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Positron annihilation in silicon in thermal equilibrium at high temperature

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Abstract. Annihilation characteristics of positrons in Si in thermal equilibrium at high temperature were studied using a monoenergetic positron beam. The line-shape parameter of the Doppler broadening spectrum for the annihilation of positrons at the surface S_s and the diffusion coefficient of positrons D_+ were determined in the temperature range 298–1618 K. At 298–1373 K, because of the thermal desorption of positrons from the surface state and a resultant formation of positronium (Ps), the value of S_s increased with increasing temperature. However, it started to decrease above 1473 K. The decrease in the value of S_s was attributed to the suppression of the Ps formation due to the surface melting. With increasing temperature, the value of D_+ was decreased by the scattering of positrons with acoustic and optical phonons, and above 1073 K, it was almost constant; the lower limit of D_+ was determined as 0.48 cm^2 s^{-1}. From the analysis of Doppler broadening spectra, the behaviour of positrons in bulk Si at high temperature are also discussed.

1. Introduction

Positron annihilation has become an established means of studying point defects in metals and semiconductors [1]. When a positron is implanted in condensed matter, it annihilates with an electron mainly into two 511 keV γ rays. The motion of the positron–electron pair causes a Doppler shift in the energy of the annihilation radiation. In a material containing defects, a freely diffusing positron may be localized in a vacancy-type defect by Coulomb repulsion from ion cores. Since the momentum profile of the electrons in such defects is different from that in the bulk, one can detect vacancy-type defects through measurements of Doppler broadening spectra of the annihilation radiation [2]. When positrons are trapped by vacancy-type defects, their lifetime increases because of a reduced electron density in such defects. Thus, one can also obtain information about vacancy-type defects from measurements of lifetime spectra of positrons.

After the implantation of positrons, they rapidly slow down to thermal energy. After this thermalization process, the transport properties of positrons are similar to those of electrons and holes. Since the behaviour of the thermalized positrons dominates the trapping process of positrons by defects [1], the study of the diffusion of positrons is important. The investigation of the positron motion also can improve the theory of the diffusion of light particles [3]. From

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a technical point of view, knowledge of the positron motion is crucial, because it relates to the efficiency in slow/monoenergetic positron production and to the resolution of a positron annihilation microscope [4].

From measurements of Doppler broadening spectra as a function of voltage across metal/semiconductor structures, the mobilities of positrons were determined for Si and Ge [5]. A monoenergetic positron beam was also used to study the diffusion process of positrons. In this technique, slow/monoenergetic positrons are implanted into a specimen at various incident positron energies, and their probability to return back to the surface was measured [4]. The results were analysed using a one-dimensional diffusion model of positrons, and information about the positron motion was derived. Using this method, Hautojärvi and co-workers [1, 6, 7] systematically studied the positron motion in semiconductors. For Si, however, the number of reports about the behaviour of positrons above 1000 K is still limited (the melting point T_m of Si is 1688 K). From another point of view, because of its importance in semiconductor technology, the surface roughening [8] and the surface melting [9] were extensively studied for Si. Because of the high sensitivity of positrons to vacancy-type defects, one might obtain information for surface atomic structures at high temperature using the mono-energetic positron beam. In the present paper, we discuss positron motion and the annihilation characteristics of positrons in Si in the temperature range 298–1618 K.

2. Experimental

The basic concept of the positron annihilation technique used in the present experiments is as follows. The energy distribution of annihilation γ rays is broadened because of the momentum component of the annihilating positron–electron pair P_L, parallel to the direction of the emitted γ rays. The energy of the γ ray is given by the relation: $E_\gamma = 511 \pm \Delta E_\gamma$ keV. The Doppler shift ΔE_γ is obtained by the relation: $\Delta E_\gamma = P_L c/2$, where c is the speed of light. The change in the Doppler broadening spectrum is characterized by the S parameter or the W parameter [2]; S and W mainly characterize the fraction of the annihilation of positron–electron pairs with the low-momentum distribution and that of the annihilation of the pairs with the high-momentum distribution, respectively.

