Hydrogen passivation for reduction of SiO₂/Si interface state density using hydrocarbon-molecular-ion-implanted silicon wafers

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The reduction in the density of SiO₂/Si interface state (Dₓ) in the isolation region and transfer transistor gate oxide is necessary to improve the performance of complementary metal-oxide semiconductor (CMOS) image sensors. In this study, we demonstrated that a hydrocarbon-molecular-ion-implanted epitaxial silicon wafer can reduce the Dₓ and Pb₀ center density in SiO₂/Si interface regions analyzed by quasi-static capacitance–voltage and electron spin resonance measurements, respectively. The Dₓ and Pb₀ center density of wafers without hydrocarbon molecular ions increased after annealing at 700 °C. On the other hand, the Dₓ and Pb₀ center density of wafers implanted with hydrocarbon molecular ions decreased after annealing at 700 °C. We also estimated the activation energy to be 1.67 eV for the hydrogen termination reactions with hydrogen molecules and Si dangling bonds at the SiO₂/Si interface. The termination effects of the hydrocarbon-molecular-ion-implanted epitaxial silicon wafers can contribute to the high electrical performance of CMOS image sensors. © 2020 The Japan Society of Applied Physics

1. Introduction

Complementary metal-oxide semiconductor (CMOS) image sensors have been widely used in ubiquitous devices such as smartphones and smartwatches. Consumer markets strongly demand for high sensitivity and speed image data processing for fabricating high-performance CMOS image sensors. ¹–⁶ However, there are some serious technical issues in manufacturing high-performance CMOS image sensors, namely, dark current noise, fixed-pattern noise, and random telegraph noise generated from CMOS image sensor circuits.

The origins of noise are SiO₂/Si interface states at transfer gate oxides, local oxidation of silicon, shallow trench isolation, and deep trench isolation. ⁷–⁹ The interface states form deep energy levels in the silicon bandgap, which can act as generation–recombination (G–R) centers. The G–R centers strongly affect the electrical performance of CMOS image sensors. Therefore, CMOS image sensor manufacturers try to reduce noise from various parts of sensor circuits using optimum circuit designs and device fabrication processes. Previously, some circuit design researchers proposed a new circuit design concept for noise reduction from CMOS image sensor circuits. ¹⁰,¹¹ However, it is insufficient to improve the noise reduction performance. Another solution is low-temperature hydrogen annealing after the back-end-of-line (BEOL) process. ⁹,¹²,¹³ We call this forming gas annealing (FGA) treatment. However, recently, multilfilm formation by atomic layer deposition has been widely applied to the BEOL process for the formation of interconnect layers. Hydrogen does not reach the SiO₂/Si interface because the deposited interconnect layers act as a diffusion barrier. Therefore, it is not effective for solving the above-mentioned technical issue. A previous study has shown the reduction in interface state density (Dₓ) at the SiO₂/Si interface by forming a SiO₂ layer after monomer hydrogen ion implantation. ¹⁴,¹⁵ However, since high-temperature annealing is repeatedly performed after forming the SiO₂ layer, a concern is that Dₓ increases when hydrogen at the SiO₂/Si interface is released again during high-temperature annealing. In addition, hydrogen does not remain in the silicon wafer after annealing owing to its high diffusion velocity. It is necessary to repeat the step of implanting hydrogen ions, which increases the device fabrication cost. Thus, the reduction in Dₓ by monomer hydrogen ion implantation is difficult to apply to any device process.

