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Defects in electron irradiated vitreous SiO_2 probed by positron annihilation

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Abstract. Defects in 3 MeV electron irradiated vitreous SiO_2 (v-SiO_2) were probed by the positron annihilation technique. For unirradiated v-SiO_2 specimens, almost all positrons were found to annihilate from positronium (Ps) states. The high formation probability of Ps was attributed to the trapping of positrons by open-space defects. The formation probability of Ps was decreased by the electron irradiation. The observed inhibition of the Ps formation was attributed to the trapping of positrons by point defects introduced and/or activated by the irradiation. From measurements of the lifetime distribution of Ps, it was found that, by the electron irradiation, the mean size of open-space defects was decreased and the size distribution of such defects was broadened.

1. Introduction

Developments of large-scale integrated devices require a precise control of defects in oxide films [1]. Characterization of SiO_2 films by a monoenergetic positron beam is of increasing interest because of the technological importance of this material [2–4]. The annihilation characteristics of positrons in SiO_2 films, however, are not yet so clear, because these studies were performed by measuring Doppler broadening profiles of the annihilation radiation. That is, the information derived from Doppler broadening profiles is rather incomplete, because of difficulties of the decomposition of various annihilation modes convoluted in Doppler broadening profiles. On the other hand, lifetime spectra of positrons can be in principle decomposed into several components corresponding to different annihilation modes. Recently, Uedono and co-workers [5, 6] reported measurements of lifetime spectra of positrons for SiO_2 films grown on Si substrates by using a pulsed monoenergetic positron beam. The time resolution of the lifetime spectra of positrons measured by the pulsed monoenergetic positron beam, however, was still lower than that measured by a conventional system using a white beam as born from the β^+ decay of nuclides designed for positron annihilation for bulk materials [7]. For this reason, the potential of the positron annihilation technique for studies in defects in amorphous SiO_2 has not been fully elucidated. In the present paper, therefore, we report the study of defects in electron irradiated vitreous SiO_2 (v-SiO_2) by conventional positron annihilation techniques.

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2. Experiment

Synthesized v-SiO_2 specimens and a fused v-SiO_2 specimen used in the present experiment were provided by Sumikin Quartz Products, Inc. The synthesized v-SiO_2 specimens were prepared by the vapour-phase axial deposition (VAD) technique. The concentrations of major impurities for these specimens are summarized in table 1. The specimens were irradiated with 3 MeV electrons up to a dose of 1×10^{18} e cm^{-2}. The temperature of the specimen was kept at $\sim$ 70 °C during the irradiation. Doppler broadening profiles of the annihilation radiation were measured by a Ge detector. Each Doppler broadening profile contains 3×10^7 counts. The positron lifetime spectra were measured by a fast–fast system with BaF_2 scintillators attached to XP2020Q photomultiplier tubes. The full width at half maximum (FWHM) of the time resolution of the system was $\sim$ 210 ps. Each lifetime spectrum contains 6×10^6 counts. All measurements were made at room temperature. The observed lifetime spectrum of positrons, $I(t)$, can be represented by

$$I(t) = \sum_{i=1}^{n} \alpha_i \lambda_i \exp(-\lambda_i t) \tag{1}$$

where α_i is the fraction of positrons annihilating with the ith lifetime, τ_i ($= 1/\lambda_i$), and n is the number of annihilation modes. $I(t)$ can be analysed by using computer codes (PATFIT and RESOLUTION) developed by Kirkegaard *et al* [8].

Table 1. The concentrations of impurities for the synthesized v-SiO_2 specimens and the fused v-SiO_2 specimen.

