



**Characterization of Multi Temperature and Multi RF
Chuck Power Grown Silicon Nitride Films by PECVD and
ICP Vapor Deposition**

by F. Semendy, N. Mark, S. Farrell, and G. P. Meissner

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14. ABSTRACT Silicon Nitride thin films were grown at 100, 150, 200, and 250 °C and at rf-power of 30, 40, 50, and 60 Watts by plasma enhanced chemical deposition (PECVD) and inductive coupled plasma (ICP) chemical vapor deposition using 450 W microwave power. The silicon nitride films were deposited on a n-type (100) 4 silicon wafer with 1-20 Ω cm resistivity. The optical, physical, and electrical properties were systematically investigated. Extensive analysis were conducted using Wollam Spectroscopic Ellipsometer, FTIR, and AFM. The temperature of deposition and rf-power played important role in the physical and optical properties of the film. Similarly the index of refraction increased with increasing deposition temperature. Silicon nitride stretching characteristic peaks Si-N, N-H, Si-H, and N-H have been observed with significant intensities as observed by FTIR spectroscopy especially in the case of the film grown under various temperatures. For the first time the results obtained from the growth of silicon nitride using PECVD and ICP under conditions, we conclude that the ICP grown films are of superior quality.					
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1. Introduction

Silicon nitride is used in a number of electronic and optical applications and is widely used in thin film dielectrics in a broad range of microelectronic, photovoltaic, and photonic devices. In addition to remarkable electronic properties, silicon nitride has high chemical and thermal stability, satisfactory optical transparency, remarkable moisture resistivity, and good mechanical strength. It is the candidate material for gate dielectric, insulation, barrier passivation, and encapsulation layers in microelectronic devices and integrated circuits wherever high-quality native oxides are not available or suitable. Another application is as an antireflection and passivation coating layer on crystalline-silicon (c-Si) solar cells. Common deposition techniques include plasma enhanced chemical vapor deposition (PECVD) and low pressure chemical vapor deposition (LPCVD). With LPCVD (1, 2) stoichiometric films of silicon nitride with low hydrogen content are obtained. However, due to high substrate temperature of typically of 700–900 °C these layers cannot be used for low-temperature applications. Therefore, PECVD (3) grown nitrides are used, which can be deposited at low temperature, usually at 250 °C. This method allows silicon nitride films to deposit at low temperature and pressure of 0.1–5 mTorr. The gases used in this method are SiH_4 and NH_3 . Even though this technique is widely used, the silicon nitride film deposited is known to be non-stoichiometric and contains substantial amount of hydrogen (4–6) and in some cases a small amount of oxygen (7, 8). The high hydrogen content results in a film that is actually $\text{Si}_x\text{N}_y\text{H}_z$, instead of Si_3N_4 . Device performance can be degraded due to the substantial presence of hydrogen in the film which can migrate into the Si– Si_3N_4 interface in devices, thus creating interface states (9, 10). The film characteristics can also be degraded by hydrogen. Thus, the silicon nitride may end up with lower density, higher etch rate, and lower thermal stability when compared to such films grown by LPCVD. In realizing and trying to overcome these kind of problems with silicon nitride films grown by LPCVD, and PECVD a new method to deposit these films was developed which was previously used to deposit silicon dioxide (11, 12). In this method silicon nitride can be deposited at a temperature of 400 °C or below at a pressure less than 10 mTorr. This new technique uses SiH_4 and N_2 gases and inductively coupled plasma (ICP) and creates high density plasma in the processing reactor. This high density plasma allows the deposition of a high quality silicon nitride film at low temperature with minimal hydrogen in the film. Some of the advantages of ICP sources over other types of high-density sources include easier scale up, and advanced automatic tuning for the source. In the hybrid ICP configuration, such as the one used in our work, it is possible through the additions of rf power to the wafer chuck, to control ion flux and ion energy independently (14). This helps the applications for dielectric film deposition by ICP. One can use gap filling techniques by high ion bombardment, by controlling the rf power in the wafer chuck, which is not possible by PECVD. With such ion energy tuning, damage sensitive devices can be easily handled in this process. Figure 1 gives the layout of an ICP and PECVD systems. The ICP

system is generally noted for low frequency of excitation, and it produces hot and cold electrons. Increased cracking molecules is easily possible and film uniformity can be obtained to the tune of 2–5% for 50 nm thick films.

