Defects in electron-irradiated Ge studied by positron lifetime spectroscopy

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The formation and annealing behavior of electron-irradiation induced defects in germanium were studied. The undoped Ge samples were irradiated at 4 K with an electron energy of 2 MeV. The fluence was varied between 10^{15} and $10^{19}\,\mathrm{cm}^{-2}$. Annealing experiments were performed from 90 K. Additional temperature-dependent positron lifetime measurements after different annealing stages were carried out to make a statement about charge states of defects and to the presence of shallow positron traps. Two main annealing stages were observed. The first between 150 and 250 K was attributed to the annealing of Frenkel pairs. The second annealing stage between 350 and 450 K was observed after irradiation with fluences above $10^{17}\,\mathrm{cm}^{-2}$ and was assigned to annealing of a complex containing a vacancy and an impurity atom. Furthermore, shallow positron traps were detected after irradiation. This defect type anneals above 330 K in a wide-temperature range.
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I. INTRODUCTION

Point defects as vacancies and interstitials influence the electrical and optical properties of semiconductors and were investigated by several methods. These defects can be produced by low-temperature electron irradiation in a reproducible manner. Many studies have been performed on annealing behavior, structure, and energy levels of irradiation-induced defects in Ge by electrical measurements and spectroscopic methods.[1,2] The results related to the annealing mechanism of close Frenkel pair,[3,4] *A* center,[5,6] and interstitial atom.[7,8] In addition, complex defect structures can result from the possible ionization-induced motion of interstitials.[9]

Vacancylike defects and complexes consisting of doping and interstitial atoms were in detail investigated in germanium after low-dose electron irradiation ($\leqslant 10^{17}\,\mathrm{cm}^{-2}$) by spectroscopic methods.[1] Undisturbed interstitials could not be proved as well. X-ray diffraction studies in electron-irradiated germanium ($\Phi \approx 10^{19}\,\mathrm{cm}^{-2}$) show that stable close Frenkel pairs are formed with a rate of about 1 cm^{-1}.[10] These defects anneal at 70 K.[1] A second annealing stage between 150 and 200 K was attributed to the neutral vacancy becoming mobil in this temperature range. The other defect complexes anneal above 400 K with the mobility of the divacancy. The annealing is completed at a temperature of 600 K.[10] The formation of larger vacancy clusters, which were detected after high-dose irradiation in the high-voltage electron microscope[11] could not be proved. It was explained by interaction reactions of defects with doping atoms.

Deep-level transient spectroscopy (DLTS) studies after electron irradiation show that impurities play an important role at the generation of irradiation-induced defects in germanium. Ito, Ito and Mizuno attributed an annealing stage at about 400 K observed by DLTS to complexes from divacancies and oxygen.[12] Fukuoka *et al.* investigated defects, which were formed by room-temperature irradiation (1.5 MeV) in As-, Sb-, and O-doped Ge, by x-rays, neutrons, and electrons.[13] An annealing stage at 260 °C was assigned to the annealing of vacancy clusters. In very pure germanium no irradiation-induced defects are detectable after a temperature treatment at 200 °C.[2]

The positron annihilation spectroscopy is a powerful method to identify vacancylike defects in electron-irradiated semiconductors. The efficiency of this method was shown in many cases earlier.[14–17] A few positron investigations of vacancylike defects in electron-irradiated Ge were done in the past. A Ge crystal irradiated with 4.4 MeV electrons at 90 K was annealed and three stages were observed. The first stage at 150 K was attributed to the vacancy-interstitial annihilation, the second between 180 and 420 K to the decay of a vacancy complex and the creation of stable complexes, and the annealing of all these defects occurs above 420 K.[18] After 3-MeV electron irradiation at 20 K the annealing behavior was attributed to the annealing of vacancy-oxygen complexes.[19,20] This defect disappears partially at 200 K, due to the migration of interstitial-related defects, and completely at 550 K when dissociation and evaporation of the complex occurs. The defect-related positron lifetime was determined to be 292 ps during the annealing between 77 and 675 K. In addition, the average positron lifetime of 279 ps was detected at 87 K after 2.5 MeV electron irradiation and was assigned to saturation trapping of positrons in irradiation-induced vacancies.[15]

Calculations of positron lifetime values in germanium result in 229 ps for the bulk, 263 ps for the monovacancy, and 316 ps for the divacancy.[21] Figure 1 shows the calculated ionization levels of the Ge vacancy in the band gap.[22] In the case of *n*-conductive or semi-insulating samples, the monovacancy is in the neutral or negative state and, therefore, detectable by positrons.