Therefore, we have developed an alternative solution to the technical issue encountered in CMOS image sensor fabrication by hydrocarbon molecular ion implantation. ¹⁶–²⁴ We have demonstrated the fabrication of a novel silicon wafer with three unique characteristics, namely, the high gettering capability for metallic impurities, the oxygen out-diffusion barrier effect and the hydrogen storage effect in the projection range of hydrocarbon molecular ions. We have already reported that our novel silicon wafer leads to a marked improvement of CMOS device key parameters, such as low dark current and white spot defect density when using the CMOS image sensor manufacturing line. ¹⁸

In addition, in a previous study, the diffusion behavior of two types of hydrogen was investigated in the hydrocarbon-molecular-ion-implanted region during isochronal annealing. ²⁴ One is molecular hydrogen (H₂) that out-diffused to the silicon lattice from the hydrocarbon-ion-implanted region during annealing below 800 °C. The other is atomic hydrogen (H) that out-diffused to the silicon lattice from the hydrocarbon-molecular-ion-implanted region during annealing above 800 °C. The study suggests that these two types of hydrogen with different diffusion behaviors can reduce Dₓ at the SiO₂/Si interface. However, it did not clearly show the hydrogen termination mechanism at the SiO₂/Si interface after isochronal annealing.

Therefore, in this study, we focus on the mechanism of hydrogen termination of the SiO₂/Si interface state after isochronal annealing analyzed by capacitance–voltage (C–V) and electron spin resonance (ESR) measurements. Previous studies have shown that Dₓ increases owing to the dissociation of hydrogen from the SiO₂/Si interface, which is predominant during annealing at 500 °C or higher.
Therefore, annealing above 500 °C could not be performed after FGA. On the other hand, it has been reported that hydrogen dissociates from the hydrocarbon-molecular-ion-implanted region by annealing at 500 °C or higher. In other words, hydrogen will be supplied to the SiO₂/Si interface from the hydrocarbon-molecular-ion-implanted region even during annealing at 500 °C or higher. Therefore, we aimed to clarify the hydrogen termination effect of the hydrocarbon-molecular-ion-implanted wafer at annealing temperatures higher than 500 °C.

2. Experimental procedure

Figure 1 shows the schematics of the flow of sample preparation and the sample structure. A p-type (100) wafer with a resistivity of 10 ohm cm was implanted with C₃H₅ ions at a dose of 3.3 × 10¹⁴ molecular ions cm⁻² at room temperature. The C₃H₅ implantation energy was 80 keV. The projection range of C₃H₅ under this condition was approximately 80 nm from the surface. Then, 5 μm thick p-type silicon epitaxial layers were grown with a resistivity of 10 ohm cm at 1100 °C. The same epitaxially grown samples without C₃H₅ implantation were prepared to compare the hydrogen termination effect of C₃H₅. Dry oxidation for 100 min at 900 °C was performed for the growth of SiO₂ layers of 25 nm thickness, as shown in Fig. 1. Then, the samples were irradiated with an electron beam of 800 keV at a dose of 2.0 MGy to increase D_it at the SiO₂/Si interface, because D_it obtained by C–V and ESR measurements was below the detection limit with or without C₃H₅ implantation immediately after the SiO₂ layer growth. The samples irradiated with the electron beam were isochronally annealed at 500 °C, 600 °C, 700 °C, 800 °C, and 900 °C for 10, 30, 60, and 120 min in nitrogen ambient. In addition, the MOS structure for C–V measurement was prepared by depositing 100 nm thick Al contacts on the oxide surface by electron beam evaporation using a mechanical mask. The Al electrode area was 6.4 × 10⁻³ cm². Post metallization annealing was performed at 400 °C for 30 min in 1 atm nitrogen ambient. D_it at the SiO₂/Si interface was determined by standard quasi-static CV measurement.²⁵,²⁶ ESR analysis of the samples was conducted to determine the density of Pb centers.²⁷–³⁰

