Formation of a layered structure in the distribution of boron atoms initiated in silicon by ion implantation

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Secondary-ion mass spectrometry has been used to study the redistribution of boron isotopes in silicon samples subjected to diffusive doping with implanted boron to a total concentration $\sim 2\times 10^{20}$ cm^{-3} of the isotopes ^{10}B and ^{11}B, followed by implantation of the isotope $^{10}B^+$ at an energy of 180 keV in a dose of 10^{16} cm^{-2}. Annealing at 900 °C gives rise to a quasiperiodic structure consisting of five clearly expressed peaks formed on the concentration profile of each isotope in the region subjected to ion implantation. The results are explained under the assumption that there is a decomposition of a supersaturated solid substitution solution of boron in silicon, initiated by an annealing out of structural defects built up during the bombardment. This process would be accompanied by the formation of precipitates containing boron with a defect-stimulated drainage of the free component of the boron to them.

The purpose of the present study was to learn about the redistribution of boron during heat treatment of silicon which had been doped uniformly with boron to a concentration exceeding the solubility limit for the annealing temperature and which had been bombarded with B^+ ions.

The distributions of the isotopes ^{10}B and ^{11}B in the bulk of the samples were studied by secondary-ion mass spectrometry with the help of a CAMECA-RIBER MIQ-256 apparatus. The primary beam of O_2^+ ions had an energy of 13 keV. This beam was scanned over an area $\approx 0.4\times 0.6$ mm^2. To eliminate a contribution from secondary boron ions ejected from the crater walls, we took the signal to be analyzed from a central region of the crater constituting 10% of the scanning area. The etching rate during the measurements was $\approx$0.5 nm/s.

The starting material in the experiment was KÉF-7.5 silicon with a (100) surface. A diffusive doping with boron was carried out at 1150 °C in the initial silicon platelets. A region of uniform doping, with a boron concentration $\approx 2\times 10^{20}$ cm^{-3}, was created down to a depth of at least 1 μm. At this temperature, this boron concentration corresponds to the limiting concentration of boron in substitution sites.[1,2] The concentrations by volume of the ^{10}B and ^{11}B isotopes ($\approx$20 and $\approx$80%) were determined in this case from the natural abundance of these isotopes. After these steps, $^{10}B^+$ ions with an energy $E = 180$ keV were implanted to a dose $D = 10^{16}$ cm^{-2} on an HV-400 apparatus.

Figure 1a shows the depth profile of the boron isotopes found after the implantation. The samples prepared in the specified manner were annealed for times ranging from 10 min to 4 h at 900 °C. At this temperature, the limiting concentration of boron in substi-

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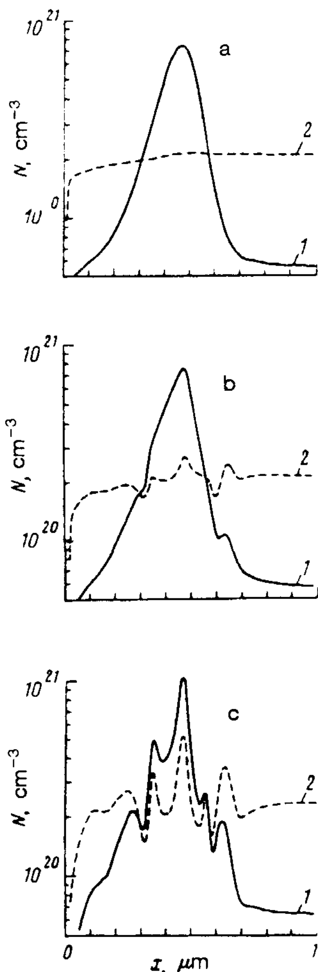

FIG. 1. Concentration profiles of boron isotopes in silicon resulting from thermal diffusion and implantation of ^{10}B ions ions ($E = 180$ keV, $D = 10^{16}$ cm^{-2}). 1—^{10}B; 2—^{11}B. a) Without annealing; b) $T = 900$ °C, $t = 10$ min; c) 900 °C, 60 min.