Perturbed angular correlation (PAC) of γ ray studies led to the conclusion that interstitials are positively charged in middle-doped *n* and *p* Ge and in highly *n*-type material ($N_D - N_A \approx 6\times 10^{17}\,\mathrm{cm}^{-3}$) they are neutral or negatively charged.[23]

In this paper we used positron-lifetime spectroscopy and

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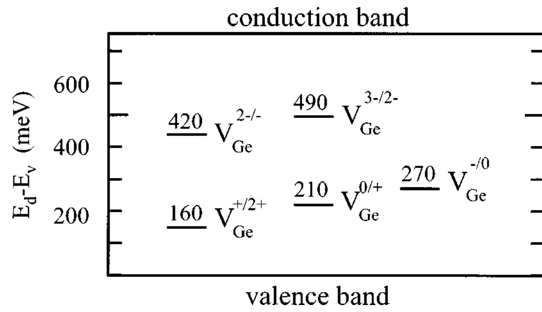

FIG. 1. The position of the ionization levels of the monovacancy in the band gap of germanium (Ref. 22).

Hall-effect measurements to investigate systematically the electron fluence dependence ($10^{15}\ldots10^{19}\,\mathrm{cm}^{-2}$) of defect formation and annealing behavior of undoped Ge.

In the following section we summarize the material and experimental details. The method of positron-lifetime spectroscopy and its specific features used to study vacancy defects in semiconductors is shortly described in Sec. III. In Sec. IV we present the results obtained, which are also discussed in this section. The presentation and the discussion of the results are divided in three sections; Sect. IV A concerns results on annealing experiments, Sec. IV B statements about the dose dependence, and Sec. IV C the temperature-dependent measurements after different annealing stages. Conclusions are drawn in Sec. V.

II. EXPERIMENTAL PROCEDURE

The samples used in this investigation were initially undoped germanium single crystals. The net-carrier concentration of the as-grown sample was determined to be $1.6\times10^{14}\,\mathrm{cm}^{-3}$ at room temperature. Electron irradiations were performed at 4 K with Van-der-Graaf accelerator in the Forschungszentrum Jülich. The incident energy of electrons was 2 MeV and different irradiation fluences between 10^{15} and $10^{19}\,\mathrm{cm}^{-2}$ were used. The samples were mounted at low temperatures (under liquid nitrogen) in a cryoheater system. The annealings were performed isochronally (15 min, 20 K steps) in the range between 90 and 600 K. After annealing at 330 K positron-lifetime measurements and annealings were carried out in a second cryoheater system in the range between 15 and 630 K. After this treatment some selected samples were annealed at higher temperatures (up to 700 K) external to the cryoheater system in a muffle furnace under vacuum. Temperature-dependent measurements after different annealing steps were also performed from 15 K.

The carrier concentration and mobility were obtained by Hall effect and conductivity measurements carried out according to van der Pauw[24] at 300 K.

The positron lifetimes were measured with a ^{22}NaCl positron source ($\approx$0.5 MBq) sandwiched between a pair of identically treated samples. We worked with a fast-fast coincidence system (time-resolution full width at half maximum $=260\,\mathrm{ps}$). The spectra contained at least 5×10^{6} counts and they were analyzed in terms of the trapping model[25,26] after the source and background correction.[27,28]

III. POSITRON LIFETIME AND DEFECT CONCENTRATION

The lifetime of positrons produced by ^{22}Na can be determined by the measurement of the time difference between the birth of the positron indicated by a γ quantum (1.27 MeV) and the death of the positron by annihilation with an electron indicated by annihilation quanta (0.511 MeV).