3. Results and discussion

Figure 2 shows the high frequency (100 kHz) and quasi-static C–V curves before electron beam irradiation (black dotted line) without C₃H₅ implantation and after electron beam irradiation without (blue dashed line) and with (red solid line) C₃H₅ implantation. There was a concern that an inversion layer would be formed in this structure without a channel stop layer. However, the high-frequency C–V curves show almost the same value of Cₘᵢₙ. Thus, it was considered that the inversion layer did not affect the D_it evaluation. On the other hand, the measured quasi-static C–V curves of wafers with and without C₃H₅ implantation were found to be different in Fig. 2. These results indicate that D_it at the SiO₂/Si interface can be calculated by the standard quasi-static method.²⁵,²⁶ Figure 3 shows the D_it of wafers after annealing at (a) 500 °C and (b) 700 °C without (blue squares) and with (red diamonds) C₃H₅ implantation and before annealing without (black circles) C₃H₅ implantation. D_it at
the midgap of the wafer with C3H5 implantation was lower than that without C3H5 implantation after annealing at 700 °C. A previous study demonstrated that the $D_{it}$ of the sample with hydrogen FGA increases with annealing above 600 °C because hydrogen dissociates from the SiO2/Si interface during high-temperature annealing. However, in this study, the $D_{it}$ of the C3H5-implanted wafer decreased after annealing at 700 °C. The result demonstrated that the $D_{it}$ of the C3H5-implanted wafer may be decreased by hydrogen out-diffusion from the C3H5-implanted region after high-temperature annealing. Then, the dependence of $D_{it}$ on annealing temperature was analyzed by the quasi-static C–V method. Figure 4 shows $D_{it}$ at the midgap of the wafers without and with C3H5 implantation after annealing. The $D_{it}$ of the wafers without C3H5 implantation decreased initially after annealing at 500 °C, but increased after annealing at 900 °C. In contrast, the $D_{it}$ of the C3H5-implanted wafer decreased with increasing annealing temperature. A previous study showed that the concentration of hydrogen that out-diffused from the C3H5-implanted region increases with annealing temperature. Thus, the $D_{it}$ of the C3H5-implanted wafer was lower after annealing at 900 °C than after annealing at 500 °C. We consider that the increase in the concentration of hydrogen that out-diffused from the C3H5-implanted region was larger than the increase in $D_{it}$ during annealing at 900 °C. Consequently, the reduction in $D_{it}$ at the SiO2/Si interface after annealing at 900 °C is considered to be the hydrogen termination effect of the C3H5-implanted epitaxial silicon wafer.

Then, we analyzed the C–V-measured samples by ESR measurement to identify the defects at the SiO2/Si interface and determine their relationship with the reduction in $D_{it}$. ESR measurement is used to analyze the density of Pb center defects, which are also known as SiO2/Si interface state defects.27–29 Most ESR studies of the SiO2/Si interface have dealt with Pb center defects.29,30 One of the Pb center signals was the Pb0 center at $g$ values of 2.0060–2.0062 for each annealed sample. A Pb0 center has a structure of three silicon bonds: ·Si≡Si3.31 In addition, to verify the relationship between $D_{it}$ and the density of Pb0 centers, we plotted $D_{it}$ at the midgap versus density of Pb0 centers at each annealing temperature in Fig. 5. The results indicate that the density of Pb0 centers determined by ESR measurement is proportional to $D_{it}$ obtained by CV measurement. In this study, electron beam irradiation was performed to increase $D_{it}$ and clarify the hydrogen termination effect on the C3H5-implanted epitaxial wafer. As shown in Fig. 5, $D_{it}$ and Pb0 center density were increased by electron beam irradiation, which can be reduced by using a C3H5-implanted silicon epitaxial wafer. Figure 5 also shows that the $D_{it}$ in the wafer without C3H5 implantation is also proportional to the density of Pb0 centers.

