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Electric field induced surface passivation of Si by atomic layer deposited Al₂O₃ studied by optical second-harmonic generation

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ABSTRACT

Recently, we have demonstrated that ultrathin (<30 nm) films of Al₂O₃ synthesized by (plasma-assisted) atomic layer deposition (ALD) provide an excellent level of surface passivation of *c*-Si which may find important applications in (high-efficiency) solar cells. In this contribution, the Al₂O₃ passivation mechanism has been further elucidated by the contactless characterization of the *c*-Si/Al₂O₃ interface by optical second-harmonic generation (SHG). SHG has revealed effective field-effect passivation of the *c*-Si surface caused by a *negative* fixed charge density of $5 \times 10^{12} \text{ cm}^{-2}$ in an annealed, 11 nm thick Al₂O₃ film while it is on the order of 10^{11} cm^{-2} in the as-deposited film which shows negligible passivation. A comparison with SHG measurements on a 84 nm thick *a*-SiN_x:H film treated in a conventional firing furnace has revealed the presence of a *positive* fixed charge density of $2 \times 10^{12} \text{ cm}^{-2}$ which further corroborates the SHG analysis and results.

INTRODUCTION

Due to the decrease of wafer thickness in crystalline silicon (*c*-Si) photovoltaics, the surface-to-bulk ratio of solar cells increases. This enhances the need of good surface passivation for both the front and rear side of the *c*-Si solar cells. In general, surface passivation can be induced (i) by reducing the amount of recombination centers at the interface (so-called chemical passivation), and (ii) by electrostatic shielding the charge carriers from the interface by an internal electric field (so-called field-effect passivation). Several thin films applied to the *c*-Si surface have shown to obtain a good degree of surface passivation by employing (i) or (ii) or by a combination of both.

Recently, excellent surface passivation by Al₂O₃ films synthesized by (plasma-assisted) atomic layer deposition (ALD) has been reported for *n*, *p* and *p*⁺ type *c*-Si [1,2,3,4]. These results were obtained after a post-deposition anneal as the Al₂O₃ films demonstrated no significant level of surface passivation in the as-deposited state. The surface passivation properties by ALD-synthesized Al₂O₃ films were also confirmed at the device level. Al₂O₃ applied at the rear of diffused emitter *p*-type *c*-Si solar cells yielded a conversion efficiency of 20.6% [5] and Al₂O₃ applied for boron doped emitter passivation at the front side of *n*-type *c*-Si solar cells yielded a conversion efficiency of 23.2% [6].

The passivation properties of Al₂O₃ films are related to *negative* fixed charge in Al₂O₃ leading to an internal electrical field at the *c*-Si interface and therefore providing

field-effect passivation [7,8]. Characterization of the field-effect passivation and the fixed charge density in the Al₂O₃ films before and even during processing could help to further unravel the passivation mechanism. In this respect, the noninvasive nonlinear optical technique of second-harmonic generation (SHG) is a very promising diagnostic. SHG, as schematically illustrated in Fig. 1, is highly interface sensitive for centrosymmetric media and allows for a contactless detection of internal electric fields, which can either be applied static fields or electric fields in semiconductor space-charge regions arising from interfacial charge separation [9,10]. The effect of electric field-induced SHG (EFISH) can be described by the second-order nonlinear polarization $\mathbf{P}^{(2)}(2\omega)$ induced by an incident electric field $\mathbf{E}(\omega)$

$$\mathbf{P}^{(2)}(2\omega) = \epsilon_0 \chi_B^{-(3)}(2\omega) : \mathbf{E}(\omega) \mathbf{E}(\omega) \mathbf{E}_{dc,bulk} \quad (1)$$

where $\chi_B^{-(3)}$ and $\mathbf{E}_{dc,bulk}$ are the third-order nonlinear susceptibility tensor and dc electric field, respectively. EFISH has been used to study charge trapping in the *c*-Si/SiO₂ system [11,12,13,14], photon induced charge trapping [15,16], as well as process-dependent charging in high- κ dielectric stacks [17,18].

