

Stability Study of Alkyl Monolayers Directly Attached to Si (111) Surface for Solar Cells Application

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Abstract: - The effect of surface recombination of the Si can be reduced by the chemical passivation technique. Formation of 1-decene monolayer was directly attached on Si (111) surfaces by using mild thermal hydrosilylation (110 °C, 20 h) in the presence of Lewis acid catalyst which uses hydrogenated (Si-H) surfaces as the precursor. Characterization by XPS and water contact angle reveals that the alkyl monolayers are covalently bonded to silicon and show better stability after exposed to air. Kelvin probe measurements show that there is charge trapping and detrapping mechanism under illumination, even the samples was tested after 21 days. The surface traps and reversible filling of charges occurs in slow rate for the passivating layers on the silicon.

Key-Words: - surface recombination, chemical passivation, 1-decene, hydrosilylation, alkyl monolayers, Kelvin probe

1 Introduction

The surface plays an important role in thin silicon solar cells where the diffusion lengths of the minority charge carriers can exceed substantially the thickness of the device. The surface and interface electronic properties of silicon, in particular, are important for the efficiency of the device. To a large extent, these properties are defined by the density and nature of the surface and interface states resulting from unsaturated dangling bonds [1]. These surface states created by dangling bonds act as recombination centers for electron and holes which may influencing the surface properties.

The effect of surface recombination of the silicon can be reduced by the chemical passivation technique. Passivation of organic monolayers have been considered as a promising passivating coating for silicon and germanium [2], where the methods to obtain covalently attached monolayers on silicon

surfaces ranging from thermal hydrosilylation, UV light, peroxides Grignard reaction and electrochemical reaction. We have chosen a thermal hydrosilylation as an initiator because it is cheaper, safer and more versatile method. These procedures to prepare the monolayers required the use of neat alkenes or alkynes [3] which is shown the chemical stability of the surface and electrical conductivity of thin layers, by attaching a layer of thickness up to 2-3 nm [4]. Passivation with organic monolayers is shown to produce surfaces that are stable in hot solvents, acids and bases [5]; they are thermodynamically and kinetically stable due to the high bond strength and low polarity of the bond [6]. Previous works also shown that these 1-alkene monolayers act as a good surface passivation with a low interface state density [4], a long minority carrier lifetime [7] and a low surface recombination velocity [7,8].

The formation of alkyl chains on the silicon surface may create a hydrophobic environment due to the strong covalent Si-C bond and not readily penetrated by water and oxygen molecules. However, the stability of the monolayer could not last for long-term. A reason is related to the passivation quality where they are remaining Si-H sites (about 45-50%) [9] and create small oxide patches after completion of an alkyl monolayer.

In this study, the similar approach of using 1-decene in the presence Lewis acid catalyst to form covalently bonded alkyl chains that act as the interface layer on silicon surface. The stability of these layers is important to be understood before any addition of light harvesting structures in the form of Langmuir-Blodgett (LB) film [10]. These passivation surfaces may create environment for the development of new solar cell concepts. The substrates are also investigated by Kelvin Probe measurements where this technique can provide information about the changes in the electronic properties i.e. band bending, charge trapping, surface dipole layer of the semiconductor surfaces.

2 Experimental

Samples of single-sided of *n-type* Si (111) wafers with 525 μm thickness were used in these experiments. The resistivity of all the wafers (float zone (FZ) quality) was in the range $\sim 27 \Omega\text{-cm}$ with guaranteed bulk lifetime $> 1000 \mu\text{s}$ and each sample were cut to 1.0 cm^2 . Solutions of 1-Decene were purchased from Sigma-Aldrich and follow by distilled twice under reduced pressure prior to use. Solution of isopropyl alcohol (IPA) (99.5%), H_2SO_4 (30%), H_2O_2 (30%) were purchased from Fisher Scientific and used directly without further purification.

