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HIGH PRESSURE – HIGH TEMPERATURE TREATMENT TO CREATE OXYGEN NANO-CLUSTERS AND DEFECTS IN SINGLE CRYSTALLINE SILICON

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Abstract. Effect of enhanced hydrostatic pressure (HP) on oxygen clustering in as-grown Czochralski silicon (Cz-Si) treated at up to 1000K – 1.6 GPa as well as on creation of defects in Cz-Si with SiO_x precipitates, HP treated at 295K – 2 GPa and at 1580K – 1 GPa, has been investigated by infrared spectroscopy, electrical, photoluminescence and related structure – sensitive methods. Treatment of Cz-Si at 720–1000K resulted in enhanced generation of oxygen – containing nano-clusters exhibiting thermal donor activity while the HP treatment at 295K and 1580K – in creation of some additional defects (non-radiative recombination centres). Above effects are related to HP – induced creation of nucleation centres for oxygen clustering in initially "defect free" Cz-Si at 720–1000K and to generation of nano-defects at the SiO_x/Si boundary in Cz-Si containing oxygen precipitates.

1. INTRODUCTION

Single crystalline Czochralski-grown silicon, Cz-Si, containing, as an unavoidable impurity, interstitial oxygen atoms, O_i, in a concentration up to above $1\cdot10^{18}$ cm^{-3}, is the basic semiconductor used in microelectronics. During its processing at enhanced temperature, HT, typically at atmospheric pressure (10^5 Pa), oxygen interstitials are subjected to different transformations. Such transformations are related to the fact that Cz-Si at room temperature represents an over-saturated Si-O solid solution. At HT, when oxygen atoms become to be sufficiently mobile, it occurs progressive clustering and precipitation of O_i, strongly dependent on initial concentration of oxygen interstitials, c_o, on temperature and time of annealing, its sequence, etc. At even higher temperatures (>1400K), the Si-O solid solution becomes to be under-saturated again, and the oxygen-containing clusters and precipitates tend to dissolve in the silicon matrix.

Oxygen clustering/precipitation at HT has been studied intensively for many years [1-3]. Schematic (simplified) character of oxygen impurity transformation in Cz-Si at annealing is presented in Fig. 1.

The temperature-induced oxygen clustering and precipitation are concomitant with stress [4], e.g. at the SiO_x precipitate / Si matrix boundary. This stress is related first of all to the larger volume (in comparison to that of the host Si atoms) of clustering / precipitating oxygen atoms; other reason of internal stress is the difference in thermal expansion coefficients of SiO_x and of Si.

The stress can be changed by subjecting Cz-Si to the treatment / annealing at enhanced pressure of ambient (HT–HP treatment) [5-7]. In effect of the HP–HT treatment of Cz-Si with previously created oxygen precipitates (Fig. 1f), the misfit, ε, and so the shear stresses at the SiO_x/Si boundary are subjected to changes, as it follows [8] from Eq. 1:

$$\varepsilon = \varepsilon_0 + \frac{K_{SiO_x}}{3K_{SiO_x} + 4G_{Si}} \times \left[\Delta T(\beta_{SiO_x} - \beta_{Si}) + HP\left(\frac{1}{K_{Si}} - \frac{1}{K_{SiO_x}} \right) \right], \quad (1)$$

where: ε_o – initial misfit, K – bulk modulus of SiO_x or Si; G_{Si} – shear modulus of Si, β – volume thermal expansion coefficient (the bottom indexes denote the material), and $\Delta T = T_{exp} - 300K$.

In effect, the HT-HP treatment can result in creation of additional defects at the SiO_x/Si boundary [9]. For the case of treatment at room temperature or for short-time treatments at higher temperatures, such additionally–created defects remain to be present at vicinity of the place of their creation.

