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Positron annihilation measurements in as-grown and alpha irradiated undoped Indium Phosphide

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Abstract*— The effects of 40 MeV alpha irradiation in undoped InP crystal and post irradiation isochronal annealing from 25 to 650^{O}C were investigated using positron annihilation spectroscopy. After each annealing the positron measurements were carried out at room temperature. Formation of radiation induced defects and their recovery with annealing temperature were investigated. The lifetime spectra of the irradiated sample were fitted with three lifetimes. Trapping model analysis was carried out with the de-convoluted lifetimes to characterize the defect states. The average positron lifetime τ_{avg}=261ps at room temperature after irradiation indicated the presence of defects and the high value of τ_2 (342ps) at room temperature suggested that the probable defects were divacancies. Two distinct annealing stages in τ_{avg} at 375 to 550^{O}C and 550 to 650^{O}C were observed. The variations in line-shape parameter (S) and defect specific parameter (R) during annealing in the temperature region 25-650^{O}C resembled the behaviour of τ_{avg} indicating the migration of vacancies, formation of vacancy clusters and the disappearance of defects between 375 to 650^{O}C.

***Index Terms*— High energy alpha irradiation, Defects, Isochronal annealing, Positron Annihilation Measurements.**

I. INTRODUCTION

The performance of semiconductor devices is greatly influenced by the presence of defects because the defects interact with the free carriers and act as scattering as well as recombination centers for carriers or as carrier traps. The effect, even if the defect concentration is small, is not negligible and can change the electrical and optical properties of the material significantly. Therefore it is very important to study the physical behaviour of these defects. Thus, the study of defects, their nature, origin, concentration in semiconductors especially in compound semiconductors have become the field of growing interest since last few years.

Defects not only exist in as-grown bulk materials, but also can be produced through irradiation with various particles such as □-particles [1], electrons [2], protons [3]-[5], neutrons [4], heavy ions [6] etc. Radiation damage in semiconductors have been studied by several investigators with different experimental techniques such as electron paramagnetic resonance (EPR) [7], Fourier transformed infrared spectroscopy (FTIR) [8], deep level transient spectroscopy (DLTS) [9], infrared Spectroscopy [10], photoluminescence (PL) [11], positron annihilation spectroscopy (PAS) [12] etc. Among these techniques, PAS can provide quite novel and direct information about microscopic identification and quantification of vacancy like defects such as the nature, size, concentration and charge state of the defects [12]-[13]. However, the positron lifetime measurements give no information on the chemical surroundings of the annihilation event and can't identify the sub-lattice of the vacancy or whether the vacancy is isolated or complexes with impurity atoms. The lifetime measurements along with Doppler broadening of the annihilation radiation can provide additional information on the nearest-neighbor atoms of the defect. Thus the combinations of these two measurements are very useful for the identification of point defects and defect complexes, especially in compound semiconductors.

Having a large band gap of 1.4eV and high electron mobility compared to Si and GaAs, Indium Phosphide has been considered as higher promising compound semiconductors for the manufacturing of different optoelectronic devices such as high frequency circuits, laser diodes, solar cells, high speed transistors, microwave oscillators, quantum well diodes, IR photo detectors etc. [14]-[18].Very few studies on the induced defect formation by ion implantation and its recovery in InP have been performed previously using different ions, energies and implantation parameters [19]-[24] still the detailed information and an indepth understanding of the defect states are far from complete today.

It was shown by Dlubek et al. [9], [25], almost for all InP samples except heavily Zn-doped (4.5x10^{18}/cm^3) sample lifetime varied between 242ps and 252ps. But for heavily Zn-doped InP, a longer lifetime of 325ps was observed. This longer lifetime was due to the neutral Zn-divacancy complexes.

In the present paper, investigations on defects simulated by 40MeV alpha beam in undoped InP were carried out using positron measurements in order to identify the vacancy type defects and their recovery behaviour with annealing temperature.