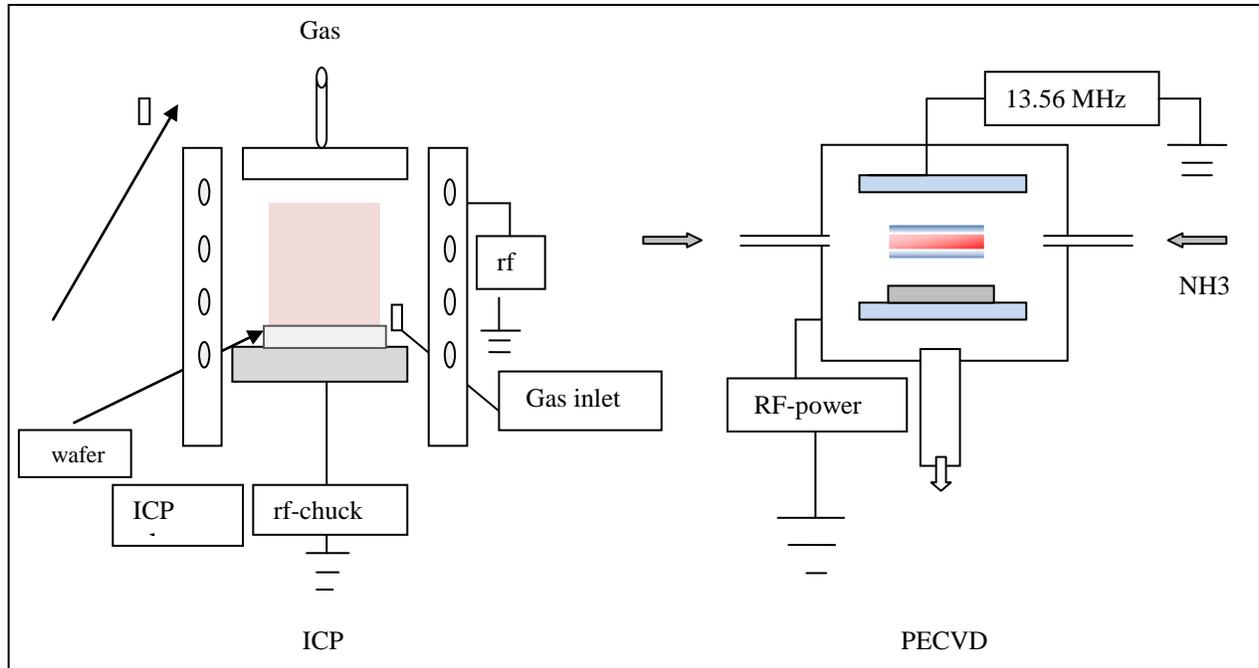


Figure 1. (a) ICP system (b) PECVD system.

In this study, silicon nitride films deposited by PECVD and ICP methods for various temperatures and rf-chuck power were characterized by ellipsometer, AFM, and FTIR. Ellipsometric study provided detailed information on the thickness of the sample, optical constants, index of refraction, AFM profiling for surface roughness, grain size, FTIR spectroscopy for information on the chemical bonds.

2. Experimental Details

The silicon nitride films were deposited on a p-type single side polished 4" wafer substrate with <100> orientation and with a resistivity of 1–20 Ω cm. The wafer was about 500 μ m thick. Prior to deposition the Si wafers were stripped of native oxide by using dilute HF solution. The deposited silicon nitride was about 2000–3000 Å . The silicon nitride in this experiment were produced by Unaxis VLR 700 ICP system and Plasma Therm PT 790 PECVD system. In the PECVD system the process conditions were as follows. The total deposition pressure was 900 mTorr, the rf power 45 W with rf of 13.56 MHz. Flow rates for SiH_4 , N_2 , NH_3 , and He were 90, 160, 3, and 488 sccm. In each case the film was grown for 30 min at various temperatures of 100, 150, 200, and 250 $^\circ\text{C}$ and in a second set of experiments the rf-chuck power

of 30, 40, 50, and 60 W were used as a variable. The ICP system is capable of depositing high quality dielectric films at a temperature as low as room temperature. The film was deposited at various temperatures of 100, 150, 200, and 250 °C at rf power of 50 W. The experiment was also done for various rf power levels of 30, 40, 50, and 60 W. The deposition rate was about 70 Å/min for the rf power of 30 W. The thickness, refractive index, optical constants and surface layout were measured at a wavelength of 633 nm using a Wollam spectroscopic ellipsometer. This optical technique measures a change in polarization as light reflected or transmitted from the material structure. The polarization change is represented as an amplitude ratio and the phase difference. The measured response depends on optical properties and thickness of individual materials. One can also compute surface roughness this system. In addition, FTIR measurements were carried out in the 400 to 4000 cm^{-1} range to obtain information on the vibrational modes Si-N, Si-H, and Si-NH bonds for the films grown under various conditions. The surface roughness was measured by AFM for 1x1 μm sizes.

3. Results and Discussion

Ellipsometric characterization provided information on thickness, deposition rate, and refractive index for silicon nitride grown using ICP and PECVD methods. Table 1 gives details on the film grown using ICP and figure 2a gives the graphical representation of the thickness and uniformity change for various power levels.

Table 1. Thickness, deposition rate, and index of refraction for ICP grown samples under various chuck power.

