

Study of the stress-related vacancy generation in silicon due to silicon nitride films

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Recibido el 10 de agosto de 1998; aceptado el 9 de noviembre de 1998

The effect of stress on the point defect concentrations (vacancies and self-interstitials) in float zone silicon substrates due to silicon nitride films, deposited by the Low-Pressure Chemical Vapor Deposition (LPCVD) technique, has been studied by using thermal treatments at 1100°C in Argon ambient. The changes in point defects concentrations were monitored by the shrinkage of surface stacking faults (OSF's) in the silicon substrates, under silicon nitride films with different stress conditions. These conditions are controlled by varying the ratio of flow rates of silane and ammonia in the deposit reaction. During thermal treatments stacking faults shrink faster under silicon nitride films with the higher stress. These results suggest that a vacancy supersaturation and a self-interstitial undersaturation exist near the silicon surface region. It was observed that the deviation in point defects concentrations from equilibrium is directly related with the stress level in the silicon nitride films. An analysis of the possible cause of vacancy generation and its relation with the film stress conditions is presented.

Keywords: Silicon nitride; vacancies; stress

Se llevó a cabo un estudio del efecto que produce el esfuerzo en películas de nitruro de silicio obtenidas por depósito químico en fase vapor a baja presión (LPCVD), sobre las concentraciones de defectos puntuales intrínsecos (vacancias y auto-intersticiales) en sustratos de silicio de zona flotante, usando tratamientos térmicos en ambiente de argón a 1100°C. Los cambios de concentraciones de los defectos puntuales se estudian a través de las variaciones del tamaño de fallas de apilamiento superficiales (OSF's) en el sustrato de silicio, bajo diversas condiciones de esfuerzo. Estas condiciones se controlan variando la razón de flujos de los gases silano y amoníaco que intervienen en la reacción de depósito. Durante los tratamientos térmicos, las fallas de apilamiento se reducen de tamaño más rápidamente bajo las películas de nitruro de silicio con el esfuerzo mayor. Estos resultados sugieren que existe una supersaturación de vacancias y una bajosaturación de auto-intersticiales en la región del silicio cercana a la superficie. Se observa que las desviaciones de las concentraciones de sus valores de equilibrio, están directamente relacionadas con el nivel de esfuerzo en las películas de nitruro de silicio. Se llevó a cabo un análisis sobre la causa probable de generación de las vacancias y su relación con las condiciones de esfuerzo de las películas de nitruro de silicio.

Descriptores: Nitruro de silicio; vacancias; esfuerzo

PACS: 61.70.Bv; 61.70.Yq; 61.70.At

1. Introduction

Nowadays, meaningful progresses in the elaboration of good quality materials and integrated circuits (IC) have been fulfilled. Materials with very-low defect volume concentrations and practically free of dislocations can be found. Nevertheless, during a semiconductor devices fabrication process several steps of oxidation, diffusion, ion implantation, and so forth, can be used which results in high concentrations of self-interstitials and vacancies in the material volume. These defects, are then promoters of macroscopic defects such as stacking faults and dislocations, moreover, they affect in an important fashion the diffusivity of the used dopants [1]. Hence, independently of the scientific importance that the survey of the point defects as part of the comprehension of the semiconductor materials conformation has, the knowledge of its different interactions with the devices fabrication processes implies a huge technological interest. One of these

aspects consists of the knowledge of the effects that dielectric films exert on the point defects concentrations and diffusivities in the substrate. It is something known that thin films, either thermally grown or deposited, which are used in silicon integrated circuits, generally develop high-levels of intrinsic stress during the deposition processes [2]. Furthermore, there exists a stress of thermal sort during the film heating or cooling. This is as a result of the difference in the thermal expansion coefficients between the film and the substrate. In this way, it is interesting to observe if there is some effect of the total stress on the point defect concentrations, and on the diffusivity of the dopants in the region close to the substrate surface.

