

## Impurity Dependence of Exfoliation in Proton-implanted Silicon

プロトン剥離現象における不純物依存性

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**Abstract:** Silicon wafers implanted at adequate conditions (such as room temperature, 80keV,  $5 \times 10^{16}$  H·cm<sup>-2</sup>) induce exfoliation phenomena after 500°C heating. In this study, for making of the influence of impurity clear, the damaged layer in the hydrogen-implanted silicon was observed with cross sectional transmission electron microscopy. And the exfoliated surface was observed by optical-microscopy and atomic force microscopy (AFM) The behaviors of three types of specimens (p++, p and n type, respectively) were compared by carrying out in furnace annealing. The quantity of defect is proportional to the concentration of dopant, and it is interesting to note that p++ and p type had a sharp peak in the depth profile, but n type had a broad peak and a second peak in a shallow region. The number of exfoliation was proportional to the dopant concentration. So was the total exfoliated area (ratio of area). The other hand, the average area of exfoliation was biggest in n type silicon and was in smallest in p++ type. It seems that the average area was not affected by dopant, while it was affected by the depth of exfoliation.

### 1. Introduction

Both the diffusion and the reactivity of hydrogen in solid materials remain a matter of lasting interest in material engineering. Hydrogen implantation in silicon has been applied to local control of recombination life time, gettering of metallic impurities and fabrication of silicon-on-insulator (SOI) wafers. In the SOI fabrication technology, the hydrogen exfoliation method introduced by Bruel<sup>[1,2]</sup> has advantages of greater uniformity of thickness of the surface layer and crystal quality than other techniques. This method involves a micro slicing process of silicon by high dose hydrogen implantation. This slicing process is usable as a valuable new micro slicing tool for such hard materials as SiC or diamond, and has also been used to obtain the transfer of thin layers from thick substrates on to different substrates<sup>[3,4]</sup>. Although this unique and useful process has been extensively developed in industrial applications during the past few years, the fundamental phenomenon and the

underlying mechanism are still not completely understood. Generally, silicon wafers implanted at room temperature (80keV,  $5 \times 10^{16}$  H·cm<sup>-2</sup>) induce exfoliation phenomena after 500°C heating. However, silicon wafers processed under some certain conditions, example for n-type silicon<sup>[5]</sup> or co-implantation<sup>[6]</sup>, do not induce exfoliation even at above 500°C heating.

In this work, we study the effect of the influence of the impurity in silicon. The damaged-layer induced in hydrogen-implanted silicon was observed with cross sectional transmission electron microscopy (XTEM) and the exfoliated surface was observed by optical-microscopy and atomic force microscopy (AFM)

### 2. Experiments

Table 1. Dopant properties of prepared silicon wafer.

Sample identifier	Dopant	Resistivity [Ω cm]	Estimated concentration [cm <sup>-3</sup> ]
p <sup>++</sup>	B	0.014	$4 \times 10^{18}$
p	B	6~8	$2 \times 10^{15}$
n	P	1~2	$3 \times 10^{15}$

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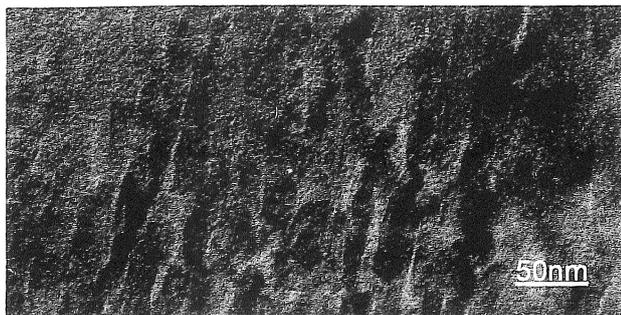


FIG. 1 XTEM micrograph of the damaged layer induced by proton implantation in p-type silicon.

