Positron Annihilation in Neutron-Irradiated p-Type Silicon

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Effects of neutron irradiation and subsequent thermal annealing on positron-lifetime spectra in p-type silicon have been studied. In unirradiated samples, two annihilation rates (4.13×10^9 sec^{-1} with an intensity of 98% and 7.14×10^8 sec^{-1} with an intensity of 2%) are observed. The annihilation rate of the dominant component becomes smaller upon irradiation. The effect saturates at high integrated neutron flux, $\sim 1 \times 10^{17}$ n/cm^2, and disappears completely upon thermal annealing at 400–500 °C. The annealing behavior is dependent on the integrated neutron flux. These results show that some neutron-induced defects act as positron traps at room temperature. Our study also gives evidence that high-order vacancy defects are formed during annealing near 300 °C. The mean electron density of the positron-sensitive defects (positron traps) is estimated from the saturation value of the annihilation rate to be about 35% less than that in the perfect crystal. The diffusion coefficient and the mobility of thermalized positrons in silicon are estimated to be about 0.16 cm^2/sec and 6.4 cm^2/sec V, respectively.

INTRODUCTION

Positron-annihilation measurements have been recently demonstrated to be a useful tool for studying defects in metallic,[1–5] ionic,[6,7] and semiconductive solids[8,9] because positrons can be trapped in some defects where they annihilate with a distinct annihilation rate. MacKenzie *et al.*[1] have found a marked temperature dependence of the positron lifetime in certain metals. They suggested that vacancy formation is responsible for the effect. Some simple calculations[10,11] based on a double-lifetime model have satisfactorily reproduced the essential feature of the measurements. In the model, positrons can be trapped by a vacancy. The trapped positron will have a slower annihilation rate than one in the perfect crystal because of lower electron density at a vacancy site. Later, Connors *et al.*[2] observed effects of thermal quenching of samples on positron annihilation in cadmium metal by measuring the angular correlation of annihilation photons. Snead *et al.*[3] have observed effects of electron irradiation on positron annihilation in iron by using the same technique. Recently, McKee *et al.*[5] have successfully measured vacancy-formation energies in various metals observing the temperature dependence of the annihilating γ angular distribution. For ionic crystals, Tumosa *et al.*[6] have studied the interactions of positrons with defects in NaCl crystals produced by proton, γ-ray, or x-ray irradiations. Furthermore, Brandt *et al.*[7] have reported that V-center formation in NaCl crystals by x rays is correlated with the reduction of the positron-annihilation center responsible for the long-lived component in the lifetime spectrum. Fabri, Poletti, and Randone[8] have observed some enhancement of the positron lifetime in silicon, germanium, and gallium arsenide upon neutron irradiation. Most recently, Cheng and Yeh[9] have reported results from a study on the effect of 5.7-MeV proton irradiation on the positron-lifetime spectrum in p-type silicon, showing that some radiation-induced defects in semiconductors can act as positron traps. All the above-mentioned studies show that positrons can be trapped in some defects in crystalline solids. Thus, the positron-annihilation method is a useful tool for studying defects in solids.

In this paper, we present some results from a study on the effect of neutron irradiations and subsequent thermal annealings on positron-annihilation rates in p-type silicon single crystals.

EXPERIMENTAL PROCEDURE

The standard positron-lifetime measurement technique was used. A Na^{22} positron source was sandwiched by two wafer-form samples in which the annihilation is to take place and the spectrum of time delays between the 1.27-MeV prompt nuclear γ ray and one of the 0.51-MeV annihilation γ ray was recorded in a multichannel time-to-pulse-height conversion and coincidence sys-

tem. Positron lifetimes in solids are typically $(0.2-2)\times10^{-9}$ sec; compared with this, the mean time between the Na^{22} β decay and the subsequent emission of a 1.27-MeV Na^{22} γ ray is 3×10^{-12} sec [12] and the time required for the thermalization of a positron is in order of 10^{-12} sec.[13] Thus the measured coincidence curve is closely equal to the ideal lifetime spectrum of thermalized positrons folded into the instrumental time resolution of the measuring system.

