Invited paper presented at 207th Electrochemical Society Meeting, Quebec City Canada, May 2005. Proc. Symp. Silicon Nitride and Silicon Dioxide Thin Insulating Films & Other Emerging Dielectrics VIII, **PV2005-01**, 148-159, Electrochemical Society, Pennington, NJ (2005).

STRESS CONTROL OF Si-BASED PECVD DIELECTRICS

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The Si-based dielectric films, silicon dioxide (SiO_2) and silicon nitride (SiN_x) prepared by plasma-enhanced chemical vapor deposition (PECVD), are used in device fabrication throughout the microelectronics and optoelectronics industries. The stress of these films can affect the electrical and optical characteristics as well as the integrity and reliability of the devices. In this paper, results are presented on a study of different techniques for stress control of these films. This includes discussion of a simple stress control method and the mechanism involved for SiN_x achieved by addition of He to the standard SiH_4, NH_3, and N_2 plasma chemistry. A designed experiment has been used to optimize a low stress SiN_x process based on this method on a commercial PECVD reactor developed for volume GaAs manufacturing.

INTRODUCTION

Key to the overall success of the plasma-enhanced chemical vapor deposition (PECVD) technique in the microelectronics and optoelectronics industries is the ability to deposit high quality and highly uniform Si-based films in large capacity reactors at suitably low temperatures (<400 °C) required for successful device fabrication. The Si-based dielectrics silicon dioxide (SiO_2) and silicon nitride (SiN_x) produced by this method, are used extensively in a wide and varying range of roles for the production of Si, III-V, and GaN semiconductor devices. Some examples of these roles include hardmask for dry etching, device encapsulation and passivation, intermetallic dielectric, gate dielectric, implant and diffusion mask, and diffusion barrier.

Mechanical stress within these Si-based dielectrics affects not only the performance of the film but also the overall device reliability. If the stress is too compressive, the film may blister or buckle. Whereas, if the stress is too tensile, the film may fracture. In both cases, the device integrity is compromised. The importance of this issue is highlighted by recent developments in microelectromechanical systems (MEMS) and planar lightwave circuits (PLCs). In some of these applications, there are requirements for PECVD SiO_2 films with thicknesses approaching 40 µm; about two orders of magnitude greater than is typically used for the examples cited earlier [1-4]. As illustrated in Figure 1, a consequence of the thicker films is the significant bowing or distortion of the wafer that occurs even with a moderate film stress of 300 MPa. This wafer distortion makes processing steps such as photolithography extremely challenging. In these applications, the deposition of dielectrics with near zero or at least very low stress is highly desirable.

Stress can also influence the electrical and optical performance of the devices. For example, it is well documented that the stress of the SiN_x layer in GaAs-based devices can impact the electrical performance and lead to degradation and possible premature failure of the device. For GaAs high electron mobility transistors (HEMTs) and metal-semiconductor field effect transistors (MESFETs), it has been demonstrated that not only the magnitude of the stress of the PECVD SiN_x passivation layer but also the stress state, compressive or tensile, can affect the device performance [5]. Stress-induced failure in SiN_x metal-insulator-metal (MIM) capacitors has also been reported [6]. Stress can also influence the optical performance of PLCs. In this application, the PECVD process needs to be optimized to minimize the undesirable stress-induced birefringence and optical mode distortion that may occur [7].

The capability of tailoring both the magnitude and where possible the stress state of the Si-based dielectric for each specific device application is extremely important. Surveying the many different applications for these dielectrics, a zero or slightly compressive film stress are the typical requirements. These films must also be mechanically stable [8-10]. The PECVD deposition parameters i.e. temperature, pressure, gas chemistry, power density and frequency of the rf excitation source, all influence the stress of the deposited film. In this paper, we will present and discuss two different practical techniques for stress control of PECVD SiO_2 and SiN_x. Namely, stress control by adjustment of film stoichiometry and by the addition of low frequency power in the PECVD reactor. In addition, a simple alternative technique to control the stress of PECVD SiN_x by adjustment of the carrier gas chemistry will also be presented. The applicability of this technique for volume manufacturing will also be demonstrated.