Undoped Si wafers with (100) orientation grown by a floating zone (Fz) method were used in the present experiments. The resistivity of the specimen was above 4000 Ω cm, and the oxygen concentration was below 1×10^{17} cm^{-3}. Before the measurements, the specimens were chemically etched for removal of surface contamination and natural oxide layers; SC1 (NH_4OH:H_2O_2:H_2O = 1:1:5) and SC2 (HCl:H_2O_2:H_2O = 1:1:5) methods were used. After such treatments, the specimens were etched by HF (HF:H_2O = 1:100), then inserted into the sample chamber within 10 min. The specimen was heated with a halogen lamp, and the temperature of the specimen was monitored with a thermocouple located below the specimen. The pressure of the sample chamber was 1–3 $\times 10^{-8}$ Torr, and it was not changed during the measurements.

The Doppler broadening spectra were measured with a Ge detector as a function of incident positron energy E. At each E, 1×10^6 counts were accumulated, and the spectrum was characterized by the S parameter. The central region of the spectrum was defined as 511 ± 0.75 keV. The specimen was heated from 298 K to 1618 K with steps of 100 K. At each measurement temperature, the deviation of the temperature was ± 0.5 K. The temperature dependences of S and W (the wing region of the spectrum was defined as from 511 ± 3.0 keV to 511 ± 6.1 keV) for the annihilation of positrons in the bulk were also measured. In this experiment, E was fixed at 25 keV, and the specimen was heated with a rate of 4 °C h^{-1}. A two-parameter multichannel analyser was used in order to measure the Doppler broadening

spectrum and the sample temperature automatically. At each temperature, about 5×10^6 counts were accumulated.

The observed relationship between S and E was analysed using the computer code VEPFIT developed by van Veen *et al* [10]. The one-dimensional diffusion model of positrons is expressed by [4]

$$D_+ \frac{\mathrm{d}^2 n(z)}{\mathrm{d}z^2} - \kappa_{eff} n(z) + P(z, E) = 0 \tag{1}$$

where D_+ is the diffusion coefficient of positrons, $n(z)$ is the probability density of positrons at a distance z from the surface, κ_{eff} is the effective escape rate of positrons from the diffusion process and $P(z, E)$ is the implantation profile of positrons. For the specimen with no defect response, κ_{eff} is related to the lifetime of positrons annihilated from the free state τ_f by $\kappa_{eff} = 1/\tau_f$. The relationship between the diffusion length of positrons L_+ and κ_{eff} is given by

$$L_+ = \sqrt{D_+/\kappa_{eff}}. \tag{2}$$

$P(z, E)$ can be described by [4]

$$P(z, E) = \frac{m z^{m-1}}{z_0^m} \exp\left[-\left(\frac{z}{z_0}\right)^m\right] \tag{3}$$

where m is the shape parameter of $P(z, E)$. Here, z_0 is proportional to the mean implantation depth of positrons $\bar{z}$ as follows: $z_0 = \bar{z}/\Gamma[(1/m) + 1]$, where Γ is the gamma function. The mean implantation depth $\bar{z}$ has a power-law dependence on E: $\bar{z} = AE^n$, where A is a constant dependent on the density of the specimen, ρ ($A = C/\rho$, C is a constant) and n is an energy- and a material-dependent constant. In the present work, the values of m, n and C were fixed as 2, 1.6 and 3.32 μg cm^{-2} keV$^{-1.6}$, respectively [11]. Using equations (1) and (3), we can calculate the fraction of positrons diffused back to the surface $F_s(E)$ and that of positrons annihilated in the bulk $F_b(E)$. Positrons implanted into the specimen might be re-emitted before being fully thermalized [4]. In VEPFIT, the fraction of the epithermal positrons at the surface is given by

$$F_{ep}(E) = \int P(z, E) \exp(-z/L_{ep})\, \mathrm{d}z. \tag{4}$$

In equation (4), it is assumed that the epithermal emission arises due to the elastic scattering with the scattering length L_{ep}. The observed S–E relationships were fitted to

$$S(E) = S_{ep} F_{ep}(E) + S_s F_s(E) + S_b F_b(E) \tag{5}$$

where S_s and S_b are the characteristic values of the S parameter for positrons annihilating at the surface and in the bulk, respectively ($F_{ep}(E) + F_s(E) + F_b(E) = 1$). Here, S_{ep} is the characteristic value of S for the epithermal positrons. From the fitting using equation (5) to the experimentally obtained S–E relationship, the values of S_{ep}, S_s, S_b and L_+ were determined.

