Sensitivity Enhancement Mechanisms in Textured Dielectric based Electrolyte-Insulator-Semiconductor (EIS) Sensors

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The possible mechanisms governing the enhancement of sensitivities of Electrolyte-insulator-semiconductor (EIS) devices upon surface modification (texturing) realized by electrostatic attachment of silica particles using 3-aminopropyl triethoxysilane (APTES) as a linker are presented. EIS devices were fabricated with a textured dielectric surface using SiO2 particles (of diameters 475, 135, and 70 nm) and screen printed Ag/AgCl electrodes. A maximum pH sensitivity of 52.4 mV/pH was achieved for the EIS device textured with 70 nm particle size while the sensitivity with the planar dielectric was 37.1 mV/pH. The APTES modification enhanced the adsorption of H+ ions by protonation of the –NH2 to –NH3+ sites as seen from the capacitance versus voltage (C-V) hysteresis voltages. UV-Vis absorption and photoluminescence (PL) spectra indicated that the surface defects on the textured surface increased with decreasing particle size. Zeta potential measurements suggested a combined acid-base behavior of textured surface with the formation of –NH3+ and –SiO– groups. The flatband voltage study showed that surface textured with 70 nm SiO2 particles provided the optimum ratio of Si-OH and –NH2 groups. The various chemical treatments during texturization did not affect the characteristics at the Si/SiO2 interface.

In this work, an attempt is made to understand the mechanism governing the sensitivity enhancement induced by physical/chemical modifications of the oxide surface with SiO2 particles using APTES as the linker. For this, the phenomena occurring at the electrolyte-oxide and oxide-semiconductor interfaces is investigated by fabricating EIS (using printed Ag/AgCl electrode) and MOS devices on planar and textured dielectric-silicon wafers. Capacitance-voltage (C-V) hysteresis curves of EIS devices are obtained to understand the effect of silanization and texturing on the sensitivity. The nature of the sensing surface (i.e., number and the type of sites) and its chemical reactivity determine the sensitivity of the EIS device. UV-Vis absorption and photoluminescence was recorded to ascertain the defects present while zeta potential measurements were made to analyze the surface charge characteristics of the textured oxide. C-V and conductance-voltage (G-V) characteristics of MOS devices were studied to examine the changes in the flatband voltage and the interface states occurring at the oxide-semiconductor interface by chemical and physical modifications.

Experimental

Fabrication.—To fabricate the MOS devices, a p-type silicon (100) wafer was heated at 900 °C for 1 h in oxygen to grow an oxide layer of a thickness of about 50 nm. The wafer was cut into small sizes (15 × 15 mm2), cleaned with RCA1 and RCA2 solutions, hydrolyzed in piranha solution and subjected to silanization with APTES in succession. Silica particles of various sizes (475, 135 and 70 nm diameter), prepared by the modified Stöber method, were then attached to the silanized wafer pieces via electrostatic attachment between the –NH2 groups on the APTES modified ZnO/Silica nanoscopic field effect transistor. Hsu et al. 11 designed a glucose sensor based on a poly-silicon wire coated with a mixture of γ-APTES and silica nanoparticles (NPs). The improved detection limit was attributed to the large specific surface area of the silica NPs cluster and the increased amount of OH-bonds. Wu et al. 12 fabricated a label free DNA detection device with sensitivity in the range 1 fM-10 μM by utilizing a mixture of UV illuminated γ-APTES and silica NPs. Further, Oh et al. 13 used silica nanowires and improved the pH sensitivity from 54.9 mV/pH to 60.2 mV/pH. Xue et al. 14 fabricated a Si/SiO2 thin-film based ion-sensitive field-effect transistor (ISFET) by using self-assembled carbon nanotubes (CNTs) and silica NPs to detect acetylcholine with high sensitivity but with poor stability due to lack of cohesiveness in CNTs and NPs. In an earlier work from our group, 15 we fabricated a textured dielectric by electrostatic attachment of silica particles to the APTES modified oxidized silicon surface and observed improvement in the sensitivity with decreasing particle size. The sensitivity enhancement of up to ~ 42% upon texturization was achieved from the base value of ~ 38.3 mV/pH obtained in the planar or non-textured dielectric.