The silicon nitride films deposited by the Vapor Phase (CVD) generally exhibit a high-stress of the tensile type. Under this sort of films, Mizuo and Higuchi [3] observed an "abnormal" diffusion in some dopants. They found that the diffusion of phosphorus and boron in an inert atmosphere in float

zone silicon wafers, under a silicon nitride film, are reduced in comparison with the diffusion observed under a SiO_2 film covered with CVD nitride. The explication of this anomalous diffusion can be given in terms of the stress in the silicon nitride films [3].

Currently, it is well established that the phosphorus and boron diffuse predominantly by interactions with silicon interstitials, and the antimony diffuses by interactions with vacancies [4]. Moreover, the growing or the reduction in size of the stacking faults generated during the thermal oxidation, depends on the relative concentration of point defects in the silicon network. Since the stacking faults are of extrinsic kind, their decreasing rate is accelerated under a vacancy super-saturation [4]. On the other hand, when there is a super-saturation of silicon self-interstitials, the stacking faults grow or are reduced more slowly than normal, *i.e.*, of the intrinsic case. In this form, the stacking faults can be used in order to monitor the concentration deviation of point defects from their equilibrium values.

In the present work, the stress effect in the Low-Pressure Chemical Vapor Deposition (LPCVD) silicon nitride films on the silicon point defect concentrations has been studied, by observing during the thermal treatment at 1100°C in Argon, the change in size of the stacking faults previously generated in the silicon surface while the stress level is varied in the nitride films. The stress level and the stoichiometry in the silicon nitride films are modified by controlling the deposition conditions, as it is the ratio of the silane and ammonia gas flow that intervene in the reaction. On the basis of the stress relaxation mechanism that is proposed, some aspects are discussed, for instance, the apparent contradiction that is experimentally observed between the stress values measured in the films before and after the thermal treatment in Argon. The silicon nitride obtained by LPCVD will be denoted subsequently as SiN_x , indicating by means of this, that, in accordance with the deposition conditions, the films can change their stoichiometry.

2. Experimental procedure

Float zone silicon wafers of *p*-type, diameter 75 mm and $375\ \mu\text{m}$ thick were used, with orientation (100) and 250 Ohm-cm resistivity. In order to nucleate the stacking faults on the silicon wafers surface, previously to the nitride depositions, Si^+ was implanted with doses of $5 \times 10^{13}\ \text{cm}^{-2}$ and energy of 100 KeV using a Balzers MPB 202 implanter. All the samples were oxidized at 1100°C during 5 h in H_2O vapor to make the stacking faults grow up to $73\ \mu\text{m}$. The resulting oxide from this oxidation was completely eliminated with HF from the surface of some wafers, and in others the oxide was etched until reaching 45 nm thick. Silicon nitride films were deposited on the wafers by using a LPCVD reactor in a pressure of 380 Torr at 700°C . In order to deposit films with different stress levels, the ratio $R_F = f_{\text{SiH}_4}/f_{\text{NH}_3}$, where *f* denotes a flow, was varied from 0.35 to 6, maintaining the total flow to 250 sccm. Also, to observe the nitride

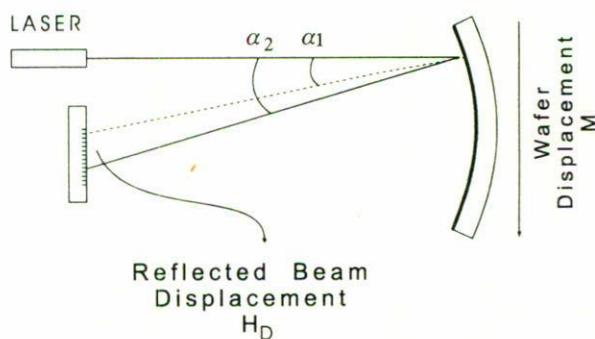


FIGURE 1. Experimental system for the measurements of the curvature radius. H_D is the laser beam displacement, M is the silicon sample displacement, $\alpha_2 - \alpha_1$ is the change of the beam angle reflected with the sample displacement.

