Characterization & Optimization of Low Stress PECVD Silicon Nitride for Production GaAs Manufacturing

Kenneth D. Mackenzie, Brad Reelfs, Michael W. DeVre, Russell Westerman, and David J. Johnson

Unaxis USA, Inc., 10050 16th North, St. Petersburg, FL 33716, Email: ken.mackenzie@unaxis.com, Tel: (727) 577-4999

Keywords: PECVD, silicon nitride, stress, manufacturing

Abstract

A designed experiment has been used to characterize and optimize a process for low-stress silicon nitride deposition using a commercial batch PECVD reactor developed for production GaAs manufacturing. The deposition process is based on a low power, low damage technique in which precise control of the silicon nitride stress is achieved by adjusting the relative concentration of N_2 and He carrier gases in the plasma. In the designed experiment, the influence of rf power, NH_3 gas flow rate, and also N_2/He concentration on film stress, deposition rate, refractive index, thickness non-uniformity, and wet etch rate resistance has been investigated. From the analysis of the design, a practical process regime has been established to achieve a highly uniform low-stress silicon nitride film with high wet etch resistance.

To understand the mechanism for stress control by the above technique, optical spectroscopic analysis of different N_2/He plasmas in the PECVD reactor has been performed. Results from this work are also presented.

INTRODUCTION

Plasma-enhanced chemical vapor deposition (PECVD) silicon nitride (SiN_x) is used extensively in the production of GaAs devices. PECVD is compatible with the low temperature constraints required for GaAs device manufacturing. With this technique, high quality SiN_x can be deposited at temperatures less than 400 °C. PECVD SiN_x is used in many different GaAs-based devices such as MESFETs, HBTs, and HEMTs. In these devices, PECVD SiN_x is typically used for passivation, encapsulation, and as a capping layer. In addition, the large dielectric constant of SiN_x makes it attractive for use as the intermetallic dielectric in MIM capacitors.

It is well recognized that the stress of the SiN_x layer in GaAs-based device structures can impact the electrical performance and lead to degradation. For GaAs MESFET and HEMT devices, it has been demonstrated that not only the magnitude of the stress but also the stress state, compressive or tensile, can affect the performance.[1] Stress-induced failure in SiN_x MIM capacitors has also been reported.[2] Therefore, the capability of tailoring the magnitude and state of the SiN_x stress required for a specific device structure is very important.

For SiN_x, a common technique to control the stress in a conventional 13.56 MHz parallel plate PECVD reactor is through the addition of low frequency power. At 13.56 MHz, SiN_x films prepared from standard gas mixtures of SiH_4, NH_3 and N_2 are typically tensile in nature. The added low frequency (< 1 MHz) component results in high energy ion bombardment of the growing SiN_x film and this results in a change of the stress state from tensile to compressive.[3, 4] In 1986, one of the authors of this paper, Johnson with co-workers at Unaxis USA Inc. (formerly Plasma Therm, Inc.) developed a He dilution method as a simpler alternative technique to control the stress of PECVD SiN_x. As illustrated in Figure 1, through the addition of He to the standard gas mixture of SiH_4, NH_3, and N_2, it is possible to control the stress from about 300 MPa, tensile through zero to about –300 MPa, compressive.

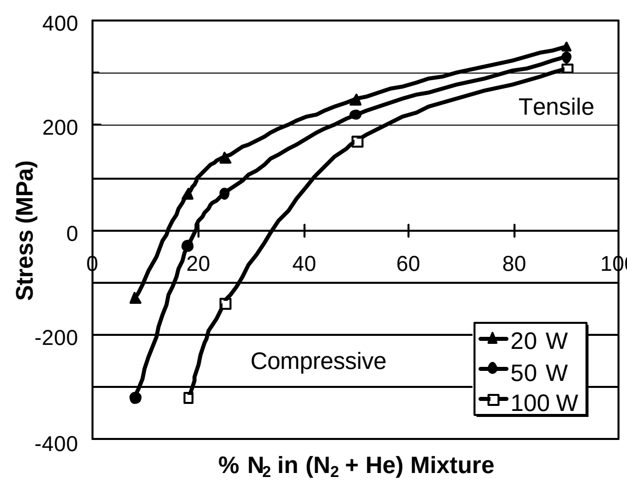

Figure 1. Stress control of PECVD SiN_x by the He dilution method. The SiN_x films were deposited at 250 °C at the different rf power levels indicated in the figure.

Plasma-induced damage during the SiN_x deposition process, resulting in physical and electronic degradation of GaAs devices is a very important issue.[5-7] Without the requirement of a low frequency power source, the possibility of damage is reduced with the He dilution method. The rf power density at 13.56 MHz is very low and typically is less than 50 mW/cm^2.