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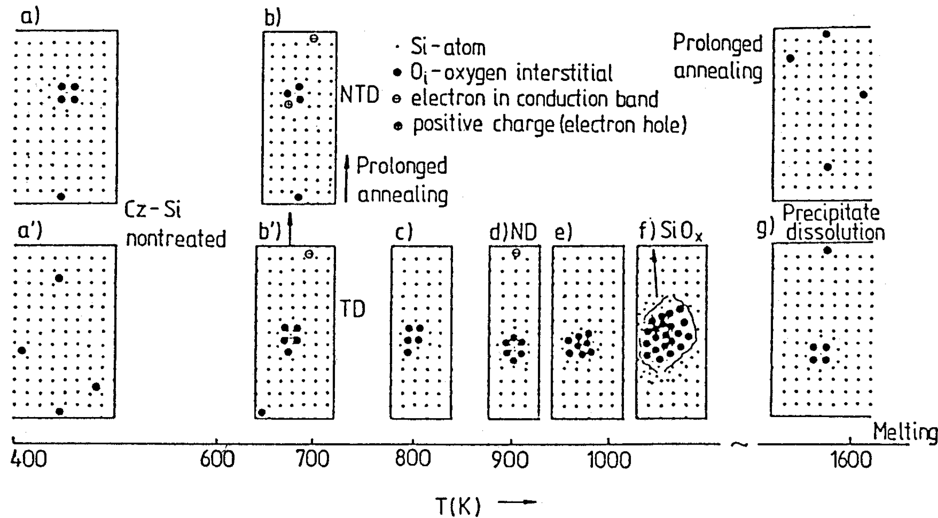

Fig. 1. Schematic draw of O_i transformation in Cz-Si in effect of annealing. TD — means thermal donor; NTD — new thermal donor; ND — new donor. Larger oxygen—containing precipitates composed of sub-stoichiometric SiO_{2-x} are created mostly in effect of consecutive annealing: at 720—1000K to create nucleation centres for oxygen precipitation, NC's, and at 1100—1400K — to cause massive oxygen precipitation on before-generated NC's.

As it follows from Fig. 1, annealing of oxygen–containing Cz-Si at comparatively low temperatures, 720–1000K at atmospheric pressure results in gradual clustering of O_i with creation of oxygen–containing nano–clusters, often with donor activity. Enhanced stress at annealing results in strong enhancement of oxygen clustering [10], most probably in effect of stress–stimulated creation of nucleation centres for oxygen clustering, NC's [11], while O_i diffusion at HT–HP seems to be retarded [12].

Effect of enhanced (hydrostatic) pressure of ambient on creation of oxygen nano-clusters in Cz-Si annealed / treated at 720–1000K (Fig. 1a-e) and on creation of nano-defects at 295, 1580K in Cz-Si containing comparatively large, preliminary created oxygen precipitates (of SiO_{2-x} composition, Fig. 1f) was investigated in the present work. Special emphasis on stress–induced creation of oxygen–silicon nano–clusters and defects has been put in this work while referring to earlier papers dealing with the HT–HP treatment effect on the Si–O system, e.g. [4, 5, 7, 8, 10-14].

2. EXPERIMENTAL

The Si samples of about 12x8x0.6 mm^3 dimension were cut from single crystal Cz-Si wafers of (001) orientation and interstitial oxygen concentration $c_o = (8 - 12)\cdot 10^{17}$ cm^{-3} (typical wafer diameter was 100 mm). To create oxygen clusters and comparatively large (>50 nm) SiO_x precipitates, some Cz-Si wafers were subjected to sequential pre-annealing at 10^5 Pa for up to 40 hrs. Typical pre-annealing conditions and some sample features are listed in Table 1. Most samples were prepared from the "A" wafers (Table 1) with initial c_o=(11–12)$\cdot 10^{17}$ cm^{-3}.

The Cz-Si samples were annealed / treated (at up to 1000K) under argon / helium hydrostatic pressure at up to 1.2 (1.6) GPa in specially designed high temperature – pressure apparatus (Fig. 2). The H and I samples (with large precipitates) were subjected to cyclic (3 cycles) HP treatment at 295K – 2 GPa in *n*-isopentane (quasi-hydrostatic conditions) and to the HT–HP treatment at 1580K – 1 GPa for 5 min.

Before and after pre-annealing and the treatment, the interstitial oxygen concentration, c_o, was measured by Fourier Infrared Spectroscopy (FTIR). Sample conductivity and concentration of carriers, N_n or N_p, was determined by the four point probe and CV methods, while the kind and density of defects – by etching in the Yang solution followed by optical microscopy as well by observation in transmission electron microscope (TEM). Photoluminescence (PL) spectra were recorded at helium temperatures using argon laser excitation (λ_{ex}=488 nm). Some sample characteristics were determined by X-ray methods.

