



# Reciprocal space mapping of silicon implanted with nitrogen by plasma immersion ion implantation

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## Abstract

Nitrogen was implanted in (001) silicon wafers using 12 kV pulses in a glow-discharge plasma immersion ion implantation (PIII) system and at 35 keV in an electron-cyclotron-resonance (ECR) PIII facility. An implantation depth of 80 nm and a retained dose of approximately  $3 \times 10^{17} \text{ cm}^{-2}$  were found, for both samples, from the nitrogen Auger profiles. Reciprocal space maps (RSMs) around the (004) and (113) Si lattice points were measured for the implanted and unimplanted Si wafers, using the high-resolution X-ray diffractometer in the triple axis configuration. An asymmetry in the reciprocal space coordinate  $Q_z$  (perpendicular to the sample surface) indicates that the implanted atoms force an increase in the Si lattice parameter in this direction. A broadening in the  $Q_x$  direction (parallel to the sample surface) was also observed, but with a less pronounced effect. For the sample implanted with higher energy, the shape of the map indicates a higher disorder in the crystal structure. © 2001 Elsevier Science B.V. All rights reserved.

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## 1. Introduction

Plasma immersion ion implantation (PIII) has recently appeared as a three-dimensional implantation method to treat surfaces of different materials like metals, semiconductors or dielectrics. During the PIII process, the ions of interest are

extracted directly from the plasma in which the samples are immersed in, by applying negative high-voltage pulses (typically 10–100 kV, 10–100  $\mu\text{s}$  duration time, 10–1000 Hz repetition rate) to the sheath formed between the plasma and the sample structure. The main advantage of PIII over other implantation methods is that, due to its immersion characteristic, it can successfully implant ions to manufactured pieces of different dimensions and/or complex shapes.

In very-low scale integration circuit technology, it is desirable to reduce the junction depth in order

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to minimize short channel effects, when scaling down the device dimensions. The PIII technique has been applied to produce ultra-shallow  $p^+-n$  or  $n^+-p$  junctions in silicon wafers [1,2]. In this case,  $BF_3$  ( $PH_3$ ) plasma sources are used for boron (phosphorous) implantation.

After implantation, the samples must be characterized to extract information about the surface and the implanted region. Among the various characterization methods available, X-ray diffraction appears as a good non-destructive one. In a previous work [3], we applied high-resolution X-ray diffraction to characterize (001)Si wafers implanted with nitrogen by the glow discharge (GD) PIII. The rocking curves around the (004)Si Bragg diffraction peak were measured before and after implantation, and a small distortion was observed for the as-implanted sample curve. The distorted rocking curve was successfully simulated by dynamical theory of X-ray diffraction, assuming a Gaussian distribution of strain through the implanted region with the central peak position and the width taken from the nitrogen profile obtained from Auger measurements.

The main objective of this work is to analyze reciprocal space maps (RSMs) measured around specific lattice points of implanted samples, to obtain information about the crystalline structure after implantation. For this purpose, silicon wafers were implanted with nitrogen at 12 keV in a glow discharge PIII system and at 35 keV in an electron-cyclotron-resonance (ECR) PIII facility. The samples were characterized by Auger electron spectroscopy in order to obtain the concentration profiles through the implanted region. We then perform reciprocal space mapping around the (004) and (113)Si lattice points, using the high-

resolution X-ray diffractometer in the triple axis configuration. These maps have the advantage of distinguishing between changes in lattice plane spacing to changes in orientation or mosaic effects, when compared to standard rocking curves.

## 2. Nitrogen-implanted silicon samples

The [001]-oriented Si wafers were implanted with nitrogen using two different PIII systems with different plasma sources. The first PIII facility, located at Laboratório Associado de Plasma – INPE (São José dos Campos – Brazil), uses a DC glow discharge (GD) plasma source with the plasma potential controlled at 70 V by an electron shower. Details about the system and the technique were published elsewhere [4,5]. Due to the chamber size, this GD PIII system is limited to a maximum high-voltage pulse of 15 kV. To implant Si wafers with higher energy, we use the ECR based PIII facility at the Research Center Rosendorf (Dresden, Germany). We expect to have  $N_2^+$  and  $N^+$  ion species in both plasma sources, but we are not able to measure their percentages. Table 1 summarizes the plasma and high-voltage pulse characteristics of the implanted Si samples analyzed in this work, together with some data obtained from Auger experiments. The difference between the delivered and retained dose is mainly due to the sputtering effect, which causes the saturation of the retained dose at approximately  $3.5 \times 10^{17} \text{ cm}^{-2}$  in both systems.

