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PRESSURE INDUCED FORMATION OF THE ELECTRICALLY ACTIVE CENTERS IN ELECTRON AND NEUTRON IRRADIATED SILICON

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Abstract. The effect of annealing at 300-500°C under enhanced hydrostatic pressure (up to 1.2 GPa) in argon ambient on the interstitial oxygen aggregation (thermal donors) in Czochralski grown silicon subjected to electron irradical vas investigated by infrared absorption and electrical techniques. Strong presure- and irradiation-enhanced changes in oxygen concentration, formation of the trinsic p-n-junctions, conversion of conductivity type due to formation of the thermal donors and thermal acceptors were found.

1. INTRODUCTION

Annealing at moderate temperatures (300 – 700°C) of silicon irradiated with electrons or neutrons leads to formation of two types of electrically active centers. The shallow thermal acceptors (TAs), beloning to the first type, are created in irradiated silicon after annealing at moderate temperature [1]. The origin of acceptors and the conditions of their formation are debatable. The well-known thermal donors (TDs) represent the second type of electrically active centers. They are formed in Czochralski-grown silicon (Cz-Si) only because interstitial oxygen is the component of TDs [2]. It has been established that the thermal donors consist of several TD families: for example, of the shallow thermal donors family

with energy of ionisation E_c - $E_i \le 30 \text{meV}$ (STDs), and of the family of classical Thermal Double Donors (TDDs) [3]. Irradiation by electrons or neutrons can affect the TDs formation. Electron irradiation in silicon leads to the creation of point defects only [4], whereas neutron irradiation results, in addition, in the formation of relatively large defect clusters, known as the disordered regions [4]. Introduction of TAs is expected to be more pronounced in neutron-irradiated silicon in comparison with the electron-irradiated one [1]. It is known that enhanced pressure (HP) of the gas ambient during annealing increases the formation kinetics of the oxygen-related TDs [5]. However, the effect of pressure on the TAs formation kinetics in the electron-irradiated silicon is practically unknown. The aim of the present report is to study and compare the HP effect on the TDs and the TAs formation in electron and neutron irradiated Cz-Si.

2. EXPERIMENTAL

P-type Cz-Si samples with a hole concentration p = (6-8)·10¹⁴ cm⁻³ and initial oxygen concentration [O_i]_o = 8·10¹⁷ cm⁻³ were used. The concentration of the interstitial oxygen, O_i, was monitored by measuring the well known absorption band at 1107 cm⁻¹, using a calibration coefficient equal to 3.14×10¹⁷ cm⁻². The electron irradiation was carried out in sets of pulses of 2.5 MeV energy, at a fluence in the range 1·10¹⁴ - 1·10¹⁷ cm⁻². The pulse duration was 400 µs and the current flowing during pulse was in the range 0.12 - 0.14 A. The interval between pulses varied so that the average sample temperature was kept below 50°C. Another group of samples was irradiated with 2 MeV fast neutrons at room temperature, at a fluence of 5×10¹⁶ cm⁻². Then the samples were heat-treated at temperatures between 300°C - 500°C, at atmospheric and enhanced hydrostatic pressure (HT-HP treatment), in argon ambient, in the HP range of 0.1 - 1.2 GPa, for 45 min. - 10 hours.

The carrier concentration was monitoring from high-frequency capacity-voltage, CV, and resistivity measurements. CV measurements were performed with the use of a mercury probe. The probe square was 4.5×10^{-2} mm². Following the HP – HT treatment infrared (IR) absorption measurements with a resolution $4 \text{ cm}^{-1} - 0.5 \text{ cm}^{-1}$ were carried out at 300 K - 11 K over a spectral range of 1500 cm⁻¹ – 200 cm⁻¹ with the aid of a Brucker IFS 113v Fourier Transform Infrared Spectrometer (FTIR).