film thickness effect on the stress, films ranging in thickness from 26 nm to 206 nm were deposited keeping constant $R_F = 1.67$. The film thickness and their refractive indices were measured with a Gaertner ellipsometer employing a He-Ne laser beam (wavelength of 632.8 nm), at an angle of 70° . In general, the refractive index values and thickness of the films were obtained as the average of at least 10 measurements in each sample. The total stress level in the silicon nitride films was determined by measuring the tension (change in curvature) produced in the silicon substrate by the deposited film. For the stress measurement, samples of $25\ \text{mm} \times 6\ \text{mm}$ were used. In this case, the stress in the film is given by [5]

$$\sigma_f = \frac{E_s}{6(1 - \nu_s)} \frac{t_s^2}{t_f} \frac{1}{R}, \quad (1)$$

where E_s is the Young's modulus, t_s is the thickness and ν_s the Poisson's ratio of the substrate, respectively; t_s is the film thickness and R the curvature radius that is produced in the silicon substrate. For the silicon, we have that $E_s = 1.689 \times 10^{11}\ \text{N/m}^2$ and $\nu_s = 0.064$ with t_s and R expressed in meters. The curvature radius was measured by an optical technique that uses a laser beam [5, 6]. The basic principle of the technique is very simple and is illustrated in Fig. 1. A laser beam is reflected on the sample surface and the displacement of the reflected beam is measured while the sample moves. The change in the displacement of the reflected beam is proportional to the change in the α angle between the incident beam and the wafer surface. For a perfectly flat wafer, the reflected beam position will be constant. If the wafer has a constant curvature, the α angle changes linearly with the relative movement of the wafer and the incident laser beam. Consequently, the beam displacement varies inversely to the curvature radius. A curve of the laser beam displacement (H_D) as a function of the wafer displacement (M), is therefore, a straight line with slope proportional to the inverse of the curvature radius, which is given by [5]

$$R = \frac{2DM}{H_D}, \quad (2)$$

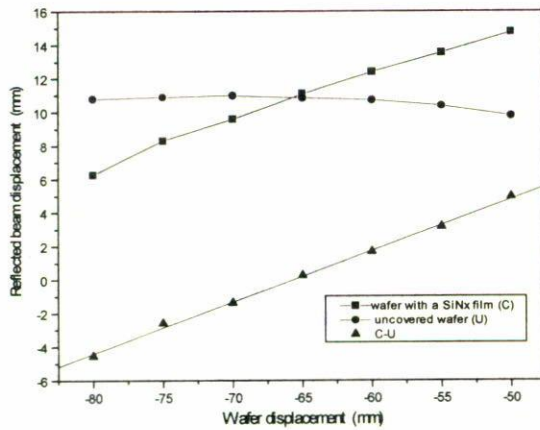


FIGURE 2. Differential method in order to measure the curvature radius of a film deposited on a substrate. The curve in the lower part indicates the point-to-point difference between the curves corresponding to the substrate with film and the uncovered substrate, respectively. This procedure eliminates the initial substrate curvature effects.

where D is the total distance traveled by the laser beam after being reflected on the sample surface. If the curvature radius is constant, the H_D vs. M graphic will be a straight line with slope p given by

$$p = \frac{2D}{R}. \quad (3)$$

The curvature radius R is considered by convention negative for samples with convex curvature (compression stress) and positive for samples with concave curvature (tensile stress). With the described measurement system the sample displacements (M) were measured with precision of ± 0.1 mm and the ones of the reflected beam (H_D) with a precision of ± 0.05 mm. In these measurements a Melles Griot model 05 LHR 991 He-Ne laser was utilized with a maximum power of 30 mW and wavelength of 632.8 nm. It must be pointed out that in order to eliminate all the possible curvature effects in the original substrate, it was necessary to use a differential technique in the calculus of the film curvature radius. For this, the point-to-point difference was fulfilled between the curves H_D - M of the silicon sample with the film deposited and that one corresponding to the uncovered sample, as indicated in Fig. 2. The calculus of R was performed for the resulting straight line.

