Stress release of PECVD oxide by RTA

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ABSTRACT

The effects of various deposition and annealing conditions of Plasma Enhanced Chemical Vapour Deposited (PECVD) oxide films on residual stress, optical index, BHF etch rate, surface roughness for thick PECVD oxide films are investigated. Rapid Thermal Annealing (RTA) and annealing in regular furnaces are both considered and compared. AFM measurements were required to measure the oxide surface roughness. Thick PECVD oxide films of 1.5 µm were deposited on 380 µm bulk silicon wafer. Starting from a conventional recipe the power, the [N_2O/SiH_4] ratio and the total gas flow were successively varied. An increase of the total gas flow or/and a decrease of power lead to an increase of the BHF etch rate and surface roughness but on the other hand decrease the residual stress. A high ratio N_2O/SiH_4 yields to oxide with low BHF etch rate but characterized by higher residual stress. RTA reduces the stress and the BHF etch rate drastically and it is more efficient than conventional annealing in standard furnace. Actually, RTA reduces stress in a very short period of time (few seconds) compared to standard annealing, and then it does not contribute to doping diffusion of the already implanted regions of the wafer. From a compressive stress of about 80 MPa, RTA of 15 s leads to a residual stress of only 30 MPa and bring the BHF etch rate to acceptable values in the range of 2000 Å/min for high N_2O/SiH_4 ratio oxide. One main application of this RTA stress release of oxide is to provide processed wafers with a bow compatible(less than 10 µm) with Chemical Mechanical Polishing (CMP) or wafer bonding.

Keywords: RTA, PECVD oxide, stress release, etch rate

1. INTRODUCTION

PECVD silicon oxide is widely used in the fabrication of microelectronic devices and microelectromechanical systems (MEMS). Due to the low deposition temperature, PECVD is very convenient for processes with low thermal budget. Furthermore, plasma instead of heat ignites the chemical reaction and allows reaching higher deposition rate. It is thus used as thick sacrificial layer in MEMS[1] fabrication process, for integrated optics applications[2] or as passivation layer in microelectronic devices.

One of the drawbacks of the PECVD silicon oxide films is the residual stress which limits the maximal thickness of the films to avoid films cracking and prevents wafer bonding or CMP because of too high wafer bow. On the other hand, PECVD allows the tuning of different process parameters like RF Power, ratio of the different precursor gases, total flow rate, or even deposition temperature. Changing these parameters gives a freedom in the choice of some properties of the deposited oxide layer like optical index, BHF etch rate, density, residual stress[3] but do not always lead to a low stress oxide. Densification by RTA overcomes this inconvenient and decreases drastically the stress[4] of the deposited layer and improves simultaneously the other characteristics such as BHF etch rate and optical index. It has the advantage of being non time consuming and compatible with a CMOS process as RTA does not induce more diffusion than doping species activation. The RTA step can even be made after an implantation step in order to combine stress release and doping activation.

In the first part of the study, the RF power, the ratio between the two precursor gases (nitrous oxide and silane 5%), and the total gas flow in the reactor chamber were varied and the effects on the oxide properties measured before and after a

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RTA step. The second part of the study shows for a given oxide the influence of the RTA time, the RTA temperature and compares it to a conventional furnace annealing.

2. EXPERIMENTAL SET-UP

Three inch diameter p-type <100> 380 µm thick bulk silicon wafers were used as substrates. The PECVD of silicon oxide was made in an OXFORD Plasmalab System 100. The size of the deposition chamber allows to process three wafers simultaneously. The chamber consists of a top electrode with high uniformity shower head gas inlet top separated by substrate electrode heated and grounded. The frequency of the RF power generator applied to the top electrode is 13.56 MHz. Through the whole batch of experiment the pressure in the chamber was kept at 1 Torr and the substrate electrode temperature was also kept constant at 300°C. The two precursor gases used to form the silicon oxide were nitrous oxide (N_2O) and silane (SiH_4) diluted in nitrogen at a concentration of 5%. In the following, silane or SiH_4 stands for silane 5%.

Variation range for the parameters were: from 200 sccm to 1050 sccm for the total flow rate, from 5 to 17 for the ratio $[N_2O]/[SiH_4]$, and from 30 W to 100 W for the RF power. The deposition time was 20 minutes, in order to get a film thickness between 10000 Å and 20000 Å, the deposition rate depending strongly on the deposition parameters.

The thickness of the deposited film is measured with a Prometrix Spectramap System 100. It uses spectral analysis of reflected white light in the 400 – 800 nm range to provide accurate and repeatable thickness measurements from 200 Å to 5 µm on 3 inch wafers. This computer controlled system collects data on 10 sites per wafer and gives the mean value of the measured thickness, the standard deviation and the range of the series of the measured points.

The residual stress of the oxide layer is deduced by a measurement from the strain induced in the silicon substrate by the oxide layer deposition (change in wafer curvature). The wafer curvature before and after the deposition is obtained using the Dektak profilometer over a long distance (50 mm). The profilometer gives the maximal deflexion of the wafer surface over the length of the scale (Figure 1).

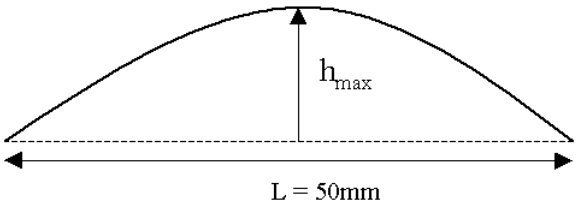


Figure 1: Evaluation of the radius curvature by profilometer scan.