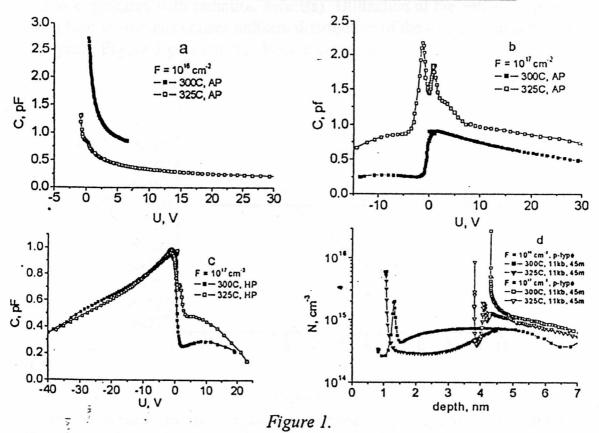
3. EXPERIMENTAL RESULTS AND DISCUSSION

The relative concentrations of the interstitial oxygen, [O_i] in silicon irradiated and HP treated in the temperature range of 300°C - 325°C are given in Table 1. Treatment at 300-325°C of irradiated silicon regularly leads to annealing of the

VO center and TO the formation of the VO₂ defect. A strong decrease in the oxygen concentration in the as-irradiated samples and in the samples annealed at 300-325°C was found despite the fact that the used temperature and time duration of the heat-treatment were suffisient for the annealing of the main part of the electrically active defects (see Fig. 1).

Table 1
The relative concentration of interstitial oxygen in silicon irradiated by electrons and HP treated at temperature of 300°C - 325°C.

Electron fluence	Treatment	$[O_i]/[O_i]_o$
1x10 ¹⁶ cm ⁻²	As irradiated	1
	300°C, 1.1 GPa, 45 min	0.92
	325°C, 1.1 GPa, 45 min	0.69
1x10 ¹⁷ cm ⁻²	As irradiated	1
	300°C, 1.1 GPa, 45 min	0.70
	325°C, 1.1 GPa, 45 min	0.79



Capacity-voltage characteristics (a – c) and carrier profiles (d) of electronirradiated samples annealed at 300 and 325°C for 45 min at different pressures. Values of electron fluence (F) are given in the figures. AP means atmospheric pressure while HP = 1.1 GPa.

Figure 1 presents the CV characteristics OFelectron-irradiated Cz-Si subjected to anneals at atmospheric pressure (AP) and to the HP treatment at 300°C - 325°C. The CV characteristic for the sample irradiated at a fluence of 10¹⁶ cm⁻² is the typical one for p-type silicon (Fig. 1a). Formation of the intrinsic p-n junction(s) is revealed in the samples subjected to electron fluence of 10¹⁷ cm⁻² (Fig. 1b, c). The p-n junctions are formed both after annealing at AP and HP. But the carrier concentration in the p-type silicon was practically not changed after such treatments (Fig. 1d).

Annealing at 450°C leads to the inhomogeneous formation of the TDs and TAs at atmospheric pressure. The domains with p- and n-type conductivity are revealed in the samples irradiated with high electron fluence of 10¹⁶- 10¹⁷ cm⁻² (Fig. 2). The presence of p-type domains and the increase in the hole concentration within domains as a function of annealing time are suggested to be related with the generation of the defect-related thermal acceptors. Formation of the domains with p- and n-type conductivity is correlated with a non-uniform distribution of oxygen. TAs are suggested to be electrically active complexes of TD-like oxygen aggregates with radiation defect(s). Utilization of the enhanced pressure during heat treatments causes uniform distribution of the carrier concentration in the crystal. Figure 3 presents the donor concentration in

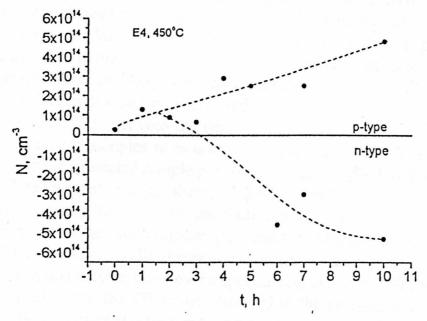


Figure 2.

