Characterization of thermally treated PECVD SiON layers

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PECVD Silicon Oxynitride (SiON) layers with different refractive indices (1.472-1.635) were grown and characterized. The as-deposited layers have good thickness uniformity (~1%) and a high homogeneity of the refractive index (~ 5x10^-4) over the wafer area. For telecommunication application, however, the optical losses of the as-deposited layers are unacceptably high. Therefore, the loss reduction upon annealing as well as the impact of the elevated temperature on the remaining layer properties has been studied. Annealed waveguides with optical losses as low as 0.2 dB/cm at \(\lambda = 1550\) nm have been realized.

Introduction
Plasma Enhanced Chemical Vapor Deposition (PECVD) for silicon oxynitride deposition is a very attractive technique for optical waveguide fabrication. Because of the hydrogenated precursors, the as-deposited SiON layers contain a large amount of hydrogen bonded as N-H and Si-H, having their stretching modes around 3340 cm\(^{-1}\) and 2280 cm\(^{-1}\), respectively. Consequently, their overtones contribute to absorption losses around 1510 and 1500 nm wavelength, respectively. Since this wavelength region is of particular interest for the third telecommunication window, the hydrogen content of the SiON layers has to be reduced significantly. It is known from literature that hydrogen can be eliminated from SiON layers by treatment at elevated temperature [1]. In this paper we will summarize our results on silicon oxynitride waveguide deposition, the thermal treatment of the layers and discuss the effect of the high temperature on optical loss and other relevant layer properties.

Deposition and characterization of SiON layers
The silicon oxynitride layers were deposited from 2%SiH\(_4\)/N\(_2\) and N\(_2\)O applying a parallel plate Electrotech 310 PECVD reactor operating in the low frequency mode (187.5 KHz). The applied plasma power was 60 W, the chamber pressure was 650 mTorr and the substrate temperature was kept at 300 \(^\circ\)C. In order to allow for prism coupling measurement, layers were deposited on thermally oxidized wafers. From prism coupling the following parameters have been extracted: layer thickness (d), refractive index (n), material birefringence (\(\Delta n_{TM-TE}\)), thickness non-uniformity over the wafer (\(\delta d\)), refractive index in-homogeneity over the wafer (\(\Delta n\)) and optical loss (\(\alpha\)). Since the prism coupling measurement has been performed at several wavelengths, 632.8, 832.0 and 1523.4 nm, we have been able to extract the wavelength dispersion with the Cauchy relation. Applying the Cauchy parameters, the refractive index at \(\lambda = 1550\) nm (n\(_{1550}\)), which is relevant for the design of telecommunication devices, has been calculated. For characterizing the SiON material by ellipsometry and IR-spectroscopy, the layers have been grown directly on <100> oriented 3” silicon wafers. In general, ellipsometry allows for fast measurement of thickness, refractive index, thickness non-uniformity and wavelength dispersion. IR-spectroscopy has been applied for the measurement of the absorption spectra of the SiON layers. Based on the measured FTIR spectra, the Si-H and N-H content in those layers has been calculated applying the N-H and Si-H absorption cross-sections as given by Lanford and Rand [2].
By varying the ratio between the input gas flows ($N_2O : SiH_4/N_2$) SiON layers with different compositions have been deposited. The refractive indices and deposition rates of several as-deposited SiON layers are given in figure 1. For SiON layers with the same compositions, the uniformity of layer thickness and refractive index as well as the material birefringence have been measured, too. The results are summarized in table I.

The thickness non-uniformity and refractive index inhomogeneity values are representing the min-max deviation of those parameters over a 50x50 mm$^2$ area on a 3” wafer. The good thickness non-uniformity, being typically in the range of 1-2%, as well as the excellent refractive index in-homogeneity, around 5x10$^{-4}$, make those layers very attractive for application in integrated optics devices. The material birefringence, given by $\Delta n_{TM-TE} = n_{TM} - n_{TE}$, whereas $n_{TM}$ and $n_{TE}$ are the refractive indices for TM and TE polarized light, respectively, is typically in the order of 10$^{-3}$. The material birefringence, which is a highly important parameter for the polarization independent device design, is caused by compressive stress in the deposited SiON layers [3].

Furthermore, the measured N-H and Si-H content of the various, as-deposited SiON layers are given in table I, too. The N-H as well as the Si-H content increases with the refractive index of the layer. Since the overtones of those hydrogen bonds cause unacceptably high losses in the for telecommunication relevant wavelength region, the amount of hydrogen has to be significantly reduced by thermal treatment, what we will discuss in detail in the next section.