Fig. 1. Schematic representation of the second harmonic generation (SHG) method used to measure the electric field in the space charge region of *c*-Si as caused by the presence of fixed negative interface charge in the Al₂O₃ film. The method, sensitive to electric fields of $>10^5 \text{ V/cm}$, is also referred to as electric field induced second harmonic generation or EFISH. SHG can be considered as the conversion of two photons with energy $\hbar\omega$ into a single photon of energy $2\hbar\omega$, a process that can occur only for high intensity (laser) radiation.

Fig. 2. High resolution transmission electron micrograph (HR-TEM) of an ALD-synthesized Al_2O_3 film (20 nm thick) yielding excellent Si surface passivation after a post-deposition anneal. The interfacial SiO_x is ~ 1.5 nm thick [3].

In this paper we characterize as-deposited and annealed $\text{Si}(100)/\text{Al}_2\text{O}_3$ structures with interfacial SiO_x synthesized by plasma-assisted ALD using spectroscopic SHG [19]. The results are compared with SHG results obtained on an $a\text{-SiN}_x\text{:H}$ film deposited with plasma-enhanced chemical vapor deposition (PECVD). The polarity of the fixed charges is confirmed to be negative for the Al_2O_3 and positive for the $a\text{-SiN}_x\text{:H}$. The fixed charge density is also deduced for both materials and it is shown that the density exhibits a strong increase after anneal for the Al_2O_3 , from a value lower than for $a\text{-SiN}_x\text{:H}$ before anneal to a value significantly higher than for $a\text{-SiN}_x\text{:H}$ after anneal. These results provide important insight into the interface passivation properties of Al_2O_3 and directly illustrate the feasibility of SHG as a contactless technique to characterize field-effect surface passivation of $c\text{-Si}$ *in situ* and during processing.

EXPERIMENTAL

Amorphous Al_2O_3 films were deposited at both sides of $275\ \mu\text{m}$ thick P-doped H-terminated $\text{Si}(100)$ wafers with a resistivity of $1.9\ \Omega\ \text{cm}$ by plasma-assisted ALD using alternating $\text{Al}(\text{CH}_3)_3$ dosing and O_2 plasma exposure at a substrate temperature of $200\ ^\circ\text{C}$. After being analyzed, the as-deposited samples were annealed for 30 min at $425\ ^\circ\text{C}$ in N_2 . More details on the preparation of the Al_2O_3 films and their analysis, demonstrating for example a atomic composition of $[\text{O}]/[\text{Al}]=1.5$, a H-content of 2 at.% and a refractive index of 1.62 at 2 eV, can be found in Ref. 20. High resolution transmission electron microscopy (HR-TEM) images, such as shown in Fig. 2, revealed the presence of an interfacial SiO_x layer of ~ 1.5 nm between the $\text{Si}(100)$ and the Al_2O_3 both before and after anneal, with the Al_2O_3 remaining amorphous after anneal [3]. With carrier lifetime spectroscopy the effective lifetimes of $c\text{-Si}$ passivated by as-deposited Al_2O_3 were determined to be <

$10\ \mu\text{s}$, which are indistinguishable from an unpassivated $c\text{-Si}$ wafer, whereas after anneal lifetimes up to 6.6 ms were obtained, corresponding to an excellent level of surface passivation [3,4,7].

An $a\text{-SiN}_x\text{:H}$ film deposited by PECVD in the OTB Solar DEP system from an $\text{Ar-NH}_3\text{-SiH}_4$ gas mixture was used for comparison purposes. This film, 84 nm thick and with a refractive index of 2.1 at 2 eV, was deposited on a $380\ \mu\text{m}$ thick B-doped H-terminated $\text{Si}(100)$ wafer with a resistivity of $8.4\ \Omega\ \text{cm}$. The sample underwent a high-temperature ($\sim 800\ ^\circ\text{C}$) firing step in a standard metallization firing furnace. More details are described in Ref. [21]. The effective lifetime of $c\text{-Si}$ passivated by the $a\text{-SiN}_x\text{:H}$ was determined to be $174\ \mu\text{s}$ with carrier lifetime spectroscopy (as measured right before the SHG experiments).

The SHG experiments were carried out at an angle of incidence of 35° using a Ti:sapphire oscillator providing radiation tunable in the 1.33 - 1.75 eV photon energy range with a pulse duration of ~ 90 fs [22]. Data were obtained at p -polarized fundamental and SHG radiation using a laser power at the sample of 40 mW (fluence $25\ \mu\text{J}\ \text{cm}^{-2}$ per pulse).

