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Study of surface exfoliation on crystalline silicon induced by Co-implantation of He and H ions*

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Abstract Crystalline n-type Si (100) wafers were implanted at room temperature with 160 keV He ions to a fluence of 5×10^{16} cm^{-2} or 40 keV H ions to a fluence of 1×10^{16} cm^{-2}, singly or in combination, followed by thermal annealing. Scanning electron microscopy (SEM), atomic force microscopy (AFM) and cross-sectional transmission electron microscopy (XTEM) have been used to investigate ion implantation induced surface phenomena and thermal evolution of micro-defects. SEM results show that successive implantation of He and H ions could induce surface blistering and exfoliation of Si at high thermal annealing temperature. AFM observations reveal that the surface exfoliation is mainly achieved at the sample depth corresponding to the projected range of H ions. XTEM observations demonstrate that the occurrence of surface blister or exfoliation on silicon can be attributed to the important role of He implantation in the thermal growth of H induced defects.

Key words crystalline Si, He and H ion implantation, surface blistering and exfoliation, bubbles growth

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1 Introduction

SOI materials have been widely used in modern large scale integrated circuits due to its unique advantages. Technologies, such as SIMOX (Separation by Implantation of Oxygen), BESOI (Bonded and Etched-Back Silicon-on-insulator) as well as Smart-cut have been considered to be the most competitive at present. The Smart-cut process designed by Bruel[1] in 1995 incorporates ion implantation, wafer bonding, and fracture to achieve a thin layer transfer. This process potentially allows SOI wafers to be produced more economically since the remaining sheared wafer can be re-polished and used again[2]. Moreover, due to its generic nature, Smart-cut enables fabrication of several other combinations of semiconductors and insulators, building of multilayer substrates and devices, and matching of materials that can hardly be grown epitaxially[3, 4].

In recent years many theoretical and experimental researches have been carried out to reduce the implantation fluence for the layer transfer of Si. It has been revealed that H and He sequential implantations can reduce both the total fluence and the thermal budget required for Si surface exfoliation as compared to the case of H single implantation[5, 6]. However, in the case of H and He co-implantation, the formation of surface damage is involved with many complex processes, such as the interaction between the two kinds of atoms, the correlation between the defects

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and H/He atoms and the defects induced by H/He atoms, which leads to much more difficulties in correctly understanding the mechanism of surface exfoliation. Therefore more experiments should be carried out in order to get a clear view of exfoliation mechanism. In addition, up to now, most co-implantation studies mainly focus on implantation at low energy (several tens of keV) and the mean projected range (R_p) for He or H ions is nearly the same. Our earlier study has shown that serious exfoliation of 1.0μm surface layer could be achieved on Si by successive implantation of 160 keV He ions and 110 keV H ions and after subsequent high temperature annealing[7]. In this study, crystalline Si were pre-implanted with higher energy (160 keV) He ions and then subjected to H implantation at lower energy (40 keV). Surface blistering and exfoliation with the thickness of about 0.50 μm have been observed after subsequent thermal annealing above 500 ℃. The possible mechanism for surface exfoliation has been tentatively discussed in combination with XTEM observations.

2 Experimental

N-type Si (100) wafers (3—6 $\Omega\cdot$cm) were implanted at room temperature with 160 keV He or 40 keV H ions, singly and in combination. In the case of co-implantation, H implant was chosen to follow He ions. The implantation fluence for He and H ions were chosen to be 5×10^{16} cm^{-2} and 1×10^{16} cm^{-2}, respectively. TRIM simulations show that the mean projected ranges for He and H ions are about 1.0 and 0.5 μm, respectively[8]. After implantation, the Si wafers were cut into smaller pieces and subjected to furnace annealing in a temperature range from 500 ℃ to 1000 ℃ in N_2 ambient for 1 hour.