Characterization.—The capacitance versus voltage (C-V) and conductance versus voltage (G-V) measurements were made with...
an impedance analyzer (Agilent 4294A). Copper wires were attached to the device (placed in holder made of Teflon) with silver paste for connecting to the impedance analyzer. C-V measurements were undertaken in the voltage range −3 V to 2 V with an AC signal of 40 mV at 1 kHz frequency after filling the reservoir with 20 μl of analyte. To calculate the pH sensitivity of the EIS device, the C-V curves were obtained with pH values in the range of 4−9. The device sensitivity was calculated using the procedure reported earlier.\textsuperscript{15} The interface states (Nss) were determined by the conductance approximation method.\textsuperscript{17} The Nss can be expressed as

\begin{equation}
N_{SS} = \frac{2}{qA} \left( \frac{G_m}{\omega C_{ox}} \right)_{\text{max}} \left( 1 - \frac{C_m}{C_{ox}} \right)^2
\end{equation}

with \( C_{ox} = C_{\text{ma}} \left[ 1 + \left( \frac{G_m}{\omega C_{\text{ma}}} \right)^2 \right] = \frac{\varepsilon_i \varepsilon_0 A}{d_{ox}} \),

where \( G_m \) and \( C_m \) are the measured conductance and capacitance, respectively, \( C_{ox} \) is the oxide capacitance, \( G_{ma} \) and \( C_{ma} \) are the maximum measured conductance and capacitance, respectively, \( \varepsilon_i \) is the insulator dielectric constant of SiO\(_2\), \( \varepsilon_0 \) is the permittivity of free space, \( A \) is the area, and \( d_{ox} \) is the oxide thickness.

UV-Vis absorption spectra were obtained with a UV-Vis-IR spectrophotometer (Varian, Carry 5000) in the wavelength range 200−800 nm to gather evidence about the presence of paramagnetic defects like E’centers, i.e., \( =\text{Si} =\text{Si} \) (paramagnetic positively charged oxygen vacancies) and \( =\text{Si} \equiv \) (neutral dangling Si bonds) on the textured silica surface.\textsuperscript{18} Photoluminescence (PL) spectra recorded using a Nd:YAG laser at a wavelength of 266 nm to obtain information about the Si–OH, Si–H, Si–O\(^-\) and self trapped exciton (STE) sites on planar and textured oxide surfaces.\textsuperscript{19,20} To study the nature of the surface charges, zeta potential measurements were conducted by the electrophoretic light scattering technique using an analyzer (Beckman Coulter, Delsa Nano C).

### Results and Discussion

**Electrical characteristics of EIS devices.**—The pH sensitivity studies of EIS devices with their drift and hysteresis are discussed in the Sensitivity, hysteresis, and drift section. And, the C-V hysteresis studies to understand the role of chemical (APTES) modification are discussed in Effect of silanization/texturing on capacitance-voltage (C-V) plots section.

**Sensitivity, hysteresis, and drift.**—The sensitivity of an EIS device is based on the change in the flatband voltage of the C-V curves with the change in the pH value of an electrolyte. The change is associated with the reaction of the hydrogen and the hydroxyl moieties present at the insulator (oxide) surface with the chemical species/groups in the analyte. The sensitivity was found to improve with texturing of the oxide surface by attaching silica particles with APTES as a linker. Moreover, the sensitivity also increased with decreasing the size of silica particles as well.\textsuperscript{15} Similar trends were observed in this work with the Ag/AgCl passivated electrodes (Table I). The Ag/AgCl electrodes are known to reduce the drift voltage significantly.\textsuperscript{21} C-V measurements were carried out for several devices with buffer solutions of pH varying from 4 to 9. Typical C-V plots of the EIS device textured with 70 nm silica particles show a linear voltage shift with increasing pH (Figure 2a). The reference voltage versus pH plot is shown in Figure 2b; it corresponds to a pH sensitivity of 52 mV/pH and a linearity of 0.99. Three such devices displayed the average sensitivity of 52.4 mV/pH. Table I lists the pH sensitivity data of all the devices.

