0.2 cm are comparable, whereby the values of the UV/O₃ oxidized samples are slightly higher (720 mV). For $T_{anneal}=900^{\circ}\text{C}$, which enables a low contact resistivity, iV_{OC} values exceeding 705 mV are reached for planar samples for both oxidation processes. Further maximum iV_{OC} values above 725 mV for the UV/O₃ oxidized samples are obtained and a significant dependency of the exposure time on the iV_{OC} has been found. If the exposure time is extended to 3 min the iV_{OC} values of these samples increase. The found dependency can be attributed to an insufficient oxide thickness that can result in a disruption of the oxide layer during the thermal treatment. For the textured samples the same interrelations result for the UV/O₃ oxidized samples, but with the addition that they are more pronounced. Maximum iV_{OC} values of 715 mV are reached. For the HNO₃ (Ref.) interface tunnel oxide the resulting iV_{OC} value is only 630 mV what indicates that a difference regarding the oxide stoichiometry might be present. (Fig. 5)

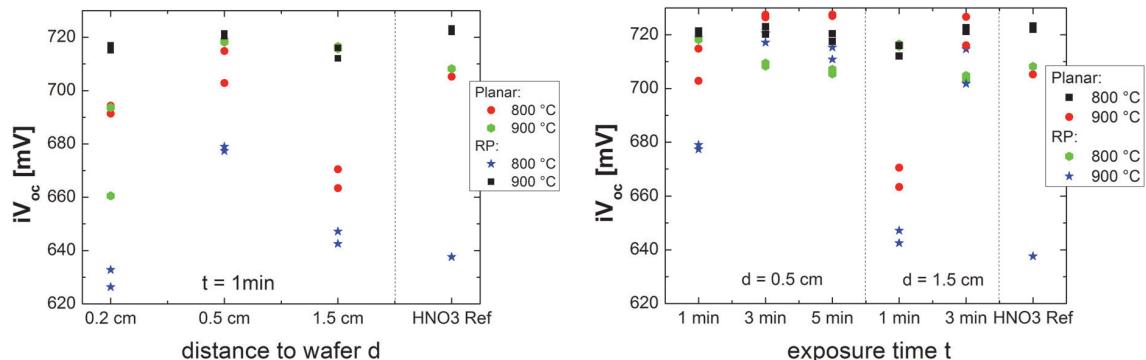


Fig. 5) iV_{OC} data of symmetrical lifetime samples of the TOPCon n-contact (a) variation of distance (d) between sample and irradiation source for 1 minute exposure time (t); (b) exposure time variation for a distance of 0.5 cm and 1.5 cm.

2.4.2. Analysis of the ultra-thin oxide layers

To get a better understanding of the requirements to ensure a good and temperature stable interface passivation a detailed analysis of the oxide properties (layer thickness, stoichiometry) is conducted by spectral ellipsometry (SE) and X-ray photoelectron spectroscopy (XPS). The layer thickness of the UV/O₃ oxides is determined by SE (measurement angle 70°) for the distances of 0.5 and 1.5 cm from the sample to the irradiation source and the exposure times 0 (reference), 30, 45, 60, 90, 120, 150 and 180 s. Prior to oxidation the native oxide layer is removed

by an HF-dip (1 wt % HF, 1 min). The measurement data are evaluated with the aid of a model for 1 mm Si and approx. 1.5 nm native oxide (Fig. 6). The obtained oxide layer thickness after the HF-dip is 1.0-1.1 nm, after 30 s exposure time 1.6 nm and 1.8-1.9 nm after 180 s. The thickness of the HNO_3 oxide amounts approx. 1.5 nm. The oxide layer thickness can vary depending on the model that is used for the evaluation and is therefore not always very precise as for example for the oxide layer thickness after the HF-Dip. Here the model does not fit and the thickness is over estimated.