In this paper, we utilize a designed experiment (DOE) to characterize and optimize a low stress SiN_x process based on the He dilution method on a commercially available Unaxis PECVD production platform developed for high volume GaAs manufacturing. Further, to understand the mechanism involved in this technique for stress control, optical spectroscopic analysis of different He/N_2 plasmas in the PECVD reactor has been performed.

EXPERIMENTAL

All the SiN_x films were prepared on 100 mm Si from gas mixtures of SiH_4, NH_3, N_2, and He. The work was done on a Unaxis Versalock® PECVD system. This fully automated cassette-to-cassette system is capable of batch handling eight 100 mm GaAs or five 150 mm GaAs wafers. The PECVD reactor is of conventional parallel plate design and uses a 13.56 MHz rf power source to generate the plasma. The wafer temperature can be controlled over a range of 100 to 350 °C. To obtain a high yield and minimize system downtime, several features in the PECVD reactor have been implemented to maintain system cleanliness. For example, both the chamber walls and the upper gas distribution electrode of the reactor are heated to minimize particulate formation during the SiN_x deposition process. In addition, an automated plasma etchback sequencer interfaced to an *in situ* optical emission spectrometer is used to achieve and consistently maintain the reactor in a clean known state.

The DOE for the process optimization was setup and analyzed using Design Expert software from Stat-Ease, Inc. A two level full factorial design on three factors was constructed for the experiments. Two repeats of the center point were included. The three factors were NH_3 gas flow rate, $N_2/(N_2+He)$ gas flow ratio, and rf power. Table I shows these factors and the factor ranges used for the DOE. The SiN_x deposition runs were randomized. All films were deposited at 300 °C. The SiH_4 gas flow rate, process pressure, and the combined N_2 and He gas flow rates were held constant during the experiments. These parameters were 16 sccm, 1200 mTorr, and 3000 sccm, respectively.

TABLE I
FACTORS AND FACTOR RANGES FOR DOE

Factor	Low	High
NH_3 Gas Flow Rate (sccm)	5	9
N_2 concentration in N_2/He mixture (%)	10	20
rf Power (W)	75	125

The measured responses were refractive index, deposition rate, thickness non-uniformity, stress, and wet etch rate. A Gaertner model L116D-PC ellipsometer was used to determine the refractive index. The deposition rate and thickness non-uniformity were measured optically with a Nanometrics NanoSpec model 4150 metrology system. The thickness non-uniformity is defined as the thickness range divided by twice the mean thickness expressed as a percentage. The thickness was measured at 25 sites on each wafer. The edge exclusion was 6 mm. A buffered oxide etch (BOE) solution of 7:1 NH_4F : HF was used for the wet-etch rate measurements. The intrinsic stress of the deposited film was determined by the wafer bow technique. These measurements were made on a Tencor model P-2 long scan profiler.

For optical characterization, 1000 Å SiN_x films were deposited. Thicker films of about 5000 Å were deposited for the stress and wet etch rate measurements.

RESULTS AND DISCUSSION

A) Designed Experiment

In Table II, the general response trends from the analyzed DOE are summarized. The up and down arrows indicate the directional change in the response resulting from an increase in a process factor, NH_3 gas flow rate, N_2 concentration in He, or rf power. Double and single arrows respectively indicate a strong or weak dependence of a response on a factor over the range investigated.

TABLE II
GENERAL RESPONSE TRENDS FROM DOE

		Responses				
		Index	Dep Rate	Thickness Non Uniformity	Stress	Etch Rate
Factors	↑ NH_3	↓↓			↑↑	↑↑
	↑ %N_2/He	↓	↑	↓↓	↑↑	↑↑
	↑ RF	↓	↑↑	↑↑	↓↓	↓↓

Within the process regime investigated, the deposition rate increases linearly with increase in rf power and to a lesser extent with increase in N_2 concentration in the plasma. The deposition rate ranges from about 100 to 150 Å/min. As might be expected, the thickness non-uniformity is found to be dependent on rf power. However, the actual non-uniformity response is more complex as there exists an interaction between the rf power and the N_2/He concentration in the plasma. At high rf power, the thickness non-uniformity is strongly dependent on the N_2/He concentration. For example at 125 W rf power, the thickness non-uniformity decreases from about ±4 to ±2 %

as the N_2/He concentration increases from 10 to 20 %. At a lower rf power of 75 W, the interaction is weak and the typical thickness non-uniformity is about ±1.5 %. The etch rate resistance of all SiN_x films measured is high implying the films are dense. The wet etch rate for all films was less than 300 Å/min.

Figure 2 shows a contour plot for the refractive index. The refractive index decreases linearly with increase in NH_3 gas flow rate. This behavior is consistent with the commonly observed result that the SiN_x film stoichiometry is fundamentally controlled by the SiH_4/NH_3 gas flow ratio. The refractive index is also weakly dependent on the N_2/He concentration and rf power.

