Chemical Vapor Deposits

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Abstract - **We have been studying the deposition of dielectric thin films using Plasma-Enhanced Chemical Vapor Deposition (PECVD) and Low-Pressure Chemical Vapor Deposition (LPCVD) processes. The study has been in the hope of developing process recipes for SiO_2 and SiN_4 thin films. To achieve this, investigations of film thicknesses, film stress, refractive index and general uniformities, were carried out.**

I. Introduction

SiN_4 and SiO_2 dielectric thin films are useful in the semi-conductor and optical industries. These films are also useful in nanofabrication, microelectronics and microelectromechanical system (MEMS) materials. Silicon Nitride films can act as protective layers for integrated circuits as well as that of a mask for the selective oxidation of silicon[1]. It is good as a hard mask due to the difficulty of oxygen to penetrate the silicon nitride.

Chemical vapor deposition (CVD) is defined as the formation of a non-volatile solid film on a substrate by the reaction of vapor phase chemicals (reactants) that contain the required constituents.[1]

A CVD process can be summarized as consisting of the following sequence of steps: a) a given composition (and flow rate) of reactant gases and diluent inert gases is introduced into a reactant chamber; b) the gas species move to the substrate; c) the reactants are absorbed on the substrate; d) the adatoms undergo migration and film-forming chemical reactions, and e) the gaseous by-products of the reaction are desorbed and removed from the reaction chamber.[1]

Two different deposition techniques were used, the main one being Plasma-Enhanced Chemical Vapor Deposition

(PECVD) and the other one being Low Pressure Chemical Vapor Deposition (LPCVD). Other types of deposition techniques include Atmospheric Pressure Chemical Vapor Deposition (APCVD) and PHoton-induced Chemical Vapor Deposition (PHCVD).

The film properties investigated were film thickness, refractive index and stress associated with the film. The conditions involved in each experiment were: time; gas flow ratio; gas selection; chamber pressure; temperature and gas flow rate. The gas flow ratio is the ratio of the flow rate of N_2 to SiH_4 and can also be termed as process conditions.

The PECVD process makes use of high plasma density and low ion energy at low chamber pressure. This allows for deposition of film at relatively low temperatures. The LPCVD process on the other hand deposits the film at relatively high temperatures and low pressures.

II. Process

Due to the sensitivity of the silicon wafers used, special care was needed in its handling to ensure that the surface remained clean and free from any dust particles.

Fig 1: Nexx machine.

When using the PECVD tool, also called the Nexx machine (fig. 1), the user would have to first run a Burn-In recipe*, this would purge the system of any lingering gases. Now prior to a user's deposition process, the user must run a Pre-Clean recipe which is supposed to condition the chamber prior to deposition. After this Pre-Clean recipe is run the user is then able to proceed to selecting, editing and running the recipe of their choice. Prior to deposition, the load lock is vented and a silicon wafer is placed on the wood chuck. The load lock is then closed and brought to transfer pressure. Once the sample reaches inside the chamber, the chamber is filled with helium and the[1]n argon gas. These gases are allowed to flow into the chamber at specific flow rates, previously determined by the recipe in use. The gases are then allowed to stabilize for a few seconds before the microwave power activates. The plasma particles cause the argon (an inert gas) to begin to react with the nitrogen and deposition of a thin silicon nitride film

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* recipe: standard process conditions for a deposition

on the wafer begins. After the recipe is complete, the sample is brought back into the load lock, which is then brought to atmospheric pressure before the sample is removed.

Fig. 2: ellipsometer

After this is done, the average thickness of film deposited on the silicon wafer is then measured using an ellipsometer (fig. 2), which also was used to calculate the refractive index of the film. A five point scan was done on the sample, in which the laser picked up five points on the sample and measured the thickness and calculated the refractive index for each of these points. The average of the five points was then taken to get the average thickness and refractive index of the sample. The thickness was measured in angstroms (10^{-10} m).

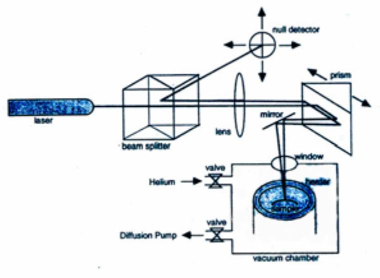

Fig. 3: curvature apparatus[3]

A curvature system (fig. 3) was used to measure the curvature of the sample before and after the deposition of the thin film. The difference of these two values, gives twice the value of the change in curvature associated with the film. A specific sample size of ¼ inch by 1 inch was necessary for the sample to be able to fit on the sample holder of the curvature machine.