Positrons may annihilate from the delocalized ground state in the perfect lattice or from localized states formed at defects being able to trap positrons. The positron lifetime of the bulk τ_b (*b*...bulk) will be measured if no trapping centers locate the positrons during their diffusion through the sample. In the case of the presence of one type of open-volume defects (e.g., vacancies), the positrons may be trapped there with the trapping rate κ. The lifetime spectrum consists then of two exponential decay components. The positron lifetime in the defect τ_d (*d*...defect) is increased and, therefore, it is larger than τ_b due to the decrease in the electron density in the defect compared to the bulk. From the experimentally obtained fitting parameters τ_1, τ_2, and I_2 (lifetimes and intensities), the positron trapping rate may be determined by means of the average positron lifetime $\bar{\tau}=I_1\tau_1+I_2\tau_2$ with $\tau_2=\tau_d$. The positron trapping rate κ_d yields the defect concentration C_d, if the constant μ (trapping coefficient) could be obtained at least once by an independent method (e.g., Hall-effect measurements),

$$C_d=\frac{\kappa_d}{\mu}=\frac{1}{\mu\cdot\tau_b}\,\frac{\bar{\tau}-\tau_b}{\tau_d-\bar{\tau}}. \tag{1}$$

The so-called trapping coefficient μ depends strongly on the defect type, defect charge, and possibly on temperature. A compilation of experimental results of the determination of trapping coefficients for semiconductors was published by Krause-Rehberg and Leipner.[29]

A special feature of semiconductors is the occurrence of charged defects. The charge state of a defect in semiconductors depends on the position of the Fermi level in the energy gap. The charge gives rise to an additional coulombic tail of the positron potential. As a result, the following situation for a vacancy appears: Positively charged vacancies are repulsive for positrons, the positron trapping at neutral vacancy defects is distinct, and it is strongly enhanced at negative vacancies at low temperatures. This temperature dependence of trapping in negatively charged vacancies is due to positron trapping in and detrapping from extended Rydberg states.[30]

Additionally, shallow positron traps are possible candidates for positron localization sites. The long-range Coulomb field around negative ions can bind positrons to Rydberg-like states with small binding energies ($\ll$ 1 eV). The positron wave function is only weakly localized. The effectiveness of these shallow positron traps increases at low temperatures due to the small binding energies. Positrons are detrapped at higher temperatures. The annihilation characteristic of positrons in shallow traps agrees usually with that of free positrons in the bulk, in contrast to open-volume defects. Therefore, only the temperature dependence of positron lifetime as a result of competition between positron trapping at vacancies and at negative ions or the decreasing positron diffusion

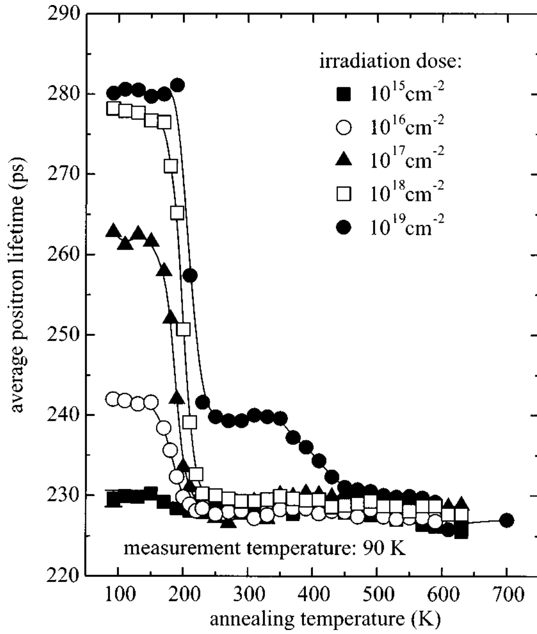

FIG. 2. The average positron lifetime as a function of the annealing temperature in germanium electron irradiated (2 MeV, 4 K) to different doses. The measurement temperature was 90 K.