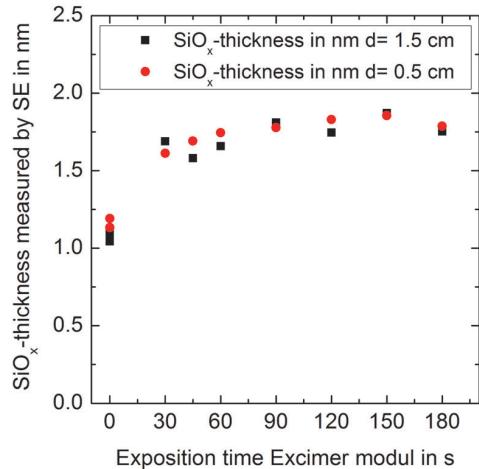


Fig. 6. UV/ O_3 oxide layer thickness determined by SE (measurement angle 70°).

A detailed analysis of the oxide stoichiometry (suboxide species of Si^+ , Si^{2+} , Si^{3+}) and the layer thickness is feasible by XPS. The analysis was carried out with a *Kratos Axis Ultra DLD* XPS instrument at an emission angle of 0°, a step size of 0.05 eV and a pass energy of 10 eV. For the measurements a monochromatized (Rowland circle monochromator) Al K_α source with an excitation energy of $h\nu=1486.6$ eV was used. The dwell time of the measurements was 75 s. The spectra obtained during the XPS analysis were not post-processed.

To obtain information about the stoichiometry of silicon oxides only the range of the binding energy between 95 and 105 eV has to be considered where the 2 peaks originating from the electrons of the 2p orbitals are found. The higher peak at approx. 99 eV corresponds to the silicon substrate (Si^{0+}), the smaller peak at approx. 103 eV to SiO_2 (Si^{4+}). Nevertheless, when studied more closely both peaks consist of several individual peaks. The silicon substrate peak consists of the 2 $p_{3/2}$ and 2 $p_{1/2}$ peak due to the spin-orbit coupling which is shifted 0.6 eV to higher binding energies. The peaks of SiO_2 and its sub-stoichiometric species Si_2O (Si^{+}), SiO (Si^{2+}), Si_2O_3 (Si^{3+}) are each shifted 3.3-4.8, 0.95, 1.75 and 2.48 eV from the Si 2 $p_{3/2}$ peak [14,15]. A survey spectrum of a Si/SiO_2 sample is depicted in Fig. 7.

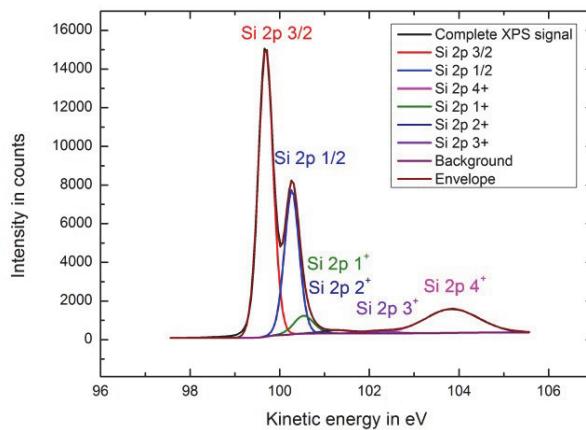


Fig. 7. XPS survey spectrum of a Si/SiO₂ sample with all silicon and SiO₂ peaks.

The oxide layer thickness is calculated according to the formula suggested by Seah and Spencer [14,15] whereby L_{SiO_2} corresponds to the attenuation length for the Si 2p photoelectrons in the SiO₂ overlayer, θ is the angle of emission of the photoelectrons from the surface normal, R_{expt} is the measured ratio of the oxide and substrate photoelectrons and R_0 is the ratio of electrons for these two states of the bulk samples. For the calculation of the oxide thickness, $L_{\text{SiO}_2} = 3.4 \text{ nm}$ and $R_0 = 0.9$ were assumed according to literature [14,15].

$$d_{\text{oxide}} = L_{\text{SiO}_2} \cos \theta \ln \left(1 + \frac{R_{\text{expt}}}{R_0} \right) \quad (2)$$

For the samples p-type 1 Ωcm (100)-oriented FZ silicon wafers of 200 μm thickness with high reflective surface have been cleaned by RCA with HF-dip last. Directly after cleaning the samples are oxidized either wet chemically in HNO₃ or by exposure to an UV/O₃ source (excimer module, $\lambda = 172 \text{ nm}$) whereby the exposure time (1 and 3 min) and the distance (0.2, 0.5 and 1.5 cm) of the sample to the irradiation source were varied. After oxidation the samples were stored under N₂-atmosphere for approx. 11 hours before XPS analysis.

