Radiation Physics and Chemistry 78 (2009) S67–S70

Contents lists available at ScienceDirect

Radiation Physics and Chemistry

journal homepage: www.elsevier.com/locate/radphyschem

Thermally induced defects in silicon irradiated with fast neutrons

A. Misiuk [a,*], W. Wierzchowski [b], K. Wieteska [c], P. Romanowski [d], J. Bak-Misiuk [d], M. Prujszczyk [a], C.A. Londos [e], W. Graeff [f]

[a] *Institute of Electron Technology, Al. Lotników 46, 02-668 Warsaw, Poland*
[b] *Institute of Electronic Materials Technology, 01-919 Warsaw, Poland*
[c] *Institute of Atomic Energy, 05-400 Otwock-Świerk, Poland*
[d] *Institute of Physics, PAS, Al. Lotników 32/46, 02-668 Warsaw, Poland*
[e] *University of Athens, Athens 15784, Greece*
[f] *HASYLAB at DESY, D-22603 Hamburg, Germany*

ARTICLE INFO

Article history:
Received 14 October 2008
Accepted 20 March 2009

Keywords:
Silicon
Neutron irradiation
Imaging
Defect structure

ABSTRACT

Defect structure of Czochralski grown (111) oriented silicon single crystals (Cz-Si), irradiated with fast neutrons (energy 5 MeV, dose $5\times10^{16}\,\text{cm}^{-2}$) and annealed at up to 1400 K, also under hydrostatic Ar pressure equal to 1.1 GPa, has been investigated by high-resolution X-ray diffraction and synchrotron topography at HASY Laboratory. The annealing, especially at 1270 and 1400 K, results in precipitation of interstitial oxygen and creation of extended defects. A thermally induced oxygen precipitation at high temperatures–pressures in neutron-irradiated Cz-Si reveals the irradiation-related history of investigated samples.

1. Introduction

Irradiation of oxygen-containing Czochralski grown silicon single crystals (Cz-Si) with fast neutrons (in what follows the neutron-irradiated Cz-Si samples are labeled as nCz-Si) and subsequent processing of nCz-Si at enhanced temperatures results in accelerated precipitation of oxygen interstitials (Varnina et al., 2001; Ma et al., 2005) present in Cz-Si at up to about 1.2×10^{18} cm^{-3} concentration (c_o). An amount of precipitated oxygen (Δc_o) is dependent on sample and irradiation parameters, among them on c_o and conductivity type, as well as on neutron energy (E), dose (D), annealing temperature (HT) and time. The oxygen-containing defects in nCz-Si prepared by neutron irradiation with $D=10^{16}$ cm^{-2} were revealed after processing at $\leq 1070\,\text{K}$ for $t\approx 10\,\text{h}$; this time decreased with increasing both the dose and temperature (Varnina et al., 2001).

Irradiation-induced oxygen precipitation in nCz-Si has been reported also to depend on the value of hydrostatic argon pressure (HP) applied during annealing (Misiuk et al., 2005). This influence of HP on Δc_o is related either to stabilization of radiation-induced defects or to a creation of additional nucleation sites for O_i's precipitation. Oxygen precipitation induced by specific thermal processing under HP of boron-doped Cz-Si irradiated with high doses of γ rays or neutrons has been suggested by Misiuk et al. (2007) and Wieteska et al. (2008) for application in dosimetry.

Thermally induced defects produced in *n*-type phosphorus-doped (111) oriented Cz-Si irradiated with medium dose ($D=5\times10^{16}\,\text{cm}^{-2}$) of neutrons and processed at HT–(HP) are investigated in the present work in more detail by high-resolution X-ray diffraction and synchrotron topography (compare the paper by Misiuk et al., 2005). The resolution of synchrotron X-ray topographic methods was on the 2–5 μm level. Much smaller precipitates can be detected, however, if they produce sufficiently large deformation of the surrounding lattice.

2. Experimental

The (111) oriented Cz-Si samples of about $14\times8\,\text{mm}^2$ dimension with initial concentration of interstitial oxygen (O_i), $c_o=9\times10^{17}\,\text{cm}^{-3}$, were cut from 1.8-mm-thick P-doped silicon wafers and irradiated with fast neutrons ($E=5\,\text{MeV}$) to $D=5\times10^{16}\,\text{cm}^{-2}$. Neutron-irradiated samples and reference non-irradiated samples were processed for 2–5 h at up to 1400 K in Ar atmosphere under 10^5 Pa (atmospheric pressure) and under pressure of 1.1 GPa.