Our measuring system is basically a commercially available Ortec γ-γ lifetime measuring system with a pair of 1-in.-diam, 1-in. long Naton 136 scintillators and two Philips 56 AVP photomultipliers. The prompt spectrum of the system at the setting for the positron-lifetime measurement for a Co^{60} source had a full-width of 360×10^{-12} sec at half-maximum (FWHM) and a decay slope of 100×10^{-12} sec. The peak-to-background ratio was about 2×10^{4}. In order to stabilize the overall system, a precise ac-line-voltage regulator was used. However, the laboratory room had no automatic temperature control system, even though it was air conditioned. Some drift in the time spectrum was found. The maximum drift observed was about 0.07×10^{-9} sec per 24 h, but typical was about 0.03×10^{-9} sec per 24 h.

The positron source consisted of Na^{22} radioactive isotopes in the form of NaCl sealed between two thin Mylar films of thickness 0.96 mg/cm^2. The source strength is about 1 μCi. The silicon samples were cut in wafer form 1 mm thick from a commercially available floating-zone refined p-type silicon ingot with a resistivity of 100 Ω cm at room temperature. The samples were polished with Al_2O_3 powder and then chemically etched.

The neutron irradiation was performed in the 1-MW Open-Pool Reactor of the National Tsing Hua University at room temperature. During irradiation, samples were sealed in an Al container placed near the reactor core. Because the fast-neutron spectrum of the reactor at the irradiation place was not known, we were forced to choose the intensity of the 1.8-μ radiation-induced band of the sample as fast-neutron flux monitors. The production rate of the 1.8-μ band under fission neutron irradiation at room temperature was obtained from the work of Cheng and Lori.[14] Our estimated neutron flux is equivalent to about 1.86×10^{11} fission neutrons per cm^2/sec. As pointed out in Ref. 14, the absolute error in their value can be about 50%. Some other error should be also considered, such as the one in divacancy production rate due to the difference in the neutron-energy spectrum. However, the relative values among all the flux measurements reported in this paper are only in error by about 5%. The 1.8-μ band intensity was measured with a Cary 14 spectrometer at room temperature. The annealing experiments were done in a temperature-controlled oven.

EXPERIMENTAL RESULTS

Figure 1 shows two typical delay-time spectra between 1.27-MeV γ rays and 0.51-MeV positron-annihilation rays from a pair of 100-Ω cm p-type silicon samples before and after 150-h neutron irradiation in the Tsing Hua reactor. By graphical analysis, each spectrum can be fitted best with two decay components plus a constant background. The two components correspond to two positron-annihilation rates. They are $(4.13\pm0.08)\times10^{9}$ and $(7.1\pm1.0)\times10^{8}$ sec^{-1} for the unirradiated samples (solid dots) and $(3.32\pm0.06)\times10^{9}$ and $(7.1\pm1.0)\times10^{8}$ sec^{-1} for the irradiated samples (open dots). The relative intensities of the two components before and after irradiation are about 98% for the faster annihilation rate and about 2% for the slower one. Neutron irradiation decreases only the annihilation rate of the dominant fast component. The irradiation has no significant effect on the relative intensities. Since the 2% component remains unchanged, we shall not discuss it further in the following.

The dominant annihilation rate in the unirradiated samples agrees closely with the most recent data[15,16] and the theoretical value of Brandt and Reinheimer,[17] and only slightly smaller than the measured value of Wiesberg and Berko.[18]

Figure 2 presents the change of the annihilation rate versus the integrated neutron flux (i.e., neutron fluence). The annihilation rate decreases with increasing integrated neutron flux, approaching

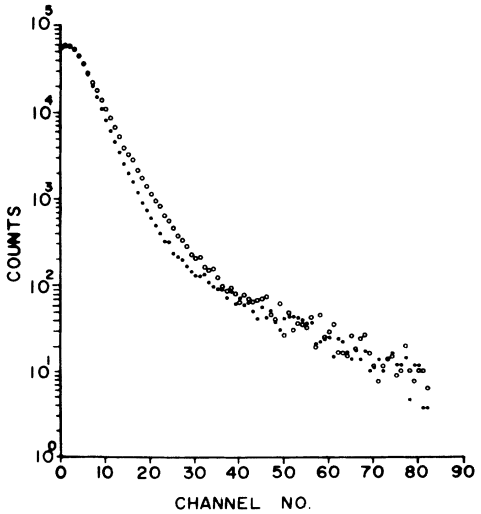

FIG. 1. Typical delay-time spectra between 1.28-MeV γ rays and 0.51-MeV positron-annihilation rays from a pair of 100-Ω cm p-type silicon samples before and after an irradiation of 1×10^{17} fast neutrons/cm^2.

