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A method to determine the pressure and densities of gas stored in blisters: Application to H and He sequential ion implantation in silicon

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A B S T R A C T

H and He sequential ion implantation of silicon followed by annealing leads to the formation of gas pressurized cavities. When close enough to the surface, they elastically deform this surface and generate blisters. Gaining knowledge of the characteristics and thermal behavior of these blisters is mandatory for the optimization of the Smart Cut™ process. In this paper, we develop the idea and demonstrate that the pressure and the concentrations of the gases inside a blister can be inferred from its actual dimensions and depth location by using simulations based on Finite Element Method (FEM) modelling. We apply this method to initiate a study on the influence of the respective fluences of H and He ions used in a sequential implantation on blistering efficiency.

Keywords:

Ion implantation
Defects in silicon
Elastic deformation

1. Introduction

Hydrogen and Helium sequential implantation in Si followed by wafer bonding and annealing allows for slicing and the transfer of Si layers from a donor substrate to a host material. The Smart Cut™ technology, based on this principle [1], is used to fabricate Silicon-On-Insulator (SOI) substrates, the starting material of many electronic and photovoltaic modern devices [2,3]. After implantation and during annealing, silicon vacancies and hydrogen atoms precipitate in the form of platelets of nanometer dimensions that progressively grow by Ostwald ripening [4,5]. When their density is large enough, the platelets further evolves during annealing and finally form highly pressurized micro-cracks [5]. When these micro-cracks are located close enough to the wafer surface, stress relaxation occurs through the formation of blisters elastically deforming the surface [6]. Alternatively, when a stiffener is bonded onto the implanted layer, such relaxations cannot occur and the in-plane stress exerted by these micro-cracks finally leads to the fracture and delamination of the whole layer, the working principle of the Smart Cut™ process. This is why the optimization of the various parameters which define a particular sequential implantation (order, energies, fluences), in order to minimize the total fluence needed to fracture the implanted layer when bonded to a stiffener, is often undertaken by studying the characteristics of the blisters formed at the free surface of this layer.

One important characteristic of these blisters is the concentration and composition of the gas they contain. Indeed, the efficiency of the process is directly linked to the proportion of the implanted dose which is ultimately stored within them. Unfortunately, up to now, a

method to measure these characteristics was lacking. In this paper, we develop the idea that the pressure and the concentrations of the gases inside a blister can be inferred from its actual dimensions and depth at which it is formed with the help of simulations based on Finite Element Method (FEM) modelling. We apply this method to initiate a study and understand the influence of the respective fluences of H and He ions used in a sequential implantation onto the blistering efficiency.

2. Samples

(001) Si wafers covered by a 25 nm-thick thermally grown SiO₂ layer (wet oxidation at 800 °C) were sequentially implanted at room temperature with He at 8 keV (Rp from SIMS not shown at about 100 nm below the surface) then with H at 3 keV (Rp from SIMS not shown at about 60 nm below the surface), so that the He ions were implanted before and deeper than H ions. The H fluence (Φ_H) was fixed at $0.6 \times 10^{16} \text{ cm}^{-2}$ while the He fluence (Φ_{He}) was 0.4×10^{16} , 0.6×10^{16} , 0.9×10^{16} or $1.2 \times 10^{16} \text{ cm}^{-2}$, depending on the samples. These samples were then annealed at 550 °C for 30 min under nitrogen in a conventional furnace.

3. Experimental results

Fig. 1 shows the images obtained by SEM of the underlying cavities (using back-scattered electrons) on the different samples (left column). On the right, the surface topographies of the same samples observed by AFM are shown. White spots in the AFM images reveal protuberances due to the presence of blisters deforming the surface. The SEM pictures are used to measure the sizes and densities of the cavities while the AFM images are used to measure their heights. The result of the statistical analysis of such SEM images is shown in Fig. 2.