RESULTS AND DISCUSSION

In Fig. 3(a) and (b) SHG spectra for p -polarized fundamental and SHG radiation are shown for the 11 nm thick Al_2O_3 film on $\text{Si}(100)$, as-deposited and after anneal, respectively. Both show a distinct resonance in the 3.3 - 3.4 eV range, indicating that the SHG response is dominated by $c\text{-Si}$ interband transitions at the E_0/E_1 critical point (CP) [23,24]. The anneal very clearly modifies the SHG spectrum; the amplitude increases with more than an order of magnitude, whereas the peak shifts from ~ 3.3 to ~ 3.4 eV resulting in a more symmetric feature.

In order to separate different contributions to the SHG response, the spectra have been reproduced using a model in which the SHG intensity is approximated by a coherent superposition of CP-like resonances with excitonic line shapes evaluated at the substrate/film interface [22,25,26]

$$I(2\omega) = \left| A_{zzz}(\omega, \theta) \sum_q \chi_{zzz,q}^{(2)} \right|^2 I_n^2(\omega) \\ \propto \left| A_{zzz}(\omega, \theta) \sum_q \frac{h_q e^{i\varphi_q}}{2\omega - \omega_q + i\Gamma_q} \right|^2 I_n^2(\omega), \quad (2)$$

where h_q denotes the (real) amplitude, ω_q the frequency, Γ_q the linewidth, and φ_q the excitonic phase of resonance q . The spectra have been analyzed in terms of tensor element $\chi_{zzz}^{(2)}$, as including the other elements contributing to p -polarized fundamental and SHG radiation, $\chi_{zzx}^{(2)}$ and $\chi_{xxz}^{(2)}$ [9], does not modify the spectral parameters significantly. The complex function $A_{zzz}(\omega, \theta)$ in Eq. (2) describes the propagation of the fundamental and SHG radiation in the film-substrate system. This film thickness dependent function includes linear optical effects, such as

absorption, refraction, and interference due to multiple reflections within the Al_2O_3 films [22].

The spectrum for the as-deposited Al_2O_3 film clearly has an asymmetric shape, indicating the presence of multiple interfering contributions. The data can be fitted very well when taking into account three contributions with their parameters listed in Table I. The individual resonances, shown in Fig. 3(a), consist of a main contribution at 3.32 ± 0.01 eV and additional contributions at 3.38 ± 0.01 eV and 3.62 eV. The resonance frequency and linewidth of the third contribution are fixed at 3.62 and 0.36 eV, as within the current experimental photon energy range the parameters of this contribution can not be determined unambiguously. These latter values have been reported by Rumpel *et al.* for $c\text{-Si}/\text{SiO}_2$ and have been attributed to a resonance related to Si interband transitions in a thin transition layer between Si and SiO_2 [27,28]. The presence of such an interface resonance seems also viable for the $\text{Si}/\text{Al}_2\text{O}_3$ system, especially considering the presence of the interfacial SiO_x layer as detected by HR-TEM (Fig. 2). Also, the parameters of the resonances at 3.32 ± 0.01 eV and 3.38 ± 0.01 eV correspond well to values reported for the $c\text{-Si}/\text{SiO}_2$ interface [28], as well as to values reported for clean and H dosed $c\text{-Si}$ surfaces [24]. In addition to the good reproduction of the experimental data, this similarity supports the validity of the fitting results.

Moreover, the SHG spectrum after anneal can also be reproduced very well by the same resonances, as shown in Table I and Fig. 3(b). The second contribution at 3.414 ± 0.004 eV is clearly dominant with an amplitude that increased by a factor of six compared to the as-deposited sample. The first contribution has redshifted to 3.25 ± 0.02 eV and has minor impact. This contribution can be assigned to interband transitions related to Si-Si bonds modified due to the vicinity of the interface with the film [23,24]. The redshift of this “modified Si-Si interface contribution” after anneal might be related to further weakening of Si-Si bonds [23,27], indicating structural changes in the (interfacial) oxide.

