Strain profile of (001) silicon implanted with nitrogen by plasma immersion

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(Received 21 December 2006; accepted 22 March 2007; published online 24 May 2007)

In this work, we investigate the strain and defect state of silicon implanted with nitrogen by plasma immersion ion implantation, with doses between 4.5×10^{16} and $8.7 \times 10^{16} \text{ cm}^{-2}$. For this purpose, we have used Auger electron spectroscopy, x-ray reflectivity, and high-resolution x-ray diffraction. Auger spectra showed that nitrogen concentration profiles broaden and shift deeper into the substrate as the dose increases. High oxygen concentration in the first 20 nm suggested the presence of an amorphous oxide layer at the sample surface, which was confirmed by x-ray reflectivity measurements. Reciprocal space maps revealed a tensile strain perpendicular to the surface, while no in-plane strain was detected. Since no significant diffuse scattering was found, randomly distributed point defects must be predominant in the strained region compared to large displacement field defects such as clusters and dislocations. $\omega/2\theta$ scans around (004) Bragg reflection were fitted using dynamical theory of x-ray diffraction. The strain profiles obtained from the best fits correlated well with nitrogen concentration depth profiles, signaling interstitial nitrogen as the main source of strain. © 2007 American Institute of Physics. [DOI: 10.1063/1.2734957]

I. INTRODUCTION

Nitrogen doping is widely employed to modify silicon properties in electronic devices. In MOSFET transistors, a nitrogen rich region at the Si/SiO₂ interface hinders boron diffusion from polysilicon gates into the channel and improves hot-electron resistance.¹ Also, nitrogen implantation in silicon and subsequent annealing is used to form buried silicon on insulator (SOI) structures.² Moreover, it was demonstrated that small concentrations of nitrogen improve float zone silicon mechanical properties through dislocation locking mechanisms.³

Doping can be achieved through plasma implantation techniques, which allow the creation of peak shaped concentration profiles and operate at relatively low temperatures, thus avoiding undesirable diffusion phenomena.⁴ Among the implantation techniques, plasma immersion ion implantation (PIII) or plasma doping, as it is also known, has recently attracted much interest.⁵ PIII is a three dimensional implantation process, which circumvents the line-of-sight restriction of ion beam implantation. Plasma implantation is simpler and cheaper than beam implantation, and has already found applications in the semiconductor industry, such as boron and phosphorous doping of silicon wafers to form shallow junctions.^{6,7} PIII has also been employed to form amorphous silicon nitride layers on silicon.⁸

High-resolution x-ray diffraction (HRXRD) is a nonde-

structive technique that can be used to characterize the structural properties of implanted layers. HRXRD measurements are strongly affected by the strain distribution created both by ions and damage in the implanted region. Reciprocal space maps, or area scans, contain a wealth of information on the lattice strain state and other defects such as lattice bending, clusters, and dislocations. The strain distribution can be obtained by fitting an x-ray line scan with the spectrum calculated using diffraction theory. This method has been widely employed in the characterization of ion beam implanted samples.^{9–13} Lattice disorder lowers the diffracted intensity, and it is usually accounted for by introducing a depth dependent Debye-Waller (DW) factor. Sometimes, the solution is not unique and two or more combinations of a DW factor and strain profiles produce equally good fits to the measured x-ray spectra. Additional information from x-ray standing wave and transmission electron microscopy can be used to eliminate this ambiguity. Furthermore, in weakly damaged silicon implanted samples, a linear dependence has been found between defect and strain depth profiles,¹⁴ and simulations are greatly simplified by considering a linear relation between the mean atomic displacement and the strain.^{10,11} A comparison with ion and defect distributions calculated using TRIM^{11,12,15} and SUPREMI¹⁶ simulation packages or measured by Rutherford backscattering,¹⁴ Auger electron spectroscopy (AES),¹⁷ or secondary ion mass spectroscopy^{11,14} is commonly made as a test of the results and to remove any remaining ambiguity.