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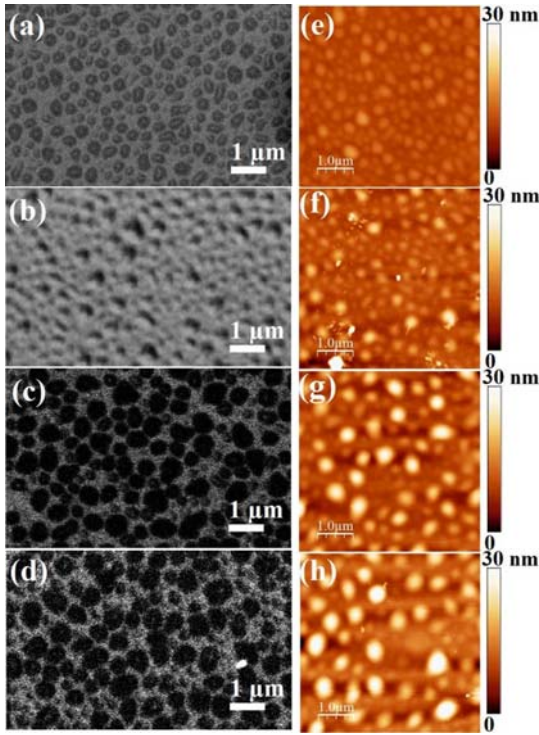


Fig. 1. SEM images of cavities (left column (a–d)) and AFM images of blisters (e, h) observed on the different samples. The $\Phi_{\text{He}}/\Phi_{\text{H}}$ ratio equals to: (a, e), 0.67; (b, f), 1; (c, g), 1.5 and (d, h), 2. The zero value of height contrast in the AFM images was assigned to the lowest (thus darkest) areas in the AFM images.

These graphs show that when the He fluence increases by a factor 3, the average diameter of the blisters increases by a factor 2, while their density decreases by a factor of 5. As a result, the surface fraction they occupy stays almost constant at about 60–55%.

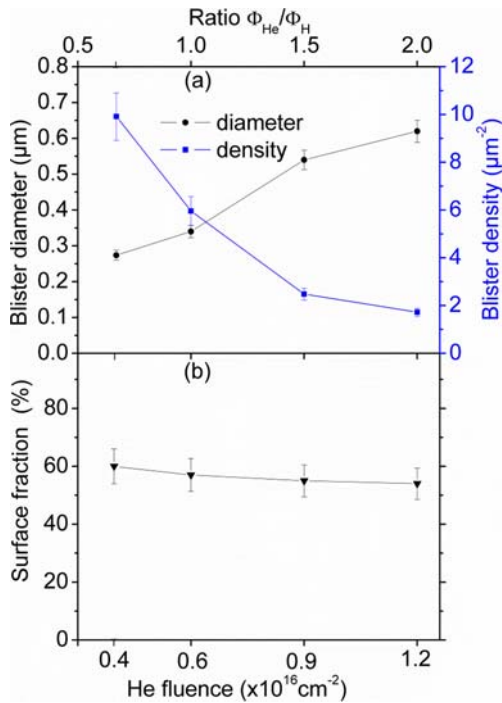


Fig. 2. (a), Average diameter (solid black circles) and density (square blue solid) of the blisters; (b), surface fraction they occupy, as a function of the He fluence. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

Fig. 3 shows the result of plotting the height of the blisters (right axis) as a function of their diameter.

This graph shows that there is a direct correlation between the diameter of a cavity and the blister height it generated and this, whatever the He fluence.

Finally, the last missing information i.e., the depth at which these cavities are located was obtained by XTEM (not shown here but same as in Ref [6]). Cavities are located at a depth of 60 nm from the wafer surface.