In order to observe the stress effect of the nitride films on the point defect concentrations, through the size changes in the stacking faults, thermal treatments were applied to the samples at 1100°C in ultra-high purity Argon ambient, for periods of time ranging from 1 to 8.5 h. After the thermal treatments, the films stress was again measured for the purpose of comparing the stress before and after the thermal treatments. Finally, after the complete removal of the nitride films and the revealing of the stacking faults with Wright solution [7], the stacking fault size was measured by using a Wild Heerbrugg MPS51 optical microscope.

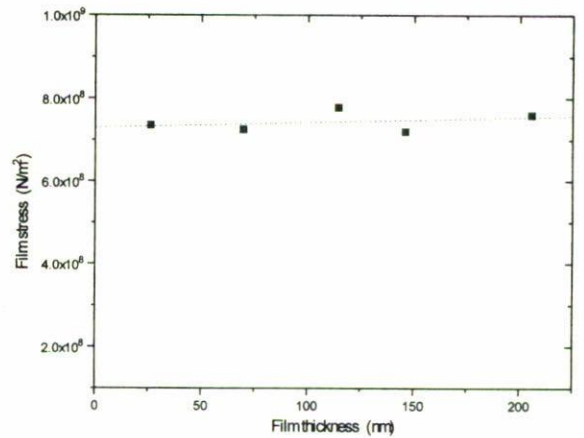


FIGURE 3. Graphic of the stress vs. thickness for LPCVD silicon nitride films.

3. Results

For the deposited silicon nitride films, a good uniformity was obtained for both the thickness and the refractive indices. The measured stress in the films at ambient temperature is tensile, which is in agreement with previous measurements [8]. In Fig. 3 the stress is shown as a function of the silicon nitride film thickness deposited with $R_F = 1.67$, for times of 10, 30, 50, 70, and 90 min. The stress is practically independent of the silicon nitride film thickness, within the experimental error, which agrees with the results of other researchers [9]. This fact is explained by observing that a thicker film will result in a curvature radius correspondingly minor. Thus, if the biaxial elastic constant $E_s/(1 - \nu_s)$ does not change, the intrinsic component of the stress is maintained for the R_F used.

On the other hand, the silicon nitride film stress is reduced whereas the R_F value is increased, as shown in Fig. 4. In this case the film thickness increased with the R_F value, however, as seen above, the film stress does not depend on the film thickness. In Fig. 5, the refractive indices of the deposited films as a function of the R_F ratio have been graphed. The stoichiometric silicon nitride film, *i.e.*, that with a refractive index of about 2.0, was obtained with a small R_F value, while the films with higher refractive indices, which indicates a higher silicon content [10], were obtained with higher R_F values.

In Fig. 6, the stacking faults behavior during the thermal treatments for the silicon nitride films with different thickness but the same chemical composition are shown. The shrinkage rate of the stacking faults under the nitride films, is practically the same for the different used thickness. On the other hand, in Fig. 7, the behavior exhibited by the stacking faults under the nitride films deposited for different R_F values is shown. In this case there exists a prevalent tendency to higher shrinkage rates of the stacking faults for lower R_F values. Figure 7 also shows the stacking faults behavior during the thermal treatment when using a composed $\text{SiN}_x/\text{SiO}_2$ film. The stack-