Growth condition	OH (ppm)	Al (pppm)	Fe (ppm)	Na (ppm)	Ca (ppm)
Synthesized (VAD)	<5	<0.04	not detected	not detected	<0.04
Synthesized (VAD)	<200	<0.04	<0.05	not detected	<0.04
Fused	~150	8.7	0.5	0.7	0.5

In (1), we usually have no prior knowledge of n. Since the number of components has ramifications for physical interpretations in the procedure for the fitting of the data to the finite-term model spectrum, one must consider the limits of statistical accuracy for which various types of analysis are possible. One of the ways to assess the reliability of the results obtained through the fitting procedure is to compare them with those obtained through the continuous lifetime analysis. Equation (1) can be expressed in a continuous decay form:

$$I(t) = \int_0^\infty \alpha(\lambda)\lambda \exp(-\lambda t)\,d\lambda. \tag{2}$$

In (2), $I(t)$ is a Laplace transformation of the decay probability density function (PDF), $\alpha(\lambda)\lambda$. The deconvolution by a Laplace inversion technique was developed by Gregory [9] into a computer code named CONTIN (PALS-2). In CONTIN (PALS-2), the PDF is obtained by using a reference spectrum of the specimen with the known positron lifetime. In the present experiment, the lifetime spectrum of a well annealed Ni spectrum ($\tau = 110$ ps) was used for the reference spectrum.

3. Results

Figure 1 shows the lineshape parameter S [10] for the synthesized and fused v-SiO_2 specimens before and after the electron irradiation. In figure 1, it was found that the value of the S parameter was decreased by the electron irradiation. The largest decrease in the value of S by the irradiation was observed for the synthesized v-SiO_2 specimen with an OH concentration of < 5 ppm.

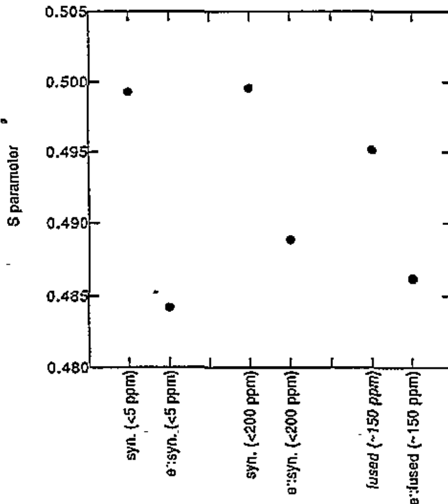

Figure 1. The Doppler-broadened lineshape parameter S for the synthesized v-SiO_2 specimens and the fused one before and after the electron irradiation.

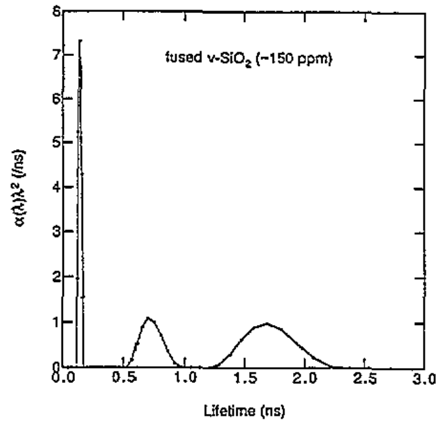

Figure 2. The positron lifetime distribution function for the fused v-SiO_2 specimen obtained by CONTIN (PALS-2).

Figure 2 shows the positron lifetime distribution function obtained by CONTIN (PALS-2) for the fused v-SiO_2 specimen before the irradiation. In figure 2, the number of peaks in the lifetime distribution was found to be three. For the other specimens, the number of peaks was also found to be three. This fact suggests that positrons in the v-SiO_2 specimens annihilated from three different states. Thus, the lifetime spectra were decomposed into three components by RESOLUTION. The results are summarized in table 2. As shown in figure 2 and table 2, the results obtained by RESOLUTION were in agreement with those obtained by CONTIN (PALS-2). In table 2, the values of τ_3 (1.70–1.76 ns) were found to be longer than the typical lifetime of positrons in crystalline solids [11]. Thus, this annihilation mode can be attributed to the pick-off annihilation of *ortho*-positronium (*o*-Ps) [12]. The presence of *o*-Ps should be associated with that of *para*-positronium (*p*-Ps), too [12]. In table 2, for the unirradiated specimens, the obtained values of τ_1 (0.127–0.131 ns) were in good agreement with the intrinsic lifetime of *p*-Ps (0.125 ns). For the irradiated specimens, however, the values of τ_1 (0.140–0.148 ns) were obviously longer than 0.125 ns. This means that the first component for the irradiated specimens is the superposition of the annihilation of *p*-Ps and that of positrons. Thus, the lifetime spectra for the irradiated specimens were decomposed into four components. Since *p*-Ps could annihilate from the pick-off process, the lifetime of *p*-Ps might be shorter than 0.125 ns. However, such a process is unlikely to drastically decrease the lifetime of *p*-Ps because of the very short intrinsic lifetime of *p*-Ps. For the unirradiated specimens, for example, the values of τ_1 were in agreement with 0.125 ns.

