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# Band alignment of SiO<sub>2</sub>/Si structure formed with nitric acid vapor below 500 $^\circ$ C

Kentarou Imamura<sup>a,b</sup>, Masao Takahashi<sup>a,c</sup>, Shigeki Imai<sup>b,c</sup>, Hikaru Kobayashi<sup>a,c,\*</sup>

<sup>a</sup> Institute of Scientific and Industrial Research, Osaka University, 8-1 Mihogaoka, Ibaraki, Osaka 567-0047, Japan

<sup>b</sup> Display Technology Development Group, Sharp Corporation, 2613-1, Ichinomoto-cho, Tenri, Nara 632-8567, Japan

<sup>c</sup> CREST, Japan Science and Technology Agency, Kawaguchi, Japan

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## ABSTRACT

A relatively thick (i.e.,  $\sim 9$  nm) SiO<sub>2</sub> layer can be formed by oxidation of Si with nitric acid (HNO<sub>3</sub>) vapor below 500 °C. In spite of the low temperature formation, the leakage current density flowing through the SiO<sub>2</sub> layer is considerably low, and it follows the Fowler–Nordheim mechanism. From the Fowler–Nordheim plots, the conduction band offset energy at the SiO<sub>2</sub>/Si interface is determined to be 2.57 and 2.21 eV for HNO<sub>3</sub> vapor oxidation at 500 and 350 °C, respectively. From X-ray photoelectron spectroscopy measurements, the valence band offset energy is estimated to be 4.80 and 4.48 eV, respectively, for 500 and 350 °C oxidation. The band-gap energy of the SiO<sub>2</sub> layer formed at 500 °C (8.39 eV) is 0.68 eV larger than that formed at 350 °C. The higher band-gap energy for 500 °C oxidation is mainly attributable to the higher atomic density of the SiO<sub>2</sub> layer of 2.46 × 10<sup>22</sup>/cm<sup>3</sup>. Another reason may be the absence of SiO<sub>2</sub> trap-states.

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## 1. Introduction

Development of low temperature formation methods of silicon dioxide (SiO<sub>2</sub>)/Si structure is of importance for improvement of electrical characteristics of semiconductor devices such as large scale integration (LSI) and thin film transistors (TFT). A gate oxide layer in LSI is generally produced by thermal oxidation of Si above 800 °C. The high temperature heating destroys shallow junction and possibly forms defect states [1,2]. A gate oxide layer in polycrystalline Si (poly-Si)-based TFT should be formed at low temperatures below 500 °C due to the use of glass substrates, and a plasma-enhanced chemical vapor deposition (CVD) method is usually employed for the formation of a gate oxide layer [3–5]. However, deposition methods cannot form a uniform thickness SiO<sub>2</sub> layer on rough poly-Si surfaces arising from laser annealing of amorphous Si thin films to crystallize [6]. Moreover, bulk and interfacial properties of the deposited SiO<sub>2</sub> laver are much worse than those formed by direct Si oxidation such as thermal oxidation. Due to these disadvantages, a thick gate oxide layer (i.e., 50-100 nm) is required in order to achieve a sufficiently good leakage current characteristic. The thick gate oxide layer increases the TFT operation

\* Corresponding author. Address: Institute of Scientific and Industrial Research, Osaka University, 8-1 Mihogaoka, Ibaraki, Osaka 567-0047, Japan. Tel./fax: +81 6 6879 8450.

E-mail address: h.kobayashi@sanken.osaka-u.ac.jp (H. Kobayashi).

voltage, leading to an increase in the power consumption, and moreover, it makes shrinkage of TFT difficult. In fact, submicron TFT has not been commercially produced so far.

Extensive studies on low temperature Si oxidation have been carried out using plasma oxidation [7–9], photo-oxidation [10,11], ozone oxidation [12,13], catalytic decomposition of oxygen [14], metal-promoted oxidation [15–17], etc. However, either thickness or quality of the SiO<sub>2</sub> layer formed by these methods is not sufficient for application to gate oxide layer in TFT.

