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Abstract:	<p>Two stage electron irradiation with thermal heat-treatments after each stage has been used for vacancy-oxygen-related defect engineering in Czochralski-grown silicon (Cz-Si). The Cz-Si samples were first irradiated at room temperature with 2.5 MeV electrons and then heat-treated at 320°C to anneal out the VO, V2O and V3O centers and generate the VO2, V2O2 and V3O2 complexes as the dominant vacancy-oxygen-related defects. Subsequently, the samples were irradiated at room temperature again and subjected to 30-min isochronal annealing in the temperature range 75-350°C. Defect evolution upon the treatments has been monitored by means of the local vibrational mode (LVM) absorption spectroscopy. From an analysis of changes in intensity of the LVM lines it is revealed that the second irradiation results in a noticeable decrease in the concentrations of the VO2, V2O2 and V3O2 complexes and an increase in the concentrations of the oxygen dimer and the VO2* defect (metastable state of VO2, which consists of the VO and Oi components). The observed defect transformations are argued to be related to interactions of the radiation-induced self-interstitial silicon atoms (I) with the vacancy-oxygen complexes via the following reactions: VO2 + I → O2i, V3O2 + I → V2O2, V2O2 + I → VO2*.</p>
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<p>Please submit a plain text version of your cover letter here.</p>	<p>Dear Editors,</p> <p>Uploaded is a file with text and original figures of our revised manuscript "Interaction of Radiation-Induced Self-Interstitials with Vacancy-Oxygen Related Defects VnO2 (n from 1 to 3) in Silicon" by L.I. Murin et al., which we would like to submit for publication in Physica Status Solidi A in a special issue with Proceedings of the E-MRS-2018 Symposium I - Materials Research for Group IV Semiconductors: growth, characterization and technological developments.</p> <p>Yours faithfully,</p> <p>Vladimir Markevich</p>
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Full Paper

Interaction of Radiation-Induced Self-Interstitials with Vacancy-Oxygen Related Defects V_nO_2 (n from 1 to 3) in Silicon

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Keywords: silicon, electron irradiation, vacancy-oxygen defects, self-interstitials, local vibrational modes

Two stage electron irradiation with thermal heat-treatments after each stage has been used for vacancy-oxygen-related defect engineering in Czochralski-grown silicon (Cz-Si). The Cz-Si samples were first irradiated at room temperature with 2.5 MeV electrons and then heat-treated at 320 °C to anneal out the VO, V_2O and V_3O centers and generate the VO_2 , V_2O_2 and V_3O_2 complexes as the dominant vacancy-oxygen-related defects. Subsequently, the samples were irradiated at room temperature again and subjected to 30-min isochronal annealing in the temperature range 75-350 °C. Defect evolution upon the treatments has been monitored by means of the local vibrational mode (LVM) absorption spectroscopy. From an analysis of changes in intensity of the LVM lines it is revealed that the second irradiation results in a noticeable decrease in the concentrations of the VO_2 , V_2O_2 and V_3O_2 complexes and an increase in the concentrations of the oxygen dimer and the VO_2^* defect (metastable state of VO_2 , which consists of the VO and O_i components). The observed defect transformations are argued to be related to interactions of the radiation-induced self-interstitial silicon atoms (I) with the vacancy-oxygen complexes via the following reactions: $VO_2 + I \rightarrow O_{2i}$, $V_3O_2 + I \rightarrow V_2O_2$, $V_2O_2 + I \rightarrow VO_2^*$.

Dedication: This paper is dedicated to our co-author Professor Bengt Svensson who died in a tragic accident on 24th July 2018, shortly after the completion of this paper. He has spent much of his professional life working on defects in semiconductors and he will be sorely missed by his many collaborators.

1. Introduction

Processes of radiation-induced defect formation and their elimination in silicon and silicon devices have been a subject of great attention since the 1960s and still are.^[1-3] A large number of experimental and theoretical results have already been accumulated in this research field motivated by the control and removal of implant damage and an understanding of defect reactions which has facilitated defect engineering in silicon. In particular, the structural, electronic, optical, and dynamical properties of a number of radiation-induced defects (RIDs) have been determined. These include defects resulting from interactions of primary lattice defects with interstitial oxygen atoms, the most abundant impurity atoms in Czochralski-grown (Cz) Si crystals. The relevance of understanding of effects of radiation-induced oxygen-related defects on performance of silicon-based electronic devices have been underlined recently in a review paper by Chroneos et al.^[3] In that review and in an earlier work (Ref. [4]) several methods of defect engineering for management of interactions of intrinsic defects with oxygen-related centers and related modifications of electrical and optical properties of Cz-Si crystals have been discussed. In the present work interactions of silicon self-interstitial atoms with some oxygen-vacancy-related defects will be considered in more details.

