Determining Dielectric Constant Variation of SiOC Low k Film Using Density Measurement

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ABSTRACT

The ability to detect small changes in dielectric constant of low k films in a fast, non-destructive and non-contaminating way is critical for production control. Density measurement was used to determine the k variation of PECVD SiOC films with k~2.86±0.1. The weight measurement technique in this study achieved an accuracy of 0.05mg and the capability to detect density changes of low k films on the order of 0.01 g/cm³. The ability of the density measurement to distinguish the three groups of SiOC low k films was as good as other characterizations such as C-V and refractive index. XPS and SIMS showed no significant change in film composition. Density and k relation was modeled with the Clausius-Mosotti equation.

INTRODUCTION

As ULSI design rules shrink to 0.18μm or smaller, interconnect RC delay and power consumption increase dramatically and start to limit the device performance [1]. Various low k ILD materials have been implemented to reduce RC delay and power consumption. PECVD SiOC film has been widely adopted as the low k of choice for the 0.13μm generation and beyond because of its low k (~2.8), high mechanical strength compared to spin-on polymers, low cost of ownership and compatibility with existing integration schemes [2].

Small variations in process parameters such as drifting plasma power or gas flows can change the dielectric constant, which in turn affects device performance. In production, it is necessary for small variations to be detected as soon as they occur. However, direct measurement of k using C-V methods such as Al dots or Hg probes is rather complicated and there are contamination concerns. Using XPS, SIMS or other methods to determine the changes in composition take a long time and are destructive. Although FTIR is an available method which is quick and non-destructive, it is not accurate enough for quantitative process control purposes. Density measurement can provide an alternative quick and clean method to characterize the changes in dielectric constant. The relationship between density and k can be described by the Clausius-Mosotti equation [3]:

\[ \frac{N_0 \alpha}{3 \varepsilon_0} = \frac{k-1}{k+2} \frac{M}{\rho} \]

(1)

Where \( N_0 \) is Avogadro’s number, \( \varepsilon_0 \) is the vacuum dielectric constant, M is molecular weight, \( \rho \) is the density of the material, and \( \alpha \) is the polarizability. The polarizability of a dielectric material can be written as the sum of three terms: electronic polarizability \( \alpha_e \), atomic polarizability \( \alpha_a \) and
dipole polarizability $\alpha_d$, which are associated with the displacement of charges locally bound in atoms, in molecules, and in the structures, respectively.

\[ \alpha = \alpha_s + \alpha_a + \alpha_d \]  

(2)

SiOC films have a lower $k$ than $\text{SiO}_2$ for two reasons. By replacing the highly polarized Si-O bonds with less polarized Si-C and C-H bonds (with electronegativity difference of 1.54, 0.65 and 0.35, respectively), $\alpha_a$ is lowered. Introducing CH$_3$ to the Si-O network reduces the density $\rho$ of the material. With both mechanisms, the $k$ value of SiOC can be reduced to be 25% lower than SiO$_2$.

In this study, we intentionally varied the $k$ of SiOC films within +/-0.1 by changing process parameters, and attempted to distinguish using density, refractive index (R.I.), Hg-probe, C-V, XPS and SIMS. Density proved to be as good as R.I. in its ability to distinguish the groups and much better than Hg-probe. XPS and SIMS results showed insignificant changes in film composition. The variations in $k$ appear to be due to changes in density $\rho$ instead of polarizability $\alpha$. The relation between density and $k$ was fitted with a Clausius-Mosotti type equation.

EXPERIMENTAL

PECVD SiOC films were deposited on 200mm Si wafers using trimethylsilane and oxygen precursors. Based on a process DOE, in which process parameters were varied by +/-10%, three different groups of SiOC films were selected to give different $k$'s as outlined in Table I. The “Low End”, “Center” and “High End” conditions resulted in $k$'s of 2.78, 2.86 and 2.97, respectively, as determined by C-V measurement. In each group, films were deposited with thickness of 1000Å, 3000Å, 5000Å and 7000Å. Two wafers were deposited at each process condition.

<table>
<thead>
<tr>
<th>Group</th>
<th>Pressure</th>
<th>RF Power</th>
<th>Spacing</th>
<th>O$_2$ Flow</th>
<th>K</th>
</tr>
</thead>
<tbody>
<tr>
<td>Low End</td>
<td>+</td>
<td>-</td>
<td>+</td>
<td>-</td>
<td>2.78</td>
</tr>
<tr>
<td>Center</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>2.86</td>
</tr>
<tr>
<td>High End</td>
<td>-</td>
<td>+</td>
<td>-</td>
<td>-</td>
<td>2.97</td>
</tr>
</tbody>
</table>

Refractive index and thickness were measured using KLA-Tencor’s UV1050 with goodness of fit >0.9. C-V measurement was performed on the MOS structure at 100 kHz using Hg-probe (CV MAP 92A by Four Dimensions, Inc.). The SiOC measurement program on the Hg probe was separately confirmed by using the Al dots C-V method.

The weight measurement was performed using Metryx’s Mentor tool. Care was taken to correct for errors caused by static electricity, thermal convection currents and, atmospheric buoyancy (caused by the volume of the wafer displacing air). Air density corrections were applied for variations in ambient temperature, pressure and humidity. A gauge study conducted by Metryx showed weighing accuracy < 0.05 mg.

RESULTS AND DISCUSSIONS

The weights of all wafers were measured before and after the low $k$ deposition. Figure 1
plotted weight vs. thickness of SiOC film. A control wafer without any deposition was measured together with the other wafers. The difference of the two measurements was 0.046mg, representing <1% of the weight of the 1000Å film.