3. Results and discussion

Figure 1 shows some examples of the S–E relationship for Fz–Si measured at different temperatures ($\bar{z}$ is shown in the upper horizontal axis). At high E (>15 keV), S approaches a constant value, indicating that almost all positrons were implanted into the bulk in this energy range and annihilated in the bulk without diffusing back to the surface. The value of S increased with decreasing E. This is due to the annihilation of positrons and of positronium (Ps) at the surface of the specimen [4]. The value of S near the surface increased with increasing temperature. This is attributed to the increase in the formation probability of Ps due to the

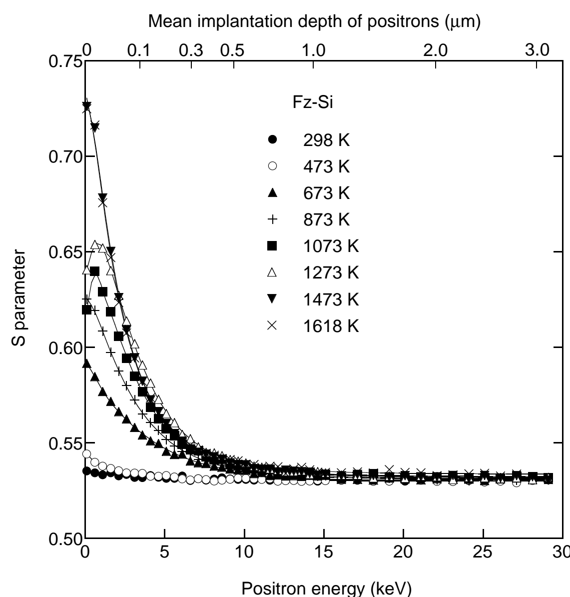

Figure 1. Examples of the *S–E* relationship for Fz–Si measured at different temperatures. The full curves are fits of equation (5) to the experimental data. The derived parameters are summarized in figures 2 and 3.

thermal desorption of positrons from the surface state [4]. The observed *S–E* relationships were fitted by equation (5). The full curves shown in figure 1 are the results of the fitting. The derived values of S_{ep}, S_s and S_b are summarized in figure 2. Using the value of L_+ and equation (2), the value of D_+ was calculated, where the temperature dependence of τ_f was assumed as $\tau_f = 216+0.005T$ ps [12]. The obtained temperature dependence of D_+ is shown in figure 3. The derived values of L_{ep} were about 10 nm. In the fitting procedure, L_{ep} can be varied without seriously affecting the goodness of fit and the values of the other fitting parameters. Kong and Lynn [13] developed the model for the epithermal positron transport employing the one-dimensional Boltzmann equation. According to their results [13], although L_{ep} is treated as the elastic scattering length in VEPFIT, it seems to be more related to the inelastic mean free path, and can be characterized as 'thermalization length'. Since the derived value of L_{ep} shows no clear temperature dependence in the present experiment, the discussion about the temperature dependence of the inelastic scattering is not given in the present paper.

The values of S_{ep} and S_s are affected by not only the change in the formation probability of Ps but also the momentum distribution of positron–electron pairs. Thus, information obtained from S leaves uncertainty. In the present paper, therefore, the behaviours of S_{ep} and S_s are discussed based on knowledge of the nature of Si surfaces. In figure 2, at 298–1373 K, because of the thermal desorption of positrons from the surface state and the resultant formation of Ps, the value of S_s increased with increasing temperature. Above 1473 K, however, it started to decrease. Fraxedas *et al* [14] reported that a Si(100) surface undergoes an incomplete melting transition at about 1400 K. The surface melting of Si is characterized by the formation of a liquid layer in about two topmost surface planes [9]. For a Si(111) surface, the onset temperature of the surface melting was also reported as 1414 K [15]. The formation probability of Ps depends on the number of positrons at the surface. Above the onset temperature of the surface melting, the thermalized positrons diffusing back to the surface could be trapped by open spaces in the