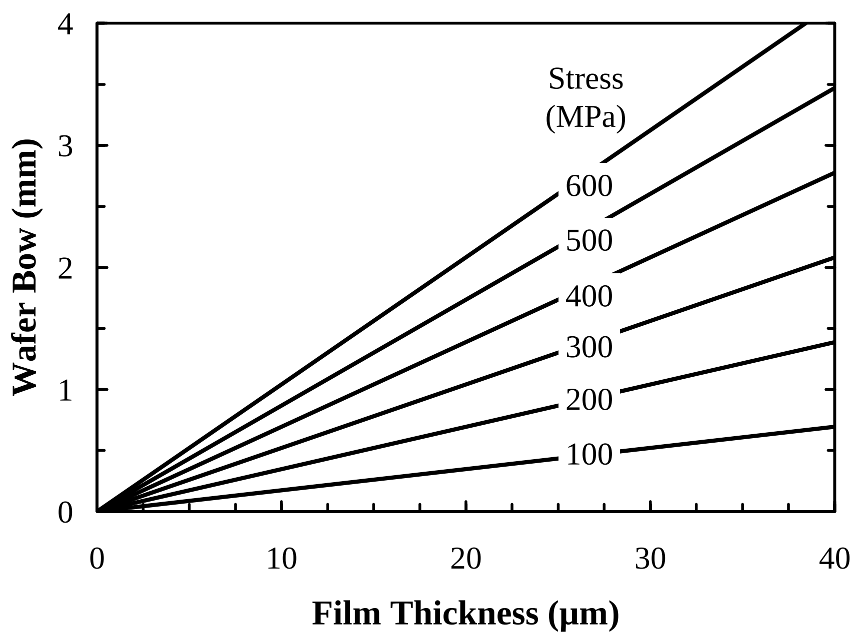


Figure 1. Calculated maximum bow of a 150 mm Si wafer *versus* deposited film thickness for the various film stress values indicated. (Bow estimated from Stoney's equation [11] using the parameters: wafer thickness 650 μm, Poisson's ratio 0.22, and Young's modulus 180 GPa).

EXPERIMENTAL

All the SiO_2 and SiN_x films for this study were prepared on a Unaxis PECVD reactor at deposition temperatures of 250 and 350 °C. The PECVD reactor is of a conventional parallel plate design and uses a high frequency (13.56 MHz) power source to generate the plasma. For some of the stress control experiments, an additional low frequency (380 kHz) power source was coupled with the 13.56 MHz power source into the plasma. Both power sources operate in continuous wave mode.

For the stress-stoichiometry experiments, three types of films, SiO_2, SiN_x, and silicon oxynitride, SiO_yN_x were deposited. The SiO_2 films were prepared from a plasma chemistry of SiH_4, N_2O, and N_2. The film stress was investigated as a function of N_2O/SiH_4 gas flow ratio. The SiN_x films were prepared from a plasma chemistry of SiH_4, NH_3, and N_2. The film stress was investigated as function of NH_3/SiH_4 gas flow ratio. The SiO_yN_x films were prepared from mixtures of SiH_4, NH_3, N_2O, and N_2. In this case, the film stress was investigated as a function of N_2O/NH_3 gas flow ratio. For all three types of film, a dilute silane source, 2% SiH_4 in N_2 was used. The deposition temperature and the process pressure were 250 °C and 900 mTorr, respectively. The applied rf power density was kept low at less than 80 mW/cm^2. Deposition rates for these processes ranged from about 100 to 1000 Å/min.

For the low-frequency experiments, SiO_2 and SiN_x films were deposited from plasma chemistries of SiH_4, N_2O and SiH_4, NH_3, N_2, respectively. In each case, the total applied power was held fixed and the film stress was investigated as the percentage of low frequency power was varied. The deposition temperature and the process pressure were 350 °C and 1200-1500 mTorr, respectively. The total applied power density was about 500 mW/cm^2. The gas flow ratios, N_2O/SiH_4 and NH_3/SiH_4 for the respective SiO_2 and SiN_x films were fixed at 30 and 0.9. An undiluted silane source was used to achieve higher deposition rates from about 500 to 3000 Å/min.

All the films for this study were deposited on 100 mm and 150 mm Si wafers. The typical thickness of the films was in the range from 1000 to 20,000 Å. The deposited films were characterized by refractive index, stress, deposition rate, deposition uniformity, and wet-etch rate. A Gaertner model L116D-PC ellipsometer was used to determine the refractive index at 633 nm. The film thickness and thickness uniformity were measured optically with a Nanometrics NanoSpec model 4150 metrology system. A buffered HF etch solution of 7:1 NH_4F:HF was used for the wet-etch rate measurements. The film stress was determined by the wafer bow technique. These measurements were carried out on a Tencor model P-2 long scan profiler. The film stress was estimated from Stoney's equation [11].

RESULTS AND DISCUSSIONS

Stress Control by Adjustment of Stoichiometry

The composition of PECVD SiO_2 and SiN_x depends fundamentally on the respective N_2O/SiH_4 and NH_3/SiH_4 gas flow ratios. As illustrated in Figures 2 and 3, by lowering the gas flow ratio which results in a slightly Si-rich film, the stress decreases for both types of film. In the case of SiO_2, the film stress, which is compressive, decreases monotonically from about -300 MPa to a low value of -50 MPa as the N_2O/SiH_4 ratio decreases from about 80 to about 10. This corresponds to a change in refractive index from 1.47 to about 1.50. For SiN_x, the film stress, which is tensile, can be adjusted from about 400 MPa to about 200 MPa as the NH_3/SiH_4 ratio decreases from about 0.9 to 0.5. This corresponds to a change in refractive index from about 1.9 to 2.1.

