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Surface damage versus defect microstructures in He and H ion co-implanted Si_3N_4/Si

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ABSTRACT

Cz *n*-type Si (100) wafers with a top Si_3N_4 layer of about 170 nm in thickness were sequentially implanted with 40 keV He ions at a fluence of $5 \times 10^{16}/cm^2$ and 35 keV H ions at fluences of 1×10^{15}, 5×10^{15} and $1 \times 10^{16}/cm^2$, respectively. Creation and evolution of surface damage as well as micro-defects have been studied. Our results clearly show that production of surface damage depends strongly on both the H implant fluence and annealing temperature. Only blistering or localized exfoliation of the top Si_3N_4 layer has been observed for post H implantation at fluences of 1×10^{15} and $5 \times 10^{15}/cm^2$ upon 800 °C annealing. However, serious surface exfoliation has been found for the $1 \times 10^{16}/cm^2$ H co-implanted samples after annealing at 450 °C and above. The exfoliation occurs at a depth of about 360 nm from the surface, which is obviously larger than the He or H ion range. Moreover, the exfoliated craters show clear two-step structures. Cross-sectional transmission electron microscopy (XTEM) observations reveal formation of micro-cracks in Si bulk and along the original interface, which is mainly responsible for the observed surface phenomena. The formation mechanism of micro-cracks has been discussed in combination of He and H implant-induced defects, impurities as well as their interactions upon annealing.

1. Introduction

Light gas ion implantation into semiconductor materials at sufficiently high fluences could induce a series of phenomena, including bubble formation, surface blistering, localized exfoliation and even layer splitting [1–3]. Such phenomena have wide applications in modern semiconductor technology. Especially, based on the surface blistering or exfoliation observed for H ion implanted Si, Bruel [4] has proposed a technique named "smart-cut" to fabricate high quality silicon-on-insulator (SOI) structures. Nowadays, this technique has been extended to synthesize various heterostructures based on gas ion implantation combined with bonding method [5,6]. Gas ion (He or H) induced layer transfer of semiconductor materials generally needs high fluence and thermal treatment temperature. Thus, many efforts have been recently made to explore the possible ways to reduce both the applied fluence and thermal treatment temperature necessary for achieving layer splitting. Actually, it has been demonstrated that sequential implantation of H ions with other ions, such as He, D, B, etc., could enhance the surface blistering and/or exfoliation [7–9]. As for the involved mechanism, it is commonly recognized that the formation of micrometer sized cracks together with the large pressure inside them is mainly responsible for occurrence of various surface phenomena [3,10,11]. Nevertheless, more impurities and defects could be introduced into solids during successive implantation of different ions, it thus makes much more complicated for understanding of the processes concerning evolution of surface damage as well as micro-defects. Therefore, the formation mechanism of pressurized micro-cracks in semiconductors under co-implantation of H with other ions is still not well understood.

Up to now, gas ion implantation-induced surface damage together with the involved mechanism has been extensively studied for Si and Si covered with a thermal oxidation layer. However, few of them have been contributed to Si covered with a top Si_3N_4 layer. Si_3N_4 is an important insulating material and widely used in the modern microelectronics and other fields. The presence of Si_3N_4 layer on the Si surface could affect the creation of defects under ion implantation. Actually, our earlier research has clearly shown that the presence of Si_3N_4 layer on Si surface could suppress the growth of He bubbles and affect the He desorption behavior in He implanted Si [12]. Moreover, by using successive implantation of $5 \times 10^{16}/cm^2$, 160 keV He and $1 \times 10^{16}/cm^2$, 110 keV H ions into the same region of Si covered with Si_3N_4 layer (i.e. Si_3N_4/Si system), surface exfoliations of the Si_3N_4 layer and implanted Si have been well separated upon annealing, [13]. The exfoliation of Si_3N_4 layer has been demonstrated to be mainly related to the large strain created around the original interface during growth of pressurized cracks in Si together with the large difference in the mechanical

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properties of Si_3N_4 and Si. Such phenomenon could be useful to fabrication of heterostructures by transfer of Si_3N_4 layer from Si to other substrates based on gas ion implantation combined with bonding method. Therefore, in order to promote its applications, it is necessary to understand the fundamental processes involved in the Si_3N_4/Si system under gas ion implantation at different conditions.

In the present study, we report the main results concerning surface damage as well as defect microstructures in Si_3N_4/Si samples sequentially implanted with He and H ions at relatively low energies (40 and 35 keV for He and H, respectively). The present results clearly shown that reducing the energies of He and H ions could lead to the localized exfoliation of the whole implanted layer, which is quite different from that that observed in 160 keV He and 110 keV H co-implanted Si_3N_4/Si system at the same fluence, i.e. $5 \times 10^{16}/cm^2$ for He ions and $1 \times 10^{16}/cm^2$ for H ions. Based on the XTEM results, the possible mechanism involved in the surface damage has been tentatively discussed and presented in view of interactions of implantation induced defects and impurities during implantation and annealing processes.

2. Experimental details

The study was performed by using (100)-oriented *n*-type Czochralski grown Si wafers (resistivity $\rho \sim 3–7\ \Omega$ cm) covered with a top Si_3N_4 layer of about 170 nm in thickness (labeled as Si_3N_4/Si). The Si_3N_4 layer was thermally grown on Si surface in a NH_3 ambient at 1150 °C for 3 h. The wafers were firstly implanted at room temperature with 40 keV He ions up to a fluence of $5 \times 10^{16}/cm^2$ in order to create a band of bubbles in Si bulk around the He range, and some of them were then subjected to implantation of 35 keV H ions at fluences of 1×10^{15}, 5×10^{15} and $1 \times 10^{16}/cm^2$, respectively. Simulations from SRIM 2008 code [14] show that implantation of 40 keV He and 35 keV H ions into Si_3N_4/Si gives a nearly coincident mean projected range (R_p) of about 260 nm. After implantation, the wafers were cut into small pieces for various analyzes. Before analyzes, the sample pieces were subjected to furnace annealing in the temperature range of 300–800 °C. The annealing was carried out in a flow of nitrogen gas for 1 h.