Then, we discuss the increases or decreases in $D_{it}$ and the density of Pb0 centers in wafers without and with C3H5 implantation after isochronal annealing. Firstly, we consider the change in the $D_{it}$ of wafers without C3H5 implantation. A previous study showed that $D_{it}$ decreases in nitrogen ambient at 500 °C.32 Here, the initial decrease in the $D_{it}$ of wafers without C3H5 implantation at 500 °C may be a passivation effect of the thermal strain induced by thermal stress in nitrogen ambient.33 However, it has been reported that $D_{it}$ is...
lower after hydrogen FGA than after annealing in nitrogen ambient. The increase in the $D_{th}$ of wafers without C$_3$H$_5$ implantation at 700 °C can be explained by two possibilities. One is $D_{th}$ generation due to Si–O bond breaking induced by the compressive stress during annealing. The other is the dissociation of hydrogen or nitrogen at the SiO$_2$/Si interface during annealing. On the other hand, the $D_{th}$ of the C$_3$H$_5$-implanted wafers decreased with increasing annealing temperature. The results suggest that the hydrogen that out-diffused from the C$_3$H$_5$-implanted region can terminate dangling bonds of silicon at the SiO$_2$/Si interface during annealing at 700 °C. Probably, the hydrogen that terminated interface state defects should also dissociate from the SiO$_2$/silicon interface of the C$_3$H$_5$-implanted wafer. However, it is considered that the concentration of hydrogen that out-diffused from the C$_3$H$_5$-implanted region is higher than that of the dissociated hydrogen during annealing at 700 °C. Indeed, a previous study has demonstrated that the concentration of hydrogen that out-diffused from the C$_3$H$_5$-implanted region at 700 °C is sufficient compared with $D_{th}$ at the SiO$_2$/Si interface. Therefore, we consider that $D_{th}$ at the SiO$_2$/Si interface obtained by C–V measurement was reduced owing to the termination of the Si dangling bonds (Si≡Si) at the SiO$_2$/silicon interface with hydrogen out-diffused from the C$_3$H$_5$-implanted region.

We then consider how the out-diffused hydrogen terminated the Si dangling bonds. Previous studies have shown that two types of hydrogen, molecular and atomic, dissociate from the C$_3$H$_5$-implanted region. In this study, a SiO$_2$ layer was formed at 900 °C for 100 min and then irradiated with an electron beam for annealing at 500 °C and 700 °C for 30 min. A previous study showed that molecular hydrogen first dissociates from the C$_3$H$_5$-implanted region. In addition, it has been demonstrated that a considerable amount of atomic hydrogen diffuses during annealing at a temperature higher than 800 °C. For this reason, molecular hydrogen diffusion predominantly occurs during the subsequent annealing at 500 °C and 700 °C.

The mechanism of the hydrogen termination effect in the hydrocarbon-molecular-ion-implanted wafer was analyzed on the basis of the assumed termination reaction at the SiO$_2$/Si interface. The dependence of the $D_{th}$ of C$_3$H$_5$-implanted wafers on annealing time and temperature was evaluated by quasi-static C–V measurement. Figure 6 shows $D_{th}$ at the midgap of the C$_3$H$_5$-implanted wafers after 500 °C, 600 °C, 700 °C, 800 °C and 900 °C annealing. The $D_{th}$ of C$_3$H$_5$-implanted wafers decreased with increasing annealing temperature and time. In the wafers with high-temperature annealing, $D_{th}$ rapidly decreased significantly. A previous study showed the effect of hydrogen passivation by FGA below 500 °C. Previous studies have never shown a decrease in $D_{th}$ with annealing time at temperatures above 500 °C. Understanding the reduction mechanism of the $D_{th}$ of C$_3$H$_5$-implanted wafers is extremely important for the application of advanced CMOS image sensors to device processing.