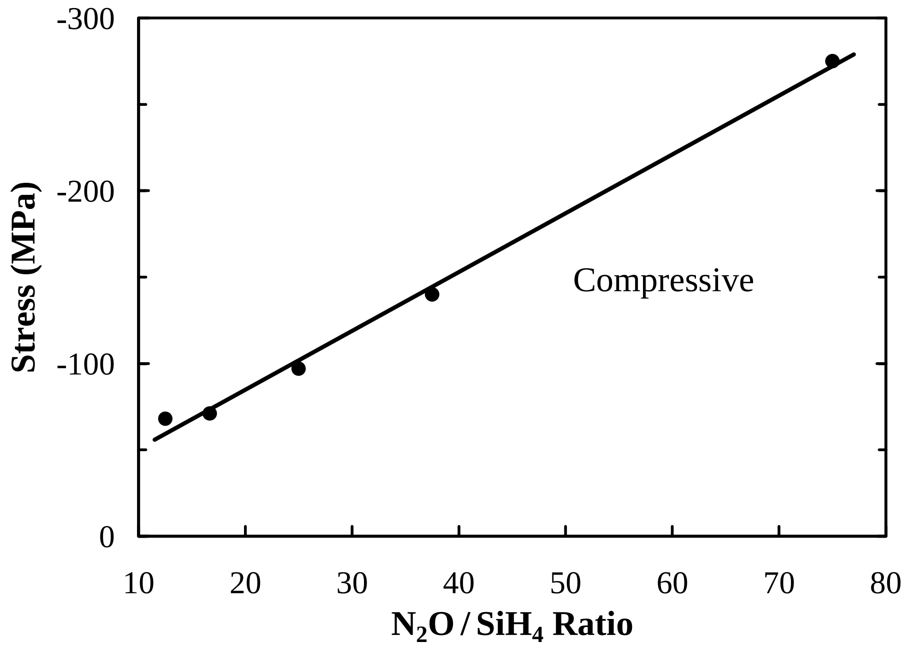


Figure 2. Stress of PECVD SiO_2 films deposited at 250 °C as a function of N_2O/SiH_4 gas flow ratio. The deposition rates were about 500 Å/min.

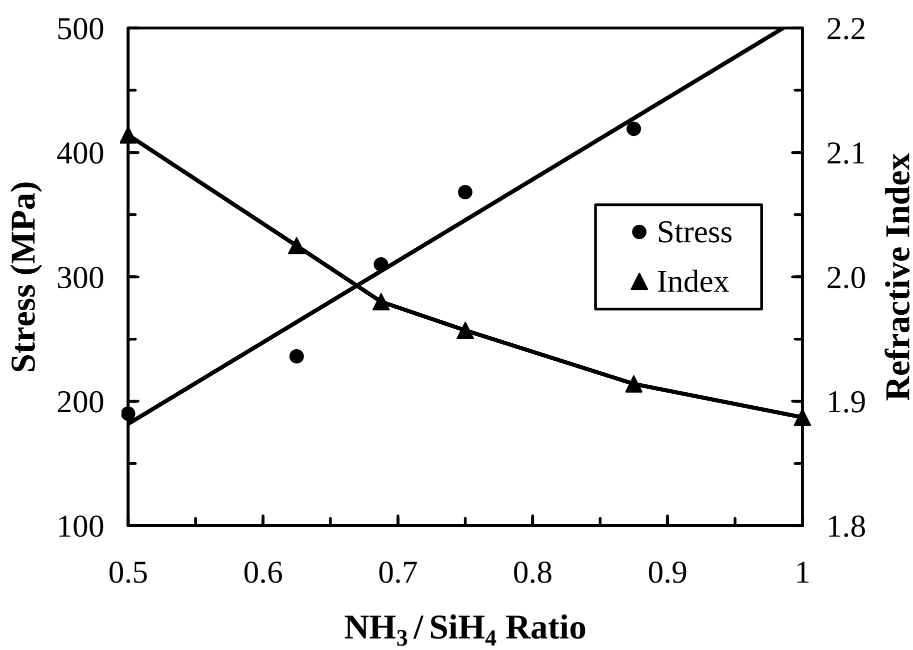


Figure 3. Tensile stress and refractive index of PECVD SiN_x films deposited at 250 °C as a function of NH_3/SiH_4 gas flow ratio. Deposition rates were about 120 Å/min.

By adding N_2O to the SiN_x plasma chemistry of SiH_4, NH_3, and N_2, it is possible to form the alloy, SiO_yN_x. Altering the SiO_yN_x stoichiometry by adjustment of the N_2O/NH_3 gas flow ratio extends the range of stress control from compressive (SiO_2) through zero to tensile (SiN_x).