Scanning electron microscopy (SEM) was used to characterize surface morphology. The SEM images were taken by using a JEOL model JSM 6700F microscopy with a field-emission gun, operating at 10 kV. Atomic force microscopy (AFM) measurements were performed to quantitatively evaluate the thickness of exfoliated layer as well as the sizes and heights of blisters. Moreover, cross-sectional transmission electron microscopy (XTEM) investigations were selectively conducted for some of the implanted and annealed samples to reveal defect microstructures. Before XTEM investigations, the samples were cut, glued, and then thinned using mechanical polishing and ion milling. XTEM images were taken at 200 kV with a JEOL 2010 microscopy.

3. Results

SEM and AFM investigations have been carried out for the Si_3N_4/Si samples singly implanted with 40 keV He ions or 35 keV H ions. No surface damage has been clearly detected even after 800 °C annealing for 1 h. However, under sequential implantation of 40 keV He and 35 keV H ions, blistering and/or localized surface exfoliation of Si_3N_4/Si have been well observed upon annealing, which show dependence on the applied fluence of H ions. For H co-implantation at a fluence of 1×10^{15} or $5 \times 10^{15}/cm^2$, surface damage has only been observed after 800 °C annealing, which mainly consists of blisters or craters resulting from the ruptured blisters. As an example, Fig. 1(a) gives the AFM image showing formation of blisters and craters on Si_3N_4/Si surface sequentially implanted with He ions at a fluence of $5 \times 10^{16}/cm^2$ and H ions at a fluence of $5 \times 10^{15}/cm^2$, and followed by 800 °C annealing. Quantitative measurement of one crater has revealed that the exfoliation occurs at a depth of about 171 nm, as shown in Fig. 1 (b), which is in good agreement with thickness of the top Si_3N_4 layer. Thus, the result clearly suggests that the craters directly result from the localized exfoliation of the top Si_3N_4 layer.

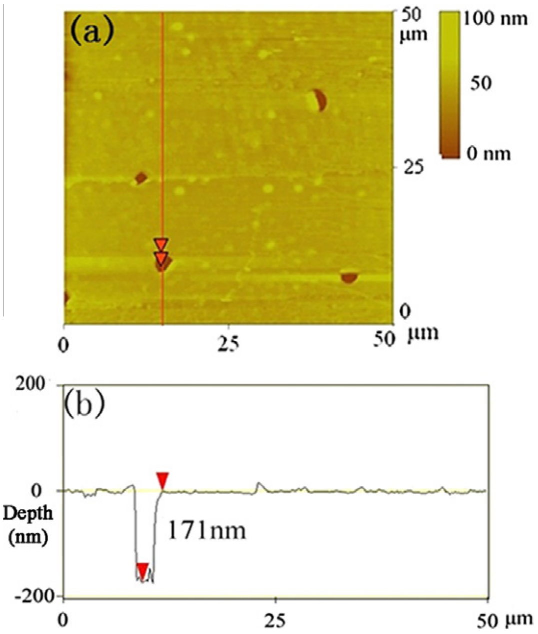

Fig. 1. AFM results of the Si_3N_4/Si sample sequentially implanted with 40 keV He ions at a fluence of $5 \times 10^{16}/cm^2$ and 35 keV H ions at a fluence of $5 \times 10^{15}/cm^2$, and followed by annealing at 800 °C for 1 h, (a) surface topography, and (b) exfoliation depth of one crater.

Fig. 2 presents the SEM images taken on the 40 keV He ion pre-implanted Si_3N_4/Si samples followed by 35 keV H ions at a fluence $1 \times 10^{16}/cm^2$ and after annealing at different temperatures. At 450 °C, a few of craters begin to appear on the sample surface, indicating occurrence of the localized exfoliation, as shown in Fig. 2(a). Increasing annealing temperature to 500 °C leads to formation of more craters on the sample surface (Fig. 2(b)), suggesting that the degree of surface exfoliation increases. By close view of the craters, one can find that the craters usually show two step structures (see one of the craters indicated by white arrow, as an example), which will be discussed in combination with the AFM results later. As the annealing increases to 600 °C, craters in larger size are usually formed owing to overlapping of the exfoliation region, as shown in Fig. 2(c). Further increase of annealing temperatures up to 700 and 800 °C gives rise to no significant change on the surface morphologies, suggesting that the degree of surface exfoliation may saturate at temperatures above 600 °C. As an example, Fig. 2(d) presents the typical SEM image taken on the He and H co-implanted Si_3N_4/Si after 800 °C annealing.

Fig. 3 gives the AFM results acquired on the He pre-implanted Si_3N_4/Si sample sequentially implanted with H ions at a fluence of $1 \times 10^{16}/cm^2$, and followed by annealing at 800 °C. One can see from Fig. 3(a) that besides formation of craters, blisters could also be observed throughout the sample surface. The craters usually show two step structures which is in good agreement with that observed by SEM. Moreover, as shown in Fig. 3(b), quantitative measurement of one crater show that the height of the shallow step is about 160 nm, quite close to the thickness of the top Si_3N_4 layer, while that of the deep step is evaluated to be about