

Properties, process control, and characterization of PECVD silicon nitrides for compound semiconductor devices

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Abstract

We demonstrate the use of scanning electron microscopy (SEM), transmission electron microscopy (TEM), and spectroscopic ellipsometry (SE) to determine thickness of silicon nitrides for compound semiconductor devices, describing in detail the accuracy and convenience of each technique. In addition to thickness, nitride composition is another process parameter that needs to be controlled. Therefore, we also discuss using UV Raman spectroscopy, Rutherford backscattering spectroscopy, and spectroscopic ellipsometry to measure composition. Finally, we discuss the correlation between electrical parameters (capacitance and breakdown voltage) and the stoichiometry of the silicon nitride used as a dielectric in a metal-insulator-metal capacitor.

INTRODUCTION

Silicon nitrides have a variety of uses in compound semiconductor devices (surface passivation, interlayer or capacitor dielectric) due to the versatility of the material when varying composition and deposition conditions. Process control and metrology accuracy is critical for successful operation of the devices. Therefore, to achieve a manufacturable technology, non-destructive, in-line monitoring is essential. Spectroscopic ellipsometry (SE) is a solution and we discuss the sample requirements for optimum thickness and composition measurements. Furthermore, we focus on various analytical techniques, i.e., scanning electron microscopy (SEM), transmission electron microscopy (TEM), Rutherford backscattering (RBS), and Raman spectroscopy that can be completed off-line and yield supporting data. [1]

PROCESS CONTROL OF NITRIDE THICKNESS USING SEM, TEM AND SE

For process control of the areal capacitance (pF/mm^2) in a metal-insulator-metal (MIM) capacitor, it is critical to determine the thickness of the silicon nitride with high accuracy and precision. On product wafers (in MIM structures), we use scanning electron microscopy (SEM) and

transmission electron microscopy (TEM), see Fig. 1. SEM measurement techniques have limited precision, since there are numerous dependent variables to produce optimum images of the sample. The variables, i.e., acceleration voltage, working distance, detector, and beam aperture, all contribute to the image process and affect the measurement precision. Under controlled operating conditions with fixed instrument settings a precision of $\pm 5\%$ can be reached together with a calibration standard. If the instrument settings are changed to improve image contrast, the errors can increase to $\pm 10\%$. Furthermore, physical thickness measurements are made difficult (and inaccurate) by the roughness of the metal plates. The TEM measurements are more accurate at high magnification ($\pm 2\%$, using the GaAs lattice spacing as a built-in calibration standard), but more cumbersome due to the lengthy sample preparation process.

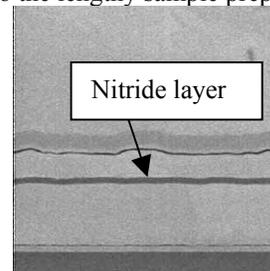


Figure 1: SEM image of a MIM capacitor.

On bare Si or GaAs test wafers, we use SE to determine the nitride thickness, either in-line on a 44-wavelength instrument (400–800 nm) with focusing optics or offline using a tabletop scanning spectroscopic ellipsometer (0.7 to 6.6 eV) with computer-controlled compensator. [2] Since our nitrides are not stoichiometric, but slightly Si-rich, a simple Cauchy or Lorentz oscillator dispersion does not produce good fits to the spectra over the 400–800 nm range of the inline ellipsometer. We therefore add an amorphous band tail at lower photon energies (Tauc-Lorentz model). In this model, the Tauc gap is the lower limit of absorption in the film. It decreases with increasing silicon to nitrogen (Si:N) ratio in the film. [3] The importance of understanding the absorption of the SiN film is the dependence of the property on composition.

In Fig. 2, data was taken on a sample with 800 Å of silicon nitride on a silicon substrate using a variable angle spectroscopic ellipsometer. The data was fitted with the Tauc Lorentz optical dispersion model and the onset of absorption or Tauc gap is 2.34 eV. Due to the small thickness of the film, the onset of absorption is not obvious in the figure, but needs to be determined by curve fitting.

