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Effect of high temperature-pressure on silicon surface layers in Si:H,He (Si:He) and Si:N

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Enhanced hydrostatic pressure (HP, up to 1.1 GPa) applied at 720 K to Czochralski silicon, implanted with hydrogen and helium (Cz-Si:H,He) or with nitrogen (Cz-Si:N) to a dose $D = 1\text{x}10^{17}\text{cm}^{-2}$ at energies E = 135-150 keV, and to reference low dose implanted ($D = 2\text{x}10^{16}\text{cm}^{-2}$) floating zone grown silicon (Fz-Si:He and Fz-Si:N), exerts pronounced effect on the structural perfection of the near surface Si layers. These layers were more disturbed in the case of the highest applied pressures, especially in Cz-Si:H,He, while creation of thermal donors was HP-stimulated both in Cz-Si:H,He and Cz-Si:N. Qualitative explanation of pressure induced effects is proposed.
Keywords: silicon surface layers, hydrostatic pressure, implantation, defects.

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I. Introduction

The silicon based structures (Si:He and Si:H, He) prepared by implantation of helium or hydrogen and helium ions into single crystalline silicon, Czochralski (Cz-Si) or floating zone grown (Fz-Si), are of considerable interest for microelectronics, mostly because their (as well as of Si:H) application for Smart Cut processing and gettering activity of the implantation disturbed areas [1]. Low dose (D) nitrogen implantation into silicon has been used to control the gate oxide thickness [2]; the nitrogen implanted silicon (Si:N) is recently considered as important component of the silicon-on-insulator structures (SOI).

In effect of implantation, and depending on the kind of implanted ion, on D and ion energy (E), as well as on other implantation and annealing parameters, the specific layered structures are produced. The Si near-top layer (typically ≤ 1 μm thick) remains relatively undisturbed while the crystallographic perfection of the deeper Si areas, containing most implanted atoms (with concentration peaking near the implanted ion projected range, R_p) becomes to be strongly disturbed or even amorphous.

In the case of Si implanted with helium or / and hydrogen, the implanted atoms are gradually removed at annealing so the sample can regain, partially or fully, its original structural perfection. Contrary to that, the implanted nitrogen atoms remain in the crystal lattice also after annealing, creating nitrogen - enriched buried layer embedded into the sample.

The top near surface Si layer in annealed Si:H,He and Si:N can retain high crystallographic perfection while the deeper one, containing most of the implanted atoms, remains to be strongly disturbed. The implanted structures are usually processed under atmospheric pressure (10^5 Pa). As it has been reported recently [3], enhanced hydrostatic pressure of ambient gas (HP) at annealing (HT - HP treatment) affects considerably the properties of such structures, both of the Si top layer and of the buried implantation disturbed areas.

The effect of HT - HP on the near surface Si layers in practically oxygen free Fz-Si and in oxygen containing Cz-Si (concentration of oxygen interstitials in Cz-Si, $c_o \approx 7.5\text{x}10^{17}\text{cm}^{-3}$) implanted with He or H + He or with nitrogen (nitrogen reacts with Si and so creates the N-containing disturbed buried layer), at low dose (D = $2\text{x}10^{16}$ cm^{-2}, Fz-Si:He and Fz-Si:N) and at medium dose (total D = $1\text{x}10^{17}$ cm^{-2}, Cz-Si:H,He and Cz-Si:N) is investigated in the present work. The Fz-Si:He and Fz-Si:N samples prepared by low dose implantation into oxygen - free Fz-Si were investigated mostly for reference purposes. The samples were treated at 720 K - HP. At this temperature not only nitrogen but also He (H + He) remain to be still present in the crystalline lattice at a concentration close to that reached at implantation [4].

