PECVD Silicon Nitride Film Property and Pre-deposition Surface Treatment Effects on MIMCAP Reliability for InGaP/GaAs HBT Applications

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Abstract

We report on the development of two silicon nitride films deposited at 300°C using PECVD processes for applications in high density metal-insulator-metal capacitor dielectrics in InGaP/GaAs devices. The two films are compared to an existing silicon nitride film previously developed on a different deposition tool under different process conditions.

In order to understand the mechanisms of their individual reliability performance, specifically in pressure cooker test (PCT/autoclave), the films are characterized for composition and chemical bonding using HFS and FTIR. The characterization indicated that in addition to film breakdown properties and refractive indices, the film composition itself also needs to be controlled in order to obtain films that can successfully withstand the PCT test. Finally, we report on the benefits of pre-deposition treatments of metal electrodes, which are believed to enhance nitride adhesion and improve the consistency of device reliability performance.

INTRODUCTION

Silicon nitride is a versatile material widely used in compound semiconductor devices as a passivation layer, an interlayer dielectric, and as a capacitor dielectric, largely due to its film properties being easily controlled and modified by varying its deposition methods and conditions [1]. Plasma Enhanced Chemical Vapor Deposition (PECVD) is among the most commonly used deposition methods because of its compatibility with GaAs processing, and its ability to produce good silicon nitride film characteristics, and electrical properties. Silicon nitride employed as a MIMCAP dielectric provides good conformality, a relatively high dielectric constant, and a high breakdown field [2-5].

EXPERIMENTAL

A. Film Development

In this study, we have developed two 900 Å silicon nitride films for high-density MIM capacitors for InGaP/GaAs HBT applications. The films were developed using a commercial multi-station PECVD tool at 300 °C and compared with silicon nitride films previously developed on a different tool under different conditions. For both tools, the gases used for silicon nitride deposition are silane, ammonia and nitrogen.

Silicon nitride film development was performed by varying the silane flow, and the ammonia to silane ratio, as well as by varying pressure, total RF power, and LF to total RF power ratio. It is reported in the literature [6] that better electrical breakdown performance is associated with decreasing Si/N ratio, i.e. lower refractive index, therefore the refractive index is kept low in the development of new films.

B. Film Characterization

Physical and optical characterizations of the silicon nitride films, including thickness, thickness uniformity, refractive index, stress and deposition rate were conducted. Thickness, refractive index, and their respective uniformity were measured using a Rudolph FE-VII ellipsometer using 49 pts with 3 mm edge exclusion, while the in-film stress was measured using a Flexus stress gauge. Additionally, chemical bonding, elemental composition and density of the films were characterized by FTIR and Hydrogen Forward Scattering (HFS). FTIR was performed with the microscope in transmission mode.

HFS gives the elemental composition independent of the bond chemistry. In the HFS experiment, a detector is placed 30° from the forward trajectory of the incident He^{++} ion beam and the sample is positioned so that the incident beam strikes the surfaces 75° from normal. In this geometry it is possible to collect light atoms, namely hydrogen, forward-scattered from a sample after collisions with the probing He++ ion beam. A thin absorber foil is placed over the detector to filter out He^{++} ions that are also forward scattered from the sample. Hydrogen concentrations are determined by comparing the number of hydrogen counts to that obtained from reference samples after normalizing by the stopping powers of the different materials. A hydrogen implanted silicon sample and a geological sample, muscovite, are used as references. During the HFS acquisition, the backscattering spectra are acquired using the 160° angle detector (with the sample in forward scattering orientation). The HFS and RBS spectra are fit by applying a theoretical model and iteratively adjusting elemental concentrations and thickness until good agreement is found between the theoretical and the experimental spectra.

Hydrogen loss is checked in the analyzed region and a short acquisition time is used when necessary. To account for surface hydrogen due to residual moisture or hydrocarbon adsorption a silicon control sample is analyzed together with the actual samples and the hydrogen signal from the control sample is subtracted from the spectra obtained from the actual samples.

