Journal of Physics: Conference Series

A new fabrication method for low stress PECVD - SiN_x layers

To cite this article: Poh Lam Ong *et al* 2006 *J. Phys.: Conf. Ser.* **34** 126

Institute of Physics Publishing
doi:10.1088/1742-6596/34/1/126
Journal of Physics: Conference Series **34** (2006) 764–769
International MEMS Conference 2006

A new fabrication method for low stress PECVD – SiN_x layers

Poh Lam Ong [1, 2], Jiashen Wei[2], Francis E.H. Tay[1, 2], Ciprian Iliescu[1, 2]

[1] Institute of Bioengineering and Nanotechnology, 31 Biopolis Way, The Nanos, Singapore 138669;
[2] Department of Mechanical Engineering, National University of Singapore, 10 Kent Ridge Crescent, Singapore 119260

Email: ciliescu@ibn.a-star.edu.sg

Abstract. This paper presents a new method of depositing low stress silicon nitride (SiN_x) with high deposition rate using a plasma-enhanced chemical vapour deposition (PECVD) system (STS, Multiplex Pro-CVD). By increasing the operating power of the PECVD system at the high frequency mode (13.56MHz), the deposition rate also increases while that the level of intrinsic stress within the SiN_x film decreases. The relationships between some of the key deposition parameters of the experiment such as chamber pressure, silane (SiH_4), ammonia (NH_3) and nitrogen (N_2) flow rates and the two important response variables namely the level of intrinsic stress and deposition rate are established within this investigation.

1. Introduction

Silicon nitride (SiN_x) has been a predominantly known material for its role in the realm of MEMS as well as in microelectronic and optoelectronic industries. It is usually used either as a passivation or encapsulation material during micromachining or packaging processes due to its excellent adherence and ions as well as moisture diffusion barrier properties [1, 2]. One such good evidence is the vast usage of SiN_x thin films in the passivation of aluminium bond pads and interconnects in IC chips from corrosion [3]. It has also been widely recognised that silicon nitride could be used to fabricate microstructures such as cantilever beams and MEMS tunable optical filters due to its good mechanical strength, high wear resistance and low absorption losses properties [4].

There are a few ways of preparing silicon nitride layers ranging from chemical to physical means such as low pressure chemical vapour deposition (LPCVD), PECVD to thermal evaporation and RF-sputtering. Besides the advantage of depositing material at a relatively lower temperature, PECVD also offers a much higher deposition rate. Yet, another benefit of the PECVD process is that it can produce near stress-free films by appropriate adjustment of the deposition parameters. For instance, one possible approach as reported by Tarraf et al [5] and van de Ven et al [6] was the consecutive deposition of alternate SiN_x layers at both high and low frequencies. This was because the compressive stress within the nitride layer deposited at the low frequency (LF) mode will normally be compensated by the tensile stress deposited at the high frequency (HF) mode and consequently achieving a near stress-free layer. Other means include the doping with Ar or He into the mixture of SiH_4, NH_3 and N_2 precursors that act as diluents such that the intrinsic stress within the SiN_x film can be reduced [7, 8].

In this study, we aimed to establish the fundamental relationships between some of the key deposition parameters of the process with the level of intrinsic stress and deposition rate using high power and high frequency (HF) mode of operation. With this set of established relationships, a possible solution of achieving low stress silicon nitride was then proposed.

2. Experimental procedure

The fabrication of the SiN_x layers was carried out using a plasma-enhanced chemical vapour deposition (PECVD) reactor (STS, Multiplex Pro-CVD). For this reactor, a pair of 8" size upper and bottom electrodes made of stainless steel was used within the deposition chamber that will be kept at high vacuum during the deposition process. The main feature of this system is its duality of RF operation modes, namely low frequency (LF) at 380 KHz and high frequency (HF) at 13.56 MHz. Also, the operating power can be adjusted to range from 0 to 1000 W in the LF mode and 0 to 600 W in the HF mode. Other features of the system include the pressure ranging from 0 to 2000 mTorr; SiH_4 flow rates ranging from 0 to 500 sccm; NH_3 flow rates ranging from 0 to 360 sccm and N_2 flow rates varying from 0 to 5000 sccm.