Although the two implanted Si samples with different energies (Z5 and Z4) showed different nitrogen Auger profiles, the implantation depth (maximum reach of nitrogen) was the same for

Table 1  
Data of the Si samples implanted with nitrogen by plasma immersion ion implantation<sup>a</sup>

Si sample	Plasma source	Pulse height (kV)	Pulse width ( $\mu\text{s}$ )	Repetition rate (Hz)	Exposure time (min)	Sample temperature ( $^\circ\text{C}$ )	Delivered dose ( $10^{17} \text{ cm}^{-2}$ )	Implanted depth (nm)	Retained dose ( $10^{17} \text{ cm}^{-2}$ )
Z5	DC-GD	12	100	670	15	300	6.0	80	3.0
Z4	ECR	35	5	1250	20	420	6.3	80	3.5

<sup>a</sup> The implantation depth and the retained dose were obtained from Auger profiles.

both samples (80 nm). The retained dose evaluated from the nitrogen Auger profiles [4] was also very similar. These peculiar results can be attributed to a combination of several effects. The etching depth due to sputtering is higher for the ECR sample (25 nm) than for the GD one (14 nm). The percentage of the ion species may be different for each plasma system, and a non-thermal diffusion of the implanted atoms may take place. At this stage, we cannot clearly address the actual reason for these results. Details about the implanted samples and their Auger profiles are being published in [6]. These two samples were chosen for the RSM analysis due to their similarity in retained dose and implantation depth.

### 3. Reciprocal space mapping

The Si samples were characterized by high-resolution X-ray diffraction using a Philips X'Pert MRD diffractometer in the triple axis configuration. In this configuration, a four-crystal Ge(220) monochromator is positioned just after the Cu X-ray tube (point focus), leading, for the incident beam, to an axial divergence ( $\Delta\omega$ ) of 12 arcsec and a wavelength dispersion  $\Delta\lambda/\lambda$  of approximately  $10^{-4}$ . Before reaching the detector, the diffracted beam passes through a Ge(220) channel-cut analyzer, which also reduces the detector acceptance angle ( $\Delta 2\theta$ ) to 12 arcsec. With the X-ray diffractometer in the triple axis configuration, the so-called RSM of a reciprocal lattice point (REL P) can be obtained by measuring a set of  $\omega/2\theta$  scans with different  $\omega$ -offset angles [7,8].

RSMs were measured around (004) and (113) RELPs of the Si lattice of the implanted samples and of an unimplanted reference wafer. The (004) maps were recorded in a  $\Delta(\omega/2\theta)$  range of  $\pm 0.15^\circ$  in steps of  $0.0005^\circ$  and in a  $\Delta\omega$  range of  $\pm 0.05^\circ$  in steps of  $0.002^\circ$ . The maps around the (113) RELP were performed using the low incident angle ( $\omega \sim 2.82^\circ$ ) with the detector at the Bragg angle of the (113) plane of Si ( $2\theta \sim 56.12^\circ$ ) with a  $\Delta(\omega/2\theta)$  range of  $\pm 0.15^\circ$  in steps of  $0.0007^\circ$  and in a  $\Delta\omega$  range of  $\pm 0.15^\circ$  in steps of  $0.004^\circ$ . We decided to use the low incident angle of the (113)

plane because it is more sensitive to surface modifications.

Fig. 1 shows the RSM around the (004) RELP of the Si wafer implanted with nitrogen at 12 keV using the GD PIII process and also, for comparison, of the unimplanted Si wafer. The maps are plotted in reciprocal space coordinates  $Q_x$  parallel

