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Frequency dependence studies on the interface trap density and series resistance of HfO_2 gate dielectric deposited on Si substrate: Before and after 50 MeV Li³⁺ ions irradiation

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1. Introduction

ABSTRACT

We report the first investigation of the frequency dependent effect of 50 MeV Li³⁺ ion irradiation on the series resistance and interface state density determined from capacitance–voltage (*C–V*) and conductance–voltage (*G–V*) characteristics in HfO₂ based MOS capacitors prepared by rf-sputtering. The samples were irradiated by 50 MeV Li³⁺ ions at room temperature. The measured capacitance and conductance were corrected for series resistance. The series resistance was estimated at various frequencies from 1 KHz to 1 MHz before and after irradiation. It was observed that the series resistance decreases from 6344.5 to 322 Ω as a function of frequency before irradiation and 8954–134 Ω after irradiation. The interface state density D_{it} decreases from 1.12 × 10¹² eV⁻¹ cm⁻² before irradiation to 3.67 × 10¹¹ eV⁻¹ cm⁻² after ion irradiation and further decreases with increasing frequency.

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Continuous scaling of gate dielectric thickness is leading to the intolerable tunneling gate leakage and power consumption, Hfbased high-k dielectric has been recognized as the most promising candidate for future advanced gate stacks in sub-45 nm node technologies due to its high dielectric constant (\sim 10), relatively wide band-gap (~5.6 eV), sufficient band offset (>1.4 eV), and thermal compatibility with Si-based processing [1-5]. Qualification of high-k devices for advanced CMOS devices and space applications may need more understanding of the charge trapping characteristics and long-term reliability of these materials [6-9]. From the literature, very little is known about the radiation hardness of these new high-k systems. The high-k systems are likely to be the heart of advanced MOS integrated circuits, particularly low power system, in the fairly near future. Therefore, the high-k system could become particularly important in radiation hard system utilized in space application.

For devices used in space systems, radiation exposure is another reliability problem. Despite large amount of ongoing research into

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alternative dielectric materials, very little work has been done to understand the radiation responses of these materials [6-9,19]. Electronic devices in space are exposed to various forms of radiation, such as electrons, protons, neutrons, and heavy ions [10]. Recent results have shown that lithium ions are one of the suitable sources for testing radiation hardness of devices for reasons - (i) lithium along with silicon and oxygen ions contribute a major percentage among the heavy ions found in space. (ii) Being triply charged, lithium ions can be accelerated to very high energies compared to protons. (iii) Lithium ions with comparatively higher mass and greater range can deliver more energy to the target atoms forming ion tracks along the oxide and the bulk [9,11–18]. These properties of lithium ions make it a promising source of radiation for radiation hardness testing of semiconductor devices. A lot of work has been done by the radiation effects community to investigate changes in Si-SiO₂ MOS-structures. With regard to the high-k systems, very little information is available on interaction of lithium ion with HfO₂-Si MOS structures [9]. With present day VLSI technology with HfO₂ as gate oxide it becomes very necessary to study the lithium interaction on these devices for reliability in radiation harsh environments and space applications.

In this paper, we report a systematic investigation of frequency dependent effect of 50 MeV Li^{3+} ions on series resistance and interface state density of HfO_2 based MOS-capacitor from C-V and G-V

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characteristics. The electrical properties of MOS devices were investigated for a range of frequencies from 1 KHz to 1 MHz at room temperature before and after Li^{3+} ion irradiation. The existence of an interfacial layer between the oxide and the semiconductor play an important role in the determination of the series resistance and interface state density [20,21]. The *C*-*V* and *G*-*V* measurements give the important information about the density or energy distribution of the interface states of the structure.