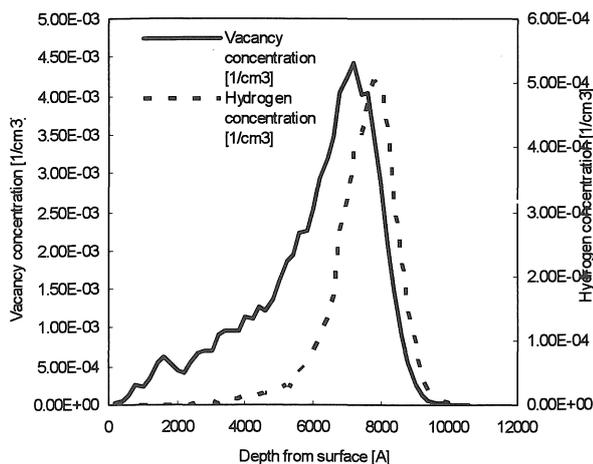
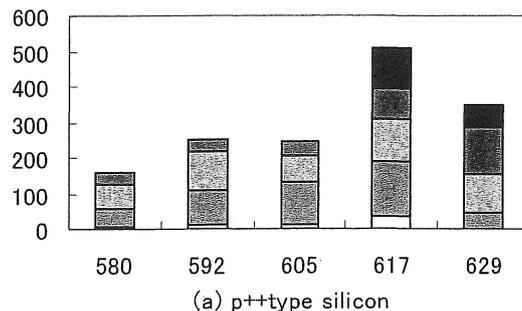
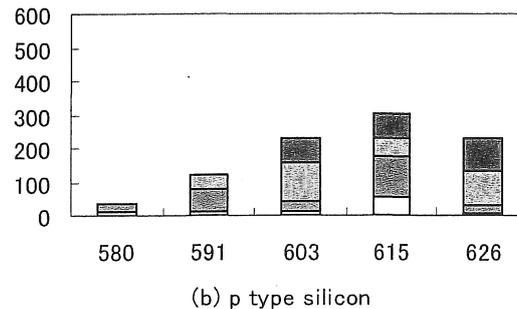


Fig.2 Hydrogen and Vacancy concentrations obtained by TRIM code simulation at 80keV hydrogen implanted in silicon

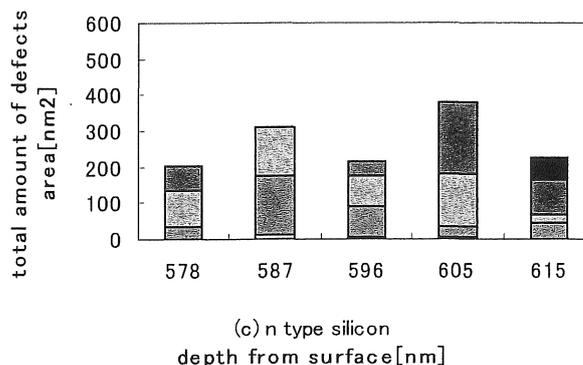
As shown in Table 1, five type samples were prepared. These are cut from  $\langle 100 \rangle$  Cz-grown silicon wafer. Hydrogen implantations were carried out with 80keV  $H^+$  ions at room temperature. The wafers were oriented  $7^\circ$  off normal to the incident ion beam in order to minimize channeling effects. The hydrogen dose was  $5.0 \times 10^{16}$  ions/cm<sup>2</sup>, this dose is high enough dose quantity required to cause blistering on the surface of a wafer or exfoliation in the bonded wafer. The damaged layers induced by hydrogen implantations were examined by XTEM using a JEOL JEM-2010 with a [011] 200kV electron beam. The cross-sectional specimens were prepared by a combination of mechanical polishing and ion thinning with 3keV  $Ar^+$  ions. These preparation processes involved gluing small pieces from a single sample face-to-face using epoxy cement and mounting into a  $\phi$  3mm brass pipe to strengthen the specimen. In order to confirm the appearance of blistering or exfoliation, the furnace annealing was carried out under  $N_2$  gas at 500°C for 30 minutes or 700°C for 60 minutes.