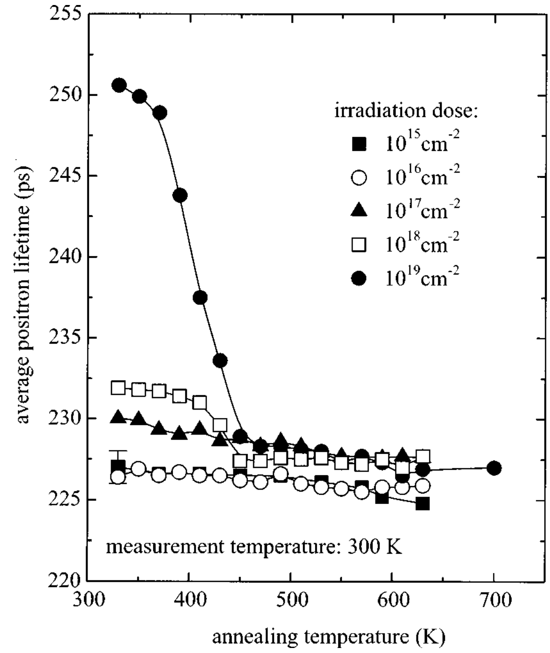

FIG. 3. The average positron lifetime in dependence on the annealing temperature in electron-irradiated Ge. The measurements were performed at 300 K.

length is an indication for the presence of shallow positron traps. The temperature dependence of the positron lifetime is dependent on the charge state of the vacancy defect and can be in various forms. The positron trapping in and detrapping from the shallow traps are analogous to those of the Rydberg states of negatively charged vacancies and can be described exactly the same way.[17] Besides negative ions, dislocation lines[31] and neutral defects (*A* center in Si)[32] can act as shallow positron traps due to their small open volume.

IV. RESULTS AND DISCUSSION

Undoped germanium grown by the Czochralski method was used for the present studies. Before irradiation, the Fermi level of the untreated material was located at $E_f - E_v = (275 \pm 5)$ meV. This position was unchanged up to an annealing temperature of 500 K.

A. Annealing experiments

Figure 2 shows the annealing curves of differently electron-irradiated Ge in the temperature range between 90 and 700 K detected by the average positron lifetime. The measurement temperature was 90 K and thus, equivalent to the temperature of the sample installation in the cryoheater system.

A main annealing stage between 150 and 250 K was observed for all of studied samples. The annealing temperature shifted to higher values with increasing dose. This can be attributed to the additional formation of more complicated defects by irradiation with a high dose. This annealing stage of the monovacancy at about 200 K was also detected in *p*-type Ge.[19]

A defect-related positron lifetime of (281 ± 2) ps was decomposed for the samples irradiated with an electron dose $\Phi \geqslant 10^{16}\,\mathrm{cm}^{-2}$. This value is typical of a monovacancy. After irradiation with $\Phi = 10^{15}\,\mathrm{cm}^{-2}$, the increase of the average positron lifetime was only 2.5 ps and the intensity of the defect component was too low to decompose a defect-related positron lifetime. The complete positron trapping in the vacancy defect was proved for the sample irradiated with the highest dose of $\Phi = 10^{19}\,\mathrm{cm}^{-2}$. The calculated value of the positron lifetime in the monovacancy of 265 ps[21] is lower than the experimental one. But also other experiments yield similar values for the monovacancy: 290, 292, and 278 ps.[19,20,15]

The Fermi-level position of about $E_f - E_v = 300$ meV was determined by Hall-effect measurements at room temperature for all of irradiated samples. In this sample state, the monovacancy should be detectable by positrons due to its negative charge according to calculations (see Fig. 1).[22]

These results agree with those of perturbed angular correlation spectroscopy (PACS) studies in electron-irradiated Ge.[33] The annealing of the vacancy was observed at 200 K and an annealing stage at 220 K was assigned to the Ge interstitial.

In addition to the main annealing stage at 200 K, a second annealing stage between 350 and 450 K was observed at a measurement temperature of 90 K for the sample irradiated with $\Phi = 10^{19}\,\mathrm{cm}^{-2}$ (see Fig. 2). If we consider the isochronal annealing curves measured at room temperature in Fig. 3, this second annealing stage was also detectable for irradiation doses $\Phi > 10^{17}\,\mathrm{cm}^{-2}$. This means, that the positron trapping in vacancies at low temperature (90 K) was influenced by the trapping in shallow positron traps. The temperature-dependent measurements of the positron lifetime (see Sec. IV C) explain this interpretation.

