A. SWIFT HEAVY IONS IN MATERIALS

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Materials science beam line has been one of the most active beam lines due to large number of experiments by different groups from universities and other research institutions. A very brief summary of the experiments conducted in this year is given.

There were experiments to study the influence of SHI at the surface and interfaces. In the electronic sputtering of graphite by 150 MeV Ag ions showed enhancement around 60 degree which was explained on the basis of hcp structure of graphite. Atomic transport across the interface was investigated in some metal/Si, and metal/Ge under the influence of electronic excitation. The observed results are in accordance with thermal spike predictions on the sensitivity of metals to the electronic excitation. It has been shown in a demonstration experiment that it is possible to carry out on-line monitoring of the mixing across the interface using the indigenously developed LAPSDT detector with kinematic correction. The mixing across Au/Ge interface was observed in the irradiated annealed sample, whereas the irradiated sample did not show mixing upto the fluence of 10^{13} ions/cm^2 of 120 MeV Au ions.

Synthesis of nanoparticles by ion beams has been of interest. Ag doped glass on irradiation and subsequent annealing showed the photoluminescence, indicating the possibility of the formation of Ag clusters. Amorphous Sio_x films were irradiated with high energy Ag ions. Pristine Sio_x films did not give any PL signal whereas the irradiated sample gave PL in visible region due to the formation of Si nanocrystals. The red shift in PL peak with fluence indicated the increase in particle size with fluence. It suggests the possibility of control on the particle size of Si nanocrystals. The effect of SHI in low dimension systems in form of lattice matched semiconductor thin multilayers and strained layer superlattices (SLS) were investigated. In SLS system, the strain in the InGaAs/GaAs system was found to be decreasing with the fluence of 130 MeV Ag ions. It was observed that SHI irradiation induces strain in lattice matched InGaAs/InP multilayer. The lattice strain in SLS was measured by high energy channeling experiment.

Effects of SHI irradiation in terms of dielectric properties, magnetic properties, hardness, etc. were studied in various ferrites, HTSC, LCMO, PZT thin films. Some of the oxides were studied by online ERDA to study the loss of oxygen with fluence. It was not noticeable within experimental error. Stoichiometry of oxygen with respect to a heavier element was determined in some of these oxide systems.

Field emission characteristic of CVD diamond films were seen to be improved by 100 MeV Ag ion irradiation. Turn on voltage was considerably reduced with the ion irradiation. Fullerene film irradiation with Au, Ni and O ions was investigated by Raman, UV-Vis optical spectroscopy and resistivity measurements. The onset of conductivity

with fluence was found to be dependent on electronic energy loss deposition. The polymerization surrounding the ion track was also investigated.

Various polymer and polymer blends were irradiated to investigate the SHI induced modifications. Latent track in fused silica generated 200 MeV Ag ion beam was imaged by TEM and the diameter was found to be about 12 nm. On-line luminescence and PL and/or TL were employed to study the SHI induced defects and color centres in Saphire, Kyanite, photo simulated luminescence (PSL) and field emission display (FED) phosphors.

Experiments on energy loss measurements of heavy ions in gases were carried out with the objective to study the applicability of Bragg's law and to study the gas solid effect. New type of experiment on transverse cooling of channeled ions was initiated, using a two dimensional position sensitive detector. The experiments on the testing of VLSI devices under the ion irradiation at low flux was undertaken to simulate the effect of space radiation on the crucial devices mounted in space vehicles.

5.2.1 Electronic Sputtering from Semiconducting HOPG: A Study of Angular Dependence

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The studies on electronic sputtering from various materials including carbon allotropes have been reported by many groups [1-3], assuming the yield to be isotropic in all the directions. Toulemonde et al recently studied the angular distribution of sputtering yield from insulating samples such as LiF and SiO_2 [4], showing an angular anisotropy. In view of the above, we have measured the angular distribution of the sputtering yield from semiconducting highly oriented pyrolytic graphite (HOPG) sample.

