X-ray Bragg Reflexion and Strain Compensation in Silicon Crystals

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(Received 11 June 1979; accepted 10 September 1979)

Abstract
Crystal-lattice strain induced by impurity doping may be compensated for by diffusion of another kind of impurity. This was tried with successive diffusion of germanium and boron into (111) silicon wafers and was confirmed in a nondestructive way from the intensity profiles of the 333 Bragg reflexion of Cu Kα X-rays. The intensity profile is related to the strain distribution along the depth through numerical computations, the programme for which has been developed and utilized previously [Fukuhara & Takano (1977). Acta Cryst. A33, 137-142] in quantitative discussions on strain and impurity concentration in silicon.

1. Introduction
Crystal-lattice distortion, resulting from impurity diffusion, ion implantation, etc., has been widely studied with X-ray diffraction methods especially for silicon crystals in connexion with material processes for solid-state devices (e.g. Larson, White & Appleton, 1978).

The intensity profile of a Bragg reflexion, i.e. rocking curve, can be a good tool to investigate the strain distributions along the depth, as shown in the previous reports (Fukuhara & Takano, 1977a, b); fairly good fits were reached between experimental rocking curves and theoretical ones computed for model strain distributions, which were assumed from physical pictures of the sample-preparation processes.

Our investigation showed that the proportionality between the strain and the impurity concentration holds, at least up to \(4 \times 10^{25}\) for boron and \(10^{26}\) atoms \(\text{m}^{-3}\) for phosphorus, and that the proportionality coefficient depends on the crystallographic orientation of the sample surface. The coefficient was determined for (111) and (100) wafers of silicon doped with boron and phosphorus, respectively; its dependence on the orientation was consistent with a prediction based on elastic stiffness constants.

The purpose of the present report is to investigate an example of strain compensation from double impurity diffusion by using rocking curves and to extend the applicability of the strain-analysis method. The method of computation is within the frame of the dynamical diffraction theory for distorted crystal lattices (Takagi, 1962, 1969; Taupin, 1964) and has been explained previously (Fukuhara & Takano, 1977a); the experimental arrangements and their characteristic features were also described there. Thus these are not repeated here.

2. Comparison between experimental data and theoretical results
In Fig. 1, an experimental rocking curve is shown for the 333 Bragg reflexion of Cu Kα X-rays with a silicon (111) wafer, which was annealed at 1423 K for 24 h after being subjected to boron-doped silicon as a diffusion source. The relative intensity of the reflexion was numerically computed with the assumption that the strain (lattice contraction in this case) had only the \((z, z)\) component of a Gaussian functional form as stated in the figure caption and that X-rays were in the pure \(\sigma\) mode of polarization. The latter assumption is consistent with the situation that the Bragg angle of this reflexion is near \(45°\). The surface value of the strain corresponds to a concentration \(1.3 \times 10^{25}\) boron atoms/\(\text{m}^3\). The curve shown here has the typical profile of such samples, i.e. consists of a sharp peak due to the thick substrate and a broader peak or hump for the distorted surface layer accompanied by subsidiary maxima.

![Fig. 1. Rocking curve of the Cu Ka 333 reflexion from a (111)Si wafer after boron diffusion. The calculated curve is for \(\sigma\)-polarized X-rays and for a strain distribution \(e_{zz} = -8.6 \times 10^{-3} \exp \left[-(z/5.1)^2\right]\), \(z\) being the coordinate along the depth in \(\mu\text{m}\). The abscissa shows the angular deviation of the incident direction measured from the exact Bragg position for the substratum. The ordinate gives the reflexion intensity relative to the incident one for the computed curve and the scale of experimental values was adjusted for the fitting.](image)

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Fig. 2 corresponds to a similar rocking curve for another sample with a lattice expansion caused by germanium diffusion. The sample was annealed at 1473 K for 14 d after being subjected to germanium-doped silicon. The surface concentration of germanium is estimated at \(8.3 \times 10^{26}\) atoms/m\(^3\) from experimental data for germanium diffusion obtained by the neutron-activation method. It should be noticed that the curve here looks roughly similar to that in Fig. 1 on reversing the direction of the abscissa; this shows that the strain distributions in these two samples resemble each other except for their signs. Since germanium hardly diffuses in silicon, the sample underwent rather serious heat treatment which presumably caused growth of some swirl defects contained in the initial as-grown crystal. This is possibly the reason why the experimental curve appears blunt and a better fitting with the theoretical estimation could not be reached here.

For the third sample, the above two diffusion processes were performed in sequence in expectation of strain compensation. The resultant rocking curve of this sample is shown in Fig. 3. The theoretical curve was worked out for a strain distribution which is a simple sum of the two strain distributions previously assumed. Although the fitting is not satisfactory, the simple peak structure and its narrow width reveal the real occurrence of the strain compensation.

For confirmation of this reasoning, a surface layer, 1.5 \(\mu\)m thick, was etched off between the germanium and the boron diffusion processes. The sample just after the etching corresponds to the rocking curve in Fig. 4, where the computation is for a truncated form of the strain distribution assumed in Fig. 2. The etching has made the curve profile smooth. The result in Fig. 5 was obtained from the sample after boron diffusion. The rocking curve here discloses an incomplete strain compensation, i.e. there is lattice contraction due to the predominance of the boron-diffusion effect.

3. Concluding remarks

As an extension of our study on strain distribution in a crystal lattice by using rocking curves of an X-ray reflexion, an example of strain compensation was examined; it is clearly seen from the change in the intensity profile that the lattice expansion due to germanium diffusion into a silicon wafer was nearly

![Fig. 2. Rocking curve of the Cu Ka 333 reflexion from a (111) Si wafer with germanium diffusion. The computed curve is for \(\sigma\)-polarized X-rays and for a strain distribution \(e_z = 8.0 \times 10^{-5} \times \exp\left[-(z/4.9)^2\right]\).](image)

![Fig. 4. Rocking curve obtained from the same sample as used for Fig. 2, except that a 1.5 \(\mu\)m thick surface layer has been removed by chemical etching.](image)

![Fig. 3. Rocking curve of the same reflexion but from another (111) Si wafer with germanium diffusion and boron diffusion performed in sequence. The strain distribution assumed for the computation is a simple sum of the two assumed for Figs. 1 and 2.](image)

![Fig. 5. Rocking curve after boron diffusion into the sample for Fig. 4.](image)
cancelled out by subsequent boron diffusion which alone would cause a lattice contraction.

Other possible pairs of species for strain compensation can be anticipated from data of atomic radii (Pauling, 1960) in tetrahedral bonding. Actually, it was confirmed by one of the authors (YT) that the pair of germanium and phosphorus also cause this compensation. Tin can also be adopted among fourth-group elements as the lattice expander of the pair, as described by Yagi, Miyamoto & Nishizawa (1970).

We have shown several examples of diffraction profiles affected by lattice-strain distributions in the present and previous reports. Thus, if proper care is taken in the design of an experiment, i.e. choice of X-rays and reflexion order, fine collimation etc., and if the form of strain distribution can be presumed, profiles of the rocking curve can give us even quantitative information to help the understanding of phenomena in wider fields related to strain distribution.

References