We have recently developed a method of low temperature formation of an SiO<sub>2</sub> layer by use of nitric acid (HNO<sub>3</sub>) [18–23]. The ultrathin SiO<sub>2</sub> layer (i.e., ~1.3 nm) formed by immersion in azeotropic mixture of HNO<sub>3</sub> with water (i.e., 68 wt% HNO<sub>3</sub>) possesses a leakage current density (e.g., 0.4 A/cm<sup>2</sup> at the forward gate bias of 1 V) much lower than that of a thermal oxide layer with the same thickness [18–21]. The thick SiO<sub>2</sub> layer can be formed by using HNO<sub>3</sub> solutions with two different concentrations, i.e., initial immersion in ~40 wt% HNO<sub>3</sub> followed by that in 68 wt% HNO<sub>3</sub> [22,23]. We have also shown that a relatively thick  $SiO_2$  layer can be formed by oxidation of Si with HNO<sub>3</sub> vapor at temperatures below 500 °C [24]. In the present study, physical properties of SiO<sub>2</sub>/Si structure fabricated with HNO<sub>3</sub> vapor have been investigated using X-ray photoelectron spectroscopy (XPS), Fourier-transform infrared absorption spectroscopy (FT-IR), and current-voltage (I-V)measurements, and the band diagrams of the SiO<sub>2</sub>/Si structure have been obtained.



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# 2. Experiments

Phosphorus-doped n-type Si(100) wafers with a  $5-10 \Omega$  cm resistivity were cleaned using the RCA method [25] and etched with a dilute hydrofluoric acid (DHF) solution. Then, the Si specimens were inserted in a furnace and heated at temperatures in the range between 300 and 500 °C. HNO<sub>3</sub> solutions with the azeotropic concentration (i.e., 68 wt%) were heated at the boiling temperature of 120 °C and the vapor was introduced to the furnace. It should be noted that the HNO<sub>3</sub> concentration in the vapor phase was also 68 wt%, which was characteristic of azeotropic mixture [19]. The metal impurity concentrations of the HNO<sub>3</sub> aqueous solutions were less than 1 ppb. After oxidation with HNO<sub>3</sub> vapor, the specimens were rinsed with ultra-pure water for 5 min at room temperature. For some specimens, aluminum (Al) dots of 0.15 mm diameter were formed by the thermal evaporation method in order to measure current-voltage (I-V) characteristics using an HP 4140B picoammeter.

XPS measurements were performed using a VG Scientific Escalab 220i-XL spectrometer with a monochromatic Al K $\alpha$  radiation source. Photoelectrons were collected in the surface-normal direction. FT-IR measurements were carried out using a Nicolet Nexus 370S FT-IR spectrometer with the incident angle of 65°.

## 3. Results and discussion

Fig. 1a shows XPS spectra in the Si 2p region for the SiO<sub>2</sub>/ Si(100) structure formed with HNO<sub>3</sub> vapor at 500 °C. Doublet peaks were due to Si  $2p_{3/2}$  and  $2p_{1/2}$  levels of the Si substrate and a broad peak in the higher binding energy region was due to the SiO<sub>2</sub> layer. The thickness of the SiO<sub>2</sub> layer was determined to be 8.8 nm from the intensity ratio between the SiO<sub>2</sub> peak and the substrate peaks [26,27], using 3.2 nm as the mean free path of photoelectrons in SiO<sub>2</sub>. The estimated SiO<sub>2</sub> thickness was nearly the same as that determined from ellipsometry. It should be noted that thermal oxidation of Si at 500 °C can form an SiO<sub>2</sub> layer with the thickness only up to ~2 nm even by long-term oxidation [28]. This result clearly shows that HNO<sub>3</sub> molecules possess a much higher oxidizing activity than oxygen molecules. The possible oxidizing species is oxygen atoms generated at the SiO<sub>2</sub> surface by decomposition of HNO<sub>3</sub> molecules:

$$2HNO_3 \rightarrow NO_2 + NO + H_2O + 2O \tag{1}$$

In the case of HNO<sub>3</sub> vapor oxidation, diffusion of oxygen atoms through SiO<sub>2</sub> is the rate-determining step, evident from a parabolic relation between the SiO<sub>2</sub> thickness and the oxidation time [24]. According to previous literature [29–31], reactions of Si or SiO<sub>2</sub> with gaseous NO<sub>2</sub> or NO to form silicon oxynitride films proceed only at high temperatures above 1000 °C. In the present study, no N 1s peak was observed after HNO<sub>3</sub> vapor oxidation, indicating that NO<sub>2</sub> and NO produced by decomposition of HNO<sub>3</sub> did not react with Si at temperatures below 500 °C.