Electrical characterization of the films includes the capacitance and its uniformity, I-V characteristics, and breakdown voltage. The film breakdown voltage is both measured on blank nitride film using Mercury probe technique and on fabricated MIMCAP structures. The bottom and top metal electrodes are formed by gold.

The standard PCT test was done at 121°C, 100% relative humidity and pressure 1 atmosphere above ambient for 96 hours.

RESULTS AND DISCUSSION

A. Film Characteristics

The newly developed films, film 1 and film 2, have refractive indices of 1.82 and 1.83; closely matched to the existing film — film 3. The new films yield an improved uniformity and process control characteristics, including thickness, stress, refractive index, and low particle density. Selected film characteristics are listed in Table I.

TABLE I
SELECTED FILM CHARACTERISTICS OF THE THREE FILMS

Films	**Film 1**	**Film 2**	**Film 3**
Mean Thickness (Å)	910	910	910
Thickness Uniformity (%)	0.48%	0.32%	1.04%
Refractive Index	1.82	1.83	1.82
R.I. Uniformity (%)	0.09%	0.04%	0.10%
Film Stress (MPa, tensile)	190	400	400
Dep Rate (Å/min)	443	750	488

B. Capacitance and Breakdown Voltage Studies

The MIMCAPs fabricated with the above films have a similar capacitance density approx. 0.7 fF/um^2. The dielectric constant ratio of film 2 and film 3 with respect to film 1 are 0.95 and 0.94, respectively. All films exhibit a breakdown field of > 8 MV/cm on fabricated 20 pF MIMCAPs. I-V characteristics of MIMCAPs were measured to 20 V to screen pre-mature failures prior to reliability tests. None of the films showed any premature failures.

C. FTIR Analysis

Each wafer was measured near its center and near one edge with FTIR to assess the film consistency across the wafers. Figure 1 shows the FTIR spectra of the silicon nitride films as deposited on a Si substrate. The FTIR characterization indicates that film 1 (red), which suffers severe breakdown voltage degradation after PCT, contains more NH and less SiH bonds than film 2 (blue) and film 3 (green), which successfully passed PCT. Film 2 and film 3 exhibit identical spectra, as can be seen in figure 1, and all three films have similar Si-N bond intensity.

The FTIR average integrated band intensities for the three films are summarized in Table II. Table III summarizes the estimated SiH and NH areal bond densities, based on the method of Lanford and Rand. Table IV shows the volumetric densities converted from areal density using film thickness measured with ellipsometer. As shown in Table IV, film 2 and 3 exhibit similar NH and SiH bond concentrations and approximately the same NH to SiH ratio, while film 1 has less SiH, more NH, and a much higher NH to SiH ratio.

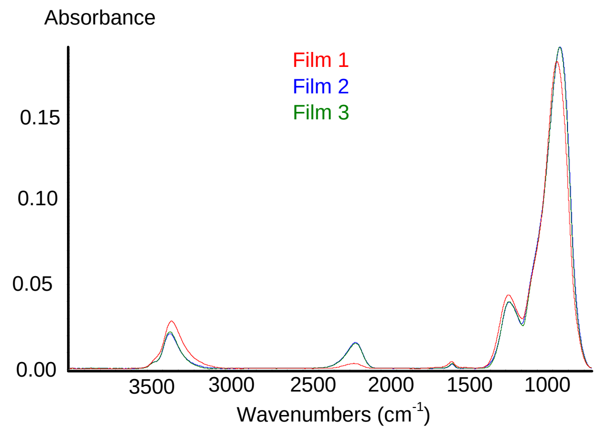

FIGURE 1. FTIR Spectra overlay. Red: film 1, which failed PCT test. Blue and green: films 2 and 3, respectively, which passed PCT test.