The curvature radius is approximated by:

$$R = \frac{\left(\frac{L}{2}\right)^2}{2 * h_{\max}},$$

where h_{max} is the maximal deflection of the wafer surface and L the scanning length. Once the strain is determined, the stress is easily calculated using the Hook's low for elastic deformation. If the film thickness is much lower than the wafer thickness, and the stress isotropically distributed through the cross section of the film, one can derive the Stoney equation[5]:

$$\sigma = \frac{E}{6*(1-\nu)} * \frac{t_{sub}^2}{t_{film}} * \left(\frac{1}{R_{post}} - \frac{1}{R_{pre}} \right)$$

where σ is the stress in the thin film, E and υ, respectively the Young modulus and Poisson coefficient of the substrate, t_{sub} and $t_{film,}$ respectively, the substrate and film thicknesses and R_{pre} and R_{post} respectively the curvature radius of the wafer before and after deposition. As the wafers do not have the same curvature radius before deposition (the h_{max} value can vary from 600 nm up to 15 µm), the curvature radius was measured before and after deposition.

Using the 1.5 µm thick films to determine the optical index of the deposited oxide with an ellipsometer would have given inaccurate results while the thickness is known modulo the ellipsometer period which is approximately 2000 Å for silicon oxide and the accuracy of the optical index is the best when the thickness of the measured layer is close to ¾ of the ellipsometer period modulo of the measured material. Thus the optical index measurement was done on films of thicknesses around 1500 Å in order to get the highest resolution.

The BHF used to measure the etch rate was prepared in the following proportions: 87% NHF_4[40%], 13% HF[49%] in volume. The etching is performed at 20°C without shaking.

An ADDAX RTA oven with a single 3 inch wafer chamber is used for the rapid thermal annealing. As observed by Carlos Dominguez *et al.*[6], the ambient gas during annealing does not have any impact on the effect of the RTA. Thus the atmosphere during RTA was pure nitrogen, with a flow rate of 4 l/min during the heating phase and a flow rate of 6 l/min during the cooling phase. The heating rate was 700 °C/s and the cooling was efficient enough to reach a temperature of 500 °C in less than 2 seconds after a heating at 950 °C. The temperatures were varied between 400 and 950 °C and the annealing time between 2 and 30 s.

3. RESULTS AND DISCUSSIONS

Oxford instrument gives a standard recipe for PECVD oxide. All the tested recipes were derived from that one, just varying one parameter at a time. The given standard oxide is deposited in the following conditions: pressure 1 Torr, power 100 W, total gas flow 600 sccm, ratio [N_2O/SiH_4]=7, table temperature 300 °C.

3.1. Gas flow

Firstly the total gas flow was varied from 200 sccm to 1050 sccm keeping all the other parameters constant (1 Torr, 100 W, 300 °C, [N_2O:SiH_4]=7). All the interesting physical properties were measured before and after a 900 °C RTA. Figures 2 to 5 plot the evolution of these properties in function of the total gas flow.

The wafers had a nice aspect after deposition, the film thickness was homogenous over the whole wafer for total gas flow over 400 sccm. Below this value, the standard deviation of the measured thickness was 3%. This effect is due to the volume of our deposition chamber, which allowing to process three wafers at a time requires a minimum gas flow through the chamber to have an homogenous diffusion of the gases inside the chamber. The deposition rate increases linearly with the total gas flow, letting us assume that all the gases react and give rise to more oxide when more reactants are present. The optical index decreases as the total gas flow increases. A low total gas flow yields a dense oxide due to a high transit time of the reactants in the reactor, characterized by a high optical index as can be predicted by the Lorentz-Lorentz[7] equation which indicates that the optical index increases as density increases and vice-versa. Inversely a high total gas flow gives a silicon oxide with a lower optical index, which can be explained by a lower density of the deposited silicon oxide, the deposition rate being higher for high total gas flow. The RTA makes the optical index drops for all the samples. It could appear to be in contradiction with the process of densification that the film suffers during the RTA, but as it is described in the literature[8,9], H and N atoms impurities that were trapped are released and the deposited film comes closer in composition to stoichiometric oxide and the optical index decreases.

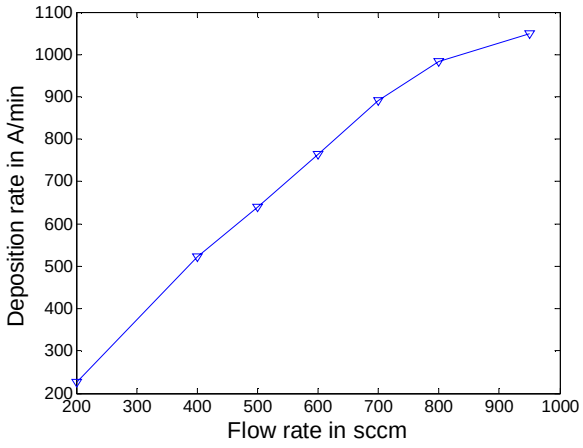


Figure 2: Deposition rate in function of the total gas flow.