Thus, the effect of the pick-off annihilation of p-Ps is considered to be negligible. In the present experiment, the value of τ_1 was fixed at 0.125 ns in the four-component analysis. The mean lifetimes of positrons, τ_m, are shown in figure 3. τ_m was calculated by the following equation:

$$\tau_m = \sum_{i=1}^{n} I_i \tau_i \tag{3}$$

where $I_i = \alpha_i \lambda_i$. In figure 3, the value of τ_m was found to decrease on electron irradiation. The largest decrease in the value of τ_m was observed for the synthesized specimen with an OH concentration of < 5 ppm. The lifetimes and the intensities are shown in figures 4 and 5, respectively. In the text, hereafter, the notations used in the four-component analysis will be used for the irradiated specimens. For the irradiated specimens, the number of annihilation modes obtained by CONTIN (PALS-2) was three. For these specimens, since the value of the intrinsic lifetime of p-Ps was close to the value of τ_2 (0.23–0.3 ns), the separation of these two annihilation modes by the numerical Laplace inversion technique is considered to need higher statistics [13]. For the unirradiated specimens, the lifetime spectra were not able to decompose into four components. Thus, the intensity corresponding to the annihilation of positrons with the lifetime of 0.23–0.3 ns is considered to be close to zero.

Table 2. The lifetimes and the intensities obtained by the three-component analysis for the synthesized v-SiO_2 specimens and the fused v-SiO_2 specimen.

Condition	Specimen	τ_1 (ns)	τ_2 (ns)	τ_3 (ns)	I_1 (%)	I_2 (%)	I_3 (%)
Unirradiated	synthesized (< 5 ppm)	0.127(2)	0.80(1)	1.758(6)	26.3(1)	21.4(5)	52.2(5)
	synthesized (< 200 ppm)	0.131(2)	0.85(2)	1.762(7)	26.7(1)	21.6(6)	51.7(6)
	fused (~ 150 ppm)	0.128(3)	0.71(1)	1.731(5)	24.8(1)	24.5(3)	50.7(4)
Irradiated	synthesized (< 5 ppm)	0.148(4)	0.541(6)	1.698(4)	21.5(3)	38.1(2)	40.4(2)
	synthesized (< 200 ppm)	0.140(3)	0.587(7)	1.721(4)	30.0(2)	31.0(2)	46.0(3)
	fused (~ 150 ppm)	0.146(3)	0.573(7)	1.701(4)	23.4(3)	32.7(2)	43.9(3)

Figures 6–8 show the positron lifetime distribution in the region between 1.2 ns and 2.4 ns for the unirradiated v-SiO_2 specimens and the irradiated ones. For the synthesized specimens, a broadening of the lifetime distribution due to the electron irradiation was observed. For the fused specimen, this was not the case.

4. Distribution

4.1. Positron annihilation in v-SiO_2

For the unirradiated specimens, the longest lifetime and the shortest lifetime were reasonably attributed to the pick-off annihilation of o-Ps and the self-annihilation of p-Ps, respectively. The value of τ_2 was also longer than the typical lifetime of positrons in crystalline solids [11]. The ratio of $I_2 + I_3$ to I_1 was calculated as 2.7–3.0 from table 2. Since these values agree with the ratio of the formation probability of o-Ps to that of p-Ps (3) [12], the second component also can be mainly associated with one of the annihilation modes of o-Ps.