2. Experiment

HfO₂ based MOS-capacitors were fabricated on a p-type 1-10 Ohm cm resistivity and (100) orientation silicon substrate. High purity HfO₂ (99.9% purity) 5-inch sputtering target supplied by M/s semiconductor technology was used to deposit the thin films in MRC rf-sputtering system. The wafers were cleaned by using standard cleaning procedure for removing organic and inorganic contaminations followed by interface oxide etched in dilute HF (1:20), rinsed in DI water and dried in dry N₂ immediately before loading in the vacuum chamber. The vacuum chamber was evacuated to the background pressure of 1.2×10^{-6} Torr. The sputtering was done in high purity argon ambient gas and the gas pressure was maintained at 6 m Torr. Thin films of HfO₂ were deposited on silicon substrate at sputtering voltage 0.8 kV for 5 min keeping film thickness 15-20 nm corresponding to equivalent oxide thickness of SiO₂ about 3.5 nm. The asdeposited films were thermally annealed at 700 °C in nitrogen ambient for 30 min. Deposition of high-k metal oxides on silicon substrates is always associated with an unintentional Interfacial Layer between high-k layer and silicon which is almost inevitable. This SiO_x interfacial layer which is due to the strong affinity of silicon to oxygen is less than 1 nm in general. These interfacial layers produce undesirable effects which can be minimized by annealing in nitrogen ambient. In our previous work [4,5], we have reported the effect of oxygen and nitrogen annealing on HfO₂ films for MOS structures where it is clearly shown that annealing in nitrogen ambient improves the performance of the devices by providing good interface between the HfO₂ and Silicon substrate. The nitrogen annealing provides a good oxygen diffusion barrier suppressing interfacial oxidation. Nitrogen annealing also improves the thermal stability of the devices. The dielectric constant (k) of the sputtered films was estimated to be \sim 18. The details are reported in our earlier publication [5]. The sputtered AlSi thin film about 700 nm, deposited on both sides of the wafer was used as top electrode and back contact. The metal film was patterned using photolithography and metal etching. The minimum contact area was $50\times 10^{-4}\,\text{cm}^2$. The MOS structures were finally annealed in forming gas at 450 °C. The film thickness was measured using Ambios step profiler and the microstructure was examined by Nanoscope II atomic force microscope in the contact mode. After this, these MOS-capacitors were irradiated by Li³⁺ ions. Samples were mounted on metallic holder. The metallic holder was then placed in a general-purpose scattering chamber at a vacuum of 10^{-6} torr. The samples were exposed with 50 MeV Li^{3+} lons for fluence $1 \times 10^{11} \text{Li}^{3+}/\text{cm}^2$ at Nuclear Science Center (Inter University Acceleration Center (IUAC)), New Delhi. The ions were beamed to fall on the front side of the device exactly at right angles. The scanning area was fixed at 1×1 cm² with magnetic scanner to obtain uniform fluence over the devices. The beam current was 1 nA. SRIM (Stopping and Range of Ions in Matter) simulations have been performed in order to investigate the energy loss of such ions in silicon. SRIM accounts for the non-ionizing energy loss of the impinging particles due to the Coulomb scattering. The range of the Li-ions in silicon is 310.24 µm, larger than the 300 micron thickness of the devices considered in this study. This ensures that Li ions which are n-type doping impurities, are not implanted in the device region of silicon substrate. In addition the highly damaged region generated at depths close to the ion range, where the non-ionizing energy loss is maximum, lies outside the silicon substrate. The nuclear energy loss $\langle dE/dx \rangle n$, the electronic energy loss $\langle dE/dx \rangle e$ of 50 MeV Li³⁺ ions in HfO₂ was calculated using SRIM 2008 and was found to be 1.514×10^{-4} and 2.734×10^{-1} , respectively [22]. The electrical characterization of the devices before and after irradiation was performed using Keithley 4200-SCS integrated system analyzer and shielding probe station.

3. Results and discussions

The use *C*–*V* characteristics of MOS capacitors provides a very sensitive tool to measure changes in the dielectrics, interfaces (gate electrode/dielectric interface and dielectric/substrate interface) and the bulk of silicon. Fig.1(a and b) presents the measured capacitance as a function of gate voltage before and after 50 MeV Li³⁺ ion irradiation for AlSi/HfO₂/*n*-Si MOS capacitors observed at 1, 10, 100, 500 KHz and 1 MHz at room temperature. From these figures, the three distinct regimes of *C*–*V* curve: accumulation, depletion and inversion before and after Li³⁺ ion irradiation are shown. The observed curves showed irradiation dispersion in accumulation and depletion region. Any change in the metal/oxide interface layer due to the radiation leads to changes in the gate capacitance, gate conductance and flat-band of the device. Similarly, Fig. 2(a and b) presents the measured conductance as a function of gate voltage before and after 50 MeV Li³⁺ ion irradiation. Since the values of



Fig. 1. The capacitance (C_m) at room temperature before and after 50 MeV Li³⁺ ions irradiation for AlSi/HfO₂/*n*-Si MOS capacitors measured at (a) 1, 10, 100 KHz and (b) 500 KHz, 1 MHz.



