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Research paper

Effect on Silicon Nitride thin Films Properties at Various Powers of RF Magnetron Sputtering

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Abstract

Silicon nitride thin films have numerous applications in microelectronics and optoelectronics fields due to their unique properties. In this work, silicon nitride thin films were produced using radio frequency (R.F.) magnetron sputtering technique at various sputtering powers. The prepared thin films were characterized with XRD, FE-SEM, FTIR, surface profiler, AFM and spectral reflectance techniques for structure, surface morphology, chemical bonding information, growth rate, surface roughness and optical properties. The results showed that silicon nitride thin films were amorphous in nature. The films were smooth and densely packed with no voids or cracks at the surface. FTIR characterization informed about Si-N bonding existence which confirmed the formation of silicon nitride films. The sputtering power showed the impetus effect on growth rate, surface roughness and optical properties of produced films.

Keywords: *AFM; FE-SEM; FTIR; R.F; Silicon Nitride; Sputtering Technique; Surface Profiler; Thin Films; XRD.*

1. Introduction

Silicon nitride films are useful in microelectronics applications [1] such as oxidation masks, protection and passivation barrier layers, gate dielectrics and interlevel insulations due their remarkable hardness, high thermal stability, chemical inertness and excellent insulation capabilities [2]. These films also play important role in optoelectronic applications such as antireflection coatings and optimization of optical performance of devices due to its capability of providing the refractive index variation from silicon oxide like (~1.46) to stochiometric silicon nitride (~2.0) and by providing transparency in ultraviolet (250nm) to infrared (9µm) regions of optical spectrum [3].

Silicon nitride films can be produced through various methods such as atomic layer deposition [4], chemical vapor deposition and sputtering [5, 6]. The advantage of sputtering technique over rest of two techniques is low temperature deposition which enables sputtering to employ the substrates that can't tolerate high temperatures. Unlike CVD technique, sputtering is safe and environment friendly process as it doesn't require any toxic gas which requires special material handling arrangements.

An extensive literature is available on the deposition of silicon nitride thin film deposition through reactive sputtering. However the sputtering in non reactive regime has not been explored completely. The benefit of non reactive sputtering is the exclusion of nitrogen gas. This in turns reduces the chance of impurity incorporation, if the purity level of nitrogen gas is not sufficient. It is well known that deposition parameters influenced the properties of thin films significantly [7]. This also holds true for sputtering deposition technique where variations in parameters such as sputtering power, sputtering pressure and target to substrate distance and had significance effects on the properties of deposited films [8-10].

Our previous work focused on influence of sputtering pressure on the properties of silicon nitride thin films deposited in non-reactive environment. In current work, the influence of sputtering power in non-reactive sputtering regime is studied. The effect of variation on the structure, surface morphology, chemical bonding information, growth rate, surface roughness and optical properties has been characterized.

2. Materials and Method

We used a RF magnetron sputtering system (SNTEK, Korea) to deposit silicon nitride thin films. The system was consisted of a main deposition chamber (60 cm in diameter and 40 cm in height) with six view ports. It was consisted of dual sputtering sources, one was connected to RF and the other was connected to DC power supply. The sputtering sources were connected with water coolant flow system in order to prevent temperature rise during deposition process. A stoichiometric Si3N4 circular target (Taewon Scientific, Korea) of 3" diameter and 3mm thickness was used as a source material. Single sided polished P-type boron doped silicon oxide coated silicon wafers (with thickness 500 µm and orientation <100>) were used as substrate. A target shield was also applied to prevent the unnecessary sputtering of target holder and screws. The angle between sputtering and substrate holder was 30o.

All substrates were cleaned through ultrasonic agitation using acetone, iso propyle alcohol (IPA) and de-ionized (DI) water (18 MΩ, Millipore USA) and dried with industrial grade nitrogen gas. The experiments were carried out in a vacuum chamber evacuated at 4.5×10-6 Torr to get high purity in thin films. It was achieved from a vacuum turbo molecular pump which was backed by rotary mechanical pump. Argon (Ar) gas with 99.99% purity was introduced in the chamber as sputtering gas. Ar flow rate was fixed at 80 sccm and working pressure of the system was kept constant at 5 mTorr. Samples were rotated constantly at 7 rpm to improve the film homogeneity. Desposition time (t) was kept fixed at 30 min

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for each sample. Experiments were conducted with following sputtering powers (a) 100 W (b) 150 W (c) 200 W (d) 250 W (e) 300 W. These process parameters are tabulated in Table 1.