The substrates were degreased in acetone and IPA solution followed by ultrasonic for 15 min. Then the substrate was treated in piranha solution (3:1 $\text{H}_2\text{SO}_4/\text{H}_2\text{O}_2$) for 30 min. This cleaning process leaves 0.6 – 1.5 nm of hydroxylated oxide on the silicon surface which is prevents recontamination of the surface. After rinsing in deionized water, the H-termination was achieved by immersion in NH_4F (40%) and followed by HF (2%). The alkyl monolayer was experimented using a mild thermal hydrosilylation of 1-alkenes, in addition Lewis acid catalyst of Ethylaluminiumdichloride ($\text{C}_2\text{H}_5\text{AlCl}_2$) [11]. The H-terminated samples were immersed in deoxygenated 1-decene with presence of Lewis acid catalyst which is carried out in glove box and heated

at 110 $^\circ\text{C}$ for 20 hours. The samples were removed from the reaction flask and washed with 1,1,1-trifluoroacetone (TFA) to neutralize residual of the catalyst. Then the sample was washed with methanol and cleaned with hexane.

The XPS spectra were recorded by Thermo Fisher ESCALAB 250 where this imaging XPS instrument has a monochromatic Al X-ray source, which allows energy resolution of 0.45 eV FWHM. This makes the identification of chemical states much more precise and have the spatial resolution is 15 microns in spectroscopy mode and 3 microns in chemical imaging mode. It also has an Al/Mg dual anode X-ray source which allows analysis areas of up to 8mm diameter. The base pressure in the instrument is $< 3 \times 10^{-10}$ mbar. Spectra were fitted with Gaussian profiles using standard procedures. The positions of all peaks were normalized to C1s at 285.0 eV. The chemical stability of the samples was investigated when exposed to air (normal laboratory conditions). The water contact angles were measured with a Kruss DSA 100 using a computer controlled contact angle calculation program supplied by the manufacturer. The measurements were carried out with water droplets of fixed size, about 1.0 μL in volume and represent an average of at least five points at room temperature under ambient humidity.

The light absorption and photon flux of the samples were uniformly illuminated by a low response of monochromic light source of Bentham spectrometer CL2 universal spectral irradiance standard beam (Bentham, Inc.) with a wavelength range 250 to 3000 nm. The surface photovoltage (SPV) was measured using Ambient Kelvin Probe System (KPSP) with software SKP 4.5 (KP technology Ltd). The work function of the semiconductor can be measured from high resolution ($< 50 \mu\text{eV}$) with characteristic of voice-coil actuator [12]. The Kelvin probe (KP) consists of a flat circular electrode placed at the top and parallel to a stationary electrode. The gold reference tip normally works vibrates and contactless to the sample. The difference of work function is determined by the value of the external voltage at null condition which is produces a contact potential difference (CPD) between the metals. The electric field created by V_{CPD} induces charges of opposite sign on the capacitor electrodes. This induces charge was detected as transient current flow in an external circuit by changing the spacing between electrodes shortly [13].

3 Results and Discussion

3.1 Characterization Monolayers

The X-ray photoelectron survey spectrum of this surface (Fig. 1) showed the binding energy of 1-decene monolayer on Si (111) sample. As a result, there are significant peak in the Si, C and O signals which is good agreement with previous work by Boukherroub and co-workers [14]. The alkyl chains of 1-decene yield a clear C 1s signal which is higher than the oxygen signal but smaller and closer to the Si signal; while a small amount of fluorine is observed after exposure to NH_4F and HF.

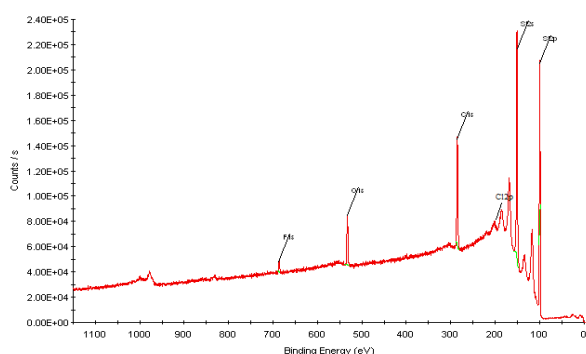


Fig. 1 XPS survey spectrum of Si(111)- $\text{C}_{10}\text{H}_{21}$ prepared by thermal hydrosilylation reaction of 1-decene.

Fig. 2(a) shows the high-resolution XP spectrum of the C 1s which consists of three components: C-C/C-H (85.6 %), C-O (8.9 %) and the rest is Si-C. The XPS C 1s spectra showed a peak located at 285.0 eV, which is assigned to C-C/C-H bonding. There was an inflection point at 283.8 eV in the spectra, indicating a peak at this binding energy. Furthermore, the inflection point at 286.5 and 283.9 eV which correspond to a chemical bonding state originating from C-O and Si-C bonds, respectively.

