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Positron annihilation lifetime in float-zone n-type silicon irradiated by fast electrons: a thermally stable vacancy defect

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Temperature dependency of the average positron lifetime has been investigated for n-type float-zone silicon, n-FZ-Si(P), subjected to irradiation with 0.9 MeV electrons at RT. In the course of the isochronal annealing a new defect-related temperature-dependent pattern of the positron lifetime spectra has been revealed. Beyond the well known intervals of isochronal annealing of acceptor-like defects such as E-centers, divacancies and A-centers, the positron annihilation at the vacancy defects has been observed in the course of the isochronal annealing from ~ 320 ºC up to the limit of reliable detecting of the defect-related positron annihilation lifetime at ≥ 500 ºC. These data correlate with the ones of recovery of the concentration of the charge carriers and their mobility which is found to continue in the course of annealing to ~ 570 ºC; the annealing is accomplished at ~650 ºC. A thermally stable complex consisting of the open vacancy volume and the phosphorus impurity atom, V_{op}-P, is suggested as a possible candidate for interpreting the data obtained by the positron annihilation lifetime spectroscopy. An extended couple of semi-vacancies, $2V_{s\text{-}ext}$, as well as a relaxed inwards a couple of vacancies, $2V_{inw}$, are suggested as the open vacancy volume V_{op} to be probed with the positron. It is argued that a high thermal stability of the $V_{s\text{-}ext}PV_{s\text{-}ext}$ (or $V_{inw}PV_{inw.}$) configuration is contributed by the efficiency of PSi_5 bonding.

1 Introduction Recent findings obtained for the first time by the low-temperature electrical measurements of the Hall effect and conductivity on the electron- and gamma-irradiated n-FZ-Si(P) materials correlate well with the formation model of the donor-vacancy pairs put forward earlier on the basis of the EPR data [1]. At the same time, a surprising temperature behaviour of mobility of charge carriers suggests large-scale elastic deformations created by the centers, supposedly, of an interstitial type in the electron-irradiated moderately doped n-type material: their cross sections are estimated to be ~ 10–12 cm^2 [1, 2]. Moreover, the picture does not fit solely into a simple annealing process of E-centers: the recovery of the charge carrier concentration and mobility proceeds in the two stages, 100-180 ºC, 200-300 ºC, followed by the last annealing stage which is accomplished ranging from ~ 570 to 650 ºC [3].

The thermally stable point defects to be annealed at this last stage are positron-sensitive centers of a vacancy type [4]. They are effective positron traps which are observed in the course of isochronal annealing ranging from $T_{anneal.} \geq 280$ ºC to $T_{anneal.} \geq 460$-500 ºC when their concentration is becoming insufficient for detecting them by the positron annihilation lifetime spectroscopy. It will be noted that thermally stable vacancy center revealed in the same n-FZ-Si(P) material irradiated by 15 MeV protons [5, 14, 15] is characterized by long positron lifetime ~ 276-294 ps, thus suggesting that the open vacancy volume related to the center is equal to the one of a pair of vacancies.

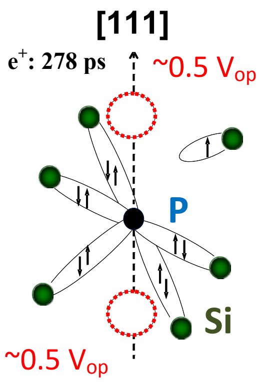

Figure 1 The thermally stable open vacancy volume V_{op} to be detected by the positron probing is suggested for interpreting the data for n-FZ-Si (P) material subjected to irradiation with 0.9 MeV electrons. A possible configuration of V_{op} involving the impurity atom of phosphorus may be provided by an extended semi-vacancy, $V_{s\text{-}ext}$, or, depending on the extent of V_{op}, by a couple of the vacancy volumes relaxed inwards, V_{inw}, so that $2V_{s\text{-}ext} \approx V_{op} \approx 2V_{inw}$. The defect-related positron lifetime τ_2 (V_{op}) ≈ 278 ps is observed under isochronal annealing up to $T_{anneal.} \geq$ 460-500 ºC. Known from the literature, the positron lifetime values calculated for a cluster of a pair of neutral vacancies are within a range from ~265 ps to ~ 299 ps (see Fig. 3).