Power (W)	Thickness (Å)	% Uniformity	Deposition Rate (Å/min)	Index	% Uniformity
30	211.7	2.19%	70.4	2.0883	0.88%
40	2408.8	2.17%	80.3	2.1009	0.93%
50	3032.9	1.68%	101.1	2.0969	0.57%
60	3365.2	1.47%	112.2	2.1132	0.47%

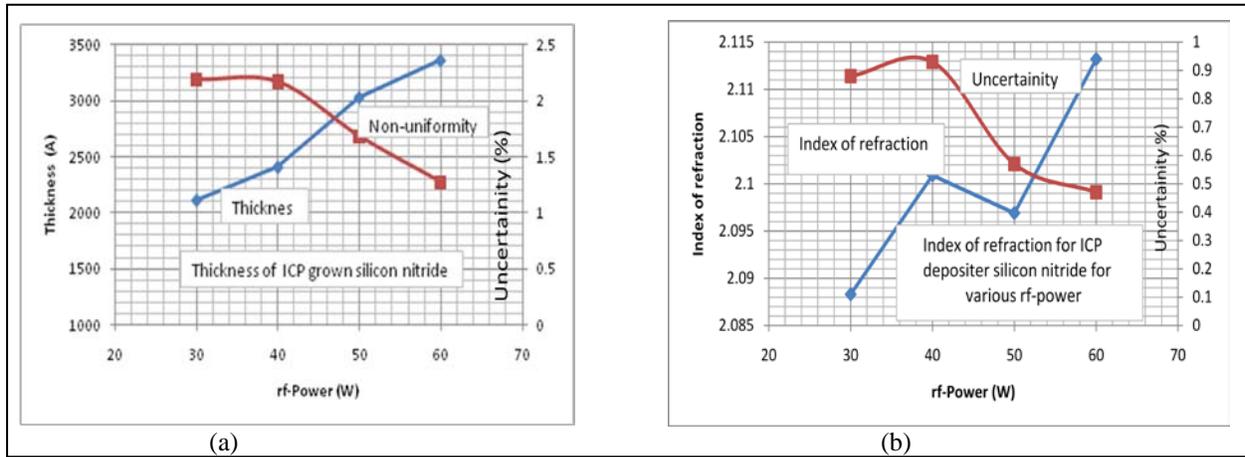


Figure 2. Silicon nitride grown by ICP (a) thickness versus power and (b) index versus power.

From the graph one can see that the thickness of the silicon nitride film is linearly increasing, whereas the uniformity change is at a higher percentage at the lower power level, while it is at a lower level at the higher power level. This means that the uniformity of the film is better at a higher power of film deposition. Figure 2b gives plot of the index of refraction for the ICP grown silicon nitride film for various power levels.

Table 2 gives the thickness, deposition rate and index of refraction for PECVD grown samples under various power levels using PECVD. Obtained values are plotted on figure 3. In the PECVD technique the thickness of the film increases as the rf power increases and the index of refraction increase nominally and settles at about 60 W as indicated by the percentage uncertainty. Figure 4 gives the thickness and index of refraction of silicon nitride grown by PECVD and ICP for various temperatures.

Table 2. Thickness, deposition rate, and index of refraction for PECVD grown samples under various power

Power (W)	Thickness (Å)	% Uniformity	Deposition Rate (Å/min)	Index	% Uniformity
30	1804.8	2.08%	60.2	1.9045	0.57
40	2181.0	2.19%	72.7	1.9208	0.71%
50	2840.5	2.54%	94.7	1.9228	1.17%
60	3142.1	1.84%	108.1	1.9185	0.65 %

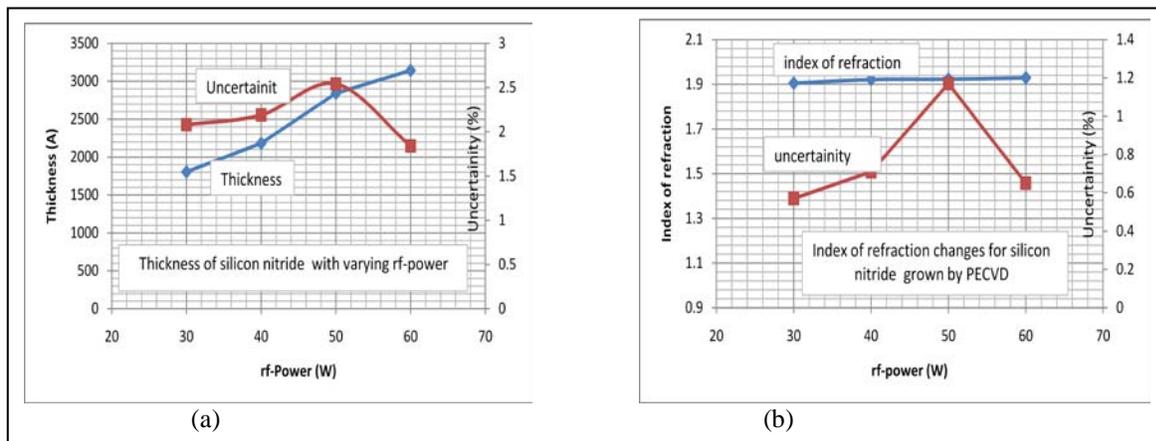


Figure 3. Silicon nitride grown by PECVD (a) thickness versus power and (b) index versus power.