The Si 2p peak shown in Fig. 2(b) has two components of Si 2p_{3/2} (Si1) and 2p_{1/2} (Si2) estimated at 100.1 and 99.4 eV, respectively. These results have three arguments by previous researchers which is used 1-decene as pre-cursor for organic monolayer on silicon surface. The existence of two components peak of Si 2p has a similar result that obtained by Boukherroub *et al.* [14] and Rosso *et al.* [15]. The Boukherroub *et al.* [14] works appear peak of Si 2p estimated at 100.1 and 99.4 eV that carried out using ultraviolet irradiation method. Rosso *et al.* [15] were acquired to demonstrate peak of Si 2p exactly at 101.1 and 100.4 eV using H-terminated by HF and heated neat alkenes at 130 °C for 6 hours.

Meanwhile, one component peak of Si 2p was achieved by Ishizaki *et al.* [16] that carried out using H-terminated by NH_4F and thermal hydrosilylation method (5 hrs at 160 °C). The energy of the primary Si 2p_{3/2} peak is at 99.4 eV compared to the native Si 2p_{3/2} peak at 99.0 eV. This indicates that a proportion of Si on the surface of this sample is H-terminated. The peak shift is the only indication of H-termination which is indicating possible way to remove completely hydrogen bonding from the surface by minimizing pre-treatment of H-terminated using either NH_4F or HF only. Furthermore, the O 1s peak in the survey spectrum does not appear to be associated with the formation of SiO_2 (which would appear at about 102 [17] – 104 eV [18]). This indicates that the silicon dioxide layer on the Si substrate had been almost removed.

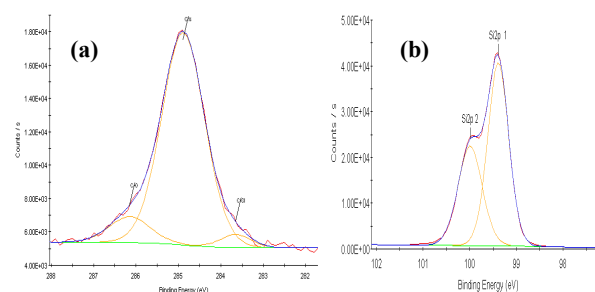


Fig. 2 High-resolution XP spectra of surfaces a) C 1s region; b) Si 2p region prepared by the thermal hydrosilylation reaction of 1-decene.

Table 1 The relative atomic concentration (%) of the elements present in the samples by XPS

Element	Relative atomic concentration (%)	
	Native	1-Decene
Si	61.0	65.8
C	9.4	27.7
O	28.9	5.7
F	0.8	6.8

Result of quantitative XPS analysis of the sample is summarized in Table 1. The surface oxygen concentration decreases greatly from 28.9 to 5.7 % due to the H-termination and organic passivation process, since the surface oxide layer is removed earlier during H-termination process. This oxygen concentration value of 5.7 % is due to contamination, most likely adsorbed water molecules during sample exposure in air. The carbon amounts on the 1-decene monolayer samples increase greatly up to 27.7 % compared with the native sample (9.4 %) which is due to contamination during sample handling in air. This increase in the surface carbon concentration supports our findings

that 1-decene molecules have been passivated on the samples.

The measurement of water contact angles is a useful tool for monolayer analysis which able to monitor the monolayer stability. If the sample is measured as a function of time, any changes on the hydrophobic surface will show significantly effect on contact angles [19].

Table 2 Water contact angles of the samples.

Samples	(°)
Native oxide	58
Si-H	84
1-decene (24 h)	104
1-decene (192 h)	101
1-decene (624 h)	93

The H-Si (111) surface shows a water contact angle of about 84° (see Table 2), in good agreement with the reported values [20]. The surface wetting properties of H-terminated surface reacted with 1-decene achieve the maximum value (104°) within 24 hours of exposure to air. These values correspond to 1-decene monolayer is closely packed methyl terminated surfaces, and agree well with reported literature values of 102° [21], 107° [22], 108° [16]. Ishizaki *et al.* [16] reported that water contact angle is strongly depend on the molecular structure of the monolayer, with the C₁₀ (1-decene) value less than 110°. Furthermore, the sample was maintaining hydrophobic surfaces (above 101°) within 192 hours before the contact angle drop to 93°. The minimal difference (~ 11°) after the sample is exposed to air for 624 hours, suggests a very low inherent reactivity of the oxide surface toward 1-decene.