In this case, the experimental steps could be classified into three main parts: cleaning of the silicon wafers, deposition of the layer of SiN_x onto the substrate and characterisation of the deposited thin film material. In all the experiments, 4", n-type silicon wafers of resistivity 1 to 20 Ω cm and crystallographic orientation (100) were used as the substrates for the PECVD process. The wafers were initially cleaned in piranha (H_2SO_4: H_2O_2 in ratio of 2: 1) at 120^{0}C for 20 minutes, rinsed with deionised (DI) water and then spun-dried. This was to remove any possible contaminants that were on the surfaces of the wafers. After that, the wafers were immersed in a classical buffered oxide etch (BOE) solution (NH_4F: HF = 7: 1) for a minute for removing of the native silicon oxide from the wafer surface. Next the initial curvature of the silicon wafer was recorded down using a stress measurement system (KLA Tencor FLX-2320). This was followed by the deposition of the SiN_x layer onto the silicon substrate using SiH_4, NH_3 and N_2 gases as the precursor reactants. The substrate temperature was kept at 300^{o}C for all experiments. For all the experiments conducted, a pre-deposition of SiN_x was always carried out immediately after a plasma cleaning process to ensure the purity of the deposited SiN_x layer. The influence of the mode of excitation frequency and RF power on both the intrinsic stress level and deposition rate was first investigated. Next, the influence of the pressure and the three gas flow rates on the two response variables conducted at high power and HF mode was considered. Finally, the characterisation of the deposited SiN_x film at the various conditions was achieved by measuring both the intrinsic stress level and the deposition rate. In this case, the deposition rate was computed by first measuring the thickness of the deposited SiN_x layer using a refractometer (Filmetrics, F50) divided by the deposition time. The intrinsic stress level within the SiN_x film was then computed by taking another measurement of the curvature of the processed wafer using the same files under the stress measurement system.

3. Results and discussion

There is accepted that the operating RF mode have a strong influence on the residual stress of the SiNx layer [5,6]. By operating at the HF mode, a tensile stress is achieved while exciting the plasma at the LF mode, yields a compressive stress state within the deposited layer. The main reason for this difference is that at the high frequency mode (13.56 MHz), only the lighter mass electrons within the plasma are free to move while the heavier ions and free radicals are kept "stationary" by the rapid changing electric field. As a result, ion and free radicals bombardments are much more intense at the lower RF mode, which not only enhance chemical reactions, but also promote a densification of the deposited SiN_x film due to the lower energy implantations of both the ions and free radicals onto the surface of the substrate. This has lead to a decrease in magnitude of the intrinsic stress level within the deposited film [6, 9, 10] since the quality of the deposited SiN_x layer is now more uniform

The influence of the RF power operating at the HF mode on the two response variables was investigated. Several combinations of gas flow rates were tested as follow:

- SiH_4: NH_3: N_2 = 120: 75: 1200 sccm
- SiH_4: NH_3: N_2 = 100: 60: 1500 sccm

- SiH_4: NH_3: N_2 = 80: 60: 1500 sccm
- SiH_4: NH_3: N_2 = 80: 60: 1700 sccm

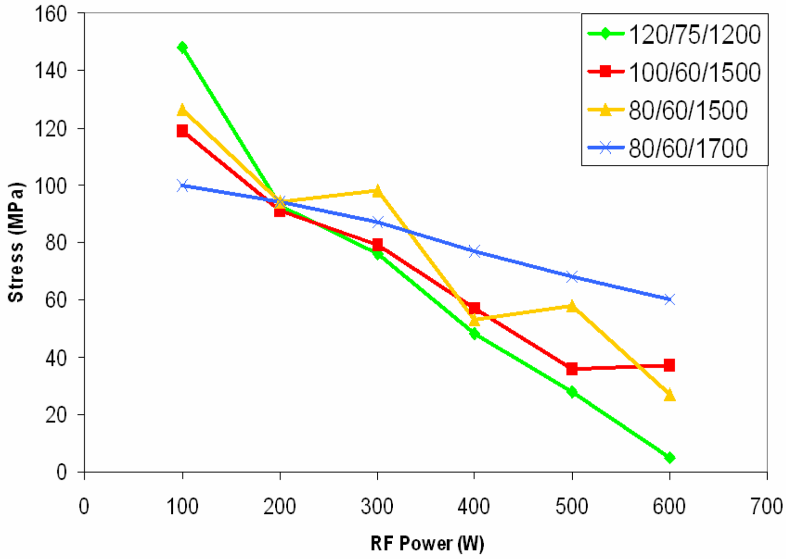

Figure 1. Influence of RF power at HF mode with the intrinsic stress level

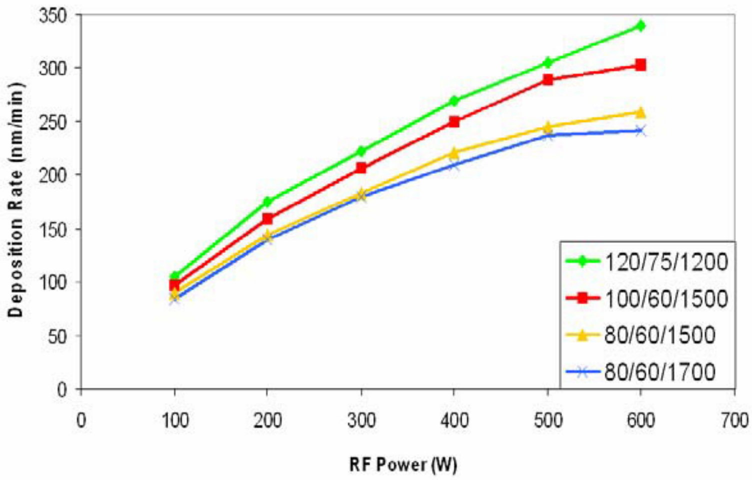

Figure 2. Influence of RF power at HF mode with the deposition rate

The chamber pressure together with the substrate temperature was kept constant at 900 mTorr and 300°C. The established relationships are shown in Figures 1 and Figure 2. The intrinsic stress within the SiN_x layer decreases and the deposition rate increases with RF power. A possible explanation for these trends is that with an increase in RF power, a larger density of higher energy electrons can be found within the plasma and as a result, higher dissociation and ionisation of the reactant gases can be expected. Consequently, a higher deposition rate will be observed. For the case of the intrinsic stress level, it can be reason out that with an increase in RF power, a greater percentage of nitrogen-related species can be expected within the plasma and hence greater incorporation of nitrogen within the deposited SiN_x layers. This will lead to a volume expansion of the deposited thin film, which accounts for the decrease in stress level.

The influence of the chamber pressure was also investigated since this becomes an important factor when the reactants were all in the gaseous phase. The high power (600 W) and high RF mode (13.56 MHz) of operation was adopted throughout this portion of the study. The pressure was varied from 700 mTorr to 1100 mTorr with the gas flow rates kept constant at SiH_4: NH_3: N_2 = 120: 75: 1150 sccm. Figure 3 depicts the established relationships. As it can be observed, both the intrinsic stress and deposition rate increases with pressure.

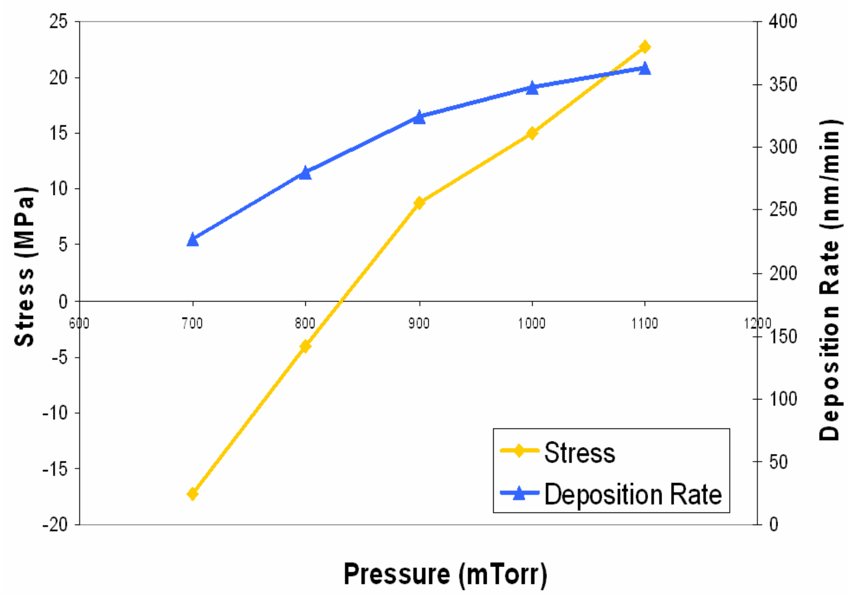

Figure 3. Influence of pressure with the intrinsic stress level and deposition rate

The reason for the higher deposition rate associated with an increase in pressure is that a higher pressurised chamber is associated with a larger amount of ions, molecules and free radicals. Hence, there will be higher chances of the reactant species within the chamber impinging onto the substrate surface with sufficient energy to cause a chemical reaction from occurring. As a result, higher deposition rate can be expected. For the case of the stress within the deposited film, it was proposed that increasing the pressure within the chamber will result in a higher percentage of nitrogen within the deposited film which results in the stress level to increase. This is because both NH_3 and N_2 contribute to the nitrogen content within the layer and that increasing the pressure will increase the percentage of these two precursors further.