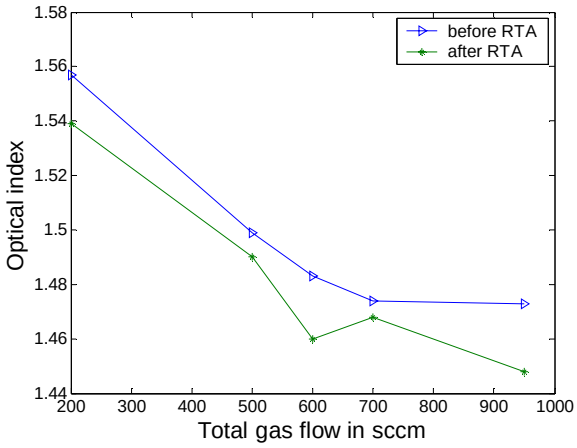


Figure 3: Optical index in function of the total gas flow.

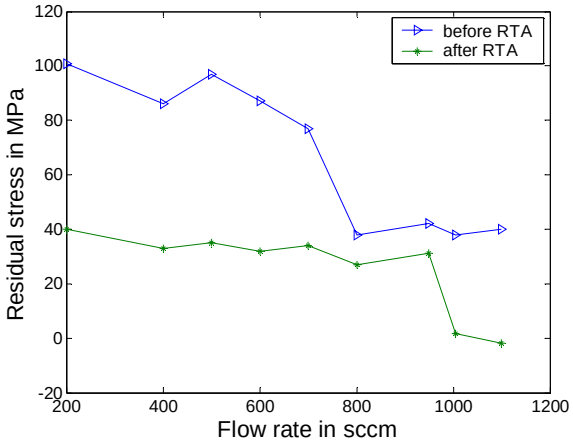


Figure 4: Residual stress in function of the total gas flow.

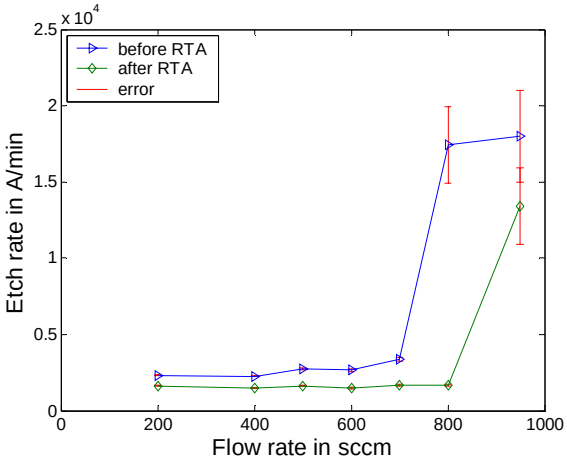


Figure 5: Residual stress in function of the total gas flow.

The residual stress and the etch rate are strongly correlated. Though a small increase in the etch rate pre and post annealing correlated to a slight decrease of the residual stress pre and post annealing when increasing the flow rate up till 700 sccm, the etch rate increases drastically when increasing the total flow rate above this value with a simultaneous drop in the residual stress. The RTA allows to keep an acceptable etch rate of 3000 Å/min at 800 sccm while having the residual stress considerably reduced from 45 to 32 MPa. Above this total gas flow, it is not possible any more to control the etch rate, the error bars on the graph indicating the non homogeneity of the etch rate. On the other hand the residual stress in that case becomes null after RTA, which can be an advantage if the films is used as passivation layer or as bonding interface. One disadvantage of those low stress high etch rate oxide is that it is not stable over the time. This point will be detailed later on.

3.2. RF Power

The power was then varied from 30 to 100 W. Total gas flow was kept at 600 sccm, pressure at 1 Torr, $[N_2O]/[SiH_4]$ at 7 and the table temperature remained constant at 300 °C. Figures 6 to 9 plot the measurements done for these samples before and after RTA.

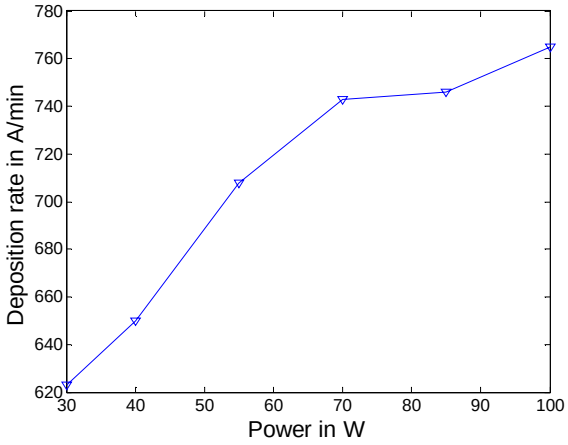


Figure 6: Deposition rate in function of the power.

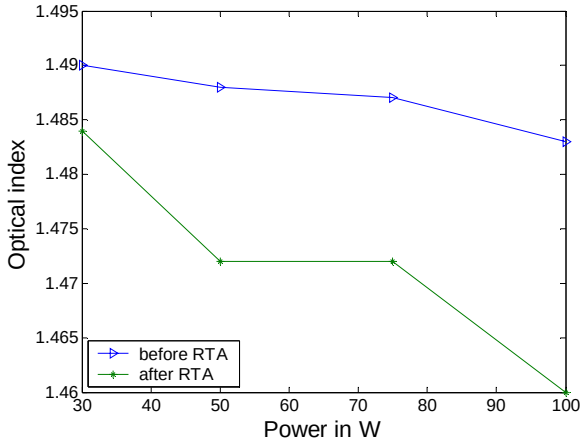


Figure 7: Optical index in function of the power.