tution positions[1,2] $\approx 6\times10^{19}$ cm^{-3}. The results are shown in Fig. 1, b and c. We see from this figure that even the 10-min annealing causes a transformation of the concentration profiles of the boron isotopes: The ^{10}B profile loses its Gaussian shape, and inflection points and additional peaks begin to appear on the wings of the profile. Simultaneously, a concentration modulation arises on the originally flat ^{11}B profile. The structural features that arise on the two concentration profiles have the same spatial coordinates. As the annealing time is raised to 1 h, five clearly expressed peaks form on the concentration profiles, along with a quasiperiodic structure. At this point, the process by which the layered structure forms essentially ends: The number and spatial positions of the peaks do not change as the annealing time is raised to 4 h. The only changes are slight increases in the boron concentration in each of the peaks. The total amount of both boron isotopes in the test samples remains constant within the measurement error. A spatial redistribution of boron is observed only in the immediate vicinity and in the region bombarded most heavily with $^{10}B^+$ ions.

We know that, in the range 700–1000 °C, the transformation and annealing out of defect–impurity associations built up during the implantation lead to both a high boron mobility[2–5] and the formation of precipitation centers.[6–8] A competition among precipitation centers in trapping the free component of the boron has the consequence that the profiles reflect only those centers that lie at distances comparable to the mean diffusion length of the free component of the boron. The process by which the layered structure forms ends with an annealing out of the radiation-induced defects. Since the coordinates of the structure are fixed by even a 10-min annealing, the diffusion coefficient of the free component in this case must be at least

$$D \approx L^2/t \approx 2 \times 10^{-13}\ \mathrm{cm}^2/\mathrm{s},$$

where $L \approx 0.1\ \mu\mathrm{m}$ is the average period of the structure, and $t = 600$ s.

The impurity profiles observed in the early stages of the annealing ($t = 10$ min) do not give sufficient information to unambiguously link the appearance of precipitation centers with exclusively an impurity supersaturation effect, since the peaks form on the $^{10}\mathrm{B}$ and $^{11}\mathrm{B}$ profiles in regions in which there is no significant supersaturation in the boron concentration. The appearance of these peaks can thus be linked with structural defects resulting from the ion implantation.

According to the data of Ref. 8, the formation of structural defects (primarily dislocations) during an annealing results not only from a restructuring of defects introduced by the irradiation but also from a local change in the density due to the implanted matter. Specifically this point was used in Ref. 8 in a discussion of the differences between the types of structural defects in the bulk of the sample subjected to bombardment. In other words, structural defects which can also arise at depths greater than the projected ion range[9] R_p make their own contribution to the formation of nucleating regions of precipitation centers.

If these regions are to be seen clearly on the concentration profiles, they must be spatially separated. This separation is determined by the energy of the implanted ions. It is likely that for this reason only a single concentration spike appeared on the profile at 900 °C in Ref. 3, where the experimental procedure differed only in energy ($^{11}\mathrm{B}^+$, 70 keV). The single spike appeared to be a superposition of at least two constituent peaks. We should point out that there was apparently no spatial separation of the boron-rich regions in Refs. 6 and 7, where an accumulation of impurity in an R_p region was observed as the result of an annealing at 950 °C of silicon samples bombarded with boron ions at energies of 25 and 40 keV in doses of $10^{16}\ \mathrm{cm}^{-2}$ and $4 \times 10^{15}\ \mathrm{cm}^{-2}$, respectively. At the same time, electron microscopy revealed two spatially separated layers of structural defects.[7]

Furthermore, we do not rule out the possibility that a buildup of impurities in local regions stimulates the development of structural defects in the crystal lattice of silicon. This factor in turn creates some further possibilities for trapping boron atoms, and the process occurs in a self-accelerated manner.

In summary, this study has shown that implantation of boron ions in a supersaturated solid substitution solution of boron in silicon leads to a redistribution of significant

amounts of both the implanted impurity and an impurity introduced by a preliminary diffusion in the course of heat treatment. The resulting profile of the distribution has a clear spatial periodicity.

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