СЕКЦИЯ 2. РАДИАЦИОННЫЕ ЭФФЕКТЫ В ТВЕРДОМ ТЕЛЕ SECTION 2. RADIATION EFFECTS IN SOLIDS

SOME FEATURES OF THE STRUCURE OF SILICON SINGLE CRYSTALS IRRADIATED BY LARGE FLUENCES OF FAST LIGHT IONS OF GASES

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Structural study of silicon irradiated by large fluences of light gas ions has shown how the degree of damage in Si, both in the ion passage region and in the braking area, increases and become more complicated with increasing energy and mass of ions. For hydrogen ions the stress line in the braking region is narrowest and its width grows almost linearly with energy. For ions of deuterium and helium, this dependence is weaker and deviates from linear. Additional stress lines depended on the value of ion current (intensity of irradiation) were observed in the pictures of selective etching. For current densities up to $0.45 \ \mu A/cm^2$, the second etched line of stresses appeared at the distance equal to the twice length of the run of protons from the surface. With an increase in the current density up to $1 \ \mu A/cm^2$, this line was not observed, at $3 \ \mu A/cm^2$ the part of Si (with thickness that corresponds to the ion path depth) in the irradiation process was exfoliated from main volume.

The number of extra etched lines is maximal for helium and depends on the temperature of irradiation. Increase in the temperature (over 100 °C) leads to disappearance of additional stress lines.

Detail study with gradual selective etching (in the step of 30 μ m) from an irradiated surface has shown the distribution of structural defects in the plane of the proton braking region of silicon, irradiated with fluence 10¹⁷ cm⁻² at $T \le 100$ °C and annealed for 0.5 h at 580 °C. With deepening into Si volume the irradiated region associated with radiation defects was gradually decreasing and completely disappeared at the depth of 150 μ m behind the braking line. At the depth of nearly 35 μ m behind the braking line, the growth layers of matrix Si were detected.

Keywords: silicon; structure; light ions of gasses; irradiation.

Introduction

Ion irradiation is so widely spread in electronic technology that it is difficult even to outline all possible applications [1]. Today, methods of modifying by ion beams the properties of materials on a nanoscale level are sufficiently developed and have theoretical justification. The size, shape, and location of regions with properties different from those of the matrix depend on the irradiation conditions: the energy and mass of the ions, the fluence and intensity, the irradiation temperature and the characteristics of the crystals [2]. Despite on the advantages of planar technology some moments of ion interaction with crystals have not yet been explained. These concerns, first of all, the features of defect formation due to large fluences of irradiation, when disordering regions are overlapped and the collective interaction between the defects appears.

The effect of high-energy (tens of MeV) light ions on the structure of Si is under investigation. First of all, except hydrogen, there were ions of deuterium and helium with energies from the less studied range. Usage of particles which energies and masses differ twice was convenient for the comparison of structural changes, induced by different ions. The second, all ions under research are gasses. And finally, the obtained experimental data concerning the features of material structure under irradiation with large fluences can be useful when building the adequate theory of interaction of the high-energy ions with crystal. Therefore, it is necessary to conduct researches in this direction to improve the understanding of these processes and to control of them further.

Experimental results and discussion

The effects of fast hydrogen ions on the properties of silicon single crystals were studied at our institute for several years. The infrared spectroscopy and structural methods have been used to investigate the types of radiation damage and to show the features of defect formation in silicon exposed to hydrogen ions [3]. Further studies of silicon, irradiated with hydrogen, deuterium and helium ions, demonstrated the specificity of its optical and structural characteristics for large fluences [4].

It is known that ions, getting into the crystal during the irradiation process, lose energy due to scattering at the electronic subsystem (excitation and ionization) and nuclear, when the energy is transferred to nuclei of the matrix and radiation defects are formed (Frenkel pairs).

In the case of the interaction of light ions of gases with Si crystal, the value of energy, where nuclear losses dominate, is several keV [2] that is, a small part of the total ion energy goes to the formation of radiation defects, the rest goes to the excitation and ionization of silicon atoms. The main defects are generated when ions are slowed down, so, its creation is uneven along the particle track and has maximum at the end of the path. The average length of the run of high energetic particles in silicon was estimated in [5].