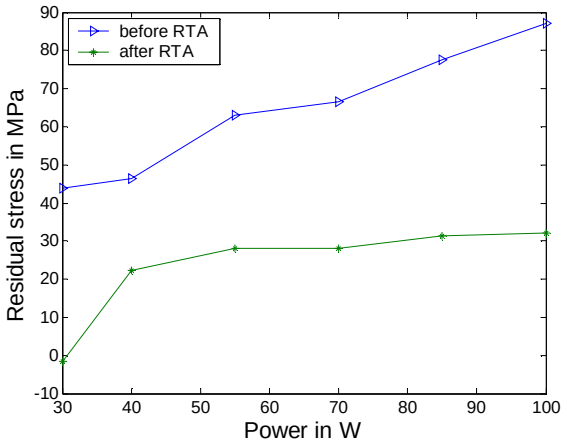


Figure 8: Residual stress in function of the power.

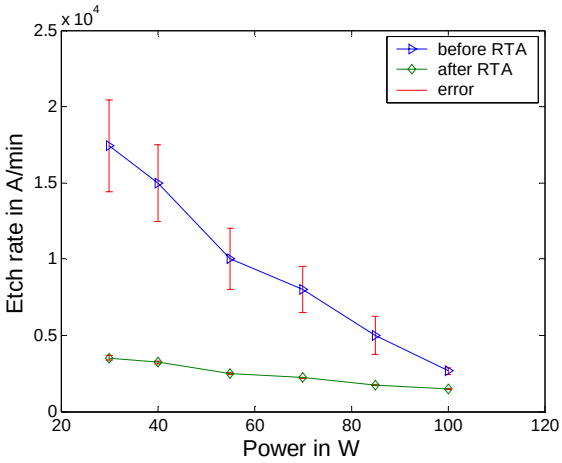


Figure 9: Etch rate in function of the power.

The deposited films have a homogenous thickness over the whole wafer, the measured value having a standard deviation around 0.7%. The deposition rate increases slightly with the power, but not of a significant manner, only 20% faster from 30 to 100 W. Increasing the power does not give real advantage for the deposition rate. The optical index before RTA decreases also slightly as the power increases but of a non significant magnitude. Previous studies[10] showed that the quality of the PECVD film strongly depends on the ratio $\eta P/F$, where η is the gas dissociation efficiency, P the power and F the total gas flow. A too low ratio will yield poor quality oxide with voids and nodules[11] and weakly bonded atoms, whether a high ratio means that the energy transfer process is well matched to the presence of ion species and leading to a well bonded atoms in the deposited layer. It explains that the value of the optical index tends toward the one of thermal oxide as the power increase. The diminution of optical index after RTA is much more pronounced for

oxide deposited at high power. At higher power, the ionization of the precursor gases is more complete and the grown oxide does not have voids or particles. During the RTA, N and H release makes the optical index drop. For low power oxide, the optical index drop due to the impurities release is compensated by the densification which makes the optical index increase, therefore is the optical index diminution smaller for low power oxide. The stress and etch rate behavior can also be explained in the same way. Stress post deposition continuously decreases when the power decreases. At small power, the atoms being weakly bonded in the silicon oxide layer, they relax the internal stress by forming voids. The rapid thermal annealing decreases always the stress by his effect of re-organization of the deposited layer. For a power of 30 W, the stress becomes null but the stress varies with the time. This point will be mentioned later on. The etch rate decreases continuously when the power increases. For low power, the etching of the deposited film in BHF is totally non uniform, the etching rate being determined with an uncertainty of 5000 Å. The silicon oxide formed at low power has weakly bonded atoms, so easily etch by HF. The RTA decreases drastically and make homogenous the etch rate. At low power, the etch rate is in the order of 4000 Å/min, which can be a handling value. The oxide deposited at 30 W seems to be a good candidate for bonding and passivation applications, except its temporal instability.

3.3. Precursor gases ratio [N2O/SiH4]

The ratio of the two precursor gases [N_2O/SiH_4] was varied from 5 to 17. Total gas flow was kept at 600 sccm, pressure at 1 Torr, power at 100 W and the table temperature remained constant at 300 °C. Figures 10 to 13 plot the measurements done for these samples before and after RTA.

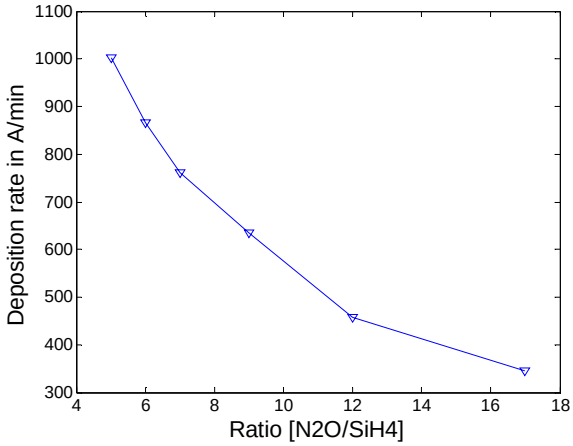


Figure 10: Deposition rate in function of the [N_2O/SiH_4] ratio.