The metal impurity concentrations in the SiO<sub>2</sub> layer formed with HNO<sub>3</sub> vapor were less than  $1 \times 10^{10}$  atoms/cm<sup>2</sup>, which was confirmed by measurements of total reflection X-ray fluorescence spectra. Avoidance of metal contaminations probably resulted from high purity HNO<sub>3</sub> solutions (metal contamination level: <1 ppb).

Fig. 1b shows the FT-IR spectrum for the SiO<sub>2</sub>/Si structures formed in HNO<sub>3</sub> vapor at 500 °C. The peaks at ~1050 and ~1250 cm<sup>-1</sup> are attributable to transverse optical (TO) and longitudinal optical (LO) phonons of asymmetric Si–O–Si stretching vibration, respectively [32–35]. Using the central and non-central force model, the vibrational frequencies of TO and LO phonons,  $v_{TO}$  and  $v_{LO}$ , are given by [32]



Fig. 1. XPS spectrum in the Si 2p region (a) and FT-IR spectrum (b) for the SiO<sub>2</sub>/Si(100) structure formed in HNO<sub>3</sub> vapor at 500  $^{\circ}$ C.

$$v_{\rm TO} = 2\pi \left[ \frac{2}{m_0} \left\{ \alpha \sin^2 \frac{\theta}{2} + \beta \cos^2 \frac{\theta}{2} \right\} \right]^{1/2}$$
(2)

$$v_{\rm LO} = 2\pi \left[ \frac{2}{m_0} \left\{ \alpha \sin^2 \frac{\theta}{2} + \beta \cos^2 \frac{\theta}{2} + \gamma^{\rm ss} \right\} \right]^{1/2} \tag{3}$$

where  $m_0$  is the mass of an oxygen atom,  $\theta$  is the Si–O–Si bond angle, and  $\alpha$  and  $\beta$  are the central and non-central force constants, respectively.  $\gamma^{ss}$  is defined as

$$\gamma^{\rm ss} = \frac{\left(Z_{\rm OS}^{\rm T}\right)^2}{\varepsilon_{\rm ox}(2m_{\rm O} + M)\rho} \tag{4}$$

where *M* is the mass of a Si atom,  $\rho$  is the density of the SiO<sub>2</sub> layer,  $Z_{OS}^{T}$  is the charge related to vibration of an oxygen atom, and  $\varepsilon_{ox}$  is the dielectric constant of SiO<sub>2</sub>. From Eqs. (2)–(4), we have

$$\rho = \mathcal{C}(v_{\rm LO}^2 - v_{\rm TO}^2) \tag{5}$$

where C is a constant. Using the values for a thick thermal SiO<sub>2</sub> layer (i.e.,  $\rho = 2.28 \times 10^{22}$  atoms/cm<sup>3</sup>,  $v_{TO} = 1090$  cm<sup>-1</sup>,  $v_{LO} = 1256$  cm<sup>-1</sup> [36]), and assuming that the observed  $v_{TO}$  was shifted by 5 cm<sup>-1</sup> due to the geometrical effect [36], C is determined to be  $5.85 \times 10^{16}$  atoms/cm. Using this value, the atomic density of the SiO<sub>2</sub> layer formed in HNO<sub>3</sub> vapor at 500 °C is estimated to be  $2.46 \times 10^{22}$  atoms/cm<sup>3</sup>.

Fig. 2 shows the valence band spectra for the SiO<sub>2</sub>/Si structure. The component due to the Si substrate (spectrum d) was subtracted from the measured spectra for the SiO<sub>2</sub>/Si structure. The Si valence band maximum was determined by the interception of the extrapolated line (dotted line in spectrum d) with the energy axis. The SiO<sub>2</sub> valence band maximum was also determined by the interception of the extrapolated line of the subtracted spectrum (dashed line in spectra a–c). Therefore, the valence band off-set energies could simply be determined by the difference between the Si and SiO<sub>2</sub> valence band maxima. The reference energy, E = 0, was determined by the apparatus Fermi level. The valence band offset energies at the SiO<sub>2</sub>/Si interfaces were estimated to be 4.48 eV for HNO<sub>3</sub> oxidation at 500 °C (spectrum b), and 4.81 eV for thermal oxidation at 900 °C (spectrum c).