Fig. 2. The measured conductance (G_m) versus gate bias (V) at room temperature before and after 50 MeV Li³⁺ ions irradiation for AlSi/HfO₂/*n*-Si MOS capacitors prepared at (a) 1, 10, 100 KHz and (b) 500 KHz, 1 MHz.

the capacitance and conductance depends on a number of parameters such as the thickness and formation of the oxide layer, series resistance and density of interface states, the explanation of this behavior of frequency dependant C-V and G-V characteristics queries whether the interface states contribute to the MOS capacitance and conductance or the charge at interface states are just following an alternating current signal. From Fig. 1(a and b), the decreasing accumulation capacitance with increase in frequency shows the frequency dispersion which indicates the presence of frequency dependent interface states. From the above discussion it can be concluded that under bias condition the interface states are responsible for the observed frequency dispersion in the C-V and G-V curves [23].

Fig. 2(a and b) shows the radiation induced changes in the measured conductance as function of gate voltage before and after 50 MeV Li³⁺ ion irradiation measured at frequencies ranging from 1 KHz to 1 MHz at room temperature. It is observed from these figures that the absence of a peak in the measured *G*–*V* characteristics indicates that series resistance is responsible for the dominant loss, completely masking the interface trap loss as observed in the *G*–*V* plot. Therefore series resistance is an important parameter which causes the major error during analyzing the accumulation region and a portion of the depletion region [23,24]. Series resistance (*R*_s) also causes a serious error in the extraction of interfacial prop-



Fig. 3. The series resistance (R_S) versus gate bias (V) at room temperature before and after 50 MeV Li³⁺ ions irradiation for AlSi/HfO₂/*n*-Si MOS capacitors measured at frequencies 1, 10, 100 and 500 KHz, 1 MHz.



Fig. 4. The dielectrics loss versus gate bias (V) at room temperature before and after 50 MeV Li³⁺ ions irradiation for AlSi/HfO₂/*n*-Si MOS capacitors measured at frequencies 10, 100 and 500 KHz, 1 MHz.

erties from the C-V and G-V measurements. The error can be minimized by measuring the series resistance and applying a correction to the measured capacitance and conductance values before the desired information is extracted. At a given frequency, most of the errors occur in the measured admittance in strong accumulation. The value of R_s can be found using the following equation:

$$R_{\rm s} = \frac{G_{\rm m,acc}}{G_{\rm m,acc}^2 + (\omega C_{\rm m,acc})^2} \tag{1}$$

where $C_{m,acc}$ and $G_{m,acc}$ are the measured capacitance and conductance in strong accumulation. Fig. 3, shows the changes in series resistance (R_s) of the HfO₂-based MOS-CAP devices before and after irradiation. The voltage dependence of the series resistance R_s was calculated from Eq. (1) as a function of gate voltage before and after Li³⁺ ion irradiation for frequencies ranging from 1 KHz to 1 MHz. As shown in Fig. 3, the series resistance is found to decrease



Fig. 5. The corrected capacitance (C_c) versus gate bias (V) at room temperature before and after 50 MeV Li³⁺ ions irradiation for AlSi/HfO₂/*n*-Si MOS capacitors measured at various frequencies: (a) 1, 10, 100 KHz and (b) 500 KHz and 1 MHz.

with increase in frequency. The series resistance values of HfO₂ based MOS structure calculated at strong accumulation region at 1, 10, 100, 500 KHz, 1 MHz are found to be 6344.5, 3254.4, 970, 911.4, 322 Ohm, respectively for our sample before irradiation. The series resistance versus frequency curves before and after irradiation was compared and plotted in Fig. 7 and data are compiled given in Table 1. The presence of the series resistance can also cause the variation in oxide capacitance. Fig. 4 shows the radiation induced changes in dissipation factor as function of gate bias voltage before and after 50 MeV Li³⁺ ion irradiation measured at frequencies from 1 KHz to 1 MHz at room temperature. The frequency dependence of the dissipation factor seems to support the resistance assumption. The dominant contribution to the loss arises from the transmission of majority carriers to and from interface states due the capture and emission of carriers by the interface states.

