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# Defects in silicon introduced by helium implantation and subsequent annealing

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#### ABSTRACT

Formation and morphology of defects, including bubbles and voids, induced in silicon by He<sup>+</sup> implantation and subsequent high temperature and pressure treatment have been studied by means of X-ray diffraction method and grazing incidence small angle X-ray scattering (GISAXS). Enhanced pressure affects the formation of voids and/or of large cavities inducing creation of faceted structures. Moreover, high pressure treatment suppresses creation of interstitial-related defects.

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#### 1. Introduction

Helium implantation into silicon produces bubbles and other defects; due to the effect of the heat treatment He out-diffuses partially creating sponge-like buried layer and leaving voids (Griffioen et al., 1987; Misiuk et al., 2009). Such voids are of considerable interest as their properties are useful for numerous applications in semiconductor devices. An ability of voids to getter metallic impurities has been demonstrated (Raineri et al., 1995). An even more important property is the introduction of the mid-gap levels (Seager et al., 1994). This property can be used to control locally the minority carrier lifetime in silicon devices; fairly important applications of this effect have been demonstrated (Grob et al., 2003). Such increasing interest has strengthened efforts to study their fundamental properties to guarantee the reproducibility required in many applications. The reproducibility in void density and morphology is imperative to control locally the lifetime and to obtain identical device characteristics across the entire single crystalline Si wafer.

Defects in silicon implanted with helium, Si:He, subjected to annealing at up to 1270 K (*HT*), also under enhanced hydrostatic pressure, *HP*, are investigated in the present work. Transmission Electron Microscopy (TEM) was as so far the most widely used technique for structural investigation of voids in Si:He. As it has been reported earlier, based on the TEM data (David et al., 2003,

Zhang et al., 1998), as-implanted Si:He indicates, for implanted He<sup>+</sup> doses, D, within the range  $(5-10) \times 10^{16}$  cm<sup>-2</sup> and energy, E=50-150 keV, the presence of severely damaged region with a dense array of small bubbles near the projected range of He,  $R_{\rm He}$ . On annealing of Si:He at higher temperatures, relatively large He-filled voids are formed while helium desorption, dependent on processing parameters, hydrostatic pressure of ambient among them, takes place simultaneously (Misiuk et al., 2002).

The aim of this work is twofold:

The first one is to contribute to the structural characterization of bubbles and voids using grazing incidence small angle X-ray scattering (GISAXS) as one of the non-destructive techniques, which is very useful for analyzing structures with dimensions in the 1-150 nm range. It means that most of the available data were obtained only on subsequent annealing of the samples. GISAXS gives an opportunity to study bubbles in the as-implanted stage of Si:He, as well as the voids created on annealing.

The second goal is to show how the high temperature–high hydrostatic pressure (*HT–HP*) treatments influence the defect structure of Si:He in a view of evolution of the voids (gas-empty cavities) from the bubbles (gas-filled cavities).

#### 2. Experimental details

Czochralski grown Si (0 0 1) crystals (Cz-Si) were implanted with He<sup>+</sup> ions at temperature below 350 K using  $E_{\text{He}}$  + = 150 keV and D = 1 × 10<sup>17</sup> cm<sup>-2</sup>. After implantation the Si:He samples were subjected to annealing for 1.5–10 h at different temperatures

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(720–1270 K) under atmospheric and enhanced hydrostatic Ar pressure, *HP*, up to 1.1 GPa.

X-ray measurements were performed using a high resolution Philips material research diffractometer (MRD) equipped with a standard laboratory source of  $\text{CuK}_{\approx 1}$  radiation in the double and triple crystal configurations. We have recorded  $2\theta/\omega$  scans and the X-ray reciprocal space maps for the 004 reflections to study the changes in structural properties for the implanted and subsequently annealed Si:He crystals. The analysis of the shape of reciprocal lattice points including character of diffuse scattering near Bragg reflection is a powerful method to study point defects or their clusters that induce lattice distortion in crystals (see e.g. Moreno et al., 2003).

GISAXS is a powerful tool for structural characterization of bubbles because of its non-destructive character and sensitivity to electron density averaged over a large volume of material.

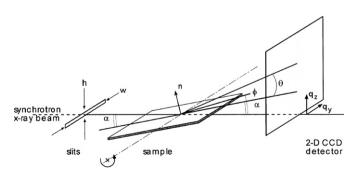
The GISAXS experiments were carried out at synchrotron facility of Elettra, Trieste, Italy, on the SAXS beamline (Amenitsch et al., 1995), using radiation with wavelength  $\lambda$ =0.07 nm and photon energy of 16 keV. A two-dimensional CCD detector with  $1024 \times 1024$  pixels, positioned perpendicular to the incident beam, was used to record the GISAXS intensity (see Fig. 1). A thin Al strip was placed in front of the 2D detector to avoid overflow of the detector in the specular plane direction where the much stronger surface scattering is present. The spectra were corrected for background intensity and detector response. The 2D GISAXS patterns represent the maps of scattering intensity in the reciprocal space. Details of the GISAXS geometry and of the data analysis were presented elsewhere (Kovacevic et al., 2006; Revenant et al., 2004).