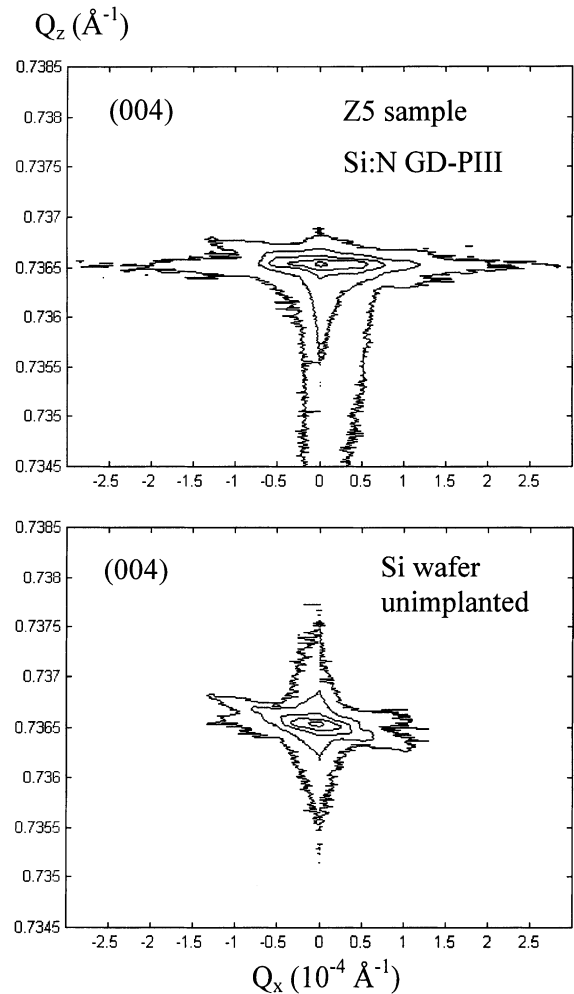


Fig. 1. RSM around the (004) lattice point of a Si wafer implanted with nitrogen by the GD PIII method at 12 kV (upper panel). For comparison, the (004) map of an unimplanted Si wafer is displayed in the same scale in the lower panel. The maps are plotted in reciprocal space coordinates  $Q_x$  parallel to the [110] in-plane azimuth and  $Q_z$  parallel to the [001] direction. The isointensity contour lines are at  $10^1$ ,  $10^2$ ,  $10^3$ ,  $10^4$  and  $10^5$  cps.

to the [110] in-plane azimuth (parallel to the sample surface) and  $Q_z$  parallel to the [001] direction (perpendicular to the sample surface). The contour lines in the maps are lines of equal intensity at different intensity values.

It is important to point out that, in the RSMs, the mosaicity (i.e. changes in orientation of the lattice planes) produces a broadening in the  $\omega$ -direction (parallel to the  $Q_x$  direction), while changes in the lattice spacing produce a variation in the  $Q_z$  direction. Since, for typical PIII process, the implanted atoms have low energies (10–50 keV), the X-ray diffraction curves only show modifications in their tails [3,9], i.e. for scattered intensities  $10^4$ – $10^3$  times lower than the maximum of the Bragg diffraction peak.

As can be observed in Fig. 1, the (004) RSM of the GD PIII implanted Si sample (Z5) exhibited a clear asymmetry in the  $Q_z$  direction, when compared to the map of the unimplanted sample. The scattered intensity increased for  $Q_z$  values lower than the point of maximum intensity and decreased for  $Q_z$  values higher than this point. This result demonstrates that the implanted atoms force an increase in the lattice parameter perpendicular to the sample surface. Incorporation of the nitrogen atoms at interstitial sites, segregation at grain boundaries or trapping in amorphous layer are some of the possible reasons for this effect. A broadening in the  $Q_x$  direction is also observed, i.e., an increase of the mosaicity in the Si lattice, but with a less pronounced effect.

The asymmetry in the  $Q_z$  direction of the implanted sample is also observed when measuring the (113) map. Fig. 2 shows the (113) RSM of the GD implanted sample Z5 together with the one of the unimplanted wafer. Since the (113) plane is inclined in relation to the sample surface, the (113) asymmetrical Bragg reflection carries information about the lattice spacing parallel and perpendicular to the surface. Similar to the (004) map, the (113) RSM of the sample Z5 shows that the variation in lattice spacing is totally perpendicular to the sample surface (in the  $Q_z$  direction), indicating an increase of the lattice parameter in this direction. However, the broadening in the  $Q_x$  direction of the (113) RSM of sample Z5 was found to be more pronounced than in the (004)

map. This fact can be explained by the lower incidence angle for the (113) reflection, which, due to the smaller penetration depth, is more sensitive to surface variations.

In order to evaluate the effect of the implantation energy in the crystalline structure of silicon, RSMs were also performed on Si samples implanted with higher energy. Fig. 3 shows the (004) and (113) RSMs of the sample Z4 implanted with nitrogen at 35 keV, using the ECR plasma source. One can observe that the shape of both RSMs is somewhat different than the respective ones of the sample implanted with 12