The corrected capacitance C_c and equivalent parallel conductance G_c for series resistance were evaluated from the relations [23]:

$$C_{\rm c} = \frac{(G_{\rm m} + \omega^2 C_{\rm m}^2)^2 C_{\rm m}}{a^2 + \omega^2 C_{\rm m}^2}$$
(2)

$$G_{\rm c} = \frac{(G_{\rm m} + \omega^2 C_{\rm m}^2)^2 a}{a^2 + \omega^2 C_{\rm m}^2}$$
(3)



Fig. 6. The calculated corrected conductance (G_c) as a function of gate bias voltage (V) before and after 50 MeV Li³⁺ ions irradiation for AlSi/HfO₂/*n*-Si MOS capacitors for various frequencies: (a) 1, 10 KHz, and (b) 100, 500 KHz and 1 MHz.



Fig. 7. Calculated series resistance and interface state density versus frequency of HfO_2 based MOS capacitor before and after Li-ion irradiation.

$$a = G_{\rm m} - (G_{\rm m} + \omega^2 C_{\rm m}^2)^2 R_{\rm s} \tag{4}$$

where C_m and G_m are the measured capacitance conductance.

Fig. 5(a and b) shows the frequency dispersion in the *C*–*V* characteristics of MOS capacitor structure after series resistance

Table 1

The values of series resistance and interface trap density for HfO_2 MOS-CAP determined from corrected *C*-*V* and *G*-*V* characteristics at five different frequencies before and after 50 MeV Li³⁺ ion.

Frequency	Before irradiation		After irradiation	
	R _s (Ohm)	$D_{\rm it}~({\rm eV}^{-1}~{\rm cm}^{-2})$	R _s (Ohm)	$D_{\rm it}~({\rm eV^{-1}~cm^{-2}})$
1 KHz 10 KHz 100 KHz 500 KHz	6344.5 3254.4 970 911.4	$\begin{array}{c} 4.6\times 10^{12}\\ 3.2\times 10^{12}\\ 2.36\times 10^{12}\\ 1.59\times 10^{12} \end{array}$	8954.73 2754.58 627.81 185.68	$\begin{array}{c} 6.609 \times 10^{11} \\ 5.29 \times 10^{11} \\ 4.07 \times 10^{11} \\ 3.81 \times 10^{11} \end{array}$

correction. The frequency dispersion at accumulation is mainly due to the presence of interface traps at the semiconductor insulator contact region. The capacitance of such a layer acts in series with the insulator capacitance causing frequency dispersion. However, negligible frequency dispersion is observed in the inversion region.

Fig. 6(a and b) shows the corrected conductance G_c characteristics as function of gate voltage before and after 50 MeV Li³⁺ ion irradiation for frequencies 1, 10, 100, 500 KHz and 1 MHz. After correction for series resistance, the conductance peaks are seen in the Fig. 6(a and b) corresponding to the depletion regions of the device. The value of interface trap density (D_{it}) is determined from this peak value. Single-frequency approximation method was used for estimation of the density of interface states from the *G–V*-measurements. Hill–Coleman method is fast and reliable method to determine the density of interface states (D_{it}) [25]. According to this method, D_{it} can be calculated using the following formula:

$$D_{\rm it} = \frac{2}{qA} \frac{G_{\rm c,max}/\omega}{\left[(G_{\rm c,max}/C\omega C_{\rm ox})^2 + (1 - C_{\rm c}/C_{\rm ox})^2 \right]}$$
(5)

where *A* is the area of the diode, ω is the angular frequency, *q* is the elementary electrical charge, $G_{c,max}$ conforms to maximum corrected *G*–*V* curve, C_c is capacitance of the diodes corresponding to $G_{c,max}$, C_{ox} is the capacitance of oxide layer in accumulation region of C_c –*V* curves. The C_{ox} was calculated through relation [24]:

$$C_{\rm ox} = C_{\rm c,acc} \left[1 + \left(\frac{G_{\rm c,acc}}{\omega C_{\rm c,acc}} \right)^2 \right] \tag{6}$$

where $C_{c,acc}$ and $G_{c,acc}$ are the corrected capacitance and conductance in accumulation region.