Table 1: Process parameters of the experiment.

Process Parameter	Value
Sputtering pressure (mtorr)	5
Target to substrate distance (cm)	14
Angle between target and substrate (deg.)	30
Argon flow (sccm)	80
Substrate rotation (rpm)	7
Base pressure (torr)	4 x 10^{-6}
Deposition time (min)	30
Sputtering power (w)	100,150,200,250,300

The deposited films were characterized with x-ray diffraction (XRD), field emission scanning electron microscopy (FE-SEM), Fourier transform infrared (FTIR) spectroscopy, surface profiling, atomic force microscopy (AFM), and spectral reflectance techniques for the structural, topographical and chemical and optical properties.

3. Results and discussions

The deposited films were first characterized for structural properties through Xpert3 PANalytical, xray diffraction system. The samples were scanned for 2 theta and intensity was measured. Figure 1 shows the XRD scan for the deposited films at various sputtering powers. The only peak appeared at around 52o which corresponds to 311 planes in silicon substrate. This proved that all deposited films were amorphous. It is consistent with literature that at room temperature the formation of silicon nitride is amorphous and the crystalline features appear at higher temperatures starting from 400oC.

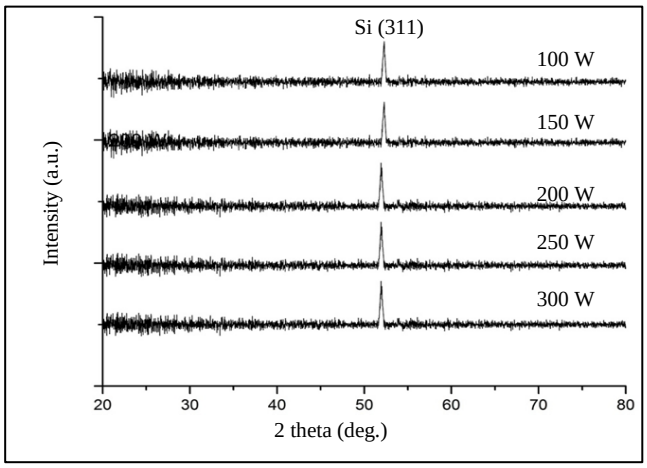

Figure1: XRD spectra of thin films deposited at various sputtering powers

Figure 2 shows the surface images acquired by the FE-SEM. All the deposited films are featureless confirming the formation of thin films. All films were found smooth and homogenous. There were no cracks and voids.

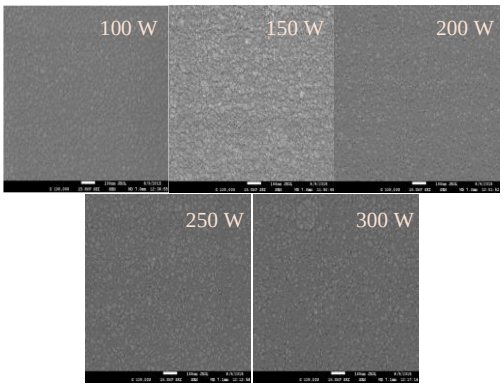

Fig. 2: Surface images of deposited films at various sputtering powers

The chemical composition of deposited films was investigated with FTIR spectroscopy system (Perkin Elmer, USA). Figure 3 shows the FTIR scan obtained from the sample deposited at various sputtering powers. It is found that the decrease in reflectance at wave number range 700 cm-1 to 850 cm-1 correspond to infrared absorption by Si-N stretching bonds.