After processing the near-surface Si layers of about 150 μm thickness were removed from both the sample sides by chemical polishing.

The sample defect structure was determined by high-resolution X-ray diffraction (recording ω and $2\theta/\omega$ scans in triple axis

---

* Corresponding author. Tel.: +48 22 5487792; fax: +48 22 847 06 31
E-mail address: misiuk@ite.waw.pl (A. Misiuk).

0969-806X/$ - see front matter © 2009 Elsevier Ltd. All rights reserved.
doi:10.1016/j.radphyschem.2009.03.087

configuration of diffractometer) and by synchrotron diffraction topography at F1 and E2 experimental stations of the DORIS III synchrotron in HASYLAB (Germany). Both white and monochromatic ($\lambda = 0.1115\,\text{nm}$) beam topographic methods in the Bragg case geometry were used. Section topography (with the application of a fine 5 μm slit and glancing angle of 5°) enabled indication of volume character of defect distribution. High sensitivity to strains associated with small inclusions and dislocation loops was provided by monochromatic beam topography taken in the 333 symmetrical reflection.

3. Results and discussion

The near-surface layers of silicon samples were chemically out-etched after processing at HT–(HP) conditions. This means that the below reported effects are predominantly related to the bulk structure of investigated samples.

For nCz-Si as well as for the reference Cz-Si samples processed for 5 h at 1070 K, both under 10^5 Pa and under high pressure, only a small fraction of O_i's precipitated ($\Delta c_o/c_o \leq 0.1$). Very few individual defects distributed within sample volume were detected appearing as dark spots visible below the dark stripe coming from the reflection of the incident linear beam from the sample surface (Fig. 1). One can suppose that the observed contrast corresponds to structural defects in as-grown samples, eventually decorated by oxygen atoms. These defects do not disturb markedly the interference pattern visible close to the slit image.

Processing of nCz-Si at 1270 K for 5 h results in massive precipitation of O_i's (Fleming et al., 2007), especially if performed under HP (Misiuk et al., 2007). This precipitation is much less pronounced in the case of reference Cz-Si processed at the same conditions. Interference-related effects remain to be still visible close to the slit image (Fig. 2). Individual defects at a medium concentration and of evidently volume distribution are detectable after processing at 1270 K under 10^5 Pa (Fig. 2A). In the case of nCz-Si processed at 1270 K under 1.1 GPa, one can detect numerous tiny defects distributed almost uniformly within the sample volume. The presence of these defects results in extended "grain like" shadow below the stripe coming from the reflection of the linear beam from the surface (Fig. 2B).

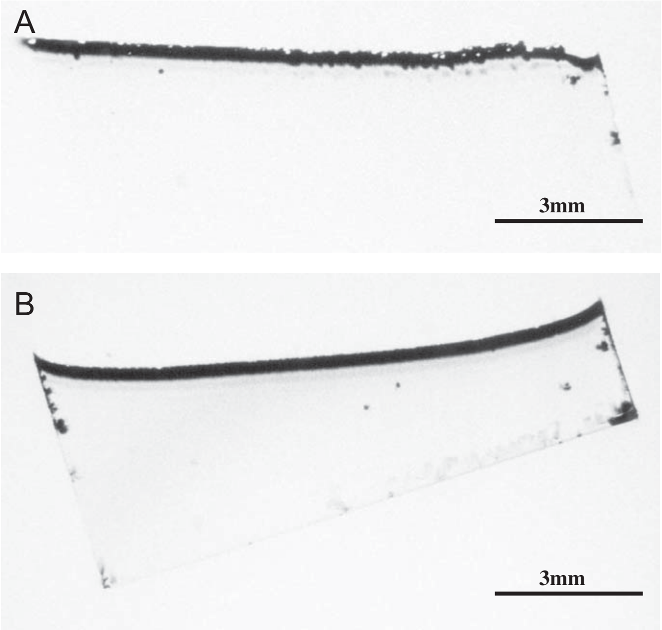

Fig. 1. White beam back reflection synchrotron X-ray section topographs of nCz-Si processed for 5 h at 1070 K under 10^5 Pa (A) and 1.1 GPa (B).