![Graph of weight vs. thickness](image)

**Figure 1.**

*Weight of SiOC low k film at different thickness.*

The density of the film was calculated using the mean film thickness and assuming a diameter of 200mm for all wafers. As seen in Figure 2, the densities of films 3000Å or thicker were approximately constant within each group, at 1.2815g/cm³, 1.3134g/cm³ and 1.3545g/cm³ for low-end, center and high-end groups, respectively. But the density of the 1000Å film was 1.0-1.9% lower than that of the thicker film within each group. The same phenomenon can be seen in the R.I. measurements of Figure 3, confirming it as a real process effect. Although the cause for the reduced density and R.I. for the thin film was not clear, the observation proved the capability of detecting density differences on the order of 0.01g/cm³.

![Graph of density vs. thickness](image)

**Figure 2.**

*Density of SiOC low k film at different thickness.*
Relative dielectric constant \( k \) was calculated using the mean capacitance and mean thickness of each wafer. Dielectric constant was plotted as a function of thickness in Figure 4. Although the three groups can be distinguished, dielectric constants had some spread within each group presumably due to Hg-probe measurement error. We were unable to measure C-V of the 1000Å films because the films became leaky after moisture absorption.

Table II lists the compositions of the three groups by XPS. There was less than 2% variation in the atomic concentrations of C, O and Si and the differences were not inconsistent with the \( k \) variation. H was another element in the PECVD SiOC film; SIMS profile showed no significant differences in H concentration, as shown in Figure 5.
Table II.

XPS results of the composition of SiOC low k films.

<table>
<thead>
<tr>
<th>Sample</th>
<th>C</th>
<th>O</th>
<th>Si</th>
</tr>
</thead>
<tbody>
<tr>
<td>Low End</td>
<td>Average</td>
<td>25.2</td>
<td>41.9</td>
</tr>
<tr>
<td></td>
<td>Std. Dev.</td>
<td>1.0</td>
<td>0.7</td>
</tr>
<tr>
<td>Center</td>
<td>Average</td>
<td>24.8</td>
<td>42.0</td>
</tr>
<tr>
<td></td>
<td>Std. Dev.</td>
<td>1.0</td>
<td>0.6</td>
</tr>
<tr>
<td>High End</td>
<td>Average</td>
<td>26.0</td>
<td>40.3</td>
</tr>
<tr>
<td></td>
<td>Std. Dev.</td>
<td>0.3</td>
<td>0.2</td>
</tr>
</tbody>
</table>

Figure 5.

SIMS profile of H in the SiOC low k films.

Without detectable changes in film composition, it was assumed that $\alpha$ and $M$ in (1) remained constant for the three groups, and the variation in $k$ with process parameters in this study was due to density differences. Density was plotted as a function of $k$ in Figure 6. A curve representing a Clausius-Mosotti-like equation ran through most of the data:

$$\rho = 2.725(\kappa - 1)/(\kappa + 2)$$

(3)
Figure 6.
Density as a function of $k$.

![Graph showing density as a function of relative dielectric constant.](image)

The molecular formula of the film can be written as SiO$_{1.24}$C$_{0.76}$ based on the mean composition from XPS data in Table II. The molecular weight $M$ is 56.96. From (1) and (3) $\alpha$ is estimated to be $9.22 \times 10^{-34} (\varepsilon \cdot m^3)$, where $\varepsilon$ is the unit of vacuum dielectric constant $\varepsilon_0$, i.e. farad/m.

A characterization’s power to distinguish different groups is defined as:

$$
Power = \frac{\text{mean(grp1)} - \text{mean(grp2)}}{\text{mean(stdev(grp1), stdev(grp2))}}
$$

(4)

The powers for Hg-probe, refractive index, XPS of C at% and density are compared to each other in table III. Density is as good as R.I., and better than other techniques.

Table III.
Power to distinguish the groups by different characterization methods.

<table>
<thead>
<tr>
<th>To Distinguish</th>
<th>Hg-probe</th>
<th>R.I.</th>
<th>C at%</th>
<th>Density</th>
</tr>
</thead>
<tbody>
<tr>
<td>Low End to Center Groups</td>
<td>3.92</td>
<td>12.09</td>
<td>0.40</td>
<td>16.41</td>
</tr>
<tr>
<td>High End to Center Groups</td>
<td>4.43</td>
<td>34.93</td>
<td>1.85</td>
<td>15.72</td>
</tr>
</tbody>
</table>

The Clausius-Mosotti equation can be applied to the complex index of refraction, $n^*$. However, the R.I. routinely measured is only the real part of $n^*$. R.I. is measured at UV frequencies, only reflecting the contribution of the $\alpha_e$ term in (2). For these two reasons, correlating R.I. to $k$ is not as easy as with density. Although R.I. measurement can distinguish the groups in this study, density is a better method to detect $k$ variation.

Hg-probe C-V measurement can give $k$ results, but it has a larger error than density measurement and presents Hg contamination concerns. Al-dot C-V is usually accurate, but it is complicated, destructive and not suitable for daily production control. XPS and SIMS could not detect the $k$ changes in this study because the mechanism for the $k$ variation was due to changes in density rather than composition.

For production process control, it is best to use the combined techniques of density measurement for $k$ and optical measurement for R.I. and thickness.
CONCLUSIONS

Process variations were introduced to deposit three groups of PECVD SiOC film with k of 2.78–2.97. The k variations were effectively determined by density measurement. The power of density to distinguish the groups was as high as R.I. and better than Hg-probe C-V measurement. It was demonstrated that the sensitivity of density measurement was on the order of 0.01 g/cm$^3$. The density and k relation can be described using the Clausius-Mosotti equation because SIMS and XPS showed no significant composition changes.

ACKNOWLEDGEMENTS

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REFERENCES