The hysteresis is important for determining the error caused in pH measurement by defects present in the insulating layer.\textsuperscript{22} It was measured by taking the difference of the initial and final reference voltages at pH 7 when the EIS device was subjected to a pH cycle of 7−4−9−7 for about 35 minutes. The drift is yet another factor that affects the stability of the device. The drift voltage was measured by exposing the EIS sensor to a standard solution of pH 7 for 1 h. The voltage shift does not decrease or increase monotonically with time, as was observed by Oh et al.\textsuperscript{13} The hysteresis and drift voltages measured for all planar and textured EIS devices are listed in Table I. While the hysteresis is reduced (2.3 mV) for the device with the surface textured with 70 nm silica particles, the minimum drift (0.7 mV) is observed for the case of large particles (diameter∼135 nm). The hysteresis and drift voltages data are comparable to those reported in the literature\textsuperscript{7,8,13} for EIS devices. For example, while investigating the sensing performance of high-k Sm\(_2\)O\(_3\) by annealing at different temperatures, Wu et al.\textsuperscript{7} observed hysteresis and drift voltages in the range 6.2−21.8 mV and 1.29−18.14 mV/h, respectively. Pan et al.,\textsuperscript{8} utilizing as deposited and annealed high-k Ho\(_2\)O\(_3\) as pH sensing films, found hysteresis and drift voltages in the range 2.2−11.6 mV and 0.47−6.45 mV/h, respectively. Oh et al.\textsuperscript{13} reported hysteresis voltages in the range 5.3−25 mV in devices with silicon nanowires and Al\(_2\)O\(_3\).

| Device   | Planar | Textured |
|----------|--------|----------|
| pH sensitivity (mV/pH) | 37.1 ± 1.5 | 45.1 ± 0.6 | 49.7 ± 1.2 | 52.4 ± 2.5 |
| Hysteresis Voltage (mV) | 9.2 | 10.1 | 2.6 | 2.3 |
| Drift/60 min (mV) | 6.3 | 2.7 | 0.8 | 3 |

Table I. Sensitivity, hysteresis voltage and drift of planar and textured EIS devices.
The hysteresis effect was not observed in the planar MOS device (figure not shown here), indicating an insignificant number of interface states present at the oxide-semiconductor interface. It can however occur in the planar EIS structure by adsorption/desorption of $\text{H}^+$ or $\text{OH}^-$ ions (Si-$\text{OH} + \text{OH}^- \leftrightarrow \text{Si-O}^-\text{H} + \text{H}^+$) at electrolyte-oxide interface.\(^{23}\) Increased hysteresis in the silanized EIS device could be due to the enhanced adsorption and desorption of $\text{H}^+$ ions on the silanized oxide surface. An increase in the pH sensitivity (42 mV/pH) with improved linearity was observed upon silanization supports the hypothesis of enhanced adsorption of $\text{H}^+$ ions by protonation of the $\text{–NH}_2$ to $\text{–NH}_3^+$ (eq. 1). Cheng et al.\(^9\) too observed slight increase in pH sensitivity by protonation /deprotonation of the amine end groups on APTES modified SnO$_2$ surfaces. The present results are also in agreement with those of Cui et al.,\(^{23}\) who reported an enhanced and linear pH response with APTES modified silicon nanowires sensor. They observed that $\text{–Si–OH}$ groups are more sensitive in the pH range 6 to 9 as compared to pH range 2 to 6, and the APTES modification provided both $\text{–NH}_2$ and $\text{–Si–OH}$ groups where the $\text{–NH}_2$ group protonated $\text{–NH}_3^+$ at the acidic pH values while $\text{–Si–OH}$ deprotonated to $\text{–Si–O}^-$ at the basic pH values.

Reduction in C-V hysteresis after texturing suggests the increased density of $\text{–Si–O}^-$ and $\text{–Si–OH}$ surface groups in the sensitivity enhancement. This will be further evident from zeta potential and flatband voltage discussed in Surface modification aspects section and Electric and dielectric behavior of MOS devices section, respectively.