II. EXPERIMENTAL DETAILS

A. Irradiation

The undoped InP sample was used for the study. A circular piece of 10mm diameter and of 0.6mm thickness was subjected to irradiation by 40MeV alpha beam to a total dose of 10^{17}alpha/cm^2 for 17 hours with an average beam current 500nA at room temperature.

B. Positron Annihilation Lifetime Spectroscopy

A ^{22}Na positron source of strength ~ 7μCi was used for the measurements. The source was deposited on a Ni foil of 5μm thickness. It was sandwiched between two identical cut pieces of sample under study. Positron lifetime measured using FAST-FAST coincidence technique having a time resolution of 290ps with ^{60}Co prompt source at positron window settings. Analysis of each positron lifetime spectrum, after proper source and background corrections, was made using standard PATFIT program [26] in accordance with the positron trapping model. Post irradiation isochronal annealing was performed at different temperatures starting from room temperature (25^{O}C) to 650^{O}C in a vacuum of 10^{-6}Torr. The annealing time was of 30minutes duration at each set temperature. The sample was cooled down to room temperature in vacuum and all the measurements were carried out at room temperature.

C. Doppler Broadening Annihilation Measurements

Doppler broadening annihilation line-shape measurements were carried out using HPGe detector having energy resolution of 1.4keV for 662keV γ-line with ^{137}Cs source. All measurements were carried out at room temperature. The annihilation line-shape parameter, S had its usual definition as the ratio of the central area (background subtracted) to the normalized total area (background subtracted) under the curve. Similarly the W-parameter was calculated as the ratio of the area of the high momentum part (wings) of the spectrum for a fixed energy interval to the normalized total area under the curve.

III. RESULTS AND DISCUSSIONS

A. Simulation by Monte Carlo Method

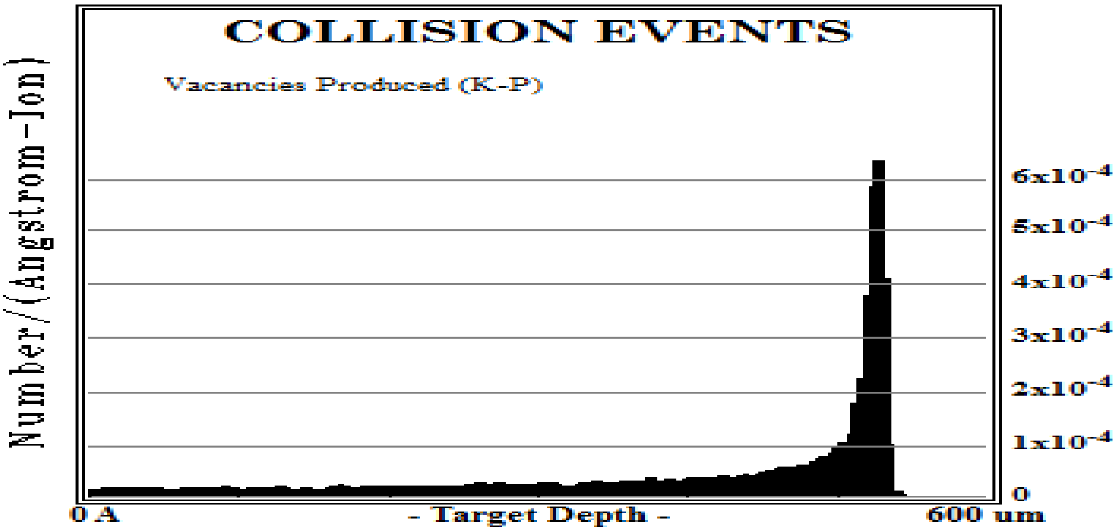

Fig 1: Variation of the concentration of vacancies produced by alpha-irradiation versus sample depth.

