Propagation and interactions of cracks in Si induced by H supply into He-filled cracks

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Abstract. The phenomena of interaction and propagation of cracks under the contribution of hydrogen were studied in (001) silicon substrate in which an array of scattered overpressurized He-plates was previously introduced at a given depth. Their propagation under subcritical regime was activated through diffusional supply of H atoms introduced by implantation/annealing. Interactions between the tips of non coplanar cracks take place in a nanometric scale; they can be of plastic-type leading to the formation of extended defects or of elastic-type resulting in deviations of crack-tip propagation. While the planar interactions facilitate the propagation of cracks, those of non coplanar-type stop them. The observations were carried out by transmission electron microscopy and the results were discussed and modelled by using concepts of elasticity and fracture mechanics.

1. Introduction

Hydrogen implantation induced cracking in semiconducting material has been extensively studied in connection with thin layers transfer technology. This process is a key issue for the fabrication of semiconductor-on-insulator (SOI) substrates – the so-called Smart-Cut® process [1]. This ion cutting method drives the lateral propagation of cracks over the whole wafer area resulting in a SOI structure when bound onto another wafer. A significant amount of research on this hydrogen ion-cut process in silicon has thus been carried out to clarify the underlying mechanisms of layer splitting [2-5]. Platelets resulting from H implantation grow during post-thermal annealing to form large microcracks in the narrow depth region around the projected range of the H ions. Their propagation and interactions lead to exfoliation of a thin layer from the wafer. Later it has been shown that the co-implantation of H and He ions allowed blistering at much lower total fluence than H implantation alone [6]. The blistering facilitation is understood as due to helium capture by the previously introduced H platelets, He acting as an overpressurizer of platelets. Even in silicon, despite significant efforts, many physical aspects of the evolution of platelets to microcracks and then their interaction into large cracks are still unclear.

This paper investigates the propagation and interactions of cracks into implanted silicon. Specific two-steps experiments were designed to allow controlled crack propagation. H atoms were introduced into pressurized He-filled cracks previously formed under particular conditions of He implantation. This original approach produces rather efficient, flat and controlled growth of cracks [7]. In this paper
previously the results obtained [7] are more detailed and completed. The resulting microstructure was examined by transmission electron microscopy (TEM) and discussed in terms of elasticity and fracture mechanics.

2. Experimental
The experiments were performed using p-type (001) Si Cz-wafers with a resistivity of 1-25 Ω cm. In the first step sample pieces were implanted at room temperature with He⁺ ions accelerated at 45 kV to a fluence \( \Phi = 1 \times 10^{16} \text{ cm}^{-2} \), and then annealed at 350 °C during 900 s. These experimental conditions are known to lead to the formation of over-pressurized He-platelike cavities (or He-plates) located at the mean projected range of He atoms (≈ 400 nm underneath the surface) [8]. In the second step of the process, the samples were implanted at 200°C with 30 keV H₂⁺ ions to a fluence \( \Phi = 0.5 \times 10^{16} \text{ cm}^{-2} \). The predicted mean depth range of the H⁺ implantation is approximately 200 nm according to SRIM calculations [9]. The as-He/H co-implanted samples as well as a reference sample containing only the He-plates were finally subjected to a final thermal treatment at 300°C for 900 s. Microstructure characterization was performed by transmission electron microscopy (TEM) in cross-sectional specimens prepared by mechanical polishing and ion milling. The observations were carried out in a JEOL JEM 2010 microscope operating at 200 kV.

3. Results
Figure 1 shows the microstructure after the first step of the procedure, i.e. the He implantation followed by the 350°C annealing [7]. As expected scattered He-plates form close to the mean projected range of He. The average distance between He-plates exceeds their mean diameter of ≈ 150 nm, resulting in a discrete planar array of plates oriented along the (100) lattice planes parallel to the surface. The surrounding fringes of the two edge-on He-plates seen on the cross sectional TEM image are ascribed to the strain-related contrast induced by the high gas pressure inside the over-pressurized He-plates [8]. The elastic strain field of the He-plate can be calculated in 2D by modelling the He-plate by a dipole of edge dislocations with an effective Burger’s vector [10]. The local strain field can be used to favour sublocal organized arrangement of H precipitates [11]. No modification of both the arrangement and the size of He-plates are observed after an annealing at 350°C suggesting that the He-plates are quite stable with respect to 350°C.