Therefore, we analyzed a termination reaction by reaction kinetic analysis in order to explore the reduction mechanism of the $D_{th}$ of C$_3$H$_5$-implanted wafers. The reaction equation for the time change in $D_{th}$ obtained from the assumed reaction model was derived and fitted with the experimental results for the annealing time dependence of $D_{th}$. Furthermore, the activation energy of hydrogen termination at the SiO$_2$/Si interface was derived by obtaining the reaction rate constant of the assumed reaction model from the fitting results. A reaction model of the hydrogen termination effect at the SiO$_2$/Si interface was assumed. The reaction between hydrogen and a silicon dangling bond at the SiO$_2$/Si interface is expressed by reaction Eq. (1). This is a reaction model in which hydrogen binds to silicon dangling bonds (Si') to form Si–H. The reaction equation obtained from reaction Eq. (1) is shown in Eq. (2).

$$\text{Si} + H \xrightarrow{k_1} \text{Si} - H$$

(1)

$$\frac{d}{dt}\text{[Si]} = -k_1\text{[Si]}\text{[H]}.$$  

(2)

[Si] corresponds to the $D_{th}$ obtained in the experiment. Previously, Reed and Plummer demonstrated the model of the molecular hydrogen reaction of $D_{th}$ at the SiO$_2$/Si interface during hydrogen FGA at low temperatures. The reaction model and reaction equation are as shown in reaction Eqs. (3) and (4).

$$2H \xrightarrow{k_2} H_2$$

(3)

$$\frac{d}{dt}\text{[H]} = -2k_2\text{[H]}^2.$$  

(4)

This reaction model is derived from the experimental results of hydrogen termination after low-temperature FGA. In addition, Eq. (5) is derived from the above Eqs. (2) and (4) to consider the hydrogen passivation mechanism at the interface during high-temperature annealing.

$$\text{[Si]} = \frac{[\text{Si}]_0}{1 + 2k_2[H]^2t}[\text{H}]_0.$$  

(5)

where $k_1$ and $k_2$ are the reaction rate constants of reaction Eqs. (1) and (3), respectively. $t$ is the annealing time. $[\text{Si}]_0$ is the initial concentration of hydrogen that out-diffused from the C$_3$H$_5$-implanted region. In addition, in a previous study, [H]$_0$ is expressed by the dissociation activation energy and the initial hydrogen concentration in the projection range of C$_3$H$_5$ as:

![Image](image_url)
\[ [H]_0 = 1.2 \times 10^{-18} \exp \left( \frac{0.78 \text{ eV}}{k_B T} \right) \text{(atoms cm}^{-3} \text{)}. \quad (6) \]

\[ k_2 \text{ is expressed by the following equation:} \]
\[ k_2 = 1.3 \times 10^{-12} \exp \left( \frac{0.75 \text{ eV}}{k_B T} \right) \text{(atoms cm}^{-3} \text{)}. \quad (7) \]

\( k_B \) is Boltzmann constant and \( T \) is temperature. Using \( k_1 \) in Eq. (5) as a parameter, we derived \( k_1 \) by fitting the model with the experimental results of the annealing time dependence of the \( D_{it} \) of the C\(_3\)H\(_5\)-implanted wafers. Figure 7 shows the fitting plots of \( D_{it} \) at the midgap of wafers with C\(_3\)H\(_5\) implantation by Eq. (5). In addition, Fig. 8 shows the Arrhenius plot obtained using \( k_1 \) at each annealing temperature. It is shown that the reaction at the SiO\(_2\)/Si interface can be expressed by assuming the reaction model and the proportion of the Arrhenius plot. \( k_1 \) for the Arrhenius plot is shown below
\[ k_1 = 7.03 \times 10^{-13} \exp \left( \frac{1.67 \text{ eV}}{k_B T} \right) \]

We discuss the obtained activation energy of 1.67 eV. Stesmans reported that the activation energy in the reaction where hydrogen molecules terminate the Pb\(_0\) centers at the SiO\(_2\)/Si interface formed by dry oxidation on Si (100) is 1.51 eV.\(^{34}\) The following is a model of the reaction of molecular hydrogen at the SiO\(_2\)/Si interface:
\[ \text{Si} :: \text{H} \rightarrow \text{Si} - \text{H} + \text{H} \]