A defect-related positron lifetime of 281 ps was decomposed in the annealing process above 350 K for our samples. This value is equivalent to this of the monovacancy (see discussion above). The formation of the divacancy by mobile

monovacancies was favored in literature[10] and the annealing stage between 400 and 450 K observed by DLTS was attributed to the annealing of vacancy clusters.[2] Another possibility of the attribution of the annealing stage to a defect is a complex containing a vacancy and an impurity. The formation of such thermally more stable complexes was also observed in silicon.[34] A further argument for it is the defect-related positron lifetime, which is typical of a monovacancy. The formation of vacancy agglomerates must result in a significant increase of τ_2.

An acceptor level at $E_v + 0.22\,\mathrm{eV}$ detected by DLTS and the annealing of this one in the temperature range between 370 and 420 K was observed in electron-irradiated undoped p-Ge.[12] A suggested model for the defect complex is a divacancy connected with an oxygen atom, although a complex consisting of a monovacancy and oxygen cannot be excluded. The introduction rate of this defect by 2-MeV electrons at room temperature is very small, about $2 \times 10^{-4}\,\mathrm{cm}^{-1}$. This means that this defect should be detectable by positrons only for irradiation doses $\Phi \geqslant 10^{18}\,\mathrm{cm}^{-2}$.

From our positron results and the comparison with results from literature, we conclude that the annealing at 400 K observed by positrons can be attributed to the annealing of V-O complexes. This statement is also supported by the following facts: The Ge samples under investigation are nominally undoped and the main impurity in Czochralski-grown Ge is oxygen. The defect-related positron lifetime agrees well with this of the monovacancy. The experiments should be repeated on ''oxygen-free'' Ge grown by the floating-zone method to confirm this interpretation. But it is impossible because of the shutdown of the accelerator used for these electron irradiations.

There are two possible defect complexes acting as the vacancylike positron trap V_2O and VO. If oxygen occupies a vacancy, the open volume of the former complex is equivalent to that of a monovacancy and the latter complex is not a vacancylike defect. Such a behavior was detected in silicon. For the V_2O complex in silicon, a defect-related positron lifetime of the monovacancy was measured[35] and the *A* center (VO) acted as a shallow positron trap in Si.[32] If oxygen substitutes a Ge site near the vacancies, the open volume of V_2O is similar to that of a divacancy and the VO defect is comparable to the monovacancy. We cannot decide which of the two defects is responsible for the annealing stage at about 400 K.

The annealing stage at 500 K observed in electron irradiated p-Ge ($\rho = 2\,\Omega\mathrm{cm}$) and assigned to a vacancy-dopand complex[19] could not be detected in undoped germanium.

B. Dose dependence

Figure 4 shows the dependence of the average positron lifetime measured at 90 K and of the corresponding positron trapping rate on the irradiation dose. The positron lifetime of defect-free bulk of $\tau_b = 227$ ps was measured in unirradiated Ge. The average positron lifetime increased with irradiation dose and it achieved the saturation value of 281 ps for $\Phi \geqslant 10^{19}\,\mathrm{cm}^{-2}$.

The vacancy concentration results from Eq. (1) by using the corresponding trapping coefficient μ. The trapping coefficient of the negative Ge vacancy was not experimentally

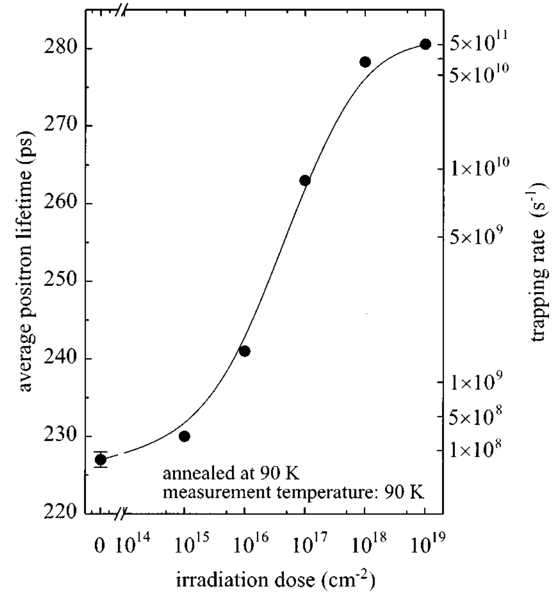

FIG. 4. The average positron lifetime and the trapping rate determined at 90 K in dependence on electron dose in Ge.

determined up to now. These irradiation experiments were not suitable for the determination of this trapping coefficient because of the lack of the exact defect introduction rates in Ge by electron irradiation. Therefore, only an estimation of vacancy concentrations and introduction rates was performed by using a trapping coefficient of $3 \times 10^{16}\,\mathrm{s}^{-1}$ determined for negative Ga vacancies in GaAs at low temperature as a lower limiting value.[36] The estimated Ge vacancy concentration increased from 3.8×10^{14} to $8.6 \times 10^{17}\,\mathrm{cm}^{-3}$ in the dose range under investigation.