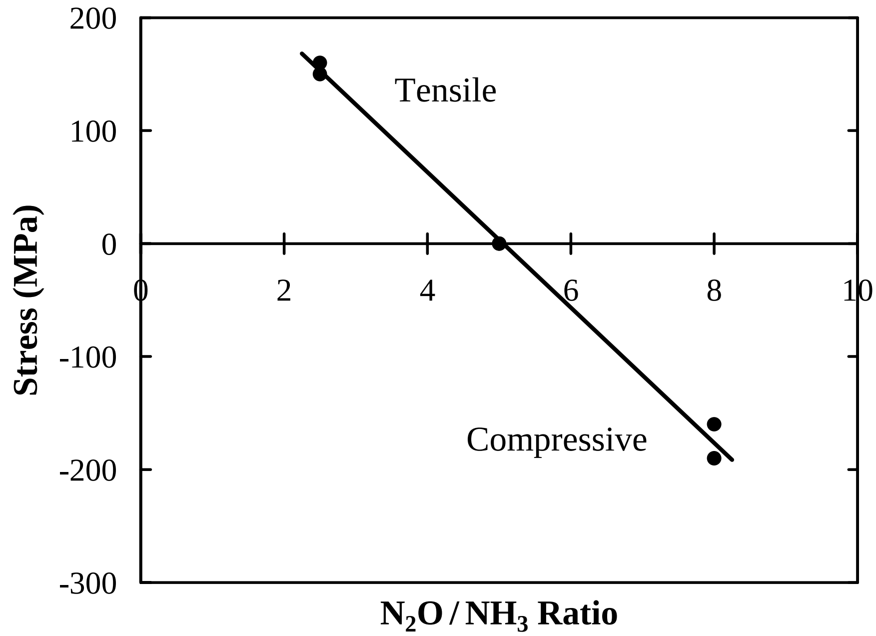


Figure 4. Variation of stress of PECVD SiO_yN_x films deposited at 250 °C with N_2O/NH_3 gas flow ratio. The deposition rates were about 300 Å/min.

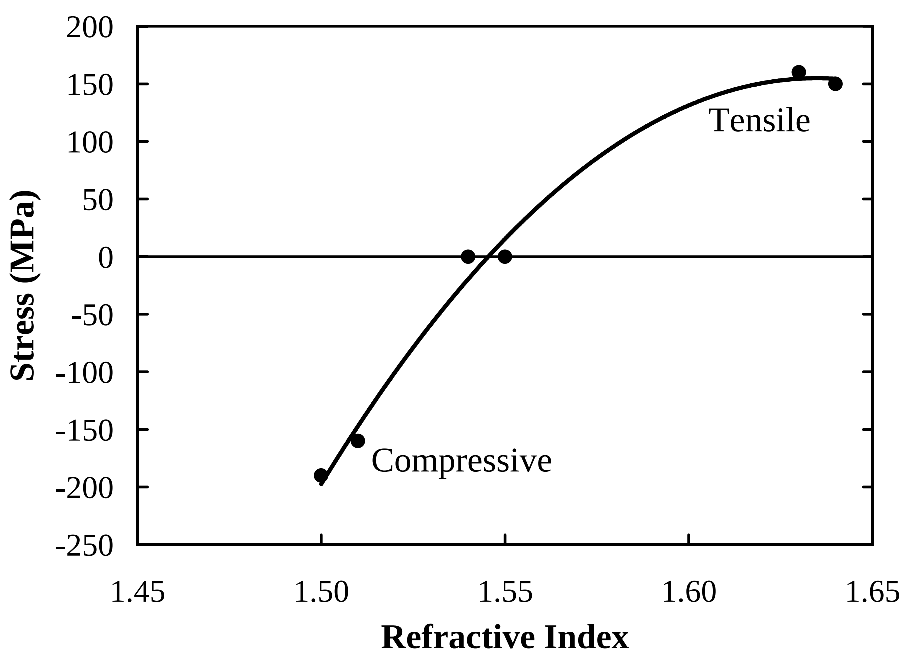


Figure 5. Stress of the PECVD SiO_yN_x films from Figure 4 *versus* refractive index.

As illustrated in Figure 4, it is relatively easy to achieve zero stress films by this method. In this particular example, zero stress occurs at a N_2O/H_3 ratio of 5. Referring to Figure

5, this corresponds to a film refractive index of 1.55. Low stress SiO_yN_x prepared by this technique has been successfully implemented in many different applications such as hardmasks on X-ray membrane masks [12].

Detailed explanations for the chemical origin of the compressive or tensile nature of the stress in SiO_2, SiN_x, and SiO_yN_x films can be found in References [13-15].