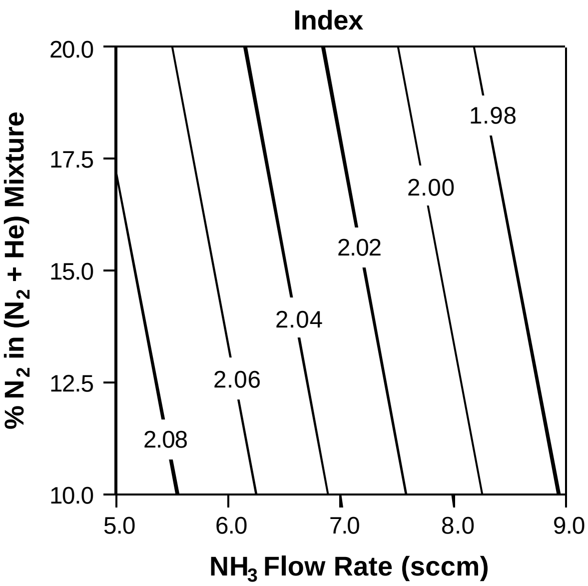

Figure 2. Refractive index as a function of NH_3 flow rate and N_2/He concentration.

As illustrated by the perturbation plot in Figure 3, all three factors have a major influence on the SiN_x film stress. In order to determine the optimum process conditions for a low stress SiN_x film, a transform was necessary to accurately model the stress response. The measured film stresses ranged from about 300 MPa, tensile to about -400 MPa, compressive.

In Table III, the film parameter criteria for a low stress SiN_x film are presented. Figure 4 maps out the predicted process space from the DOE for these criteria as a function of NH_3 gas flow rate and N_2/He concentration in the plasma. These results clearly indicate that a practical process regime exists to achieve a low-stress silicon nitride film to meet the desired criteria.

B) Stress Control Mechanism

Examination of the optical emission spectra of the deposition plasma provides important insight concerning the

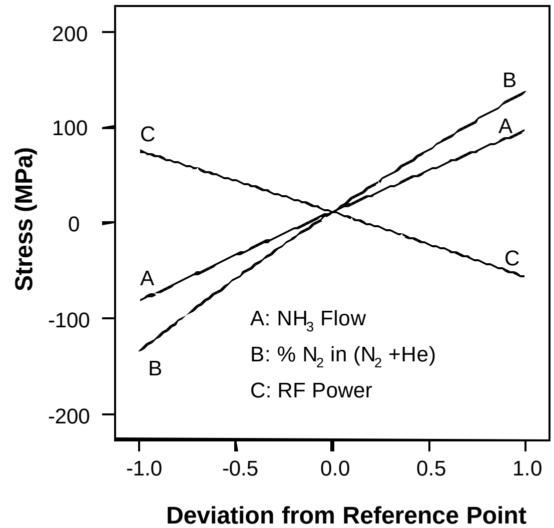

Figure 3. Perturbation effects on SiN_x stress from factors, NH_3 flow, N_2/He concentration, and rf power.

TABLE III
CRITERIA FOR LOW STRESS PROCESS OPTIMIZATION

Film Parameter	Range
Stress (MPa)	-100 to +100
Refractive Index	2.0 to 2.05
Thickness Non-Uniformity (%)	< ± 2.5
Wet Etch Rate (Å/min)	< 300

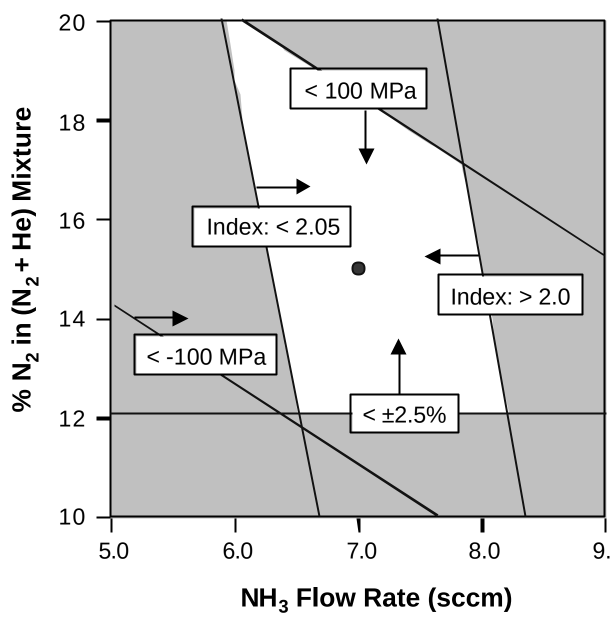

Figure 4. Overlay plot for an optimized low stress SiN_x process. Non-shaded area shows the optimized process regime. Point in center denotes the center point of the design.