(a) p++ type silicon



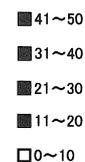
(b) p type silicon



(c) n type silicon

depth from surface [nm]

FIG. 3 Depth distributions of estimated defects area in damaged layer obtained by high resolution XTEM. (a): p++ type silicon, (b): p type silicon, (c): n type silicon



Defect length

The properties of blistering or exfoliation were examined by conventional optical microscopy and AFM

### 3. Results and discussion

Figure 1 shows the XTEM micrograph of a damaged layer induced by hydrogen implantation with the  $5.0 \times 10^{16}$  Hcm-2 doses. The depth of layer approximately 0.7 $\mu$ m below the surface correspond to the depth of peak of vacancy and hydrogen concentration obtained by simulation as shown in Fig.2. The contrast of damaged layer is formed by lattice distortion as  $\{100\}$  platelets and  $\{111\}$

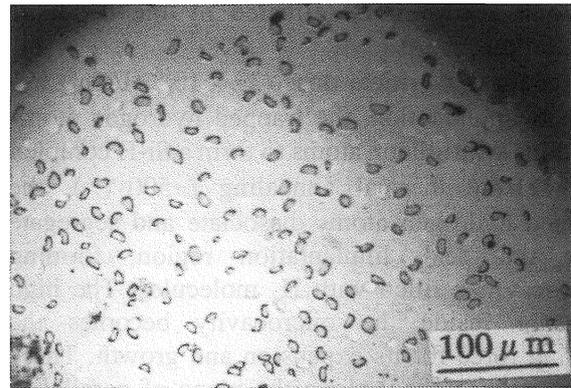
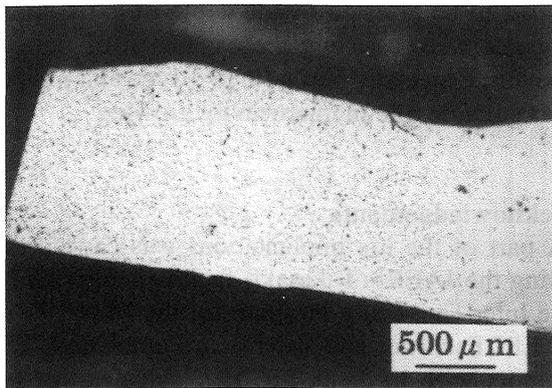


FIG. 4. Surface blistering observed with optical microscopy. (p type silicon surface implanted  $5.0 \times 10^{16}$  ions/cm<sup>2</sup> proton and 500°C annealing)

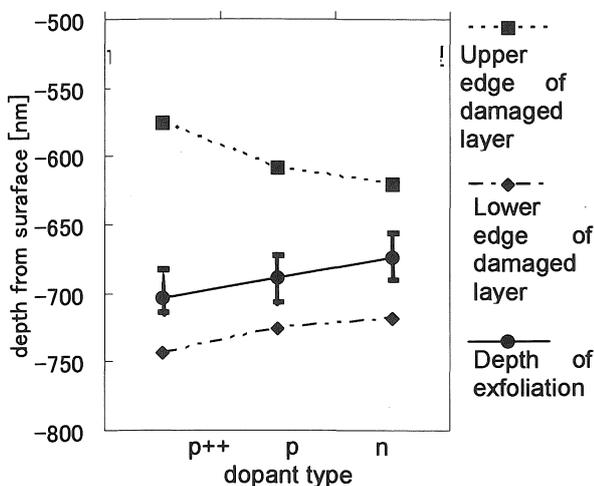


FIG. 5. The depth of exfoliated location and the damaged layer.