Surface modification aspects.—UV-Vis absorption spectroscopy.—UV-Vis absorption spectra were analyzed to gather information about the defects generated by the chemical and physical modifications at the dielectric surface. Figure 4 shows the UV-Vis absorption spectra of planar, silanized and textured oxide surfaces. Emergence of an absorption band in the wavelength range of 300 – 350 nm was observed after silanization of the planar oxidized wafer. Strong absorption peaks observed around 230–240 nm in the textured surface could be due to paramagnetic defects like E’ centers, i.e., $\equiv\text{Si–Si}\equiv$ (paramagnetic positively charged oxygen vacancy) and $\equiv\text{Si}\bullet$ (neutral dangling Si bond), as reported by Rahman et al.\(^{18}\) A slight redshift in the absorption peak resulting with decrease in particle size from 475 nm to 135 nm is possibly associated with the decrease in the number of nearest neighbors which could de-stabilize E’ centers and reduce the energy required for excitation of electrons.\(^6\) Enhanced absorption intensity with the lowering of the particle size can be correlated with the increased surface density of particles on the textured surface.\(^6\) Thus, the generation of defects upon texturing may improve the chemical

Effect of silanization/texturing on capacitance-voltage (C-V) plots.—In EIS devices, C-V hysteresis is caused by the phenomena at the dielectric-electrolyte interface due to adsorption/desorption of $\text{H}^+$ and $\text{OH}^-$ ions and/or at the dielectric-semiconductor interface due to the interface states. The pH sensitivity improves with increase in adsorption/desorption but reduces with increase in the number of interface states. Although silanization is reported to enhance the pH sensitivity and the linearity of field effect based sensors,\(^9,10,23\) not much systematic work is reported so far. In order to understand the role of APTES modification on pH sensitivity, C-V hysteresis measurements were conducted in the devices with planar, silanized and textured (with particles of 135 nm diameter) dielectrics at pH 4 by sweeping the voltage up from $-3 \text{ V}$ to $2 \text{ V}$ and then down from $2 \text{ V}$ to $-3 \text{ V}$. Figure 3 clearly shows an increase in hysteresis upon silanization up to 76 mV from the base value of 28 mV (for planar EIS) and a decrease subsequently upon texturing to an intermediate level of 56 mV. Lue et al.\(^{24}\) attributed the hysteresis effect and the reduced pH sensitivity to the large number of interface traps present at the oxide-semiconductor interface due to the lattice mismatch in the Si/Si$_3$N$_4$ based EIS. However, in the present case, both the C-V hysteresis and the pH sensitivity increased upon texturing, suggesting the hysteresis phenomenon in the EIS devices was induced by the silanization instead of any increase in the interface states at the oxide-semiconductor interface.

Surface modification aspects.—UV-Vis absorption spectroscopy.—UV-Vis absorption spectra of planar, silanized and textured oxide surfaces. Emergence of an absorption band in the wavelength range of 300 – 350 nm was observed after silanization of the planar oxidized wafer. Strong absorption peaks observed around 230–240 nm in the textured surface could be due to paramagnetic defects like E’ centers, i.e., $\equiv\text{Si–Si}\equiv$ (paramagnetic positively charged oxygen vacancy) and $\equiv\text{Si}\bullet$ (neutral dangling Si bond), as reported by Rahman et al.\(^{18}\) A slight redshift in the absorption peak resulting with decrease in particle size from 475 nm to 135 nm is possibly associated with the decrease in the number of nearest neighbors which could de-stabilize E’ centers and reduce the energy required for excitation of electrons.\(^6\) Enhanced absorption intensity with the lowering of the particle size can be correlated with the increased surface density of particles on the textured surface.\(^6\) Thus, the generation of defects upon texturing may improve the chemical

Figure 2. (a) C-V curves at 1 kHz frequency at different pH values of the electrolyte and (b) Sensitivity plot for an EIS device with dielectric textured with 70 nm sized particles.

Figure 3. C-V hysteresis curves of planar, silanized and textured (70 nm) EIS devices measured at 1 kHz.
reactivity of the oxide surface with the electrolyte and enhance the sensitivity.

Photoluminescence (PL) measurement.—Figure 5 shows the PL spectra of planar silicon oxide surface before and after silanization. It reveals the presence of three sites on the planar surface emitting at wavelengths 632 nm, 520 nm and 410 nm corresponding to Si–O–, Si–OH and Si–H, and the oxygen-deficient centers (ODC), respectively.\(^{19}\)

Also, one more emission peak was observed at wavelength 390 nm which may be associated with Si dangling bonds present either in the silicon rich SiO\(_2\) film or at Si/SiO\(_2\) interface as reported elsewhere.\(^{26,27}\) But, all the emission peaks diminish after silanization due to blocking of respective surface sites by the amine groups.

