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Stress-induced transformation of microdefects in neutron irradiated Czochralski silicon

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Abstract

Microstructure of neutron irradiated Cz-Si has been examined after out-annealing of irradiation-induced defects and pressure treatment at 1170 K-1 GPa. X-ray diffuse scattering and defect dimensions are related to oxygen concentration and to stress-induced oxygen precipitation. The pressure treatment results in diminished diffuse scattering and defect dimensions. © 2001 Elsevier Science Ltd. All rights reserved.

Keywords: Neutron irradiated Cz-Si; High pressure–high temperature; X-ray diffraction

1. Introduction

Si monovacancies, V, produced by neutron irradiation, are mobile and, besides annihilation or pairing (to produce divacancies, V_2), are trapped by O interstitials, O_i, to form vacancy-O_i pairs, VO. Such pairs are manifested by absorption at 828 cm^{-1} (NA-centres). Annealing at 670–770 K results in decreased intensity of the 828 cm^{-1} band and in absorption at 887 cm^{-1}, attributed to VO_2. Further annealing leads to formation of the V_nO_m bands [1]. At about 1040 K all bands disappear except that at 1106 cm^{-1}, related to presence of O_i. One would expect that annealing of neutron irradiated Cz-Si results in restoration of its structure; this statement, however, was questioned [2].

Determination of the effect of neutron irradiation and annealing on the Cz-Si microstructure is investigated in this work, also after subjecting to high temperature–pressure treatment (HT–HP). Just the Cz-Si defect structure was proven to be extremely sensitive to the HT–HP treatment [3].

2. Experimental

The initial O_i concentration, c_o, and the carbon concentration, c_c, in the investigated samples are presented in Table 1. Two, A and B, batches of samples were prepared. The A samples (non-irradiated) were annealed at 1170 K-10^5 Pa or HT–HP treated at 1170 K-1 GPa for 5 h. The B samples were irradiated by fast neutrons (dose 1×10^{17} cm^{-2}) at 320 K (the effect of thermal neutrons, with $E<0.4$ eV, was avoided by putting the samples inside Cd foil) and subjected to isochronal step-anneals for 15 min at 10^5 Pa (10 K steps), from 670 to 1040 K. Afterwards the B samples were subjected to the same treatment as the A samples: at 1170 K-1 GPa for 5 h.

The sample properties were determined by X-ray and

Table 1
Sample designation, corresponding to different content of carbon and oxygen in investigated samples

Designation	Conductivity	$c_c(\pm0.2)\times10^{-16}$ (cm^{-3})	$c_0(\pm0.3)\times10^{-17}$ (cm^{-3})
I	n	5.9	7.6
II	p	9.3	8.2
III	p	15.2	7.7
IV	p	4	8.7

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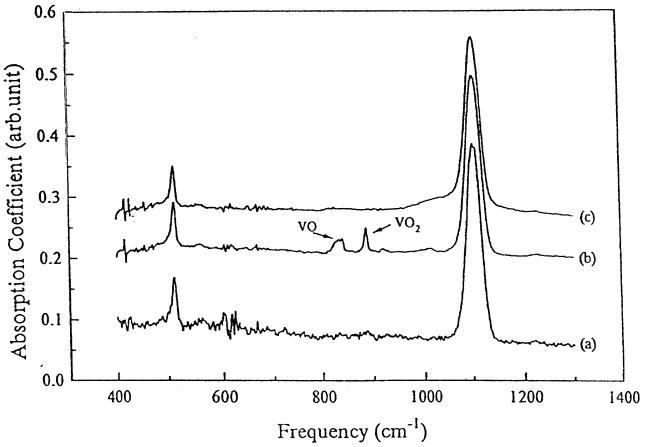

Fig. 1. IR spectra of neutron-irradiated BIV samples: (a) just after irradiation by neutrons; (b) after annealing at up to 670 K, and (c) after completion of isochronal annealing at up to 1040 K.

infrared (IR or FTIR) methods. The defects sizes were calculated from X-ray diffuse scattering data in the Huang range [4].

3. Results and discussion

The IR spectra of BIV samples are presented in Fig. 1. Reciprocal space maps (RSMs) of the AIII and BIII samples (the last subjected to neutron irradiation and out-annealing) as well as of AI and BI ones are presented in Figs. 2 and 3. The A samples indicate the presence of defect clusters, of 170–230 nm dimension (Table 2). Annealing of the A samples at 1170 K-10^5 Pa results in a slight increase (<10%) of c_0 (compare Tables 1 and 3).