The samples were irradiated with 130 MeV Ag beam incident perpendicular to the sample surface. Sputtered carbon atoms were collected on 10mm x 5mm Si catcher foils which were kept at a distance of 5 cm from the samples. The catcher foils were mounted on the secondary electron suppressor in such a way, so that all the catchers were equidistant in the horizontal plane. These were mounted at an angle of 10 to 90 degrees

from the beam direction. The sample was irradiated for a fluence of $9x10^{14}$ ions/cm^2 to collect sufficient carbon on small catchers for ERD analysis. Since the exposure time was long, the beam was scanned over an area of 5mm x 10 mm to avoid any damage to the sample. After the irradiation was complete, a 10 nm Al layer was deposited on the sample to avoid the collected carbon getting sputtered during subsequent ERDA study. A dummy was also placed in the chamber and contribution from contaminants was subtracted while calculating the actual quantity of sputtered carbon.

The carbon collected on the catcher foils is analysed by ERDA technique using 1.3 MeV Ar beam from 6 MV Pelletron at Max Planck Institute, Germany. Since the deposited carbon layer is of the order of a few nanometres, the analysis of the sputtered carbon was done using a high resolution spectrometer [5] set up. The sample was kept at an angle of 20^0 to the beam direction and the recoils were detected at an angle of 40^0. The energy spectra was obtained by projecting energy from the carbon band from the two dimensional spectra. The ERD spectra for the ten catcher foils was recorded and for clarity, the spectra for only five catchers at alternate angles is shown in Fig. 1. The peak in the spectra shows the sputtered carbon.

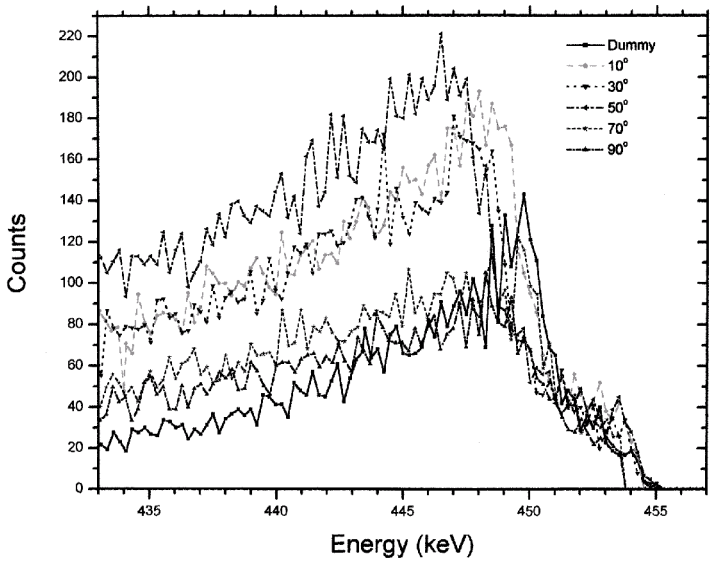

Fig. 1 : ERDA spectra upto a depth of 20 nm for five cathcer foils

The total carbon sputtered is calculated for each of the catcher foil from the area under the curve in the spectra and the same is plotted in Fig. 2. The total quantity of the sputtered carbon doesn't vary much for the 10^0, 20^0 and 30^0 catchers with a total content of approximately $4.2x10^{16}$ atoms/cm^2. There is a rise in the carbon content in the next two wafers with 50^0 catcher showing a maximum content of $5.6x10^{16}$ atoms/cm^2. The catchers at 70^0, 80^0 and 90^0 show sudden decrease with content going down to $0.2x10^{16}$ atoms/cm^2, a content even lower than the catchers at 10^0. We have calculated the average sputter rate as $5.5x10^3$ atoms/ion. This value is higher than the value obtained by S. Ghosh et al [6]. The observed increase in sputtering yield is expected to be due to the large ion fluence as

compared in earlier study, which is closer to the sputtering yield value for amorphised carbon [7]. The analysis of the distribution shows an anisotropic distribution with moderate sputtering at smaller angles. The spectra is fitted to a distribution $C=A\cos^{1.3}\theta+B\exp(-(\theta-53)^2)$, with a χ^2 value of 0.07, which shows that a Gaussian peak lies at around 53^0 on a distribution which otherwise has a over-cosine distribution. We feel the distribution shows the existence of a pressure pulse, due to which, for perpendicular beam incidence, the ejected atoms are more likely to travel along the axis at 60^0 to the surface normal, for hcp structure of HOPG with [001] surface.