In this work, we investigate the strain and defect state of

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silicon wafers implanted with nitrogen by plasma immersion ion implantation. For this purpose, we produced two identical sets of samples varying the nitrogen pressure inside the PIII chamber to obtain different implantation doses. One set was analyzed by AES to find the elemental composition depth profiles. The AES nitrogen profiles allowed the determination of the retained doses. The second sample set, with counterparts corresponding to the ones with AES data, was investigated by high-resolution x-ray diffraction and grazing incidence x-ray reflectivity. Reciprocal space maps around symmetric (004) and asymmetric (224) Si lattice points were measured to determine major strain and defect features. The strain profile of the implanted samples was determined by a best fit procedure, which compares the spectra calculated by the dynamical theory of x-ray diffraction with the $\omega/2\theta$ scans measured around the (004) Si Bragg peak. The amorphous layer with high oxygen concentration at the sample surface, observed in the AES depth profiles and confirmed by x-ray reflectivity measurements, was considered in the fitting process as a zero strain layer with a very high Debye-Waller factor.

II. IMPLANTATION

The experimental apparatus for performing the PIII treatment of the polished surface of Si wafers is described in detail elsewhere.¹⁸ It consists of a 30 l stainless steel SS304 vacuum chamber with a diffusion pumping system, a nitrogen gas injection system, a plasma source, and a high voltage pulser. For the plasma source, we used a glow discharge with controlled plasma potential, which provides plasma densities in the order of 10^9 – 10^{10} cm⁻³ and temperatures around 5 eV. The high voltage pulser is a commercial hard tube type unit RUP-4 that can deliver negative pulses of up to 30 kV, a maximum of 1.1 kHz frequency, and pulse duration from 1 μ s to 1 ms.

For nitrogen implantation, we used 2.5×2.5 cm² pieces of (100) Si *n*-type wafers with a resistivity of 1 Ω cm. The wafers were RCA cleaned in order to remove organic residue and native oxide, and were loaded in the PIII system after 24–48 h. This procedure allowed the formation of a thin oxide layer that shall act as a barrier for the retention of the implanted nitrogen.¹⁸ The Si wafers were fixed on a SS304 block support of $2.5 \times 2.5 \times 8$ cm³ with two flat SS304 sheets, which allow the exposure of the polished side of the wafers to the PIII ion flux.

Two identical sets of samples were prepared, one for AES and the other for x-ray investigation. Nitrogen pressures during implantation varied between 3×10^{-4} and 7×10^{-4} mbar, which lead to plasma densities ranging from 2×10^9 to 6×10^9 cm⁻³. The implantation was carried out with applied voltage pulses of 10 kV during 50 μ s at a frequency of 300 Hz. Implantation time was fixed to 30 min in all cases.

III. CHARACTERIZATION

A. Auger electron spectroscopy

Elemental composition of the nitrogen implanted Si samples was determined as a function of depth using Auger

TABLE I. Nitrogen retained dose and concentration profile peak position of implanted Si samples, determined from Auger electron spectroscopy. Maximum and integral strain values obtained from the best fits to the measured x-ray diffraction spectra are also shown.

Sample	Dose (10^{16} cm ⁻²)	N peak position (nm) ^a	Maximum strain (10^{-3})	Integral strain (nm ⁻¹)
R37	4.5	14.9	6.7	0.118
R02	5.9	18.6	3.6	0.072
R22	7.5	19.5	2.8	0.071
R13	8.0	31.2	2.3	0.070
R31	8.7	29.3	3.8	0.085

^aMeasured from the substrate surface

electron spectroscopy (AES) combined with Ar sputtering. For this purpose, we used a Fisons Instruments Surface Science system model Microlab 310-F. The retained dose was computed from the AES nitrogen profile using the method described in Ref. 19, and was found to vary between 4.5×10^{16} and 8.7×10^{16} cm⁻², as displayed in Table I.

Figure 1 shows the AES depth profile for the samples with minimum and maximum doses. Besides the implanted nitrogen atoms, impurities such as oxygen and carbon are also present in the silicon matrix. There is a high oxygen concentration at the sample surface, surpassing that of silicon in the first 10–20 nm. Oxygen comes from both the native

FIG. 1. Elemental composition depth profiles of two nitrogen implanted silicon samples with different doses, as determined by Auger electron spectroscopy. Concentrations of oxygen and carbon impurities are also shown. Solid lines are guides to the eyes. The insets detail the implanted nitrogen depth profiles and their fits, which were used as starting strain distributions.

FIG. 2. (a) X-ray reflectivity curve measured for sample R31. Calculated spectra for two sample models: (b) with one SiO₂ layer on top of the Si substrate, and (c) adding a ten layers linear transition region between the SiO₂ cap layer and the Si substrate. Spectra were shifted in the vertical for better visualization.

oxide layer and the residual gases in the implantation chamber. High oxygen concentrations suggest the presence of an amorphous layer at the sample surface.