The resonance around 3.40 eV is a clear signature of EFISH originating from the bulk space-charge region (SCR) in the $c\text{-Si}$ [24]. The Si SCR is predominantly caused by fixed charge in the Al_2O_3 , as schematically illustrated in Fig. 1. The drastic increase of the EFISH contribution after anneal is a clear indication for the increase of the fixed charge density Q_f in the Al_2O_3 . The electric field at the interface with Al_2O_3 resulting from the doping of the $c\text{-Si}$ substrates ($2.5 \times 10^{15} \text{ cm}^{-3}$) can be estimated to be $< 10 \text{ kV cm}^{-1}$ [29,30], which is too low to have a significant effect on the SHG response [10,24]. As shown in Table I, the phase difference between the contribution due to the Si-Si interface bonds and the EFISH contribution is $\sim \pi$, both before and after anneal, which indicates a *positively* charged Si SCR [28], and thus *negative* fixed charge in the Al_2O_3 . Electrical characterization of Al_2O_3 films has revealed the presence of negative fixed charge situated predominantly at the interface with interfacial SiO_x [8,31,32].

The intensity of the EFISH contribution resulting from the CP modeling reflects the magnitude of the electric field $E_z^{DC}(z)$ in the Si SCR and can be used to quantify the negative Q_f in the Al_2O_3 before and after anneal. To achieve this, the negative Q_f can be related to the electric field $E_z^{DC}(z)$ in the Si SCR by numerically integrating Poisson's equation. Subsequent integration of $E_z^{DC}(z)$ over the SCR, taking into account the penetration and escape depths of the fundamental and SHG radiation, gives the EFISH electric field and, hence, the EFISH intensity [10,24]

$$I^{\text{EFISH}}(2\omega) \sim \left| \tilde{\chi}^{(3)} : \mathbf{E}(\omega) \mathbf{E}(\omega) \int_{-\infty}^0 e^{-i(K_z + 2k_z)z} E_z^{DC}(z) dz \right|^2, \quad (3)$$

where K_z and k_z are the complex wavevector components of the SHG and fundamental radiation perpendicular to the $c\text{-Si}/\text{Al}_2\text{O}_3$ interface, respectively. To relate the EFISH intensity to absolute values of Q_f in the Al_2O_3 , a 26 nm annealed Al_2O_3 film on Si(100) with a known negative

Fig. 3. SHG spectra for (a) an 11 nm as-deposited Al_2O_3 film on Si(100), (b) an 11 nm annealed Al_2O_3 film on Si(100), and (c) an 84 nm “fired” $a\text{-SiN}_x\text{:H}$ film on Si(100). The solid lines are fits to the data using a superposition of two or three CP-like resonances. The dashed lines represent the individual resonances. Note the difference in vertical scale.

Table I. Parameters of the critical point resonances as obtained from the fits to the SHG spectra for the passivation films deposited on Si(100): 11 nm Al₂O₃ as-deposited and after anneal, and 84 nm a-SiN_x:H after “firing”. In the analysis φ_i is set to 0. Parameter values in italic were fixed in the analysis.

Sample	Modified Si-Si interface contribution			EFISH contribution				Oxide related interface contribution			
	h_1 (arb. units)	$\hbar\omega_1$ (eV)	$\hbar\Gamma_1$ (eV)	h_2 (arb. units)	$\hbar\omega_2$ (eV)	$\hbar\Gamma_2$ (eV)	φ_2 (π rad)	h_3 (arb. units)	$\hbar\omega_3$ (eV)	$\hbar\Gamma_3$ (eV)	φ_3 (π rad)
Al ₂ O ₃ as-dep.	0.31 ± 0.05	3.32 ± 0.01	0.121 ± 0.009	0.19 ± 0.07	3.38 ± 0.01	0.09 ± 0.01	0.9 ± 0.1	0.23 ± 0.12	3.62	0.36	0.33 ± 0.07
Al ₂ O ₃ anneal	<i>0.31</i>	3.25 ± 0.02	<i>0.121</i>	1.21 ± 0.05	3.414 ± 0.004	0.114 ± 0.004	1.00 ± 0.06	0.23	3.62	0.36	0.33
a-SiN _x :H “fired”	0.13 ± 0.02	3.32 ± 0.01	0.090 ± 0.008	0.65 ± 0.03	3.419 ± 0.007	0.116 ± 0.004	0.01 ± 0.03	-	-	-	-