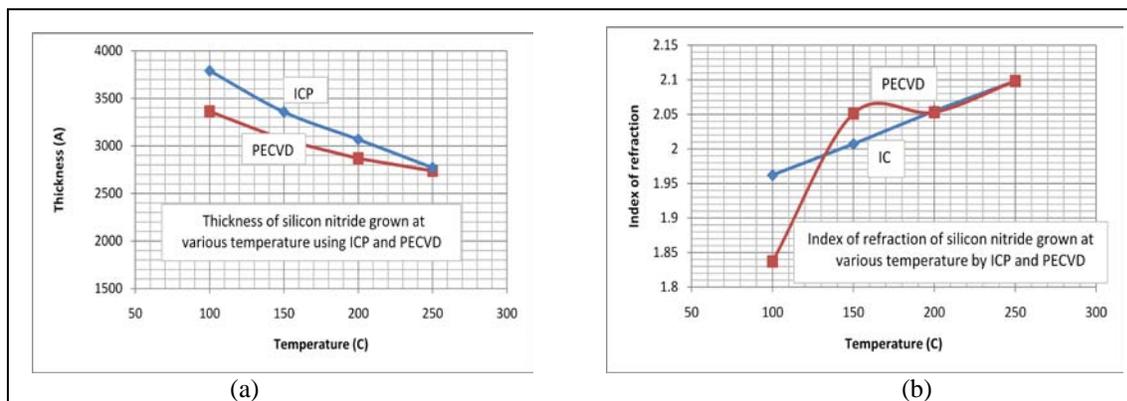


Figure 4. (a) Thickness of (b) index of refraction of silicon nitride grown by ICP and PECVD for various temperatures.

Observe that the thickness of silicon nitride decreases as the temperature increases in both PECVD and ICP growth, whereas the index of refraction increases as the growth temperature goes up. This is indicative of the nature of the material grown. As the temperature increases, more of the real silicon nitride films are grown instead of the growth of other silicon bonded species. Also observe that the index change is linear for the ICP grown material—indicative of the better quality of ICP technique. At higher temperature, closer to 250 °C, the film is more of Si-N bonded material as indicated by the FTIR spectra reported later. Spectroscopic ellipsometer measurements provide additional information on the surface, as shown in figure 5. These surface plots were obtained after making a seventeen (17) point or more laser measurements. From the figure we see difference in thickness as well as the distribution of the varying thickness. It is interesting to note that the thickness deviation has decreased, and that the uniformity is closer to the mean value.

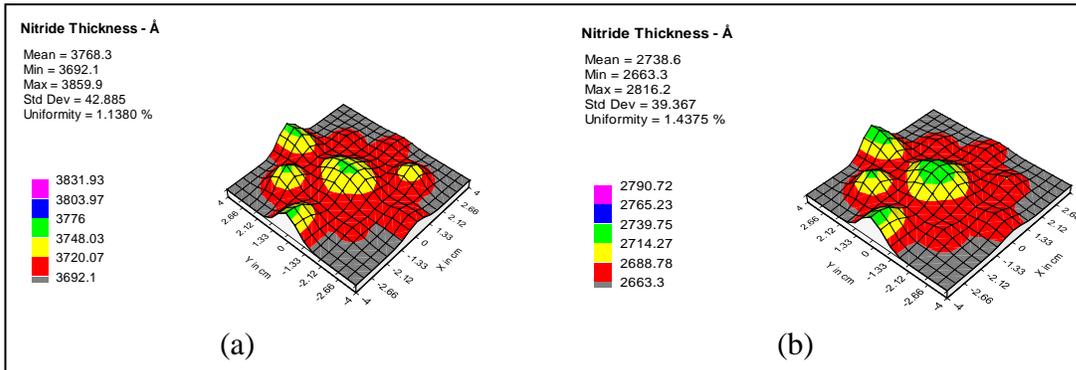


Figure 5. Surface plot of thickness of silicon nitride deposited by ICP at (a) 100 °C and (b) 250 °C.

Optical constants were measured using the ellipsometer in each case and the two samples deposited by ICP at 100 °C and 250 °C are shown in figure 6.

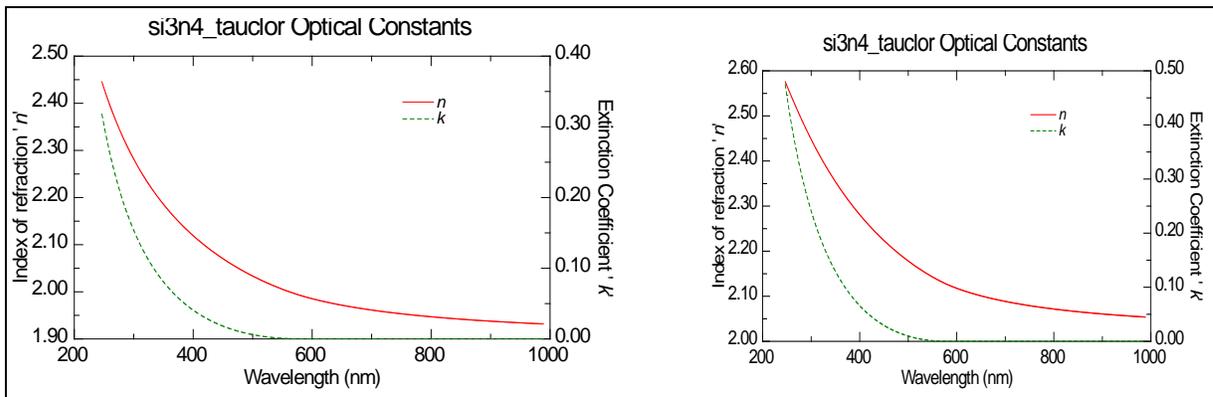


Figure 6. Optical constants including index of refraction and extinction coefficient for silicon nitride grown by ICP (a) grown at 100 °C (b) grown at 250 °C. Red curve shows the index of refraction while green dotted line indicate the extinction coefficient.