Fig. 3 shows the *I–V* curves in the forward bias region for the  $\langle Al/SiO_2/Si(100) \rangle$  MOS diodes. The SiO<sub>2</sub> thickness is 8.8 nm both for HNO<sub>3</sub> oxidation at 350 °C (curve a) and 500 °C (curve b), and 8.5 nm for thermal oxidation at 900 °C (curve c). The leakage cur-



**Fig. 2.** Valence band spectra for the SiO<sub>2</sub>/Si(100) structure formed with the following conditions: (a) in HNO<sub>3</sub> vapor at 350 °C; (b) in HNO<sub>3</sub> vapor at 500 °C; and (c) in oxygen at 900 °C. The valence band spectrum for the Si substrate is shown for reference (d).



**Fig. 3.** *I*–V curves for the  $\langle AI/SiO_2/Si(100) \rangle$  MOS diodes with the SiO<sub>2</sub> layer formed with the following conditions: (a) in HNO<sub>3</sub> vapor at 350 °C; (b) in HNO<sub>3</sub> vapor at 500 °C; and (c) in oxygen at 900 °C.

rent density for curve a was high in the bias region below  $\sim 5$  V. For curve a, the semi-logarithmic plot of the current density divided by the electrical field vs. the square root of the electrical field, i.e.,  $\ln(J/E_{ox})$  vs.  $\sqrt{E_{ox}}$ , in the low bias region was linear, indicating that the leakage current flowed with the Poole–Frenkel mechanism [37]:

$$J_{\rm PF} = \sigma(0) E_{\rm ox} \exp\left[\frac{-q\left(\phi_{\rm t} - n\sqrt{qE_{\rm ox}/\pi\varepsilon_{\rm ox}}\right)}{kT}\right]$$
(6)

where  $\sigma(0)$  is the electrical conductivity at low electrical field,  $E_{\text{ox}}$ ,  $\varepsilon_{\text{ox}}$  is the permittivity of SiO<sub>2</sub>, and  $\phi_{\text{t}}$  is the trap energy with respect to the SiO<sub>2</sub> conduction band. The value of 3.9 was used for the dielectric constant of the SiO<sub>2</sub> layer employed in the present study. (The trap energy,  $\phi_{\text{t}}$ , is determined to be 0.57 eV from the intercept of the plot.)

The *I*–*V* curves of the SiO<sub>2</sub> layer formed in HNO<sub>3</sub> at 500 °C and in oxygen at 900 °C do not follow the Poole–Frenkel mechanism but follows the Fowler–Nordheim mechanism, indicating the absence of trap-states. The *I*–*V* curves in the bias region between 5.7 and 6.5 V for 350 °C HNO<sub>3</sub> vapor oxidation also followed the Fowler–Nordheim mechanism [34]:

$$\frac{J_{\rm FN}}{E_{\rm ox}^2} = A \cdot \exp\left(-\frac{B}{E_{\rm ox}}\right) \tag{7}$$

$$A = \frac{q^2 m}{16\pi \hbar m^* \phi_{\rm b}} = 1.55 \times 10^{-6} \frac{m^*}{m} \frac{1}{\phi_{\rm b}}$$
(8)

$$B = \frac{4\sqrt{2m^*q}\phi_b^{3/2}}{3\hbar} = 6.83 \times 10^7 \left(\frac{m^*}{m}\right)^{1/2} \phi_b^{3/2} \tag{9}$$

where  $m^*$  is the effective mass of an electron in the Si conduction band, m is the static electron mass, and  $\phi_b$  is the barrier height at the SiO<sub>2</sub>/Si interface. In the present study, 0.5 was used for  $m^*/m$ . For the Fowler–Nordheim mechanism, the plots of  $\ln (J_{FN}/E_{ax}^2)$  vs.  $1/E_{ox}$  become linear as shown in Fig. 4. From the slopes of the plots, the barrier height at the SiO<sub>2</sub>/Si interfaces is estimated to be 2.40 eV for 350 °C HNO<sub>3</sub> vapor oxidation, 2.76 eV for 500 °C HNO<sub>3</sub> vapor oxidation, and 3.27 eV for 900 °C thermal oxidation. From the Si resistivity of ~7  $\Omega$  cm, the Si Fermi level is calculated to be located at 0.27 eV below the Si conduction band minimum. Using these val-



Fig. 4. Fowler–Nordheim plots for the  $\langle Al/SiO_2/Si(100)\rangle$  MOS diodes with the  $SiO_2$ layer formed with the following conditions: (a) in HNO3 vapor at 350 °C; (b) in HNO3 vapor at 500 °C; and (c) in oxygen at 900 °C.

ues, the conduction band offset energy at the SiO<sub>2</sub>/Si interfaces can be determined as described below.