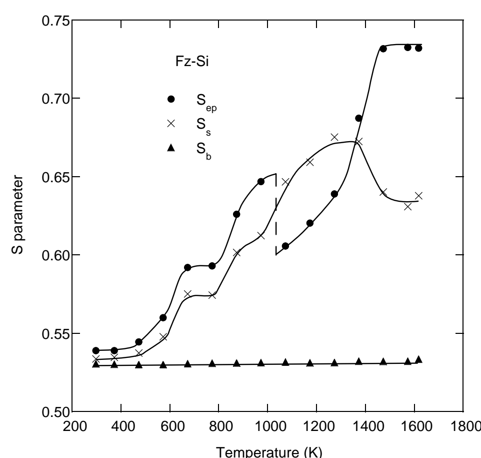

Figure 2. Temperature dependences of the characteristic values of S for the annihilation of the epithermal positrons S_{ep}, the thermalized positrons at the surface S_s and in the bulk S_b.

liquid layer and as a result, the number of positrons that are available for the Ps formation could decrease. The characteristic value of S for the annihilation of positrons in the liquid layer is expected to be larger than S_b [12], but is thought to be smaller than the characteristic value of S for the annihilation of Ps. Thus, the decrease of S_s above 1473 K can be reasonably explained by such a model. In the same temperature range, no decrease of S_{ep} was observed. Because the kinetic energy of the epithermal positrons is high, their probability of being trapped by open spaces in the liquid layer is thought to be lower than that of the thermalized positrons.

A thin oxide film is known to be formed on the surface of Si by chemical etching using dilute HF solution. For such an oxide film, the oxygen desorption (in the SiO_2 form) starts above 800–900 K [16]. Thus, the effect of the oxide film on S is likely to be neglected above 1000 K. Another major contamination at the surface is thought to be carbon. Effects of annealing on the structure and the composition of thin carbon films were reported by Martin-Gago *et al* [17]. In their report, after heating the carbon film deposited on the Si surface to 1223 K, both the grafitization and the formation of SiC were found. Once the SiC film is formed, it is expected to be stable even at high temperature. In the present experiments, after the measurement, the specimen was replaced from the sample chamber, and characterized using Auger electron spectroscopy with Ar^+-ion sputtering, although no clear evidence of the formation of SiC (and SiO_2) was obtained. As mentioned above, thin oxide films on Si become unstable at 800–900 K, indicating that the Si surface is etched away by the oxygen desorption. Therefore, above 1000 K, the effect of the surface contamination on S is considered to be small. In figure 2, in the temperature range between 298 K and 973 K, S_{ep} increases with increasing temperature, and suddenly decreases at 1073 K. However, that is not the case for S_s. Since this temperature range (973–1073 K) is close to the onset temperature of the oxygen desorption, the effect of the surface contamination on S cannot be neglected. Thus, the discussion of S_{ep} and S_s below 1000 K is not made in the present paper.

In figure 3, the value of D_+ decreased with increasing temperature. Mäkinen *et al* [6] and Soininen *et al* [7] reported that D_+ varies as $T^{-\frac{1}{2}}$ in the temperature range between 30 and 500 K. This means that the diffusion of positrons is controlled by the interaction between positrons and longitudinal acoustic phonons. In their report, at 500–1000 K, the temperature dependence of D_+ deviated from the $T^{-\frac{1}{2}}$ dependence. This fact was explained by the onset of the optical-phonon scattering. In the band structure of positrons, the lowest positron state

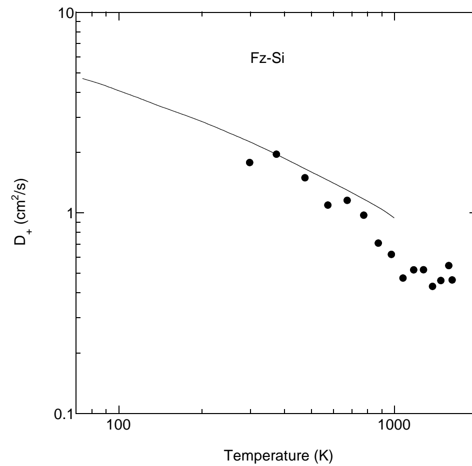

Figure 3. Temperature dependence of the diffusion coefficient of positrons D_+. The full curve is reproduced from the results obtained by Mäkinen *et al* [6] and Soininen *et al* [7].

