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Passivation Layers Deposition: The Effect of Temperature and Nitrogen Flow Rate on the Thin Film Formation

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ABSTRACT

Taguchi method was performed on a series of ultra-thin Si$_3$N$_4$ films in order to study the parameter affecting the Nitride growth. The samples of ultrathin Si$_3$N$_4$ were prepared through a dry nitridation/oxidation method using a high temperature furnace. There are three levels of temperature used, that are 750, 800 and 950$^\circ$C. The samples were grown in 0.333 litre/min, 0.667 liter/min and 1 liter/min nitrogen flow rate and with variation in time that is 1, 2 and 3 minutes. The thickness characterization was done by ellipsometer. The thickness of the nitride was obtained and is ranging from 1 to 5 nm. All the data were interpreted using Taguchi method to optimize the processer parameters to fabricate Si3N4 films. The most affecting factors in producing an ultra-thin silicon Nitride using a high temperature furnace.

Key words: Ultra-thin silicon nitride; Taguchi’s method; LPCVD; Microfabrication; MEM Devices

Introduction

The most critical role of silicon Nitride is, its use as a MEM devices for dielectric or used as various isolation layer in semiconductor fabrication. The influence of N-atoms at the Si (100)/dielectric interface would be detrimental to the electrical properties of devices and the methods were therefore optimized for preventing N-atom diffusion to the interface (Bahari, M., Riaziyan and R. Ahmadnejad, 2009; Marras, I.D., et al., 2004; Bahari, A., 2008; Adam, T., et al., 2013). Another demand is that the Nitride thickness must be controlled to the desired thickness that matches the design specifications of the standard fabrication requirements. Considering the application of ultra thin silicon Nitride film with the thickness of a few atomic layers to stacked gate dielectrics in CMOS devices for example, even single atomic steps at the interface or small roughness on the surface should be eliminated (Adam, T., U. Hashim, K.L. Foo, 2013; Adam, T., et al., 2013; Adam, T., et al., 2013; Adam, T., et al., 2013; Adam, T., U. Hashim, 2012; Adam, T., U. Hashim, 2012). The thickness of the nitride must be less than 3 nm to be considered as an ultra thin film (Adam, T., U. Hashim, 2012). This is in a very small scale so any different in thickness even if 1 nm would give a different characteristic (Adam, T., U. Hashim, 2012; Adam, T., U. Hashim, 2012; Adam, T., et al., 2012; Hashim, U., et al., 2012). Therefore uniformity of the Nitride is a must for achieving a good thin film Nitride (Al-Mufti, W.M., et al., 2012; Adam, T., U. Hashim, 2012; Adam, T., U. Hashim, P.L. Leow, 2012; Adam, T., U. Hashim, 2012; Hashim, U., et al., 2012; Hashim, U., et al., 2012; Rao, B.S., et al., 2012). The thickness must be sufficiently uniform across the wafer, wafer to wafer and from run to run (Adam, T., U. Hashim, P.L. Leow, 2012; Hashim, U., T. Adam, 2012; Adam, T., U. Hashim, U.S. Sani, 2012; Hashim, U., et al., 2012). Even though nitridation at higher temperature, has been reported as effective to reduce the surface roughness, but still it is insufficient and yet should be improved. In this experiment, Taguchi method is used to find and examining the interactions between factors (Adam, T., et al., 2012; Hashim, U., et al., 2012). The equilibrium between levels of different factors, robust tolerance design, and costs is based on two main concepts proposed by Taguchi: quality loss functions and signal/noise ratio (S/N). According to Taguchi’s quality engineering philosophy and methodology, there are three important steps in designing a product or process: system design, parameter design and tolerance design. The aim of system design is to create a product that indeed possesses the properties intended for it at the planning stage (Adam, T., et al., 2012; Hashim, U., et al., 2012; Xiao, H., 2001; Peng, L.-H. et al., 2005; Bryant, M., 2004; Phadke, Madhav S., 1989; Hashim, U., A.B. Shahrrul, 2012). This involves the development of a prototype, choice of materials, parts, components, assembly system and manufacturing processes, so that the product fulfills the specified conditions and tolerances at the lowest costs. The thickness of the nitride must be less than 3 nm to be considered as an ultra thin film. With the Taguchi methods perform on a series of nitride, the thicknesses of all possible combination of parameters were able to be predicted. This will

Materials And Methods

Details of the experimental setup are summarized as follows. The substrate is <100> silicon wafers. The pre-nitridation cleaning sequence consisted of H₂O₂-based solutions of NH₄OH and HCl with appropriate DI water rinses, followed by a dip in dilute HF and a final DI water rinse (Adam, T., U. Hashim, 2012; Chee, P.S., et al., 2012; Adam, T., et al., 2012; Adam, T., et al., 2012; Adam, T., U. Hashim, 2012; Hashim, U., et al., 2012). The wafers were then dried using nitrogen and immediately loaded in the nitridation furnace with nitrogen flowing. The silicon nitride is grown by dry nitridation method using a high-temperature furnace. The silicon nitride layers had being grown from the range of 1 to 3 minutes with temperatures at 750, 800 and 950°C and with the differences in nitride flow rate. The nitride flow rates used are 0.333, 0.667 and 1 litre/minute. Upon reaching the target nitridation temperature, the furnace ambient was then switched to dry nitridation/oxidation. The parameters are arrange in an standard L9 orthogonal array, which means for any pair of columns, all combinations of factor level occur an equal number of times. Detail of the parameters is shown in table 1. The thicknesses were then measured using elipsometer. All the thickness data obtained had been interpreted using Taguchi’s method to achieve the best parameter of acquiring the thickness below 3 nm.

