

Effective passivation of silicon surfaces by ultrathin atomic-layer deposited niobium oxide

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Effective passivation of silicon surfaces by ultrathin atomic-layer deposited niobium oxide

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This letter reports on effective surface passivation of *n*-type crystalline silicon by ultrathin niobium oxide (Nb₂O₅) films prepared by atomic layer deposition (ALD) and subjected to a forming gas anneal at 300 °C. A champion recombination parameter J_0 of 20 fA/cm² and a surface recombination velocity S_{eff} of 4.8 cm/s have been achieved for ultrathin films of 1 nm. The surface pretreatment was found to have a strong impact on the passivation. Good passivation can be achieved on both HF-treated *c*-Si surfaces and *c*-Si surfaces with a wet-chemically grown interfacial silicon oxide layer. On HF-treated surfaces, a minimum film thickness of 3 nm is required to achieve a high level of surface passivation, whereas the use of a wet chemically-grown interfacial oxide enables excellent passivation even for Nb₂O₅ films of only 1 nm. This discrepancy in passivation between both surface types is attributed to differences in the formation and stoichiometry of interfacial silicon oxide, resulting in different levels of *chemical passivation*. On both surface types, the high level of passivation of ALD Nb₂O₅ is aided by *field-effect* passivation originating from a high fixed negative charge density of $1\text{--}2 \times 10^{12} \text{ cm}^{-3}$. Furthermore, it is demonstrated that the passivation level provided by 1 nm of Nb₂O₅ can be further enhanced through light-soaking. Finally, initial explorations show that a low contact resistivity can be obtained using Nb₂O₅-based contacts. Together, these properties make ALD Nb₂O₅ a highly interesting building block for high-efficiency *c*-Si solar cells. *Published by AIP Publishing.* <https://doi.org/10.1063/1.5029346>

Effective passivation of surface defects in crystalline silicon (*c*-Si) solar cells by the deposition of thin films is a prerequisite for achieving high energy conversion efficiencies. Silicon oxide,¹ silicon nitride,² amorphous silicon³ and aluminum oxide⁴ have been the commonly-employed passivation layers. In recent years, these classical passivation materials have been joined by a range of other metal oxide passivation materials, notable examples being titanium oxide,^{5–7} tantalum oxide,⁸ gallium oxide,⁹ hafnium oxide,¹⁰ molybdenum oxide¹¹ and zinc oxide.¹² This expanding library of passivating materials not only allows us to gain a deeper understanding as to which materials are able to form a high-quality interface with *c*-Si with a low defect density, but also enables the use of passivation materials with enhanced or additional functionalities, such as antireflection properties and lateral conductivity. Most notably, this search for novel materials has been sparked by the growing interest in so-called passivating contacts which not only passivate the Si surface, but also selectively extract holes or electrons from Si.¹³ Such passivating contacts allow for full-area passivation of the Si surface and avoid the use of highly recombination-active local metal-Si contacts, thereby enabling high efficiencies using lean full-area processing. Besides the more well-known passivating contacts based on doped amorphous¹⁴ and polycrystalline¹⁵ silicon, there is significant interest in dopant-free metal oxide-based passivating contacts such as aforementioned titanium oxide⁵ and molybdenum oxide.¹⁶

In this letter, we expand on this growing list of passivation materials by demonstrating effective surface passivation

of *c*-Si by niobium oxide (Nb₂O₅), a material so far not known for its ability to passivate *c*-Si. Nb₂O₅ thin films were prepared by atomic layer deposition (ALD) and subjected to post-deposition anneal in forming gas to activate the passivation. It is shown how the use of a wet-chemically grown SiO₂ interlayer can further enhance the passivation level and insights into the underlying passivation mechanisms are presented. The salient features of this passivation material are the strong negative fixed charge and the ability to passivate silicon using ultrathin films of 1 nm without the use of any further capping or hydrogenation layers. Furthermore, it will be shown that the surface passivation can be enhanced further through light-soaking, leading to a champion recombination parameter J_0 of 20 fA/cm². Finally, initial explorations show that a low contact resistivity can be obtained using Nb₂O₅-based contacts. Together, these properties make ALD Nb₂O₅ a highly interesting building block for high-efficiency *c*-Si solar cells.