To gain insight into the microstructure of these defects, we have undertaken a study of the pattern of temperature dependencies of the positron annihilation lifetime (PAL) in the electron-irradiated n-FZ-Si(P); the study of isochronal annealing of material subjected to irradiation with 0.9 MeV electrons underlies this experiment. It is argued that the thermally stable positron-sensitive centers which are detected by PAL spectroscopy up to the limit of instrumental sensitivity (at ≥ 460-540 ºC) are the complexes of a vacancy type comprising the phosphorus impurity atom (see also Fig. 1).

2 Experimental

2.1 Samples and electron irradiation Single crystal of silicon was grown by the floating-zone technique (n-FZ-Si[P=7 × 10^{15} cm^{-3}]). According to data of IR spectroscopy, the concentration of oxygen was ~ 2 × 10^{16} cm^{-3}. The concentration of carbon was 7 × 10^{15} cm^{-3}. The similar material has been used earlier for EPR studies [6].

To avoid unwanted formation of various multi-vacancy defects the samples were irradiated with electrons at 0.9 MeV in a resonant transformer accelerator on a target in a water stream. The pulse repetition frequency was 490 Hz, and the pulse duration was 330 µs. The power of the electron flow was I = 7.5 × 10^{13} electrons/cm^2 s; the dose of irradiation was 1 × 10^{16} electrons/cm^2. The irradiation temperature did not exceed 30 ºC.

2.2 Electrical measurements The Hall effect measurements have been conducted as a complementary ones to that of positron annihilation lifetime. In a similar way this Si material has been studied earlier [4, 5]. The temperature dependences of the concentrations of charge carriers n(1/T) and mobility have been measured over the range of ~ 4.8-300 K (some example of analysis of these data one could find, e.g., in [1-3]). Analysis of n(1/T) functions with the use of equations of the charge balance gives us an opportunity to evaluate separately both the total concentration of shallow donor centers and the concentration of compensating acceptors (see, e.g. [1] and references therein). The data obtained enables one to estimate the concentrations of the produced defects as well as their levels in the forbidden gap: the estimated concentration of the centers to be discussed in this paper was ~ 10^{15} cm^{-3} prior to the beginning of the isochronal annealing which was carried out ranging from ~ 60 to 700 °C. The positron annihilation experiments were conducted for the same material which had been used for the electrical measurements.

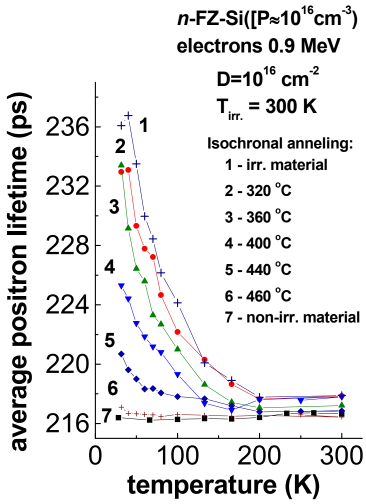

Figure 2 The pattern of the temperature dependencies of the average positron lifetime (τ_{av}) in n-FZ-Si(P) material irradiated with 0.9 MeV electrons and subjected to the isochronal annealing. The stage related to the thermally stable centers from 320 to 460 ºC is presented. The stages of annealing in the intervals ~ 80-170 ºC, ~180-280 ºC will be discussed elsewhere.