Fig. 2 : Angular distribution of the sputtering yield showing total carbon collected on all ten catcher foils

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5.2.2 Formation of $Au_{0.6}Ge_{0.4}$ Alloy Induced by Au-Ion Irradiation of Au/Ge Bilayer

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Ion beam mixing using swift heavy ion (SHI) irradiation is a field of recent interest due to its fundamental importance. A very few works have been reported in this direction. In this work, we have studied the formation of oriented $Au_{0.6}Ge_{0.4}$ alloy by irradiating Au/Ge bilayer films with 120 MeV Au ions at a fluence of 1×10^{13} ions/cm^2 and by subsequent vacuum annealing.

Thin films of Au (42.5 nm) and Ge (38 nm) were deposited on Si (100) substrates by electron-beam evaporation technique. The films were irradiated at Nuclear Science Centre using 120 MeV Au^{9+} ions at room temperature at different fluences in the range of 1×10^{12} to 1×10^{13} ions/cm^2. The pristine as well as the irradiated samples were later vacuum annealed at 360°C for different time durations. We have characterized the samples by Rutherford backscatetring spectrometry (RBS), x-ray diffraction (XRD), scanning electron microscopy (SEM), and transmission electron microscopy (TEM). The beam was scanned over the entire surface area to get a uniform irradiation.

RBS analyses before and after irradiation of the Au/Ge bilayer structure showed no change even up to the highest fluence. However, annealing the irradiated samples at 360°C in vacuum up to 5 h showed vigorous reaction across the Au/Ge interface. The results have been shown in Fig. 1 and we observe that the Au signal height decreases nearly by a factor of three and Ge as well as Si signal positions move nearer to their surface positions.

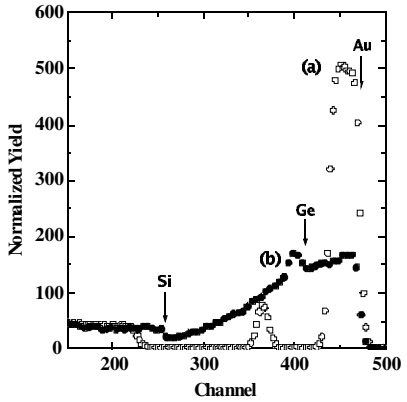

Fig. 1 : RBS spectra of Au/ge bilayer structures: (a) irradiated with 120 MeV Au ions at a fluence of 1×10^{13} ions/cm^2 and (b) irradiated as well as vacuum annealed at 360°C for 5 h

XRD measurements also show the similar trend. We observed only the Au reflections of (111), (200) and (220) indicating the polycrystalline nature of Au. However, in case of the irradiated and annealed sample, a strong peak evolves, which was identified as (800) reflection of $Au_{0.6}Ge_{0.4}$ alloy phase. These results have been depicted in Fig. 2. Since no other peak of this alloy phase is seen, we have predicted the formation of oriented Au-Ge alloy phase.

SEM analyses on the pristine sample show continuous film with no special feature although the irradiated and annealed sample shows a lot of isolated island structures of ~2 μm in size. The island structures as seen in Fig. 3 are surrounded by black regions, which have been found out to be Si (from energy dispersive x-ray analysis measurements). This explains the very special shape and features of the corresponding RBS spectrum (Fig. 1(b)). If we assume that the islands are made up of Au-Ge alloy as seen from the XRD measurements, their isolated nature helps the RBS probing beam to see Si through the gap between them. Also it is the alloy formation with a little amount of unreacted Au left at the top for which the Ge signal appears almost at its surface position. Broadening of the Au and Ge signals is also typical of island formation and alloying.

To confirm the oriented nature of the Au-Ge alloy, we performed the TEM measurement and selected area diffraction (SAD) pattern obtained from one of the isolated islands indeed shows the single crystalline nature.