Monte Carlo method based SRIM-2008 program [27] was used to simulate 40MeV alpha irradiation induced defects in InP. The concentration of vacancies produced by irradiation versus sample depth is shown in figure 1. It was observed that the vacancies had a depth distribution along the alpha implantation path. The induced vacancy concentration around the implanted path mainly lay up to $6\text{x}10^{-4}$Number/Angstrom-Ion. The largest concentration of defects was around the end of the range of alpha, where the depth was about 523μm. The positron implantation depth R was calculated from the relation: $R^{-1} = 17\rho/E^{1.43}$ (cm^{-1}) [28], where ρ was the InP sample density and E was the largest positron energy. R = 51.34μm was obtained by considering $\rho = 4.81\text{g/cm}^3$ and E = 0.545MeV. It indicated that the vacancies induced by alpha irradiation were under the detection area of positron.

B. As-grown sample

PATFIT analysis of the positron lifetime spectrum for reference undoped InP sample, after proper background subtraction and suitable source correction yielded the positron lifetime value as τ_b=242ps at room temperature for a single component fit with a good variance. The value agreed well with the bulk value as obtained in several previous observations [9, 25, 29-30].

C. Post-irradiation

During irradiation depending on particle type and energy, formation of defects and their interactions were possible. 40MeV alpha beam introduced both P and In related defects because the threshold displacement energy for P and In are ~ 8eV and ~ 4eV respectively [31]. The probable defects were therefore vacancy at In-site (V_{In}), vacancy at P-site (V_P), antisites (P_{In}, In_P) and higher order defects. After irradiation, as the temperature increased the defects became mobile and they either disappeared by recombination i.e. forward annealing or migrated to reorder themselves to form larger size vacancies i.e. reverse annealing. For irradiated sample 3-component lifetime decomposition carried out by PATFIT program throughout all annealed sample spectra with a good variance. The shortest lifetime τ_1 was assigned to de-localized positrons annihilated in the bulk of the sample, with I_1 being the corresponding intensity. The intermediate lifetime τ_2 with intensity I_2 was attributed to positrons annihilated at the defect sites. The presence of a long lifetime component, τ_3 varied from 1-2.5ns having very low intensity I_3 was observed which might be due to the formation of positronium at the surface. Similar observations were presented in the previous studies by different groups [32]-[35]. Dannefaer et al. observed the similar defects of very low intensity while studying different semiconductors [32]. Mari et al. explained the presence of long component as due to positron annihilations in air [33]. Chen et al. [34] and Chaudhuri et al. [35] in their studies discussed about the formation of positronium which needs a large open volume. Thus the presence of long lifetime component can be related to either the formation of an amorphous layer at the sample surface or voids within the sample. The previously mentioned SRIM program showed that the largest vacancy concentration was mainly around the end of the alpha ion range (about 523μm). It suggests that, the defects in the range will overlap each other and an amorphous layer will easily be formed. Also long lifetimes were observed only for the irradiated sample not for the reference sample in our measurements. Thus the presence of the long lifetime component might be due to the formation of positronium at amorphous layer. However, the possibility that the long lifetime component, τ_3 of very low intensity was due to the surface effect of the irradiated sample could not be ruled out. So, further investigation is still needed to study the origin of the long lifetime component.

C.1. Trapping model analysis

Two-state trapping model analysis [36]-[37] was performed with the deconvoluted lifetimes to study indepth about positron trapping sites. These two states corresponded to a bulk state and a defect related state. According to this model

$$I_1+I_2=1 \tag{1}$$

$$K=I_2/I_1[\tau_b\tau_d/(\tau_d-\tau_b)] \tag{2}$$

$$\tau_1^{TM}= \tau_b/(1+K\tau_b) \tag{3}$$

Here, $\tau_1=\tau_b$=242ps i.e. the bulk lifetime of Indium Phosphide & $\tau_2=\tau_d$ i.e. the defect lifetime of positron. Here K is the positron trapping rate from bulk to the defect sites. Also I_1 and I_2 are the corresponding intensities of τ_b and τ_d respectively.

