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# *X*-RAY DIFFRACTION EFFECTS IN THE CASE OF IMPLANTED SILICON SINGLE CRYSTALS

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The effects of *X*-ray diffraction by a layer of silicon implanted with a $10^{15}$ ions/cm$^2$ dose of 60-keV boron ions are studied. Use was made of the methods of *X*-ray automonochromatization by an oscillating slit and a two-crystals *X*-ray spectrometer. The reflectivity and lattice constant are found to be larger for the implanted layer, and the broadening of the diffraction curve by about three times for this layer shows that there are lattice defects in it.

The development of the implantation technique as a method of doping semiconductors gives rise to the need for determining the type of damage occurred during the implantation process in the crystal lattice. When they penetrate the target, the implanted ions lose energy due to Coulomb interactions with its atoms (elastic collisions) and interactions with the bound and free electrons in it (inelastic collisions). In silicon, in the case of implantation of the usually used impurities within the ion energy range up to several hundred keV, mainly collisions of the first kind are responsible for the formation of defects in the structure of the target [1, 3, 4, 7].

In the case of heavy ions the mean retardation path is short, namely, it barely exceeds the dimensions of the region in which atoms are knocked out avalanche-wise from the lattice site positions; the lattice defects are contained within one big cascade elongated in the direction of ion motion. Owing to the high activation energy for the recombination of knocked-on atoms and vacancies in silicon at room and lower temperatures, they do not annihilate as in the case of higher implantation temperatures. In the Si crystal a region of amorphous structure, having dimensions of several tens of angstroms, becomes formed around each ion path. There are quite a number of point defects (bi-vacancies and intersti-

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tial atoms captured by impurities) in the vicinity of such an amorphous region; this is due to the diffusion of defects from the cascade region.

With increasing dose of bombarding ions the multitude of amorphous regions formed by the individual ions increases until an implanted layer of amorphous structure is obtained at the semiconductor surface when the dose reaches $10^{14}$ to $10^{16}$ ions/cm$^2$, depending on the ion mass. This amorphous structure may be visualized as being formed from the structure of an ideal Si single crystal by rotations of its tetrahedrons and a change in the Si–Si spacing from 2.35 Å for the ideal crystal to 2.45 Å for the amorphous layer [2].

The structure of amorphous layer formed by the implantation of heavy ions has been the subject of numerous researches which made use of electron microscopes, EPR, absorption in the infrared, and backscattering of protons and alpha particles [3, 4].

In the case of light ions, such as boron ions, the mean retardation path in a silicon crystal is much longer than the dimensions of the regions of avalanches of atoms knocked out of their lattice positions. Along the track of such an ion, much longer than for a heavy ion, highly deformed regions of the individual cascades become formed. As opposed to the former case, they do not produce a single big-cascade region. The dose of light ions required for producing an amorphous layer is larger than that of heavy ions. This is because the stability of the small isolated cascades is lower than that of the huge cascade formed in heavy ion bombardment.

The defect structure of amorphous layers formed by implantation is complex. Regions of very different structure appear, and to describe them exactly there must be some knowledge available regarding the ion retardation mechanisms, the concentration and spatial distribution of the introduced impurities and defects, and the effect of crystal orientation on the produced defects [4]. This structure bears considerable influence on the process of formation of dislocations, point defect accumulations and impurity precipitates in the recrystallization of implanted layers, necessary when acquiring semiconducting devices. In turn, such defects change the properties of such devices considerably [5], and knowledge of the structure of implanted layers prior to recrystallization may enable the process to be so conducted as to have a minimum number of defects.

This work is an attempt to assess the possibilities of $X$-ray techniques in studying the structure of boron-implanted layers prior to their recrystallization.

The object under scrutiny was a single-crystalline slice of silicon 0.3 mm thick cut in such a way that its largest surface was approximately parallel to the $\{111\}$ planes. The platelet surface was very smooth, achieved by precise mechanical polishing with the use of diamond pastes having grains smaller than 0.25 μm in diameter. During the implantation of the boron ions a part of the platelet was shielded. The orientation of the shield edge, providing the boundary between the implanted section and the non-implanted section, was chosen at random. The ion energy was 60 keV, and the dose $10^{15}$ ions/cm$^2$. A new method of $X$-ray topography was used, namely, the method of $X$-ray autochromatization by means of an oscillating slit [6].

Figure 1 presents the image of the surface of the examined silicon platelet obtained by this method (the 111 reflection, characteristic Cu$K_{\alpha_1}$ radiation). The photograph distinctly shows the difference in reflectivities of the implanted ($A$) and non-implanted ($B$)

regions. This effect is due to the creation of a difference in the extinction in the two sections. In the case of the almost ideal crystal there is a strong influence of primary extinction on the intensity of the diffracted beams. Due to the bombardment with the 60-keV boron ions a disturbed layer is formed; the maximum impurity concentration is at a depth of approx. 2000 Å [1]. The deformed structure of this layer causes the effect of extinction to become lessened. Hence, the intensity of the diffracted beam is stronger in the implanted region than in the non-implanted one.