Oxygen in silicon is one of the most effective traps for mobile mono-vacancies, di-vacancies and tri-vacancies.^[1-12] The vacancy-oxygen complex (VO), also called the A-center, was the

first radiation-induced defect identified in irradiated Si crystals.^[5] By now, its properties have been thoroughly studied.^[3,6] More recently, the properties of the divacancy-oxygen (V_2O) and trivacancy-oxygen (V_3O) complexes have been studied in detail.^[7-11] It is found that the VO , V_2O and V_3O centers disappear upon annealing of the irradiated Si samples at temperatures above 300 °C and their disappearance is accompanied by the appearance of vacancy-oxygen complexes containing two oxygen atoms (VO_2 , V_2O_2 and V_3O_2). All of these complexes give rise to vibrational absorption bands which can be observed in the infra-red absorption spectra.^[3,4,6,10,12-18]

The VO_2 defect plays an important role in processes of aggregation of oxygen atoms and vacancies during growth of silicon crystals by Czochralski techniques and upon formation of oxide films and oxygen precipitates in Cz-Si wafers.^[6,19,20] Therefore, the formation and elimination mechanisms and properties of this center have been extensively studied.^[3,6]

According to the VO_2 structural model, in the minimum energy configuration two oxygen atoms saturate all four broken bonds of a Si vacancy.^[3,6] In this configuration, both oxygen atoms occupy equivalent positions inside the vacancy, each of them is connected with two silicon atoms and gives rise to an absorption band at 895 cm^{-1} .^[6, 12] For a long time it was thought that the VO_2 defect could not be electrical active. However, in 2003, bistability of the VO_2 complex was discovered.^[15] It has been found that there is another, metastable, configuration of this center - VO_2^* , in which one oxygen atom is inside the vacancy, while the second atom occupies an interstitial position at the center of the nearest Si-Si bond. The total energy of the defect in the metastable configuration is only 0.25 eV higher than the energy of the ground state of the VO_2 complex, so at high temperatures a noticeable fraction of the complexes can be in the metastable VO_2^* state. Unlike the minimum energy electrically inactive VO_2 state, the VO_2^* complex is an acceptor with an energy level located at about 0.05 eV below the conduction band edge. The absorption bands at 928 and 1004 cm^{-1} have

been assigned to the neutral state of the VO_2^* defect, while the bands at 967 and 1023 cm^{-1} have been linked to the negatively charged center.^[16, 17]

In electron-irradiated Cz-Si crystals, the formation of the VO_2 and VO_2^* complexes was found to occur either during annealing in the temperature range 300-400 °C of Cz-Si crystals irradiated at room temperature, or upon so-called hot ($\sim 350\text{ °C}$) irradiation (see Ref. [17] and references therein). Recently, it has been argued that two infrared absorption lines at 924 and 1016 cm^{-1} , which were observed in absorption spectra of Cz-Si samples subjected to irradiation with fast neutrons and annealing in the temperature range 200-300 °C, are related to the VO_2^* defect.²¹ It should be noted, however, that the arguments presented in Ref. [21] for the assignment of the lines at 924 and 1016 cm^{-1} to the VO_2^* center are not very solid. In this paper we give further evidence for the assignment of the absorption bands at 928 and 1004 cm^{-1} to the VO_2^* defect and report on the formation of this complex and oxygen dimers (O_{2i}) in the room temperature region as a result of irradiation of Cz-Si samples containing the V_nO_2 vacancy-oxygen centers, including the recently identified V_2O_2 and V_3O_2 complexes.^[13, 14]

2. Experimental Details

The samples used in the study were prepared from phosphorus doped *n*-type Czochralski-grown (Cz-Si) crystals ($\rho = 20\text{ }\Omega\cdot\text{cm}$) with measured concentrations of interstitial oxygen (O_i) and substitutional carbon (C_s) atoms being $1 \times 10^{18}\text{ cm}^{-3}$ and $\leq 5 \times 10^{16}\text{ cm}^{-3}$, respectively. Irradiation with fast electrons ($E = 2.5\text{ MeV}$) was carried out at nominal room temperature with a dose of $1.28 \times 10^{18}\text{ cm}^{-2}$. To generate the VO_2 , V_2O_2 and V_3O_2 defects in high concentrations the samples were annealed at 320 °C for 30 hours. Then a second room

temperature irradiation was carried out with a dose of $3.5 \times 10^{17} \text{ cm}^{-2}$ and the samples were subjected to 30-min isochronal annealing in the temperature range 75-350 °C with 25 °C increments.