![Figure 1. Cross-sectional bright-field TEM image showing two edge-on overpressurized He-plates observed in He-implanted silicon (1x10^{16} \text{ cm}^{-2}, \text{ RT}) and then annealed (350°C, 900s) [7].](image)

After the formation of the He-plates, the samples were implanted with H₂⁺ ions at 200°C and then annealed at 300°C during 900 s. A general view of the developed microstructure is provided in figure 2. Nano-sized H-platelets are observed around a depth of 200 nm. However, at a depth of ≈ 400 nm, where the He-plates were originally located, a chain of long and quite flat cracks is now observed. This shows that some H atoms diffused towards the He-plates initiating a process of rectilinear crack propagation.
Figure 2. Bright field cross sectional TEM image of sample submitted to the total process, i.e., after He-implantation (45 keV, $1 \times 10^{16}$ cm$^{-2}$, RT, annealed 350°C for 900s), and then H$_2$-implantation (30 keV, $0.5 \times 10^{16}$ cm$^{-2}$, 200°C, annealed 300°C for 900s) [7]. The upper part shows in detail the different types of H-platelets as formed.

Various types of interaction between the long cracks are observed, see figures 3 and 4. These interactions can be of plastic or elastic type. In case of plastic interactions dislocations and nanotwin structures connecting the crack tips are observed, figure 3. For interactions of elastic type, deviations of crack propagation are observed; the two approaching cracks merge by redirecting the direction of propagation against each other, figure 4. Experimentally the elastic-type interaction seems to be effective for distances of approach lower than 50 nm; the spatial extension of the interaction is thus limited around the tip of crack [7].

4. Discussion
Most of the He-plates formed during the first step of the process form along (001) plans parallel to the surface due to the implantation in-plane stress [10]. For simplicity the He-plate was treated as a penny-shaped crack of radius $d$ in an infinite elastic medium subjected to an internal pressure $p$ according to the linear elastic fracture mechanism. The He-crack stability up to 350°C shows that the stress intensity factor at the crack tip, $K_I$, does not overcome the fracture toughness, $K_{IC}$, of the medium ($=1.19 \times 10^6$ Nm$^{-3/2}$ in silicon [12]) required to propagate the crack in unstable manner. Therefore, this suggests that:

$$p < p_{max} = K_{IC} \left( \frac{2}{\pi d} \right)^{1/2}$$

(1)
where $d$ is the diameter of the crack. Conversely the pressure is larger than the pressure for a crack in equilibrium given by [13]:

$$p > p_{\text{min}} = \left( \frac{2\pi \mu \gamma}{(1-\nu)d} \right)^{1/2}$$

(2)

where $\mu$ is the shear modulus, $\nu$ the Poisson’s ratio and $\gamma$ the surface energy. So, the catastrophic propagation of a 150 nm He-crack (introduced by the first step of the process) controlled by the fracture toughness would require an additional overpressure of maximal value $\Delta p_{\text{max}} = p_{\text{max}} - p_{\text{min}} \approx 200 \text{MPa}$ resulting in the second step of the process (H$_2$ diffusion facilitated by the out-of plane strain of the He-plates). According to the ideal gas law the increase of pressure in the He-plate is directly proportional to the amount of added molecular hydrogen. Thus only few percents of implanted H atoms would require diffusing towards the He-plates to initiate the crack propagation via the increase of the inner gas pressure. However the role of hydrogen can be of chemical nature by breaking the strained Si-Si bonds at the crack-tip. In this case the Si-H formed should influence the internal structure of the crack and therefore should assist the crack propagation. This last scenario is more likely because it was shown that He not only leads to a high internal pressure but also induces the reconversion of molecular H$_2$ into bound Si–H [14]. Similarly recent dynamical simulations confirmed the growth of H-induced platelets via subcritical stress-corrosion mechanism rather than any significant H$_2$ pressure buildup [15]. In this last case the propagation of cracks takes place under subcritical conditions (i.e., with $K_I < K_{IC}$). The additional contribution of H is thus an effective process for the growth of large cracks. Moreover the rectilinear cracks suggest flat final surface in the ion cutting process.

**Figure 3.** Example of plastic-type interaction between two cracks propagating towards each other. The HRTEM shows the microtwinn-like defect between the crack-tips [7].

**Figure 4.** Example of elastic interaction where both crack tips run around each other (a) schematic of the process [14], (b) and [7] (c) TEM examples
During the growth of cracks, when the distance between two parallel crack tips becomes small enough complex phenomena of interaction are observed (figure 2). According to the different TEM observations, when two cracks get closer, they can interact in a plastic way blocking so brutally their growth, figure 3. This mechanism of relaxation of elastic strain by plastic deformation is the origin of the formation of structural defects such as nanotwins. The formation of structural defects requires high stresses at the crack-tips. TEM observations showed that the nucleation of perfect dislocations requires stresses larger than 1 GPa [16]. However, when the stress between a pair of parallel cracks is not quite important for satisfying the plastic deformation conditions, the cracks continue their expansion while remaining subjected to the interaction of elastic type, leading to a deviation of their path, figure 4. This behaviour of attractive-type can be explained by taking into account modifications of the stress intensity factor $K$ of a given crack-tip due to the elastic forces induced by the approaching second collinear crack-tip. According to Gross and Seelig [17] and assuming equilibrium conditions (i.e, equation (1) $\sim$ equation (2)) the $K$-factors, $K_I$ (mode-I loading) and $K_{II}$ (mode-II loading), of two parallel cracks of midpoint distance $L$ and of interaction angle $\theta$ are given by:

\[
K_I \approx (\pi\beta) \left[ 1 + \frac{1}{8} \alpha^2 (2 \cos 2\theta - \cos 4\theta) \right]
\]

\[
K_{II} \approx \frac{\pi}{8} \beta \alpha^2 \left[ -\sin 2\theta + \sin 4\theta \right]
\]

where $\beta = \left( \frac{\mu}{1-\nu} \right)^{1/2}$ is a constant characteristic of the matrix and $\alpha = \frac{d}{L}$ an adimensional term characterizing the distance of interaction. For large distance $L$ and for coplanar cracks ($\theta = 0$) $K_I$ prevails (mode-I loading) and cracks develop in a rectilinear way to form long straight cracks as observed (figure 2). However, for cracks of vertical separation $Z$, when interacting, deviations from a straight path occur; the angle at which a propagating crack is deviated is mainly controlled by the $K_{II}$ factor [17]. Figures 5 and 6 show the variations of the reduced factors $K_I/\beta$ and $K_{II}/\beta$ respectively as function of both $\alpha$ and $\theta$.

As seen on figures 5 and 6 when two crack-tips approach (for $Z \neq 0$), both $\alpha$ and $\beta$ increase and a first phase of slight increase is observed for both $K_I$ and $K_{II}$ resulting in minor modifications of propagating parameters. However, with decreasing interaction distance $K_{II}$ becomes negative and increases in absolute value (for $\theta < \pi/6$) which means a change (attraction) in the prospective direction of crack growth [18]. Both crack-tips run thus around each other and each of them merges with the other crack as observed in figure 4. Conversely, along their interaction, $K_I$ decreases suggesting a reduction in the propagation kinetics of cracks. The dotted lines in figures 5 and 6 show two possible paths of interaction. Furthermore $K_{II}$ reaches maximal values when cracks are close and for $\theta = \pi/3$. Larger values of $Z$ result in larger values of $\theta$ for a given value of $\alpha$ causing thus a reduction in the interaction strength and therefore limiting the cutting effects for the layer separation. More work is needed to determine more precisely the transition stress at the crack-tips between plastic and elastic interaction.

The formation of coplanar cracks being ideal to form straight cracks but not realistic due to the implantation straggling. This limitation can be overcome by introducing well localized sites of nucleation as observed in strained interfaces [19].
Figure 5. Plot of the reduced factor $K_I / \beta$, equation (3), as function of $\alpha$ and $\theta$. The dotted lines show different examples of interaction.

Figure 6. Plot of the reduced factor $K_{II} / \beta$, equation (4), as function of $\alpha$ and $\theta$. $K_{II}$ shows a change in the sign for $\theta = \pi/6$ which means a qualitative change in the prospective direction of crack growth.

The diffusion of H can also be performed by using a plasma hydrogenation process. Plasma hydrogenation based techniques have also been studied for their use in the transfer of thin films [20] Figure 7 shows a long and straight crack formed by the diffusion of H during the plasma hydrogenation into the previously introduced He-plates. This last point confirms that the He-plates can be used as precursors of cracks whose propagation under subcritical regime is activated through diffusional supply of H atoms.

Figure 7. Cross-sectional bright-field TEM image showing a long and sharp crack observed after He-implantation (45 keV, 1x10^{16} cm^{-2}, RT, annealed 350°C for 900s) and plasma hydrogenation at 300°C during 1h. (S denotes the surface)
5. Conclusion
The propagation and interactions of cracks in Si were observed within a nanoscopic size scale by using a specific co-implantation process. Starting from overpressurized He-plates, subcritical propagation of cracks was activated through additional H atoms and micrometer sized rectilinear cracks were obtained. This type of He-based engineering could be used to reduced the amount of H required for the propagation of cracks and thus for the layer transfer. Plastic or elastic interactions of these cracks were observed and studied. Plastic interactions result in the formation of dislocations and twin-like extended defects due to a high shear stress of the two interacting cracks. Elastic interactions cause deviations in their path resulting in an interlacement of their tips as predicted by the calculations. The roughness of the as-split layer should be controlled by the interactions of propagating cracks.

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