TABLE II
FTIR AVERAGE INTEGRATED BAND INTENSITIES IN THE FILMS

Bond	**Absorption Peaks (cm^{-1})**	**Film 1**	**Film 2**	**Film 3**
OH (possible)	3470	--	0.02	0.02
NH	3350	4.7	3.04	3.01
SiH	2100-2175	0.38	2.1	1.87
NH_2	1550	0.13	0.09	0.11
SiNH	1180	2.7	2.2	2.3
SiN	825-875	41	42.4	41.4

TABLE III
CALCULATED BOND CONCENTRATIONS (BONDS/CM^2)

Bond	Film 1	Film 2	Film 3
NH	8.90E+17	5.70E+17	5.20E+17
SiH	5.10E+16	2.80E+17	2.50E+17

TABLE IV
CALCULATED VOLUMETRIC BOND CONCENTRATIONS (BONDS/CM^3)

Bond	Film 1	Film 2	Film 3
NH	3.40E+22	2.10E+22	1.90E+22
SiH	1.90E+21	1.04E+22	9.30E+21

D. HFS Analysis

Figure 2 and 3 show the RBS and HFS spectra obtained from film 2 as an example. The theoretical depth profile illustrated in figure 4 is used to generate the theoretical spectra in figure 2 and 3. As seen in figure 4, the elemental concentrations of Si, N and H are relatively constant with depth. The atomic concentrations of the three films are summarized in Table V. The atomic densities, adjusted for the real film thickness measured by ellipsometer, are also listed in the Table, along with the mass densities calculated from the atomic density and the composition of the three films.

HFS characterization indicates that the failed film is the closest to Si_3N_4 stoichiometry with Si to N ratio of 2.9/4. Film 2 and film 3 are Si rich with Si to N ratio at 3.4~3.5/4. All three films contain around 34 at.% hydrogen. Although the refractive index is mainly determined by the Si/N ratio in the film, refractive index can also be influenced by the density, which may explain the similar refractive index for all three films.

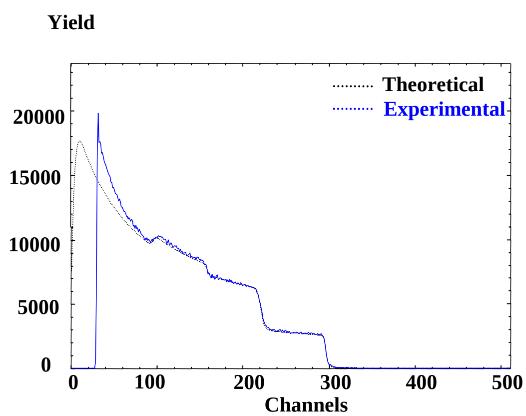

FIGURE 2. RBS spectra collected on film 2 using the 160 degree detector.

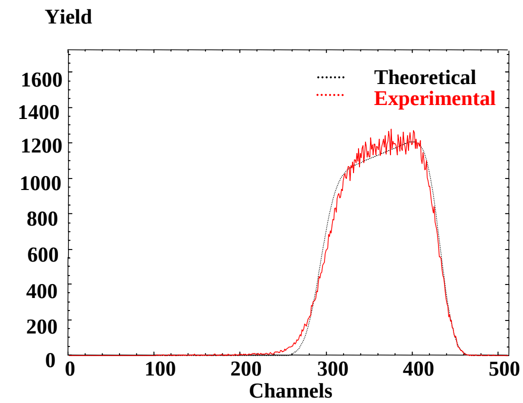

FIGURE 3. HFS spectra collected on film 2 using the 30 degree detector.

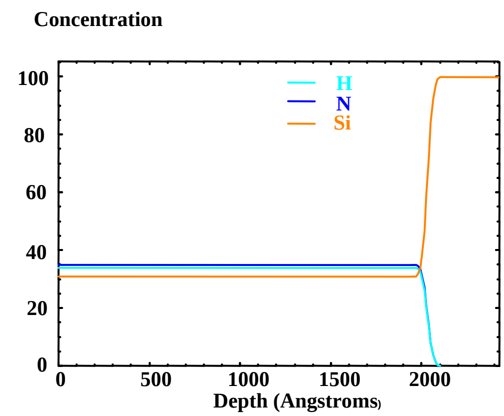

FIGURE 4. Theoretical depth profile of film 2 based on fit to experimental data from both RBS and HFS spectra.