Dannefaer *et al* [14] reported that the lifetime spectrum for single-crystal quartz (α-SiO_2) can be decomposed into three components, where the longest lifetime was 1.4 ns with an intensity of 1.5%. This component can be attributed to the pick-off annihilation of o-Ps.

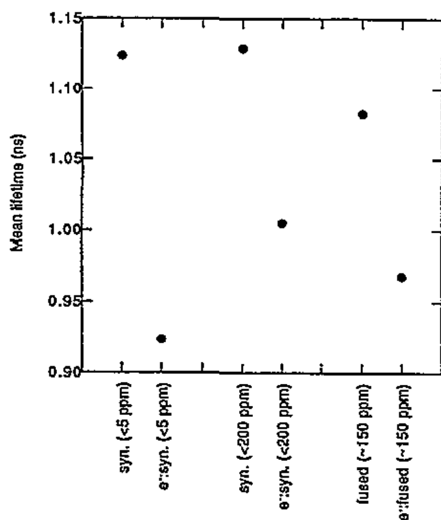

Figure 3. The mean lifetimes of positrons, τ_m, for the synthesized v-SiO_2 specimens and the fused one before and after the electron irradiation.

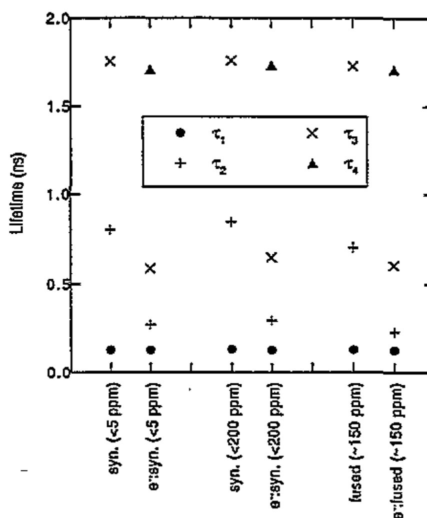

Figure 4. The lifetimes for the v-SiO_2 specimens before and after the electron irradiation. The lifetime spectra of positrons for the unirradiated specimens were decomposed into three components, and those for the irradiated ones were decomposed into four components.

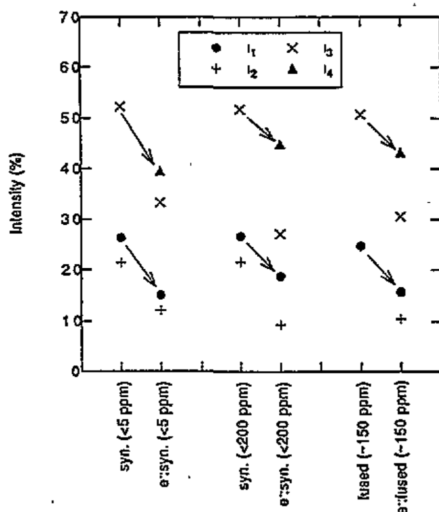

Figure 5. *The intensities for the v-*SiO_2 *specimens* before and after the electron irradiation.

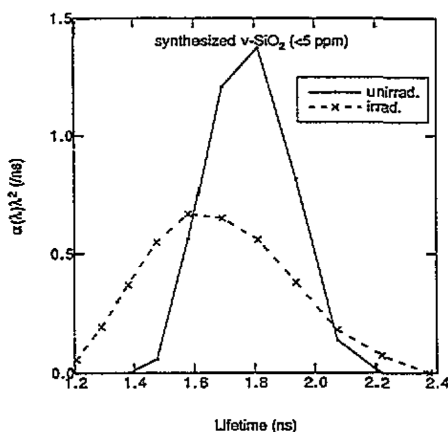

Figure 6. *The lifetime distribution corresponding to the* pick-off annihilation of *o*-Ps for the synthesized v-SiO_2 specimen with an OH concentration of < 5 ppm.