The insets in Fig. 1 detail the implanted nitrogen concentration profiles, which are peak shaped and asymmetric. Note that the maximum of the nitrogen concentration shifts to deeper regions when the dose rises, as shown in Table I. Also, the nitrogen concentration profile widens and becomes more symmetric, characteristic of a diffusion process. At implantation temperatures below 300 °C, as used here, thermal diffusion and transient-enhanced diffusion are minor effects.^{20,21} Radiation-enhanced diffusion (RED) could be the reason for the observed behavior. RED relies on the defects created during implantation, which are mobile and able to enhance diffusion for a short time, before they are annealed or trapped. This kind of diffusion has already been detected for several dopants implanted in silicon.^{21,22} Besides, RED is known to increase with the implant dose,²¹ an effect which could explain the evolution of the nitrogen profiles.

B. X-ray reflectivity measurements

Grazing incidence x-ray reflectivity measurements were made to get more insight into the high oxygen concentration layer at the sample surfaces. The measurements were performed using a high-resolution x-ray diffractometer (Philips X'Pert MRD) with the Cu x-ray tube in line focus. The optics for the incident beam consists of a Soller slit, a 1/32° divergence slit, and a Ni-foil attenuator. The reflected beam passes through a parallel beam collimator, a 0.1 mm anticatter slit, and a flat crystal graphite monochromator before reaching the gas proportional detector. Prior to the measurements, the sample height and the goniometer zero points ($2\theta = \omega = 0$) were precisely adjusted. The reflectivity spectra were recorded with a $\omega/2\theta$ scan between $\omega = 0.05^\circ$ and $\omega = 2^\circ$. An attenuator has been used for angles lower than a predefined angle to avoid the detector damage due to a very intense incident radiation.

The x-ray reflectivity curves were measured for all samples. Curve (a) in Fig. 2 shows the reflectivity spectrum measured for sample R31. Weak interference fringes can be observed, slowly damping for higher ω . All measured spectra

FIG. 3. Reciprocal space maps measured around the (004) and (224) Si lattice points of a nitrogen implanted sample (R37), plotted in reciprocal space coordinates Q_x in the [110] direction, parallel to the sample plane, and Q_z along the [001] direction, perpendicular to the sample surface. The isointensity contour lines are at 4, 20, 200, 4000, and 40 000 cps.

exhibited similar features, which indicate the presence of a graded transition region between the oxide layer and the silicon substrate. The measured spectra were fitted with the ones calculated with two sample models. The first sample model considered the presence of a SiO₂ layer on top of the Si substrate. Curve (b) in Fig. 2 shows the spectrum that best reproduced the measurement, obtained with a SiO₂ layer of 22 nm and a SiO₂-Si interface roughness of 3 nm. However, in this model the oscillations vanish for higher ω due to the relatively high interface roughness. A second more detailed sample model considered a ten layer linear transition region between the Si substrate and the SiO₂ cap layer. The best fit included an 8 nm transition region and a 17 nm SiO₂ cap layer with a roughness of 6 Å [curve (c) in Fig. 2]. This sample model yielded spectra which closely resembled the measurements, proving the oxide nature of the surface layer with a graded smooth interface toward the Si substrate, as already suggested by Auger measurements.

C. High-resolution x-ray diffraction analysis

In order to determine the strain state of the implanted samples, high-resolution x-ray diffraction measurements were performed in a Philips X'Pert MRD diffractometer. A four-bounce Ge(220) Bartels monochromator was placed at the x-ray Cu tube point-focus output, providing a highly collimated monochromatic beam with an axial divergence $\Delta\omega = 12$ arc sec and a wavelength dispersion $\Delta\lambda/\lambda \sim 10^{-4}$.

1. Reciprocal space mapping

Reciprocal space maps (RSM) around (004) and (224) Si lattice points were measured through repeated $\omega/2\theta$ scans with different ω offsets, using a three-bounce crystal Ge(220) channel-cut analyzer in front of the gas-filled proportional detector, which reduced its acceptance angle to 12 arc sec.