3. RESULTS AND DISCUSSION

Effect of HT–HP Treatment at 720–1000K on Creation of Oxygen–Containing Nano-Clusters. The initial (as-grown) Cz-Si samples with $c_o = (8–12)\cdot 10^{17}$ cm^{-3} were free of defects detectable by TEM as well as by optical microscopy after chemical selective etching. However, in the case of samples with the highest O_i content, pre-annealing at 920–1000K – 10^5 Pa resulted in creation of nano–defects [14], recognisable by selective etching (Table 1) as well as TEM. The presence of a very large density of small dislocation loops of 10–30 nm size was detected for the E and G samples after pre-annealing (Fig. 3a, b).

Table 1. Designation of samples, pre-annealing conditions (at 10^5 Pa), c_o, carrier concentration, *N*, and total density of defects, *d*.

Sample	*Pre-annealing,* 10^5Pa [K, hrs]	c_o [$\cdot 10^{17}$, cm^{-3}]	*Conductivity and N* [$\cdot 10^{15}$, cm^{-3}]	*d* [cm^{-2}]
A	–	11 – 12	p, 1.9	–
B	720, 10	11.3	n, 2.2	$4\cdot 10^3$
C	720, 40	10	n, 5.1	$9\cdot 10^3$
D	920, 10	11.8	p, 1.3	$4\cdot 10^3$
E	920, 20	11.9	p, 1.8	$3.5\cdot 10^3$
F	920, 40	11.5	p, 0.7	$3\cdot 10^3$
G	1000, 20	8.5	p, 1.5	$1.2\cdot 10^6$
H	920,20+1320K,20	6	p, 2.0	$3\cdot 10^6$
I	1000,20+1320K,20	3.2	p, 2.0	$1.5\cdot 10^6$

The O_i concentration in the samples subjected to the HT – HP treatment was dependent on temperature and pressure as well as on pre-annealing conditions, decreasing typically with HT and HP (Fig. 4). It did not concern, however, the sample treated at 920K, for which c_o increased slightly with HP.

The annealing/treatment at 720–1000K – (10^5 Pa – 0.1 GPa) results in some drop of c_o (compare Table 1 and Fig. 4). This effect is related to structural inhomogeneities present "from the very beginning" in the samples (Fig. 1a, Fig. 3a,b) and to precipitation of oxygen on them (very limited because of low mobility of O_i at $\leq$1000K). Oxygen clustering occurs simultaneously, creating small clusters of gradually growing (with temperature and time of annealing / treatment) dimension, still (up to 1000K) below about 10 nm. Such small oxygen clusters were not detected by optical observation after selective chemical etching even for the Cz-Si samples treated at 1000K (*d* not dependent on HP, Table 2) as well as by TEM. Electrical measurements of such samples indicate, however, strong dependence of carrier concentration on HP (Figs 5 and 6).

The carrier (electron) concentration change, $\Delta N_n = N_{HT\text{-}HP} - N_{initial}$, for Cz-Si samples treated at 720K–HP for 10 hrs is presented in Fig. 5 (compare Table 1). Increased concentration of electrons in conduction band is connected with creation of thermal donors, TD's [10, 11]. The initial, as-grown A sample (see Table 1 and curve 1 in Fig. 5) changed its conductivity type in effect of annealing / treatment at 720K and so of creation of TD's. The N_n value for the sample treated at 1.5 GPa was four times higher than for that annealed at 10^5 Pa, the difference in TD's concentration for such samples was equal to about $1\cdot 10^{16}$ cm^{-3} while the respective difference of c_o was equal to about $1\cdot 10^{17}$ cm^{-3}. It allows to estimate the number of oxygen atoms contained in the cluster exhibiting the TD's activity, as equal to about 10.