The scattered GISAXS intensity is a sum of the contributions from the single scattering particles and its dependence on the scattering angle is proportional to the Fourier transform of the shape and size of particles (voids). When the particles are close to each other, the particle–particle correlation also influences the scattered intensity distribution. These two different contributions can be formally factored out in the scattered intensity as the form factor P(q) and the structure factor S(q):

$$I(q) \approx P(q)S(q)$$
 (1)

where  $\overrightarrow{q}$  is the scattering wave vector  $(\overrightarrow{q} = \overrightarrow{k}_f - \overrightarrow{k}_i, |q| = \frac{4\pi \sin\theta}{\lambda}, \overrightarrow{k}_i, \overrightarrow{k}_f$  are the wave vectors of the incident and scattered beams, respectively).

The form factor P(q) depends on the shape and size of the particles, while the structure factor S(q) includes the particle to particle correlation. For non-interacting particles, the structure factor has no effect on the scattering and thus S(q) = 1 in that case.



**Fig. 1.** Experimental setup for GISAXS measurements: h – the height and w – the width of the beam defining slit;  $\alpha$  – the grazing angle of incidence;  $\theta$  – the scattering angle;  $\varphi$  – the azimuth angle.

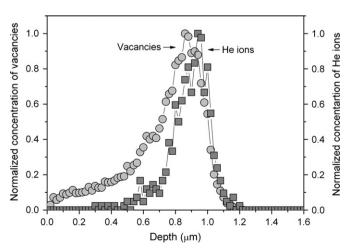
#### 3. Results and discussion

In this study, the implantation conditions (energy and dose) were chosen in order to induce formation of the bubbles, i.e. gasfilled cavities. It is well known that, for the He<sup>+</sup> implanted doses higher than  $1 \times 10^{16}$  cm<sup>-2</sup>, He agglomerates in the form of bubbles. The bubbles should be localized in a region where a high vacancy concentration is located. The normalized depth profile of helium ions and the corresponding vacancy profile obtained by SRIM code (http://www.srim.org) for a He<sup>+</sup> ion implantation at 150 keV with  $D=1 \times 10^{17}$  cm<sup>-2</sup> are shown in Fig. 2. For 150 keV  $He^+$  ions,  $R_{He+}$  equals to about 0.98  $\mu m$ . In comparison to the He ions distribution, the distribution of vacancies, produced at implantation, reaches the maximum placed closer in respect to the surface (Raineri et al., 2000). Ion implantation introduces into Si plenty of vacancies and interstitials. They are highly mobile even at low temperatures, and will therefore migrate for a long distance. Finally, they can either disappear from the material by recombination or become trapped by the impurity atoms. It is well established that vacancy in silicon tends to pair with impurities (Claeys and Simoen, 2002, Springer, Berlin).

The interaction of helium atoms with vacancies on implantation has been previously observed by several photoluminescence (PL) studies (Bak-Misiuk et al., 2005; Raineri et al., 2000). The PL peak at about 0.92 eV (Bak-Misiuk et al., 2005) has been ascribed to the vacancy clusters filled with helium (bubbles) and also the broad PL peak at about 0.94 eV (Raineri et al., 2000) has been ascribed to the bubbles. The broadening of the peak has been described by the presence of clusters with different sizes.

X-ray methods were applied to obtain information about the overall Si:He sample structure while more detailed structure of the bubbles was determined by GISAXS. X-ray reciprocal space maps and  $2\theta/\omega$  scans of the Si:He samples are presented in Figs. 3 and 4, respectively. Numerous interference fringes were observed (Figs. 3 and 4) for the as-implanted sample, confirming its layered structure, composed of the relatively perfect top Si layer (shot through by implanted He $^+$ ) and of the disturbed buried layer containing implanted helium. The polycrystalline buried layer introduces strain generating the additional peaks. Observation of the fine structure means that the interface between the top and implanted layer is smooth.

After processing at 720 K, the X-ray diffuse scattering, related to the presence of point defects as well as to the thickness fringes



**Fig. 2.** Normalized depth profile of helium ions and the corresponding vacancy profile obtained by SRIM code for a  $He^+$  implantation at 150 keV to a dose  $1 \times 10^{17}$  cm<sup>-2</sup>.

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