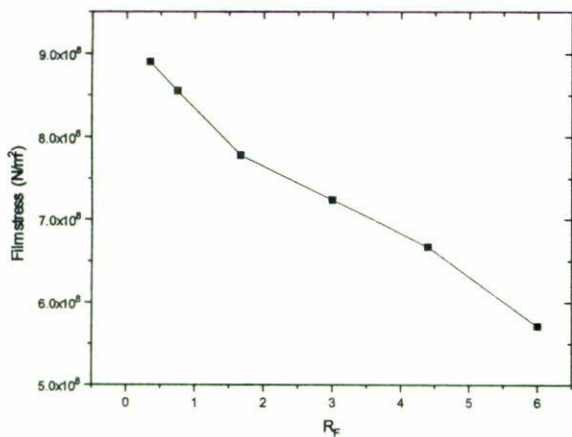


FIGURE 4. Stress in LPCVD silicon nitride films as a function of the flow ratio $R_F = f_{SiH_4}/f_{NH_3}$ of the reaction gases. As mentioned in the text, the film thickness increases with the R_F value.

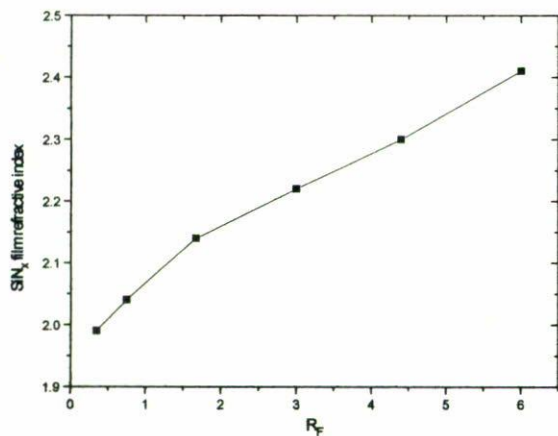


FIGURE 5. Refractive index for the LPCVD silicon nitride films, as a function of the flow ratio $R_F = f_{SiH_4}/f_{NH_3}$ of the reaction gases.

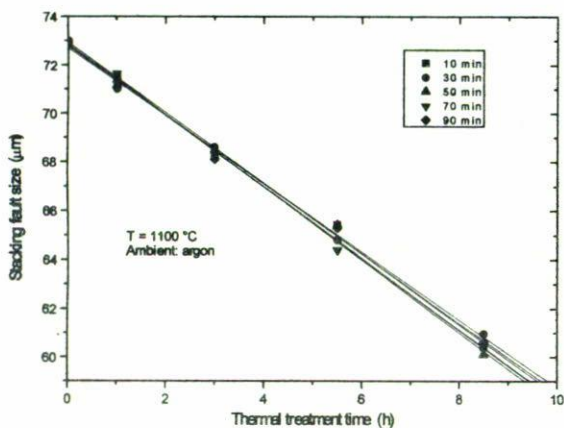


FIGURE 6. Average stacking faults length as a function of time in the thermal treatment at 1100°C in Argon ambient, for different deposit times of the LPCVD silicon nitride films.

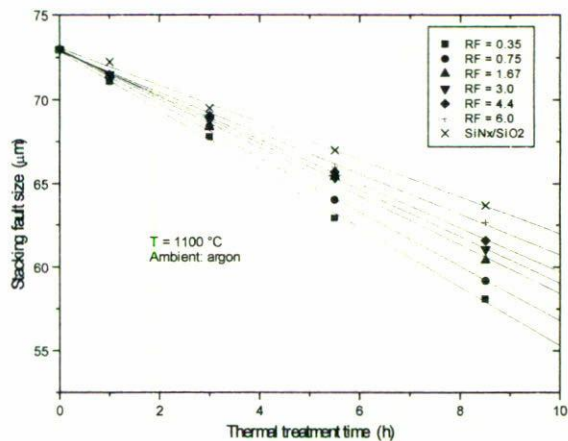


FIGURE 7. Stacking faults length as a function of time in the treatment at 1100°C in Argon ambient, for different $R_F = f_{SiH_4}/f_{NH_3}$ values used in the LPCVD silicon nitride films deposit. The upper curve presents the case for the compound SiN_x/SiO₂ film, being the SiO₂ 45 nm thick and $R_F = 0.75$ for the SiN_x film.