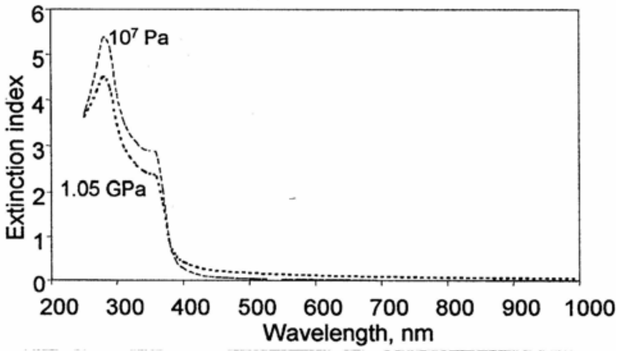

Fig. 1. Dependence of extinction index of near-surface Si layer in Cz-Si:H,He treated at 720 K for 10 h under 10 MPa and 1.05 GPa on wavelength applied in SE.

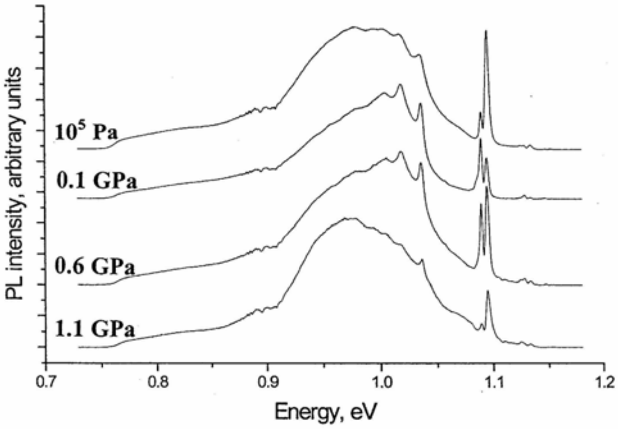

Fig. 2. PL spectra of Cz-Si:H,He samples HT - HP treated for 1 h at 720 K under 10^5 Pa, 0.1 GPa, 0.6 GPa and 1.1 GPa.

II. Experimental

The Si:He, Si:H,He and Si:N samples were prepared by implantation of Fz-Si and Cz-Si wafers (that last of p-type, N_p = 4.3 - 9.5x10^{14} cm^{-3}) with H_2^+, He^+, N^+ or N_2^+ (Table 1). After implantation the wafers were cut into the samples of about 12x8 mm^2 dimension and subjected to annealing / HT - HP treatment (typically at 720 K, for 1 h or 10 h) under 10^5 Pa (N_2 atmosphere) or at HP up to 1.2 GPa in Ar atmosphere [5]. The as implanted and annealed / treated samples were investigated by spectroscopic ellipsometry (SE, at two angles, 65^o and 75^o, λ = 250-1000 nm), photoluminescence (PL, measurements at 6-10 K, excitation with Ar laser, λ = 488 nm) and C-V electrical methods. Some results obtained by X-Ray synchrotron reflection topography (at ID 19 beam line, ESRF) and Transmission Electron Microscopy on sample cross sections (XTEM) are also presented.

III. Results and discussion

Results of investigations of Fz-Si:He, Cz-Si:H,He and Si:N and their discussion are below presented in two sections and summarised in the next chapter.

I. Cz-Si:H,He and Fz-Si:He (reference) samples

As it follows from synchrotron X-Ray reflection topography, the as-implanted Cz-Si:H, He structures (see Table 1) were flat while the reference Fz-Si:He ones - distinctly warped (as often observed for the Fz-grown Si). No defects were resolved in these structures by synchrotron X-Ray topography, both before and after the HT - HP treatment. Most probably this resulted from very high density of the implantation-induced defects and so from overlapping strain fields originating from numerous structural disturbances. As it results from XTEM, the implanted hydrogen and helium atoms in the low temperature (HT ≤ 780 K) treated Cz-Si:H, He samples created continuous implantation disturbed buried layers at about 500 nm depth containing mostly the H- or He- filled cavities / bubbles (see also [6]).