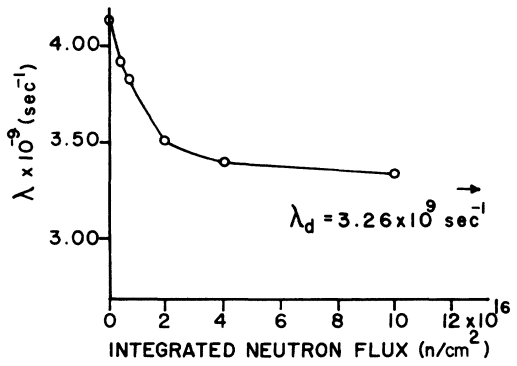

FIG. 2. Annihilation rate vs integrated neutron flux.

a saturation limit at the large integrated neutron flux.

For our discussion of the data, we apply the lifetime spectrum in the solid with defects acting as positron traps derived first by Brandt, [19,20]

$$n(t)=\left(1-\frac{\kappa}{\kappa+\lambda_c-\lambda_d}\right)e^{-(\kappa+\lambda_c)t}+\frac{\kappa}{\kappa+\lambda_c-\lambda_d}\,e^{-\lambda_d t}\,, \tag{1}$$

where $n(t)$ is the fraction of positrons surviving at time t, λ_c the annihilation rate in the perfect part of the crystal, λ_d the annihilation rate in the defect, and κ the capture rate of positrons by the defects. If the difference between λ_c and λ_d is not large compared to the time resolution of the instrument, the presence of the defect will manifest itself only through a decrease of the mean annihilation rate. [10,11,20] Then the measured value of the annihilation rate can be written as

$$\lambda=\left(\int_0^\infty t\,\frac{dn(t)}{dt}\,dt\right)^{-1}. \tag{2}$$

Using Eq. (1), we have

$$\lambda=\frac{\lambda_d(\lambda_c+\kappa)}{\lambda_d+\kappa}\,,$$

or

$$\frac{\lambda-\lambda_c}{\lambda_d-\lambda}=\frac{\kappa}{\lambda_d}=\frac{\mu C_d}{\lambda_d}=\frac{\mu\eta\varphi}{\lambda_d}\,, \tag{3}$$

where μ is the positron capture rate per defect in unit volume, C_d is the defect concentration, η is the number of defects per unit volume produced by an incident neutron per cm^2, and φ is the integrated neutron flux received by the sample. Applying Eq. (3) to our data in Fig. 2, we obtain $\lambda_d=3.26\times10^9$ sec^{-1}. This value is close to the observed value of 3.32×10^9 sec^{-1} from the samples irradiated with 1×10^{17} n/cm^2. Figure 3 depicts $\Delta\equiv(\lambda-\lambda_c)/(\lambda_d-\lambda)$ versus the integrated neutron flux. The slope corresponds to $\mu\eta/\lambda_d$. From the measured slope and the experimental value of λ_d, we obtain $\mu\eta=4.5\times10^{-7}$ (sec cm n)$^{-1}$, where μ is in cm^3/sec defect and η is in defect/cm n. It should be noticed that μ, η, and λ_d are average quantities, because the spectrum of defect size in neutron-irradiated silicon is not known.

Figure 4 presents 30-min isochronal-annealing data from three pairs of samples irradiated with various neutron fluences. The dependence of the annealing behavior of positron-sensitive defects on neutron integrated flux appears clearly in the figure. The data from a pair of samples irradiated with 1×10^{17} fast neutrons/cm^2 (the upper curve) show the existence of one annealing stage at 450 °C. This is different from the data obtained from two pairs of samples irradiated with neutron integrated flux substantially less. The annealing data of the samples irradiated with 4×10^{16} fast neutrons/cm^2 (the middle curve) indicate the existence of three annealing stages around 150, 300, and 400 °C. The annealing data of the other pair irradiated with 2×10^{16} fast neutrons/cm^2 (the lower curve) also indicate the existence of these three annealing stages. It should be noticed that in annealing stage of 300 °C, the increase of Δ upon annealing was observed.

DISCUSSION

A. Neutron-Irradiation Effect

It is well known that fast-neutron irradiations introduce lattice defects in silicon crystals. The observed decrease of the annihilation rate of the dominant component in silicon due to neutron irradiations gives evidence that positrons can be trapped in some radiation-induced defects in which the electron density is lower than that in the perfect bulk of the crystal. These traps are vacancy-type defects. As the concentration of the defects increases with the irradiation, an increasing fraction of positrons is trapped. Finally, almost all positrons annihilate with an electron in a defect.