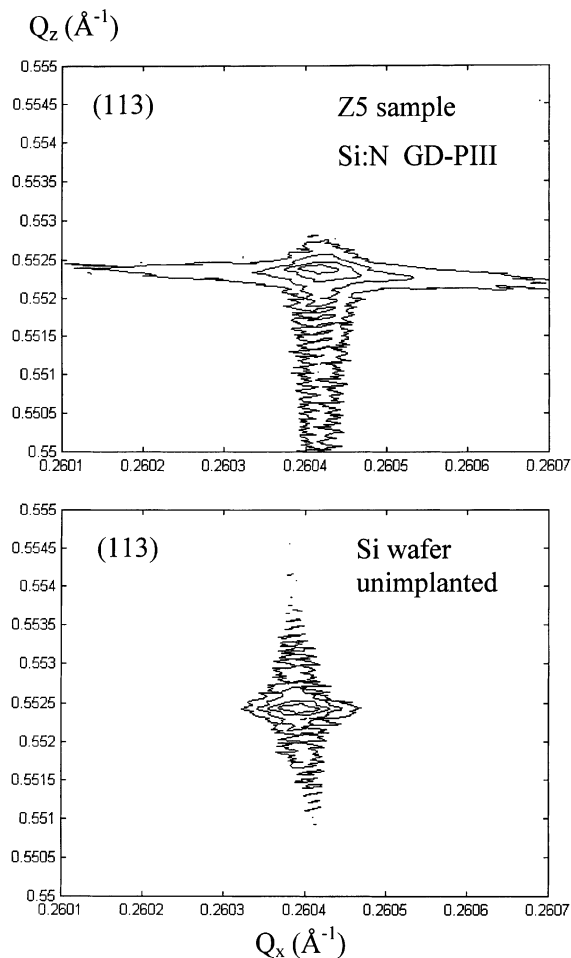


Fig. 2. The same as in Fig. 1 for the (113) RELP. The four contour lines correspond to  $10^1$ ,  $10^2$ ,  $10^3$  and  $10^4$  cps.

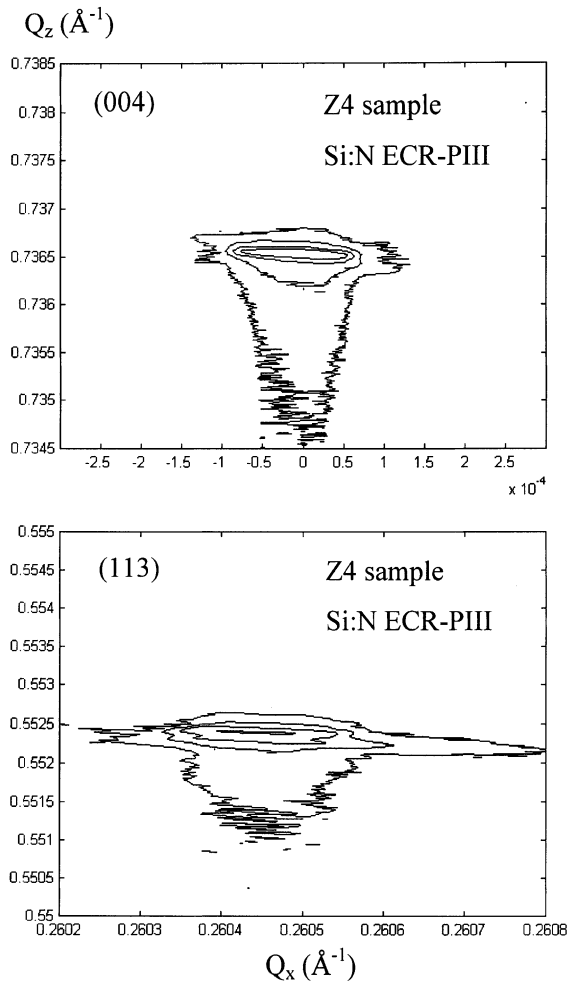


Fig. 3. RSMs around the (004) (upper panel) and (113) (lower panel) lattice points of a Si wafer implanted with nitrogen at 35 keV using an ECR plasma source. The contour lines are at intensities of  $10^1$ ,  $10^2$ ,  $10^3$  and  $10^4$  cps.

keV (Z5). The asymmetry in the  $Q_z$  direction still remains, but it is smaller than for the sample Z5, and the X-ray scattered intensity is now distributed over a larger area in both RSMs. The shape of the RSMs shown in Fig. 3 indicates that the higher energy of implantation produces more disorder in the Si lattice. We also observe a reduction by a factor of almost five in the intensity of the (004) and (113) Si Bragg peaks for this sample, indicating that the atoms with higher energy really produce more damage to the crystal

structure. This damage to the sample implanted with higher energy seems not to produce a mosaic structure in the lattice. The RSM of a typical mosaic-structured lattice would be ellipse-like curves with the principal axis perpendicular to the  $\omega/2\theta$  direction, i.e. perpendicular to the line which connects the respective RELP to the origin of the reciprocal space [10,11].

#### 4. Conclusions

We analyzed the (004) and (113) RSMs of (001)Si wafers implanted, by the PIII method, with nitrogen at 12 and 35 keV. An asymmetry in the  $Q_z$  direction (perpendicular to the sample surface) indicates that the implanted atoms force an increase in the lattice parameter in this direction. A broadening in the  $Q_x$  direction (parallel to the sample surface) is also observed, but with a less pronounced effect. The shape of the RSMs indicates that the nitrogen atoms implanted at 35 keV produces more damage to the Si lattice, but the disorder does not produce a mosaic structure. From these results, we expect that the RSM analysis would also give useful information when applied to annealed samples.

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