SE measurements confirmed that the treatment of Cz-Si:H, He at 720 K at low pressure (10 MPa) for 10 h resulted in a creation of layered structure with only slightly disturbed 650 nm thick top Si layer and the 190 nm thick buried strongly disturbed layer containing hydrogen and helium; clear interferences were detectable after that treatment. The treatment of Cz-Si:H,He at 720 K under much higher pressure (1.05 GPa) resulted in a markedly decreased wavelength-dependent extinction index (Fig. 1) while the structural disturbances reached almost the sample surface; the SE ψ and Δ characteristics (related also to the presence of buried 200 nm thick implantation-disturbed layer) indicated clear interference pattern.

Annealing / HT - HP treatment of Cz-Si:H, He at 720 K for 1 h resulted in a very wide PL band at 0.9 -

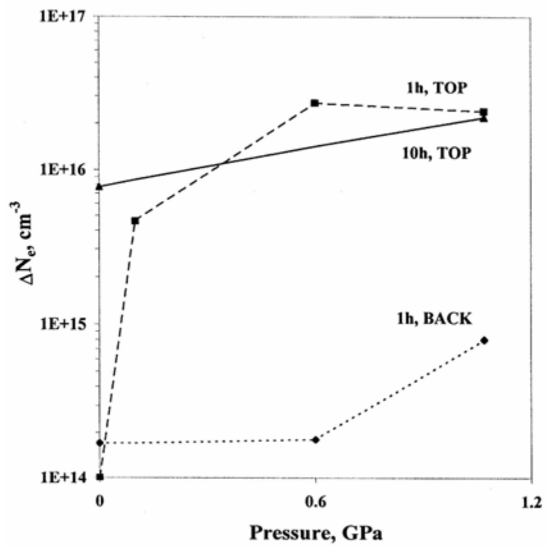

Fig. 3. Dependence of ΔN_e (ΔN_e = $|N_{HT-HP}$ - $N_I|$, where N_{HT-HP} - the carrier concentration after the HT - HP treatment and N_I - the carrier concentration of the as-implanted (initial) sample) on HP applied during treatment of Cz-Si:H,He samples at 720 K - HP for 1 and 10 h, determined at TOP (implanted) and bottom (BACK) sample surfaces.

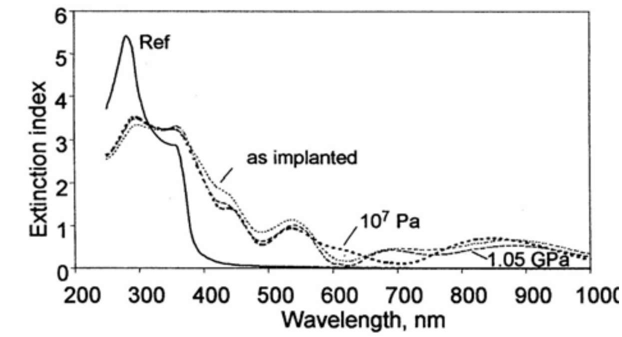

Fig. 4. Dependence of extinction index of near-surface Si layer in Cz-Si:N, as implanted and treated at 720 K for 10 h, under 10 MPa and 1.05 GPa, on λ applied in SE. Typical dependence of silicon extinction index on wavelength (Ref, solid curve) was taken from the J.A. Woolam Co. WVASE 32 program v 3.386, tabulated at UNL (Lincoln University, Nebrasca, USA).

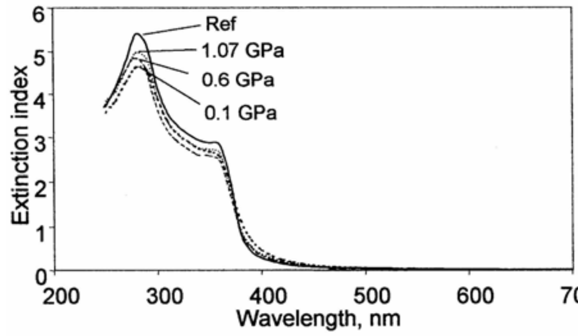

Fig. 5. Dependence of extinction index of near-surface Si layer in Fz-Si:N treated at 720 K for 10 h under 0.1 GPa, 0.6 GPa and 1.07 GPa on λ applied in SE. Typical dependence of silicon extinction index on wavelength (denoted as Ref) is also presented (see Fig. 4 caption).