Stress Control by Addition of Low Frequency Power

A common technique to control the film stress of PECVD Si-based dielectrics in a conventional parallel plate reactor operating at 13.56 MHz is through the addition of low frequency power. For example, as illustrated by the results in Figure 6, the addition of low frequency (380 kHz) power causes a controlled change in the stress state from tensile to compressive for SiN_x films. As shown in Figure 7, the addition of low frequency power also places SiO_2 in a compressive state. The origin of this behavior is well documented, both experimentally and theoretically [16-18]. The high-energy ion bombardment that occurs through the addition of low frequency (< 1 MHz) is responsible for the compressive stress of the deposited films. The densification of the film caused by the ion bombardment causes the film to expand against its volume and hence results in a compressive stress.

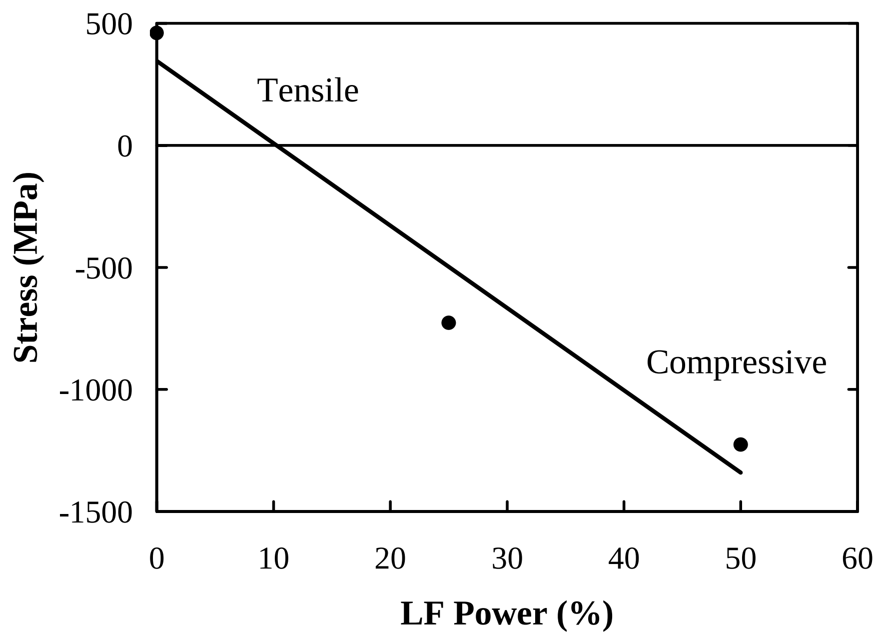


Figure 6. Stress of PECVD SiN_x films deposited at 350°C as a function of added low-frequency power. The deposition rates were between 500 and 1000 Å/min.

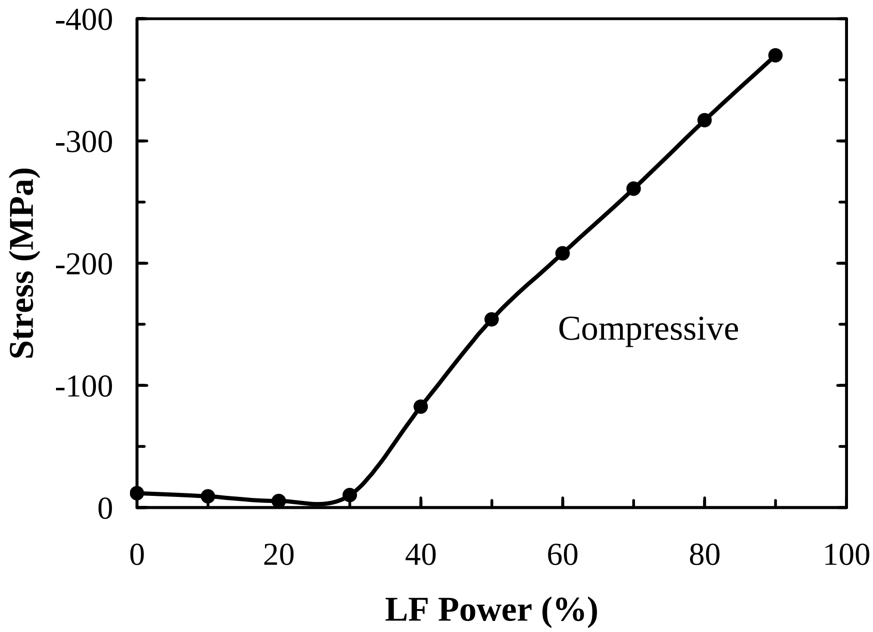


Figure 7. Stress of PECVD SiO_2 films deposited at 350°C as a function of added low-frequency power. The deposition rates were about 3000 Å/min.