Figure 2 shows a comparison between τ_1^{TM} and the experimental lifetime τ_1 for the irradiated sample. The short-lived component τ_1 was found to be much less than τ_b. The behaviour could be understood with the help of the two-state trapping model. This model said that the shortest-lived component τ_1 was obtained from the annihilation at the bulk of the material, i.e. the annihilation from the perfect lattice, but this value was modified by the rate at which positrons were trapped by defects. Therefore, the observed annihilation rate λ_1 ($=1/\tau_1$) was given by $\lambda_1=\lambda_b+K$, λ_b being the bulk annihilation rate. Hence, the behaviour of τ_1 could be explained with this relation. It was evidenced from the figure that the values of τ_1^{TM} at all the temperatures were much higher than the corresponding experimentally observed τ_1 values, indicating that a two-state trapping model was not adequate in the present case. Similar type of results had been observed by Chaudhuri et al. [35].

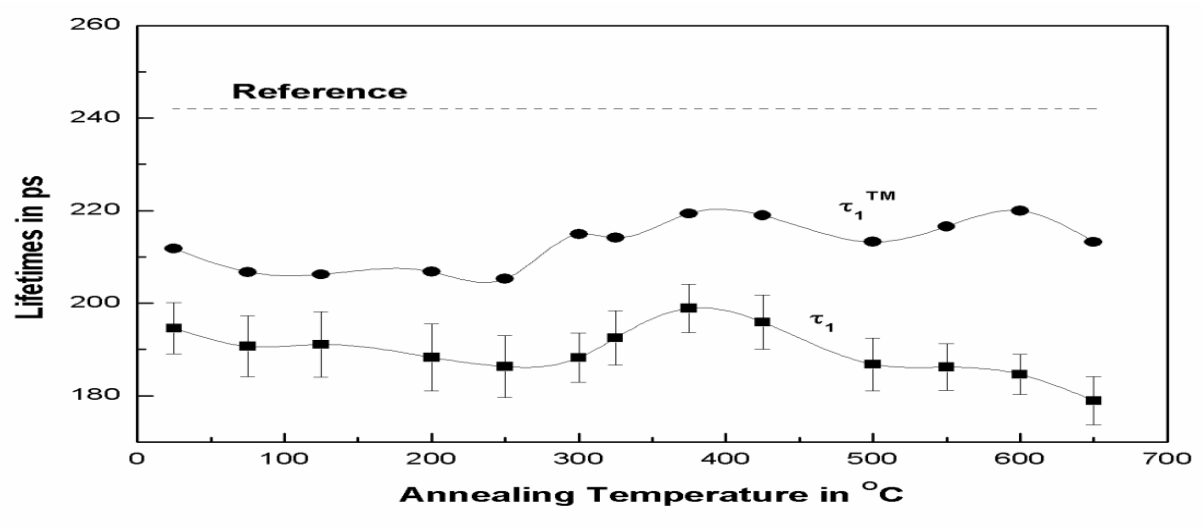

Fig: 2 Comparison of experimental value of τ_1 and calculated value of τ_1 from two-state trapping model (τ_1^{TM}).

After irradiation, at room temperature the defect related lifetime component (τ_2) had been found to be 342ps with intensity 42%, which was a typical divacancy defect lifetime with a ratio of τ_2/τ_b ~ 1.41 in InP by taking the bulk lifetime as 242ps. So due to high energy alpha irradiation with a high dose of 10^{17}alpha/cm^2, divacancy type defects were formed.