The implantation and subsequent annealing induced surface morphologies have been studied by a scanning electron microscopy (SEM). The SEM observations were carried out with a JSM-6700F field-emission electron microscopy, operating at 10 kV. Meanwhile, an atomic force microscopy (AFM) was used to quantitatively characterize the three-dimensional surface structures, the thickness of exfoliated layer and the height of blisters. Finally, cross-sectional transmission electron microscopy (XTEM) observations were performed to investigate the microstructures of defects in He and/or H implanted Si after high temperature annealing. XTEM images were taken at 200 kV with a JEOL 2010 microscopy.

3 Results and discussion

SEM observations have revealed that no significant surface phenomena, such as blistering, exfoliation, and etc. have been created on 160 keV He-only or 40 keV H-only implanted samples even after thermal annealing up to 1000 ℃. As an example, Fig. 1(a) gives the typical SEM image obtained on Si implanted with 160 keV He ions and followed by 1000 ℃ annealing. However as for the Si co-implanted with He and H ions, the surface morphologies proceed a series of significant changes, which depend strongly on the annealing temperature. For the annealing temperatures lower than 500 ℃, same as the single ion implantation, only flat and smooth surface has been observed. Nevertheless, as increasing annealing temperature to 600 ℃, as shown in Fig. 1(b), surface exfoliation is clearly found, leaving a large quantity of irregular craters on the surface. Fig. 1(c) presents the typical SEM image on the co-implanted Si followed by 800 ℃ annealing. It can be clearly seen that besides the irregular craters, the surface morphology like the ruptured blisters has also been found (e.g. marked by white arrow). Further increasing the annealing temperature to 1000 ℃ leads to occurrence of three

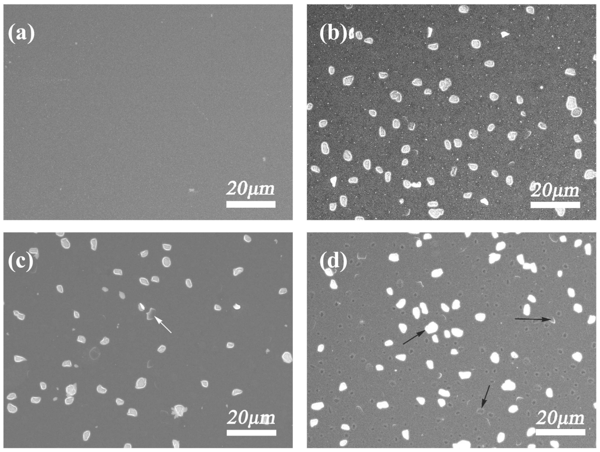

Fig. 1. Typical SEM images taken on various implanted and annealed Si. (a) He-only implantation after 1000 ℃ annealing; (b) He and H co-implantation after 600 ℃ annealing; (c) He and H co-implantation after 800 ℃ annealing; (d) He and H co-implantation after 1000 ℃ annealing.

kinds of surface characteristics, including blistering, exfoliation and the rupturing of blisters (marked by black arrows), as shown in Fig. 1(d).

The three-dimensional structures and thickness of the exfoliated surface have been characterized by using AFM for He and H co-implanted Si after annealing at 800 ℃. The typical results are shown in Fig. 2. As compared with SEM observations, AFM images can present more clear information on Si surface morphologies. For 800 ℃ annealing (Fig. 2(a)), successive implantation of He and H ions leads to formation of both surface blisters and irregular craters on Si surface. In order to acquire the quantitative information on the height of blisters and the depth of the craters, AFM measurements has also been performed for one of the blisters or craters (marked with black arrows in Fig. 2(a)). The obtained results are shown in Fig. 2(b). It is obvious that the depth of the crater is about 474.0 nm, which is quite close to the R_p ($\sim$ 0.5μm) of H ions according to TRIM simulations[8]. Therefore, it can be concluded that surface exfoliation is mainly achieved at the sample depth corresponding to the R_p of H ions.