Nb₂O₅ thin films with a thickness ranging from 1 to 5 nm were prepared by thermal atomic layer deposition (ALD) in an Oxford Instruments FlexAL reactor using (tert-butyli-mido)-tris(diethylamino)-niobium [(CH₃)₃CNNb(N(C₂H₅)₂)₃, TBTDEN] as precursor and H₂O as reactant. This process, based on earlier work in the literature,¹⁷ as well as the material properties will be published elsewhere.¹⁸ The deposition temperature was varied between 150 and 300 °C, over which the growth-per-cycle decreased from 0.40 to 0.25 Å. Mirror-polished 285 μm floatzone *n*-type silicon (100) wafers with a nominal resistivity of 3 Ωcm and a wet-chemical SiO₂ layer

grown by Radio Corporation of America (RCA) cleans 1 and 2 were used as substrates.¹⁹ Spectroscopic ellipsometry (SE) measurements revealed a thickness of ~ 1.3 nm for this wet-chemical oxide. Depositions were performed on either *c*-Si substrates from which the wet-chemical oxide was stripped by dipping in 1% HF for one minute or on *c*-Si substrates with the wet-chemical oxide. The samples were subjected to post-deposition annealing treatments in a forming gas environment (10/90 H₂/N₂) at 300 °C in a Jipelec rapid thermal anneal furnace. X-Ray diffraction measurements revealed that all films in this study were amorphous and remained amorphous during annealing. The passivation quality was evaluated using a Sinton WCT-120TS quasi-steady-state photoconductance (QSS-PC) setup. The Nb₂O₅ film thickness was determined by fitting SE data (Woollam M-2000U) to a Tauc-Lorentz model. No growth delay was observed on either substrate type. Light-soaking was performed at room temperature using a 165 lumen light emitting diode (LED) [Samsung 3432 1.8t (FH341A)] at a distance of ~ 10 cm. The interfacial silicon oxide was studied by X-ray photoelectron spectroscopy (XPS) using a Thermo Scientific KA1066 spectrometer. The induced *c*-Si equilibrium band bending was probed by surface photovoltage (SPV) measurements using 905 nm laser excitation.²⁰

The passivation provided by 5 nm Nb₂O₅ films prepared at deposition temperatures ranging from 150 to 300 °C has been studied. In Fig. 1, the evolution of the minority carrier lifetime of these symmetric samples upon annealing in forming gas at 300 °C is shown for both (a) HF-treated *c*-Si substrates and (b) *c*-Si substrates with an RCA-grown SiO₂ layer. As can be seen, although none of the samples provide passivation in the as-deposited state, a high level of surface passivation can be achieved by annealing the samples. Interestingly, for HF-treated surfaces, the level of attainable surface passivation is clearly higher for films prepared at lower deposition temperatures. Specifically, the highest level

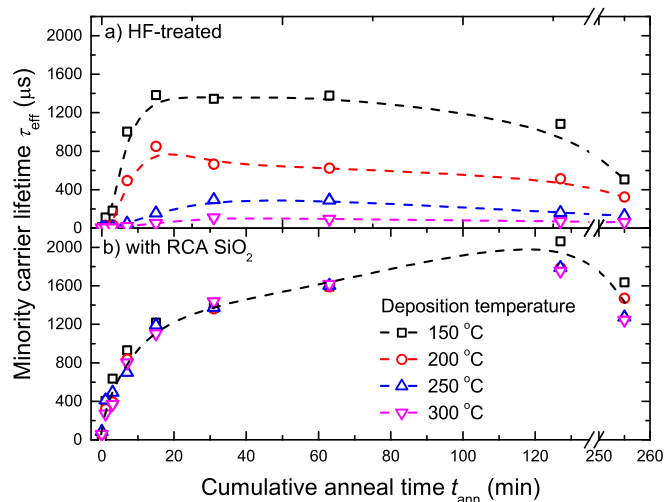


FIG. 1. Minority carrier lifetime at an injection level of 10^{15} cm⁻³ as a function of annealing time for symmetrical lifetime samples with 5 nm Nb₂O₅ films, prepared at various deposition temperatures. *n*-type *c*-Si wafers that were either (a) HF-dipped prior to deposition or (b) had an RCA-grown SiO₂ interface layer were used as substrates. Dashed lines are guide to the eye. In (b), only one guide to the eye is given as the observed trend is very similar for all deposition temperatures.