The observed asymmetry of $2\theta/\omega$ scans for nCz-Si processed at 1270 K for 5 h (Fig. 3) indicates on the lattice constant spread, especially for the sample processed under 10^5 Pa (Fig. 3A). The relatively high-X-ray diffuse scattering intensity observed for this sample (Fig. 4A) can be related to the presence of defect clusters or small dislocation loops (e.g., Patel, 1975; Shalimov et al., 2007).

Processing of nCz-Si at 1400 K under 10^5 Pa as well as under HP (Figs. 3B, 4B and 5) produces much less defects distributed within the nCz-Si volume. The defect structure of nCz-Si processed at 1400 K is comparable with that in reference non-irradiated Cz-Si subjected to similar processing (observe the respective scans, Figs. 3C and 4C).

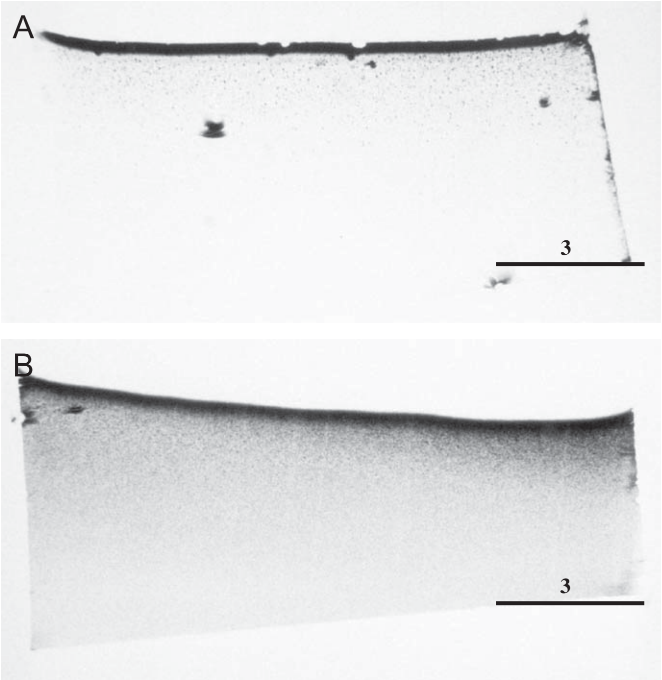

Fig. 2. White beam back reflection synchrotron X-ray section topographs of nCz-Si processed for 5 h at 1270 K under 10^5 Pa (A) and under 1.1 GPa (B).

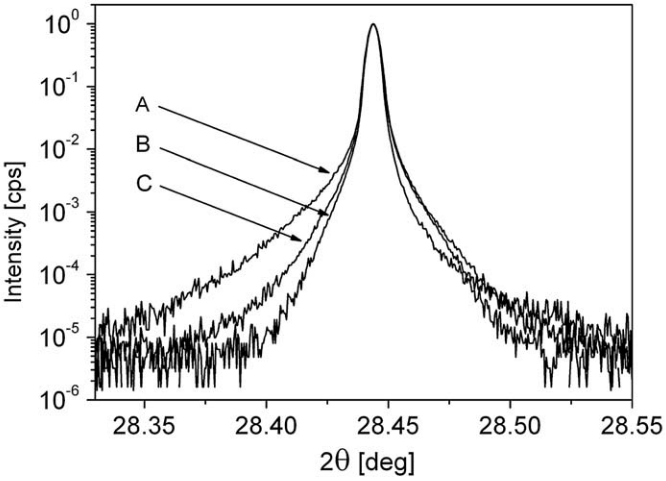

Fig. 3. $2\theta/\omega$ scans (Cu$K_{\alpha 1}$ radiation, 111 reflection) of nCz-Si processed for 5 h at 1270 K under 10^5 Pa (A) and for 2.2 h at 1400 K under 1.1 GPa (B). Scan C concerns reference Cz-Si processed for 5 h at 1400 K under 1.1 GPa.