Q_f of $(1.3 \pm 0.1) \times 10^{13} \text{ cm}^{-2}$, as measured in a corona charging experiment was used for calibration [7,19]. This value of Q_f yields an electric field at the position of the interface of $E_z^{DC}(0) = 2.1 \text{ MV cm}^{-1}$, corresponding to a strong EFISH contribution. Using this calibration, the EFISH intensity deduced from the CP modeling can be related to a negative fixed charge density of $Q_f = 2.5 \times 10^{11} \text{ cm}^{-2}$ for the as-deposited 11 nm thick Al₂O₃ film, whereas after anneal Q_f has increased to $5.4 \times 10^{12} \text{ cm}^{-2}$ for this sample. These values, which are in good agreement with results obtained by conventional capacitance-voltage analysis [7,32], indicate that the field-effect passivation improves significantly upon anneal.

In Fig. 3(c) the SHG spectrum for p -polarized fundamental and SHG radiation is shown for the 84 nm thick a-SiN_x:H film on Si(100). This spectrum also shows a distinct resonance around 3.4 eV which indicates again that c -Si interband transitions at the E_0/E_1 critical point (CP) dominate the SHG response. The data can be fitted very well when taking into account two contributions consisting of a Si-Si interface contribution at $3.32 \pm 0.01 \text{ eV}$ and EFISH contribution at $3.419 \pm 0.007 \text{ eV}$. The EFISH contribution is clearly dominant and the parameters of the fit are listed in Table I. The phase difference between the contribution due to the Si-Si interface bonds and the EFISH contribution is ~ 0 , indicating a negatively charged Si SCR and therefore *positive* charge in the a-SiN_x:H film. Using the aforementioned calibration data, the EFISH intensity deduced from the CP modeling can be related to a fixed charge density of $Q_f = 1.6 \times 10^{12} \text{ cm}^{-2}$. The presence of positive charge in a-SiN_x:H is well known and also the value of Q_f is within the range typically reported for a-SiN_x:H yielding good surface passivation of c -Si [33,34,35]. These results corroborate therefore the SHG analysis and confirm that an annealed Al₂O₃ film has a higher level of fixed charges than a-SiN_x:H and that the fixed charges have the opposite polarity. Consequently, the higher fixed charge density in Al₂O₃ and the related higher level of electric field induced surface passivation explains the fact that Al₂O₃ outrules the level of surface passivation obtained with a-SiN_x:H. However, we also want to stress that there are clear indications that the chemical passivation by Al₂O₃ is also more effective than for a-SiN_x:H [8,19].

CONCLUSIONS

Al₂O₃ and a-SiN_x:H thin films deposited on Si(100) have been studied by spectroscopic SHG. The plasma-assisted ALD Al₂O₃ films contain fixed charge with a *negative* polarity and with a density increasing from $\sim 10^{11} \text{ cm}^{-2}$ before anneal to $10^{12} - 10^{13} \text{ cm}^{-2}$ after anneal. The “fired” PECVD a-SiN_x:H film contains a *positive* fixed charge with a density of $1.6 \times 10^{12} \text{ cm}^{-2}$. These experiments confirm therefore that the c -Si passivation properties of Al₂O₃ and a-SiN_x:H can be contributed to an effective electric field induced surface passivation effect. Moreover, for annealed Al₂O₃ the excellent passivation properties recently observed are a result of the increase in the internal electric field after the anneal while also an additional reduction of interface defect seems to be important [8,19].

The results demonstrate the feasibility of using SHG for contactless investigation of electric field effect induced passivation mechanisms of c -Si. Importantly, the technique can in principle also be applied *in situ* and during processing. This kind of analysis is inaccessible by conventional techniques such as capacitance-voltage measurements, while the SHG studies do not require a minimum film thickness. Finally, we want to note that the SHG technique was recently also successfully employed for *in situ* studies on the growth of amorphous and epitaxial silicon films on c -Si [36,37,38], a subject that is of high relevance for the passivation of c -Si surfaces by a-Si:H films.

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