TABLE V
ATOMIC CONCENTRATIONS AND DENSITY OF FILMS MEASURED BY HFS

Films	Film 1	Film 2	Film 3
H	33.5	34	34
N	38.5	35	35.7
Si	28	31	30.3
Density (at/cc)	9.06E+22	8.29E+22	8.42E+22
Denstity (g/cc)	2.04	1.92	1.93

E. Reliability Studies

A pressure cooker test (PCT) was performed on bare die containing discrete MIMCAPs to test the nitride films' reliability performance. I-V characteristics were measured up to 100 V post PCT test to measure the breakdown voltage and leakage current. Film 3 has a typical failure rate of 0% ~ 10% post PCT. Film 1, however, suffers a severe breakdown voltage degradation after PCT, with a typical failure rate of 5 times or greater than the worst obtained from film 3. Film 2 exhibits a larger variability in lot-to-lot performance than film 3, with the failure rate ranging from 0% to around 20%. Pre and post PCT I-V characteristics on failed capacitors show there is no significant degradation of leakage current up to 20 V prior to breakdown. To improve the reliability consistency of film 2, pre-deposition treatment was explored, as discussed below.

F. Pre-deposition Treatment

Pre-deposition treatment, including H_2 anneal and NH_3 plasma treatment, were evaluated on film 2 and shown to improve the consistency of the reliability performance of MIMCAPs. The breakdown failure rate from film 2 with the pre-deposition treatment is compared to film 3 (without any pre-deposition surface treatment) and summarized in Table VI. The notable benefit of pre-treatment points out that the interface between nitride and the metal electrode (Au) plays a crucial role in MIMCAP reliability. H_2 anneal was done ex-situ in a rapid thermal anneal (RTA) system at a maximum temperature of 300°C to avoid damaging InGaP/GaAs wafers, and NH_3 plasma pre-treatment was done in-situ prior to the nitride deposition, utilizing the capability of the PECVD tool. We propose a possible mechanism for the improvement -- organic and oxidic contamination of the metal surface is removed by reacting with H_2 or by NH_3 plasma, which enhances the nitride adhesion to the metal electrode and reduces the delamination of nitride in the high pressure, high temperature, and high humidity environment of PCT test.

TABLE VI
COMPARISON OF FILM 2 INCORPORATING PRE-DEPOSITION TREATMENTS WITH FILM 3

Films	**Film 3**		**Film 2 with in situ NH_3 Plasma Treatment**		**Film 2 with ex situ H_2 Anneal**	
Wafer Number	1	2	3	4	5	6
Post PCT Breakdown Voltage Fail rate	13.3%	6.7%	0%	0%	0%	6.7%

More extensive reliability tests including THB (Temperature of 85°C, Humidity of 85% relative humidity, with bias applied, for 1000 hrs), PCT and thermal cycling (150°C) were performed on packaged circuit die and showed better or comparable performance from film 2 with pre-deposition NH_3 plasma treatment compared to film 3 without any pre-treatment.

CONCLUSIONS

Two PECVD silicon nitride films that were developed from a new deposition tool were compared with the existing silicon nitride film from a different, older tool, for use in high density MIMCAPs for InGaP/GaAs applications. All films possess matching or similar refractive indices, dielectric constants, and breakdown voltages. The difference observed between the films in reliability performance and the corresponding difference in film composition lead the author to believe that film composition, particularly the Si to N ratio, is crucial for MIMCAP reliability and that film composition needs to be closely controlled. Pre-deposition treatments, including in situ NH_3 plasma treatment and ex situ H_2 treatment, were evaluated and shown to improve the MIMCAP reliability and reduce performance variability. The benefit of pre-treatment likely can be attributed to the removal/reduction of oxidic and organic contamination at the metal surfaces through reactions with H_2 or NH_3 plasmas, which enhances the nitride adhesion and prevents/reduces the peeling of nitride in the harsh test environment of heat, pressure, humidity and bias.

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ACRONYMS

HBT: Heterojunction Bipolar Transistor
MIM: Metal-Insulator-Metal
RBS: Rutherford Backscattering Spectroscopy
HFS: Hydrogen Forward Scattering Spectroscopy
FTIR: Fourier Transform Infrared Spectroscopy