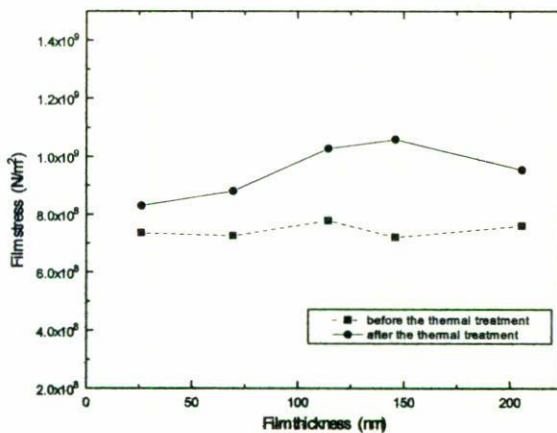


FIGURE 8. Stress in the LPCVD silicon nitride films as a function of the thickness before and after the thermal treatments at 1100°C in Argon ambient.

ing faults are reduced in size more slowly for the nitride films with a low-stress level (big R_F values) and for the composed SiN_x/SiO₂ film, than for the nitride films with a high-stress level (small R_F values). It should be pointed out that the stacking faults density observed in all samples maintained approximately constant during the thermal treatments.

Figure 8 shows the stress values in the silicon nitride films with different thickness, before and after the thermal treatments in Argon. An interesting aspect that is further discussed below is the fact that the stress in the films is in general increased after the thermal treatments.

4. Discussion of results

By observing the behavior shown by the stacking faults under thermal treatments at 1100°C in Argon ambient, and ac-

According to the characteristic of these defects of reducing their size in presence of a vacancy excess [4], it is clear that the presence of stress in the nitride film results in a vacancy supersaturation and a consequent self-interstitial undersaturation near the silicon surface region. The vacancy supersaturation level depends on the magnitude of the film stress and is manifested by the amount of shrinkage rate of the stacking faults.

This analysis suggests that the silicon nitride films deposited in the high-stress conditions, that is, for small R_F values (see Fig. 7), correspond to a nonequilibrium situation for the point defect concentrations. In other words, the stoichiometric silicon nitride films deposited by using small R_F values (low-silicon content), cause that stacking faults, in the high-temperature treatments, to shrink faster than in the corresponding case to the films obtained when using higher values for R_F (high-silicon content). Therefore, under a silicon nitride film in the silicon region near the interface Si/SiN_x, there is a vacancy supersaturation and a corresponding self-interstitial undersaturation, whose deviations of equilibrium increase by increasing the stress-level in the film.

These considerations let us think on a probable cause for the vacancy generation under the shown experimental conditions. When a silicon nitride film, (it is also observed in SiO₂ films) either deposited or thermally grown on a silicon substrate, is subjected to a thermal treatment in an inert ambient to temperatures above the deposition or growing temperature, it tends in general to reduce its stress through a relaxation process. Since under these conditions, as seen above, a vacancy excess and a corresponding self-interstitial deficit are generated, it can be supposed that the vacancies in the substrate are consequence of the interstitial transport to the nitride film, which reduces its stress by absorbing silicon atoms in its structure. It is noted that this cause of vacancy generation is compatible with the fact that silicon nitride films deposited by using high R_F values, are the ones that show the lowest stress values.

In regard to this point, it is interesting to observe the effect of introducing a thermal-grown SiO₂ film between the silicon substrate and the silicon nitride film. In Fig. 7, the fault behavior corresponding to this case is shown and it is observed that the presence of SiO₂ decreases the shrinkage rate of the stacking faults. In agreement with the above mentioned, this means that a SiO₂ film decreases the stress in the silicon substrate, existing a lower vacancy concentration than in the case of using only a silicon nitride film. The explanation to this result is that the SiO₂ has a compressive-kind stress, thus compensating the nitride tensile-stress. Furthermore, when the temperature increases, the SiO₂ behaves as a viscous flow that gives rise to a relaxation mechanism of the SiN_x/SiO₂ system. Once the stress equilibrium state in the system has been reached, by means of viscous flow of oxide, dislocation generation, etc., it will tend to establish a relationship between the resulting stress and the vacancy concentration generated in the silicon substrate. In like manner,

the silicon transport to the nitride film, will result in a gradual (additional) reduction of the total stress during the high temperature thermal treatment.