3.2 Charge Trapping Mechanism

The charge trapping and detrapping of electrons on the native oxide and 1-decene monolayers was investigated by Kelvin probe measurements. There are two advantages of Kelvin probe method on monitoring of surface modification. The surface charging can be measured by continuously monitoring the position of the Fermi level of the illuminated semiconductor. The samples also can be measure in the dark which is able to provide information about the decay process upon deactivation of light. The surface photovoltage (SPV) measurements were take place at wavelength of 900 nm by setting a maximum slit opening of intensity at 8.00 mm. The minus sign of work function represent of the work function of the tip is

larger than the silicon surface where the *n-type* silicon material was used in this study.

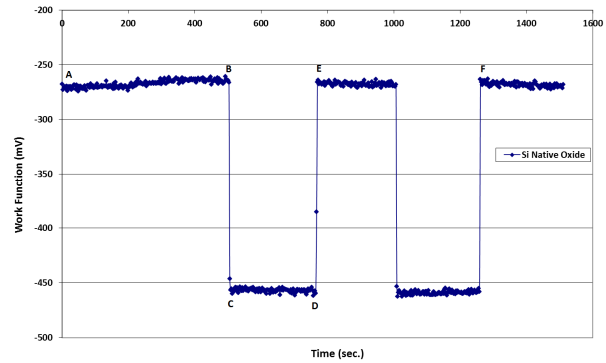


Fig. 3 SPV measurements on silicon *n-type* native oxide at 900 nm wavelength.

For silicon native oxide sample, an initial time of work function from A to B is in a dark as shown in Fig. 3. A fast drop of work function of -181.8 mV is observed when the sample is illuminated from B to C. The fast drop is due to the excess electrons and holes produced by the light in the conduction and valence band that flattening of the band of the semiconductor. Under illumination, the bands are flattened (from C to D) which is no evidence of trapping of electron in the surface states of silicon native oxide. At D, the light is extinguished, the band flattening will disappear. The work functions up immediately to E which is equal in magnitude to BC. This mechanism is reflects to a normal recombination process due to recombination of excess holes and electrons in the valence and conduction bands. We observed that the work function at E is almost similar which is indicating no detrapping process is take place. Since the relaxation time in-between measurement just 200 seconds, mechanism of on and off the illumination is repeated as similar step A-B-C-D-E where a similar of SPV plot is observed. The work function is almost back to baseline as recorded earlier in B after 500 second. At F, a slightly higher than A which is indicates the process starts to reach a thermodynamically stable state again. A similar mechanism of charge trapping was explained earlier by Haynes and Hornbeck [23].

Upon illumination, the surface charge can be calculated as

$$Q_{sc} = \sqrt{2q\epsilon_s N_d (\psi_{bi} - V_{cpd} - kT/q)} \quad (1)$$

$$\psi_{bi} = V_{cpd} + kT \ln(N_c/N_d) \quad (2)$$

where ϵ_s is dielectric constant of Si, $kT/q = 0.025$ V, N_d is $1.7 \times 10^{14} \text{ cm}^{-3}$, N_c is $2.8 \times 10^{19} \text{ cm}^{-3}$ and ψ_{bi} is built-in voltage. The surface charge is calculated about $3.24 \times 10^{10} \text{ eV/cm}^2$ for Si native oxide.

A sample of 1-decene monolayer on the silicon surface was characterized after 48 hours exposed to air and plotted in Fig. 4. We observed that the SPV difference of 1-decene monolayer is much lower compared to the silicon native oxide. For example at 900 nm wavelength, the SPV of the 1-decene monolayers on silicon surface (48 and 504 hours) demonstrated a small work function of -25.3 mV and -31.3 mV, respectively. A difference of SPV value is indicating a variance of band bending due to the surface modification on the samples. A large SPV of silicon native oxide reflects the distribution of carrier density in the surface states which is caused by surface recombination of excess holes and electrons. A smaller SPV of 1-decene monolayers is due to surface passivation by replacing the silicon-oxygen bonding with silicon-carbon bonding on the dangling bonds. Indirectly, it may reduce surface recombination as well as band bending of the samples. The lower Q_{sc} of alkene monolayers are recorded about $2.64\text{--}2.66 \times 10^{10} \text{ eV/cm}^2$ for both samples which explains the excellent passivating properties of the organic monolayers.