IR absorption analysis was carried out using a Bruker IFS 113v spectrometer. A spectral resolution of 0.5 or 1.0 cm^{-1} was used and the spectra were recorded at about 20 K and at room temperature (RT). The shape of the bands was analyzed by fitting the spectra with Lorentzian components with contributions due to silicon isotopes (^{29}Si and ^{30}Si) being taken into account.^[22]

3. Experimental Results and Discussion

Spectrum 1 in **Figure 1** is a fragment of the low-temperature absorption spectrum for a Cz-Si sample annealed at 320 °C for 30 hours after irradiation with 2.5 MeV electrons (the absorption spectrum of this sample measured at room temperature before annealing can be found in Ref. [23]). It can be seen from spectrum 1 that the main radiation-induced defects in this sample after annealing are the VO_2 , V_2O_2 and V_3O_2 vacancy-oxygen related complexes. This is evidenced by the high intensity of the absorption bands with their maxima located at 895.5 cm^{-1} (VO_2),^[6, 12] 829.3 cm^{-1} (V_2O_2) and 844.4 cm^{-1} (V_3O_2).^[13, 14] After the second irradiation of the annealed sample with 2.5 MeV electrons at room temperature, the intensities of all the above mentioned bands have decreased. On the other hand, increases in the intensity of the bands with their maxima at 835.8 cm^{-1} (VO complex),^[6] 928.4 cm^{-1} and 1003.8 cm^{-1} (VO_2^* complex),^[15-17] as well as the band with its maximum at 1012.4 cm^{-1} , which is an LVM due to the oxygen dimer,^[24] occur. It is obvious that a strong increase in the concentration of the VO complex resulting from the second irradiation is related to the capture of the radiation-induced mobile vacancies by interstitial oxygen atoms whose concentration is still rather high

in the sample studied. An increase in the oxygen dimer concentration is evidently due to an interaction of the silicon self interstitials (I) with the VO₂ defects via a reaction VO₂ + I → O_{2i}.^[25] It is reasonable to expect that not only VO₂ but V₂O₂ and V₃O₂ complexes are also effective traps for the Si self-interstitials and the following reactions can occur:

Evidence for the above reactions is provided by the results shown in **Figure 2**.

To further confirm the assignment of the bands observed at 928.4 cm⁻¹ and 1003.8 cm⁻¹ to the VO₂* complex, we carried out a 30-minute isochronal annealing with steps of 25 ° C in the temperature range 75-350 ° C using a sample subjected to the second irradiation at RT (see spectrum 2 in Figure 1). An analysis of the absorption spectra measured after each annealing step has showed that heat-treatments at 275 °C and above have resulted in a decrease in the intensity of the bands at 928.4 cm⁻¹ and 1003.8 cm⁻¹ with a correlated increase in the amplitude of the band at 895.5 cm⁻¹ (**Figure 3**). This effect is due to the transformation of the VO₂ defect from the metastable VO₂* state to the minimum energy configuration with two oxygen atoms inside a vacancy. An equilibrium distribution between the VO₂* and VO₂ states can be attained at annealing temperatures above 275 °C. The presence of a high potential barrier between these states prevents the re-configuration of the defect at lower temperatures.^[15-17]

Figures 4 and 5 show the results of a more detailed analysis of the low-temperature absorption spectra (see Figure 1) in the range of wave numbers between 810 and 855 cm⁻¹. A fitting procedure using Lorentzians was used to analyze the absorption data. For each band the presence of all three Si isotopes (²⁸Si, ²⁹Si and ³⁰Si) was taken into account upon fitting.^[22] For clarity, only the fitting sub-curves corresponding to the ²⁸Si-O-²⁸Si vibration units are shown in Figures 4 and 5, but the main fitting curves accounts for all the contributions. On the

basis of the data obtained, the absorption coefficients for all main LVM bands observed in the absorption spectra were determined. The absorption coefficient values are presented in Table 1 alongside the changes in their values as a result of the second 2.5 MeV electron irradiation with a dose of $3.5 \times 10^{17} \text{ cm}^{-2}$. The data on the changes in the absorption coefficients of the LVM bands confirm the suggestion that the increase in the concentration of VO_2^* and O_{2i} complexes is due to an interaction of the radiation-induced silicon self-interstitial atoms with the VO_2 , V_2O_2 and V_3O_2 vacancy-oxygen complexes.