is at the Γ point [18]. Soininen *et al* [7] suggested that the zeroth-order optical deformation-potential parameter for intraband scattering vanishes for an s-like band minimum at the Γ point, but the first-order optical-phonon processes are still possible. Because of the interaction between positrons and optical phonons, D_+ approximately follows the power law $T^{-\frac{3}{2}}$ [7]. The results reported by Mäkinen *et al* [6] and Soininen *et al* [7] are reproduced and shown in figure 3 as a full curve. Below 1000 K, although the full curve is higher than the value of D_+, the temperature dependence of D_+ obtained in the present experiments was close to that reported by the previous experiments [6, 7]. Mäkinen *et al* [6] and Soininen *et al* [7] obtained the value of D_+ from the fitting of the S–E relationships for Au/Si and SiO_2/Si structures. Considering the difference in the experimental conditions, the difference between the results obtained by the present and the previous experiments [6, 7] is acceptable. Above 1073 K, the decrease of D_+ was found to be saturated. In figures 2 and 3, since no correlation between the values of D_+ and S_s or S_{ep} was observed, the change in the surface condition is unlikely to affect the value of D_+. The diffusion of positrons is known to be affected by the presence of an electric field in the subsurface region [19]. To discuss the effect of the band banding on the value of D_+, Soininen *et al* [7] numerically solved the Poisson equation, and concluded that the motion of positrons in undoped Si is field free. Thus, the same is true in the present experiment. Table 1 summarizes the values of L_+, D_+ and the mean free path of positrons l_+ at 278 K and 1618 K, respectively. The value of l_+ was calculated by the semi-classical random-walk theory: $D_+ = (1/3)v_{th}l_+$, where v_{th} is the thermal velocity of positrons. Uedono *et al* [20] reported the temperature dependence of D_+ for Ge, and the decrease in the value of D_+ was found to be saturated above 800 K. The ratio of the onset temperature of the saturation, T_{sat}, to T_m (T_{sat}/T_m), was calculated as 0.66, and this value is close to that for Si (0.64). Using the data reported in [20], table 1 summarizes the diffusion related parameters for Ge at 298 K and 978 K. For Si and Ge, the lower limit of D_+ is considered as 0.3–0.5 cm^2 s^{-1}, and the corresponding values of l_+ were an order of the interatomic distances. In metals with low T_m, such as Bi, the temperature dependence of D_+ in the liquid phase is well explained by assuming that l_+ is determined by the atomic disorder (an order of interatomic distance), and should thus be approximately temperature independent [3]. For Si and Ge, upon increasing temperature, the decrease of D_+ is thought to saturate when l_+ is close to the interatomic distance.

Table 1. The diffusion length of positrons L_+, the diffusion coefficient of positrons D_+ and the mean free path of positrons l_+. The results obtained for Ge [20] are also shown. The highest temperatures T_{high} in the thermal equilibrium measurements for Ge and Si are 978 K and 1618 K, respectively.

	Si			Ge		
Temperature (K)	L_+ (nm)	D_+ (cm^2 s^{-1})	l_+ (nm)	L_+ (nm)	D_+ (cm^2 s^{-1})	l_+ (nm)
298	200 ± 20	1.8	5.8	236 ± 2	2.4	10.1
T_{high}	104 ± 1	0.48	0.66	89 ± 1	0.34	0.79