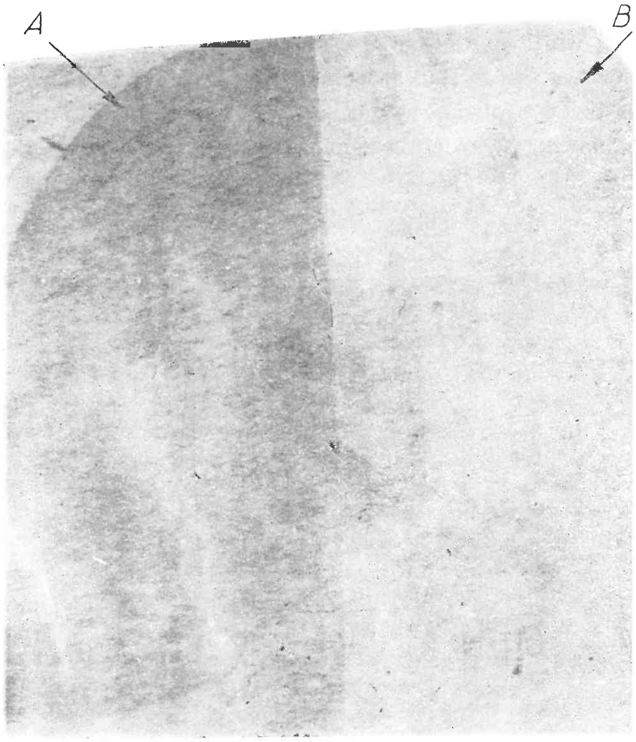

Fig. 1. $X$-ray diffraction image of {111} surface of silicon single crystal. $A$ — implanted region, $B$ — non-implanted region

In order to find what kind of structural changes occur in the surface layer of the implanted crystals, use was made of a two-crystal $X$-ray spectrometer[1] set in the (3-3) position. The monochromator was a dislocations less silicon crystal of {111} orientation, placed relative to the primary beam under an angle allowing the reflection 333 for Cu$K_{\alpha 1}$ radiation to be obtained. From the monochromator the beam impinged under the same angle onto the examined crystal of {111} orientation. Thus, the shape of the diffraction curve for the 333 reflection was measured. The detector was a scintillation counter with a wide-window photomultiplier. This arrangement was thought to be particularly suitable for this kind of measurement because

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[1] Built at the Laboratory of Roentgenography and Roentgenospectroscopy of the Institute of Physics, Polish Academy of Sciences, by K. Godwod, M. Sc.

1) it gives a very narrow diffraction peak for ideal Si crystals (angular width about 2.6″ and peak reflectivity about 65%), and

2) this curve is almost totally insensitive to the spectral distribution of the radiation used (the "parallel" system) and to the content of the various polarization components in the radiation (the angle $\theta$ is nearly 45°), and as such may be exactly calculated for ideal crystals.

The very small width of the curve especially made the arrangement very sensitive to quite minute turns of the crystal lattice and changes in the spacing between planes in the examined crystal. Each of the regions seen in Fig. 1 was examined separately in two crystal positions; namely, such that the boundary between the two regions (*A*) and (*B*)