One aspect of interest related to the vacancy generation under the stress conditions is constituted by the possible sources of these defects. In this case, there are two possibilities. The first possibility is the silicon region near the SiN_x/Si interface and the second is the interface itself. Regarding the first possibility, it must be observed that the tensile stress present in the nitride film will give rise to a compressive stress in the substrate, which can reduce the enthalpy of formation of vacancies and increase that corresponding to self-interstitials [11]. Nevertheless, in accordance with the reported in the literature, the stress produced in the substrate by a uniform silicon nitride film is practically negligible [12]. This was verified in this work by means of a simulation by employing a bi-dimensional model of the SUPREM IV [13] applied to a continuous film of silicon nitride, *i.e.*, with no steps. According to the Heemyong model [11], which is based on the changes of point defect concentrations and diffusivities in the presence of a stress field in silicon, it is possible to explain phenomena like the phosphorus retarded diffusion in a compressive stress field. However, in order to be able to explain this phenomenon it is necessary a stress level in the silicon of the order of 10⁹ N/m², like the ones generated in the nitride film edges, situation that is not applicable in this work. Thus, it is not probable that formation energies of point defects be altered and therefore, their equilibrium concentrations. Moreover, it should be observed that the thickness of the silicon nitride films does not influence their stress (see Fig. 3), but it does affect the substrate stress level, which is not noticed in the stacking faults behavior.

The other possibility is the SiN_x/Si interface acting as a source or a drain of point defects whether injecting vacancies to the substrate or absorbing self-interstitials from the same. Both conditions are compatible with the observed experimental results, that is, with an excess of vacancies that cause a stacking fault shrinkage greater than the one corresponding to the intrinsic case. If the interface absorbs interstitials, which can happen by means of some kind of reaction at the silicon surface, then it will result in a vacancy supersaturation by Frenkel defect (interstitial-vacancy pair) generation in the silicon volume in response to the interstitial deficit. However, the Frenkel defect generation in the silicon volume requires more energy than interface vacancy generation, which will result in a slower generation process in the silicon volume. This same argument was used by Fahey *et al.* [14], to determine whether there is a vacancy injection or an interstitial deficit during the silicon thermal nitridation. On the other hand, the interface acting as a source to inject vacancies into the silicon volume, it will result in an interstitial deficit through the natural reaction of recombination between interstitials and vacancies. From this point of view, it is more probable the vacancy injection of the SiN_x/Si interface in order to explain in a consistent way a vacancy supersaturation in the silicon volume.

In relation to the probable cause of vacancy generation that explains the stacking fault behavior during the thermal treatments, it has been pointed out that the nitride film absorbs silicon atoms from the substrate as a consequence of the natural tendency of the film to reduce its stress by means of a high-temperature relaxation process. Then, after the thermal treatments it would be expected to obtain lower film stress values than before the treatments. Nevertheless, this effect was not observed in our experiments. The stress values measured for the silicon nitride films increased after the thermal treatments, as it was shown in Fig. 8 for the nitride films with different thickness values. In order to adequately explain this result, it is necessary to analyze the behavior of the stress in relation to the possible structure changes in the silicon nitride films, when these are subjected to temperatures above the deposition one.