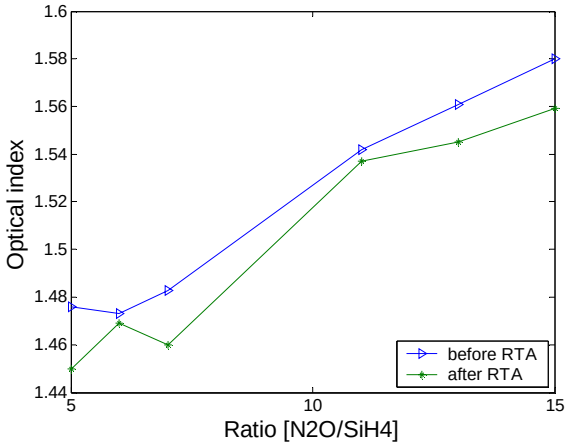


Figure 11: Optical index in function of the [N_2O/SiH_4] ratio.

The deposition rate increases when the silane partial pressure increases. The limiting factor for oxide deposition rate is the quantity of Si ions presents in the plasma. The optical index decreases with the augmentation of Si ratio in the plasma. At high [N_2O/SiH_4] ratio, more Si-O bonds are formed whether at low [N_2O/SiH_4] ratio more Si-H bonds are formed[12,13]. Although a small [N_2O/SiH_4] ratio gives an oxide of optical index close to the one of thermal silicon dioxide, Figures 12 and 13 show that an oxide made of plasma with small a [N_2O/SiH_4] ratio is of poor quality. For too high silane concentration (below a gas ratio of 6), the etch rate increases and is not homogenous over the whole wafer surface. We observe conjointly with the etch rate increase a decrease of residual stress. For Si-rich PECVD silicon oxide[14] the bond reaction energy is lower than for stoichiometric silicon dioxide, explaining the higher reactivity with BHF. As for the previous samples the RTA decreases the optical index, the residual stress and the BHF etch rate. The mechanisms involved are the same as for the two previous batches of experiences. For a gas ratio of 6, the etch rate is brought back from 5000 to 2000 Å/min, which is an acceptable value to control oxide etching in a process. Null stress is not achieved by varying the gas ratio.

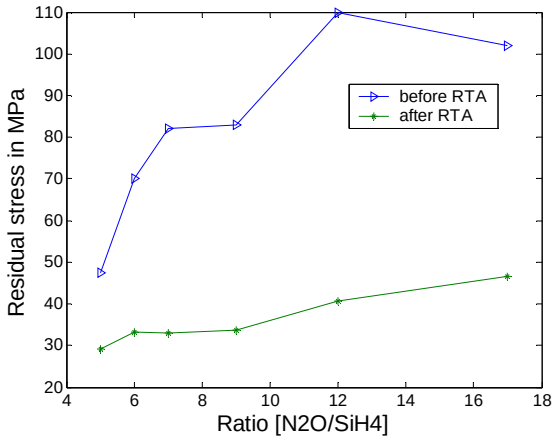


Figure 12: Residual stress in function of the [N_2O/SiH_4] ratio.

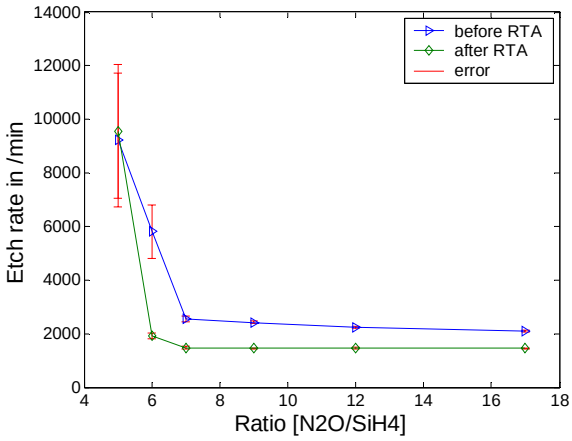


Figure 13: Etch rate in function of the [N_2O/SiH_4] ratio.

3.4. Effects of RTA

Various RTA times and temperatures were tested on samples with an 1.5 µm thick oxide layer. The experimental conditions for the deposition of this layer were: 300°C, 100 W, 600 sccm, ratio [N_2O/SiH_4]=7, 1 Torr. The RTA times were varied from 3 to 30 s (Fig. 15) and compare with a conventional annealing of 30 min. at 900°C in 1.5 l/min N_2. The RTA temperature was increased from 400 to 950°C (Fig. 14). Etch rate and residual stress were then measured.

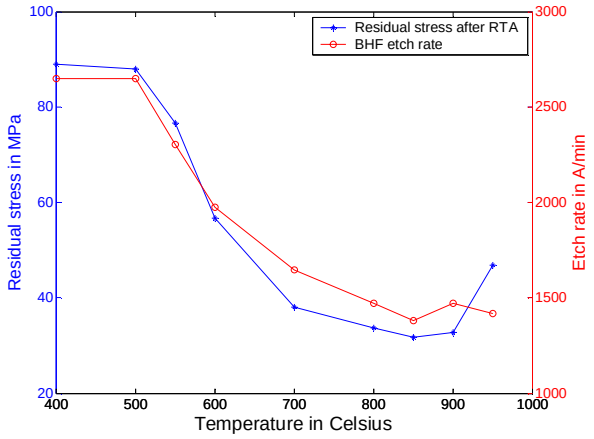


Figure 14: Effect of RTA temperature on residual stress and etch rate.