The corresponding defect introduction rates decreased with increasing dose (from 10^{15} to $10^{19}\,\mathrm{cm}^{-2}$) and they were between 0.38 and 0.09 cm^{-1}. This behavior could be due to the increasing damage density, and thus due to the more probable recombination of defects. But no positron trapping in shallow positron traps was considered for this estimation of the vacancy introduction rate. This means that the declared values represent only lower limits of this introduction rate. The existence of shallow positron traps is discussed below (Sec. IV C).

C. Temperature-dependent investigations

Temperature-dependent measurements of the average positron lifetime were carried out for unirradiated material and for germanium irradiated with different doses after annealing at 230, 330, 450, and 570 K. The positron lifetime of bulk (227.5 ± 2) ps was measured in the untreated sample in the range from 100 to 550 K. Below 100 K the positron lifetime increased to 230 ps at a sample temperature of 20 K. From this behavior we can conclude that the unirradiated sample contained negatively charged open-volume defects of low concentration. With a trapping coefficient of $3 \times 10^{16}\,\mathrm{s}^{-1}$,[36] the defect concentration can be estimated to $4 \times 10^{14}\,\mathrm{cm}^{-3}$. This defect is at least stable up to the highest measurement temperature of 550 K and can be a complex of a vacancy and an impurity. Besides oxygen, also carbon is an important impurity in Cz-Ge and can form defect complexes of this concentration during the growth.

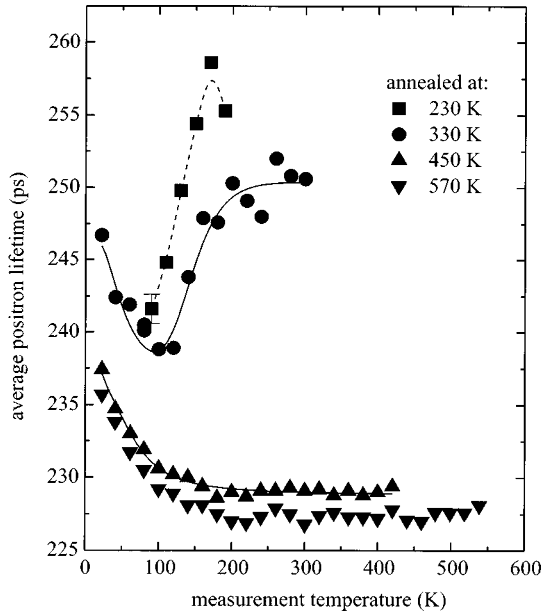

FIG. 5. The average positron lifetime as a function of the sample temperature in undoped Ge irradiated with 2-MeV electrons to a dose of $\Phi=10^{19}\,\mathrm{cm}^{-2}$ and after different annealing steps. The solid lines correspond to the trapping model. After annealing at 330 K negative V-O complexes and shallow positron traps are considered and after annealing at 450 K only negative vacancy defects are taken into account.

The positron lifetime was dependent on the temperature for the irradiated samples after all of the annealing steps. The temperature dependencies were most distinctly marked after irradiation with the highest dose and are discussed in the following. Figure 5 shows the average positron lifetime as a function on the sample temperature in undoped Ge irradiated with 2-MeV electrons to a dose of $\Phi=10^{19}\,\mathrm{cm}^{-2}$ and after isochronal annealing at 230, 330, 450, and 570 K.

After annealing at 230 K the positron lifetime distinctly decreased with decreasing sample temperature between 170 and 90 K. The main annealing stage is completed in this sample state and the dominating open-volume defect is a V-O complex (see discussion in Sec. IV A). The course of the positron lifetime indicated the existence of shallow positron traps. The positron trapping in these defects increased at low temperatures.