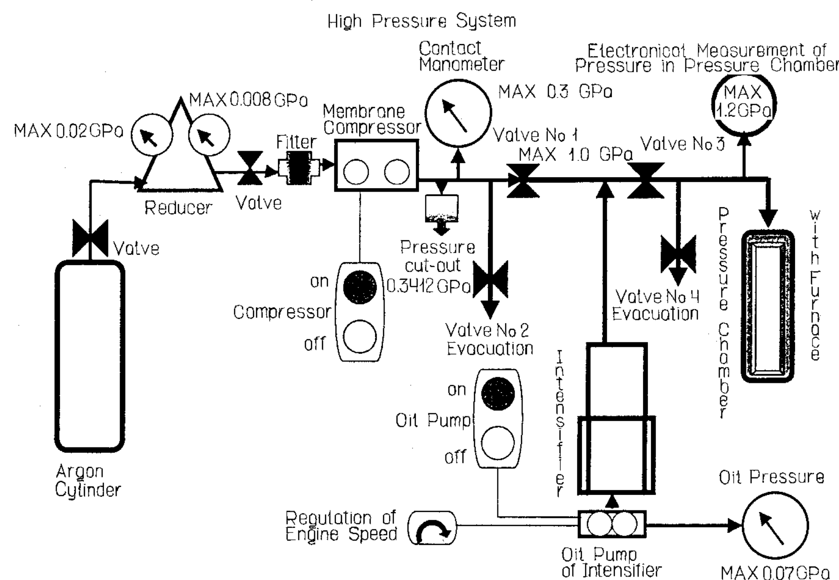

Fig. 2. Schematic draw of the system for HT—HP treatment. Treated samples are placed in the 35 mm high alumina crucible of about 10 mm diameter.

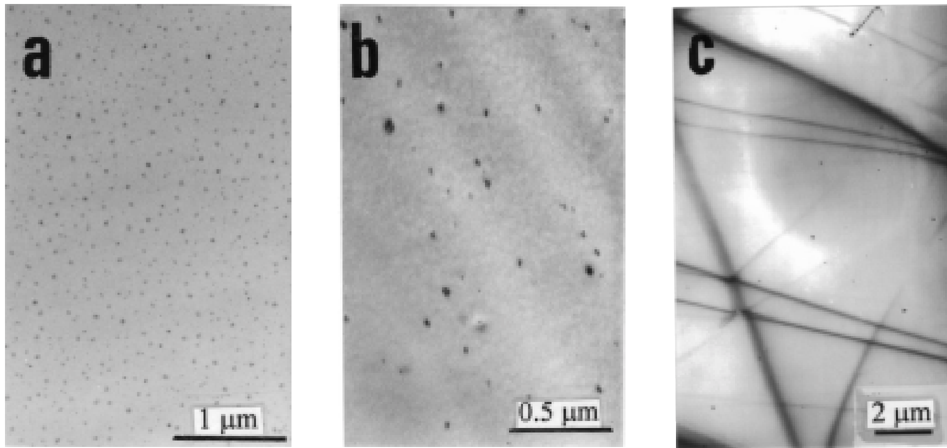

Fig. 3. TEM images of the starting samples (subjected to pre-annealing at 10^5 Pa): a — sample pre-annealed at 920K (E in Table 1); b — at 1000K (G in Table 1); c — at 920K + 1320K (H in Table 1).

It is worth to note that the samples pre-annealed at 720K and so with considerable "initial" concentration of TD's (samples B and C) and at 920K (and so with some "initial" concentration of new donors, ND's, sample D, Table 1) indicated similar ΔN_n dependence on HP. It suggests that the mechanism of TD's creation at 720K–HP differs from that accepted for TD's creation at 10^5 Pa (associated with enhanced mobility of O_i just at about 720K [1]).

The I sample (subjected to sequential pre-annealing, the final step at 1320K), indicated no TD's creation in effect of annealing / treatment at 720K (curve 5 in Fig. 5).

The changes of concentration of holes, calculated as $\Delta N_p = |N_{HT\text{-}HP} - N_{initial}|$ for the Cz-Si samples treated at 920K–HP for 10 hrs, are presented in Fig. 6 (compare Table 1). Decrease of the N_p value is caused by compensation of holes by ND's created in effect of annealing / treatment at 870–920K [1, 10, 11].

The initial, as – grown A sample (see Table 1 and curve 1 in Fig. 6) indicate decrease of N_p in effect of annealing / treatment at 920K and so of creation of ND's. The N_n value (concentration of ND's created in effect of the treatment) for the sample treated at 1.2 GPa was about nine times higher than for that annealed at 10^5 Pa for the same time (10 hrs). Comparing increase of the ND's concentration for the A sample treated at 920K–1.2 GPa with that annealed at 10^5 Pa (curve 1 in Fig. 6), and the c_o data from Fig. 4 (no change of c_o within experimental uncertainty, $\pm 0.5\cdot10^{17}$ cm^{-3}), one can estimate that oxygen clusters, "supplying" one electron into the Si conduction band, are composed of about one hundred oxygen atoms.

As it follows from Fig. 6, the maximum ND's concentration was obtained for Cz-Si samples treated at HP≈1.2 GPa (for 10 hrs).