Carrier concentrations as a function of annealing time obtained from CV measurements for p-type and n-type domains in the sample irradiated with $F = 1 \times 10^{17}$ cm⁻² and annealed at 450°C.

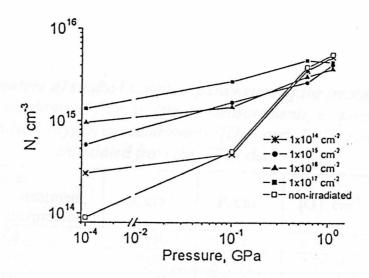


Figure 3.

Donor concentration in silicon irradiated with electrons and treated at 450°C for 10 hours as a function of the pressure. Electron fluences are given in the figure.

silicon irradiated with electrons and treated at 450°C for 10 hours, as a function of pressure. Electron fluence was a parameter. For the samples annealed at atmospheric pressure only domains with TDs were taken into consideration in Fig. 3.

An increase in the TD formation rate with pressure occurs in the non-irradiated crystals. Preliminary irradiation leads to the higher electron concentration at low pressure and to the lower electron concentration in the case of high pressure ~1GPa. The decrease in the electron concentration after HP treatment of electron irradiated samples is most likely connected with the presence of TAs and TDs simultaneously. But in this case both TAs and TDs have uniform distributions in the crystal. The difference between the electron concentrations in the non-irradiated and the irradiated samples is caused by the introduction of TAs. The TAs concentration for the irradiated sample pre-treated with HP of ~ 1 GPa is estimated from Fig. 3 to be equal or larger than $(1-1.5)\times10^{15}$ cm⁻³.

Lets now consider the results for the neutron-irradiated samples. The carrier concentration n, the oxygen concentration $[O_i]$, and the phosphorous concentration P, obtained from resistivity and IR absorption measurements are presented in Table 2.In the IR absorption spectra, taken over a spectral region of 250-550 cm⁻¹ at 11 K, the sharp lines related to the TDDs are observed in the samples heat-treated at 450° C. The total concentration of some specific members of TDDs (v=2-5), estimated from the absorption data, are given in Table 2. The excess in electron concentration above the phosphorous concentration in the initial sam-

Basic parameters of studied samples: parameters of the treatments, carrier concentration obtained from resistivity measurements, n, phosphorous (P) and interstitial oxygen concentrations ([Oi]) and TDDs concentration calculated from the FTIR data, N_{TDD} .

Sample: n° irradiation + treatment, temperature/time/pressure	n, cm ⁻³	P, cm ⁻³	[O _i], cm ⁻³	N _{TDD} , cm ⁻³
initial	8.2×10^{14}	5.0x10 ¹⁴	8.5x10 ¹⁷	-
n° + 450°C/10h/1GPa	2.4×10^{14}	3.7x10 ¹⁴	7.7×10 ¹⁷	5.86x10 ¹⁴
450°C/10h/1GPa	84.8x10 ¹⁴	5.0x10 ¹⁴	8.6x10 ¹⁷	27.0x10 ¹⁴

ple is most likely connected with the presence of shallow thermal donors (STDs). The STDs concentration in initial samples is equal to 3.2×10^{14} cm⁻³. The decrease in phosphorous and oxygen concentrations in the neutron irradiated samples is caused by the formation of complexes with radiation-produced vacancies. The carrier concentration in this sample was lower than the total of the phosphorous and TDDs concentrations (9.56×10^{14} cm⁻³). As seen from Table 2, the high concentration of TDDs (27.0×10^{14} cm⁻³) and OF STDs (52.8×10^{14} cm⁻³) is introduced in the HT-HP treated non-irradiated sample. It is therefore concluded that the production of TDDs in neutron-irradiated Cz-Si during HP-HT treatment at 450 °C is strongly reduced. This effect can be caused by the formation of TAs. TAs concentration in this case would be equal or higher than 6×10^{14} cm⁻³. This value (if compared with the corresponding values for electron-irradiated samples) is found to lower lower than expected [1].

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