1.05 eV (indicating also local maxima) and in the sharp PL peak (peaks) at about 1.09 eV, that last related to the interband transition in the B-doped silicon (Fig. 2). The origin of PL peaking at about 0.98 eV can not be explained unambiguously; possibly it is related to the presence of specific oxygen containing centres (the I and A lines at 0.9652 eV and 0.9697 eV [7]) or / and of silicon self-interstitials (the W centre [8] with PL peaking at about 1.02 eV). The dependence of the relative intensity of PL at 1.09 eV on HP (Fig. 2) can be interpreted as a proof of increased concentration of non-radiative recombination centres in the samples treated at 720 K - 0.1 / 1.1 GPa for 10 h.

The similarly treated reference Fz-Si:He samples indicated the presence of PL peaks at about 0.88 eV (the dislocation - related D2 peaks ?) and at 0.93 eV (the dislocation - related D3 peaks). No PL at 0.88 eV was detected for the Fz-Si:He sample treated under 1.1 GPa for 1 h only; that sample indicated the much lowered intensity of PL at 0.94 eV. Comparing the PL spectra of the HT-HP treated Fz-Si:He and Cz-Si:H,He samples with these reported for similarly HT - HP treated Fz-Si:H [9] and Cz-Si:H [10], one can conclude that just the presence of shallower implanted (in comparison to He^+) hydrogen atoms ($R_{pH} < R_{pHe}$, Table 1) exerts dominating effect on the observed PL spectra of Cz-Si:H,He treated at 720 K - HP.

The treatment of Cz-Si:H, He at 720 K - HP results in the dramatically increased concentration of electrons in the conduction band related to creation of oxygen - containing thermal donors (TDs), especially near the sample surface (the data marked as TOP in Fig. 3). The pressure-induced TDs compensate the initially p-type material and convert it into the n-type one after the treatment for 1 h under 0.1 GPa (at the sample top) or under 1.1 GPa (at the sample back); the treatment for 10 h resulted into the mentioned conversion even after the treatment under comparatively low pressure, at 10 MPa. The HP effect on creation of TDs, reported at first for the case of bulk Cz-Si [11], has been confirmed also for Cz-Si:H [12]. One must admit, however, that not only

Table 1.

Characteristics of investigated samples: sample designation, growth method (Czochralski growth, Cz, 001 orientation, or Floating zone growth, Fz, 111 orientation), interstitial oxygen concentration (c_o), kind of implanted ion, energy (E), atomic dose (D), projected ion range (R_p).

Sample designation	Growth method	c_o, cm^{-3}	Implanted ion	E, eV	D, cm^{-2}	R_p, nm
Cz-Si:H,He	Cz	$7.5x10^{17}$	$H_2^+ + He^+$	135 / 150	$5x10^{16} + 5x10^{16}$	700 / 880
Cz-Si:N	Cz	$7.5x10^{17}$	N_2^+	140	$1x10^{17}$	180
Fz-Si:He*	Fz	$<2x10^{16}$	He^+	150	$2x10^{16}$	980
Fz-Si:N*	Fz	$<2x10^{16}$	N^+	150	$2x10^{16}$	360

* Reference samples.