of passivation is reached after annealing the sample prepared at 150 °C for 15 minutes at 300 °C. Prolonged annealing (>4 hours) leads to depassivation of *c*-Si, which points to a competition between passivation and depassivation upon annealing. Such competition has been observed previously for Ga₂O₃⁹ and a-Si:H.²¹ Interestingly, Fig. 1(b) shows that the use of an RCA-grown SiO₂ interlayer enables improved passivation and renders the kinetics of passivation independent of the deposition temperature over the examined range. The best minority carrier lifetimes are achieved after a longer annealing time of 127 min. The very similar kinetics for all these samples suggest that it is the activation of the passivation of the RCA oxide, most likely by hydrogenation, that is responsible for the increase in lifetime, and that this process is rather independent of the deposition temperature of the Nb₂O₅ layer. This also implies that the variation of the maximum attainable lifetime on HF-treated surfaces with deposition temperature is not due to differences in the material properties of the Nb₂O₅ layer itself, but rather due to a difference in the nature of the interface (oxide) formed during deposition and/or post-deposition annealing.

Besides improving the passivation quality, the use of RCA-grown SiO₂ can also enable good passivation for ultrathin Nb₂O₅ films down to 1 nm. As shown in Fig. 2(a), in the case of HF-treated surfaces, negligible passivation is achieved for ultrathin films of 1–2 nm, whereas the passivation quality increases strongly for thicker films. This is very similar to the case of ALD Al₂O₃, for which it has been shown that the chemical passivation quality strongly decreases for films thinner than 5 nm.²² Surprisingly, in the case of RCA-treated surfaces, the passivation quality is independent of film thickness, in stark contrast to the results of HF-treated surfaces. Nonetheless, the presence of at least some Nb₂O₅ is a prerequisite for obtaining passivation as it was verified that an RCA-treated surface without Nb₂O₅ does not yield any passivation, even after annealing.

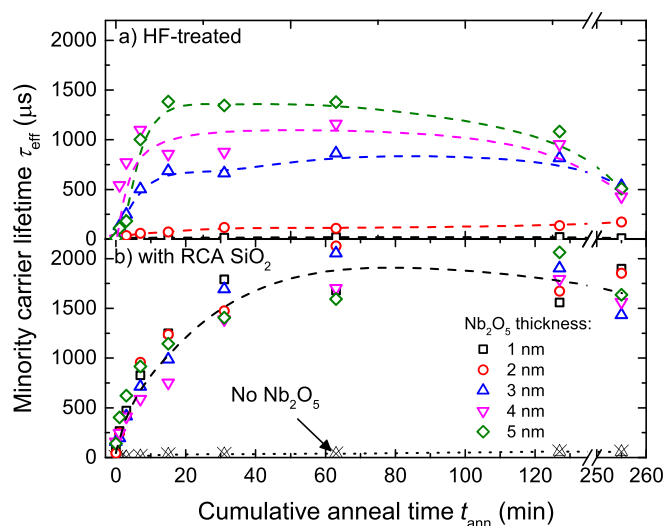


FIG. 2. Minority carrier lifetime at an injection level of 10^{15} cm⁻³ as a function of annealing time for symmetrical lifetime samples with Nb₂O₅ films of various thicknesses prepared at 150 °C. *n*-type *c*-Si wafers that were either (a) HF-dipped prior to deposition or (b) had an SiO₂ interface layer grown by RCA cleans were used as substrates. Dashed lines are guide to the eye. In (b), only one guide to the eye is given as the observed trend is very similar for all film thicknesses.

To understand these differences between both substrate types, the nature of interfacial silicon oxide, and the influence of annealing thereon, has been studied by XPS for 5 nm Nb_2O_5 films. Specifically, the oxidation state of *c*-Si has been examined, shown in Fig. 3. As can be seen, at all deposition temperatures, the *c*-Si surface of HF-treated substrates is slightly oxidized during deposition, with somewhat stronger oxidation occurring at higher deposition temperatures (dashed lines). After the one hour anneal treatment, *c*-Si oxidizes further, as witnessed by the increase in peak intensity, especially for the films prepared at higher temperatures (solid lines). Moreover, the formed silicon oxides appear to be substoichiometric as all peaks lie close to the Si^{3+} state. The XPS spectrum of an RCA-treated surface shows a much stronger oxide signal in the as-deposited state, which appears to be near-stoichiometric SiO_2 , as the peak lies closer to the Si^{4+} state. This shows that the RCA treatment leads to a well-defined oxide, which also does not significantly change upon annealing. Therefore, by forming a well-defined RCA oxide prior to deposition, the resulting passivation is rendered independent of the deposition temperature. Nonetheless, as Fig. 2(b) showed that no passivation is obtained by annealing RCA-treated surfaces without Nb_2O_5 in forming gas, it is thought that the Nb_2O_5 layer, even when it is only 1 nm thick, plays a strong role in the hydrogenation of the interfacial oxide. Although most annealing experiments were performed in forming gas, initial experiments have indicated that passivation can also be achieved by annealing in N_2 . A very similar lifetime value of 346 μs was reached after 1 min of annealing a 5 nm Nb_2O_5 sample prepared at 150 $^\circ\text{C}$ on an RCA-treated surface in N_2 . This suggests that besides the potential forming gas environment, Nb_2O_5 itself can also act as a source of hydrogen. Elastic recoil detection (ERD) measurements have shown that a 20 nm ALD Nb_2O_5 film prepared at 200 $^\circ\text{C}$ contains 3.4 at. % of H in the as-deposited state. This is in the same range as reported for ALD Al_2O_3 , for which the embedded H is known to serve as the source of hydrogen for interface hydrogenation of *c*-Si during N_2 annealing.⁴