Figure 6 clearly indicate that the silicon nitride film is settling quickly to a value of index of refraction higher than two (2) or close to it at higher temperature. A similar behavior was also observed in the case of PECVD deposited Si_3N_4 . Atomic force microscopy scanning for a $1 \times 1 \mu\text{m}$ size has given sufficient information about the quality of the film surface. Figure 7 gives the AFM scan pictures of two samples grown by ICP, one at 100 °C and the second one at 250 °C for scan size of $1 \times 1 \mu\text{m}$. Clearly, the 250 °C grown sample is smoother and planar compared to the 100 °C sample.

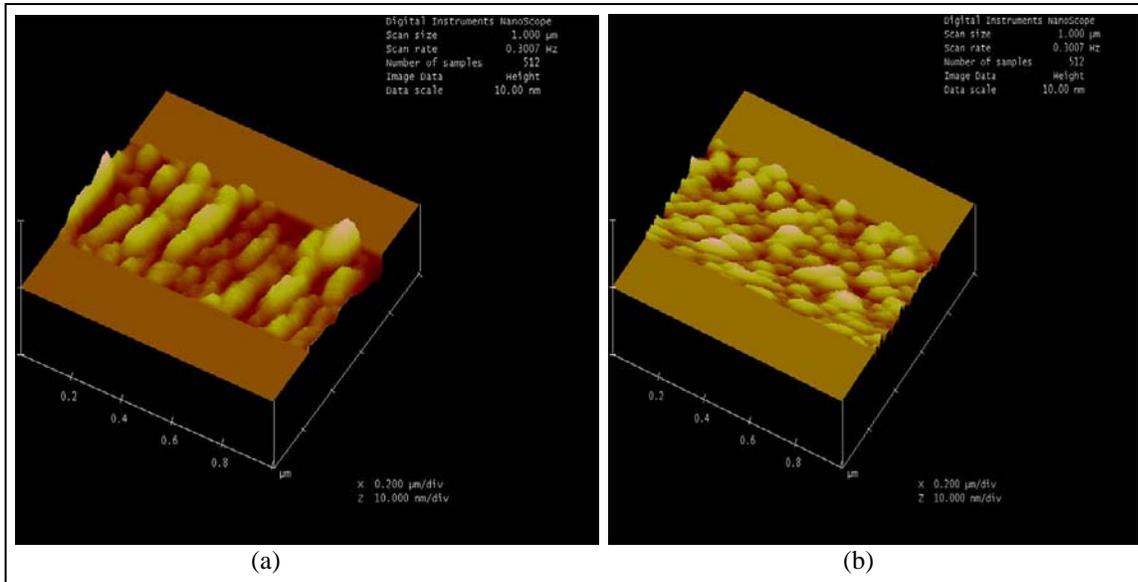


Figure 7. AFM pictures 1x1 μm size scan for samples grown by ICP at (a) 100 $^{\circ}\text{C}$ and (b) 250 $^{\circ}\text{C}$.

Figure 8 gives the AFM scan pictures of three samples grown at 100, 150, and 250 C by PECVD. The surface smoothness and uniformity changes as the temperature of growth increases and at 250 C the film is of similar quality with prevalent silicon nitride material.

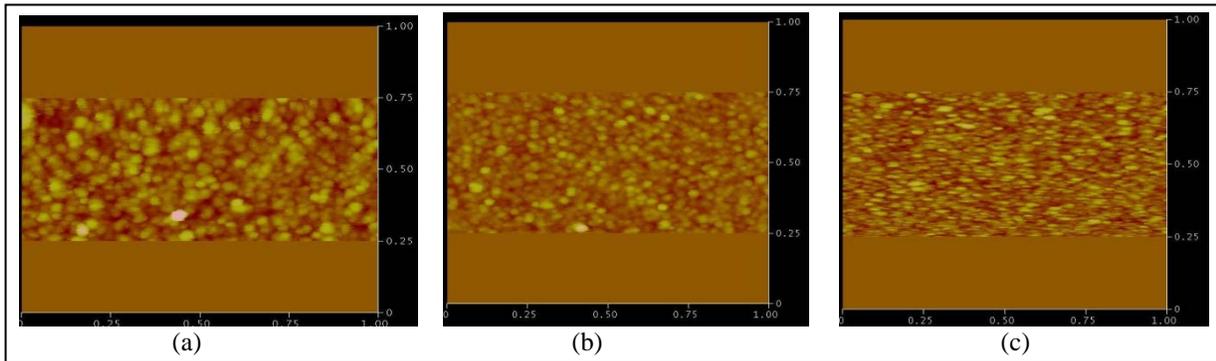


Figure 8. AFM pictures 1x1 μm size scan for samples grown by PECVD at (a) 100 $^{\circ}\text{C}$, (b) 150 $^{\circ}\text{C}$, and (c) 250 $^{\circ}\text{C}$.