After the temperature treatment at 330 K, the sample could be cooled down to 20 K and the temperature dependence of the average positron lifetime was typical of competitive positron trapping in negatively charged vacancy defects and in shallow positron traps. With decreasing temperature, the positron lifetime decreased and reached at 100 K a minimum, then $\bar{\tau}$ increased again at lower temperatures. The behavior was similar to that in electron-irradiated silicon.[32]

The deep positron trap in the samples under investigation is a V-O complex (see discussion in Sec. IV A), which is the dominating vacancy defect after annealing at 330 K. The negative charge state of this defect agrees with the results of DLTS investigations. An acceptor level at $E_v+0.22\,\mathrm{eV}$ was determined.[12] Shallow traps are negatively charged centers without open volume or neutral defects with a small open volume. In undoped Ge is the only possibility of the interstitial as an intrinsic defect. But this defect anneals at 220 K.[33] Therefore, we conclude that an impurity acts as a shallow positron trap. A possible candidate is a defect containing oxygen such as the VO complex (*A* center) in silicon,[32] for example.

The fit according to the trapping model taking into account a negatively charged vacancylike defect ($\tau_d=281\,\mathrm{ps}$) and a negatively charged shallow positron trap described qualitatively right the temperature-dependent courses of $\bar{\tau}$ after annealing at 330 K (see the solid line in Fig. 5). Fits taking into account only negative vacancies or neutral vacancy defects and shallow traps did not agree with the temperature dependence of the average positron lifetime. The errors of the fit parameters were big due to their mutual dependence. An estimation of the most important parameters was obtained: the positron binding energy to shallow traps $E_{st}=70\,\mathrm{meV}$, the concentration of shallow traps, $C_{st}=2\times10^{17}\,\mathrm{cm}^{-3}$, the positron binding energy to Rydberg states of the V-O complex $E_R=10\,\mathrm{meV}$, and the concentration of this defect $C_V=1.5\times10^{17}\,\mathrm{cm}^{-3}$.

After annealing at 450 K, the temperature behavior of the positron lifetime changed. Above 200 K, the positron lifetime was constant and the value was close to the bulk lifetime of defect-free germanium. Below 200 K, $\bar{\tau}$ increased by 10 ps. One can conclude from these results that the shallow positron traps annealed in the temperature range between 330 and 450 K, and that then negative vacancylike defects of a low concentration were the dominating positron traps. A fit taking into account only a negatively charged vacancylike defect is shown as a solid line in Fig. 5. A defect concentration of $C_V=3\times10^{16}\,\mathrm{cm}^{-3}$ and a binding energy to the Rydberg states of $E_R=14\,\mathrm{meV}$ could be determined. The binding energy agreed quite good with that value obtained after annealing at 330 K.

The temperature-dependent course of the average positron lifetime was shifted to lower values of lifetime after annealing at 570 K compared to that after the 450-K treatment. In the temperature range above 150 K the average lifetime was equal to the bulk value, and below 150 K $\bar{\tau}$ still increased. The vacancy defects did not yet anneal complete. A similar behavior was observed for unirradiated Ge and thus, the defects induced during crystal growth made also a contribution to the temperature dependence.

V. CONCLUSIONS

Annealing experiments in undoped germanium after 2-MeV electron irradiation at 4 K showed two annealing stages. The main stage at 200 K could be attributed to the mobility of vacancies and interstitials, and therefore, to the recombination of Frenkel pairs. The existence of V-O complexes was observed for Ge irradiated to doses of $\Phi\geqslant10^{18}\,\mathrm{cm}^{-2}$. The disappearance of these defects was detected in the annealing range at 400 K.

The vacancy introduction rate after 4-K irradiation and annealing at 90 K decreased with increasing electron dose. The temperature of the main annealing stage was simultaneously shifted to higher values. This can be explained by intensified recombination of defects and by formation of more complex defects with increasing damage density.

Besides vacancylike defects, shallow positron traps were proved by temperature-dependent positron lifetime measurements in electron-irradiated Ge. These irradiation-induced defects were formed by acceptorlike impurities or neutral substitutional impurities with a small open volume and annealed out in the temperature range between 330 and 450 K.

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