2.3 Positron lifetime and annihilation radiation spectra processing The average positron annihilation lifetime $\tau_{av}(T)$ was obtained using a spectrometer with high time resolution determined to be 215 ps (see [5] and references therein). The number of registered events of electron-positron annihilation was (4-6) × 10^6 in a single PAL spectrum which was recorded ranging from 30 to 300 K. We used ^{22}Na isotope as the positron source which was sandwiched between two identical 2.5 µm Al foils. Its contribution to the PAL spectra did not exceed ~ 6%. The parameters of the source contribution τ_{short}≈380 ps (~98.7%) and τ_{long} ≈ 4200 ps (~1.3%) have been fixed in the data processing. The average positron lifetime τ_{av} (T) was used for determining the short and long positron lifetimes, τ_1 and τ_2, respectively, via their intensities I_1 and I_2:

$$\tau_{av}(T)=\tau_1 I_1+\tau_2 I_2 \ , \qquad (1)$$

where $I_1 + I_2 = 1$. The τ_1 (I_1) is mainly related to the positron annihilation in the bulk of the material [9]; in this paper we will analyzed the characteristic long defect-related $\tau_2(I_2)$ lifetime reconstructed by deconvolution of the average positron lifetime. The programs LT9 and LT10 were applied taking into account the function of the experimental time resolution [4, 5].The multi-exponential fitting under the condition of unconstrained $I_1\tau_1$ and $I_2\tau_2$ values was used. Under this condition of data processing the values $\tau_2(T, T_{anneal.})$ vary insignificantly at the beginning of annealing of the thermally stable defects from $\tau_2(T_{anneal.} = 280$ ºC) ≈ 285 ps to τ_2 ($T_{anneal.}$=360 ºC) ≈ 281 ps (the fitting is good, the variance of the fit did not exceed Var. ~1.03; see [9]). On the whole, these conditions of fitting are preserved for PAL spectra obtained for the measuring temperatures up to $T_{meas.}$ = 166 K. This fact in itself suggests strongly that the pattern of τ_{av} (T, $T_{anneal.}$) spectra belongs to one kind of thermally stable defects of a vacancy type (Fig. 2). Due to decreasing the concentration of defects the processing of data gives the value $\tau_2(T_{anneal.}$=400 ºC) ≈ 271 at quite reliable variance of the fit, Var. ~1.06 (see Fig. 3). For the annealing temperatures ≥ 440 ºC the data processing requires special constrained conditions for reconstructing the characteristic τ_2 values: see τ_{av} ($T_{anneal.}$ ≥ 440 ºC) in Fig. 2.

The PAL spectra (PALS) were recorded after each step of the isochronal annealing of the samples (h = 40 °C/t_h =10 min) in vacuum conditions ~10^{-4} Torr.

3 Thermally stable radiation centers: PALS

3.1 Elimination of A-centers and divacancies A possible competitor to P impurity in formation of impurity-vacancy complexes is O impurity which may form the oxygen-impurity pairs called A-centers; however, the production rate of these defects is observed to be very low in FZ-Si under ~ 1 MeV electron irradiation [17]. The concentration of A-centers capable of producing a detectable long-lived component of the positron lifetime in the irradiated FZ-Si material at an oxygen content on the order of 10^{16} cm^{-3} is extremely small owing to a small formation cross section of A-centers, which is two orders of magnitude smaller, e.g., than that of E-centers (see [1-3] and references therein). Besides, A-centers disappear completely by ~ $T_{anneal.}$~ 350 ºC under isochronal annealing [18].

The production rate of divacancies is established to be ~3 × 10^{-3} cm^{-1} for 0.9 MeV electron irradiation at RT [19] and their concentration at the chosen dose of irradiation 10^{16} electrons/cm^2 is too small for being detected by PALS.

Moreover, in contrast to annealing VV$^+$ defect to be observed by EPR [20], in n-type FZ-Si(P) material subjected to 4 MeV electron-irradiation the defect reactions associated with complete elimination of divacancies occur at the annealing temperature ~ $T_{anneal.}$~ 300-350 ºC [21]. Also, the interval ~ $T_{anneal.}$~ 205-285 ºC has been observed for annealing of singly negative and doubly negative divacancies in the diffusion oxygenated FZ-Si material subjected to irradiation with 15 MeV electrons [22]. Thus, we arrive at a conclusion that at the annealing temperatures $T_{anneal.}$ > 280 ºC both the A-centers and divcancies can not give a detectable contribution to the average positron lifetime τ_{av} (T, $T_{anneal.}$) in the investigated material.