4. Gas pressure inside a cavity: FEM model

In our model (implemented using Comsol Multiphysics 4.3), a crack is initially described as a cavity of oblate spheroidal shape, as experimentally observed [6], characterized by a major and a minor axis, its diameter and its thickness, respectively. The crack is embedded within a semi-infinite (001) Si substrate covered by a 25 nm-thick oxide layer and located at 60 nm below the wafer surface (as deduced from SIMS and observed by XTEM). The in-plane stress of the oxide layer is biaxial and compressive and was set at a value of 0.36 GPa as expected for such an oxide [7]. The wafer surface is initially set unstressed in the vertical direction and free to move in every direction. The substrate is rigidly fixed at the bottom of the model structure while the vertical “walls” limiting the substrate are only allowed to move vertically. The pressure exerted on the internal surface of a crack of given diameter was adjusted until reproducing through elastic relaxation the actual (measured) height of the blister. Both the volume offered by a blister and its internal pressure can be obtained from this simulation. Fig. 4 shows a hybrid plot evidencing the vertical displacement and radial stress fields characterizing a blister after relaxation.

Being able to extract from FEM the gas pressure generating a blister of given height, Fig. 3 (left axis) can be replotted to show the univocal relation linking blister diameter and gas pressure inside it.

5. From pressure to concentrations

Given the high pressures deforming the blisters, the ideal gas law cannot be used to deduce the gas concentrations. Instead, we use the experimental data reported in [8,9]. From the results shown in these references, one can see that hydrogen molecules are more efficient than helium atoms to generate pressure. A given high pressure can be obtained either by a minimum molar concentration of hydrogen molecules, a maximum concentration of helium atoms or an intermediate concentration of a mixture of both gases.

The total concentration of molecules $C_{\text{HeH}_2}(P_i(d_i)) = xC_{\text{He}}(P_i(d_i)) + (1 - x)C_{\text{H}_2}(P_i(d_i))$ contained within a blister cavity “i” of diameter d_i and volume V_i can be obtained by combining the data from Fig. 3 and

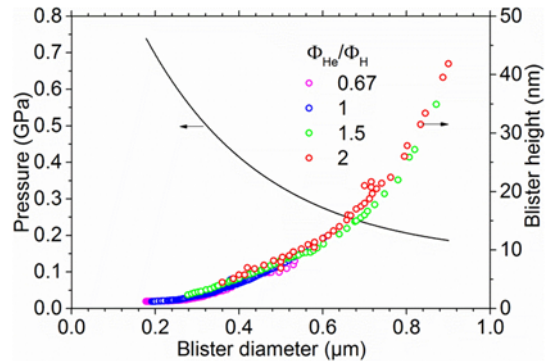


Fig. 3. Right axis, height of a blister as a function of its diameter. Left axis, after transformation using FEM, gas pressure inside that blister as a function of its diameter. (compilation of all He fluences).

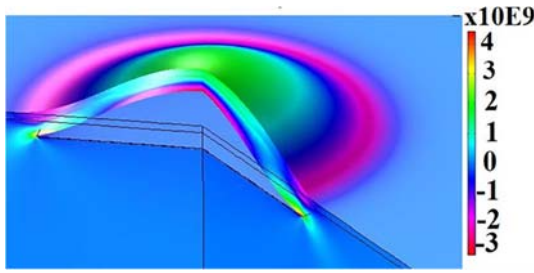


Fig. 4. Typical shape and radial stress fields characterizing a blister in equilibrium with its internal pressure. The vertical dimension was enlarged by a factor of 5 for a better visualization. Unit is Pa.

those found in [8,9], provided the respective molar fractions x and $(1 - x)$ of the two gases are known.

The areal density of molecules, i.e. the number of molecules contained within the blisters and found within a surface area S , is given by

$$\rho_{\text{HeH}_2} = \sum_i C_{\text{HeH}_2}(P_i(d_i))V_i/S,$$

with $\rho_{\text{HeH}_2} = x\rho_{\text{He}} + (1 - x)\rho_{\text{H}_2}$.

We have plotted in Fig. 5 using blue or red solid squares respectively, the areal density of molecules that would be needed to generate the observed blisters using exclusively He or H_2 molecules, and this for the different He fluences studied in this work. It follows that the density needed for any mixture of both gases to generate the same blisters would lie between these two extreme bounds.

