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Positron annihilation studies in Li-implanted alumina

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Abstract. Depth dependent Doppler broadening of annihilation radiation (DBAR) measurements are carried out for a sample of Li ion implanted in alumina. The effect of Li ion implantation and the subsequent isochronal annealing at the temperatures up to 1100 °C on the Doppler broadening annihilation parameters (S-parameter) are studied. The S-parameter around the Li implantation depth (∼191 nm) increased with annealing temperature up to 700 °C and reduced beyond. The results suggest possible Li cluster formation in annealed sample.

1. Introduction

Metal or semiconductor nano-clusters are well known for their interest in optical properties as well as their scope in the fields of quantum dots [1, 2]. Ion implantation followed by annealing is one of the popular techniques used for synthesizing embedded nano-clusters in a host matrix [3]. It has been used to synthesize clusters of Si in SiO_2; Se, Cd and CdS in Al_2O_3; Li, Zn, Ag and Au in MgO;etc. [3-5]. Superiority of ion beam implantation technique is due to its ability to implant insoluble ions near the surface of host matrix. If the impurity is insoluble in host matrix then on thermal annealing they form precipitates followed by nano-clusters or nanocrystals [3].

Positron annihilation spectroscopy with the tunable energy slow positron beams is a very useful technique to probe specific depths. When heavy ions are implanted in a matrix, their implantation is localised around their range. Beam based positron annihilation studies are used to understand the radiation damage/defects created due to ion bombardment [4, 6]. Positron annihilation spectroscopy is also a very useful technique in studying the embedded systems. In many favourable cases, the differential positron affinity between the host matrix and embedded particles enables the selective study of the embedded systems. More positive the difference in positron affinities of the cluster and host, better is the confinement of positron in clusters. The positron annihilation studies on the effect of radiation and heavy ion implantation in semiconductors are well documented [7-9]. Besides the radiation effects, the Li clusters formed in high band gap materials like MgO were studied using this differential positron affinity between MgO and Li clusters to the advantage. The difference in the positron affinities causes most of the (92 %) positron localisation and subsequent annihilations inside the Li clusters, despite low percentage of metal doping (Li/MgO ∼ 1.3 %)[4]. Similar studies were reported on metal nano precipitates growth in alloys using coincidence Doppler broadening technique. About 90 % positrons were shown to annihilate in Cu nano precipitates in Fe even at very low concentration of Cu doped in Fe (Fe:Cu 1 % Cu)[10]. Although previous studies confirm formation of metal or semiconductor clusters inside the insulators in certain combinations, mechanism of cluster formation, dependency of growth rate of clusters on annealing temperature, defect formation due to irradiated ion beam and their annealing behaviour etc. are not fully understood. In present studies DBAR measurements have been carried out on Li ion implanted alumina for better understanding of these phenomenons.

2. Experimental Technique

Li ions of 50 keV from Low Energy Accelerator Facility (LEAF) at Bhabha Atomic Research Centre (BARC) have been implanted in single crystal α-Al_2O_3. The samples are of about 2.54× 2.54 cm^2 in area and 0.1 cm in thickness. The beam current was 200 nA and irradiation was done for 22 hours on about 1 × 1 cm ^{2} area of sample. Hence, the total fluence of implantation was 1×10^{16} ions/cm^2. The temperature change in the sample due to implantation of ions was not monitored. The ion implanted sample was isochronally annealed for 30 minutes at various temperatures in air. DBAR measurements are carried out using the slow positron beam at the Radiochemistry Division of BARC. The energy of the positrons is varied by floating the target to requisite voltage. A 30 % relative efficiency HPGe with resolution of 1.2 keV for 514 keV of ^{85}Sr is used for Doppler broadening measurements. The S-parameter is evaluated as fractional peak area in the region of 511±0.68 keV.

3. Results and Discussion

The range of 50 keV Li ions in the alumina matrix calculated using SRIM-2013 is about 191 nm. The S-parameter is evaluated from the DBAR spectra and the S-parameter profiles in the as received alumina and Li implanted alumina in the positron energy range up to 20 keV are shown in figure 1. The mean implantation depth of the positrons at various energies is calculated in nanometer (x) using equation 1, where ρ is the density of alumina in g/cm^3 (3.95 g/cm^3), and E is the energy of positrons in keV.

$$x(nm) \quad = \quad \frac{40}{\rho(g/cm^3)} \times E^{1.6}(keV) \tag{1}$$

The mean implantation depth of positron are also shown in figure 1. The calculated mean implantation depth of positrons of energy 6.3 keV corresponds to the range of 50 keV Li ions in the alumina matrix. The range of Li ions in the matrix is also indicated in figure 1.

As seen from figure 1 the S-parameter values in Li implanted sample are lower than that of as received sample till the depth corresponding to the positron energy value of 12 keV and there is no difference between the two samples beyond 12 keV. The lowering in S-parameter in Li implanted sample is maximum around the region of Li implantation, where the linear energy transfer of Li ions is also maximum. This indicates that the lowering in the S-parameter might be either due to the creation of vacancies by the Li ion or presence of Li impurity. The positron trapping in Al vacancies would enhance positron annihilation with comparatively higher energy valence electrons of oxygen, thereby reducing the S-parameter. The sample was annealed for 30 minutes in air at $500, 700, 900$ and 1100 °C temperatures and characterised by DBAR technique between each temperature change. The corresponding S-parameter profiles at these annealing temperatures are shown in figure 2 along with representative error bars. The relative variation of normalised S-parameter at 191 nm depth with annealing temperature is given in inset of figure 2.