C.2. Isochronal annealing studies

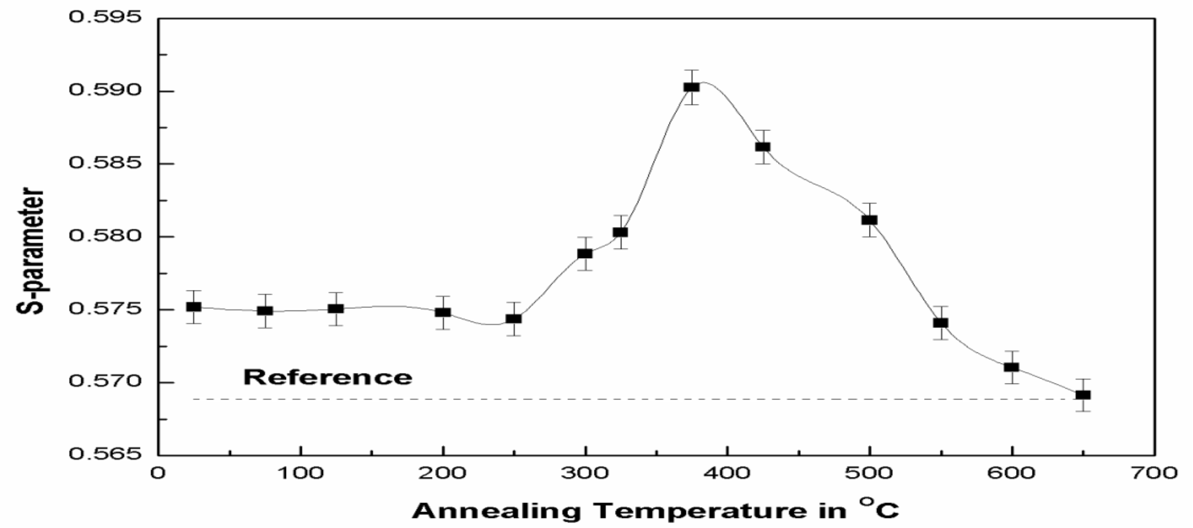

Fig: 3 Variation of S-parameter with annealing temperature in alpha irradiated undoped InP. The dotted line is for unirradiated reference InP sample.

The variations in the Doppler broadening line-shape parameter, 'S' with annealing temperature is shown in figure 3 for alpha irradiated InP. S-parameter detects the change in nature of trapping sites. Figure 4 shows a plot of the defect specific parameter, 'R' versus the annealing temperature. The R-parameter was defined as R= $(S_d - S_b)/(W_d - W_b)$, where W represented the core annihilation (wings). The suffix'd' represented the irradiated sample and 'b' represented the pre-irradiated sample i.e. the bulk sample. R-parameter is independent of the concentration of defects and only depends on the size of the trapping sites. It increases with the size of the trapping site which is predominant among several other trapping centers present at any time. The value of R-parameter is a minimum for mono-vacancies / vacancy loops.

The variation of S-parameter with annealing temperature showed the recovery of radiation induced defects over a broad range of temperature from 25-650^{O}C. The dotted line in figure 3 represented the S value for the reference InP sample. Irradiation in semiconductors is known to cause atomic displacements, resulting into the formation of vacancy-interstitial pairs. High energy alpha irradiation in high band-gap InP introduced defects which were more complex than those induced by other lighter particle irradiations such as electrons. This was due to the fact that the ion implantation caused cascade damage to produce even higher order defects. This was directly evidenced from