Figure 3 shows typical (004) and (224) RSM of the implanted Si crystals. Both maps show intensity distributions that peak at the (004) and (224) Si reciprocal lattice points and rapidly decay in all but the $-Q_z$ direction. Observe that the intensity distributions are vertically shaped, extending

FIG. 4. Reciprocal space map measured around the (004) Si Bragg peak for an implanted sample and for a bare Si substrate. The isointensity contour lines are at 4, 20, 200, 4000, and 40 000 cps.

over $0.007 \text{ \AA}^{-1}$ in the Q_z direction and only $0.0003 \text{ \AA}^{-1}$ in the Q_x direction. The elongation of the intensity distribution is generated by the strain in the implanted region. The fact that this elongation occurs toward smaller Q_z values indicates that the Si lattice in the implanted region is under tensile perpendicular strain, i.e., the lattice constant is expanded in the direction normal to the surface. A tensile strained implanted layer suggests that nitrogen is occupying interstitial sites in the silicon lattice. This behavior is typical from implanted nitrogen and is the cause of nitrogen generally showing low electrical activity.^{23,24}

The vertical intensity distribution around the (004) reflection, shown in the left panel of Fig. 3, also indicates that there is almost no mosaicity in our implanted samples.²⁵ The same distribution is observed around the (224) asymmetric reflection, showing that the implanted region preserves the in-plane lattice parameter of the substrate.²⁶ Hence, a study of the strain state of the implanted samples may be carried out considering only the strain perpendicular to the sample surface, as will be described in Sec. III C 2.

Figure 4 shows RSMs around the (004) reflection for both an implanted sample and a bare silicon substrate. Diagonal and vertical streaks in bare silicon RSM correspond to analyzer and sample crystal truncation rods (CTRs), respectively. CTRs arise from the sudden end of a perfect crystal at its surface. Though less resolved, CTRs are also present in the implanted sample maps. However, the sample CTR is much weaker after implantation, which is due to the presence of a strained region at the surface, i.e., the perfect crystal beneath the strained region has no longer a sudden end.

(004) RSMs of implanted samples show no significant diffuse scattering as compared to nonimplanted Si substrate. Besides, increased surface sensitive measurements around (113) peak at grazing incidence $\omega=2.84^\circ$ showed no additional diffuse scattering from implanted samples compared with bare Si. Diffuse scattering around reciprocal lattice points may arise from defect clusters and dislocations. Although synchrotron radiation is ideal for observing diffuse scattering, it has also been detected using conventional x-ray sources, but for much more energetic beam implanted ions and frequently after some annealing treatment.^{27–30} Zymierska *et al.*²⁷ observed diffuse radiation in 3 MeV N implanted Si, coming from the displacement fields of Si_xN_y clusters. They ascribed the formation of Si_xN_y clusters to local heating above 1400°C , which is the temperature needed for this phase formation.²⁷ The absence of any measurable diffuse

FIG. 5. $\omega/2\theta$ scans around the (004) Si Bragg diffraction peak for samples with an increasing dose of implanted nitrogen. The solid lines are the best fits to the measured spectra, calculated using the dynamical theory of x-ray diffraction. The curves are shifted in the intensity axis for better visualization.

scattering in the RSMs of our samples indicates that randomly distributed point defects in the form of implanted ions and dislodged host atoms are predominant.³⁰ Extended defects like dislocations are much less likely, and we discard the presence of Si_xN_y clusters. None of these arguments conflict with the presence of the mentioned amorphous oxide layer, because the latter merely contributes to the background.

2. Strain determination

To find the perpendicular strain distribution in the implanted region, $\omega/2\theta$ scans around the (004) Si Bragg reflection were measured. This time, a $1/16^\circ$ slit was placed in front of the detector to improve the signal to noise ratio, still maintaining a reasonable resolution.

Figure 5 shows measured $\omega/2\theta$ scans for five samples with different nitrogen implanted doses. All spectra exhibit an intense narrow peak at the (004) Si Bragg reflection, with a broad shoulder at the lower angle side. As follows from the RSM discussion, the shoulders arise from the strain in the implanted region, and their position at the lower angle side of the main peak indicates a higher perpendicular lattice parameter, i.e., a tensile perpendicular strain in all cases.

Finding the strain profile in the implanted region involves a recursively fitting process that proposes a strain profile, calculates the corresponding x-ray spectrum, and com-

pares calculated and measured spectra through a logarithmic intensity χ^2 test. The fits to the AES nitrogen concentration profiles, plotted in the insets of Fig. 1 for the samples with maximum and minimum doses, were taken as starting strain distributions. Prior to the calculation step, the strain profile in the implanted region is divided in 20 discrete layers of 50 Å each, parallel to the surface. A constant strain is assumed within each discrete layer. Subsequently, the x-ray spectrum is calculated using a program based on the Takagi-Taupin formalism of diffraction.³¹ A logarithmic intensity χ^2 minimization procedure was used to obtain the strain profile that best reproduces the measured spectrum.