Hill–Coleman equation for the extraction of interface trap density was applied on *C*–*V* and *G*–*V* curves before and after irradiation for 1, 10, 100, 500 KHz and 1 MHz. The generation of interface states from defects such as dangling bonds at the insulator-substrate interface and the energy states in the silicon band gap is dependent on the chemical composition of the interface. The D_{it} , calculated using the single frequency approximation method (Hills method for different) to study the electrical properties of interface are plotted as a function of frequency before and after Li ion irradiation as shown in Fig. 7 and also summarized in Table 1. It can be observed that the interface traps density decreases when the frequency increases from 1 KHz to 1 MHz before and after Li ion irradiation.

The reaction at metal gate/HfO₂ interface contributes to the changes in EOT (Equivalent Oxide Thickness) which directly reflects on the accumulation capacitance in turn giving rise several other changes in the electrical properties of the MOS structures. Hence a change in either flat-band (V_{FB}) or oxide capacitance (C_{OX}) would also indicate the characteristic changes in a reactive layer at the gate dielectric interface. Additionally, the effect of the irradiation on interface layers at the metal/dielectric and dielectric/substrate interface, subsequently affects the gate conduction. Since the range of Li ion in AlSi/HfO₂/silicon system is

calculated to be 310.24 μ m, the damage induced by high energy Li³⁺ ions were estimated to be distributed in more deep regions at the silicon/dielectric interface as compared to metal/dielectric interface.

4. Conclusion

The frequency dependent effect of 50 MeV Li³⁺ ion irradiation on the series resistance and interface state density determined from capacitance-voltage (C-V) and conductance-voltage (G-V) characteristics in HfO₂ based MOS capacitors prepared by rf-sputtering are discussed. The forward and reverse bias capacitance-voltage (C-V) and conductance-voltage (G-V) characteristics of HfO₂ based MOS capacitor prepared by rf-sputtering were measured at 1, 10, 100, 500 KHz and 1 MHz frequencies before and after 50 MeV Li³⁺ ion irradiation. The effects of changes in the series resistance (R_s) and interface traps density (D_{it}) of HfO₂ based MOS capacitor on C-V and G-V characteristics before and after irradiation dose are investigated. The series resistance values of HfO2 based MOS structure calculated before irradiation at strong accumulation region at frequencies 1, 10KHz, 50, 100, 500 KHz, 1 MHz are found to be 6344.5, 3254.4, 970, 911.4, 322 Ohm, respectively and after irradiation vary as 8954.73, 2754.58, 627.81, 185.68 and 138.37 Ohm at 1, 10, 100, 500 KHz and 1 MHz, respectively and it found to decrease as a function of frequency. Similar effect has been observed after irradiation. The presence of the series resistance could also cause the variation in oxide capacitance. The interface state density values of our sample before Li ion irradiation vary as 4.6×10^{12} , 3.2×10^{12} , 2.36×10^{12} , 1.59×10^{12} , and 1.12×10^{12} , at 1, 10, 100, 500 KHz and 1 MHz, respectively and after irradiation vary as 6.609×10^{11} , 5.29×10^{11} , 4.07×10^{11} , 3.81×10^{11} and 3.67×10^{11} eV⁻¹ cm⁻² at 1, 10, 100, 500 KHz and 1 MHz respectively. It is also found to decrease as a function of frequency before and after irradiation. The higher values of capacitance resulting from the D_{it}, are in equilibrium with the semiconductor that can follow the ac signal. It is shown that the performance of HfO₂ based MOS-capacitors are improved after the 50 MeV Li³⁺ ion irradiation.

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