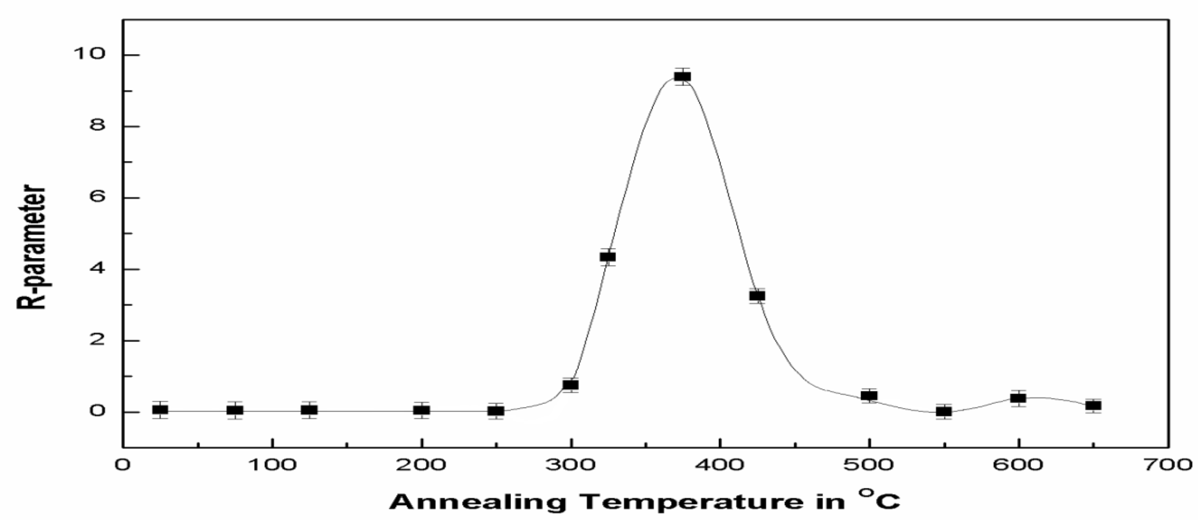

Fig: 4 Variation of R-parameter with annealing temperature in alpha irradiated undoped InP.

The increase in S-parameter value of irradiated sample compared to un-irradiated bulk sample as shown in figure 3.The probable defects could be vacancies at In-site (V_{In}), vacancies at P-site (V_{P}), antisites and other higher order defects. In figure 3, S value was almost stable up to 250^{O}C. In this temperature region i.e. from 25 to 250^{O}C R-parameter was also stable as in figure 4 and indicated that the predominant defect size was unchanged. A rise in S-parameter had been observed in the temperature range 250 to 375^{O}C. Alam et al. [38] discussed that a rise in S value was usually expected due to the migration of vacancies and clustering of those vacancies. However, Karjabinen et al. [39] had explained for such rise as due to the disappearance of some traps giving low shape broadening and retaining a large supply of defects with high broadening effects, presumably dislocations. During this temperature range i.e. 250 to 375^{O}C, R-parameter increased significantly and reached to a maximum at 375^{O}C. So the possibility of dislocations could be ruled out because R-parameter for dislocations is known to be a minimum, even less than that for mono-vacancies [40]. Keeping these arguments in mind, conclusion can be drawn that this reverse annealing stage in S-parameter might be due to the migration of vacancies and formation of vacancy clusters. It showed that the positron trapping rate for such clusters was sufficiently large so that this reverse annealing took place. The increase in R-parameter indicated the formation of higher order defects like divacancies or vacancy clusters. Beyond 375^{O}C, S-parameter started decreasing and at around 650^{O}C it acquired a value close to the un-irradiated bulk value. This decrease indicated the breaking up of vacancy clusters into vacancies in the process of annealing out of defects. In this temperature region R-parameter also decreased and it agrees well with the behaviour of S-parameter. The constant values of R in the temperature range 500-650^{O}C indicated that the defects had been annealed out.

The positron average lifetime had the high statistical accuracy and was independent of decomposition of the spectra; it was utilized to investigate the annealing behaviour of alpha-irradiation induced defects with temperature range between 25 to 650^{O}C.