The curve 4 in Fig. 6 corresponds to the C sample, pre-annealed at 720K for 40 h. Pronounced increase of the compensating electrons concentration with HP is related rather to HP-suppressed "killing" of TD's

Table 2. Density, d [cm^{-2}], of saucer pit defects (SPD), of prismatic dislocation centres (PDC) and of oxygen precipitates determined by optical observation after etching in the Yang solution for p-type Cz-Si sample with initial c_0=7.8·10^{17} cm^{-3} treated at 1000K for 5 hrs.

HP	d_{SPD}	d_{PDC}	d_{OP}
10^5 Pa	$2.5\cdot10^4$	–	–
10^7 Pa	$1.3\cdot10^4$	–	–
0.1 GPa	$1\cdot10^4$	$3\cdot10^2$	$2.5\cdot10^2$
0.6 GPa	$7\cdot10^3$	–	–
1.0 GPa	$1\cdot10^4$	$3\cdot10^2$	–

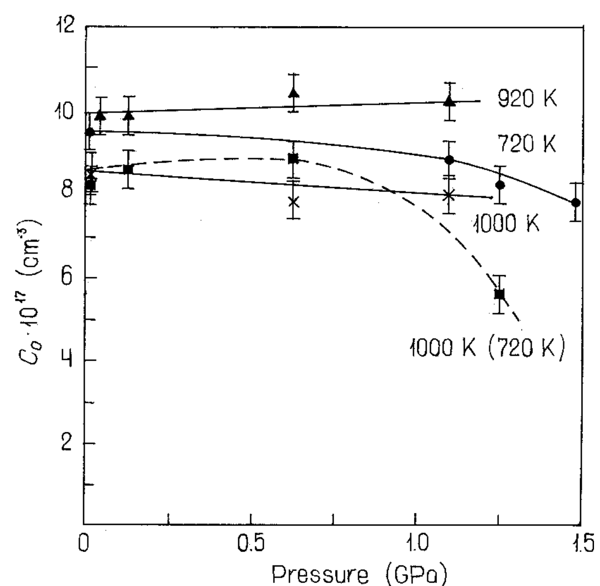

Fig. 4. Dependence of intersitial oxygen concentration, c_o, on temperature and pressure of treatment for samples A (Table 1) and for sample C (dashed line). Treatment time — 10 hrs; treatment temperatures are marked.

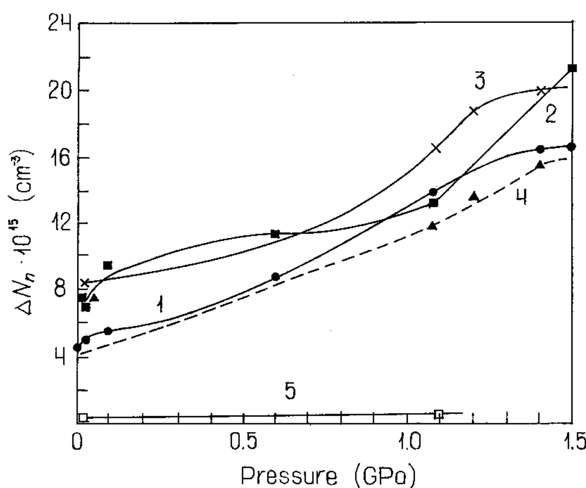

Fig. 5. Electron concentration change, $\Delta N_n = |N_{HT\text{-}HP} - N_{initial}|$, as a function of HP for Cz-Si samples treated at 720K–HP for 10 hrs: 1 — sample A; 2 — sample B; 3 — sample C; 4 — sample D; 5 — sample I (Table 1).

(being stabile just at about 720K) at 920K while stress–stimulated creation of ND's is also participating in observed effect.

The PL spectra (taken at 295K) of the Cz-Si samples annealed / treated at 720K and 870K are presented in Fig. 7. Especially the HP–treated samples indicate presence of the PL band at 0.79 eV; most interesting is the appearance of this (very weak) band for the sample treated at 870K–1.2 GPa, not reported previously. Contrary to earlier observation [15], the intensity of this band increased with HP. It has been suggested [15] that the "0.79 eV defect" represents one particular kind of TD's (in fact, different kinds of species are responsible for the TD's activity). So presence of this band can be considered as indication that just creation of specific cluster / defect is favoured at particular conditions.