The above scan pictures clearly indicate the size and change of the clusters of silicon nitride as the temperature changes. As with the deposition temperature, the quality of the film improves and particles get closer and able to form similarly sized particles and finally collapse to form uniform layer. Surface free energy gets reduced only through smoothing effects, decreasing feature heights and decreasing distance between features. Film roughness also was measured for PECVD and ICP deposited samples as shown in figure 9. Figure 9(a) shows the RMS value for PECVD grown sample at various temperatures. The RMS value decreased from the higher value to a lower value as the temperature increases.

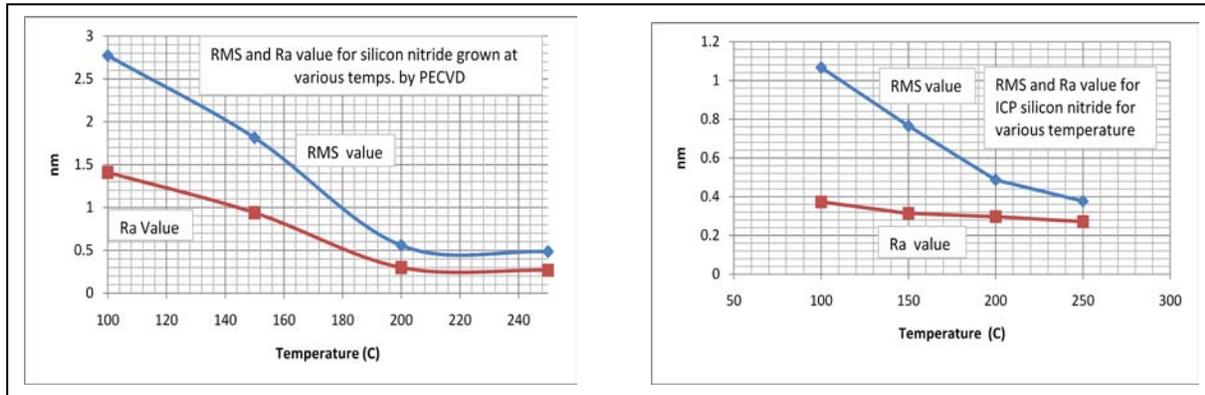


Figure 9. AFM obtained RMS and Ra value for silicon nitride grown by PECVD and ICP at various temperatures.

As the temperatures increases the RMS value decreases as seen above, indicative of the quality of the film. Note that the ICP deposited film has a lower RMS value at 250 °C, indicating its better quality of smoothness.

Figure 10 shows the RMS and RA values for PECVD and ICP grown silicon nitride under various rf-chuck power. It is clearly seen that the RMS and RA values in both cases are much lower that grown for various temperatures. The RMS of ICP grown sample is lower than that of similar samples grown for various temperatures. However, in both cases the ICP grown samples have much lower RMS values indicating better surface quality.

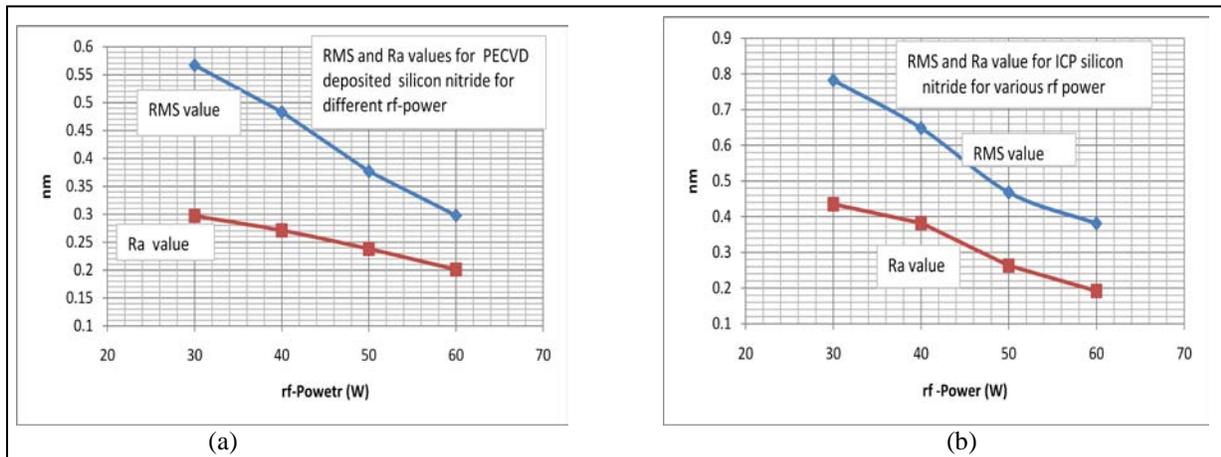


Figure 10. AFM obtained RMS and RA value for silicon nitride grown by (a) PECVD and (b) ICP at various rf-power.

