Residual stress analysis of SiO$_2$ films deposited by plasma-enhanced chemical vapor deposition

Jin-Kyung Choi$^{a,*}$, J. Lee$^a$, Ji-Beom Yoo$^a$, Jong-Sun Maeng$^b$, Young-Man Kim$^b$

$^a$Department of Materials Engineering, Sungkyunkwan University, 300 Chunchun-dong Jangan-gu, 440-746 Suwon, South Korea
$^b$Department of Metallurgical Engineering, Chonnam National University* 300, Yongbong-dong Buk-gu, 500-757 Kwangju, South Korea

Abstract

We investigated the residual stress of SiO$_2$ film using a beam bending method. The effects of deposition parameters such as Si precursor, growth temperature, N$_2$O flow rate and RF power on the residual stress and growth characteristics of SiO$_2$ film were estimated. As the growth temperature increased, residual stress of SiO$_2$ film with HMDS decreased from $-235$ to $-120$ MPa while the residual stress of SiO$_2$ with SiH$_4$ showed the opposite trend. As the flow rate of N$_2$O increased, the residual stress of SiO$_2$ film with HMDS changed from $-493$ to $48$ MPa. As RF power increased, the residual stress of SiO$_2$ film with HMDS changed from $153$ to $-16$ MPa. Residual stress of SiO$_2$ film with HMDS was lower than that of the SiO$_2$ film with SiH$_4$.

Keywords: Residual stress of SiO$_2$; Hexamethyldisilazane; Plasma enhanced chemical vapor deposition

1. Introduction

SiO$_2$ film is particularly useful for dielectric isolation and chemical passivation. But this material has problems like a high residual stress of 100–400 MPa [1], resulting in degradations of functioning and reliability of devices. It is important to reduce the residual stress of SiO$_2$ especially for microelectromechanical systems (MEMS) and other optical devices incorporated with SiO$_2$ film. Information on the residual stress depending on the precursor and growth conditions provides a way to reduce residual stress of the SiO$_2$ film. The analysis of the residual stress of the SiO$_2$ film deposited by chemical vapor deposition (CVD) using SiH$_4$ and tetraethoxysilane (TEOS) has been widely reported [2–5]. However, there were few studies on the residual stress of the SiO$_2$ film deposited by plasma-enhanced chemical vapor deposition (PECVD) using hexamethyldisilazane (HMDS).

We investigated the residual stress of SiO$_2$ film using a beam bending method. The effects of the deposition parameters such as silicon precursors, growth temperature, gas composition, RF power on the residual stress and growth characteristics of SiO$_2$ film were investigated. The residual stress of the deposited SiO$_2$ films is estimated by measuring the curvature of the specimen before and after the film deposition. Growth rate and etch rate of SiO$_2$ film were measured by ellipsometry, α-step and BOE solution to analyze the residual stress of the film.

2. Experiments

Silicon oxide films were deposited on Si (100) substrates using PECVD where a resistively heated substrate was employed. Hexamethyldisilazane (HMDS) or SiH$_4$ (10% diluted in Ar) was used as a Si source. Nitrous oxide (N$_2$O) was used as an oxygen precursor. Ar was employed as a carrier gas. Each sample was cleaned by a piranha (H$_2$SO$_4$: H$_2$O$_2$ = 4:1) solution and 6:1 buffered oxide etch (BOE) prior to deposition. The chamber was evacuated to a pressure of less than
10 m Torr while the substrate was heated up to the growth temperature. After the temperature was stabilized, reactant gases were supplied to the chamber. The typical flow rates of the Ar, nitrous oxide (N₂O) and HMDS or SiH₄(10%) were 170, 3.75, 8 and 5 sccm, respectively. HMDS bubbler temperature was 35°C. An RF generator operating at 13.56 MHz and matching network supplied RF power. After the film was deposited, purging was performed.

The film thickness is measured with an α-step, or ellipsometry. The etch rates were measured with 6:1 BOE at room temperature. The index of refraction was measured by ellipsometry of which the monochromatic wavelength was 632.8 nm and the angle of incidence was 70.00°. The residual stress of the SiO₂ film is measured by the beam bending method where the curvatures of the substrate were measured before and after the film deposition.

3. Results and discussion

Effects of growth temperature on the residual stress and properties of SiO₂ films with HMDS were investigated and shown in Fig. 1. For the deposition of SiO₂ film, the flow rates of N₂O, HMDS, and carrier gas (Ar) were 10, 8 and 170 sccm, respectively. The pressure was 1 Torr and RF power was 75 W. The growth temperature varied from 210 to 270°C. As shown in Fig. 1a, the compressive stress decreases from -235 to -120 MPa with the increase in deposition temperature. Weiss pointed out [6] that there was a strong correlation between the deposition temperature and the amount of hydrogen incorporated in the film and also suggested that the hydrogen concentration decreased with the increase in temperature. Low hydrogen concentration results in a contraction of the film and hence resulted in a tensile stress [4]. Low compressive stress may be due to the low hydrogen concentration at a high growth temperature. The growth rate decreases as the growth temperature increases at a temperature higher than 250°C. However, the growth rate remains constant at 1.6 μm/h at a growth temperature below 250°C. The film quality evaluated by etch rate was improved by increasing the growth temperature; it suggested that a denser SiO₂ film was formed at high temperature than at low temperature.