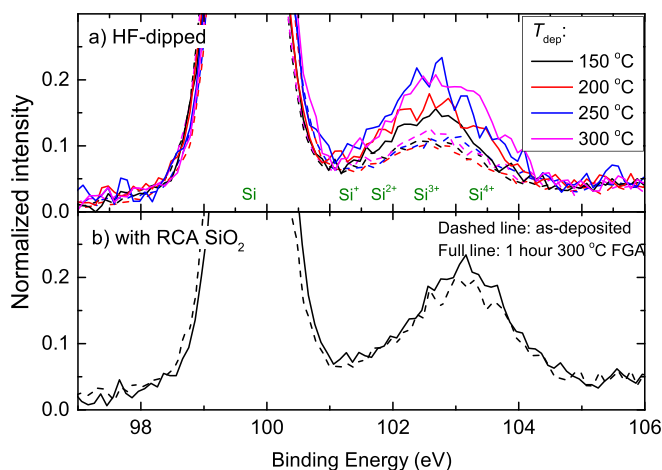


FIG. 3. XPS spectra of the Si $2p$ peaks for 5 nm Nb_2O_5 films prepared on either (a) HF-treated substrates or (b) substrates with an RCA SiO_2 layer. The approximate peak positions for the oxidation states of Si are indicated by green text.²³ Dashed lines denote as-deposited samples, where full lines are for samples after one hour of FGA at 300 $^\circ\text{C}$.

In addition, part of the excellent passivation of ALD Nb_2O_5 is attributed to strong *field-effect* passivation. SPV measurements have revealed a strong hole inversion layer at the surface of the 2.3 Ωcm *n*-type wafers for 5 nm Nb_2O_5 films prepared at 150 $^\circ\text{C}$, after forming gas annealing at 300 $^\circ\text{C}$. Specifically, induced band bending values of (847 ± 23) and (853 ± 24) meV on HF- and RCA-treated surfaces, have been measured. These induced band bending values are on par with those obtained by hole-selective MoO_x and *p*-type *a*-Si:H contacts that mostly rely on induced band bending for their hole-selectivity.²⁴ The fixed charge densities Q_f associated with these band bending values have been calculated using the Girisch model,²⁵ resulting in fixed charge densities of $(-1.6 \pm 0.8) \times 10^{12} \text{ cm}^{-3}$ and $(-1.8 \pm 0.9) \times 10^{12} \text{ cm}^{-3}$ for HF- and RCA-treated surfaces, respectively. The calculated values are very similar to those obtained for thermal ALD Al_2O_3 .⁴ The similar *c*-Si band bending for Nb_2O_5 and Al_2O_3 is further confirmed by SPV measurements of Al_2O_3 on *n*-type *c*-Si ($5 \times 10^{15} \text{ cm}^{-3}$) giving values of about 840 mV. Interestingly, given the insensitivity of the passivation quality to the Nb_2O_5 film thickness on RCA-treated surfaces, these films should exhibit similar levels of *chemical* and *field-effect* passivation for all film thicknesses. Since this even holds for films down to at least 1 nm in thickness, a major contribution of bulk fixed charge in Nb_2O_5 can be excluded. This suggests that the fixed negative charge either resides at the $\text{SiO}_2/\text{Nb}_2\text{O}_5$ interface, as is the case for Al_2O_3 ,²² or is present at the air/ Nb_2O_5 interface. For the latter case, it could be expected that the field-effect passivation would decrease with increasing film thickness due to screening. However, at least for the thickness range studied in this work, no such dependency has been observed.