In addition to the stress control afforded by this technique, another significant advantage is the densification that can be achieved for films deposited under high rate conditions. In conventional 13.56 MHz PECVD, the quality of films deposited under high rate conditions is generally inferior to films deposited at a lower rate. Typically, films prepared under high rate conditions have a high wet-etch rate indicative of much lower density and possible porosity. Figure 8 shows the wet-etch rate data for the SiN_x films from Figure 6, which were deposited at relatively high rates of 500 to 1000 Å/min. The wet-etch rate decreases by almost an order of magnitude with increasing applied low frequency power. Similarly, as shown in Figure 9 for SiO_2 films from Figure 7, which were deposited at a high rate of 3000 Å/min, the wet-etch rate decreases by approximately a factor of 3 with increasing applied low frequency power. The very low stress (< -25 MPa) observed for SiO_2 deposited at low frequency power levels less than 30%, combined with a high wet-etch rate, is indicative of highly porous films.

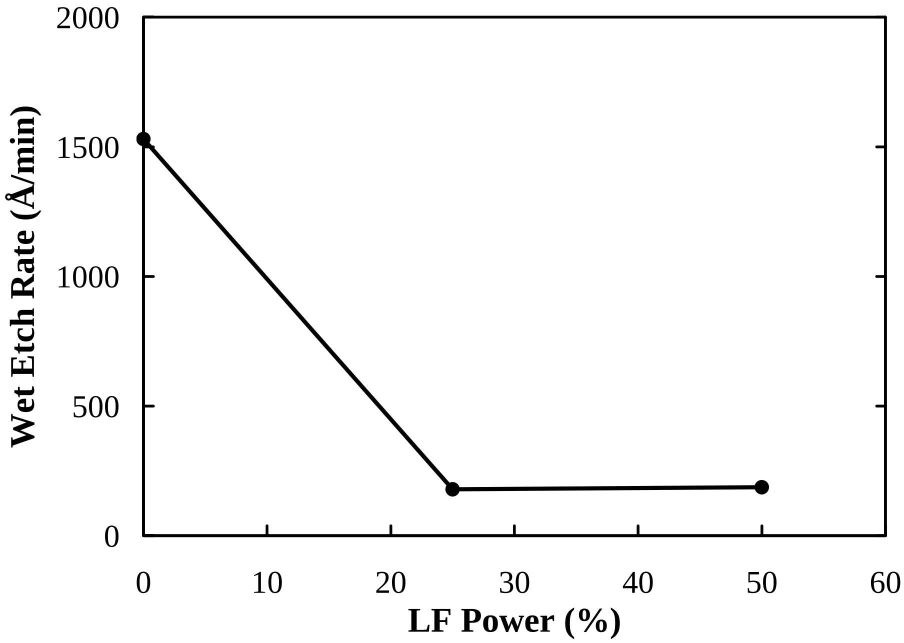


Figure 8. Variation of wet-etch rate of PECVD SiN_x deposited at 350 °C with added low-frequency power. The data refers to the same series of films used in Figure 6.

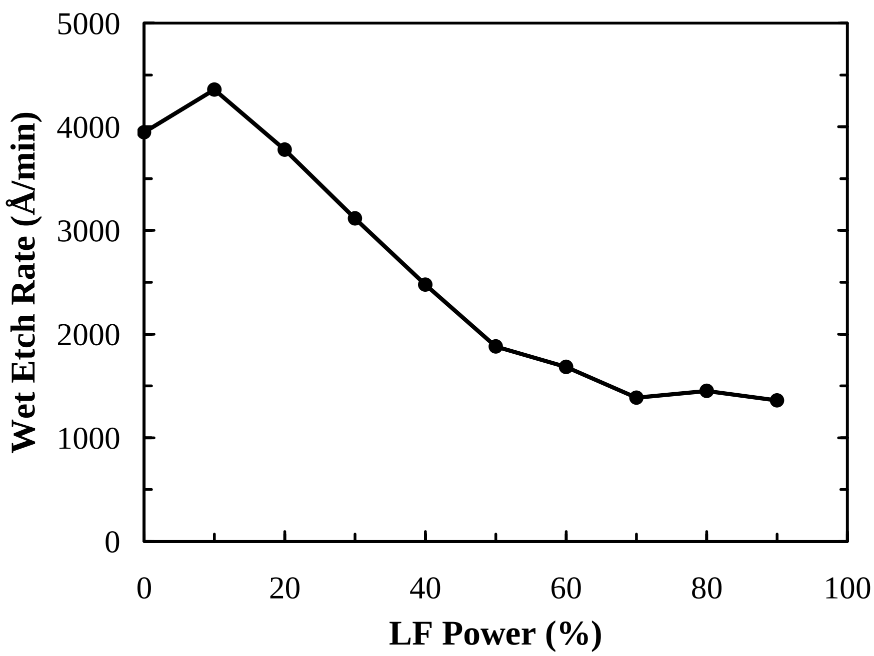


Figure 9. Variation of wet-etch rate of PECVD SiO_2 deposited at 350 °C with added low-frequency power. The data refers to the same series of films used in Figure 7. For reference, the measured wet-etch rate of thermally grown SiO_2 is about 800 Å/min.