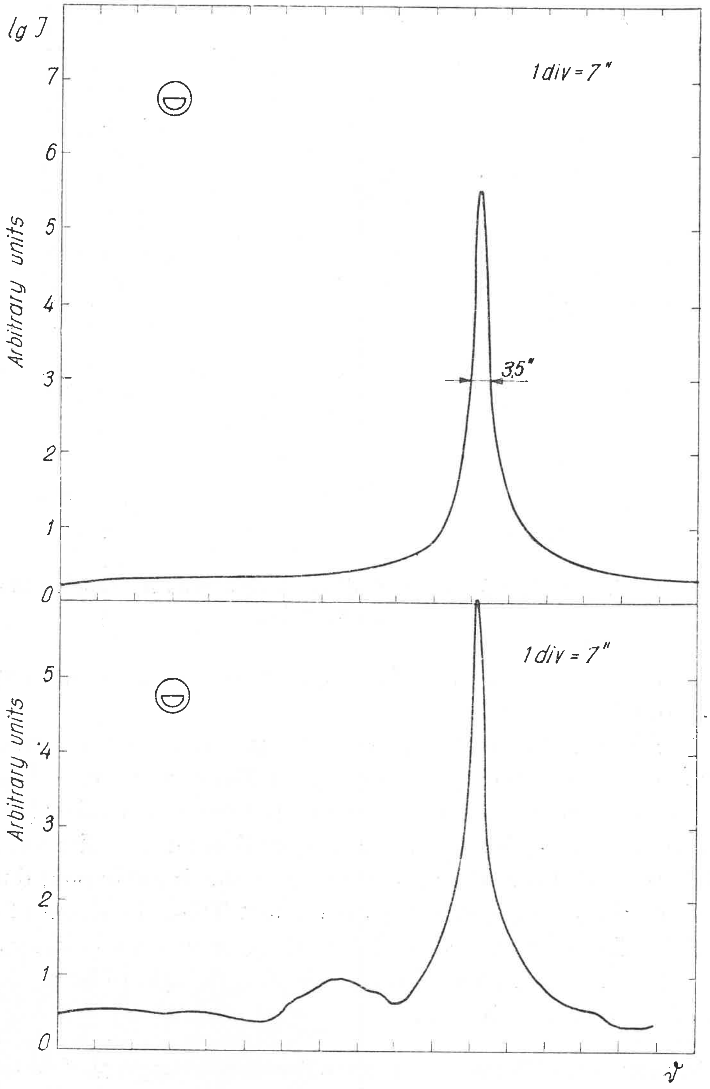

Fig. 2

is either perpendicular or parallel to the vertical axis of the spectrometer. In the first alignment the curve shown in Fig. 2 was obtained. It is apparent that the upper diffraction curve, corresponding to the non-implanted section *B*, is almost symmetrical and has a half-width of 3.5″. The lower part of the figure presents the diffraction curves obtained in the case of the implanted section of the crystal. We see that apart from the principal diffraction curve, the shape of which remains unchanged, a small peak of half-width near 8″ appears on the side of the smaller angles. If the crystal is rotated by 180° in its plane an identical system of peaks is obtained, which may be proof that the additional interference line comes from a crystal "phase" of a somewhat different lattice constant. The quantitative share of this phase in the interference phenomenon must be very small since the ratio of the intensity of the additional line and that of the principal peak is about 0.001. This effect could be revealed thanks to the detecting unit which allows Bragg reflections to be recorded in a logarithmic scale. The observed shifts show that the lattice constant becomes larger in the implanted section (*A*) of the crystal, and the change amounts to about $1.3 \times 10^{-4}$ Å along the ⟨111⟩ direction. Such an increase in lattice constant would be in conformity with theoretical predictions as far as the direction of changes in concerned [2][2].

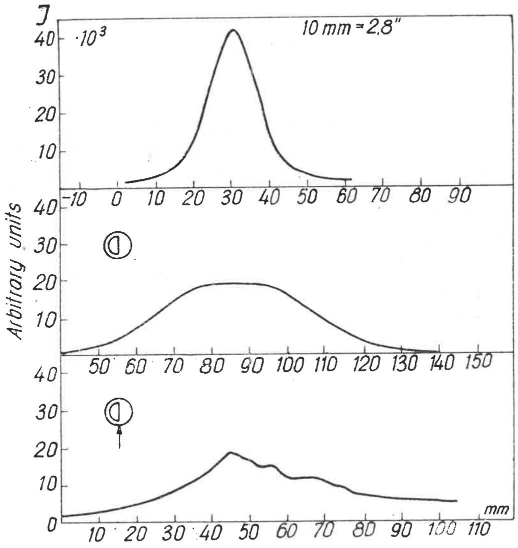

Fig. 3

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[2] The smaller changes in the lattice constant of the implanted layer observed in this work as compared with theoretical predictions [2] are presumably due to incomplete amorphization of the implanted layer at the $10^{15}$ ions/cm$^2$ dose and implantation at room temperature [1, 3]. As far as the authors know, the predictions of theory [2] have not been checked experimentally for implanted layers.

Other causes which could explain the appearance of the additional peak were also considered, namely, segregation of the impurities and thermal effects. As stems from research carried out by various methods (backscattering of alpha particles and protons, EPR, electrical and optical properties), in the case of layers implanted in Si at room temperature and without annealing we encounter a structure deformed in an unorderly way. In such a case segregation of impurities does not occur [1, 4]. On the other hand, any additional peaks associated with thermal effects would have to have much larger half-widths.

In the other case, *i.e.* when the boudary between the regions ($A$) and ($B$) was parallel to the vertical axis of the spectrometer, the diffraction curves were much broader for the implanted section, their half-widths being approx. 13.6″. This is seen in Fig. 3. The lower and middle curves come from different parts of the implanted section ($A$) of the crystal. The upper curve in Fig. 3, on the other hand, corresponds to the non-implanted section ($B$) of the crystal, and its half-width is 3.9″. The broadening of the diffraction curve for the implanted section observed in this case implies that there is directional deformation of the platelet in this section of the crystal, namely, bending about the axis parallel to the boundary. A similar effect was observed by Bubakova and Schmid [8] in the case of silicon bombarded with protons.

The experiments carried out now demonstrate the applicability of $X$-ray methods in precision investigations having the purpose of determining the degree of order and the real structure of implanted layers.

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