4. Conclusion

The processes of formation of radiation-induced defects upon electron irradiation of Cz-Si crystals containing the VO_2 , V_2O_2 and V_3O_2 vacancy-oxygen complexes have been studied. An increase in the concentration of the VO_2 complex in the metastable VO_2^* configuration as well as in the oxygen dimer O_{2i} concentration occurred in the samples as a result of the irradiation. It is concluded that these phenomena are due to the interaction of the silicon self-interstitial atoms generated by irradiation with the VO_2 , V_2O_2 and V_3O_2 vacancy-oxygen complexes. The results obtained on radiation-induced formation of VO_2^* complexes are important from the point of view of additional confirmation of the identification of the bands at 829.3 cm^{-1} and 844.4 cm^{-1} as related to the V_2O_2 and V_3O_2 complexes, respectively.

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Numerous and enlightening discussions with Prof. J.L. Lindström on the subject of oxygen in silicon are gratefully acknowledged as well as his experimental assistance during the initial stage of the study. The work was partially funded by Belarusian Republican Foundation for Fundamental Research (project No F17MC-022) and in the UK by the EPSRC contract EP/M024911/1.

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Figure 1. Fragments of infrared absorption spectra recorded at 20 K on a Cz-Si sample, which was subjected to the following treatments: 1) irradiation with 2.5 MeV electrons at room temperature to the accumulated dose of $1.28 \times 10^{18} \text{ cm}^{-2}$ with following annealing at 320 °C for 30 hours; 2) subsequent irradiation with 2.5 MeV electrons at room temperature to the accumulated dose of $3.5 \times 10^{17} \text{ cm}^{-2}$. The spectra are shifted on the vertical axis for clarity.

Figure 2. Difference absorption spectrum obtained by subtraction of spectrum 1 from spectrum 2 in Figure 1.

Figure 3. Difference absorption spectrum obtained by subtraction of the spectrum recorded after annealing at 250 °C for 30 min from the spectrum recorded after annealing at 275 °C for 30 min for a Cz-Si sample which before these anneals was subjected to a two-stage irradiation with 2.5 MeV electrons with a heat-treatment at 320 °C for 30 hours between the irradiation stages. The original spectra were recorded at 20 K.

Figure 4. Spectral analysis of a fragment of the absorption spectrum recorded at 20 K for a Cz-Si samples which was subjected to irradiation with 2.5 MeV electrons at room temperature to the accumulated dose of $1.28 \times 10^{18} \text{ cm}^{-2}$ with following annealing at 320 °C for 30 hours. Circles are experimentally measured points and solid lines are fitting lines of Lorentzian form. For each band the presence of all three Si isotopes (^{28}Si , ^{29}Si and ^{30}Si) is taken into account upon fitting. For clarity, only the fitting sub-curves corresponding to $^{28}\text{Si-O-}^{28}\text{Si}$ units are shown, but the main fitting curve accounts for all the contributions.

Figure 5. Spectral analysis of a fragment of the absorption spectrum recorded at 20 K for a Cz-Si sample which was subjected to irradiation with 2.5 MeV electrons at room temperature to the accumulated dose of $1.28 \times 10^{18} \text{ cm}^{-2}$ with following annealing at 320 °C for 30 hours and subsequent irradiation with 2.5 MeV electrons at room temperature to the accumulated dose of $3.5 \times 10^{17} \text{ cm}^{-2}$. Circles are experimentally measured points and solid lines are fitting lines of Lorentzian form. For each band the presence of all three Si isotopes (^{28}Si , ^{29}Si and ^{30}Si) is taken into account upon fitting. For clarity, only the fitting sub-curves corresponding to $^{28}\text{Si-O-}^{28}\text{Si}$ units are shown, but the main fitting curve accounts for all the contributions.

Table 1. Absorption coefficients (in cm^{-1}) in the maxima of the LVM absorption bands due to oxygen-related defects in a Cz-Si sample which was subjected to irradiation with 2.5 MeV electrons at room temperature to the accumulated dose of $1.28 \times 10^{18} \text{ cm}^{-2}$ with following annealing at 320 °C for 30 hours and subsequently to irradiation with 2 MeV electrons at room temperature to the accumulated dose of $3.5 \times 10^{17} \text{ cm}^{-2}$. Positions of the LVMs (in cm^{-1}) at 20 K for the analyzed oxygen-related defects are indicated in the Table.

Sample treatments	V_2O_2	VO	V_3O_2	VO_2	VO_2^*	VO_2^*	O_{2i}
	829.3	835.8	844.2	895.5	928.4	1003.7	1012.4
1 st Irradiation + annealing at 320 °C for 30 h	0.23	0.02	0.12	1.04	0.03	0.02	0.05
2 nd irradiation	0.15	0.69	0.05	0.54	0.08	0.05	0.17
Difference in amplitudes of LVMs	-0.08	+0.67	-0.07	-0.5	+0.05	+0.03	+0.12