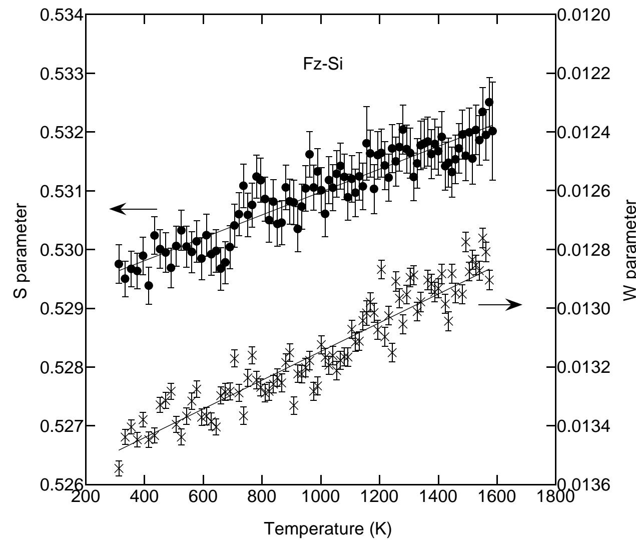

Figure 4. *S* parameter and the *W* parameter as a function of temperature; *E* was fixed as 25 keV.

Figure 4 shows *S* and *W* as a function of temperature for the Si specimen. In this experiment, *E* was fixed as 25 keV, so that the values of *S* and *W* shown in figure 4 are the characteristic values for the annihilation of positrons in the bulk. In figure 4, the observed weak temperature dependences of *S* and *W* agreed with the results reported in the previous studies [12, 21]. The temperature coefficients of *S* and *W* were found to be constant. Thus, it can be concluded that the effect of the trapping of positrons by thermal equilibrium defects on *S* or *W* is negligible.

In order to interpret the change in Doppler broadening spectra in more detail, we calculated the difference between the spectra measured in the low- and the high-temperature regions as follows. To increase statistical accuracy, we integrated the spectra measured at 313–490 K (average temperature = 402 K) into one spectrum and those measured at 1482–1585 K (average temperature = 1534 K) into another. The integrated Doppler broadening spectrum at 402 K is shown in figure 5; the horizontal axis shows ΔE_γ. In figure 5, it can be seen that the spectrum mainly consists of the narrow and the broad components. The broad component clearly observed at $\Delta E_\gamma > 3$ keV corresponds to the annihilation of positrons with core electrons, and the narrow component at $\Delta E_\gamma < 3$ keV corresponds to the annihilation of positrons with valence electrons [22]. The difference between the Doppler broadening spectra at 402 K and 1534 K is also shown in figure 5; the spectrum at 402 K was subtracted from that at 1534 K. For the subtracted spectrum, the negative values at 3 keV $< \Delta E_\gamma < 6$ keV are

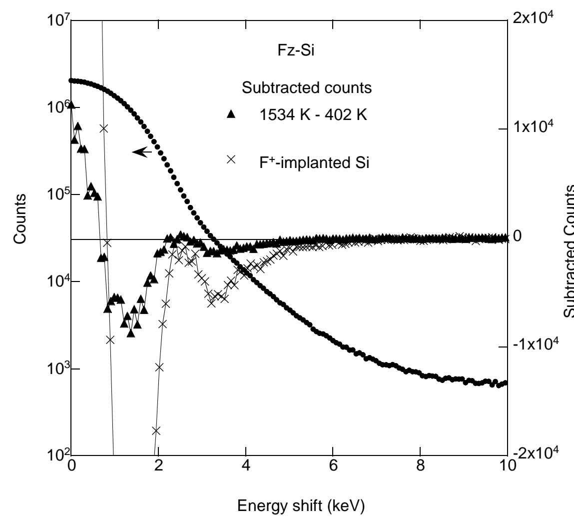

Figure 5. Doppler broadening spectrum measured at 402 K; E was fixed at 25 keV. The difference between the spectra measured at 402 K and 1534 K is also shown; the spectrum at 402 K was subtracted from that at 1534 K. For a comparison, the subtracted spectrum corresponding to the annihilation of positrons trapped by vacancy-type defects (mainly V_2) is also shown [23].

attributable to the decrease in the annihilation probability of positrons with core electrons at 1534 K, and the behaviour of the subtracted counts at $\Delta E_\gamma < 3$ keV is due to the narrowing of the spectrum corresponding to the annihilation of positrons with valence electrons.