In Table 3 the percentage of the single silicon peaks and the calculated oxide layer thickness is presented for all analyzed samples. The data evaluation was carried out with the software CasaXPS.

Table 3. Percentage of the single silicon peaks and calculated oxide layer thickness.

oxide	Si 2p 3/2	Si 2p 1/2	Si 4 ⁺	Si 1 ⁺	Si 2 ⁺	Si 3 ⁺	(Si1+Si2+Si3)+	Si4+/rest	Oxide thickness [nm]
HNO ₃ Ref.	52.69	26.34	14.65	4.90	0.88	0.54	6.32	0.17	0.59
UV/O ₃ (0.5 cm, 1 min)	54.02	27.01	12.49	4.69	1.24	0.55	6.48	0.14	0.50
UV/O ₃ (0.5 cm, 3 min)	52.01	26.00	15.75	4.40	1.16	0.68	6.24	0.19	0.64
UV/O ₃ (1.5 cm, 1 min)	54.85	27.42	11.66	4.45	1.16	0.47	6.08	0.13	0.47
UV/O ₃ (1.5 cm, 3 min)	52.69	26.34	14.83	4.44	1.04	0.65	6.13	0.17	0.60
UV/O ₃ (0.2 cm, 1 min)	52.70	26.34	14.79	4.44	1.12	0.61	6.17	0.17	0.60

The comparison of the spectra of the HNO_3 and the UV/O_3 oxides indicates that the Si^{1+} peak is more pronounced (Fig. 8), whereas the Si^{2+} and Si^{3+} peaks are less pronounced for the HNO_3 oxide (Fig. 9). The Si^{4+} peak intensity is in correlation with the layer thickness for all oxides.

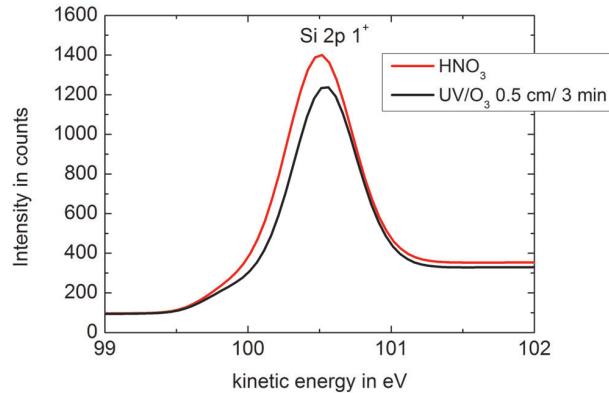


Fig. 8. Comparison of Si^{1+} peak intensity for HNO_3 oxidized and for UV/O_3 oxidized sample ($d=0.5$ cm, $t=3$ min).

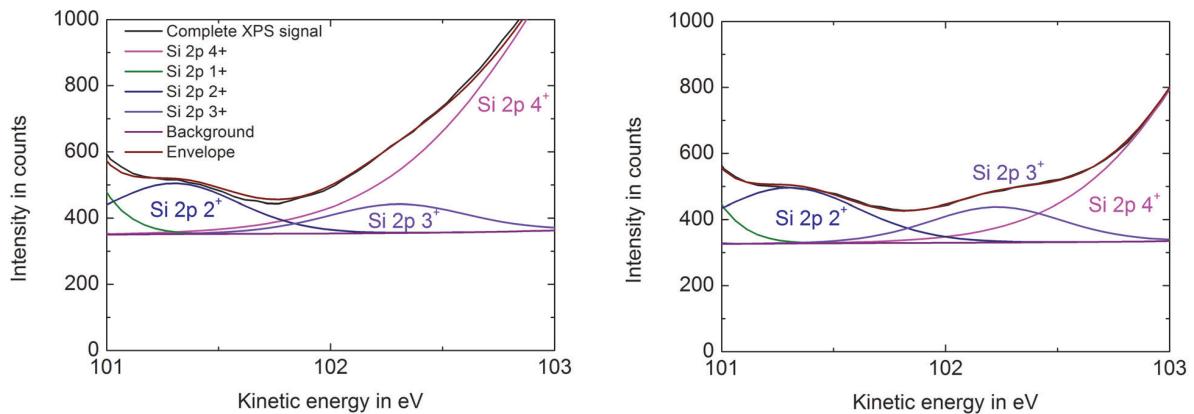


Fig. 9. Si^{2+} and Si^{3+} peaks of HNO_3 oxidized sample (left); Si^{2+} and Si^{3+} peaks of UV/O_3 oxidized sample ($d=0.5$ cm, $t=3$ min) (right).