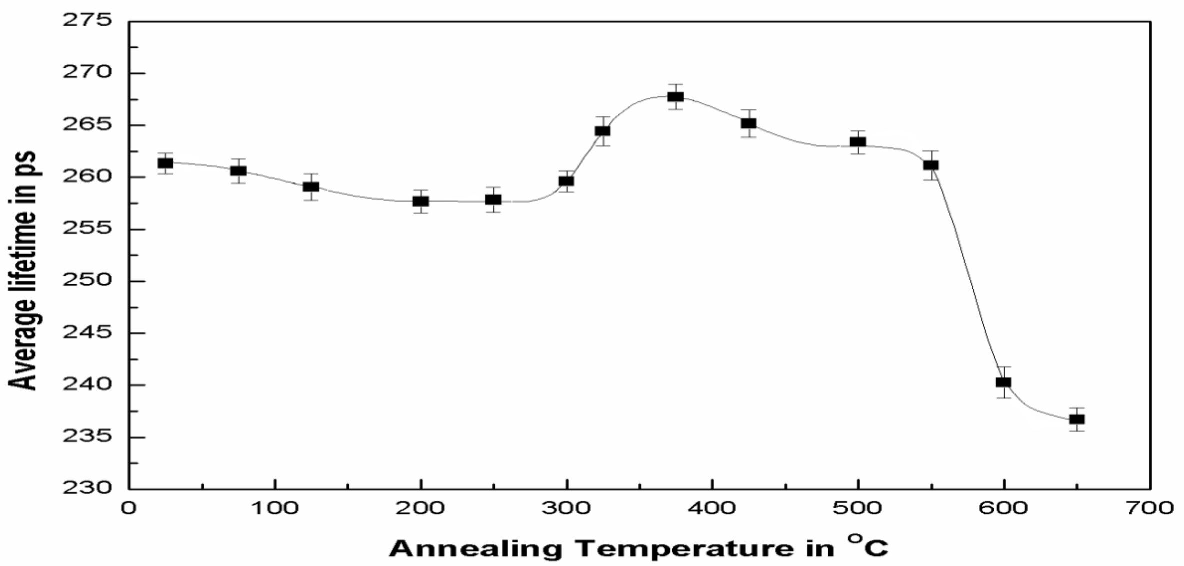

Fig: 5 Variation of the average positron lifetime (□$_{avg}$) with annealing temperature.

Figure 5 shows the variation of average lifetime (τ_{avg}) with annealing temperature. The average lifetime was calculated using the formula $\tau_{avg}= (\tau_1 I_1+ \tau_2 I_2+ \tau_3 I_3) / (I_1+I_2+I_3)$. Since τ_{avg} also gave the average defect contribution, its variation with annealing temperature was expected to be more or less same as with the S-parameter. At room temperature the observed average lifetime τ_{avg} = 261ps i.e. 19ps higher than the reference value which indicated the presence of vacancy like defects due to high energy alpha irradiation. In the temperature region 25 to 250^{O}C average positron lifetime value was stable. It agreed with the variation of S and R parameters. From 250^{O}C the average lifetime gradually increased and reached its maximum 268ps at 375^{O}C due to the migration of vacancies and reordering to more stable configurations. In this temperature region S and R parameters both followed similar behaviour. Beyond 375^{O}C, τ_{avg} gradually decreased via two stage recovery of radiation induced defects i.e. in the temperature region 375-550^{O}C and a sharp annealing stage in the temperature region 550-650^{O}C. Finally beyond 600^{O}C, average positron lifetime approached almost the bulk value indicating the process of annealing out of radiation induced defects to be completed. The variation in average lifetime resembled the behaviour of S-parameter and R-parameter. The above discussion on the S-parameter, R-parameter and the average lifetime gave the overall variation of microstructural defects with annealing temperature. The defect related lifetime τ_2 and the corresponding intensity I_2 were to be observed carefully for a better understanding of the defect dynamics.