For the α-SiO_2 specimen, the intensity corresponding to the annihilation of Ps is far less than that for the v-SiO_2 specimens. In order to find out the annihilation characteristics of Ps in α-SiO_2 and v-SiO_2, Uedono *et al* [15] measured the two-dimensional angular correlation of positron annihilation radiations (2D ACAR) for both the α-SiO_2 and v-SiO_2 specimens. The former specimen is the same one as measured by Dannefaer *et al* [14]. For the α-SiO_2

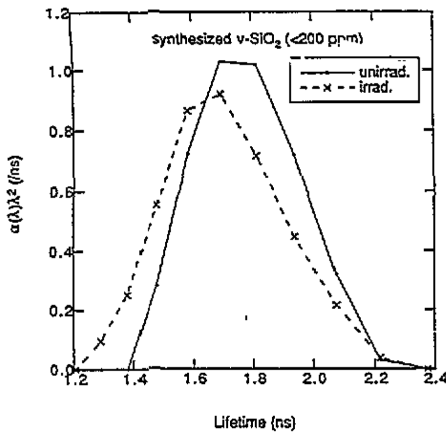

Figure 7. The lifetime distribution corresponding to the pick-off annihilation of o-Ps for the synthesized v-SiO_2 specimen with an OH concentration of < 200 ppm.

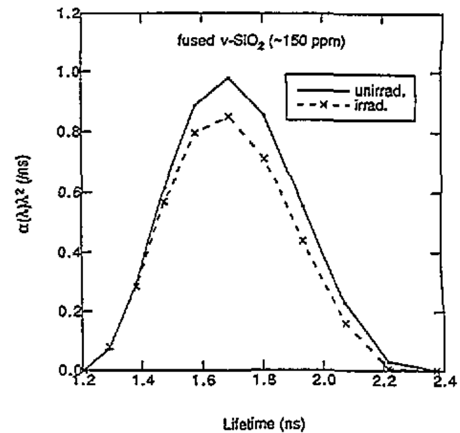

Figure 8. The lifetime distribution corresponding to the pick-off annihilation of o-Ps for the fused v-SiO_2 specimen with an OH concentration of ~ 150 ppm.

specimen, the self-annihilation of p-Ps from the delocalized state was observed. Thus, the longest component for the α-SiO_2 specimen is considered to be due to the annihilation of o-Ps from the delocalized state. For the v-SiO_2 specimens, however, this was not the case. Uedono and Tanigawa [16] also reported measurements of 2D-ACAR spectra for v-SiO_2 specimens with porous structures. From the measurements, it was found that Ps formed in the v-SiO_2 specimens annihilates with a broadened momentum distribution and that the momentum distribution of Ps was sensitive to the size of pores. These facts were attributed to the momentum uncertainty due to the localization of Ps in a finite dimension of open-space defects. Thus, it can be concluded that almost all Ps formed in the v-SiO_2 specimens is trapped by open-space defects, and its formation is enhanced by the trapping of positrons by open-space defects. In the present experiment, therefore, for the unirradiated specimens, the second and the third components can be attributed to the lifetimes of o-Ps trapped by open-space defects. The difference between the value of τ_2 and that of τ_3 might be due to a difference in the size of such defects.

4.2. The inhibition of Ps formation in the electron irradiated specimens

In figure 4, the value of the longest lifetime for the unirradiated specimens, τ_3, is seen to be close to that for the irradiated ones, τ_4. This fact suggests that the origin of the third component for the unirradiated specimens is the same as that of the fourth component for the irradiated ones. In figure 5, it can be seen that the values of I_4 for the irradiated specimens were smaller than those of I_3 for the unirradiated ones. The value of the intensity corresponding to the self-annihilation of p-Ps, I_1, was also decreased by the irradiation. The change in the formation probability of Ps should be observed in both the annihilation probability of p-Ps and that of o-Ps. The average ratio of the decrease in the shortest component to that in the longest component was calculated as 0.9, which is close to unity. Thus, it can be concluded that the formation probability of Ps in the v-SiO_2 specimens was decreased by the electron irradiation.