With irradiation of Si crystals by 6.8 MeV hydrogen ions and 27.2 MeV helium ions, the ion path length was about 360 μ m, and for 13.6 MeV ions of deuterium it was 780 μ m.

Our metallographic studies of the silicon irradiated by significant fluences of the light-gas ions that detect the distribution of defects along the direction of irradiation show that the greatest stresses of silicon lattice are observed in the region of ion braking (as predicted by the theory of interactions) [4]. However, for large fluences (exceeding 10¹⁶ cm⁻²), the layered distribution of stresses is observed both in the run region for the ions and beyond their braking lines. Thus, the irradiated crystal can be conventionally divided into three regions with different properties: run, braking, behind stopping line.

13-я Международная конференция «Взаимодействие излучений с твердым телом», 30 сентября - 3 октября 2019 г., Минск, Беларусь 13th International Conference "Interaction of Radiation with Solids", September 30 - October 3, 2019, Minsk, Belarus By means of X-ray topography and selective etching, it is shown how the degree of damage in Si, both in the ion passage region and in the braking area, increases and become more complicated with increasing energy and mass of ions More orderly and narrower lines of stress associated with defects were observed in silicon irradiated with hydrogen ions, and their number and location relative to the braking line depend on the fluence (intensity) of the ions.

The change of the width of Si lattice stress line in the braking area, depending on the energy and mass of particles, is shown in Fig. 1 (values of the stress line's width for proton energies of 0.32 MeV and 3.2 MeV were received from the analysis of [2]).



Fig. 1. Dependences of the width of Si lattice stress line in the braking area of ions on their energy (*a*) and mass (*b*). Points 1 and 2 are taken from [2]

One can see that for hydrogen ions the width of the stress line grows almost linearly with energy. For ions of deuterium and helium, this dependence weakens, deviating from a linear to a decreasing side. At the same time, in the images of the selective etching of the irradiated crystal, additional etched bands appear in silicon in the ion run region. It should be noted that the size of the regions in which the formation of «quasiperiodic» additional bands, parallel to the line of ion braking, appears to be approximately the same for hydrogen and helium ions and is equal 760–780 microns, but the number of lines increases with increasing mass of ions (two for hydrogen, against 7–8 for helium).

In general, the dependence of the occurrence of additional lines on the value of the ion current (that is, on the intensity of irradiation) was revealed. Thus, for protons with current densities $\leq 0.45 \ \mu A/cm^2$ at a distance equal to the twice length of the run of protons from the surface, a second etched line of stresses, parallel to the braking line, was observed. With an in-

crease in the current density of protons up to 1 µA/cm². this line was not observed. At a current density of 3 µA/cm² under similar cooling conditions (the sample was cooled only by the running water) the part of silicon (with a thickness that corresponds to the path depth) at the passage of ions was exfoliated from the main volume. This indicates that temperature in the braking region of silicon reached a value sufficient to release hydrogen from Si-H bonds and to fill vacancy complexes in the process of irradiation. According to [3], hydrogen was released from Si-H bonds at $T \approx 600$ °C, and formed bubbles that grew and bursted to form voids (pores), the size and density of which were determined by the energy of protons and the intensity of irradiation. At small current densities of ions of 0.25–0.45 µA/cm², the irradiated part of silicon was exfoliated only after the annealing of irradiated silicon at 600 °C, that is, the effect was determined by the temperature of the annealing of the sample.

The results obtained for the protons of various energies indicate that the features of the silicon structure should be determined not only by the temperature of irradiation or annealing, but also by its distribution over the sample in the process of irradiation, since it can affect on the defect structure in all areas (run, braking, behind the braking part).

With gradual etching (in the step of 30 microns) of silicon from the irradiated surface, the data for the distribution of structural defects in the plane of the proton braking region were obtained by X-ray topography and selective etching methods. The Si structure (previously irradiated with fluence 10^{17} cm⁻² at $T \le 100$ °C and annealed for 0.5 hours at the temperature of 580 °C) was studied. Under these conditions hydrogen begins to free from the Si-H bonds [3]. These results help to understand the possible behavior of hydrogen in Si at elevated temperature during irradiation of the sample.