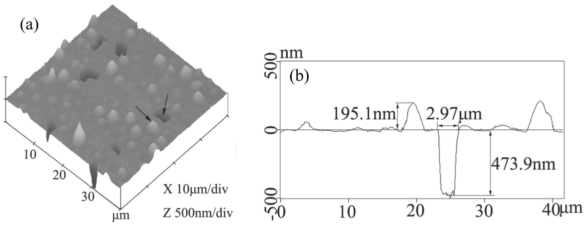

Fig. 2. (a) AFM images and (b) the related results obtained on Si co-implanted with 5 × 10^{16} cm^{-2}, 160 keV He and 1×10^{16} cm^{-2}, 40 keV H ions followed by annealing at 800 ℃.

In order to understand the possible mechanisms of surface exfoliation involved in He and H co-implanted Si, XTEM observations have been carried out for some of the implanted and annealed Si samples. The typical XTEM images are presented in Fig. 3. 160 keV He ion implantation into Si followed by 800 ℃ annealing creates a well-defined defect band, which is centered at a depth corresponding to the range of He ions ($\sim$ 1.0 μm). The band is mainly made up of the isolated cavities and a few dislocations, as shown in Fig. 3(a). Detailed measurements reveal that the cavities in the band have diameters ranging from 4.0 to 25.0 nm with a mean value of about 11.0 nm. For the case of 40 keV H ion implantation followed by 800 ℃ annealing (Fig. 3(b)), XTEM observations only reveal some dislocation loop-like defects. No cavities can be clearly seen. However, for Si co-implanted with both He and H ions and after 800 ℃ annealing, two defect bands have been found, as shown in Fig. 3(c). The shallower band is mainly located at the depth of about 0.5 μm from the surface, which corresponds to 40 keV H ion implantation. The band mainly consists of a few large cavities (size around 90 nm) and cracks surrounded by a great amount of large dislocations. The deeper defect band is mainly distributed at a depth of about 1.0 μm from the sample surface, which results from 160 keV He ion implantation. Close view of this defect band, one can clearly see that the band is mainly made up of cavities together with a few large dislocations, as shown in Fig. 3(d). Detailed measurements show that the cavities have an average diameter of about 6.0 nm. Therefore, as compared with He or H only implanted Si, the sequential implantation of He and H ions into Si has significant effects on thermal growth of both He and H-implant-induced defects during subsequent annealing. It seems to promote the growth of cavities and dislocations in the H-implanted region accompanied by suppressing thermal growth of cavities in the He-implanted region. Such effect plays very important roles in the observed surface blistering or exfoliation.

It is well-known that high fluence H or He ion implantation into Si followed by thermal annealing could lead to formation of bubbles or cavities around ion range[9, 10]. The formation and growth of bubbles or cavities are closely related to the implanted gas atoms and implantation induced vacancy-like defects (mainly divacancies). The thermal growth of bubbles is usually via either Ostwald ripening[11] or migration and coalescence processes[12]. If the implantation fluence is high enough, a narrowly confined layer of cavities or cracks could be formed, which allows the implanted gas to segregate into them upon annealing, and lead to growth and an eventual intersection of the cavities or cracks to form two continuous internal surface. The final exfoliation results from the formation of such two continuous internal surfaces together with a large gas pressure[6, 13].

As confirmed by XTEM observations, although 160 keV He ion implantation together with high temperature annealing gives rise to a well-defined cavity band in Si region close to the range of ions (Fig. 3(a)), such cavities are mainly in smaller size and dispersedly distributed. No intersection of such cavities can be achieved. Meanwhile, for single H ion implantation, subsequent high temperature only induces creation of a few dislocations situated at the end of H ion ranges (Fig. 3(b)). Therefore, no surface exfoliation or even blistering could be expected in such cases. However, for Si sequentially implanted with He and H ions, both He and H defect bands undergo significant changes during subsequent annealing. According to XTEM results (Fig. 3(c) and Fig. 3(d)), the presence of He ion implantation promotes the growth of cavities and a large dislocations in H-implanted region, while the presence of H implantation seems to suppress the growth of He cavities. Such effects could be attributed to interactions of both gas atoms and implantation induced defects, as discussed in the following.