The PL emission spectra of the oxide surface textured with SiO\(_2\) particles (of diameter 475, 135 and 70 nm) are shown in Figure 6 together with that of the silanized surface for the purpose of comparison. Blue, green and red emissions observed in all the textured samples provide evidence for the presence of surface defects. Increase in the peak intensity was also observed with the decreasing size of the SiO\(_2\) particles. This confirms increase in the number of oxygen-deficient centers (ODC), i.e., =Si–Si= and =Si–O–Si–O–Si=, Si–OH and Si–H, and Si–O– groups responsible for the blue, green and red emissions, respectively.\(^{18}\) Moreover, no wavelength shift of peaks with change in the particle size indicates absence of confinement effects (minimum particle diameter being 70 nm). In contrast, Glinka et al.\(^{20}\) observed confinement effects with fumed silica nanoparticles of diameters 7 nm and 15 nm. The increase in defects on the textured surface with decreasing particle size is consistent with other observations.\(^{16}\) Thus, the increase in the number of surface defects improves the buffer capacity and, in turn, enhances the pH sensitivity of the EIS device.

Zeta potential measurements.—The surface charge characteristics of bare and modified surfaces exposed to the electrolyte were determined by zeta potential measurements. The aim was to understand the nature of the changes occurring in the potential of the SiO\(_2\) surface after silanization with APTES and texturing by silica particles. Zeta potential measurements were conducted on planar, silanized and textured (with particles of diameter 70 nm) dielectric surfaces showed zeta potential value of \(-36.87 \text{ mV}\), \(+31.87 \text{ mV}\), and \(-9.23 \text{ mV}\), respectively. The surface becomes positively charged after silanization which indicates the presence of \(-\text{NH}_2^+\) groups.\(^{28}\) It was noticed that the surface becomes negatively charged again after texturing by silica particles. These results are consistent with other reports.\(^{28–30}\)

For example, Yamada et al.\(^{28}\) found the zeta potential of bare SiO\(_2\) and APTES modified surfaces as negative (\(-70 \text{ mV}\)) and positive (\(+50 \text{ mV}\)), respectively at pH 6. Kamada et al.\(^{29}\) obtained zeta potential value of \(-25 \text{ mV}\) at pH 6 for 20 nm size SiO\(_2\) NPs. Feifel et al.\(^{30}\) found negative zeta potential (\(-41.44 \text{ mV}\)) for bare SiO\(_2\) NPs and positive (39.63 mV) upon APTES modification. For these results, the textured surface showed combined acid-base character due to the different dissociation constants of \(-\text{NH}_2^+\) and \(-\text{Si–OH}\) groups which results as modified isoelectric point\(^{31}\) (Effect of silanization/texturing on capacitance-voltage (C-V) plots section). One could infer enhanced adsorption of H\(^+\) and OH\(^-\) ions at lower and higher pH values, respectively provided by chemical and physical modifications, enhance the pH sensitivity of the EIS devices and this is consistent with the hypothesis (mixed surface functionality of APTES modified ZnS/Silica surface due to presence of \(-\text{NH}_2^+\) and \(-\text{Si–OH}\) groups) given by He et al.\(^{10}\) This inference is further supported by the flatband voltage shift observed in textured MOS devices discussed later in Capacitance and flatband voltage measurements section.