mechanism responsible for compressive stress by the He dilution method. Shown in Figure 5 are two 13.56 MHz plasma spectra, pure N_2 and 10 % N_2/He. These correspond to deposition conditions associated with tensile and compressive films. Two emission lines at 391.4 nm and 427.8 nm are present in the 10 % N_2/He plasma that do not exist in the pure N_2 plasma. These lines are assigned to N_2^+ ions and indicate the presence of these ions in the 10 % N_2/He plasma. As shown in Figure 5, these N_2^+ spectral lines are also present in a 380 kHz pure N_2 plasma. SiN_x films prepared from SiH_4, NH_3, and N_2 at this lower frequency are confirmed to be in compression. This is strong evidence that the presence of N_2^+ ions is associated with the mechanism for film compression in the He dilution method. These results are consistent with the findings of Loboda and Seifferly[8] who inferred from residual gas analysis that He enhances the creation of N^+ species in the plasma resulting in increased incorporation of N bonding in the SiN_x film. This results in compressive stress due to the volume expansion of the SiN_x film.

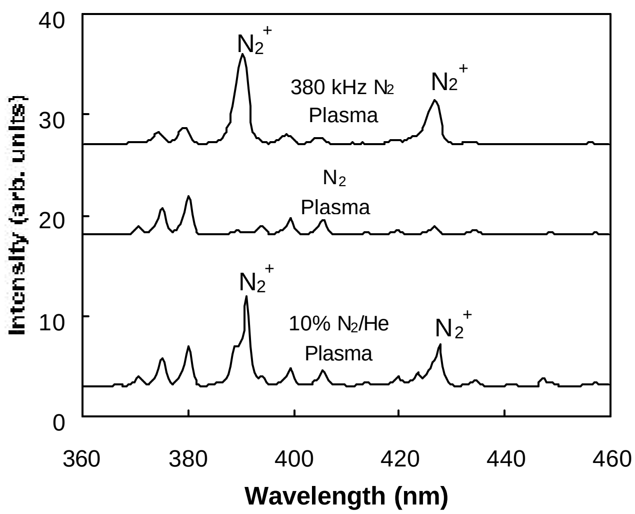

Figure 5. Optical Emission Spectra for 13.56 MHz plasmas of N_2 and 10 % N_2 in He. Also shown is a spectra of a low frequency, 380 kHz N_2 plasma. Spectra are displaced vertically for clarity.

CONCLUSIONS

Using the He dilution method, a low stress SiN_x process has been successfully optimized on a commercially available batch PECVD reactor. Highly uniform SiN_x films amenable to damage sensitive device fabrication have been demonstrated. Additionally these films have excellent wet etch resistance. Based on comparison of optical emission spectra, the mechanism responsible for compressive stress appears to be similar to that involved in low frequency PECVD SiN_x deposition.

ACKNOWLEDGEMENTS

The authors gratefully acknowledge the technical support from Lee Heckerd, Michael Moore, John Nolan, D. C. Cassaway, and B. Stites during the course of this work.

REFERENCES

[1] E. Y. Chang, G. T. Cibuzar, and K. P. Pande, IEEE Trans. Electron Devices **35**, 1412 (1988).

[2] D. Suh and J. Y. Kang, J. Vac. Sci. Technol. B **20**, 717 (2002).

[3] D. W. Hess, J. Vac. Sci. Technol A **2**, 244 (1984).

[4] D. L. Smith, A. S. Alimonda, and F. J. von Preissig, J. Vac. Sci. Technol. B **8**, 551 (1990).

[5] Y. B. Hahn, J. W. Lee, K. Mackenzie, D. Johnson, D. Hays, C. R. Abernathy, and S. J. Pearton, Electrochem. and Solid-State Lett. **1**, 230-232 (1998).

[6] J. W. Lee, K. D. Mackenzie, D. Johnson, R. J. Shul, Y. B. Hahn, D. C. Hays, C. R. Abernathy, F. Ren, and S. J. Pearton, J. Vac. Sci. Technol. A **17**, 2183 (1999).

[7] B. Luo, F. Ren, C. S. Wu, S. J. Pearton, C. R. Abernathy, and K. D. Mackenzie, Solid-State Electron. **46**, 1359-1365 (2002).

[8] M. J. Loboda and J. A. Seifferly, J. Mater. Res. **11**, 391 (1996).

ACRONYMS

PECVD: plasma enhanced chemical vapor deposition
SiN_x: silicon nitride
GaAs: gallium arsenide
DOE: Design Of Experiment
BOE: Buffered Oxide Etch
HBT: Heterojunction-junction Bipolar Transistor
HEMT: High Electron Mobility Transistor
MESFET: Metal Semiconductor Field Effect Transistor
MIM: Metal-Insulator-Metal
rf: radio frequency