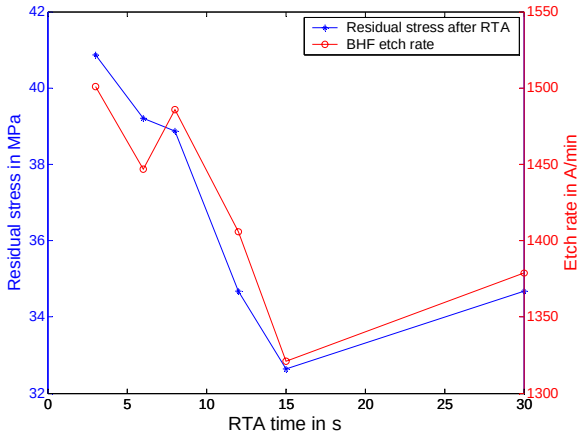


Figure 15: Effect of RTA time on residual stress and etch rate.

Below 500°C RTA has no impact on the deposited oxide. No reconstruction or impurities release occurs, as shown by the stability of the two measured parameters. As the temperature increases up till 950°C stress and etch rate are decreasing with the same behaviour. The literature[15,16] provides many explanations for this change in properties. The non stoichiometric $SiO_xN_yH_z$ film will release H and N atoms during the annealing and experiences the following network reaction:

$$SiO_x \rightarrow \left(x/2\right)SiO_2 + \left(1 - x/2\right)Si$$

Below 700°C, H is released but the bonds Si-O do not suffer any re-arrangement. Above 700°C re-organisation of Si-O in SiO_2 occurs. In our experiences we did not measure the concentration of Si-H, Si-N, Si-O bonds, but we measured the macroscopic properties. A RTA of 900°C during 15 s seems to be enough to reach the best properties that one can get for a given oxide. The etch rate and the residual stress do not change significantly above 900°C. Although teh measurement in Figure 15 are quite noisy, it appears that the RTA time has also an influence. The residual stress and the BHF etch rate reach their minimal value in 15 s, respectively 33 MPa and 1330 Å/min. Taking into account the measurement error, one can say that the etch rate and the residual stress do not vary any more above 15 s. After conventional furnace annealing (30 min. 900°C under nitrogen atmosphere) the residual stress reaches a value of 40 MPa and the BHF etch rate a value of 1290 Å/min. It appears clearly that RTA is the best method to anneal a PECVD oxide. The gain in etch rate reduction are similar to those get from conventional furnace annealing, the gain in stress reduction are even better and the thermal budget is fairly low and does not induce extra diffusion. As will be shown in the following section, the grain size increases during RTA and the roughness decreases.

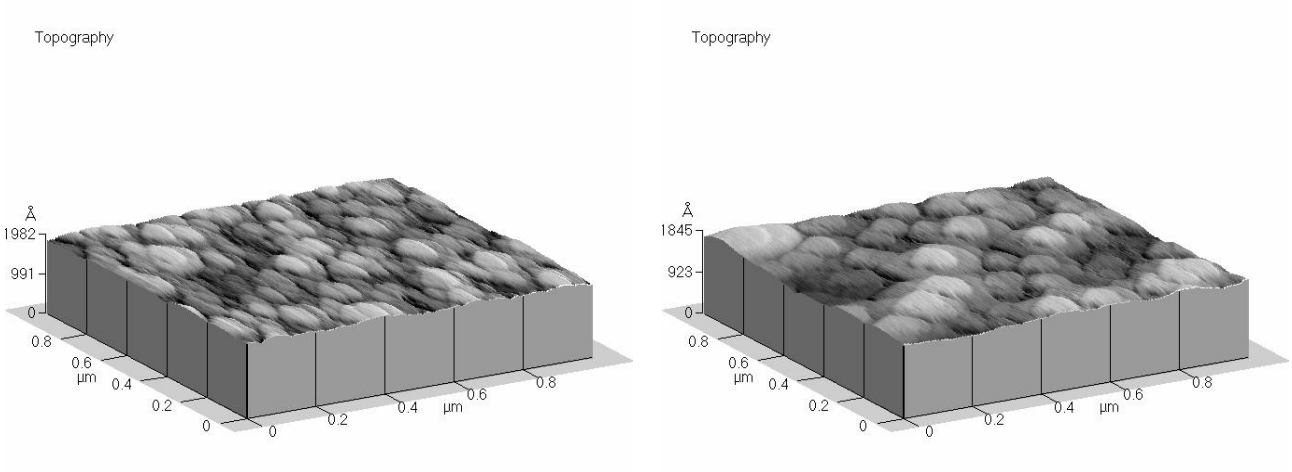
3.5. Roughness measurement

AFM was used to study the roughness before and after annealing. Table 1 shows the average and root mean square roughness for different samples before and after RTA. The temperature and pressure during deposition were for all of these samples 300°C and 1 Torr, respectively. As previously stated, a low $\eta P/F$ ratio yields an oxide with weakly bonded atoms, larger grains and voids. The roughness measurement confirms this fact: the oxide deposited at 100 W, 600 sccm and with a gas ratio of 7 presents a smaller roughness than the one deposited at 30 W and than the one deposited with a total gas flow of 1050, keeping the other parameters constant. Changing the gas ratio has also an influence on roughness but not so strong than varying power or total gas flow. A Si-rich oxide has a larger roughness than stoichiometric oxide. RTA has, depending on the quality of oxide, a positive or negative impact on roughness. It decreases a bit the roughness for the oxide deposited at 30 W and 100 W with a gas ratio of 7 and a total gas flow of 600 sccm, but increases the roughness in the other case.