Finally, it was found that the passivation of 1 nm ALD Nb_2O_5 can be further enhanced by light-soaking. As shown in Fig. 4, an enhancement of the passivation is observed after light-soaking both sides of the substrate for one minute, leading to a reduction in J_0 from 27 to 20 fA/cm^2 . This effect was observed to have already saturated after one minute of

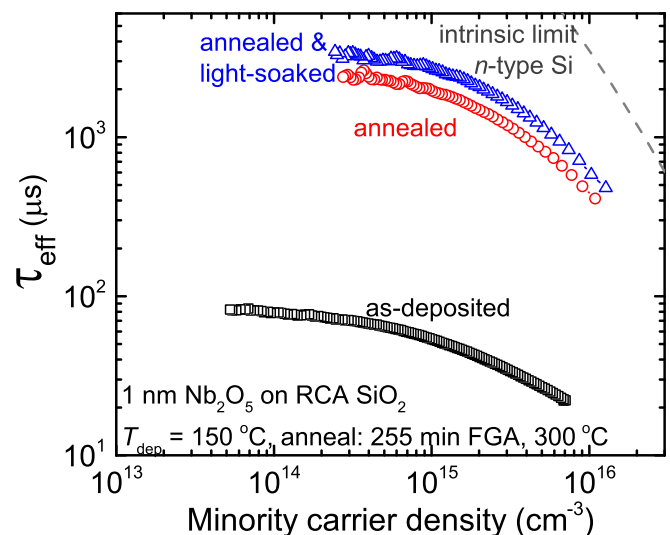


FIG. 4. Effective excess minority carrier lifetime for 1 nm Nb_2O_5 prepared on an RCA-grown SiO_2 interlayer. Data for as-deposited, annealed and annealed plus light-soaked Nb_2O_5 are shown. The intrinsic lifetime limit was calculated from the parametrization of Richter *et al.*²⁷

light-soaking. Similar behavior has been observed for ALD Al_2O_3 and TiO_x , where light-soaking is thought to increase the field-effect passivation by the injection of additional negative charge into the passivation layer.^{6,26}

As alluded to in the Introduction, the exploration of novel passivation materials is also of high interest to the field of passivating contacts. Interestingly, initial explorations on the contact resistivity of *n*-type *c*-Si/ Nb_2O_5 /Al using the Cox and Strack method already show that proper *n*-type contacts can be made with this material. For 2 nm of Nb_2O_5 on HF-treated *c*-Si, a reasonably low contact resistivity of $0.12 \Omega\text{cm}^2$ was achieved after a 15 min anneal treatment in forming gas at 300°C . Note that the formation of a good *n*-type contact by ALD Nb_2O_5 might not be directly expected given its negative charge. However, similar behavior has been observed, for example, in the case of TiO_2 , a negatively charged dielectric that forms an electron-selective contact after coming into contact with Al metal.^{5,6} The contacting behavior will be the subject of more in-depth studies, but these initial results already demonstrate that ALD Nb_2O_5 can not only passivate *c*-Si, but also has potential in the field of passivating contacts.

In conclusion, ALD Nb_2O_5 has been shown to provide effective passivation of *c*-Si on both HF- and RCA-treated surfaces, and insights into the passivation mechanism in terms of *chemical* and *field-effect* passivation have been provided. On both surface types, the passivation by Nb_2O_5 films is aided by a strong hole inversion layer on *n*-type *c*-Si. However, the kinetics of the activation of *chemical* passivation upon annealing by the growth and hydrogenation of interfacial silicon oxide is markedly different for both surface types. On HF-treated surfaces, substoichiometric interfacial silicon oxide forms during deposition and further grows upon annealing. On such surfaces, the passivation quality is observed to improve with increasing film thickness and decreasing deposition temperature. Conversely, the use of an RCA-grown interfacial silicon oxide enables a higher passivation level. Moreover, on such substrates, the passivation kinetics are much less dependent on the growth temperature and film thickness. Specifically, the excellent passivation level is persistent for films as thin as 1 nm. Furthermore, the sensitivity to light-soaking provides an additional route for improving the passivation performance of ALD Nb_2O_5 . Together with the possible low contact resistance to *c*-Si, this makes ALD Nb_2O_5 an interesting building block for passivating contact structures for high-efficiency solar cells.

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