The influences of SiH_4, NH_3, and N_2 flow rates were also studied in this work. As usual, the high power (600 W) and high RF mode (13.56 MHz) of operation was adopted throughout the remaining investigation. Pressure was kept at 900 mTorr within this section of the experiment. In the case of the influence SiH_4 flow rate, NH_3, and N_2 flow rates were kept constant at 60 and 1500 sccm respectively while the SiH_4 flow rate was varied between 70 to 120 sccm. The relationship is presented in the graphs on Figure 4. The deposition rate increases while the stress drops drastically with SiH_4 flow rate. The reason for the decrease in intrinsic stress is that there is a higher incorporation of silicon within the deposited film with the increase in SiH_4 flow rate and this result in the layer to have a composition closer to that of on the substrate. As a result, the stress level is much lower since there is a lesser mismatch of the structure within the material. The deposition rate increases since there is a higher proportion of silicon-related species within the plasma and hence a higher amount of reactants for deposition.

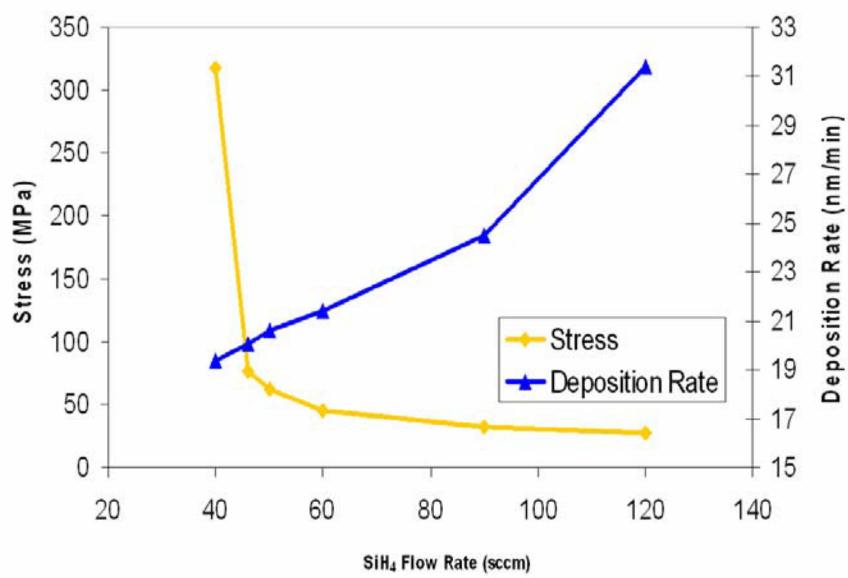

Figure 4. Influence of SiH_4 flow rate with the intrinsic stress level and deposition rate

For the study of the influence of NH_3 flow rate, the SiH_4 and N_2 were kept constant at 120 and 2200 sccm respectively while the NH_3 flow rate was varied between 45 to 100 sccm. The established relationships are presented in Figure 5. In this case, the deposition rate decreases while the stress increases with NH_3 flow rate. One proposed reason is that of the higher inclusion of nitrogen content within the deposited nitride layer. This was derived from the fact that an inverse relationship exist between the influences of the SiH_4 and NH_3 flow rates on both the deposition rate and the intrinsic stress. Increasing the NH_3 flow rate will result in a higher percentage of nitrogen-related species within the plasma. As a result, the increasingly deposited material will have a composition deviating from the one on the substrate and hence decreasing the number of nucleation sites that are responsible for film growth. This will then impede the deposition rate. For the case of intrinsic stress, increasing the NH_3 flow rate will result in a rise in the nitrogen content within the deposited film and as a result, intensify the non-uniformity within the deposited material. This will then aid in the stress level and consequently, increase the stress level.

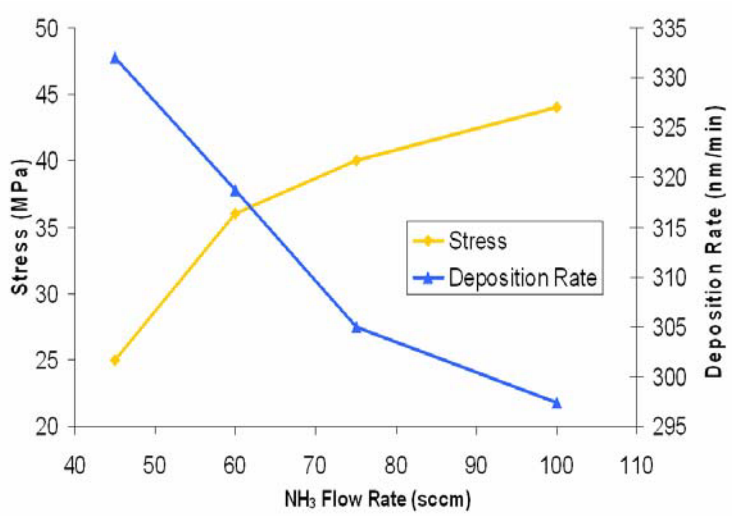

Figure 5. Influence of NH_3 flow rate with the intrinsic stress level and deposition rate