3.2 Annealing of thermally stable radiation centers For reasons given above we are compelled to attribute the average positron lifetime $\tau_{av}(T, T_{anneal.})$ to a thermally stable defect of a vacancy type. Furthermore, the end of the temperature interval of the annealing stage of these defects T_{anneal} ≥ 460 ºC, whose detection by PALS is limited just by the positron diffusion length and the concentration of defects (see below, Section 5), is within a dominating high-temperature stage of recovery of the electrical activity of P impurity atoms and mobility of charge carriers which is completed by T_{anneal} ~ 650 ºC [3, 4, 16]. This fact necessitates involving the atoms of phosphorus impurity into the configuration of defects under discussion (see Fig. 2): reasoning from this knowledge we suggest that the long-lived component $I_2\tau_2$ (V_{op}) of the average positron lifetime τ_{av} is related to the same thermally-stable centers.

After irradiation the average positron lifetime $\tau_{av}(T)$ is longer than the one, $\tau_b(T)$, obtained for initial non-irradiated reference sample: $\tau_{av}(T) >> \tau_b(T) \approx 217$ ps, cf. curves 1 and 7 in Fig. 2.

The increase of the average positron lifetime $\Delta\tau_{av}$ (T) = $\tau_{av}(T)$-$\tau_b(T)$ is generally accepted to attribute to the positron which annihilates in its localized state at the vacancy open volume (V_{op}).

According to modern conceptions, the thermalized positron probes the open volume of a defect of a vacancy type and the efficiency of emission of the annihilation gamma-quanta is due to the defect-related characteristic lifetime τ_2 (V_{op}). The probability of this emission is determined by the temperature-dependent positron trapping rate due to the cascade phonon-assisted positron trapping [14].

The decomposition of the thermally stable radiation centers begins at T_{anneal} ~ 320 ºC (cf. curves 1 and 2 in Fig. 2): this process continues up to detecting of the rise of the average positron lifetime $\tau_{av}(T)$ from ~ 200 to ~ 40 K after the annealing of material at ~ 460 ºC (cf. curves 6 and 7 in Fig. 2: the difference between $\tau_{av}(T)$ dependencies is detected even at the annealing temperature ~ 540 ºC).

No doubts that the pattern of the $\tau_{av}(T)$ dependencies within the isochronal annealing stage ΔT_{anneal} ~320-500 ºC is due to the positron trapping by the defect comprising open vacancy volume V_{op}: the annihilation 2γ-quanta emitted from the defects and related to the long-lived positrons produce the long-lived component $I_2\tau_2$ (V_{op}) of the average positron lifetime τ_{av} (see Fig. 2, curves 2-6).

4 The scaling of open vacancy volume V_{op} of thermally stable center

The magnitude scaling of the open vacancy volume V_{op} in the units of the number of vacancies may be done by comparison of the defect-related positron lifetime $\tau_2(V_{op})$ with the values obtained by calculations for the vacancy cluster [7]: the fitting function

$$\tau(n) = 220 + 230[1 - \exp(-n/5)] \quad (2)$$

has been used; the data are shown in Fig. 3. Also, the *ab initio* calculated lifetime of a positron trapped at a small vacancy cluster is presented in Fig. 3 as a function of the number of vacancies (n); see [8, 9] for more detail.

The defect-related positron lifetime values τ_2 (V_{op}) reconstructed from the average positron annihilation lifetime τ_{av} (T=40 K, $T_{anneal.}$) within the interval of annealing temperatures from 320 to 400 °C is equal to ≈ 278 ± 2 ps; see the data shown by the horizontal lines in Fig. 3: the abscissa values of the crossing points of the lines and the data of calculations indicate the magnitude of n between n=1 and n=2. It means that the vacancy open volume V_{op} to be probed with the positron is equal to the volume of a pair of vacancies relaxed inwards; alternatively, two semi-vacancies whose volume is extended due to outwards relaxation might be considered as the value of the open vacancy volume V_{op}. These options are shown in Fig. 1.