Results and Discussions

The aim of system design is to create a product that indeed possesses the properties intended for it at the planning stage. This involves the development of a prototype, choice of materials, parts, components, assembly system and manufacturing processes, so that the product fulfills the specified conditions and tolerances at the lowest costs. The thickness of the nitride must be less than 3 nm to be considered as an ultra-thin film. With the Taguchi methods perform on a series of nitride, the thicknesses of all possible combination of parameters were able to be predicted. This will reduce the cost and time of the actual experiment.

![Graphical representation of control factors and their levels](image)

**Fig. 1:** Graphical representation of control factors and their levels

Graph presented in Figure 1 is the S/N value of each factor that is affecting the nitride thickness. The graphical representation is also convenient for drawing qualitative inferences and choosing the optimum level. The optimum level of factor can be achieve by choosing the highest S/N ratio for each factor, this way the nitride thickness below 3nm is achieved. The slope of each factor shows the amount of changes that affect the thickness of the silicon nitride. The statistical design of experiment was used to evaluate three process variables that are temperature, time and the amount of flow rate. The analysis of the graphical value indicated that the temperature is the most significant factor in nitride growth. Time and nitride flow rate also effect the nitride thickness.
Fig. 2: Shows the profile of the silicon nitride (Si₃N₄) on the surface of substrate

Fig. 1. Shows the thickness parameters of ultra-thin silicon nitride grown using a high temperature furnace, the thickness varies from 1 nm to 5 nm. From the graph, all of the nitride thicknesses are linearly increasing with the process time. As shown in figure 2, the nitride grown at 700°C with 1 minute time in 0.333 l/min nitride flow rate will have a thickness of 1.13 nm, and by increase the time to 2 minutes, the thickness will increased to 1.37 nm. The difference is about 0.25 nm. Then the increase of time to 3 minutes will increase the thickness to 1.67 nm. The difference of the thickness is about the same. The thickness is also dependent with temperature and nitride flow rate. The slope in fig. 1 represents the growth rate of nitride. The higher the process temperature, the higher the slope value will be and thus will increase growth rate of the thin nitride. The increase in nitride flow rate will also affecting growth rate the same way as the process time. However, the thickness of nitride grown in 1 l/min flow rate will decrease the thickness to less than the nitride grown in 0.667l/min instead of increasing the nitride thickness.

Fig. 3: Shows the distribution of the layers on the wafer

Fig. 4: Show the profile of the deposited silicon-nitride with optimized temperature and nitrogen flow rate produced surface roughness of less than 10 nm
**Fig. 5:** Show the profile of the deposited silicon-nitride with non optimized temperature and nitrogen flow rate with surface roughness between 10 to 20nm.

By LPCVD nitride deposition primarily stoichiometric silicon nitride with low tensile stress is formed (Adam, T., et al., 2012; Hashim, U., et al., 2012; Adam, T., et al., 2012; Weir, E. et al., 1999; Chung, J-H., et al., 2006; Darder, M., et al., 2006; Xiao, H., 2001) and this can be seen in figure 2 and 3. By increasing the gas flow of nitrogen silicon-rich silicon nitride is formed. The process can be adjusted so that this nitride and coupled with temperature variation is almost stress free and good uniformity can be formed. In combination with the excellent properties of thermal silicon nitride and the degree of the uniformity we obtained is well suited for the purpose being deposited. However many particles are formed during such a process and it requires a lot careful experimental process to minimize surface roughness, this can be seen in fig. 4 and 5. The figure 4 show less than 10nm which is an excellent results and fig. 5 shown a surface roughness between 10 and 20. In conclusion, thermal nitride has high integrity than most CVD Nitride film and so far has demonstrated high uniformities, less defect and high dielectric strength than deposited nitride thin film. Thermal nitride is normally grown in a diffusion furnace at a high temperature using either wet or dry growth method. Dry nitride growth rate is much slower than wet for this reason dry deposition are primarily used for thin nitride and oxide where high uniformity and high dielectric strength are obtained.

**Conclusion:**

The statistical design of experiment was used to evaluate three process parameters that are temperature, time and the amount of nitrogen flow rate. The analysis of the graphical value in figure 2 indicated that the temperature is the most significant factor in nitride growth. Time is factor dependence to temperature. If higher temperature were used, the time will of course need to be shortened to achieve an ultra thin nitride. It is merely a constant rate which affects significantly only to the nitride thickness and the amount of the thickness increase is about the same. The point at higher amount of flow rate (1 l/min) will eventually reduce the nitride thickness compare to the nitride grown in 0.667 l/min flow rate, we believe that the statistical design of experiment can be employed to control and optimize the effect of all factors in ion implantation, deposition CVD/LPCVD processes so that it will make the realization of achieving ultra thin silicon nitride surface come true as it is becoming even more challenging as circuits are made denser and all of the dimensions are reduce correspondingly.

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