In figure 1, the value of S is seen to be decreased by the irradiation. Since the annihilation from the p-Ps state produces γ rays with very sharp energy width, the decrease in the formation probability of Ps decreases the value of S. Thus, the observed decrease in

the value of S on electron irradiation was also attributed to the inhibition of Ps formation. Uedono *et al* [17] reported that the value of S corresponding to the annihilation of positrons in SiO_2 films was decreased by γ-ray irradiation. Recently, a similar behaviour of S for x-ray- or γ-ray-irradiated SiO_2 films was reported by Khatri *et al* [18]. Fujinami and Chilton [4] and Uedono *et al* [19] reported that the value of S drastically decreased on ion implantation into SiO_2 films. From the present experiment, it is clear that the annihilation characteristics of positrons in SiO_2 films were dominated by the behaviours of Ps because almost all positrons annihilate from the Ps states in amorphous SiO_2. The decrease in the value of S for damaged SiO_2 films reported by the previous papers, therefore, can be attributed to inhibition of Ps formation.

After the irradiation, for the synthesized v-SiO_2 specimens with OH concentrations of < 5 ppm and < 200 ppm, the values of τ_2 were obtained as 0.27 ± 0.03 ns and 0.30 ± 0.04 ns, respectively. For the unirradiated specimens, the lifetime close to ~ 0.3 ns was not observed. Thus, the second component for the irradiated specimens can be attributed to the annihilation of positrons trapped by defects introduced by the electron irradiation. Since the formation of Ps needs larger open spaces or a lower electron density than the volume of or electron density around point defects, the trapping of positrons by such defects is considered to suppress the formation of Ps. Thus, the observed inhibition of the Ps formation is considered to be due to the trapping of positrons by point defects introduced by the electron irradiation.

After the irradiation, the formation probability of Ps for the synthesized v-SiO_2 specimen with an OH concentration of < 5 ppm was lower than that for one with an OH concentration of < 200 ppm (figure 5). For low-purity v-SiO_2 specimens, a similar relationship between the formation probability of Ps and the concentration of hydroxyl groups has already been found by Uedono *et al* [20]. Galeener and co-workers [21, 22] reported that the concentration of E′ centres for x-ray-irradiated v-SiO_2 specimens depends on the concentration of OH groups. The E′ centre is known to be a point defect observed by measurements of electron spin resonance (ESR), and it was assigned to an electron trapped by a tribonding Si atom, which is similar to the structure of an asymmetrically related O vacancy [23]. They reported that the E′ signal increased with decreasing concentration of OH groups at low irradiation dosage. This fact was attributed to the activation of pre-existing defects from x-ray irradiation, where this activation involves the transfer of an electron or hole to or from previously existing broken bonds. These pre-existing broken bonds are considered to be suppressed by OH groups. They also suggested that this activation mechanism can be applied to the formation of other point defects such as non-bridging O hole centres (NBOHCs) or preoxyl radical O hole centres (PROHCs). Because of the small open spaces of NBOHCs or PROHCs, however, these defects are unlikely to be attributed to the annihilation of positrons with τ_2 (~ 0.3 ns). The charge states of the O vacancy, V_O, were calculated by Rudra and Fowler [24] using molecular-orbital techniques. They showed that V_O introduces two levels (0/+ and +/2+), and possibly the third one (−/0) into the band gap of SiO_2. They also suggested that the neutral V_O is a hole trap. Hole traps can reasonably be expected to also trap positrons, because of the same sign of electric charge for both a hole and a positron. Thus, the origin of the inhibition of the Ps formation by the electron irradiation might be associated with the introduction of V_O. Dannefaer *et al* [14] suggested that positrons would not be trapped by V_O because of the small open spaces of V_O. They suggested that the most probable candidates for point defects that can trap positrons are Si vacancies, V_{Si}, and O divacancies V_OV_O. Thus, it can be concluded that V_{Si}, V_OV_O and relaxed V_O are candidates for the point defects introduced and/or activated by the electron irradiation.