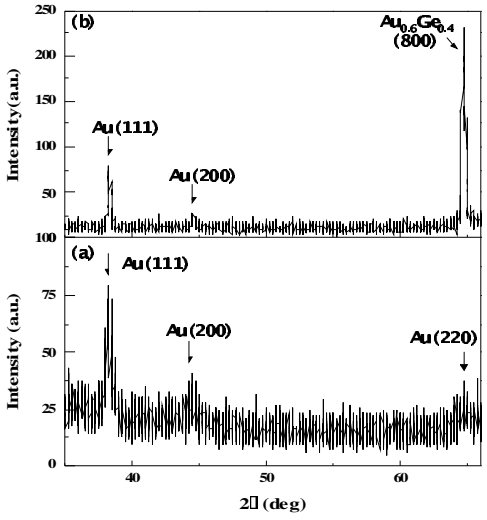

Fig. 2 : XRD patterns of Au/Ge bilayer structures: (a) irradiated with 120 MeV Au ions at a fluence of 1×10^{13} ions/cm^2 and (b) irradiated and vacuum annealed at 360°C for 5 h

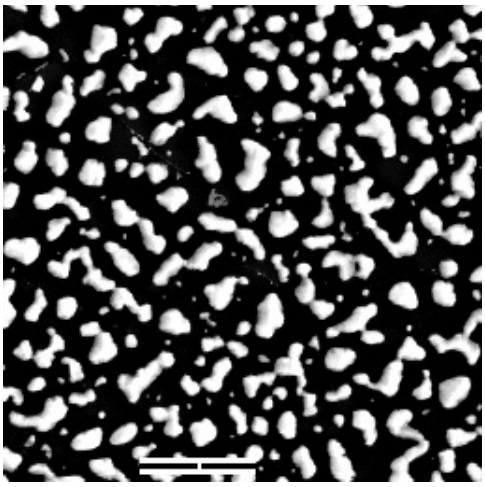

Fig. 3 : SEM micrograph of Au/Ge bilayer structure after irradiation and vacuum annealing (as described earlier). The bar size indicates 10 μm

5.2.3 Swift Heavy Ion Induced Interface Modification in Ni/Ge

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Metal/semiconductor based systems are good candidates for case study for ion beam mixing using swift heavy ion (SHI) irradiation due to the fundamental importance as well as wide applications. Recently, we have reported SHI induced oriented Au-Ge alloy formation. In this direction, we have taken up the SHI induced msiing study in Ni/Ge system. In this work, we have studied the formation of Ni_2Ge alloy phase by room temperature irradiation of Ni/Ge bilayer films with 100 MeV Au ions up to a fluence of 1×10^{14} ions/cm^2.

Thin films of Au (46.5 nm) and Ge (38 nm) were deposited on Si (100) substrates using UHV electron-beam evaporation set-up. The films were irradiated at Nuclear Science Centre using 100 MeV Au^{7+} ions at room temperature (RT) and liquid nitrogen temperature (LT) with different fluences in the range of 1×10^{12} to 1×10^{14} ions/cm^2. We have characterized the samples by Rutherford backscatetring spectrometry (RBS) and cross-sectional transmission electron microscopy (XTEM). The beam was scanned over the entire surface area to get a uniform irradiation.

RBS analyses before and after RT irradiation of the Ni/Ge bilayer structure showed mixing to take place (corresponding to the highest fluence), which is evident through the shift in the RBS peaks from the base line, peak broadening and a kink in the rising part of the Ni signal. The results have been shown in Fig.1. Likewise, we observe the mixing for LT irradiated sample although the extent of mixing is less than that of RT. From the broadening of the Ge peak, we have calculated the rate of mixing corresponding to the fluence of 1×10^{14} ions/cm^2, which turn out to be 99.6 nm^4 and 323.9 nm^4 for LT and RT, respectively. However, the very fact that the Ge signal height also decreases, there may be irradiation induced surface inhomogeneity associated with the Ge surface.

XTEM measurements show the top layer to be amorphous, while the underneath Ni layer is a polycrystalline one. Thickness of these two layers closely matches with that obtained from RBS simulation. High-resolution lattice image indicates the presence of (111) and (200) planes of Ni. Similar trend is observed from x-ray diffraction measurements. XTEM micrograph collected from the RT irradiated sample shows a lot of non-uniformity in the top Ge layer and a small layer (~6-7 nm), having a different orientation and grain size as compared to the Ni layer, to get formed between the polycrystalline Ni and the Ge layer. Calculations yield two different d-spacings of 0.291 nm and 0.254 nm, which closely match with the (102) and (200) planes of Ni_2Ge phase.