Even though the SPV of sample 1-decene monolayer 504 hours exposed in air is slightly higher, but it is still maintain below SPV value obtained by Bin *et al.* [24]. This study reported that Si-Decene (C_{10}) was prepared without catalyst where the SPV rapidly changed from 80 to 160 mV (characterized by Kelvin Probe). It indicates the surface potential increases due to water or oxygen molecules are capable of penetrating through the shorter non-branched monolayers and oxidizing the Si surface. Thus, a low band bending with the existence of Lewis catalyst in the formation alkene monolayer on the silicon surface has proved and prolongs the stability against the oxidation in air [11, 25]. These results were supporting the XPS spectra and contact angle results that reported earlier and shows these monolayers maintaining a low inherent reactivity of the oxide surface toward Si-C bonding. A similar finding is observed by Honeyman and co-workers [26] where the decene monolayer shows least amount of oxidation that took place after 19 days.

A baseline of work function for both samples was change indicates a difference distribution of

carrier's concentration before it reach equilibrium in KP measurements. Trapping of charges at the illuminated sample surface will shift the Fermi level of the 1-decene monolayer sample. An initial time of work function (from G to H) and (from M to N) is in a dark. Under illumination, a fast drop of work function of -25.3 mV and -31.3 mV is due to the light-induced flattening of the bands of the samples (from H to I and N to O), respectively. The subsequent slow exponential increase in work function is due to slow extra charging of the surface as observed from I to J. The generated excess electron and holes in the silicon filled the interface traps that were the defects located at the alkene/silicon interface and possibly electron charge were trapped in the interface layers. This mechanism is also observed but very slow changing as shown from O to P. When the shutter was closed (at J and P), the work function suddenly rises to K and Q. We observed the work function is up immediately which is equal in magnitude to HI and NO. The work function is gaining an optimum point of work function before it decay over time for both wavelengths.

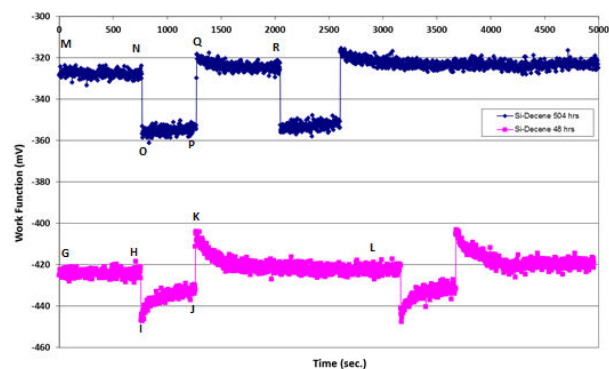


Fig. 4 SPV measurements on 1-decene monolayers of silicon surface (after 48 and 504 hours exposed in air).

The mechanism of light extinguished is reflects to a normal recombination process due to recombination of excess holes and electrons in the valence and conduction bands. At K and Q, the charge detrapped quickly when the shutter was closed where the charge is depends on the surface potential of the interface layers. At L and R, the work function is almost back to baseline which is evidence of slow states occurred in the alkene/silicon interface layers. Although 2 samples of the alkene monolayers was measured at two different time, the mechanism of trapping and detrapping are still occurred with small changes in surface potential. It is evidence the alkene monolayers is stable, strong affinity to the silicon and anchors the molecules to it.

4 Conclusion

We had investigated the stability of organic monolayers of 1-decene on Si surface for more than 21 days via contact angle and kelvin probe measurements. The significant results of XPS spectra show the covalently bound alkyl monolayers had a well passivated by presence of carbonyl groups on the monolayer surface. The structures of silicon-carbon (Si-C) bonding via 1-decene monolayers is possibly recommended for possess a functional group (LB films) that has a strong affinity to the substrate and anchors the molecules to it. The mechanism of charge trapping and detrapping by kelvin probe measurement has shown that the stability of whole monolayer surface and very low inherent reactivity of the oxide surface toward 1-decene. A smaller SPV of 1-decene monolayers indicates a low of band bending which reduces the number of surface charge filled on the passivating surface of the silicon.

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