The obtained oxide layer thickness after the HF-dip is 0.2 nm. The thickness of the HNO_3 oxide amounts to approx. 0.59 nm. The oxide growth velocity of the UV/O_3 oxides depends on both the distance to the irradiation source and the exposure time. The resulting oxide layer thickness is higher, the smaller the distance between sample and irradiation source and the longer the exposure time. The discrepancy to the oxide thickness determined by spectral ellipsometry is a known phenomenon described in literature [15].

Comparing only the UV/O_3 oxides the following correlation of the peaks of the sub-stoichiometric species are found: Si^{2+} (SiO) peak decreases with exposure times, Si^{3+} (Si_2O_3) and Si^{4+} (SiO_2) peaks increase with the exposure time, the Si^{1+} (Si_2O) peak remains unchanged. The lower the distance between sample and irradiation source the higher the resulting amount of Si^{3+} (Si_2O_3).

The comparison of the iV_{OC} and the XPS data indicates that a certain minimum oxide layer thickness and a low amount of silicon rich sub oxide species (Si_2O)/high amount of oxygen rich sub oxides species (Si_2O_3) that are close to the structure of a stoichiometric SiO_2 are required to obtain a good interface passivation that is stable also for $T_{anneal} = 900^\circ C$.

The lower iV_{OC} data of the samples oxidized close to the irradiation source ($d= 0.2$ cm, $t= 1$ min) are supposed to result from the strong UV irradiation dose close to the surface. To quantify the interface defect density SPV measurements are required.

3. Conclusion

It is demonstrated that organic residues resulting from the wafering and masking the surface during alkaline texturing can be removed by UV/O_3 (Hg vapor lamp) exposure. Particularly for a long exposure time and high O_3 concentration fingerprints are completely and the glue residues mostly removed.

Pre-passivation conditioning is possible with both examined oxidation methods (UV/O_3 and HNO_3) resulting in an improvement of emitter saturation current in combination with both investigated passivation stacks. The improvement of passivation is expected to result from a lower interface defect density due to the interfacial oxide layer.

It has been shown that by UV/O_3 exposure grown ultra-thin SiO_x layers are well suited as tunnel oxides. High iV_{OC} values exceeding 720 mV on planar samples and 710 mV on textured samples are obtained with UV/O_3 grown tunnel oxides for both tested T_{anneal} . A short exposure time of only 3 min is sufficient to achieve a good interface passivation, therefore the here investigated process represents a promising, simple and cost effective application for the generation of tunnel oxides.

XPS is a suited technique for a detailed analysis concerning oxide properties (stoichiometry, oxide layer thickness). Inter-relations between these and the improvement of passivation quality are demonstrated.

The comparison of the iV_{OC} and the XPS data indicates that a certain minimum oxide layer thickness and high amount of oxygen rich suboxides that are close to the structure of a stoichiometric SiO_2 are required to obtain a good interface passivation that is stable also for a $T_{anneal} = 900^\circ C$.

The interface defects that are supposed to lower the iV_{OC} data of the samples oxidized close to the irradiation source ($d= 0.2$ cm, $t= 1$ min) are supposed to result from the strong UV irradiation dose close to the surface. To quantify the interface defect density SPV measurements are required.

In summary $UV/ozone$ based light sources offer a simple applicable cost effective possibility for surface cleaning and conditioning.

4. Outlook

A more efficient removal of contaminants is expected by the use of an excimer module and will be studied in future experiments.

The here investigated tunnel oxides will be further characterized by CV measurements of a MOS diode or a TLM test structure to determine the leakage current and the distance dependent contact resistivity. These investigations might contribute to deeper insight concerning defects at the interface or in the SiO_2 layer which can result by the irradiation with high energetic photons [16]. Further the UV/O_3 based tunnel oxide layers will be tested and compared to the HNO_3 oxides in a TOPCon cell device structure.

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