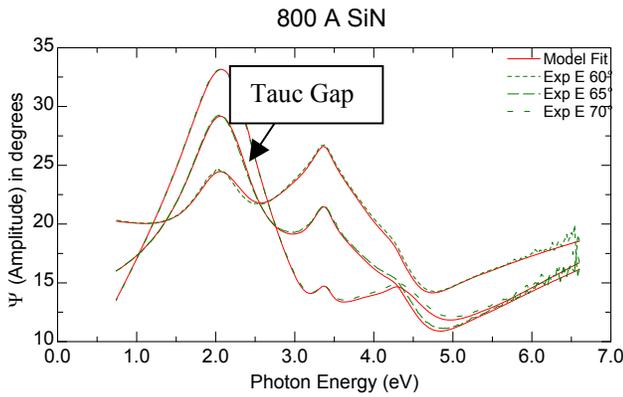


Figure 2: Psi (amplitude of the complex reflectance ratio) as a function of photon energy for an 800 Å SiN film on Si.

Fig. 3 shows ellipsometric data taken on a sample with 4000 Å SiN on a silicon substrate. The SiN film begins to absorb around 2.3 eV and becomes opaque around 3.5 eV. A thicker film contributes to less error in determining film thickness due to the higher number of optical interference fringes, over which data can be analyzed. Figure 4 is a simulation of reflectivity data taken on a 4000 Å SiN film. Typically, in a fabrication environment reflectivity measurements are utilized due to the minimal time required for analysis. However, when testing the robustness of the reflectivity model with different thickness values it took a 10% change in the thickness before the model began to fail. Furthermore, there were notable numerical instabilities for large changes in the thickness values. For more precise metrology, a single point ellipsometric measurement should probably be performed initially and modeled using the Tauc Lorentz dispersion model. The optical data determined from the ellipsometric model should then be used as the initial values for the reflectivity model on other points of the wafer (if across-wafer uniformity measurements are needed).

Ellipsometry measurements of silicon nitrides on GaAs substrates are equally straightforward, except for complications below the band gap (1.4 eV) for double-side polished wafers. [4] Although these measurements are possible, there is no added benefit for process control and one should consider the higher cost of GaAs substrates. Measurements on MIM product (bottom metal plate plus nitride dielectric, no top metal plate) are challenging, because the bottom plate has a rough surface and because the non-uniform composition of the bottom electrode makes the optical response difficult to interpret.

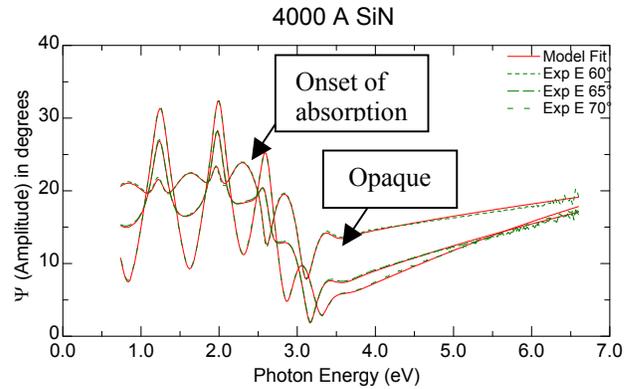


Figure 3: Psi (amplitude of the complex reflectance ratio) measured as a function of photon energy for a 4000 Å SiN film on Si.

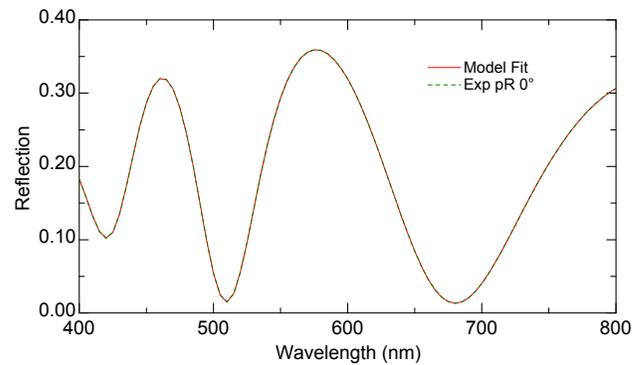


Figure 4: Simulated reflectivity data as a function of wavelength for a 4000 Å SiN film on Si.

NITRIDE COMPOSITION DETERMINED BY SE, RBS, AND RAMAN TECHNIQUES

The composition of Si-rich nitrides is difficult to measure. We have had some success using Rutherford backscattering (RBS), but the technique is tedious, destructive, and requires a large ion beam accelerator. Furthermore, it gives the elemental composition independent of the bond chemistry. We found that excess Si in the nitrides leads to amorphous Si-Si clusters, which have observable vibrations in UV Raman spectroscopy (325 nm excitation wavelength, above the band gap of the nitride). Since the a-Si Raman peak intensity depends on the absorption coefficient (which also depends on composition), we use the ratio of the a-Si to the Si-N vibration peak intensity as a parameter that scales with composition. This ratio scales inversely with the Tauc gap, determined by spectroscopic ellipsometry, which can be used as an in-line indicator for composition. The hydrogen content for the samples was determined using FTIR transmission spectroscopy from the intensity of the Si-H and N-H vibrational bands.