The mechanism of TD's creation is still not established finally, even for TD's produced at 10^5 Pa [1]. Our earlier FTIR results have suggested that the TD's created under enhanced stress conditions are identical, as it concerns IR absorption, with the "traditional" TD's created at atmospheric pressure (thermal double donors, TDD's [11]). Other measurement [16] confirmed, however, also the stress – stimulated creation of shallow TD's at $\approx E_c - 0.035$ eV and of deep donors at $\approx E_c - 0.1$ eV. It of high probability that the "pressure stimulated TD's" are generated by the mechanism proposed in [11]. Accordingly to it, TD's (as well as ND's ad other oxygen clusters) are nucleated on "initially existing" structural irregularities [17] in Cz-Si, e.g. within clouds of O_i's, activated at HP. So different TD's and ND's would be produced at HP: the "atmospheric ones" and the "HP ones", in different proportions. Such suggestion is confirmed by phenom-

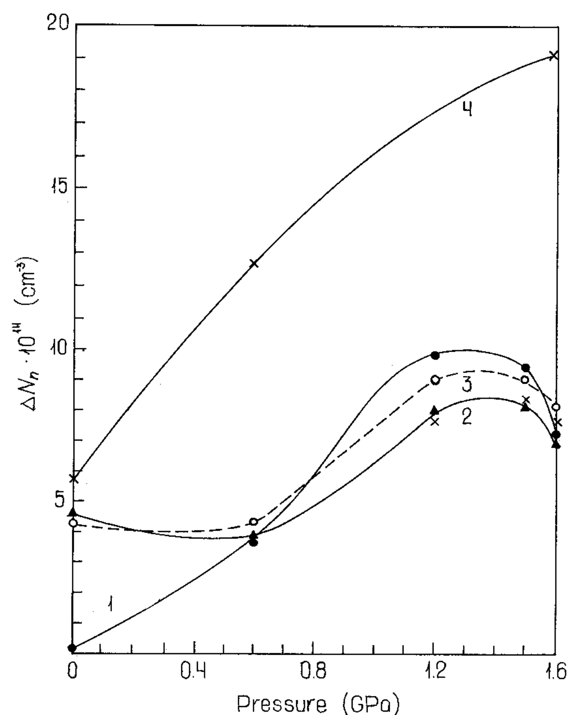

Fig. 6. Hole concentration change, $\Delta N_p = |N_{HT\text{-}HP} - N_{initial}|$, as a function of HP for Cz-Si samples treated at 920 °K – HP for 10 hrs: 1 — sample A; 2 — samples D (triangles) and F (crosses); 3 — sample E; 4 — sample C (Table 1).

enon stated in this work: "additional" TD's (small oxygen nano-clusters) were generated at HP even in the case of samples subjected to prolonged pre-annealing at 720K–10^5 Pa (Figs. 5-7). Creation of "HP TD's" did not occur in the case of sample in which most oxygen interstitials precipitated in effect of sequential pre-annealing (e.g. sample 5 in Fig. 5) and so above mentioned very small structural irregularities were removed (out-annealed). Still the above explanation of HP effect on creation of oxygen – containing nano – clusters in Cz-Si remains to be of qualitative character and so demands experimental and theoretical confirmation.

Effect of Cyclic HP and of Short – Time HT – HP Treatments on Cz-Si Containing Large Oxygen Precipitates.

The Cz-Si samples subjected to two-step pre-annealing (H and J in Table 1) were chosen for investigation of stress–induced creation of defects at the oxygen precipitate/Si matrix boundary. Such samples contained rather large oxygen precipitates, as revealed by TEM (Fig. 3c). Concentrations of oxygen precipitates was lower in the sample I while their dimensions were larger as that for the H sample, as it follows also from the lower value of O_i content remaining to be present in the Si crystal lattice after pre-annealing. It must be stressed that oxygen precipitates / defects of different kinds and sizes are created and so co-existing in the