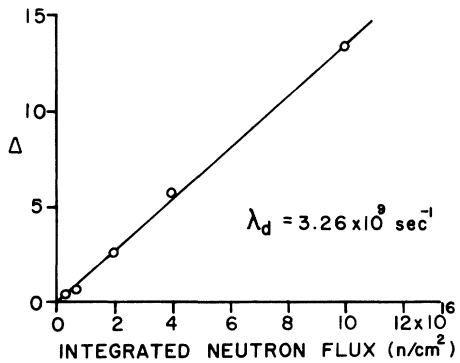

FIG. 3. Value of Δ vs integrated neutron flux.

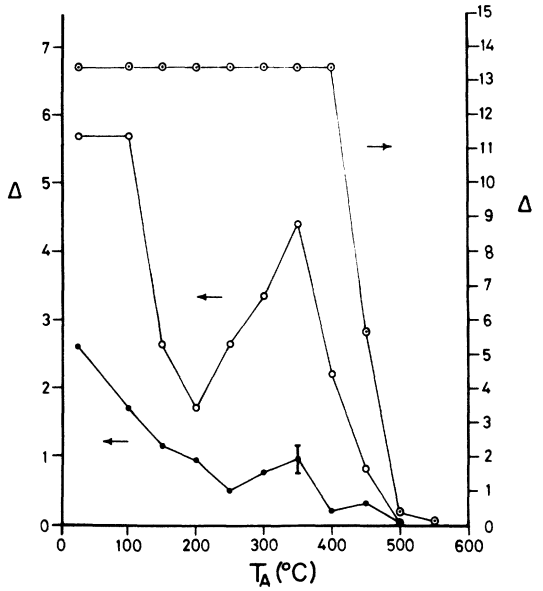

FIG. 4. Isochronal-annealing data from three pairs of samples irradiated with 2×10^{16} (darkened circle), 4×10^{16} (open circle), and 1×10^{17} fast neutrons/cm^2 (circle with a dot).

Then no further decrease of the annihilation rate can be observed, in accord with the saturation effect shown in Fig. 2. The saturation value of $\lambda = 3.26\times10^9$ sec^{-1} is equal to λ_d. This is indicative of the mean electron density of the defect which has been already pointed out in our previous paper.[9]

The annihilation rate in a semiconductor can be written in the form[17]

$$\lambda = (12.0/r_s^3)\{1+f(r_s,\ E_g)[\xi(r_s)-1]\}, \qquad (4)$$

where $\xi(r_s)$ is the enhancement factor, $f(r_s, E_g)$ is the correction factor due to the existence of an energy gap E_g in a semiconductor, and $r_s = (3/4\pi n_e)^{1/3}$ (n_e is the mean valence electron density of the material in atomic units; $n_e = 0.0315$ for silicon). From the observed value of λ_c with $r_s = 1.98$, $f(r_s, E_g) = 0.903$,[17] and $[\xi(r_s)-1]\alpha r_s^{2}$[17] for Si, Eq. (4) can be approximated as

$$\lambda = (12.0/r_s^3)(1+0.42r_s^2)\ . \qquad (5)$$

If one assumes that Eq. (5) is valid also for the defect and sets $\lambda = \lambda_d$, one can estimate a mean electron density of the defect with no trapped positron; the result is $0.65n_e$. This estimate is based on two simplifying assumptions: (i) Eq. (5) applies in the defect as well as in the crystal bulk, and (ii) the screening length of the potential owing to the positron in the defect is smaller than the linear dimension of the defect. This is a rough estimate. Extensive calculations are needed for obtaining more detailed values than the presented one.

It is of interest to note that the statistical weights for larger defects in the defect distribution produced by neutron bombardments will increase, when the integrated flux is larger than a certain value at which the overlapping of damaged regions becomes important. Presumably the mean electron density of a defect containing larger number of vacancies is lower than that of a defect composed of a smaller number of vacancies. Thus the saturation value of the annihilation rate deduced from low-integrated-neutron-flux data should be larger than that obtained from very-high-integrated-neutron-flux experiments.