It is seen from the figure that average S-parameter around Li implantation depth increases with annealing temperature and is maximum in the sample annealed at 700 °C. On further increase in the annealing temperature (1100 °C), the average S-parameter starts to decrease and the value falls even below the un-annealed sample. The increase in the S-parameter upon annealing at 500 °C or 700 °C could be attributed to either the agglomeration of the defects

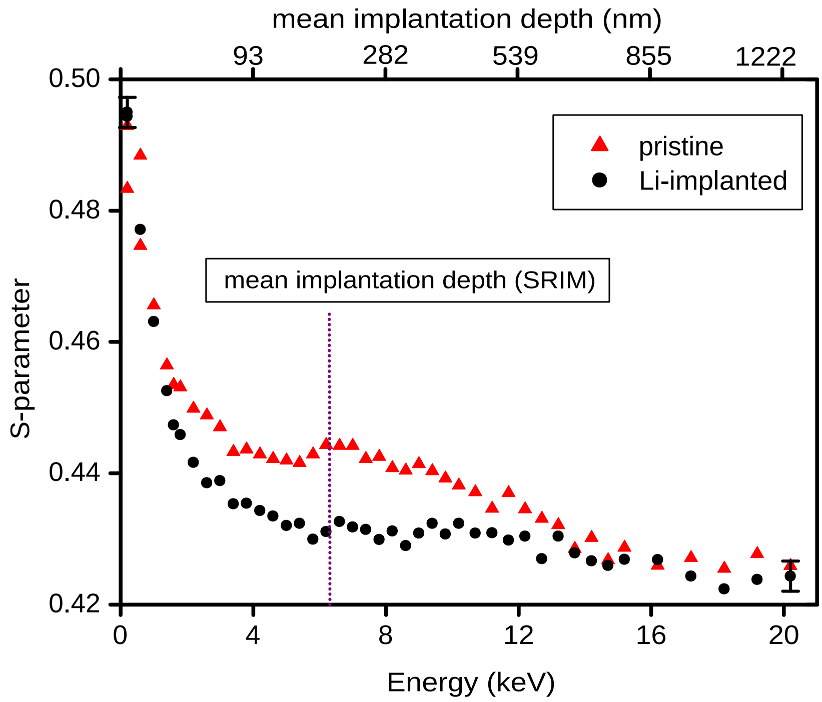

Figure 1. Depth dependent S-parameter profile with incident positron energy for the pristine sample and irradiated with Li-ion beam of 1×10^{16} fluence. Representative error bars are shown.

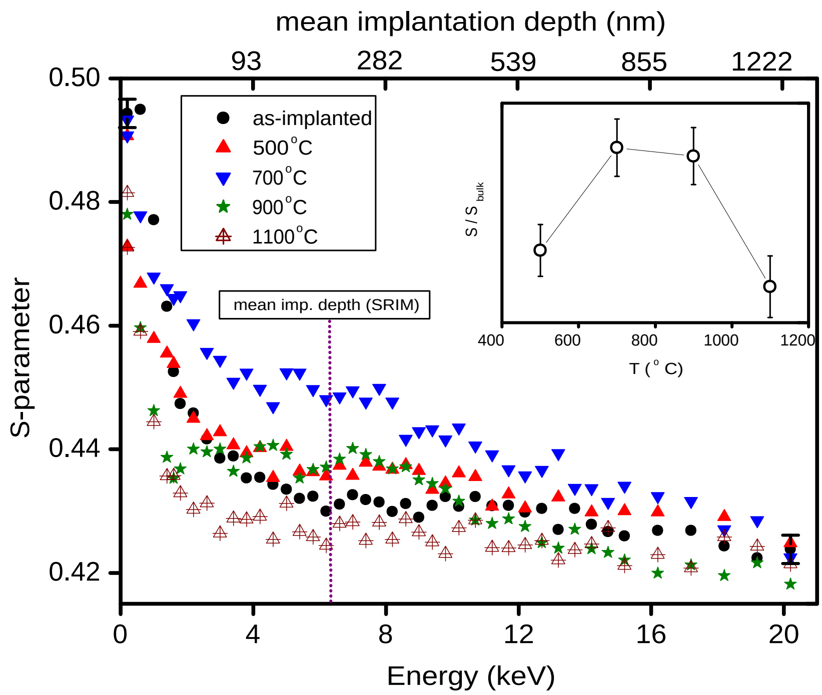

Figure 2. S-parameter profiles in Li implanted (1×10^{16}) alumina annealed at different temperatures. Inset shows variation in S-parameter at the implantation depth of Li-ions.

or positron trapping in the Li clusters formed. Similar behaviour of S-parameter was observed previously in the case of Li-ion implanted MgO on isochronal annealing and was attributed to the confinement of positron in the Li-clusters [4]. Similar phenomenon is also expected in the present case. On annealing at still higher temperatures (900 °C) the S-parameter has decreased. Have the changes in S-parameter with annealing temperature been due to agglomeration or annealing out of defects, the S-parameter should have been equal to the as received sample at highest annealing temperature. Lower S-parameter than even the as irradiated alumina in the case of the sample annealed at 1100 °C suggests that Li clusters might have migrated or dissolved leaving behind vacancies. Present experimental results suggest that the formation of Li clusters upon annealing up to 700 °C, and their breaking and migration beyond this temperature is more possible. However theoretical and experimental studies at higher Li fluences may be necessary to confirm this conclusion.

4. Conclusion

The Li ion implantation in alumina reduced the S-parameter around the Li implantation depth due to creation of large number of vacancies. Upon isochronal annealing, the S-parameter increases till 700 °C and then reduces beyond that. The increase in the S-parameter can be explained either as annealing out of the defects or positron trapping in Li-cluster formed upon annealing. However, the reduction in the S-parameter at higher annealing temperatures can not be explained based on defects annealing. Though Li-cluster formation and migration of these cluster at higher temperatures is the likely reason for the observed positron systematics, further theoretical studies are required to support the conclusion.

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