Because of the negative charges of NBOHCs or PROHCs, these defects might react with Ps. A rapid reaction between these defects and *o*-Ps could decrease the value of I_4. However,

because of the short intrinsic lifetime of p-Ps (0.125 ps), such a reaction is unlikely to occur for p-Ps. Since the decrease in the value of I_1 and that of S were observed for the irradiated specimens, the reaction between Ps and NBOHCs or PROHCs is unlikely to be possible for the above-mentioned reason. In addition, such a reaction could cause spin exchange from o-Ps to p-Ps. However, from measurements of age–momentum-correlation spectra, Uedono *et al* [20] concluded that the decrease in the value of I_4 was not due to the spin exchange from o-Ps to p-Ps. Dannefaer *et al* [14] found that the lifetime was close to 0.3 ns for an amorphous SiO_2 specimen. They suggested that this component might arise from shallow positron traps in amorphous SiO_2. The details of the annihilation characteristics involving shallow traps in v-SiO_2 are not clear in this stage.

For the irradiated specimens, the values of τ_3 were close to the lifetime of positrons trapped by vacancy clusters in crystalline solids [11]. For these specimens, the values of $(I_3 + I_4)/I_1$ were calculated as 3.8–4.8. This means that the third component for the irradiated specimens is the superposition of the annihilation of o-Ps and that of positrons. The lifetime of such positrons is considered to range between the value of τ_2 (~ 0.3 ns) and that of τ_3 (~ 0.6 ns). In figure 5, it can be seen that the value of I_3 for the irradiated specimens was larger than that of I_2 for the unirradiated ones. Thus, the inhibition of Ps is attributed not only to the trapping of positrons by monovacancy- or divacancy-type defects but also to that by vacancy clusters.

4.3. The lifetime distribution of o-Ps obtained by a Laplace inversion technique

For the synthesized specimens (figures 6 and 7), it was found that the mean lifetime of o-Ps was decreased and the lifetime distribution was broadened by the electron irradiation. The effect of the electron irradiation on the lifetime distribution for the specimen with an OH concentration of < 5 ppm was larger than that for the specimen with an OH concentration of < 200 ppm. This result is in good agreement with that shown in figures 1, 3 and 5. Since the lifetime of o-Ps decreases with decreasing size of open-space defects [25], the observed decrease in the value of the mean lifetime of o-Ps and the broadening of the lifetime distribution can be attributed to the decrease in the size of open-space defects and the broadening in the size distribution of such defects, respectively. The open-space defects in the v-SiO_2 specimens might be a sink for atoms injected into the bulk matrix by the electron irradiation. In addition, the activation of defects near the open-space defects might increase the pick-off annihilation rate.

The effects of impurities on the annihilation characteristics of positrons were observed. After the irradiation, the value of τ_2 (0.23 ns) for the fused specimen was smaller than that for the synthesized specimens (0.28 ns). In figure 8, no drastic change in the lifetime distribution due to the electron irradiation was observed for the fused specimen. Since amorphous structures of v-SiO_2 depend on several factors, such as the thermal history of specimens and impurities, the origin of the different annihilation characteristics of positrons between the synthesized specimens and the fused one is not clear at this stage. The formation probability of Ps for the fused specimen after the irradiation is close to that for the synthesized specimen with an OH concentration of < 200 ppm. Thus, it is considered that the inhibition of the Ps formation was mainly dominated by the concentration of OH groups in v-SiO_2.

5. Conclusions

We have presented the study of defects in 3 MeV electron irradiated v-SiO_2 specimens by measurements of lifetime spectra of positrons and those of Doppler broadening profiles of

the annihilation radiation. The positron annihilation technique was shown to be sufficiently sensitive for the detection of defects in v-SiO_2. For unirradiated v-SiO_2 specimens, almost all positrons were found to annihilate from the Ps states. The high formation probability of Ps was due to the trapping of positrons by open-space defects. For the electron irradiated specimens, the formation probability of Ps was decreased. These facts were attributed to the trapping of positrons by vacancy-type defects introduced and/or activated by the electron irradiation. A decrease in the size of open-space defects and a broadening in the size distribution of such defects were observed after electron irradiation. The present investigation shows that positrons provide a sensitive and non-destructive probe for the detection of defects in amorphous SiO_2.

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