With the aim of checking a possible correlation between positron-annihilation features and lattice defects, Fabri *et al.*[8] performed one irradiation with an integrated neutron flux of about 2×10^{18} n/cm^2 on silicon crystals. The annihilation rate of the dominant component was decreased from $(3.9\pm0.4)\times10^9$ to $(2.8\pm0.4)\times10^9$ sec^{-1} upon the irradiation. Our saturated value of 3.26×10^9 sec^{-1} is slightly larger than their value after the irradiation, supporting the above prediction. The recent observation of the decrease of the annihilation rate of the trapped positron in voids in molybdenum with integrated neutron flux is another example.[21] However, no voids in silicon have been observed at an integrated neutron flux about 5000 times larger than the one used in our study.[22] And the production rate of the divacancy in silicon is constant under fission neutron irradiations in the integrated flux range similar to ours.[14] No overlapping of the damaged regions is predicted in our study. Therefore, under our conditions, λ_d should be constant, independent of the integrated neutron flux.

B. Defect Production and Positron Mobility

From Fig. 3, we have obtained the value of $\mu\eta = 4.5\times10^{-7}$ (cm sec n)$^{-1}$. If one knows the capture rate, one can find the value of η, or vice versa. Neither theoretical calculation nor experiment on the positron capture rate by a defect in a semiconductor has been published. Since the situation for defects produced by fast-neutron irradiations is very complicated, the observed value of $\mu\eta$ should be considered to be a mean over $\mu\eta$ of all positron-sensitive defects in the crystal. Nevertheless, with some reasonable assumptions, valuable information about the behavior of thermalized positrons can be obtained. It is known that the defects produced by fast neutrons are in the form of damaged regions which are defect rich and crystalline.[14] We first assume that each damaged region acts as a defect unit, i.e., a positron trap, because the production rate of the damaged region per unit flux can be estimated to be 0.15 cm^{-1} using a known scattering cross section of 3 b.[14] This leads to $\mu = 2.9\times10^{-6}$ sec^{-1} per region. Then we assume that the dam-

aged regions are spherical with an average radius of 150 Å.[23,24] According to Dienes and Vineyard,[25] the capture rate per trap in a solid with spherical traps can be written as

$$\mu = 4\pi DR ,$$

where D is the diffusion coefficient of the positron and R is the effective capture radius of the trap (i.e., the damaged region). Using this, we have $D = 0.16$ cm^2/sec. This is approximately the diffusion coefficient of the thermalized positron in silicon.

Using the Einstein relation between diffusion coefficient and the mobility of a charged particle in solids (i.e., $\mu_p/D = e/kT$, where μ_p, e, k, and T are mobility, charge, the Boltzmann constant, and the temperature, respectively), we find $\mu_p = 6.4$ cm^2/sec V for the thermalized positron. This result may be correct within the order of magnitude. However it does give two interesting features. First, the thermalized positron in silicon is not very mobile in comparison to the electron and the hole in silicon, since the mobilities of the electron and the hole are 1700 cm^2/sec V and 350 cm^2/sec V at 300 °K.[26] Second, the thermalized positron in silicon diffuses faster than its counterpart in KCl, since the diffusion coefficient of the positron in KCl is reported to be $\sim 5\times10^{-4}$ cm^2/sec.[27] By comparison, positronium in fused quartz diffuses much slower than the positron in crystalline solids, since the diffusion coefficient in fused quartz is reported to be about 4.5×10^{-5} cm^2/sec.[20]

C. Defect Annealing

From Fig. 4, it is clear that the observed annealing behavior is dependent on the integrated neutron flux received by the samples. We discuss first the results from the sample pairs irradiated with 2×10^{16} n/cm^2 and 4×10^{16} n/cm^2 (lower and middle curves in Fig. 4) because the gross features of the annealing for these pairs are similar to each other and are different from that of the sample pair irradiated with 1×10^{17} n/cm^2 (upper curve in Fig. 4). The radiation effect on λ is saturated in the latter.

For the sample pairs irradiated with 2×10^{16} and 4×10^{16} n/cm^2, there are three annealing stages. The temperature ranges of these stages can be roughly stated to be ~150, ~300, and ~400 °C. The decrease in Δ in the first stage can be attributed to the decrease of defects accessible to positrons. There are two possible sources responsible for the decrease. One is the disappearance of the defects upon annealing. The 1.8-μ absorption band arising from the neutral divacancy[14] in these samples disappeared upon annealing in the temperature range of this first stage. The other is that some defects accessible to positrons before annealings become inaccessible because of the rise of the position of the "local" valence band in the damaged region with respect to the Fermi level of the crystal following the annealing of divacancies and other possible defects. In p-type silicon, a rise of this type will change the defect charge state toward the positive side.