5 Discussion: microstructure of thermally stable radiation centers The high-temperature stage of isochronal annealing which begins at ~ 320 °C (see curve 2 in Fig. 2) and continues (according to Hall effect measurements) up to ~ 600 °C [3] is related to a thermally stable center whose decomposition is accompanied by recovering the electrical activity of the impurity atoms of phosphorus [3, 16]. Starting from these data we suggest considering a complex of the phosphorus impurity atom (P) which is tied to the open vacancy volume V_{op} (see Fig. 1).

The defect-related component $I_2\{\tau_2(V_{op}) \approx 278$ ps$\}$ vanishes slowly up to the instrumental limit of its detecting in the average positron annihilation lifetime at $T_{anneal.} \geq$ 460-500 °C indicating the presence of the open vacancy volume V_{op} in the microstructure of this thermally stable phosphorus-vacancy complex (see Fig. 3). It will be noted that the defect-related positron lifetime value $\tau_2(V_{op}) \approx$ 278 ps itself does not allow us to establish whether the P impurity atom is involved in the microstructure of the thermally stable center. This issue may be clarified by probing with positrons the ion cores of atoms tied to V_{op}.

It is believed that the defect-related positron lifetime τ_2 (V_{op}) reflects the electron density contacting the positron, mainly, inside the open volume (see, e.g. [9]). When comparing $\tau_2(V_{op})$ values with the theoretical data shown in Fig. 3, one should remember that the vacancy open volume V_{op} detected by PALS is tied to P impurity atom, whereas theoretically an isolated vacancy open volume is treated.

An assumption about a shift of the P impurity atom towards the open vacancy volume (V_{op}) allows one to give a possible interpretation of the data obtained (Fig. 1). The magnitude scaling suggests that the characteristic lifetime $\tau_2(V_{op})$ shown in Fig. 3 might formally be referred to an extended semi-vacancy $V_{s\text{-}ext}$, i.e., τ_2 (V_{op}) ≈ $\tau(V_{op}{\sim}2{\times}V_{s\text{-}ext})$, or to a relaxed inwards vacancy, in which case $\tau_2(V_{op}) \approx \tau$ $(V_{op}{\sim}2{\times}V_{inv.})$.

The V_{op}-P complex introduces the long-distance elastic strains affecting the carrier mobility: the estimated cross section of area of the elastic strains is ~ 10^{-12} cm^2 [1, 3]).

The positron trapping cross section of the thermally stable center under discussion has the same order of magnitude. Indeed, the concentration of the positron traps N_d to be detected by the positron trapping rate $k \approx \sigma(e^+){\times}N_d{\times}v(e^+)$ (v is the positron velocity) must be limited by the positron diffusion length $l_+ \leq (3/4\pi N_d)^{-1/3}$ and we obtain $\sigma(e^+) \approx (1.1–0.9){\times}10^{-12}$ cm^2 (see, e.g. [5, 15] for more clarity). This means that the center is effective positron attractor and that it possesses a negative effective charge in the investigated moderately doped n-type FZ-Si.