The model includes a Debye-Waller factor M , which modifies the structure factor by the exponential factor e^{-M} as follows:

$$M = \frac{8\pi^2 \sin^2 \theta_{\text{Bragg}} \langle u^2 \rangle}{\lambda^2},$$

where u is the static atomic displacement component perpendicular to the lattice plane. The DW factor accounts for the disorder in the implanted region and lowers the intensity of the Bragg reflection.¹⁰ To account for the amorphous oxide region, we have considered an unstrained layer with a very high DW factor on top of the strain profile. We assume this amorphous layer to extend from the sample surface up to the point where the oxygen Auger profile reaches 25 at. %, which gives us thicknesses between 12 and 26 nm, in agreement with x-ray reflectivity results.

The best fits are shown as solid lines in Fig. 5 for our five samples implanted with increasing doses of nitrogen. Figure 6 shows the strain profiles corresponding to the best fits, together with Auger nitrogen and oxygen concentration profiles, for comparison. There is a good agreement between strain and nitrogen concentration distributions, except for the surface region of high oxygen concentration, which was considered amorphous and hence not strained. These similarities confirm the assumption that interstitial nitrogen is the main source of strain in our samples.

In the strained region, a model in which the mean atomic displacement is proportional to the strain was considered. As already mentioned, this is a good approximation in low damage cases and has been successfully used before.^{10,11,14} However, calculations lead to very small, nonsignificant DW factors. This means that below the amorphous layer the implanted region is rather ordered, at least where the DW factor is concerned. This result correlates well with the absence of significant diffuse scattering of the RSM's, indicating the lack of extended field defects.

Table I shows maximum and integral strain values for all our samples implanted with different doses. Except for sample R31, both maximum and integral strain values decrease for increasing doses. It is important to note here that our previously published data,¹⁷ where maximum and integral strain values of 2×10^{-3} and 0.033 nm^{-1} , respectively, were found for a PIII nitrogen implanted dose of $15 \times 10^{16} \text{ cm}^{-2}$, follows this trend too. There are two mechanisms that could be responsible for the observed strain decrease for higher doses. The first is a self-annealing effect, i.e., a reduction of the strain due to damage during implan-

FIG. 6. Strain profiles of nitrogen implanted silicon samples corresponding to the best fits shown in Fig. 5. An unstrained highly disordered surface layer accounts for the amorphous region. For comparison, nitrogen (squares) and oxygen (circles) Auger depth profiles are also plotted.

tation, already observed in Ge implanted silicon samples.¹⁰ The second possible mechanism is that a growing fraction of the implanted nitrogen begins to occupy substitutional sites as the dose increases. Nitrogen atomic radius is smaller than silicon radius. Therefore, the presence of some nitrogen in substitutional sites will partially compensate tensile strain generated by nitrogen in interstitial sites, justifying the observed behavior.

IV. CONCLUSIONS

Auger electron spectroscopy and high-resolution x-ray diffraction were used for the compositional and structural characterization of PIII nitrogen implanted silicon crystals. AES nitrogen concentration profiles were peak shaped, with

maxima between 15 and 29 nm depth from the surface. The implanted doses ranged from 4.5×10^{16} to $8.7 \times 10^{16} \text{ cm}^{-2}$. Both AES and x-ray reflectivity measurements indicated the presence of an amorphous SiO_2 layer of approximately 20 nm at the sample surfaces. RSMs showed that the implanted region is tensile strained in the direction normal to the surface. The lack of significant diffuse scattering indicated that randomly distributed point defects are predominant compared to large displacement field defects such as clusters and dislocations. The strain profiles were obtained by a best fit procedure, which compares the measured x-ray spectra with the ones calculated within the framework of dynamical theory of x-ray diffraction. Close resemblance between the calculated strain profiles and AES nitrogen concentration profiles outside the amorphous region signal interstitial nitrogen as the main source of strain.

ACKNOWLEDGMENTS

The authors are very grateful to FAPESP (Grant No. 01/12746-0) and CNPq (Grants No. 150101/2003-1 and 142292/2004-4).

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