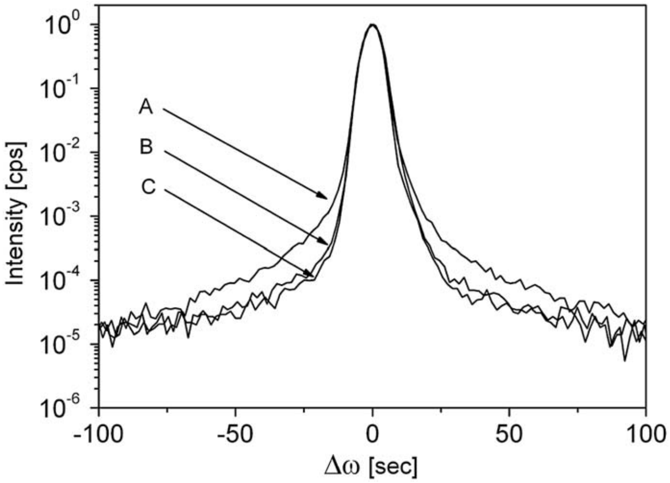

Fig. 4. ω scans in triple axis configuration ($CuK_{\alpha 1}$ radiation, 111 reflection) for nCz-Si processed for 5 h at 1270 K under 10^5 Pa (A) and for 2.2 h at 1400 K under 1.1 GPa (B). Scan C concerns reference Cz-Si processed for 5 h at 1400 K under 1.1 GPa.

While only non-numerous individual defects are visualized by synchrotron topography after processing of nCz-Si at 1400 K under 10^5 Pa (Fig. 5A and B), lack of interference fringes and extended contrast close to the slits still suggests the presence of small unresolved individual defects created in effect of processing at 1400 K under high pressure (Fig. 5C).

In the case of low concentration of individual defects, their presence does not disturb much the interference fringes in the section topographs. Lack of interference fringes and extended contrast of the stripe for the samples processed at 1270 and 1400 K (Figs. 2 and 5) may be interpreted as indicating on the presence of unresolved small inclusions in a high concentration. This means that numerous tiny individual defects, uniformly distributed in the samples volume, are produced in nCz-Si, especially after processing at 1270 K under high pressure.

Processing at 1400 K produces less oxygen-related defects, probably because of enhanced solubility of oxygen at so high temperature (compare Misiuk et al., 2005). Uniform distribution of precipitation-induced defects was evidently indicated by the Bragg case-section topographs revealing the defects located along the narrow beam intersecting the crystal. The defects concentration was the highest in nCz-Si processed at 1270 K under 1.1 GPa.

As also suggested earlier (Misiuk et al., 2005; Wieteska et al., 2008), neutron irradiation and subsequent HT–(HP) processing result in massive HT, HP-induced precipitation of oxygen interstitials at the nucleation sites (such as vacancies, *V*, and, especially, their complexes with oxygen, *V*O or V_mO_n) produced by irradiation.

4. Conclusions

The results presented now concern the earlier unreported case of P-doped *n*-type Cz-Si irradiated with medium dose ($D = 5 \times 10^{16}\,\mathrm{cm}^{-2}$) of fast energy neutrons and processed at 1070–1400 K under 10^5 Pa and 1.1 GPa. Processing of nCz-Si under enhanced hydrostatic pressure, especially at 1270 and 1400 K, results in massive precipitation of interstitial oxygen on irradiation-induced nucleation sites with a creation of extended defects. One can consider this effect as useful for post-radiation dosimetry applicable for Si-based elements.