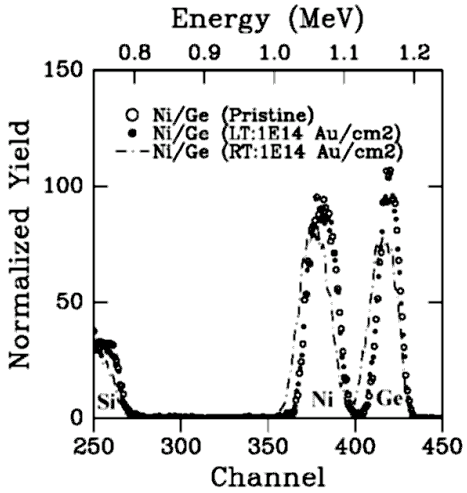

Fig. 1 : RBS spectra of Ni/Ge bilayer structures: Pristine, 100 MeV Au irradiated at RT and LT

We attribute these effects to the swift heavy ion (SHI) irradiation induced mass flow in the amorphous Ge layer followed by creation of a large number of point defects in the Ni layer. Although typically Ni is insensitive to SHI irradiation, S_n induced being cumulative, response of the Ni layer would be quite different in contrast to the bulk. As a matter of fact, S_n being almost equal to the value corresponding to low energy Kr and Ar ions, where Ni-Ge alloy phase formation was reported earlier, it is quite obvious that, in the presence of a large number of point defects created by S_n , S_e-induced effects would

lead to enhanced atomic movements across the highly reactive Ni-Ge interface to cause high mixing rate. For LT, less atomic mobility leads to lesser amount of mixing.

5.2.4 Study of swift heavy ion irradiation effects in metal/Si systems

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In case of metallic system it has been observed that the damage production due to electronic energy loss S_e can take place only above a certain threshold value [1,2]. This threshold value greatly varies form metal to metal. Aim of the present work is to compare S_e sensitivity of different metals in the form of thin films by studying their intermixing with Si under identical conditions of irradiation, and to get information about the structural evolution of intermixed region using depth selective XANES measurements. Metals chosen for the present study are tungsten (highly insensitive to S_e), iron (having intermediate sensitivity to S_e) and titanium (having high sensitivity to S_e).

It is well known that in thin films the factors like grain size and defect density significantly depend on the deposition parameters like vacuum condition, substrate temperature and deposition rate. A meaningful comparison between thin films of different metals can only be done by preparing different films under identical conditions. Keeping in mind the above aspects, a single multilayer sample was prepared having the following nominal structure: silicon(substrate) /Cr(20nm)/ Au(60nm)/ Si(12.5nm)/ W(3.0nm)/ Si(12.5nm)/ Fe (3.0nm)/ Si(12.5nm)/ Ti(3.0nm)/ Si(12.5nm). The bottom Au layer is deposited in order to achieve formation of x-ray standing waves in the multilayer structure above it [3,4].

The sample was irradiated with 100 MeV Au ions at the 15UD Pelletron at the Nuclear Science Center, New Delhi. It was analyzed using the techniques of x-ray reflectivity, x-ray standing wave and depth selective x-ray absorption near edge structure (XANES). Measurements were done at the ID32 beam line of ESRF, Grenoble, France. Depth selectivity in XANES measurements was achieved by doing measurements under x-ray standing wave conditions: total reflection of x-rays from the Au layer results in setting up of standing waves inside the multilayer. As the scattering vector $\boldsymbol{q}$ is varied, the positions of antinodes shift. By making the center of an antinode to coincide with either the center or the interfacial region of a layer, structural information from different regions of a layer can be obtained. For XANES measurements, energy of the x-rays was scanned across the absorption edge of a particular element up to an energy of 300 eV beyond the

absorption edge under constant ***q*** condition. Reflectivity measurements were also done using laboratory x-ray diffractometer with Cu K_α radiation.