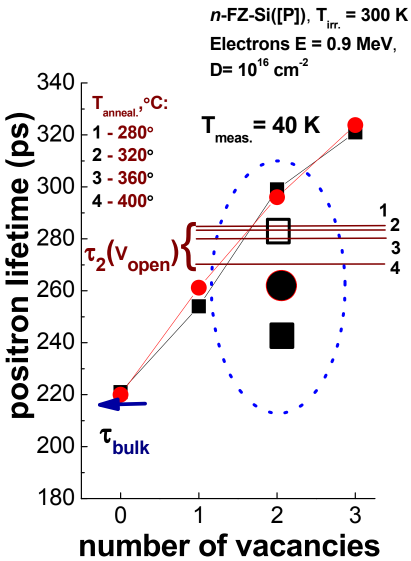

Figure 3 The number of vacancies (n) in the microstructure of the thermally stable point defect revealed in the electron-irradiated silicon n-FZ-Si single crystals: $1 < n \leq 2$ (see the area of crossing of the lines in the ellipse which is shown to guide the eye: it includes the calculated and experimental data). The thin horizontal lines indicate the values of the restored defect-related positron lifetime $\tau_2(V_{op})$ within the interval of the isochronal annealing from 280 to 400 °C. The lifetime measured for the bulk of the crystal ($\tau_{bulk} \approx 217$ ps) is shown by the arrow. The calculated positron lifetimes for the unrelaxed (small symbols) and relaxed (larger symbols) vacancy clusters in the neutral charge states are as follows: small squares: Ref. [10]; small dots: the values estimated by Eq. (2); larger solid and open squares: [8] and [11], respectively; dot: [12].

A possible microstructure of the vacancy-phosphorus complex might be characterized by a distorted symmetry D_{3d}: in this case the P atom is surrounded by six Si atoms. A gain in energy seems to be enough for providing a high thermal stability because the position of P atom in V_{op}-P complex enables it to form a quasi-molecule PSi_5 (Fig. 1). In case the complex captures one more electron all six states are occupied, thus creating additional energy gain;

partly, this issue for the phosphorus-vacancy pair has been discussed in [13]. The ion-covalent bonding of P and Si atoms facilitates deforming the complex towards octahedral symmetry (O_h) due to the shift of the neighbouring atoms.

It should be emphasized, that the calculations performed ab initio for PV pair in its neutral charge state result in the well-known configuration of EPR-sensitive E-center [23]. In this case the bonding of P atom with three nearest neighbours creates PSi_3 quasi-molecule which underlies the model of E-center having C_{3v} symmetry [6, 24]: this defect is eliminated at $T_{anneal.}$~ 175 °C from the material at the early stage of isochronal annealing. According to the results of calculations based on DFT in LDA approximation [23], in neutral PV pair P atom prefers substitutional configuration, however, partly with tangible relaxation and with a slight shift of P atom into a split configuration with D_{3d} symmetry. The complex of this symmetry when P atom is tied to the open volume V_{op} which is larger than the one of a vacancy has not been analyzed in [23].

6 Conclusion For the first time the annealing of the thermally-stable point defects created by electron irradiation in the moderately doped n-type silicon grown by floating-zone technique has been investigated by measuring the temperature-dependent positron annihilation lifetime (PAL).

Low-temperature Hall effect measurements [3, 16] displays a recovery of the donor properties of the atoms of phosphorus which correlate with the decrease of the concentration of the positron-sensitive vacancy defects up to the limit of sensitivity of PAL spectroscopy at ≥ 500 ºC. This correlation suggests involvement of the phosphorus impurity atom in the microstructure of the vacancy defects under study. The scaling transformation of the defect-related positron annihilation lifetime $\tau_2(V_{op}) \approx 278$ ps enables us to consider a complex of the phosphorus impurity atom which is tied to the open vacancy volume: V_{op} - P.

A configuration of the defect having distorted D_{3d} symmetry (or octahedral O_h symmetry when the center traps additional electron) is discussed. A couple of semi-vacancies (relaxed outwards) to be tied to the impurity atom of phosphorus, $V_{s\text{-}ext}\,PV_{s\text{-}ext}$, or, in case a larger extent of the open vacancy volume V_{op} takes place, the equivalent configuration with the relaxed inward vacancies, $V_{inw}PV_{inw,}$ enable us to interpret the data obtained. Larger number of ion covalent P-Si bonds (five; PSi_5) in the positron-sensitive V_{op}-P complex having distorted D_{3d} (or O_h) symmetry (in contrast to three P-Si bonds in EPR-sensitive phosphorus-vacancy pair, i.e. in E-center possessing C_{3v} symmetry) appears to underlie a high thermal stability of the V_{op}-P complex.

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