

SYNCHROTRON X-RAY DIFFRACTION STUDIES OF Ar-IMPLANTED SILICON AFTER THERMAL ANNEALING

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The research of silicon implanted with swift heavy ions has both cognitive and practical aspects. High energy (several MeV/nucleon) significantly increases the penetration range of ions and the number of generated defects enabling the observation of many physical phenomena. That in particular concerns the studies of the implanted layers by means of X-ray diffraction methods, which are very effective in this case.

The present studies were performed for silicon implanted with argon ions with a total energy of 117 MeV (3 MeV/nucleon), additionally thermally annealed in a complex thermal cycle. X-ray investigations of silicon implanted with high-energy (swift) Ar and Kr ions were previously described in [1–4], including former research of the present sample with conventional X-ray methods published in [4]. It is well known that thermal annealing strongly affects the state of point defects introduced by the implantation [5,6] causing partial annihilation of vacancies and interstitials. In some cases it can cause the formation of point defect clusters leading to the formation of the dislocation loops. The present investigation included a number of complementary X-ray methods exploring both white and monochromatic synchrotron radiation.

The dislocation-free silicon single crystal was grown by the Czochralski method. The (110) oriented 3-mm thick plate studied was implanted with a 5×10^{14} ions·cm⁻² dose of Ar ions, with the total energy 117 MeV (corresponding to 3 MeV/nucleon) of the Ar ion beam, from a $K = 160$ cyclotron at the Heavy Ion Laboratory of the Warsaw University. The beam current was equal to 50 enA. The implantation was performed at room temperature by a uniformly defocused beam. The annealing of the implanted specimen in high vacuum ($\sim 10^{-10}$ Torr) was divided into several steps.

The synchrotron investigations were performed at HASYLAB and included both white and monochromatic beam experiments. The white beam investigations were realized at the F1 line and included Bragg-case section and projection topography. The application of the Bragg-

case section topography allowed to follow the distribution of defects at different depth in the sample.

A monochromatic beam at the E2 line with a wave length of 1.1 Å was used for recording rocking curves and taking topographs. The beam was monochromatised by successive 333 and 511 symmetrical reflections from silicon crystals. The topographs were taken in 440 symmetrical reflection at different angular settings on both sides of the rocking curve. The high intensity of the source in the selected spectral range allowed limiting of the probe beam size to less than 50 µm and examination of the changes of rocking curves in different regions of implanted area.

Thanks to large thickness close to 4 mm, the sample was practically not bent and synchrotron monochromatic beam topographs revealed the whole area illuminated by the beam of 2 mm × 10 mm. A series of topographs were taken at different angular settings. In case of the topograph taken on the low angle flank, we observed characteristic concentric interference fringes similar to those described by us [7,8] which were not visible in the topograph taken at the high angle flank of the rocking curve.

The monochromatic beam topographs revealed also relatively strong contrasts similar to the segregation fringes often observed in silicon crystals. In the present case, when the crystal was relatively low doped, these fringes are not connected with the segregation of the impurity but more probably are due to oxygen precipitates, which may be formed during the annealing. The very interesting feature of the fringes is that their contrast was not inverted in the topographs taken for opposite flanks of the maximum. The most probable reason of this contrast are the changes of the local rocking curve connected with diffuse scattering especially a broadening of the rocking curve near its bottom as it was discussed by Kubena and Holý in [9,10].

The rocking curves recorded from the area exhibiting the fringes were of significantly different character. They exhibited a series of maxima with increasing periodicity, slightly similar to those observed by us in case of proton

implantation [8]. The number of the interference maxima was larger in the rocking curves recorded from the regions closer to the center of the fringes. Also the topographs taken for the different angular settings on the low angle side of rocking curve maximum exhibited different number of fringes decreasing with the angular departure from the maximum.

Differently than in the case of silicon implanted with light proton or helium ions [7,8], a good correspondence between the experimental and numerically calculated theoretical rocking curves cannot be obtained approximating the strain profile by the distribution of created point defects obtained using TRIM 95 program. In particular the rocking curves do not indicate any noticeable change of lattice parameter close to the surface. More satisfactory results provides approximation of the strain profile by the distribution of the implanted argon ions but in this case the strain maximum is too narrow and causing too large period of the interference maxima.

A good position of the interference maxima was obtained using a 1.5 times wider strain profile which was calculated from the initial distribution of ions from TRIM95 by numerical integration of the diffusion equation. This formal transformation may be apologized by a probable diffusion of argon atoms caused by high temperature and initial presence of point defects [11].

The theoretical rocking curves, corresponding to the experimental ones, were calculated. The experimental rocking curves exhibit worse resolution of the interference maxima and significant broadening of the main peak, caused most probably by long-range strains and irregularities of strain distribution. This effect makes a more exact fitting of the theoretical rocking curve and eventual extraction of diffuse scattering practically impossible. We report a series of double exposed white beam Bragg-case section [12,13] and projection topographs recorded from different regions of the implanted area with the sample step-wise translated by 2 mm. The corresponding section topographs in two chosen reflections produce two significantly separated stripes. Using the evaluation described in [12], we found that the lower stripe corresponds to the layer situated approximately at 30 μm (the calculated mean range of 117 MeV Ar ions in Si crystal) while the upper stripe is connected with the reflection from the surface.

The two stripes are visible in the whole implanted area and not only in the area producing interference fringes in plane wave topographs. It may be noticed that the both stripes exhibit a certain structure, which may be interpreted either as the presence of extended dislocation loops or gaseous inclusions. We may also notice the presence of insulated contrasts connected with previously mentioned striation-like contrasts. This effect, mostly similar to observed by us in thermally annealed silicon [14], evidently confirm the presence of the defects connected with oxygen precipitates. These defects may be interpreted either as large not quite coherent oxygen precipitates or the dislocation loops connected with the oxygen precipitates.

In conclusion, synchrotron plane wave topography revealed an area providing characteristic fringes connected with locally increasing strain induced by swift heavy ion implantation. The presence of the strain produced additional interference maxima in the local rocking curves measured with a point probe beam of small diameter. The number of additional interference maxima was different in different curves and was the largest for the curve recorded in the center of previously mentioned fringes.

It was possible to obtain a reasonable correspondence of theoretical and experimental rocking curves approximating the strain profile by the distribution of introduced atoms transformed assuming the diffusion of argon atoms causing some broadening of the strain maximum. The experimental rocking curves exhibited worse resolution of the interference maxima and significant broadening of the main peak. It was caused most probably by long-range strains and irregularities of strain distribution.

The use of Bragg-case section topography revealed some characteristic contrasts, which may be eventually interpreted as connected with dislocation loops in the mostly deformed layer. Some insulated contrasts interpreted as due to oxygen precipitates were connected with the striation-like contrasts observed in the presently studied sample.

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