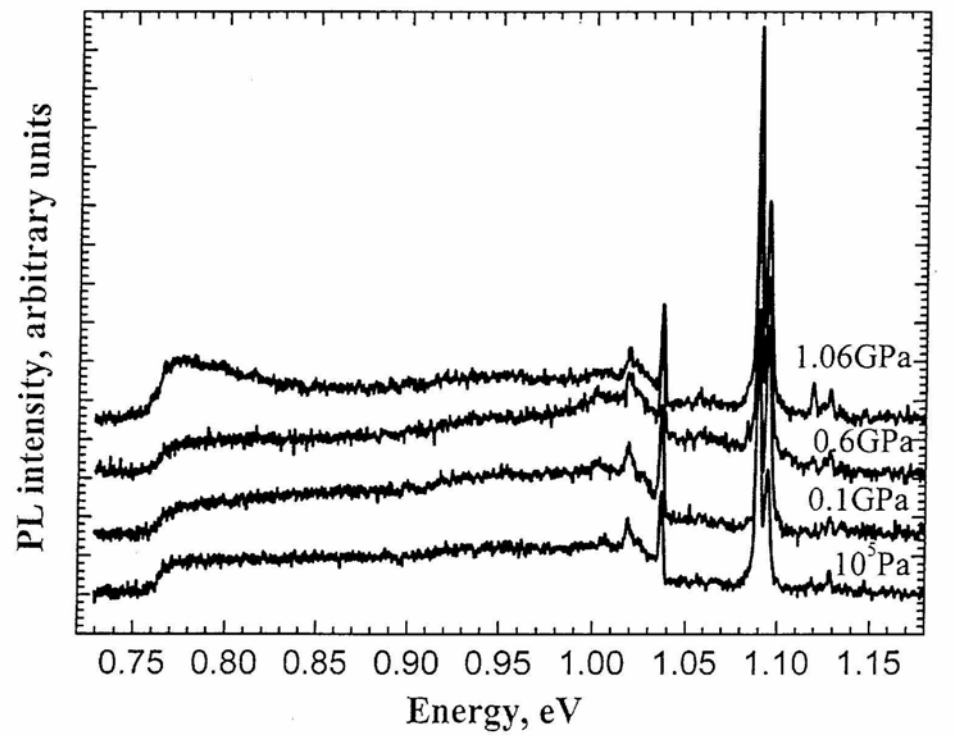

Fig. 6. PL spectra of Cz-Si:N samples HT - HP treated for 1 h at 720 K under 10^5 Pa, 0.1 GPa, 0.6 GPa and 1.06 GPa.

enhanced pressure but also the presence of hydrogen stimulate strongly the creation of TDs [12]. Comparing ΔN_e determined at the Cz-Si:H sample top (containing hydrogen atoms) with that at the sample back (where almost no hydrogen is present), one can judge that HP exerts dominating effect on TDs generation in the near - surface hydrogen - containing sample areas (Fig. 3). By comparing ΔN_e for the samples treated for 1 h with those processed for 10 h, one can speculate that lower ΔN_e (in relation to that for the sample treated for 1 h) detected for Cz-Si:H, He treated at 720 K - HP for 10 h is related to the more disturbed structure of the samples subjected to more prolonged treatment (this worsened crystallographic perfection is also confirmed by the SE and PL data).

II. Cz-Si:N and Fz-Si:N (reference) samples

Similarly to as prepared Fz-Si:He and Cz-Si:H, He, the as-implanted Cz-Si:N samples were flat while the reference Fz-Si:N ones - warped. For the same reason as in the case of Fz-Si:He and Cz-Si:H, He, no individual defects were resolved in these structures by X-Ray synchrotron topography, both before and after the HT - HP treatment. As it results from XTEM, the implanted nitrogen atoms in the HT - HP treated samples create continuous buried layer near R_p; that layer contains also the implantation - induced defects [3]. The Cz-Si:N samples treated at 720 K - HP for 10 h indicate very strong dependence of their complex extinction index on HP (Fig. 4) while the Fz-Si:N ones (implanted with lower nitrogen dose - Table 1) show, after the treatment at 720 K - HP for 10 h, much smaller effect of HP on the refraction and extinction indexes (Fig. 5). The observed SE changes in the last samples indicate on implantation-induced partial amorphization of Si within the implanted area while the structure of Cz-Si:N treated at 720 K - HP for 10 h was strongly disturbed also in the near-surface area: from R_p to the sample surface.