Effects of growth temperature on the residual stress and properties of SiO₂ films using SiH₄ were investigated and shown in Fig. 2. The flow rates of N₂O, SiH₄, and carrier gas (Ar) were 10, 5 and 170 sccm, respectively. The pressure was 1 Torr and RF power was 75 W. The growth temperature changed ranging from 230 to 290°C. As shown in Fig. 2a, the stress changes from 62.5 to -159 MPa with the increase in deposition temperature. Unlike SiO₂ film with HMDS, stress becomes more compressive with growth temperature. The growth rate and etch rate decreases as the growth temperature increases. The difference in the dependence of residual stress on temperature between SiH₄ and HMDS precursors is now under investigation.

Effects of the N₂O flow rate on the stress, growth rate and etch rate are shown in Fig. 3. The SiO₂ film was deposited using SiH₄ under the conditions that the growth temperature was 270°C, carrier gas (Ar) was 150 sccm and pressure was 1 Torr and RF power was 75 W. The N₂O flow rate varied from 2.75 to 5.75 sccm. As the N₂O flow rate increases, stress of the SiO₂ films with SiH₄ changes from -62.5 to -159 MPa. Variation of stress with N₂O flow rate may be due to the incorporation of oxygen into the interstitial site of the
SiO₂ film. It is reported that the incorporation of the oxygen into the lattice structure could cause the film to be more compressive [8]. The growth rate increases first and remains constant at the N₂O flow rate larger than 4.75 sccm and the etch rate shows a similar trend with N₂O flow rate. The refractive index of SiO₂ film at the high N₂O flow rate was 1.46.

Effects of N₂O flow rate on the residual stress, properties of SiO₂ film with HMDS are shown in Fig. 4. The SiO₂ film was deposited using HMDS under the same conditions as the SiO₂ film with SiH₄ shown in Fig. 3. As the flow rate of N₂O increased, the residual stress changed from ~493 to 48 MPa. Variation of residual stress of SiO₂ using HMDS with N₂O flow is different from that using SiH₄. The mechanism for the different dependence of residual stress on N₂O flow rate is now under investigation. However, we can get the SiO₂ film with very small residual stress of 48 MPa at the growth rate of 0.7 μm/h. As the N₂O/HMDS ratio increased, the growth rate increased first and saturated later like SiO₂ with SiH₄. The increase in growth rate with N₂O is due to the increases in oxygen supply. Saturation in the growth rate implies that the growth rate is not limited by oxygen supply but limited by Si supply. The etch rate increased with the increase in N₂O/HMDS ratio [7].

Fig. 5 shows the variation of residual stress and properties of SiO₂ film with RF power. Flow rate of SiH₄, N₂O and carrier gas (Ar) was 5, 4.75 and 150 sccm, respectively. Temperature and pressure were 250°C and 1 Torr, respectively. The RF power varied from 25 to 100 W. The linear increase in the growth rate with RF power may be due to the increase in the dissociation of gas molecules. The etch rate and index of refraction of the SiO₂ film is independent of RF power. As the RF power increased, the SiO₂ film with SiH₄ becomes more compressive. The residual stress of SiO₂ film changed from ~221 to ~720 MPa. This result may be attributed to the increase in the incorporation of oxygen into the SiO₂.

Fig. 6 shows the effect of RF power on the growth
rate, stress and etch rate of SiO₂ film using HMDS. The SiO₂ film was deposited under the conditions that growth temperature was 250°C, N₂O/HMDS ratio was 15:8, carrier gas (Ar) was 170 sccm and pressure was 1 Torr. The RF power varied from 25 to 100 W. As RF power increased, residual stress of SiO₂ film with HMDS changed from 153 MPa to a compressive stress of −16 Mpa. At the low RF power, low concentration of oxygen results in a tensile stress film [1]. As the RF power increases, dissociation of N₂O increases, resulting in increases in the oxygen incorporation in the SiO₂ film. Effects of oxygen incorporation on the residual stress of SiO₂ film were already explained. A linear increase in the growth rate with RF power may be due to an increase in dissociation of gas molecules. The etch rate of film significantly decreased with increasing RF power. As the RF power increased, index of refraction of SiO₂ film becomes that of thermally grown oxide resulting from sufficient dissociation of gases. Improvement of SiO₂ film quality with RF power is responsible for the reduced etch rate. The energetic ions are also responsible for the observed improvement of oxide film properties since they can induce local bond rearrangement resulting in dense stable films with low stress [9]. The growth rate of 3.3 μm/h was obtained at an RF power of 100 W.

4. Conclusions

As the growth temperature increased, stress of SiO₂ films using SiH₄ became more compressive but the residual stress of SiO₂ using HMDS changed from −235 to −120 MPa. As the flow rate of N₂O increased, stress of SiO₂ films using SiH₄ becomes more compressive. Residual stress of the SiO₂ film using HMDS changed from −493 to 48 MPa with an increase in the flow rate of N₂O. As the RF power increased, SiO₂ film with SiH₄ becomes more compressive. Residual stress of the SiO₂ film with HMDS changed from a tensile stress of 153 MPa to a compressive stress of −16 MPa. Generally, stress of SiO₂ films using SiH₄...
becomes more compressive. Residual stress of SiO$_2$ film using HMDS was lower than that of SiO$_2$ film using SiH$_4$.

Acknowledgements

This work is supported by an international collaboration program through IITA.

References


Fig. 6. Effect of RF power on growth characteristic and properties of SiO$_2$ films with HMDS: (a) growth rate and etch rate; and (b) stress.