The growth of the positron-sensitive defect at the second stage (~300 °C) can be attributed to two possible processes. The first is the formation of new high-order vacancy defects (e.g., trivacancies, tetravacancies, and pentavacancies[28]) following long-range outward migrations and combinations of smaller vacancy defects (e.g., single vacancies and divacancies). The single vacancies and the divacancies can be created and emitted from the damaged region during the annealing. Some divacancies can also exist outside of the damaged region before the annealing.[29] The second possible process is that some vacancy defects (larger than the divacancy) originally hiding in the inner part of the damaged regions become accessible to positrons following the outward migration of themselves and/or of other defects. In the present study, we have observed that the value of Δ after 350 °C annealing for the sample pair irradiated with 4×10^{16} n/cm^2 is about four times greater than that for the sample pair irradiated with 2×10^{16} n/cm^2 (see Fig. 4). This result indicates that the first process is the dominant process, because the first process should lead to a prediction that the value of Δ is proportional to the square of integrated neutron flux.

The third annealing stage (~400 °C) is attributed to the complete disappearance of all positron-sensitive defects in the samples.

There have been several works observing the growth of defects in the temperature range of 250–380 °C in neutron-irradiated silicon. Jung and Newell[30] observed four EPR spectra in neutron-irradiated silicon which appeared upon annealing around 250 °C, reached maxima in intensities around 350 °C, and then disappeared around 450 °C. However, they reported that the defects were oxygen dependent. In our samples, the interstitial-oxygen 9-μ absorption band was not detectable, indicating the oxygen concentration must be less than 1×10^{16} atoms/cm^3. Therefore the correlation between our positron-sensitive defects in this annealing stage (~300 °C) and those EPR centers is not proper. Recently Lee, Kim, and Corbett[31] have reported the observation of several new EPR spectra in neutron-irradiated silicon which are considered to be intrinsic defects, independent of impurities. One EPR center (Si-A7) was observed after the annealing at 270 °C. No further information on this EPR center has been reported, so far. Furthermore, Chen *et al.*[32] have reported that several new absorption bands appear upon annealing

around 300–380 °C and disappear around 400–500 °C in neutron-irradiated or 30–50-MeV electron-irradiated silicon samples ($\rho \gtrsim \Omega$ cm). They have concluded that the defects causing these bands are impurity independent and consist of at least a triple vacancy or a double divacancy. Are the infrared-sensitive defects related to the positron-sensitive defects observed in our study? This is still an open question.

Now we are going to discuss the data from the sample pair irradiated with 1×10^{17} n/cm^2 (see upper curve in Fig. 4). We have observed no change in Δ in the sample pair upon annealing up to 400 °C. This observation should not be considered as an indication that there was no change in defect concentration and/or defect forms upon the annealings. As a matter of fact 1.8-μ band in the samples disappears upon annealing in the temperature range from 100–250 °C, similar to that in other samples of unsaturated cases. The constancy in Δ only indicates that the total concentration of the positron-sensitive defect is still equal to or higher than a certain saturation concentration at which most of positrons annihilate themselves with electrons in the defects. However, the constancy may indicate that the mean electron densities of simple defects (stable below 250 °C, e.g., the divacancy) is roughly the same of high-order vacancy defects (formed after 300 °C thermal annealings). The major defect annealing in the saturated samples happened at 450 °C which is higher than that in the unsaturated samples, indicating again the existence of effect of integrated neutron flux (or concentration effect) on defect annealing behavior. This is similar with the result of Stein[33] that the annealing of an electrically active defect is dependent on integrated neutron flux.

CONCLUSIONS

From our present study, the following conclusions have been made.

(i) Some neutron-induced defects can act as positron traps at room temperature in silicon. They are vacancy-type defects.

(ii) The observed saturation effect on the annihilation rate at high-neutron fluences also supports the trapping effect.

(iii) The mean electron density of the positron-sensitive defect can be deduced from the observed saturation value of the annihilation rate. The deduced value from our study is about 35% less than that in the perfect part of the crystal.

(iv) The diffusion coefficient and the mobility of the thermalized positron in silicon can be roughly estimated from our experimental data. They are 0.16 cm^2/sec and 6.4 $\text{cm}^2/\text{sec V}$, respectively.

(v) Multivacancy defects are considered to be formed during the annealing stage around 300 °C.

(vi) Most of the positron-sensitive defects in silicon disappear upon annealing around or below 450 °C.

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