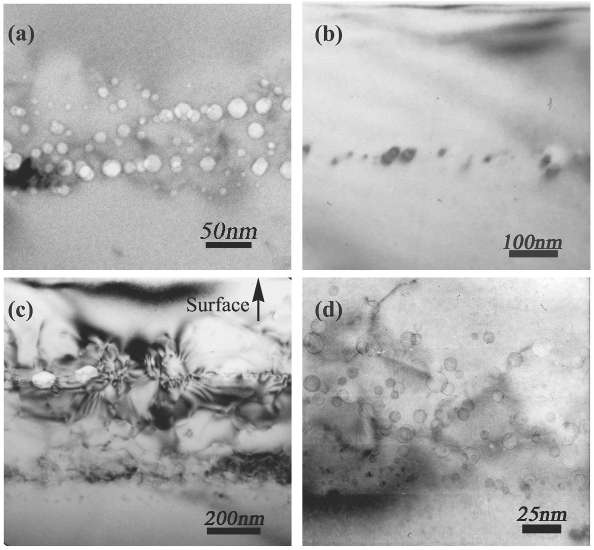

Fig. 3. XTEM images showing the defect microstructures in various implanted Si after 800 ℃ annealing (a) He ion implantation; (b) H ion implantation; (c) He and H ion co-implantation; (d) close view of the He defect band shown in (c).

It has been well recognized that both H and He ion implantation could introduce the gas atoms and create various defects (mainly vacancy-like defects) along the ion range. Strong physical interactions (gas coalescence, pressure and fracture) and chemical interactions (bond breaking, internal surface passivation) could occur between such gas atoms and defects, which depends strongly on the thermal budget[14]. Such interactions may significantly affect the thermal evolution of defects in different regions. In our case, although 160 keV He and 40 keV H ions were implanted into the same Si, the induced defects are distributed separately due to the different penetrating depths. Thus, there should be more vacancy-like defects existing in H-implanted region, as compared with H only implantation. Meanwhile, the pre-implantation of He could also introduce certain amount of He gas atoms in H-implanted region. The presence of He atoms in H region could assist in stabilizing vacancy-like defects in such a region. Since the formation and growth of bubbles are mainly via exchanging both vacancy-like defects and gas atoms, the introduction of additional vacancy-like defects and gas atoms in H-implanted region should promote cavity growth. Moreover, thermal annealing could give rise to desorption of He atoms from He-induced bubbles and thus diffusion to the sample surface[15]. During diffusion processes, these He atoms can be recaptured by H defects, leading to further pressure increase in bubbles there and thus a large strain field around. In fact, the presence of a large stain field around the H bubbles or cavities has been demonstrated by XTEM observations (Fig. 3(c)). As a result of the size increase of H bubbles, the average size of He bubbles reduces remarkably owing to loss of vacancy-like defects and He atoms in He-implanted region, which can be seen in Fig. 3(d). Therefore, as mentioned above, the large cavities, narrow cracks in combination with strong gas pressure could eventually induce the surface blistering and even exfoliation at the projected range of H ions. In fact, the AFM observations have demonstrated that the thickness of exfoliated Si layer is about 474 nm, which is quite consistent with the depth of H cavity band.

4 Conclusions

In conclusion, by using techniques of SEM and AFM, we have studied the thermal evolution of surface damage on Si implanted by 5×10^{16} cm^{-2}, 160 keV He ions and/or 1×10^{16} cm^{-2}, 40 keV H ions. Surface blistering and exfoliation have only been observed on He and H co-implanted Si, which show strong dependence on annealing temperature. The surface exfoliation has been found to occur at a depth corresponding to the range of H ions (~ 0.5 μm). According to XTEM observations, the observed surface exfoliation is mainly owing to the promotion effects of He implantation on thermal growth of H-implant induced defects.

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