Process conditions	**Rms roughness before RTA**	**Average roughness before RTA**	**Rms roughness after RTA**	**Average roughness after RTA**
30 W [N_2O/SiH_4]=7 600 sccm	40.7 Å	32.4 Å	39.2 Å	32.2 Å
100 W [N_2O/SiH_4]=7 600 sccm	35.6 Å	28.4 Å	34.4 Å	27.8 Å
100 W [N_2O/SiH_4]=5 600 sccm	38.6 Å	31.2 Å	39.9 Å	32.2 Å
100 W [N_2O/SiH_4]=7 1050 sccm	45.2 Å	35.7 Å	53.6 Å	42.3 Å

Table 1: Surface roughness before and after RTA

AFM pictures show that for all the samples the grain size is increased during RTA (Fig. 16). The pictures presented here are for the oxide deposited at 1050 sccm, 100W, [N_2O/SiH_4] ratio of 7.



(a) (b)

Figure 16: topography before RTA

3.6. Bondability

One the purpose of this stress release technique is to decrease the bow of the wafer in order to enhance CMP quality on the wafer, bonding interface for packaging or 3-D microstructure fabrication. To achieve bonding, the wafer must have a bow less than 15 µm and a surface root mean square roughness less than 5 Å. In the best case the root mean square roughness is 34.4 Å. It is too high to make wafer bonding. The wafer will have to be polished until a surface roughness of 5 Å is reached, to achieve good bonding. The maximal bow allowed for a uniform CMP is 25 µm. Wafer bonding is the limiting factor for maximal wafer bow in our case. To decrease the wafer bow, on can either decrease the film thickness or decrease the residual stress in the wafer. For the applications requiring a thick film, the only solution is to decrease the stress. For a wafer with a initial bow of 5 µm (which is quite common), the bow after deposition for a 1.5 µm thick film with a residual stress of 35 MPa is around 20 µm. The only solution to achieve bonding is to use oxides made at low power or total gas flow or small $[N_2O/SiH_4]$ ratio. Oxide deposited at high total gas flow (around 1000 sccm) or low power (30 W) can achieve null stress but as is presented in the next section this kind of oxide is not stable over time. Si-rich oxide ($[N_2O/SiH_4]$=5) presents also a strong diminution of the stress (around 20 MPa) and has also a bow acceptable for CMP and wafer bonding in the range of thickness investigated.

3.7. Time evolution

As mentioned above, we also observed variations of stress for low density oxides. The variations have been measured after deposition and after RTA for two different oxides. Conserving a total gas flow of 1050 sscm, we used the following deposition parameters: 1 Torr, 100 W, 300 °C. The thickness of the silicon oxide film is 15000 Å.

First, we studied silicon oxide with nitrous oxide and silane flow of 850 sccm and 155 sccm respectively ($[N_2O]/[SiH_4]$ = 5). The logarithmic function of time for stress measured after deposition is illustrated in Figure 17. Since there is a plateau above 200 min, we distinguish two time constants. At first, the residual stress increase rapidly to approach the final value. Secondly, the time constant varies very suddenly and takes a value near to infinity. We assume that it exists an upper limit to the residual stress of the oxide layer while the time evolution curve almost fits to a function of the type:

$$\sigma = \sigma_\infty - K \cdot \exp\left(\frac{-t}{\tau}\right),$$

where $K = \sigma_\infty - \sigma_0$, σ_0 is the initial residual stress and σ_∞ its final value and τ the time constant of the phenomenon.

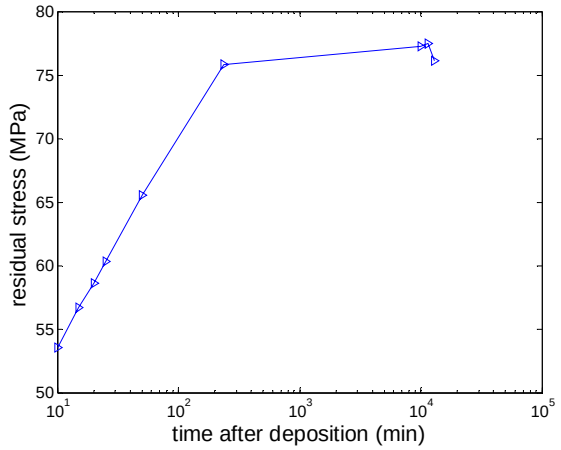


Figure 17: Residual stress in function of time after deposition.