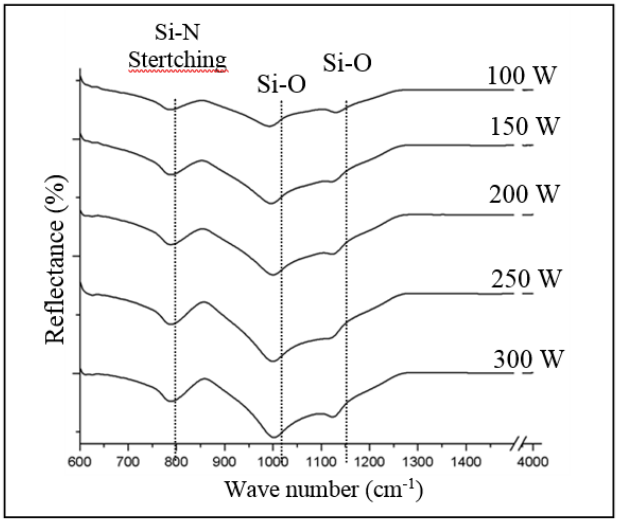

Fig. 3: FTIR spectra of silicon nitride thin films at various sputtering powers

This confirms the formation of silicon nitride as expected from the exploitation of stoichiometric silicon nitride sputtering target as a source material. However, the decrease in reflectance at 1000cm-1 and 1140 cm-1 reveals the absorption by Si-O bonding. The FTIR scan shows the formation of SiO and SiO2 within the deposited films. The Silicon oxide signal is believed to come from substrate which is coated with SiO2.

The average thicknesses of deposited films were measured with an Alpha Tencor, USA surface profiler. A portion of substrate was covered using kapton tape prior to deposition process. Due to this, a step was created when the tape was removed after the deposition. The thickness of film was found by carefully measuring the difference between heights of covered and exposed surfaces of substrate. The average growth rate (G) was found by dividing measured thickness (d) with the deposition time (t).

$$G = d/t. \quad (1)$$

Table 2 shows the average growth rates of silicon nitride films as a function of various sputtering powers. The average growth rate increased with increase in sputtering power.

Table 2: Average growth rate of silicon nitride thin films at various sputtering powers

Sputtering Power (W)	Average growth rate (G) (nm/min)
100	2.5
150	3.6
200	4.4
250	5.7
300	6.5

In sputtering technique, the mean free path of sputtered atoms plays an important role. The sputtered atoms lose energy during their travel to substrates due to collisions with Ar molecules. These collisions increased the probability for these atoms to be scattered away before reaching the substrates. At high sputtering powers the deposition rates are higher. This is because as the sputtering power increases, the kinetic energy of sputtered atoms increases. These energetic atoms have large tendency to reach the substrate.

The surface morphology of silicon nitride thin films is shown in Figure 4. The scan size for thin films deposited at various sputtering powers was kept fixed at 1µm x 1 µm. The values of several roughness parameters are tabulated in Table 3. The effect of sputtering power is significant on surface roughness as arrangement of sputtered atoms is also a function of sputtering power. The sputtered atoms with low energy have fewer tendencies to diffuse on substrate. Therefore, the growth is expected to follow Volmer Weber model in its initial stages with high surface roughness. As the sputtering power was increased, the sputtered atoms got more energy which helped them to travel more on substrate and they diffused properly. This showed that the growth started to follow the Stranski-Krastanov model and surface roughness was reduced due to initially layer by layer formation until the sputtering power of 250W. The surface roughness was found increase at 300W which can be explained as the formation of islands after layer formation as happens in Stranski Krastanov growth model.

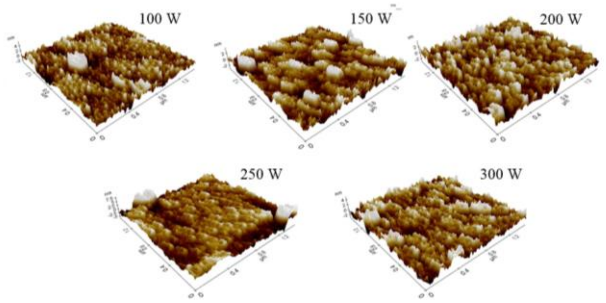

Fig. 4: Surface topographic scan of silicon nitride thin films at various sputtering powers

The refractive index as function of sputtering power was measured using spectral reflectance technique. The refractive index of films at wavelength (λ) 638nm are shown in Table 3.