Stress Control of SiN_x by Adjustment of the Carrier Gas Chemistry

As illustrated in Figure 10, it is possible, through the addition of He carrier gas to the standard admixture of SiH_4, NH_3, and N_2, to control the stress of SiN_x from about 300 MPa tensile through zero to about -300 MPa, compressive. Without the requirement of a low frequency source, the possibility of plasma-induced damage is reduced with this He dilution method, which operates at a low rf power density of less than 50 mW/cm^2.

Plasma-induced damage during the SiN_x deposition process, resulting in physical and electronic degradation is an extremely important issue for III-V and GaN devices [19-24]. Interesting work on this topic was recently reported by Tan et al., [25]. In their work, it was found that compressive SiN_x films prepared by the He dilution method for passivation resulted in no damage of the underlying AlGaN/GaN heterostructure field-effect transistors. When the compressive SiN_x passivation layers were prepared by addition of low frequency power to the plasma, severe nonreversible damage occurred to the AlGaN/GaN transistors due to the high-energy ion bombardment.

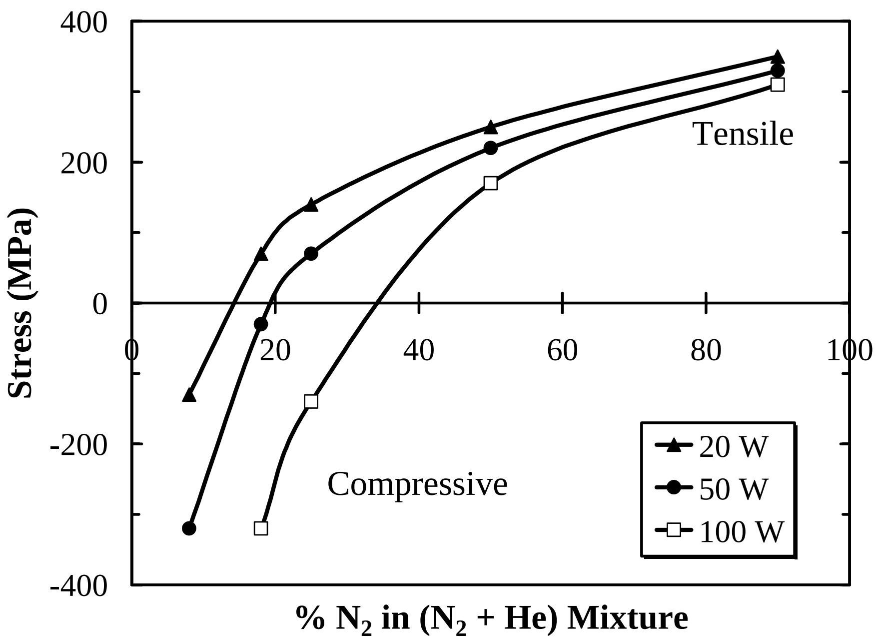


Figure 10. Stress control of PECVD SiN_x by the He dilution method. The SiN_x films were deposited at 250°C at the different rf power levels indicated in the figure. The deposition rates were about 120 Å/min.

Examination of the optical emission spectra of the deposition plasma provides important insight concerning the mechanism responsible for compressive stress by the He dilution method. Shown in Figure 11 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 [26]. As shown in Figure 11, 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 [27] 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.

The exact role of He in the plasma deposition chemistry is the subject of continued interest [13, 28-32].