Electric and dielectric behavior of MOS devices.—Capacitance and flatband voltage measurements.—Electrical and dielectric characteristics of the textured samples were undertaken with MOS instead of EIS devices to obviate the effects of the reference electrode, the electrolyte and the interface. The C-V and G-V plots of MOS devices fabricated with planar, silanized and textured (with particles of diameter 475, 135, and 70 nm) SiO\(_2\) surface are shown in Figures 7 and 8, respectively. Silanization and texturization have a bearing on the accumulation capacitance and the flatband voltage. The negative flatband voltage shift observed in the MOS devices with silanized vis-à-vis the planar surface can be attributed to the presence of positively charged amine groups, formation of dipoles at the metal/APTES interface, and changes in the metal work function. This finding is in agreement with Park et al.\(^{32}\) who reported an increase in ruthenium (Ru) metal work function by 0.1 eV on APTES modification of oxide surface.
and a shift in the flatband voltage. Mokarana et al.\textsuperscript{33} studied the effect of APTES modification in vertical devices of self-assembled hybrid organic/inorganic monolayers on tungsten polyoxometalates (POMs) and observed decrease in the capacitance. Further, addition of APTES reduces the conductance and acts as an insulator. The amount of decrease in the capacitance depends on the dielectric constant and the thickness of the APTES layer. The changes observed here may be attributed to a lower dielectric constant as well as additional thickness (~1 nm) of silane.\textsuperscript{34} The increase in capacitance of the MOS device with the textured oxide surface vis-à-vis the silanized surface is due to the cumulative effect of increase in (i) surface area, (ii) effective oxide thickness, and (iii) charge storage capacity of the SiO$_2$ particles. The positive flatband shift suggests the covering of the amine groups by the negatively charged species created on the surface by texturing. Further increase in the capacitance and the flatband voltage observed with rise in particle size arises due to increased surface roughness and negative charge developed, respectively. G-V characteristics in all the cases show good conductance peaks in the depletion region near the negatively charged under basic conditions and lead to a shift in flatband voltage up to a saturation point at high pH values because of charge accumulation. On the other hand, the –NH$_2$ groups associated with the APTES layer are sensitive to H$^+$ ions causing depletion of charge carriers in acidic medium. The sensitivity in that case will be limited by extent of depletion. Thus, better pH sensing in basic as well as the acidic media arise with the combination of surface sites associated with APTES and silica particles following chemical and physical modification, respectively. Niu et al.\textsuperscript{31} reported that a ratio of 7/3 of silanol sites to amine sites on Si$_3$N$_4$ surfaces provided the maximum pH sensitivity. Therefore, one can conclude that the surface texturing with 70 nm silica particles provides the optimum ratio of –Si-OH and –NH$_2$ groups which showed the maximum sensitivity.

Interface states ($N_{ss}$).—Interface states (traps) present at the dielectric-semiconductor interface cause hysteresis in the C-V characteristics of the EIS devices and, in turn, influence the pH sensitivity.\textsuperscript{24} In order to study the effect of texturization, the density of the interface states ($N_{ss}$) at the oxide-semiconductor junction was obtained from C-V and G-V plots using the Hill-Coleman method.\textsuperscript{17} Table II lists the values of $N_{ss}$ of various MOS devices. A slight decrease in $N_{ss}$ observed after silanization can be attributed to the dipoles emerging at the metal-oxide interface because of the charged amine groups present. Upon texturing, the $N_{ss}$ value increases toward that of the planar MOS as silica particles suppress the effect of the amine sites. This indicates that the various chemical treatments during texturization did not affect the characteristics at the Si/SiO$_2$ interface.

Conclusions

EIS devices fabricated with textured oxide surfaces utilizing screen printed Ag/AgCl electrodes exhibit reduced drift and hysteresis effect with maximum pH sensitivity of 52.4 mV/pH. While silanization causes increase in C-V hysteresis due to pronounced adsorption of H$^+$ ions due to strong proton affinity of –NH$_2$ groups, attachment of SiO$_2$ particles leads to reduced C-V hysteresis associated with the covering of –NH$_2$ groups by the SiO$_2$ NPs to an intermediate level between planar and silanized EIS devices. Thus, the C-V hysteresis behavior indicates the combined acid-base character of the textured surfaces. The effect of texturing is to enhance the number of surface sites required for the chemical interactions of the oxide and the electrolyte. UV-Vis absorption and PL studies confirmed the enhanced surface sites with decreasing particle size without observing any confinement effect. The zeta potential measurements confirm the combined acid-base character of the textured surface with the formation of –NH$_3^+$ and –Si–O$^-$ groups. An optimum ratio of silanol and amine surface sites is essential for better pH sensing in both acidic and basic media. Therefore, the surface textured with 70 nm SiO$_2$ particles was thought to have provided the optimum ratio of the silanol and the amine surface groups. Interface states ($N_{ss}$) deduced for MOS devices suggested...
that texturing with silica particles did not affect Si/SiO2 interface, and confirms that the changes only occurred at electrolyte/dielectric interface.

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