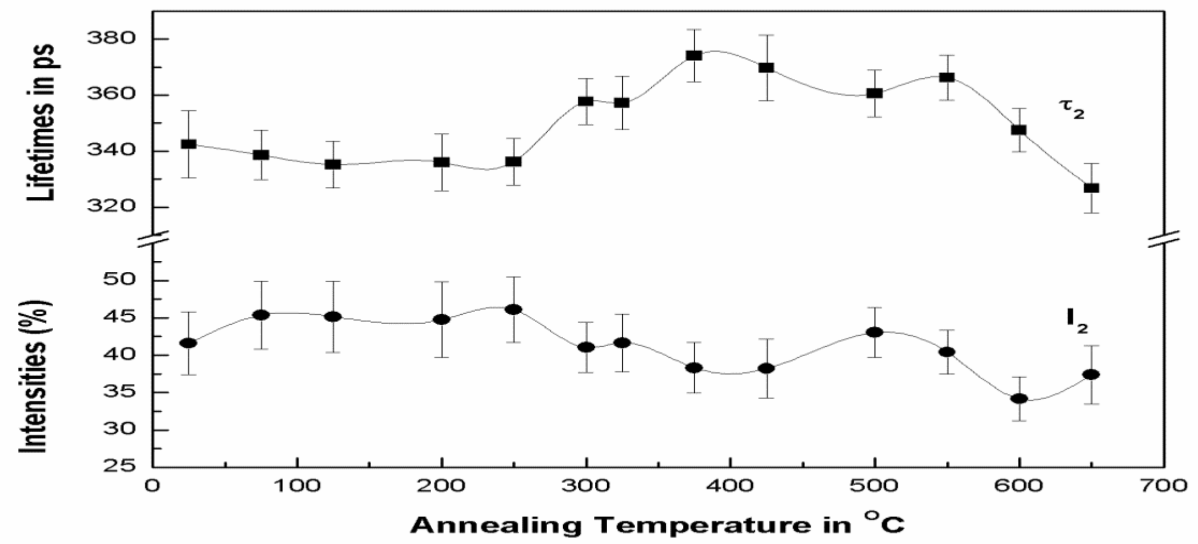

Fig: 6 Variation of defect related lifetime τ_2 and corresponding intensity I_2 with annealing temperature.

Figure 6 shows the variation of τ_2 and I_2 with annealing temperature. τ_2 was stable in the temperature range 25-250^{O}C which was attributed to the stability of the divacancies. Intensity was also almost stable in that region. In the temperature region 250 to 375^{O}C, τ_2 gradually increased to a maximum of 374ps i.e. 1.54times greater than bulk lifetime. This rise in lifetime value was attributed to the migration of divacancies, clustering of those divacancies and the formation of higher order vacancy clusters. The intensity I_2 also dropped in this temperature region which supported the agglomeration of vacancies and hence the fall of number of trapping sites. Beyond 375^{O}C and up to 650^{O}C, τ_2 decreased steadily via two annealing stages i.e. in the temperature region 375 to 500^{O}C and 550 to 650^{O}C indicating the dissociation of vacancy clusters and their dissolution leading to the annealing out of defects. Stability in the variation of I_2 was observed in that region. While discussing the isochronal annealing behaviour of the defect related lifetime τ_2, the influence of lattice relaxations had not been considered because in the presence of large open volume defects the lattice relaxations could be neglected [41]-[42].

IV. CONCLUSION

The reduced bulk lifetime τ_1 calculated from a two-state trapping model did not give a satisfactory fit to the experimentally observed τ_1 values. Monte Carlo method gave the depth distribution of vacancies induced by 40MeV alpha irradiation. Positron lifetime measurements indicated the formation of divacancies at room temperature. Average lifetime, S-parameter and R-parameter were more or less stable from room temperature (25^{O}C) to 250^{O}C which indicated that the predominant defect size was unchanged i.e. the stability of divacancy type defects. In the temperature region 250 to 375^{O}C, both S- and R-parameters increased gradually to a maximum indicating the migration of vacancies and clustering of those vacancies. In this temperature region defect related lifetime (τ_2) increased to a maximum of 374ps indicating the clustering of divacancies and the formation of higher order vacancy clusters. Beyond 375^{O}C, average lifetime, S-parameter and R-parameter started decreasing and finally at 650^{O}C reached almost the reference value indicating that the defects were annealed out.

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