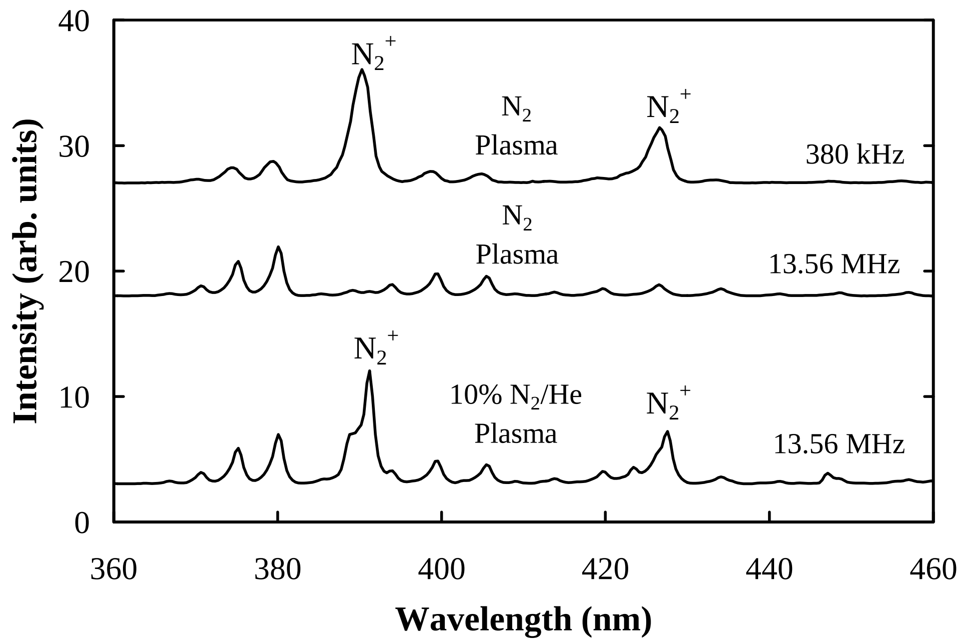


Figure 11. Optical Emission Spectra for 13.56 MHz plasmas of N_2 and 10% N_2 in He. Also shown is a spectra of a low frequency, 380 kHz N_2 plasma. Spectra are displaced vertically for clarity.

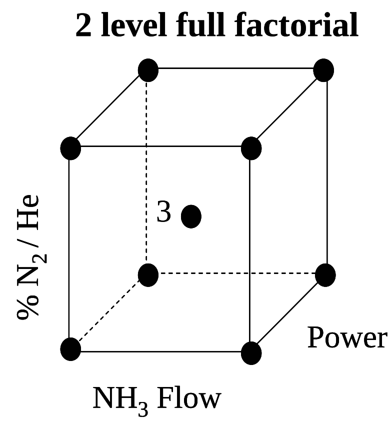
<u>Optimization of Low Stress Silicon Nitride based on the He Dilution Method</u>

Table I summarizes the typical film property requirements for a low stress SiN_x film. As outlined in Figure 12, a two level full factorial designed experiment has been implemented to optimize a low stress SiN_x process on a Unaxis large capacity batch PECVD reactor designed for volume GaAs manufacturing [33].

TABLE I. 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 13 maps out the predicted process space from the designed experiment for the criteria in Table I 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.



Factors	
NH_3 Gas Flow	5 – 9 sccm
%N_2 in (N_2 + He)	10 – 20 %
rf Power (W)	75 – 125 W

Responses
Stress
Refractive Index
Thickness Non-uniformity
Wet Etch Rate
Deposition Rate

Constants	
SiH_4 Gas Flow	16 sccm
Process Pressure	1200 mTorr
Total N_2, He Gas Flow	3000 sccm
Temperature	300 °C

Figure 12. Factors, responses, and constants for the two level full factorial designed experiment for optimization of the low stress SiN_x process.

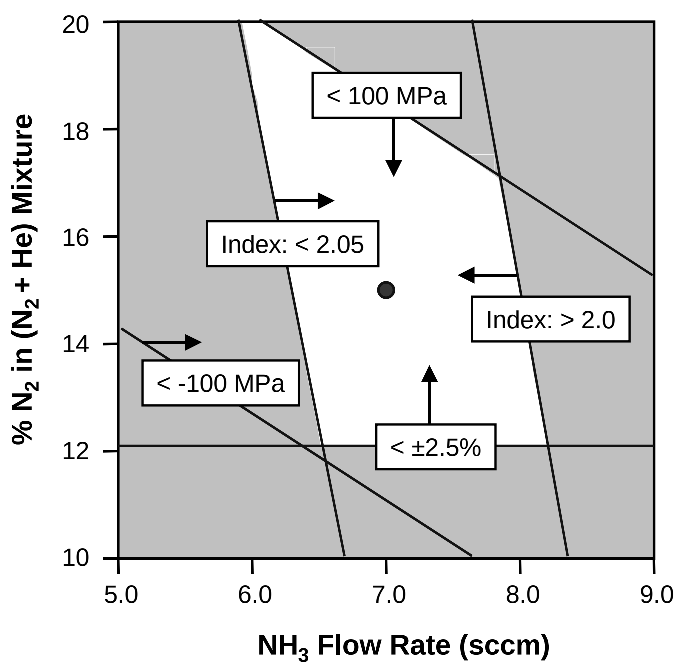


Figure 13. Overlay plot for an optimized 300°C low stress SiN_x process. Non-shaded area shows the optimized process regime. Point in center denotes the center point of the design.

CONCLUSIONS

PECVD is a highly versatile technique for stress control of Si-based dielectrics. The stress can be customized for a particular application by simple adjustment of the gas composition in the plasma or by addition of low frequency power in a conventional 13.56 MHz PECVD reactor.

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, John Nolan, Michael Moore, and Michael Todd.

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