FTIR studies were used to estimate the chemical bonding conditions in the deposited silicon nitride films. The IR spectra was recorded in the transmission mode in the range of 400–4000 cm^{-1} as shown in figure 11. In all cases the spectra show strong absorption around 850–860 cm^{-1} that can be identified with the stretching vibration mode of the Si-N bond (10). The large value of the full width half maximum (FWHM) in our study agreed with previously reported values (15).

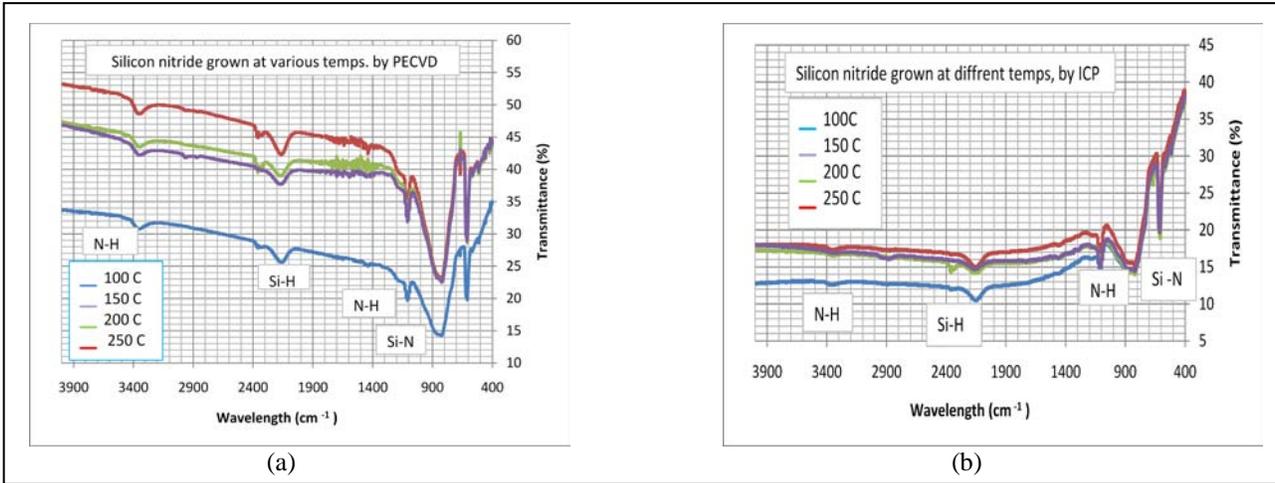


Figure 11. FTIR spectrum of silicon nitride grown by PECVD and ICP at various temperatures.

Additional small absorption bands observed around 480 cm^{-1} corresponds to the Si-N breathing mode. In addition we note the N-H bending mode around 1160 cm^{-1} , Si-H stretching bonds around 220 cm^{-1} , and N-H stretching mode around 3340 cm^{-1} . There may be some small peaks present in the spectra corresponding to Si-O stretching mode at 1080 cm^{-1} which cannot be distinguished from the large Si-N stretching mode. A peak at 1140 cm^{-1} is also present which is due to an artifact of silicon substrate. Si-N stretching band mode is prominent and low at 100 C and increase substantially for $150, 200,$ and $250\text{ }^{\circ}\text{C}$. Figure 12 gives the FTIR spectrum of silicon nitride grown at various rf-chuck power .

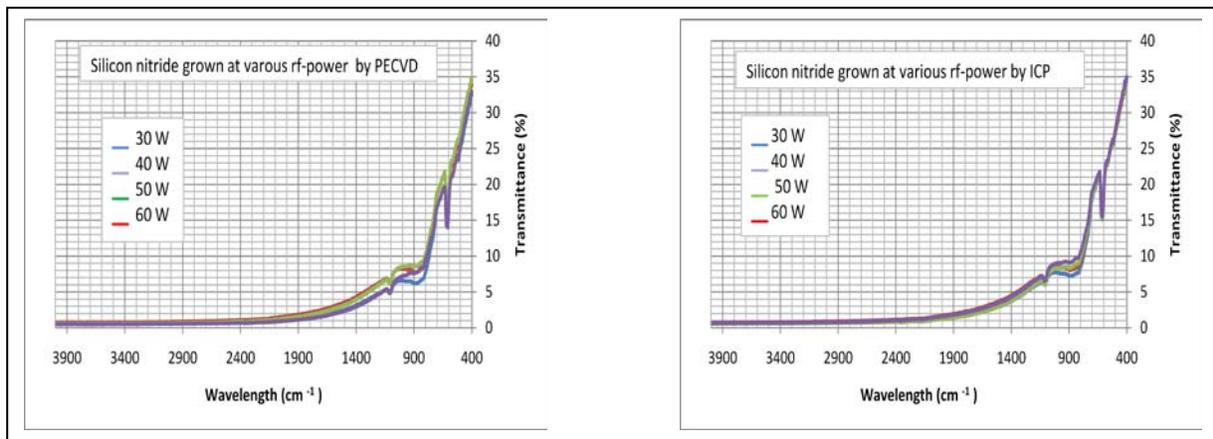


Figure 12. FTIR spectrum of silicon nitride grown by PECVD and ICP at various rf-chuck power.