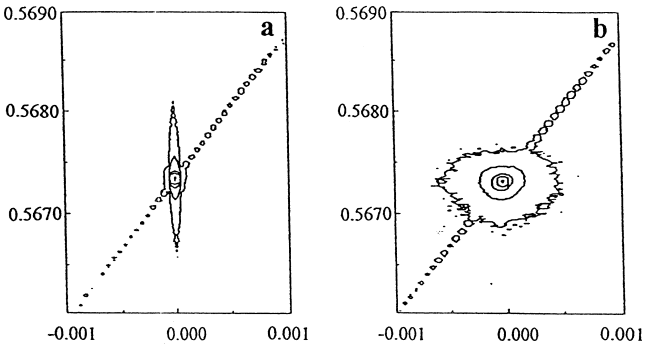

Fig. 2. RSMs of AIII (a) and of BIII (b) samples.

Table 2
Defect dimension d [nm] determined from diffuse scattering intensity in the Huang range

Designation	A	A after HT	A after HT–HP	B	B after HT–HP
I	230	190	150		320
II			165		270
III	210	180	160	295	240
IV	170	160	140	240	200

The d-values decrease in effect of annealing at 1170 K-10^5 Pa (Table 2). The HT–HP treatment of the A samples resulted in decrease of c_0 (Table 3); the d-value for the AI sample decreased to 150 nm. The B samples, out-annealed after neutron irradiation, indicate the decreased c_0 values (e.g. c_0 for the BI sample decreased to 6.5×10^{-17}cm^{-3}, Table 3). The HT–HP treatment of the B samples also resulted in decrease of c_0. For the samples with a similar c_c but of different c_0, the defect dimensions decreased with increased c_0.

It is generally assumed that out-annealing of neutron irradiated Si at up to 1040 K-10^5 Pa results in complete removal of the IR-detectable defects (Fig. 1), so the microstructure of such samples is expected to become again similar to that of the initial ones. However, as it follows from RSM's and FTIR data (Fig. 2, Tables 2 and

Table 3
Concentration of oxygen interstitials, c_0 [$\times10^{-17}$, cm^{-3}], in A and B samples

Designation	A after HT	A after HT–HP	B	B after HT–HP
I	7.9	7.2	6.5	5.9
II	8.5	8.0	7.6	7.1
III	8.2	7.7	7.2	6.6
IV	9.4	9.1	8.6	8.2

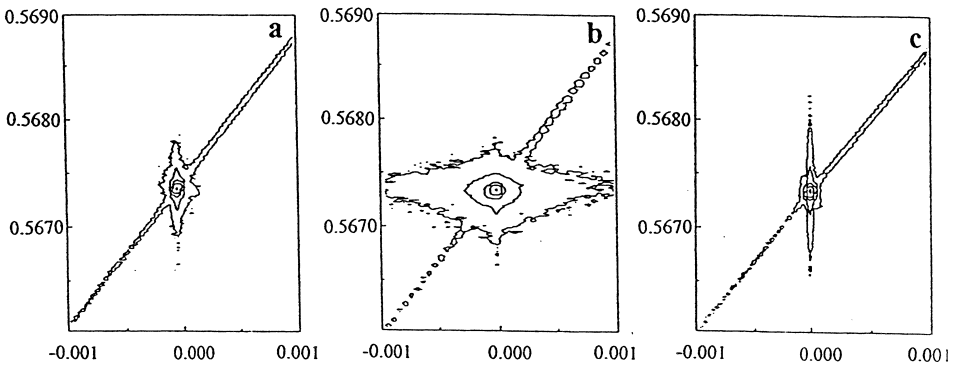

Fig. 3. RSMs of samples: HT–HP treated AI (a) and BI (b) as well as of AI, annealed at 10^5 Pa (c).

3), X-ray diffuse scattering from the B samples is distinctly different from that of the non-irradiated A samples.

Neutron irradiation followed by out-annealing resulted in increased intensity of diffuse scattering and in larger defect sizes (Table 2). Diffuse scattering was less pronounced for the HT–HP treated B samples; the defect dimensions decreased as compared to that for the untreated samples.

However, they still remained larger for the irradiated samples than that for the unirradiated ones. Influence of the HT–HP treatment on the defect dimension and diffuse scattering was more pronounced than that of annealing (Tables 2 and 3, Figs. 2 and 3). Decrease of c_0 for the B samples subjected to the HT–HP treatment can result from the stress-induced precipitation of oxygen on the nucleation centre for oxygen precipitation still present in Cz-Si out-annealed at up to 1040 K. Such nucleation centres are activated at HT–HP [5]. In effect of neutron irradiation with out-annealing, small structural irregularities are created, not observed on the IR spectra but 'serving' as the nucleation centres for stress-induced oxygen precipitation. In effect of the HT–HP treatment of B samples, the comparatively small oxygen-containing defects in a high concentration (as compared to the A samples) are created. The observed decrease of defects dimension is related probably to the decrease of strain fields surrounding the defects. It means that strain at the defect/Si matrix boundary is modified for the 'transformed' (in effect of the HT–HP treatment) defects, probably because of changed composition of the defect material creating lower a misfit (in relation to the Si matrix).

Acknowledgements

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