By the same analysis method used in the present work, Uedono *et al* [23] characterized the Doppler broadening spectrum for F^+-implanted epitaxial Si. In this experiment, the value of S and the lifetime of positrons for the annihilation of positrons in damaged region were obtained as 1.034 S_b and 298 ps, respectively. These values agree with the characteristic value of S and the lifetime of positrons for divacancies V_2 [1, 24–26], respectively. Thus, the mean size of the open volume of defects in the damaged region was estimated to be close to the size of V_2. To study the effect of the trapping of positrons by vacancy-type defects (mainly V_2) on the Doppler broadening spectrum, we subtracted the spectrum for the annihilation of positrons from the delocalized state from that for the annihilation of positrons in the damaged region. The result is shown in figure 5; the upper and lower parts of the spectrum are not shown (the overall spectrum was shown in figure 5 in [23]). In the subtracted spectrum for the F^+-implanted specimen, the positive values at $\Delta E_\gamma < 1$ keV are attributed to the lowering of the crystal symmetry due to the trapping of positrons by V_2, and the negative values at 3 keV $< \Delta E_\gamma < 6$ keV are attributed to the decrease in the annihilation probability of positrons with core electrons due to such an annihilation mode [23].

From measurements of an x-ray diffractometer, Okada and Tokumaru [27] obtained the temperature dependence of the thermal expansion coefficient for Si in the temperature range between 300 K and 1500 K. Using their result, we calculated the ratio of the lattice parameter at 1534 K to that at 402 K as 1.005. In figure 5, for the subtracted spectra for the thermal equilibrium measurement and for the F^+-implanted Si specimen, the areas at 3 keV $< \Delta E_\gamma < 6$ keV were calculated, then their ratio was determined as 0.2. This means that, at 1534 K, the decrease in the annihilation probability of positrons with core electrons corresponds to 20% of the decrease that is due to the trapping of positrons by V_2. Considering the large difference between the wavefunction of positrons with the Bloch state and that strongly

localized in the vacancy-type defects [1], this value (20%) is thought to be large as only the effect of the lattice expansion on the annihilation probability of positrons with core electrons. For more detailed discussion, the theoretical calculation of the momentum distribution of positron–electron pairs in Si at high temperature is desirable. However, the results shown in figure 5 suggest that the measurements of Doppler broadening spectrum provide unique information about the interaction between positrons and phonons.

4. Conclusions

We have discussed annihilation characteristics of positrons in Si in thermal equilibrium using the monoenergetic positron beam. From measurements of Doppler broadening spectra of the annihilation radiation as a function of incident positron energy, the characteristic value of the S parameter for the annihilation of positrons at the surface S_s and the diffusion coefficient of positrons D_+ were determined in the temperature range between 298 K and 1618 K. At 298–1373 K, the value of S_s increased with increasing temperature. This is due to the thermal desorption of positrons from the surface state and the resultant increase in the formation probability of Ps. Above 1473 K, however, the value of S_s started to decrease. This fact was attributed to the suppression of the Ps formation due to the trapping of positrons by open spaces in the liquid layer at the surface.

Below 1000 K, the observed temperature dependence of D_+ was in good agreement with that reported by Mäkinen *et al* [6] and Soininen *et al* [7]. According to their discussion, the temperature dependence of D_+ is attributed to the scattering of positrons by acoustic and optical phonons. Above 1073 K, the decrease of D_+ is saturated. At 1618 K, the value of D_+ was determined as 0.48 $cm^2\ s^{-1}$, and the corresponding mean free path of positrons was calculated as 0.66 nm. The ratio of the onset temperature of the saturation, T_{sat}, to T_m was obtained as 0.64. In the measurements of D_+ for Ge [23], the saturation of the decrease in D_+ was also observed above 800 K ($=0.66T_m$), and the values of D_+ at 298 K and at the highest temperature in the measurement were close to those for Si. Thus, for Si, the scattering mechanism of positrons is considered to be the same as that for Ge, and the lower limit of D_+ for those semiconductors was determined as 0.3–0.5 $cm^2\ s^{-1}$.

Precise measurements of Doppler broadening spectra for the annihilation of positrons in the bulk were performed in the temperature range between 298 K and 1585 K. The temperature coefficients of S and W were found to be constant in the measured temperature range. This means that the effect of the annihilation of positrons trapped by thermal equilibrium defects on S and W is negligible.

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