This difference or change in curvature is then used in the Stoney equation, in order to derive the stress associated with the film. ΔK represents half the change in curvature or the difference of the two curvatures.

The Stoney Equation

$$\Delta\sigma = \frac{Y_s d_s^{\,2}}{6 d_f} \Delta K$$

d_f = film thickness
d_s=substrate thickness
σ = biaxial film stress
Y_s= biaxial modulus of the substrate
= 180.5 GPa

The recipes used with this technique were variations of the standard Nexx-Sin recipe, with either alterations in the deposition time or in the flow rate of nitrogen into the system, experiments were also run with both these variables changed. The PECVD technique is useful when film needs to be deposited on substrates that cannot stand up to relatively high temperatures.

For the LPCVD technique, the careful preparation of the samples was once again necessary and curvature measurements done before and after deposition. An idle recipe is run prior to deposition and this allows for the ventilation of the chamber. For deposition a nitride recipe was used and after deposition the idle recipe was run again. The machine is left in idle until the next run. The overall process time is longer than the PECVD technique due to the length of time required to pump down and ventilate the system in addition to time required for the system to reach a temperature that is safe for ventilation and handling of the silicon wafer. Generally, the film produced using this technique is of a relatively high quantity.

III. Results

A standard recipe for the deposition of a silicon nitride film thickness of 1000 Å was used. The time was altered through a range of 200s to 1400s. It was noticed that the refractive index varied and as the range sought was between 1.9-2.1 the refractive index (n_f) was therefore out of specification. The graph in fig. 4 shows these results.

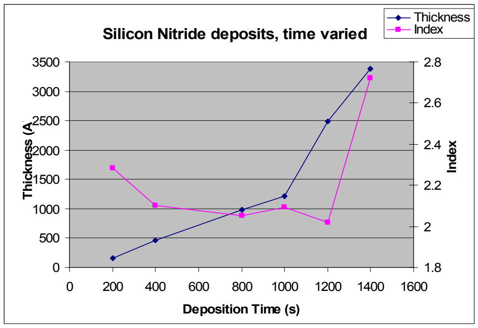

Fig. 4: initial findings

As a result of these findings, we decided to run the experiments again this time varying the gas flow ratio into the system, by simply running the experiment with different flow rates of nitrogen into the system. The flow rate of nitrogen was varied from 2 sccm* to 8 sccm, at 2 sccm intervals. This experiment was run twice, the first with a deposition time of 200s and the second

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* one standard cubic centimeter per minute (sccm) is defined as a flux of one cm^3 of gas per minute at 273 °K and 760 torr.

with a deposition time of 400s. This was in the hope of gaining knowledge as to what affects the range of refractive index. The graphs obtained based on the results of this experiment can be seen in fig. 5 below. The change in recipes as a result is shown in fig. 6 below.

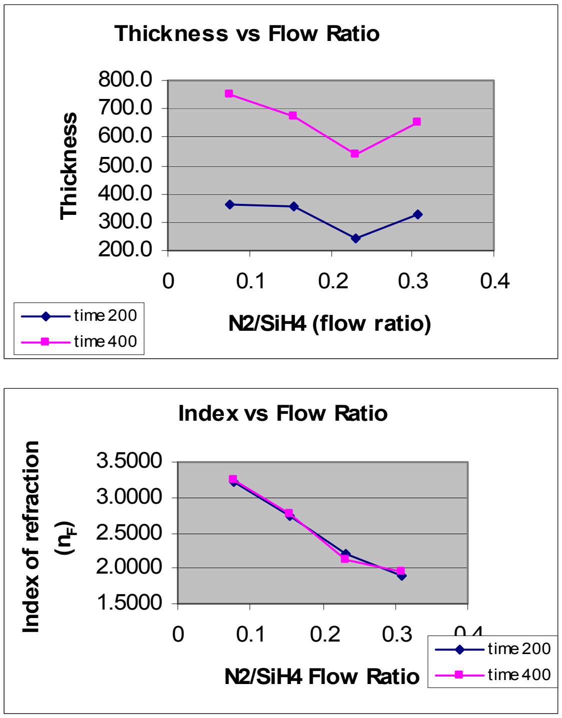

Fig. 5: graphs showing index and thickness vs. time

Old recipe	New recipe
SiH_4: 26 (sccm)	SiH_4: 26 (sccm)
Ar: 20 (sccm)	Ar: 20 (sccm)
N_2: 6.3 (sccm)	N_2: 2,4,6,8 (sccm)

Fig. 6: recipes

From the results obtained in those experiments we then decided to target increasing thickness while keeping the refractive index (n_f) within its specification. To achieve this we varied both time and the gas flow ratio. The quantities of which were approximated based on previous data gathered. The following recipes were used:

- Time: 200 s, N_2: 7 sccm
- Time: 400 s, N_2: 7 sccm
- Time: 600 s, N_2: 6 sccm
- Time: 1000 s, N_2: 6.3 sccm
- Time: 1200 s, N_2: 6 sccm
- Time: 1400 s, N_2: 6 sccm

The results of these experiments are shown in fig. 7 below.