A simultaneous fitting of reflectivity and fluorescence data yielded reliable information about the structure of the multilayer before and after irradiation. Particularly, the fluorescence data can yield unambiguous information about the positions of the metal layers. This information was used as input for fitting the reflectivity data. The final fitted values of electron density profiles in the two samples are shown in Fig. 1. Irradiation results in two significant modifications: substantial densification of the multilayer, and intermixing of the metal layers with Si.

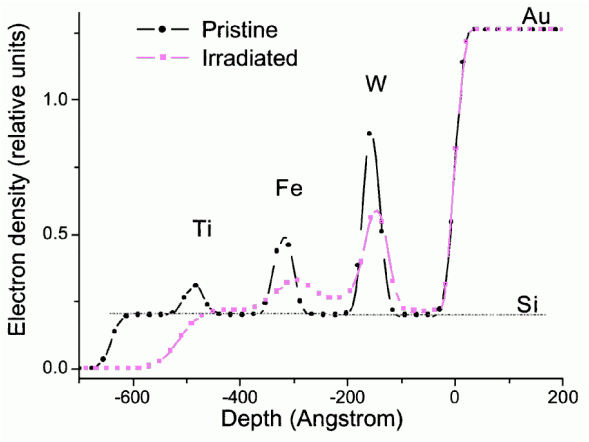

Fig. 1 : Electron density profile in the pristine as well as irradiated specimens as obtained from the combined fitting of the x-ray reflectivity and fluorescence yield data

Table 1 : Position of the centres of different layers relative to the top surface of the Au layer. Results are presented for both pristine as well as irradiated samples

Layer	Position of the center of metal layer in nm	
	Pristine	Irradiated
Top surface	63.9	51.5
Ti	48.4	43.1
Fe	31.8	29.3
W	15.4	14.5

Table 1 gives the position of the centers of different metal layers before and after irradiation. The over all thickness (above the Au layer) reduces from 64.0nm to 51.5nm, i.e. a reduction by ~20%, which is substantial. It may be noted that densification is not uniform throughout the multilayer; while the reduction in the thickness of the Si layer below W is only ~6%, that in thickness of Si layer between Fe and Ti layers is ~17%. Since the mixing of W with Si in rather small, a reduction of ~6% in the thickness of Si layer below W may be attributed to actual densification of Si layer. A larger reduction in the Si layer thickness between W and Fe or Fe and Ti layers is expected to be a combined ef-

fect of densification and metal silicide formation. A significantly large reduction of the thickness of the top Si layer from 14.0nm to 7.0nm evidences an electronically mediated sputtering.

Perusal of Fig. 1 shows that irradiation results in significant intermixing of the metal layers with Si. However, there is large variation in the efficiency of intermixing of different metals. While Ti and Fe exhibit large intermixing with Si, intermixing of W layer is rather small. An estimation using SRIM code suggests that the S_e values for 100 MeV Au ions in Ti, Fe and W are respectively 21 keV/nm, 30 keV/nm, and 34 keV/nm. While in Ti the S_e value is above the corresponding threshold, in Fe and W layers it is below the thresholds. Thus, in case of Ti a large intermixing is as per expectation. On the other hand, in case of Fe the observed intermixing can be understood in terms of a reduction in the threshold S_e value in thin film as compared to that in bulk metal. As discussed in the introduction, a decrease in the electron mobility due to scattering from the grain boundaries and surface/interfaces and a possible modification in the electron-phonon interaction can result in a decrease in threshold S_e value in Fe film as compared to the bulk. In case of W film small intermixing suggests that even after reduced electron mobility, the threshold S_e value remains above 34 keV/nm. Since in W even the nuclear energy loss is substantial (1.2 keV/nm), the observed mixing may be attributed to the same. Thus, In all the three metals damage/intermixing is observed at S_e values well below the threshold value in bulk. However, their relative sensitivity to electronic energy loss is maintained.

XANES measurements were done in order to elucidate the structure of the Fe and W layers before and after irradiation. Measurements were done under standing wave condition in order to get selective structural information about the center of a layer as well as the interfacial region. Absence of any sharp features in the XANES of irradiated specimen suggests that the layer has become amorphous after irradiation. In addition we notice a close similarity between the experimental XANES with the simulated XANES of an amorphous structure based on $FeSi_2$.