Unlike the case of the film grown at various temperatures, we can clearly see that the FTIR spectrum is very similar in both cases and also that the features are uniformly similar. This can be due to increased incorporation of nitrogen atoms in the silicon nitride network layer which change the composition of the deposited film from the silicon rich to nitrogen rich environment. Observe a decrease in N-H bond concentration as the rf power goes up $2160\text{--}2220\text{ cm}^{-1}$ also

decreases as the applied power increases and for the 40–60 W, because the concentration becomes lower.

4. Conclusions

In summary, we have investigated the influence of temperature and rf-chuck power on the growth of silicon nitride films by PECVD and ICP growth techniques and compared the results for the first time and conclude that the ICP grown silicon nitride films are of better quality compared to PECVD grown film. We have deposited good quality silicon nitride films at 100, 150, 200, and 250 °C temperatures and at 30, 40, 50, and 60 W rf-chuck power. We observed thickness of the silicon nitride film linearly increasing, whereas the uniformity change is at a higher percentage at the lower power level, while it is at a lower level at a higher power level. This indicates that the uniformity of the film is better at higher power of film deposition. The morphology of the films for the various growth conditions were analyzed through AFM. Surface quality substantially improved as the temperature of growth increased. The RMS value showed substantial change as the temperature of growth went up. FTIR studies were provided for the chemical bonding conditions in the deposited silicon nitride films. The IR spectra was recorded in the transmission mode in the range of 400–4000 cm^{-1} and in all cases the spectra showed strong absorption around 850–860 cm^{-1} that can be identified with the stretching vibration mode of the Si-N bond. The large value of the FWHM in our study agreed with previously reported values. Additional small absorption bands observed around 480 cm^{-1} corresponds to the Si-N breathing mode. In addition, we observed the N-H bending mode around 1160 cm^{-1} , Si-H stretching bonds around 220 cm^{-1} , and N-H stretching mode around 3340 cm^{-1} . There may be some small peaks present in the spectra corresponding to Si-O stretching mode at 1080 cm^{-1} which cannot be distinguished from the large Si-N stretching mode. A peak at 1140 cm^{-1} is also present which is due to an artifact of silicon substrate. Si-N stretching band mode is prominent and low at 100 °C and increase substantially for 150, 200, and 250 °C. In both cases of ICP and PECVD grown materials with variable rf-chuck power, the FTIR spectrum showed consistently non-prominent peaks indicative of the quality of films.

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List of Symbols, Abbreviations, and Acronyms

c-Si	crystalline-silicon
ICP	inductively coupled plasma
LPCVD	low pressure chemical vapor deposition
FWHM	full width half maximum
PECVD	plasma enhanced chemical vapor deposition

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1	PM TIMS, PROFILER (MMS-P) AN/TMQ-52 ATTN B GRIFFIES BUILDING 563 FT MONMOUTH NJ 07703		
1	US ARMY INFO SYS ENGRG CMND ATTN AMSEL IE TD A RIVERA FT HUACHUCA AZ 85613-5300		
1	COMMANDER US ARMY RDECOM ATTN AMSRD AMR W C MCCORKLE 5400 FOWLER RD REDSTONE ARSENAL AL 35898-5000		

NO. OF
COPIES ORGANIZATION

ATTN RDRL SEE I P FOLKES
ATTN RDRL SEE I P TAYLOR
ATTN RDRL SEE I P UPPAL
ATTN RDRL SEE I S FARRELL
(3 COPIES)
ATTN RDRL SEE I S SVENSSON
ATTN RDRL SEE I W BECK
ATTN RDRL SEE I W SARNEY
ATTN RDRL SEE I Y CHEN
ATTN RDRL SEE M G DANG
ATTN RDRL SEE M G GARRETT
ATTN RDRL SEE M M REED
ATTN RDRL SEE M M WRABACK
ATTN RDRL SEE M N BAMBHA
ATTN RDRL SEE M N DAS
ATTN RDRL SEE M P SHEN
ATTN RDRL SEE M W CHANG
ATTN RDRL SEE O N FELL
ATTN RDRL SEE O P PELLEGRINO
ATTN RDRL SEE P GILLESPIE
ATTN RDRL SEG N MARK
(3 COPIES)
ATTN RDRL SER E A DARWISH
ATTN RDRL SER E P SHAH
ATTN RDRL SER L A WICKENDEN
ATTN RDRL SER L B NICHOLS
ATTN RDRL SER L E ZAKAR
ATTN RDRL SER L M DUBEY
ATTN RDRL SER L M ERVIN
ATTN RDRL SER L S KILPATRICK
ATTN RDRL SER P AMIRTHARAJ
ATTN RDRL SER U C FAZI
ATTN RDRL SER U J COSTANZA
ATTN RDRLSEE M
M TAYSING-LARA
ADELPHI MD 20783-1197

TOTAL: 76 (74 HCS, 1 CD, 1 ELECT)