We now focus on the amount of implanted gases which could contribute to the pressurization of the blisters. Resulting from the debonding of two successive planes, cavities and blisters offer large internal (001) surfaces hosting large concentrations of dangling bonds which hydrogen atoms passivate. The density of dangling bonds provided by these (001) planes equals $\gamma = 4/a_{\text{Si}}^2$ where a_{Si} is the lattice parameter of silicon. Each dangling bond is passivated by one hydrogen atom. Thus, the total number of H_2 molecules used to passivate the

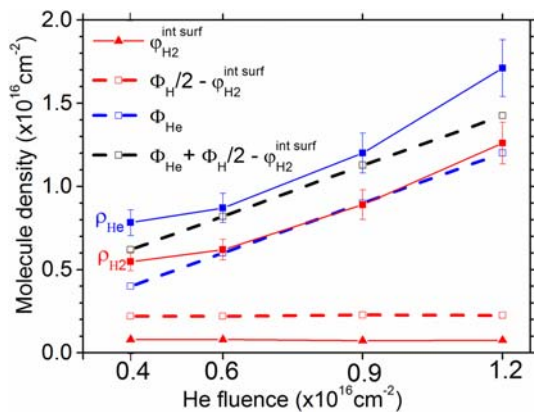


Fig. 5. Blue and red solid squares correspond to the upper and lower limits of molecule density of a mixture consisting of He and H_2 molecules and needed to pressurize the observed blisters. The red solid triangles show the density of H_2 molecules needed to passivate the internal surfaces of the blisters. Open symbols show the helium (in blue), the H_2 (in red) and the total (in black) densities available from the implanted fluences for pressurizing blisters. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

internal surfaces of the blisters found within the area S is given by

$$\phi_{\text{H}_2}^{\text{int.surf}} = \sum_i \frac{\gamma S_i}{2S} \equiv \sum_i \frac{\pi d_i^2}{a_{\text{Si}}^2 S}.$$

The values of $\phi_{\text{H}_2}^{\text{int.surf}}$ are also plotted in Fig. 5 (red solid triangles).

Thus, only some part of the implanted H fluence ($\Phi_{\text{H}}/2 - \phi_{\text{H}_2}^{\text{int.surf}}$) is left available to eventually pressurize the blisters (red dashed line) while the whole He fluence is available (blue dashed line).

From this graph, it is obvious that the blisters we have observed in this study cannot be pressurized by one type of gas only. None of the red and blue dashed lines stays between the two red and blue plain lines. Moreover, the molecule density of H_2 being small, this demonstrates that blisters are necessarily filled by a gas mixture in which He plays a major role.

Alternatively, if we plot the total molecular density of H and He available to pressurize the blisters $\Phi_{\text{He}} + \Phi_{\text{H}}/2 - \phi_{\text{H}_2}^{\text{int.surf}}$ (in black dashed line), we note that this line stays within the boundaries fixed by the two blue and red plain lines. In other words, the incorporation of all the implanted H and He ions within blisters would perfectly explain our observations. Moreover, the fine examination of the “distance” between the black dashed line (total available fluence) and the red plain line demonstrates that more than 95% of the gases initially available must be stored in the blisters.

From this observation, the relative concentrations of the gases filling the blisters can be found and are obviously very close to the relative fluences used in the experiments.

6. Conclusions

Cavities and blisters are formed after sequential H and He implantation. In this paper, we have shown how to extract the gas pressure contained within a blister of given dimensions from the FEM simulations of the elastic relaxation of silicon when exposed to the vertical stress exerted by a pressurized cavity. We show that the height of a blister developing from a cavity located at a given depth only depends on its diameter. Calculating the molecule densities that would be required to fill the blisters we have observed, we demonstrate that they are necessarily pressurized with a mixture of both gases and that almost all the implanted fluences end up within the blisters, at least for the experimental conditions studied in this work. Finally, increasing the He fluence with respect to that of H mainly results in an increase of the diameters and of the heights of the blisters and thus of the volume they offer.

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