Table 3: The refractive index of deposited thin films at various sputtering powers

Power (W)	Refractive index
100	1.47
150	1.50
200	1.69
250	1.63
300	1.78

4. Conclusion

Silicon nitride thin films were deposited using RF Magnetron sputtering technique in non reactive environment. The effect of sputtering power on film properties was investigated. The produced films were characterized using XRD, FE-SEM, FTIR, surface profiler, AFM and spectral reflectance techniques for structure, surface morphology, chemical bonding information, growth rate, surface roughness and optical properties. The xrd spectra for the silicon nitride films contain no diffraction peaks and hence were amorphous in nature. The surface morphology characterization confirmed the formation of smooth and dense films. The FTIR spectra confirmed the existence of silicon and nitrogen bonding as a evidence of silicon nitride film formation. The growth rate of films was deduced by measuring the thickness through surface profiling technique. It was found that growth rate of sputtered films increased with the increase in sputtering power. The surface roughness studies informed about the optimized values of sputtering power. The spectral reflectance characterization for refractive index at 638nm of wavelength showed the increase in refractive index with the increase in sputtering power. The tuning of refractive index made the silicon nitride thin films a promising candidate for optoelectronics applications.

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References

[1] Ulvestad A, Andersen HF, Mæhlen JP, Prytz Ø & Kirkengen M (2017), Long-term cyclability of substoichiometric silicon nitride thin film anodes for Li-ion batteries. *Scientific reports* 7(1), 13315.

[2] Dergez D, Schneider M, Bittner A & Schmid U (2015), Mechanical and electrical properties of DC magnetron sputter deposited amorphous silicon nitride thin films. *Thin Solid Films* 589, 227-232.

[3] Torchynska T, Khomenkova L & Slaoui A (2018), Modification of Light Emission in Si-Rich Silicon Nitride Films Versus Stoichiometry and Excitation Light Energy. *Journal of Electronic Materials* 47(7), 3927-3933.

[4] Knoops HC, Braeken EM, de Peuter K, Potts SE, Haukka S, Pore V, & Kessels WM (2015), Atomic layer deposition of silicon nitride from Bis (tert-butylamino) silane and N2 plasma. *ACS applied materials & interfaces* 7(35), 19857-19862.

[5] Ishii Y, Kaneko T, Okimura K, Shindo H & Isomura M (2017), Fabrication of amorphous silicon nitride thin films by radio-frequency sputtering assisted by an inductively coupled plasma. *Thin Solid Films* 624, 49-53.

[6] Yota J, Hander J & Saleh AA (2000), A comparative study on inductively-coupled plasma high-density plasma, plasma-enhanced, and low pressure chemical vapor deposition silicon nitride films. *Journal of Vacuum Science & Technology A: Vacuum, Surfaces, and Films* 18(2), 372-376.

[7] Mustafa MK, Iqbal Y, Majeed U & Sahdan MZ (2017, January), Effect of precursor's concentration on structure and morphology of ZnO nanorods synthesized through hydrothermal method on gold surface. In *AIP Conference Proceedings* 1788(1), 030120.

[8] Alnoor H, Pozina G, Khranovskyy V, Liu X, Iandolo D, Willander M & Nur O (2016), Influence of ZnO seed layer precursor molar ratio on the density of interface defects in low temperature aqueous chemically synthesized ZnO nanorods/GaN light-emitting diodes. *Journal of Applied Physics* 119(16), 165702.

[9] Majid A & Bibi M (2018), Wet Chemical Synthesis Methods. In *Cadmium based II-VI Semiconducting Nanomaterials*, (pp. 43-101). Springer, Cham.

[10] Argile C & Rhead GE (1989), Adsorbed layer and thin film growth modes monitored by Auger electron spectroscopy. *Surface Science Reports* 10(6-7), 277-356.