Fig. 2 shows the X-ray topography of the silicon sample taken in the irradiation plane at the depth of 315-510 microns from the irradiated surface. Given that the braking line of 6.8 MeV protons is ~ 360 um. the data are obtained for 45 µm (a) and 10 µm (b) to the braking line, as well as $35 \mu m$ (c) and $150 \mu m$ (d) behind the braking line. It can be seen that with the dippening into the Si volume, the irradiated by the protons area associated with radiation defects is gradually decreasing and completely disappears at a depth of ~ 510 µm from the irradiated surface (nearly 150 µm behind the braking line). The layers of growth of the matrix silicon were detected at the depth of about 395 µm that is nearly 35 µm behind the proton braking line (Fig. 2, c). In our opinion, these growth layers were decorated with hydrogen in the presence of oxygen, carbon and other growth impurities and defects. Such layers were not detected with subsequent etching at the depth more than ~ 150 microns behind the braking line of the protons (Fig. 2, d).

It is believed that in the process of irradiation by protons, the maximum number of defects is accumulated in silicon in the braking area and its wavy profile is formed, which extends from the braking line to the depth of silicon. The process is accompanied by the interaction of defects with impurities, the formation of complexes and, as a result, the propagation of the concentration profile of defects and its damping.

As it was obtained earlier [6] the braking lines of

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light-gas ions consist primarily of voids, the size and concentration of which and, accordingly, the width of the etched lines, are determined by the energy, the mass of the ions, and irradiation temperature. The increase in the temperature (more than 100 °C) leads to the disappearance of additional stress lines and, possibly, to a more even distribution of defects in both regions in relation to the line of ion braking.



Fig. 2. Series of X-ray topograms of the Si structure in the irradiation plane after gradual etching at the depth of about 315–510 μ m from the irradiation surface (near the braking area (*a*, *b*) and behind it (*c*, *d*)) after annealing at 580 °C for 0.5 h of the irradiated silicon by protons with fluence of $\Phi = 10^{17}$ cm⁻²

Micrographs of the irradiated silicon, which display changes in the structure near the braking line of protons at the corresponding depths, showed that the run region of the protons in silicon remains crystalline with detected typical defects, while modifying behind braking line. With the growth of the mass of ions, the structure of silicon in the region of run and braking is complicated, which is most clearly observed for helium.

The stress line of braking of helium ions (as seen at the significant increase [6]) was a strongly damaged region of silicon consisting of cavities (shells) of various sizes and shapes, etched as a continuous layer, and in the form of individual clusters, accompanied by the formed dislocation loops. Micrograph of run region for helium ions in silicon taken on the raster electron microscope showed that its structure is not monocrystalline, but fragmentary with the presence of ordered lines parallel to the braking line of helium ions.

After irradiation by deuterium ions, the braking band of ions (after selective etching) expanded and became more complicated closer to the center of the irradiated region. Near this band from the side of the unirradiated volume the packing defects appeared. These defects in the centre of the sample, where the flux density of the deuterium ions and, accordingly, the irradiation temperature was maximal intersected the braking band at almost the right angle. Such process is obviously associated with the presence of dislocations in the initial silicon, their movement and interaction with radiation defects during irradiation (under conditions of high temperature and large concentrations of radiation defects).

It was found that closer to the center of the sample the additional etched lines of stresses appeared, due to probably by intense heating. The etched lines of stresses did not occurred in the region of the silicon behind the braking band of deuterium ions, which may be connect with high energy release and, accordingly, with high temperature of the sample during irradiation.

Conclusions

It was established that for hydrogen ions the stress line in the braking region is narrowest and its width grows almost linearly with energy. For ions of deuterium and helium this dependence deviates from linear and in the pictures of the selective etching the additional etched bands appear.

It was found that the presence of dislocations in Si in the process of deuterium ion irradiation leads to their movement and intersection of the braking line due to the formation of stacking faults.

To summarize, it can be state that the structure of silicon irradiated by large fluences of the ions of gases with MeV energies undergoes significant changes in the region of ion braking, as well as on both sides of it (range of run and behind braking area), and the magnitude of the changes is determined by the mass and energy of the ions, as well as the temperature of irradiation. This applies in particular to the braking line, which consists of cavities, the size and concentration of which determines its width (the most complicated and the widest - for helium). The number of extra etched lines is also maximal for helium and depends on the temperature of irradiation. The increase in the temperature (over 100 °C) leads to the disappearance of additional stress lines and, possibly, to the more even distribution of defects in both regions in relation to the braking line of ions.

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