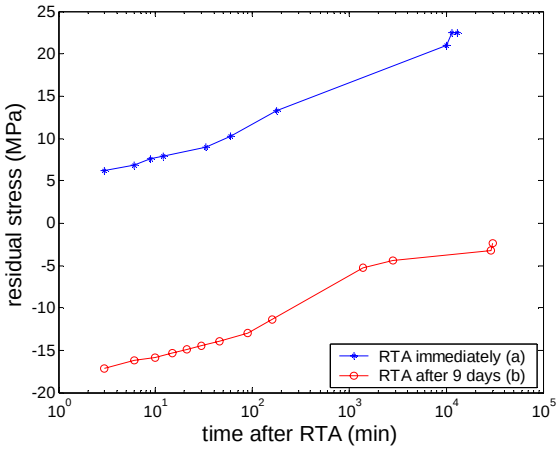


Figure 18: Residual stress in function of time after RTA.

In Figure 18, we illustrated the influence of the time between deposition and RTA on the final stress. The curve (a) was obtained by applying RTA treatment (950 °C, 2 sec) immediately after deposition. The curve (b) was obtained by submitting the wafer to a rapid thermal annealing 9 days after deposition. The influence of the time between deposition and RTA involves a shift of the residual stress. These results are quite surprising, since the stress is much more elevated nine days after deposition and this produces a final stress actually smaller and even negative (tensile stress instead of compressive stress). The evolution of stress after RTA is also logarithmic with a single time constant.

Second, the same experiment has been donewith an other silicon oxide which had been deposited with the following gas flows: $[N_2O]$=880sccm and $[SiH_4]$=125, giving $[N_2O]/[SiH_4]$=7, all other parameters being kept identical to the previous one. Results are illustrated in Figures 19 & 20.

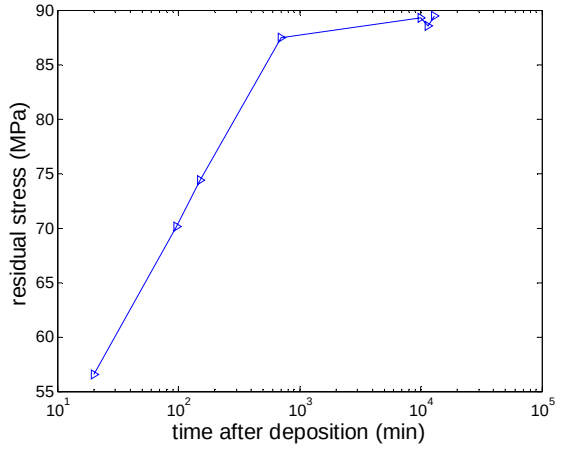


Figure 19: Residual stress in function of time after deposition.

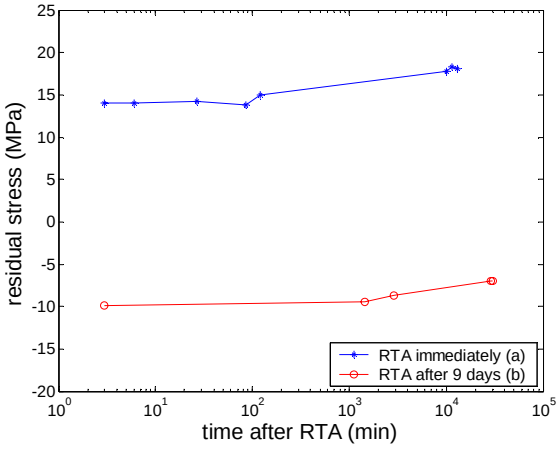


Figure 20: Residual stress in function of time after RTA.

The behavior of the wafer is qualitatively the same as the precedent. However, the time constants are different: the stability is reached at about 700 min. The shift (between (a) and (b)) of the stress curve is also observed. But this one is much more stable over the time. In both cases, the loss of stress by waiting nine days before applying RTA is about 25 MPa.

4. CONCLUSION

PECVD oxide with low etch rate and low residual stress was achieved. We tuned all the deposition parameters to get an oxide with the desired quality. It appears that the ratio $\eta P/F$, where η is the gas dissociation efficiency, P the power and F the total gas flow plays a crucial role. For high values of this ratio, the oxide presents no cracks or voids, small grains and low surface roughness, as shown by the AFM measurement. These oxides have also a low BHF etching rate, and an medium residual stress after deposition. When this ratio is low, the oxide quality degrades. It becomes easily etched in BHF and the residual stress although being very low is not stable over time. Deposition rate is strongly dependent on the silane flow in the deposition chamber. It means high flow rate or low $[N_2O/SiH_4]$ ratio yields high deposition rate. RTA was then tested to decrease the residual stress and the etch rate. There is a strong correlation between etch rate decrease and stress release. At a temperature of 900 °C, the lowest value for the stress and etch rate is achieved for 15 s RTA. RTA releases the nitrogen and hydrogen atoms trapped in the oxide and makes the oxide grains grow in size. RTA will decrease the residual stress from 80 MPa after deposition to a value of 30 MPa. The etch rate is divided by a factor of two, from 3000 Å/min down to 1500 Å/min. Those values are achieved for oxide of good quality, stable over the time. Such oxides are good candidates for sacrificial layers in MEMS technology as the etching rate is low and reproducible and the residual stress not too high. Oxides grown at to low $\eta P/F$ ratio or at low $[N_2O/SiH_4]$ ratio which have a lower residual stress after deposition get a null or tensile stress after RTA but the BHF etch rate still very high, in the range of 10000 Å/min.. The inconvenient of such oxides is that they are not stable over time. The stress value is stabilized after a period of time corresponding to ten days. They will in a future study be tested as interface layer for wafer bonding or as sacrificial layer for CMP.

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