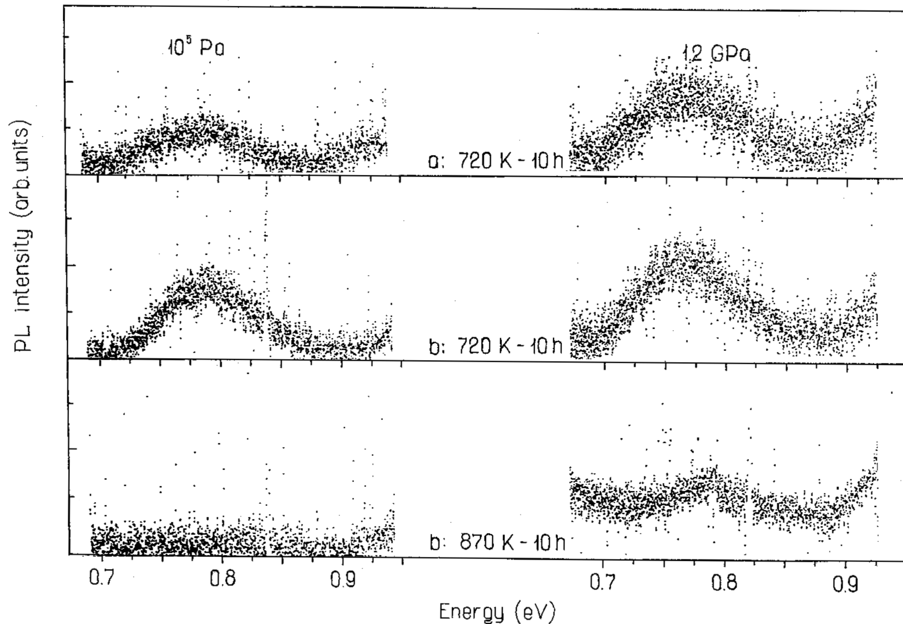

Fig. 7. PL spectra of Cz-Si samples annealed at 10^5 Pa and treated at 1.2 GPa for 10 hrs at 720K and 870K: a — initially p — type Cz-Si with $c_0=8\cdot10^{17}$ cm^{-3}, pre-annealed at 720K — 10^5 Pa for 96 hrs; b — as-grown n-type Cz-Si samples with initial $c_0=9\cdot10^{17}$ cm^{-3}. PL was measured at 295K.

pre-annealed Cz-Si samples. Just oxygen precipitates were most numerous in the H and I samples, while their mean size was larger in the I sample.

PL spectra of the H–type samples, as–prepared (pre-annealed) and treated at 295K – 2 GPa (3 pressure cycles) are presented in Fig. 8. The bands at 0.81 eV, 0.87 eV and 0.95 eV most probably correspond to dislocation–related *D*1, *D*2 and *D*3 lines [7]; their intensities decrease slightly in effect of the HP treatment.

PL spectra of the H samples annealed/treated at 1580K are presented in Fig. 9 (no marked transformation of O_i is expected to occur during the treatment for 5 min.). The intensities of *D*1, *D*2 and *D*3 lines decreased markedly in effect of the treatment at HP (compare the spectra 1 and 2, the first one in Fig. 9 was enlarged).

Presence of the dislocation–related PL lines in the as-prepared H sample confirmed its structure: the sample contained oxygen precipitates (prismatic dislocation centres, PDC, emitting dislocation loops, of $2\cdot10^3$ cm^{-2} density, as it followed from chemical selective etching). In effect of the cyclic HP treatment and especially of the short–time HT – HP treatment the intensity of dislocation–related PL lines decreased. It can be considered as an evidence of stress–stimulated creation of some small (point–like?) defects acting as non–radiative recombination centres. Such defects are most probably created at the SiO_x / Si boundary at HP (HT-HP) [18] because the misfit value at this boundary is reaching (for the largest precipitates) the critical value [9] for creating of "additional" defects (Eq. 1).

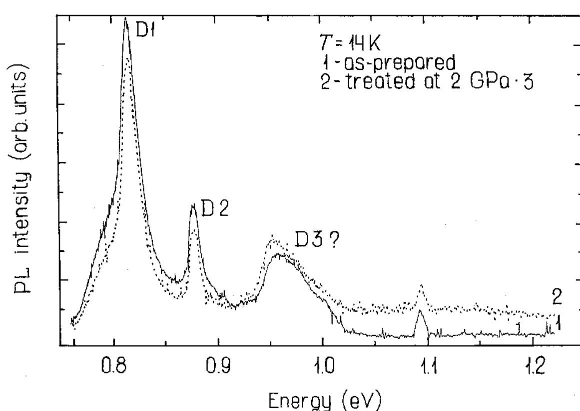

Fig. 8. PL spectra of Cz-Si samples (H in Table 1): 1 — as-prepared; 2 — treated at 295K — 2 GPa (3 pressure cycles).