Fig. 5 shows the band diagrams of the SiO<sub>2</sub>/Si structure. The conduction band (or valence band) offset energies are determined to be 2.13 eV (4.48 eV) for 350 °C HNO<sub>3</sub> vapor oxidation (Fig. 5a), 2.49 eV (or 4.80 eV) for 500 °C HNO<sub>3</sub> vapor oxidation (Fig. 5b), and 3.00 eV (or 4.81 eV) for 900 °C thermal oxidation (Fig. 5c). (The conduction band off-set energies are estimated using the barrier height obtained from the Fowler-Nordheim plots minus the energy difference between the bulk Si conduction band and the Si Fermi level.) Therefore, the SiO<sub>2</sub> band-gap energies are estimated to be 7.71 eV for 350 °C HNO<sub>3</sub> vapor oxidation, 8.39 eV for 500 °C HNO<sub>3</sub> vapor oxidation, and 8.91 eV for 900 °C thermal oxidation. The estimation method of the conduction band offset energy from the Fowler-Nordheim plots (i.e., indirect method in contrast to the valence band offset energy estimation method using photoelectron spectroscopy) may give lower values than the real values, leading to underestimation of the band-gap values. We think that these variations of the band-gap energies mainly arise from the difference in the SiO<sub>2</sub> atomic density. The high atomic density results in high interaction between atoms in SiO<sub>2</sub>, and the high interaction



Fig. 5. Band diagrams of the SiO<sub>2</sub>/Si structures with the SiO<sub>2</sub> layer formed with the following conditions: (a) in HNO3 vapor at 350 °C; (b) in HNO3 vapor at 500 °C; and (c) in oxygen at 900 °C

increases the energy difference between the bonding states (i.e., valence band) and anti-bonding states (i.e., conduction band). The distinct difference in the electronic structures between the oxides formed at 350 and 500 °C is mainly due to the variation of the SiO<sub>2</sub> atomic density. Another reason may be the presence and absence of trap-states in SiO<sub>2</sub> formed at 350 and 500 °C, respectively. Namely, for the former SiO<sub>2</sub> layer, the Fowler-Nordheim tunneling may occur from the Si conduction band to the SiO<sub>2</sub> trap-states, leading to underestimation of the conduction band off-set energy.

The SiO<sub>2</sub> layer formed with HNO<sub>3</sub> vapor contains high concentration OH species [20]. OH species disturbs SiO<sub>2</sub> network formation, probably resulting in a decrease in the band-gap energy. For this reason, the SiO<sub>2</sub> layer formed with HNO<sub>3</sub> vapor at 500 °C possesses a band-gap energy smaller than the thermal oxide layer in spite of the higher atomic density. (The atomic densities are higher in the order of (b) > (c) > (a).)

In the present study, the effective mass for all the SiO<sub>2</sub> layers is assumed to be identical in spite of the different electronic structures. In the present paper, we use  $m^*/m = 0.5$ , and the barrier heights are estimated to be 2.40 and 2.76 eV, respectively, for HNO<sub>3</sub> vapor oxidation at 350 and 500 °C. When  $m^*/m$  is assumed to be unity, the barrier heights are calculated to be 1.90 and 2.19 eV, respectively, for 350 and 500 °C oxidation.

### 4. Conclusions

The SiO<sub>2</sub> layer with  $\sim$ 9 nm thickness is formed in HNO<sub>3</sub> vapor at temperatures lower than 500 °C, and the valence and conduction band offset energies are determined from measurements of valence band spectra and the Fowler-Nordheim plots, respectively. The band-gap energies of the SiO<sub>2</sub> layer formed at 500 and 350 °C are estimated to be 8.39 and 7.71 eV, respectively. The difference in the band-gap energy is attributable to (i) presence and absence of trap-states in the SiO<sub>2</sub> laver formed at 350 and 500 °C. respectively, and (ii) various SiO<sub>2</sub> atomic density.

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