Annealing / HT - HP treatment of Cz-Si:N (Table 1) at 720 K for 1 h resulted in the sharp PL peaks at about 1.09 eV related to the interband transition in the B - doped silicon (Fig. 6) confirming good structural perfection of the samples in spite of high implanted dose of nitrogen. In this respect these samples are of better structural perfection if compared to the similarly treated Cz-Si:H,He samples (Fig. 2). The PL peaks at about 1.02 eV and 1.035 eV (both of unknown origin) do not indicate clear dependence on the treatment pressure. Also the origin of weak PL at about 0.77 eV can not be explained at present.

The Fz-Si:N samples treated at 720 K - HP indicate the PL peaks at about 0.81 eV (the dislocation-related D1 peak) with the intensity increasing with HP (no PL at 0.81 eV was detected for the sample treated under 0.1 GPa) and at about 0.93 eV (the dislocation – related D3 peak). The Fz-Si:N sample treated under even higher temperature (920 K) and the highest pressure (1.1 GPa) indicates the presence of PL only at about 0.87 eV (the D2 dislocation-related line).

The treatment of Cz-Si:N at 720 K - HP for 1 h results in the HP-dependent increase of electron concentration in the conduction band (Fig. 7), related to HP - stimulated creation of TDs and evidenced by the decreased concentration of holes (still the samples treated at 720 K - HP for 1 h remained to be p -type). The effect of the Cz-Si:N treatment at 720 K - HP for 1 h was the same at the sample surface and its back, which can be

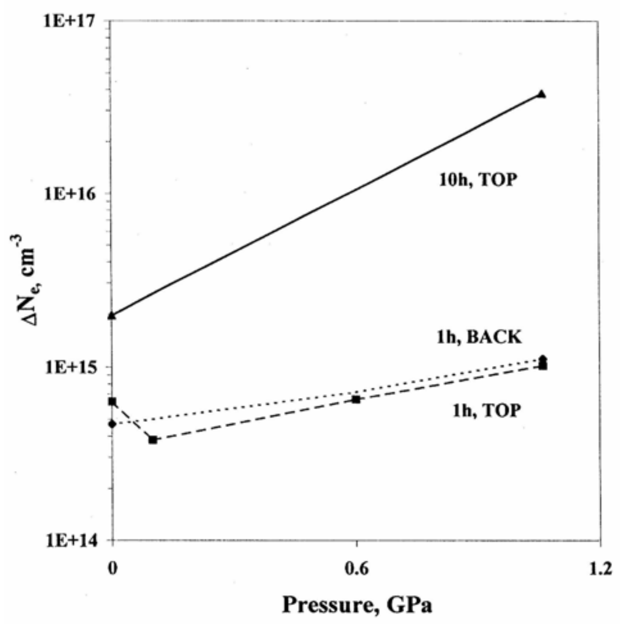

Fig. 7. Dependence of ΔN_e on HP applied during treatment of Cz-Si:N samples at 720 K - HP for 1 and 10 h, determined at top (implanted) and bottom (back) sample surfaces.

considered as an evidence that the TDs creation rate (being to some extent related with the sample structural perfection) is similar at the implanted and non-implanted wafer sides. The treatment at 720 K for 10 h results in the conversion of sample conductivity to n - type related to a creation of TDs in the sample volume.