A previous study demonstrated that the molecular hydrogen out-diffused from the C\(_3\)H\(_5\)-implanted region at annealing temperatures below 900 °C.\(^{24}\) In addition, we clarified that the change in \( D_{it} \) at the SiO\(_2\)/Si interface was enhanced by electron beam irradiation and proportional to that in the density of Pb\(_0\) centers obtained by ESR measurement. Therefore, we found that hydrogen molecules out-diffusing from the C\(_3\)H\(_5\)-implanted region can reduce \( D_{it} \) at the SiO\(_2\)/Si interface. The diffusion rate of hydrogen molecules in silicon wafers is high, and the molecules can easily reach the SiO\(_2\)/Si interface. As shown previously, hydrogen easily dissociates from the SiO\(_2\)/Si interface during annealing at temperatures higher than 500 °C. The analysis of hydrogen termination was limited to low-temperature annealing below 500 °C. However, the activation energy of 1.67 eV for hydrogen termination in high-temperature annealing above 500 °C has been derived assuming two reaction models for the SiO\(_2\)/Si interface and hydrogen.

In this study, the hydrogen termination effect of the C\(_3\)H\(_5\)-implanted silicon epitaxial wafer, which has not yet been reported, reduced \( D_{it} \) at the SiO\(_2\)/Si interface. It was found that the activation energy of the hydrogen termination reaction for the silicon dangling bonds at the SiO\(_2\)/Si interface even in high-temperature annealing is close to those in previous studies. The hydrogen termination effect in terms of \( D_{it} \) reduction under annealing conditions from 500 °C to 900 °C is compatible with device processes, which has recently been progressing to lower temperatures.

On the other hand, the C\(_3\)H\(_5\)-implanted region also contains carbon atoms, which form carbon complexes that act as the gettering and trapping sites for metal impurities and hydrogen, respectively.\(^{19-23}\) If carbon diffuses from the carbon complexes to the device active region, carbon can form the deep energy levels that cause the dark currents in CMOS image sensors.\(^{35}\) However, since carbon diffuses more slowly in silicon than in hydrogen, it is possible to reduce the effect of carbon depending on the annealing conditions. Therefore, we believe that the results of this study are meaningful for device processes that tend to use lower temperatures.

The results suggest that hydrogen molecules out-diffusing from the projection range of hydrocarbon molecular ion can efficiently terminate \( D_{it} \) at the SiO\(_2\)/Si interface. This is an important characteristic that contributes to the reduction in \( D_{it} \) at the SiO\(_2\)/Si interface, which is required for high-performance CMOS image sensors.

4. Conclusions

We demonstrated that C\(_3\)H\(_5\)-implanted epitaxial silicon wafers can reduce \( D_{it} \) and Pb center density determined by CV and ESR measurements, respectively. The \( D_{it} \) and Pb center density of wafers without C\(_3\)H\(_5\) implantation increased after annealing at 700 °C. On the other hand, the \( D_{it} \) and Pb center density of C\(_3\)H\(_5\)-implanted wafers decreased after annealing at 700 °C. These results indicate that C\(_3\)H\(_5\)-implanted silicon epitaxial wafers have the passivation effect through hydrogen termination, which decreases \( D_{it} \) at the SiO\(_2\)/Si interface. In addition, we derived the activation energy of 1.67 eV for the hydrogen termination reaction with hydrogen molecules and Si dangling bonds at the SiO\(_2\)/Si interface. We clarified that hydrogen molecules out-diffusing

Fig. 7. (Color online) Fitting plots of \( D_{it} \) at the midgap of wafers with C\(_3\)H\(_5\) implantation by Eq. (5).
from the hydrocarbon-molecular-ion-implanted region can reduce $D_{it}$ at the SiO$_2$/Si interface. We conclude that the hydrogen termination effect of the hydrocarbon-molecular-ion-implanted epitaxial silicon wafer can contribute to high electrical device performance, such as low noise or dark current owing to the interface state at the SiO$_2$/Si interface of CMOS image sensors.

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