Finally, for the influence of N_2 flow rate, the SiH_4 and NH_3 were kept constant at 120 and 75 sccm respectively while the NH_3 flow rate was varied between 1150 to 2200 sccm. In the N_2 flow rate context, both the deposition rate and the intrinsic stress relationships were similar to that of NH_3 one as presented in Figure 6. As a result, the previously hypothesised influence of nitrogen content on both the deposition rate and intrinsic stress level might be correct. Also, since we were operating at high RF power, the dissociation of triple-bonded N_2 molecule could be possible.

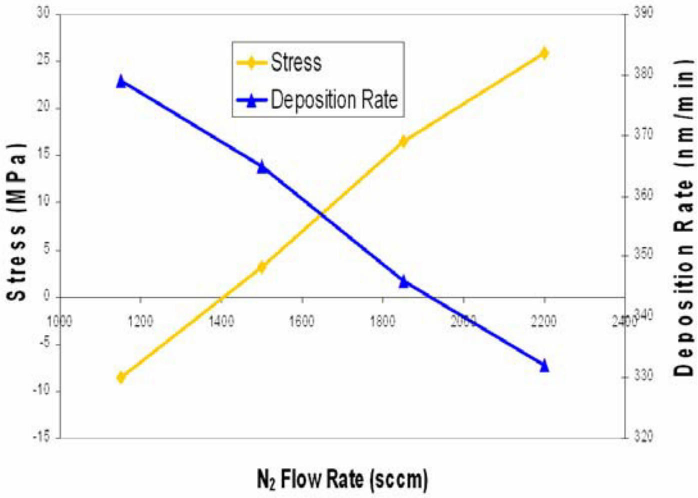

Figure 6. Influence of N_2 flow rate with the intrinsic stress level and deposition rate

4. Conclusion

Fabrication of low stress PECVD-SiN_x layers was performed and investigated using a novel method of deposition, i.e. high power (600 W) and high RF mode (13.56 MHz). Other key deposition parameters such as pressure, SiH_4, NH_3, and N_2 flow rates were also investigated in this work. A low stress and high deposition rate SiN_x layer could be achieved using high RF power (600 W) at the high frequency (HF) mode (13.56 MHz). Next, by fine-tuning the various gases flow rates, a very low stress state ranging from 0 to 20 MPa and a high deposition rate varying from 250 to 350 nm/min could be attained. This finding is crucial to the reliable usage of the fabricated SiN_x thin film in MEMS applications.

References

[1] Lin H, Xu L Q, Chen X, Wang X H, Sheng M, Stubhan F, Merkel K H and Wilde J 1998 Thin Solid Films **333** 71
[2] Wuu D S, Lo W C, Chiang C C, Lin H B, Chang L S, Horng R H, Huang C L and Gao Y J 2005 Surf. and Coat. Technol. **198** 114
[3] Yota J, Janani M, Camilletti L E, Kar-Roy A, Liu Q Z, Nguyen C and Woo M D 2000 IEEE 76
[4] Huang H, Winchester K, Liu Y, Hu X Z, Musca C A, Dell J M and Faraone L 2005 J. Micromech. Microeng. 2005 **15** 608
[5] Tarraf A, Daleiden J, Irmer S, Prasai D, Hillmer H 2004 J. Micromech. Microeng. **14** 317
[6] van de Ven E P, Connick I W, Harrus A S 1990 Proc. of the VLSI Multilevel Interconnection Conference (VMIC) 94
[7] Loboda M J, Seifferly J A 1996 J. Mater. Res. **11** 391
[8] Machenzie K D, Reelfs B, Devre M W, Westerman R, Johnson D J 2004 Chip **10** 26
[9] Smith D L, Alimonda A S and von Preissig F J 1990 J. of Vacuum Sci. & Technol. B **8** 551
[10] Hess D W 1984 J. of Vacuum Sci. and Technol. A **2** 244