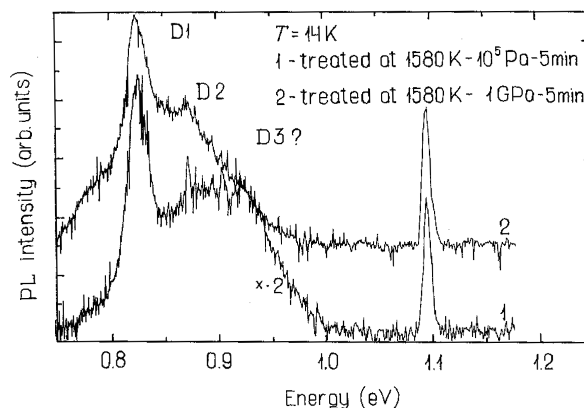

Fig. 9. PL spectra of Cz-Si samples (H in Table 1): 1 — annealed at 1580K — 10^5 Pa for 5 min.; 2 — treated at 1580K — 1 GPa for 5 min.

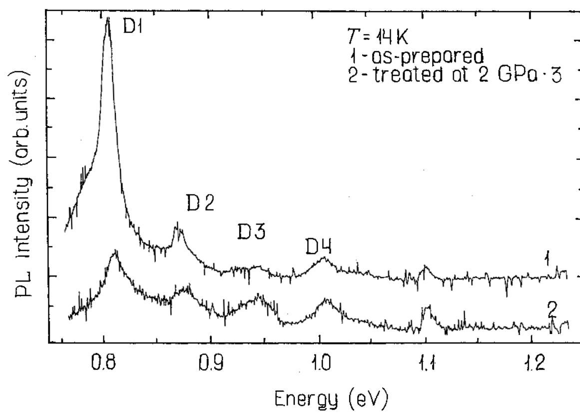

Fig. 10. PL spectra of Cz-Si samples (I in Table 1): 1 — as-prepared; 2 — treated at 295K — 2 GPa — (3 pressure cycles).

Creation of such additional defects occurs more easily at HT because the critical misfit value for creation of defects would be lower at HT.

PL spectra of the as-prepared (1) and of the treated (2) at 295K – 2 GPa (3 pressure cycles) I samples (with the largest oxygen precipitates) are presented in Fig. 10. The HP treatment caused marked decrease of intensity of the *D*1, *D*2, *D*3 and *D*4 dislocation–related lines. Explaining this effect by creation of non-radiative recombination centres is even more straightforward for the I sample because it contained more large defects fulfilling, at HP, the conditions for creation of additional defects at the SiO_x / Si boundary.

The structural perfection of such samples (with comparatively large SiO_x defects) was even more worsened in effect of the short – time HT – HP treatment at 1580K as it follows from X-ray measurements (Table 3). The value of static Debye-Waller factor, L_{660}, increases and that of X-ray anomalous transmission, I_a, decreases for the increased concentration of defects [8, 19]. The treatment at 1580K – 1 GPa resulted in slight improvement of crystal lattice perfection of the G sample (containing small oxygen nano–clusters created in effect of pre-annealing at 1000K). Contrary to that, the same treatment of the I sample (containing much larger defects) resulted in pronounced worsening of structural perfection.

Above presented results (and also that in some earlier works [8, 18]) can be considered as a proof of the HP-induced creation of defects on before–created oxygen–containing precipitates in Cz-Si. Still additional experiments are needed to answer emerging questions (e.g. would it be possible to reach, in direct experiments at 295K – HP, conditions for creation of "additional" defects at all oxygen clusters / Si matrix boundaries, in accordance with the criterion A [9]).

Table 3. Effect of HT — HP treatment (for 5 min.) on c_0, static Debye — Waller factor L_{660} and on X-ray anomalous transmission I_a for pre-annealed Cz-Si samples (see data for samples G and I, Table 1).

Sample (Table 1)	HT - HP	c_0 [$\cdot 10^{17}$cm^{-3}]	L_{660} [$\cdot 10^{-3}$]	I_a [arb.units]
G	1580K–10^5Pa	8.6	–	110
G	1580K–1GPa	8.6	–	120
I	1580K–10^5Pa	8.6	26	78
I	1580K–1GPa	7.9	33	67

CONCLUSIONS

High temperature – high pressure treatment of oxygen – containing single crystalline Czochralski silicon, Cz-Si, makes it possible to create different oxygen – containing nano – clusters and defects in the volume of this material. In spite of extended experimental activity, some important (also for application in microelectronics) questions remain to be answered.

On the other hand, hitherto obtained results on Cz-Si suggest usefulness of the elaborated HT – HP treatment approach for solving similar problems in material science.

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