Table I: Refractive index in-homogeneity, thickness non-uniformity, material birefringence and N-H and Si-H content of PECVD SiON layers with different refractive indices

<table>
<thead>
<tr>
<th>$n_{1550}$</th>
<th>$\Delta n$ (x10$^{-4}$)</th>
<th>$\delta d$ (%)</th>
<th>$\Delta n_{TM-TE}$ (x10$^{-4}$)</th>
<th>N-H [at%]</th>
<th>Si-H [at%]</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.4722</td>
<td>6</td>
<td>1.7</td>
<td>11</td>
<td>1.99</td>
<td>0.54</td>
</tr>
<tr>
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<td>1.1</td>
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<td>7.82</td>
<td>1.4</td>
</tr>
<tr>
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<td>5</td>
<td>2</td>
<td>16</td>
<td>15.20</td>
<td>2.57</td>
</tr>
<tr>
<td>1.6347</td>
<td>5</td>
<td>1.2</td>
<td>26</td>
<td>15.99</td>
<td>5.38</td>
</tr>
</tbody>
</table>

Anneal treatment of SiON layers

In order to study the effects of the anneal treatment, layers with different refractive indices ($n_{1550} = 1.472 – 1.635$) and different layer thickness ($300 – 2600$ nm) have been deposited. In the first place, the optimum annealing time has been determined. It was found that after 3 hours annealing, the layer properties would not change significantly anymore. Therefore, the different layers to be studied have been annealed each for 3 hours in a nitrogen atmosphere at annealing temperature ranging from 400 to 1150°C.
A typical example of the FTIR spectrum of as-deposited and annealed SiON layers with a certain composition ($n_{1550} = 1.527$) is shown in figure 2a. It can be clearly seen that the N-H peak is eliminated after annealing at 1150°C. This corresponds well with the loss measurements obtained from as-deposited and annealed SiON layers with the same composition (figure 2b). The peak loss at 1510 nm wavelength is reduced from 11 dB/cm for as-deposited samples to about 0.6 dB/cm after annealing at 1150°C. Moreover, the optical loss around 1550 nm wavelength is reduced to below 0.2 dB/cm upon annealing at 1150 °C, what makes this low refractive index waveguide material very attractive for application in communication devices. However, for the PECVD SiON layers with a refractive index exceeding 1.584, cracks have been observed at annealing temperatures of 1000 °C and higher.

The N-H and Si-H content of the various SiON layers annealed at different temperatures is shown in figure 3 a) and b), respectively. The different behavior of the higher ($n = 1.585 / 1.635$, nitride-like) and lower ($n = 1.472 / 1.527$, oxide-like) refractive index material with respect to the change in N-H content at annealing temperatures up to 1000°C is striking. As earlier discussed [1], the nitride-like material appears to release hydrogen at lower temperature than the oxide-like. This effect was attributed to the cross-linking between N-H and Si-H in the nitride-like material, what is in line with our measurement.
Furthermore, the refractive index change and the relative thickness change (figure 4) have been measured as function of the anneal temperature. From these results, the two steps involved in the annealing process can be clearly distinguished: (1) At temperature below 1000°C the hydrogen bonds are broken and species are diffusing out of the layer. From the strong decrease of the refractive index in the lower temperature range it might be concluded that not only hydrogen is removed, but also other species such as NH(g) evaporates [4]. (2) At temperatures above 1000°C the material is sintered, what can be explained by the combination of a strongly increasing refractive index and shrinkage of the thickness.

**Conclusion**

By varying the N₂O /SiH₄ gas flow ratio of the PECVD deposition process we have fabricated silicon oxynitride layers with various compositions (n₁₅₅₀ =1.472-1.635) having good uniformity and reproducibility. The behavior of these layers upon thermal treatment has been studied in detail. Next to reduction of hydrogen content and optical loss, the change of the refractive index and of the layer thickness upon annealing has been determined. Exact knowledge of these parameters is a highly important input for device designers. Regarding the hydrogen content, we have observed that the N-H and Si-H stretching mode absorption bonds in the PECVD layers, which contribute to the optical losses in the third telecommunication window, are removed upon annealing at 1150°C. At this annealing temperature, the optical losses around 1550 nm wavelength are reduced to below 0.2 dB/cm for slab-type waveguides with a refractive index around 1.527. Therefore, it can be concluded that this material is well suited for application in telecommunication devices. For the higher refractive index material, however, cracks have been observed upon annealing at elevated temperatures. This means that fabrication of low loss high index SiON will remain a challenging feature for future research.

**References**