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5.2.5 Mixing in Cu/Ge system by swift heavy ions

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In the present work, we have investigated the ion beam mixing in the Cu/Ge system using high energy ions. The samples of Cu/Ge bilayer were irradiated by 120 MeV Au and 140 MeV Au ions to fluence of $1x10^{13}$ and $3x10^{13}$ ions/cm^2 and multilayer by 120 MeV Au ions to fluence of $1x10^{13}$ and $1.7x10^{13}$ ions/cm^2. The concentration profiles at the interface were determined by Rutherford backscattering spectrometry (RBS) of pristine and irradiated samples. The experimental data of bilayer were fitted with Gaussian error functions. The observed increase of the variance $\Delta\sigma^2(\phi) = \sigma^2(\phi) - \sigma^2(0)$ was taken as a measure for the mixing effect and a mixing rate defined as $k = \Delta\sigma^2(\phi)/\phi$, was estimated. The pristine and irradiated Cu/Ge multilayers samples were annealed at 200^0C temperature. The irradiated annealed samples showed considerable mixing in comparison to pristine annealed due to radiation effected diffusion.

RBS spectra of the as-deposited and irradiated samples revealed that the lower edge of the Al and front edge of Ge are not showing any change indicating that there is no mixing at this interface. But the Ge and Cu interfaces indicate that the mixing occurred at this interface which increases with the fluence. The lower edge of the Cu shows mixing with the Si at the fluence $3x10^{13}$ ions/cm^2 at both the energies. The electronic stopping power, energy density (F_D) deposited in the elastic atomic collisions and mixing rate at the fluence $3x10^{13}$ ions/cm^2 for the both energy are shown in table 1. It shows that energy density (F_D) deposited by 120 MeV Au and 140 MeV is almost same but the difference between electronic stopping powers is about 2keV/nm for both the energies. Higher mixing rate for higher stopping power shows that mixing increases with increase in electronic energy loss. The mixing efficiency are $k/S_e \approx 3.94$ and 4.85 nm^5/keV by 120 MeV and 140 MeV Au ions respectively. Kraft et al. [1] reported mixing efficiency of 0.2 nm^5/keV at the Ni/Si and 2.1 nm^5/keV at the ZnO/SiO_2 interface for the samples irradiated at liquid nitrogen temperature. While insulators are very sensitive to the electronic energy loss of the impinging ions and amorphous tracks are formed ever at rather low S_e values of the order of 1-20 keV/nm [2], metals were found to be only slightly affected by high energy ions and usually exhibit high threshold values of some tens of keV/nm. We got a higher mixing rate in comparison to [1,3,4]. This may be due to the radiation-enhanced diffusion during irradiation at the room temperature

The Cu/Ge multilayer sample shows very little mixing at the fluence $1.7x10^{13}$ ions/cm^2. The pristine and irradiated samples were subsequently annealed in the vacuum at 200^0C up to 30 minute, which is below the eutectic temperature. The pristine annealed samples show very little change in comparison to irradiated annealed sample. For the irradiated samples, when sufficient thermal energy is supplied to the system during post irradiation annealing, atomic mobility across the interface is enhanced due to SHI induced defects. Thus, radiation-affected diffusion causes higher/significant atomic motion across the interface. RBS spectra of the samples irradiated with two different fluences $1x10^{13}$ and $1.7x10^{13}$ ions/cm^2, are annealed at 200^0C up to 30 minute. Irradiated annealed sample at fluences $1.7x10^{13}$ ions/cm^2 shows higher mixing but irradiated and annealed sample at fluence $1x10^{13}$ ions/cm^2 does not show significant difference from that of pristine annealed. At the higher fluence more defects are present, which are more mobile during annealing.