In Fig. 5, Raman data is shown from a set of LPCVD films with varying excess silicon concentrations. Note that samples 6 and 9 were measured to have the same silicon to nitrogen ratio by RBS, but differing Tauc gap values by

spectroscopic ellipsometry. Raman spectroscopy explains this discrepancy by demonstrating the films vibrational behavior is dependent on the content of a-Si clusters, leading to different Tauc gaps even for the same Si/N ratio.

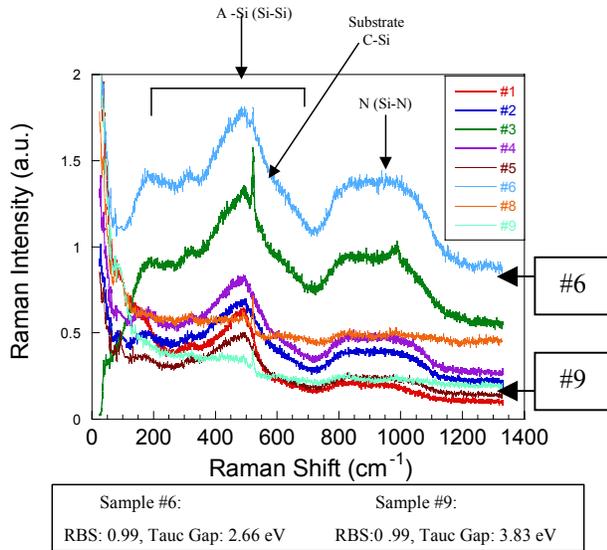


Figure 5: Raman intensity (a.u.) versus phonon energy (Raman shift) for nine LPCVD silicon nitrides on silicon substrates, showing the presence of amorphous Si-Si (200-500cm-1) and Si-N (800-1100cm-a) vibrations. This allows a quantitative determination of the a-Si content, which scales inversely with the Tauc band gap of the film.

CORRELATION BETWEEN FILM COMPOSITION AND ELECTRICAL PARAMETERS

After establishing spectroscopic ellipsometry (using the Tauc-Lorentz dispersion model) for robust inline process control for thickness and composition (with SEM, TEM, RBS, and Raman spectroscopy as offline backup techniques for confirmation of the inline results), we were able to investigate correlations between film composition and electrical parameters of the nitride dielectric. Three nitride layers with constant NH₃, but different silane flows were grown. Spectroscopic ellipsometry was completed on blanket films on silicon and electrical measurements were performed on MIM product wafers (Table 1). While this work is still ongoing, we found conclusive evidence for the increase of the capacitor breakdown voltage with decreasing excess Si content (higher band gap). The decrease in Tauc gap with increasing silane flow is associated with an increase of the extinction coefficient (higher absorption due to lower band gap) and a similar increase in the refractive index. However, no clear trends for specific capacitance were found.

Sample	Standard	Nitride +5% SiH4	Nitride - 5% SiH4
Thickness (Å)	1931	1927	1945
Tauc Gap (eV)	2.91	2.81	3.02
Capacitance (pF/mm ²)	322.73	322.56	317.81
Capacitor Breakdown Voltage (V)	59.86	57.70	63.76
Index of refraction at 400 nm	2.17	2.20	2.13
Extinction Coefficient at 400 nm	0.01	0.02	0.02

Table 1: Thickness, Tauc gap, capacitance, capacitor breakdown voltage, and complex refractive index for standard silicon nitride in comparison to nitride films with higher and lower silane flow. The capacitor breakdown clearly increases with increasing Tauc gap (decreasing Si/N ratio).

CONCLUSION

Spectroscopic ellipsometry is an excellent option for in-line quality control of silicon nitrides in compound semiconductor manufacturing. The analysis of nitrides on MIM capacitors has led to the correlation of electrical parameters and film composition.

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ACRONYMS:

- MIM: Metal-insulator-metal
- SEM: Scanning electron microscopy
- TEM: Transmission electron microscopy
- SE: Spectroscopic Ellipsometry
- RBS: Rutherford backscattering spectroscopy
- FTIR: Fourier transform infrared transmission spectroscopy