IV. Summary

Enhanced hydrostatic pressure applied during the treatment at 720 K of Cz-Si:H, He and Cz-Si:N prepared by implantation with the respective ions to a total dose $D = 1x10^{17}cm^{-2}$ at similar energies (E = 135 - 150 keV) exerts pronounced effect on the features of the near surface Si layers. The same has been stated for the reference low dose implanted samples (implantation into Fz-Si). In particular, for Cz-Si:H,He (implanted with H and He effusing gradually during the treatment) it has been stated that:

- extinction index of Si surface layer decreases with HP (SE results);
- PL results can be interpreted as indicating on enhanced creation of small defects / structural disturbances in the surface layers after the treatment under growing HP; the PL spectra are strongly dependent on D while the implanted hydrogen atoms are mainly responsible for observed PL;
- the presence of hydrogen in the near-surface layer affects markedly the pressure stimulated creation of thermal donors.

In the case of Cz-Si:N (implantation with N remaining in the crystal lattice during the treatment at 720 K, with probable creation of Si_3N_4 [13]) it has been stated that the treatment at 720 K - HP results in:

- almost pressure independent extinction index being, however, much lower in comparison with that for reference Si; this can be interpreted as an indication of marked structural disturbances (the presence of implanted nitrogen profile tail) in the as implanted samples as well in these treated at 720 K - HP;
- gradual increase of the PL peaks intensity with HP for the Fz-Si:N samples; enhancement of that intensity can be related to a worsened structural perfection of just these samples. However, the similarly treated higher dose implanted Cz-Si:N samples do not indicate mentioned structure worsening.
- HP stimulated creation of thermal donors, non dependent on the presence of nitrogen in the sample surface areas.

The HT-HP treatment of Si:H, He at 720 K results in the retarded out - diffusion of H/He so these atoms create structural defects (gas filled cavities) in the more extended, also near - surface areas of Si:H, He. Moreover, such defects are active in respect of oxygen gettering; in turn the gettering process effects in the enhanced creation of defects, especially in the case of oxygen - containing Cz-Si:H, He [4]. Strongly enhanced pressure - stimulated creation of TDs in Cz-Si:H, He subjected to the short (1 h) treatment is difficult to be explained. Possibly it is related to the joint effect of hydrogen [12] (present in the near surface sample areas in a higher concentration in Cz-Si:H,He subjected to the treatment for 1 h only in comparison with that treated for 10 h) and of enhanced hydrostatic pressure on creation of TDs.

The HT- HP treatment of Si:N at 720 K results in even more pronounced worsening of the structural perfection, owing also to the retarded diffusion of nitrogen and to creating of structural defects by these atoms, the process affected by tuning the shear stresses at the buried N-containing layer / Si matrix by hydrostatic pressure.

The presence of nitrogen seems to exert only minor effect on the HP stimulated creation of thermal donors, even within the strongly disturbed nitrogen-containing top layer of the Cz-Si:N samples. The same behaviour has been reported for bulk Cz-Si admixed with nitrogen during crystal pulling [14].

Still above explanation of the HT - HP effect on silicon surface layers in Si:H, He and in Si:N is qualitative and somewhat speculative. More adequate explanation of observed effects in Si:H, He and Si:N would demand future research.

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Ефект висока температура-стиск на поверхневих пластах кремнію у Si:H,He (Si:He) і Si:N

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Підвищення гідростатичного стиску (ГС, вверх до 1,1 ГПа) застосовували при 720 K у кремнії, отриманому методом Чохральського, осадженого в атмосфері водню, гелію (Cz-Si:H,He) або азоту (Cz-Si:N) до $D = 1x10^{17}$см$^{-2}$ при енергіях E = 135-150 кеВ, і до рекомендованої дози осадження ($D = 2x10^{16}$см$^{-2}$) зони топлення при вирощуванні кремнію (Fz-Si:He і Fz-Si:N), тиск прикладали до виникнення ефекту на структурній досконалості у приповерхневих пластах Si. Ці пласти були сильно порушені у випадку найсильніших тисків, особливо у Cz-Si:H,He, в той час як утворення термічних донорів було ТС-стимульоване як у Cz-Si:H,He так і в Cz-Si:N. Пропонується якісне пояснення ефектів, спричинених стиском.