In the ideal elastic regime, the total stress of the film measured at some given temperature is represented as [5]

$$\sigma_f = \sigma_i + \sigma_{th}, \quad (4)$$

where the intrinsic stress, σ_i , is related to the internal structure and composition and is determined by the deposition conditions, and σ_{th} is a thermal component which results from the difference in the thermal expansion coefficients of the film and substrate. The thermal component is given by the expression [5]

$$\sigma_{th} = \frac{E_f}{(1 - \nu_f)} (\alpha_s - \alpha_f) (T - T_d), \quad (5)$$

where α_s and α_f are the thermal expansion coefficients of the substrate and film, respectively; E_f is the Young's modulus of the film, ν_f is the Poisson's ratio of the film, and T_d and T are the deposition and the stress measurement temperature, respectively.

When the temperature of a deposited SiN_x film is increased, its stress will also tend to increase. This is due to the thermal component of the stress which grows with the temperature since $\alpha_s > \alpha_{\text{SiN}_x}$. If the temperature increase does not generate any structural change in the film, then the stress behavior can be explained, and even predicted, through the simple elasticity laws such as Eqs. (4) and (5), knowing the elastic constants of the material. Nevertheless, temperature increases greater than the film deposition temperature can induce irreversible changes in the film structure which are associated to the reduction of hydrogen in the film [15]. The LPCVD silicon nitride always contains hydrogen, usually at atomic concentrations around 7% bonded to silicon or nitrogen as Si-H or N-H, respectively. The loss of hydrogen during high temperature treatments, has been taken as a direct evidence for chemical and compositional changes (structural reordering and densification) in the film [15].

Thus, the positive hysteresis effect observed in the film stress after the 1100°C treatment can be explained, according to expression (5), by assuming a decrease in the silicon

nitride thermal expansion coefficient and a saturation of the biaxial constant as a consequence of loss hydrogen. In fact, Jansen *et al.* [16] found a decrease of the silicon oxide thermal coefficient in oxide films and a saturation of its biaxial constant as a result of reducing the hydrogen content in the oxide films. It should be pointed out that effects of positive hysteresis in stress have also been observed in Plasma Enhanced Chemical Vapor Deposition silicon nitride films at temperatures above the deposition temperature, and explained on the basis of loss hydrogen during the thermal treatment [17].

A possible way of avoiding or reducing the hysteresis effects in the stress of a film subjected to a thermal treatment, it would consist in depositing or growing the film to the same temperature at which the treatment is effected. In this form, no additional phenomenon could be activated (for instance a chemical one) that altered the stress behavior during the thermal treatment. This effect can be directly verified from the experimental results of Ahn *et al.*, on the phenomenon of antimony increased diffusion in silicon under thermal silicon nitride films [18]. Ahn *et al.* observed that the antimony diffusivity decreases with the time of treatment in Argon at 1100°C, which was the same temperature of the film growing. Taking into account that antimony diffuses mainly by a vacancy mechanism and that its diffusivity depends directly on the vacancy supersaturation produced by the stress in the film, this fact indicates that there exists a stress reduction with the time of treatment due to the interstitial migration from the substrate. In this case, there are no alterations in the film stress due to composition changes, that is why it is possible to observe the net stress relaxation effect.

5. Conclusions

From the stacking fault shrinkage observation on the silicon surface during treatments at 1100°C in Argon ambient, it was found that there is a vacancy supersaturation and a self-interstitial deficit under the silicon nitride films, due to the presence of stress in these ones. The equilibrium deviation of the point defect concentrations is related with the stress-level in the silicon nitride film. The increase observed in the stress of the films after the thermal treatments is explained on the basis of the structural reordering and densifying that the silicon nitride films suffer at temperatures above the one of the deposit.

Acknowledgments

The authors thank to M.S. Carlos Zúñiga Islas and Chem. Israel Fuentes Tapia for the help provided regarding the ion implantation and silicon nitride film deposits by LPCVD processes, in the facilities of the Microelectronics Laboratory of the Instituto Nacional de Astrofísica, Óptica y Electrónica. The work hereby was achieved with support of CONACyT.

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