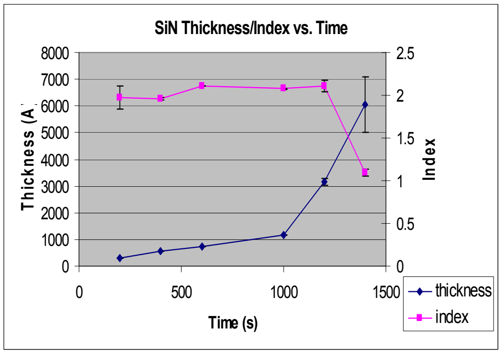

Fig. 7: graph showing thickness and refractive index vs. time.

From the graph you can see that were able to successfully increase the thickness of film deposited while keeping the refractive index within its specified range.

The experiments on Silicon Dioxide however were good and did not need any adjustments to the gas flow ratio or process conditions in order to maintain a satisfactory range for the refractive index. These experiments were done using a standard recipe for silicon dioxide and varying time through a range of 200s to 800s. These results can be seen in the graph in fig. 8.

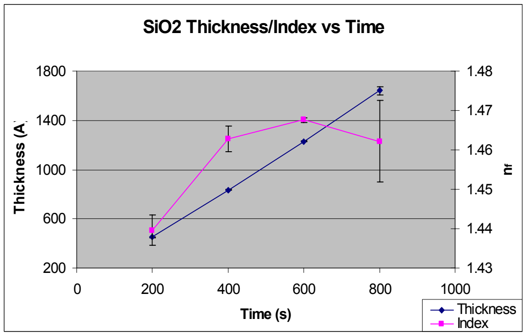

Fig. 8: silicon dioxide, thickness and index vs. time

The stress experiments were also done using variations of the standard recipe for a thickness of 1000 Å of silicon nitride. Once again, the flow rate of nitrogen into the system was varied form 2 sccm to 8 sccm and the results are shown in the graph in fig. 9. The positive y-axis represents tensile stress while the negative y-axis represents compressive stress.

A tensile film has an inherent tendency to contract if the substrate were not present. A compressive film would, under similar circumstance, expand.[2]

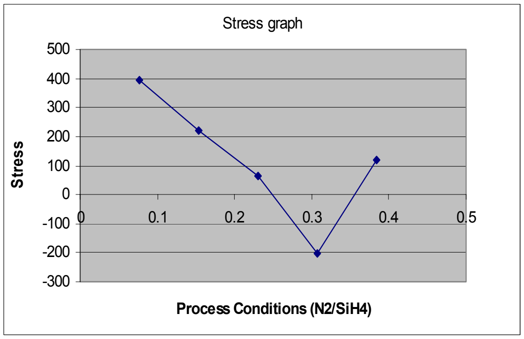

Fig. 9: stress vs. process conditions

IV. CONCLUSION

The general pattern observed throughout our investigations was that thickness can be increased simply by increasing the length of deposition time. However, in order to maintain the refractive index within its specific range of 1.9-2.1, the gas flow ratio must also be adjusted. From the data collected, it was seen that an increase in the gas flow ratio lead to a decrease in the refractive index. Further investigations are needed on the way gas flow ratio can be controlled by controlling the flow rate of nitrogen into the system because the experiment done at time 1400s shows that the refractive index dropped to roughly 1.1 (fig 7), instead of remaining within the range. Also similar investigations need to continue using the LPCVD technique.

Further stress studies must also be done using both techniques before any conclusions may be drawn. We can only wonder if the minimum point shown on the stress vs. process conditions graph (fig. 9) is in fact the film's compressive maximum but further investigations are needed to know for certain.

V. References

[1] S. Wolf, R.N. Tauber, Silicon Processing for the VLSI Era, Volume 1 – Process technology. (1986) Lattice Press.

[2] Plasma-Therm, Inc. "Application notes"

[3] J. A. Mullin, Viscous flow and structural relaxation in amorphous Selenium Thin films. (2000). (p. 19)

VI. Acknowledgments

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