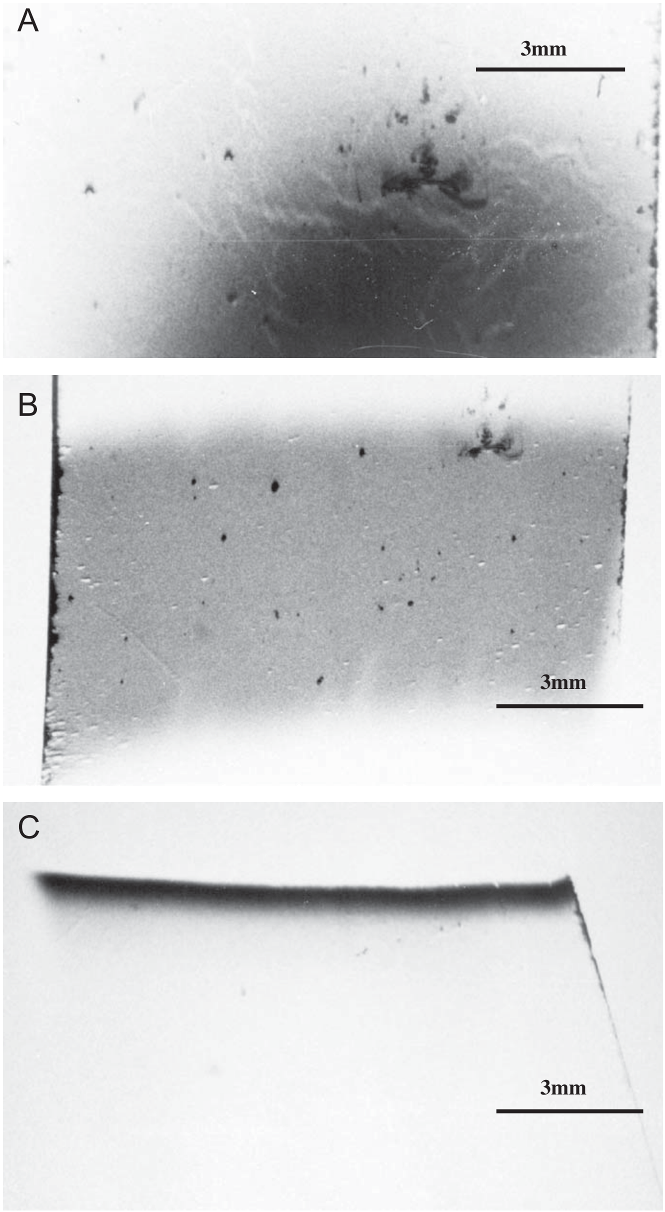

Fig. 5. Synchrotron X-ray topographs of nCz-Si processed for 5 h at 1400 K under 10^5 Pa, and investigated using monochromatic beam, 333 reflection (A), and white beam projection (B). (C): Back reflection section topograph after processing for 2.2 h at 1400 K under 1.1 GPa.

References

Fleming, R.M., Seager, C.H., Lang, D.V., Cooper, P.J., Bielejec, E., Campbell, J.M., 2007. Effects of clustering on the properties of defects in neutron irradiated silicon. J. Appl. Phys. 102, 043711-1–043711-13.

Ma, Q.Y., Li, Y.-X., Chen, G.-F., Yang, S., Liu, L.-L., Niu Ping, J., Chen, D.-F., Li, H.-T., 2005. Accelerated oxygen precipitation in fast neutron irradiated Czochralski silicon. Chin. Phys. 14, 1882–1885.

Misiuk, A., Surma, B., Bak-Misiuk, J., Londos, C.A., Vagovič, P., Kovačević, I., Pivac, B., Jung, W., Prujszczyk, M., 2007. Revealing the radiation-induced defects in silicon by processing at enhanced temperatures–pressures. Radiat. Meas. 42, 688–692.

Misiuk, A., Surma, B., Londos, C.A., Bak-Misiuk, J., Wierzchowski, W., Wieteska, K., Graeff, W., 2005. Oxygen precipitation and creation of defects in neutron irradiated Cz-Si annealed under high pressure. Phys. Status Solidi (C) 2, 1812–1816.

Patel, J., 1975. X-ray diffuse scattering from silicon containing oxygen clusters. J. Appl. Cryst. 8, 186–191.

Shalimov, A., Shcherbachev, K.D., Bak-Misiuk, J., Misiuk, A., 2007. Defect structure of silicon crystals implanted with H^{2+} ions. Phys. Status Solidi (A) 204, 2638–2644.

Varnina, V.I., Groza, A.A., Litovchenko, P.G., Starchik, M.I., Shmatko, G.G., Marchenko, L.S., Semenjuk, A.K., Litovchenko, A.P., 2001. The radiation defect influence on oxygen precipitation in thermally treated silicon. Ukr. Phys. Zhurn. 46, 205–210.

Wieteska, K., Misiuk, A., Wierzchowski, W., Bak-Misiuk, J., Romanowski, P., Surma, B., Capan, I., Yang, D., Shalimov, A., Graeff, W., Prujszczyk, M., 2008. Revealing the defects introduced in N- or Ge-doped Cz-Si by γ irradiation and high temperature–high-pressure treatment. Acta Phys. Polon. A 114, 439–446.