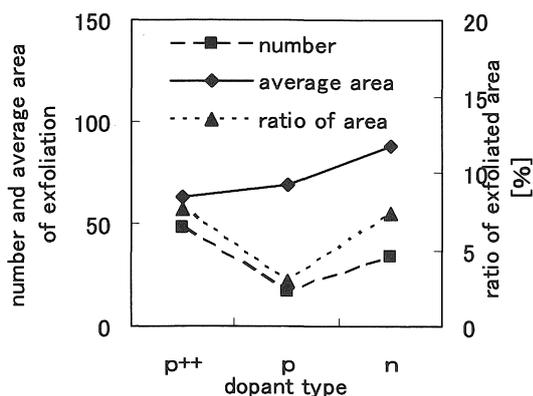


FIG. 6. number, average area and ratio of exfoliated crater on the surface of proton implanted silicon.

platelets. Accordingly, (100) and (111) defects are classified into five groups depending on the depth, and their size and density are investigated respectively in detail. The five areas located at equal intervals in the depth direction in the damaged layer

are all 20nm×20nm. Defect sizes are defined by calculating the average diameter of the defects in each area in the high-resolution micrograph taken along the [110] direction; defect densities are defined as the number of defects counted in each area in the micrograph. Figure 3 shows the defects area distribution calculated from defect densities and defect diameter length. The p++ type silicon had much defects than other type, Comparing the p type and n type silicon, n type has much defect than p type. These results mean quantity of defect is proportional to the concentration of dopant. And, it is interesting to note that p++ and p type had a sharp peak in the depth profile, but n type had a broad peak and a second peak in a shallow region.

Three types (p++, p and n wafers) were given a furnace annealing at 500°C for 30minutes in N<sub>2</sub> gas ambient. Exfoliation or blistering was observed on the implanted surface as shown in Figure 4. Figure 5 shows the relation of the depth of damaged layer obtained by XTEM and depth of exfoliation obtained by AFM. As mentioned above, p++ type had widest damaged layer and n type had shallowest one. It seemed that, in p++ type and p types, the depth of exfoliation corresponded to the hydrogen concentration peak in Figure 2. But the depth of exfoliation in n type was in shallower location in the damaged layer than others and the depth seemed to correspond to the depth of peak of vacancy concentration. Numbers of exfoliation events and areas of exfoliated crater on the surface of implanted samples were measured from micrograph obtained by optical microscopy as shown in Figure 6. The number of exfoliation was proportional to the dopant concentration. So was the total exfoliated area (ratio of area). The other hand, the average area of exfoliation was biggest in n type silicon and was in smallest in p++ type. It seems that the average area was not affected by dopant,

while it seems to be affected by the depth of exfoliation.

After these a critical dose ( $3 \sim \times 10^{16}$  H/cm<sup>2</sup>) hydrogen implantation, the trapped hydrogen atoms combine with silicon atoms to form a Si-H complex [7,8]. During thermal annealing ( $\sim 500$  °C), the trapped hydrogen atoms dissociate and segregate near the peak implantation region, forming microcavities filled with H<sub>2</sub> molecules. The high pressure inside the microcavity becomes the driving force for its expansion and growth. These microcavities grow along the plane of parallel to the surface during annealing. Afterward, all the microcavities are linked together; blistering or exfoliation is obtained in the Si wafer. In these actions, dopants were assisting the expansion of defects growth. Especially boron (in p++ type and p type) was suitable role in microcavity growing.

#### 4. Concluding remarks

Quantity of defect was proportional to the concentration of dopant. p++ and p type had a sharp peak in the depth profile, but n type had a broad peak and a second peak in a shallow region. p++ type had widest damaged layer and n type had shallowest one. In p++ type and p types, the depth of exfoliation corresponded to the hydrogen concentration peak, while the depth of exfoliation in n type was in shallower location in the damaged layer than others and the depth seemed to correspond to the depth of peak of vacancy concentration. The number of exfoliation was also proportional to the dopant concentration. The other hand, the average area of exfoliation was biggest in n type silicon and was in smallest in p++ type the average area was not affected by dopant, while it

seems to be affected by the depth of exfoliation. dopants were assisting the expansion of defects growth. Especially boron (in p++ type and p type) was suitable role in microcavity growing.

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