Table 1 : Ion, energies E, electronic stopping power S_e, energy density F_D deposited by nuclear collisions and mixing rate k at the fluence $3x10^{13}$ ions/cm^2 at the interface of Ge/Cu. For the calculation of mixing efficiencies average values were taken into account

Ion	E (MeV)	S_e (keV/nm) for Ge/Cu	F_D (keV/nm) for Ge/Cu	$\Delta\sigma^2$ (nm^2)	k (nm^4)
Au	120	19.07/32.7	0.17/0.39	30.6	102 ± 19.67
	140	21.08/36.2	0.17/0.35	41.7	139 ± 24.11

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5.2.6 Mixing Induced by Swift Heavy Ion Beam at (Ti, Ni) / Si Interface

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The present work deals with the study of ion beam mixing induced by the electronic excitation of Ti, Ni and Si by the incident swift heavy ion. The mixing at the interface is studied by RBS technique and X-ray measurements. The mixing in Ni/Si system is studied followed by thermal annealing of the interfacial region.

A single layer of Ni [~15 nm] and Ti [~18 nm] was deposited by e- gun evaporation on Si (100) substrate at $4x10^{-8}$ Torr at NSC, New Delhi. The films were irradiated with 95 Mev Au ions at RT to a dose of 10^{13}ions/cm^{2}using NSC Pelletron, having beam over the 1 cm^2 area at a current of 1 *pna.* Using the TRIM calculation, the electronic energy loss has been calculated, 20.43 *keV/nm* for 95 Mev gold ions in Ti and 30.505 *keV/nm* in Ni.

RBS spectra of Ni and Ti on Si (100) were recorded for pristine and irradiated samples. In case of Ni/Si the analysis of the RBS spectra does not show any significant mixing or it might not be resolvable in the RBS spectra below the resolution of 10 nm. RBS studies conform the presence of 15 nm of Ni and 18 nm of Ti over Si.

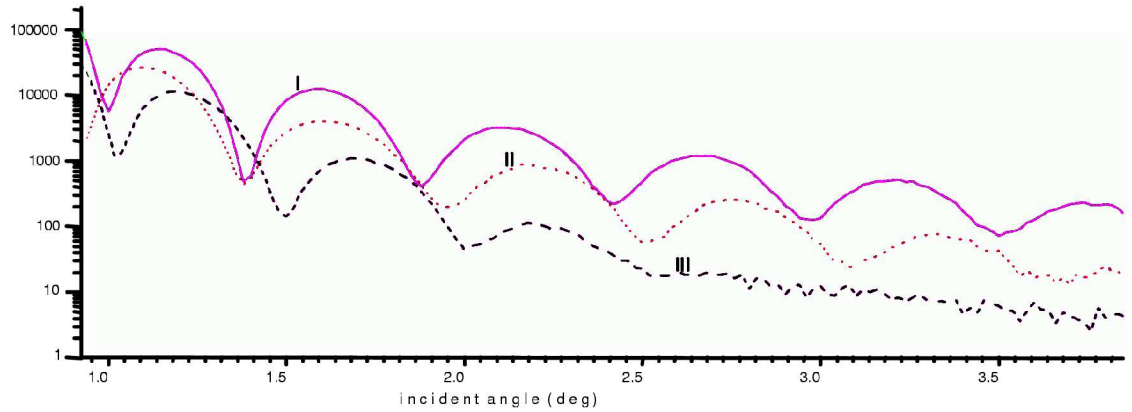
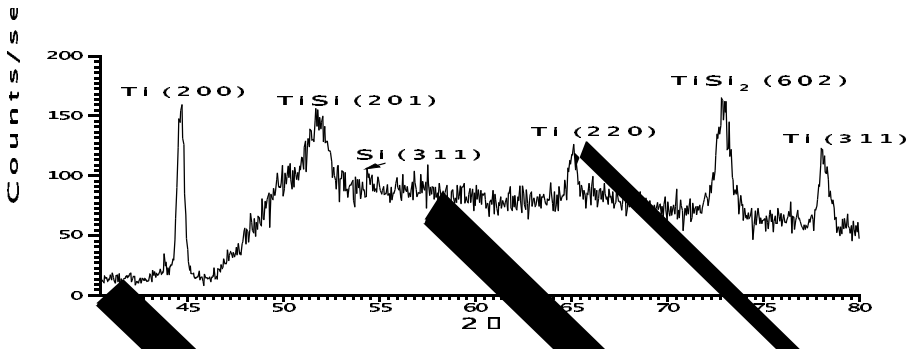


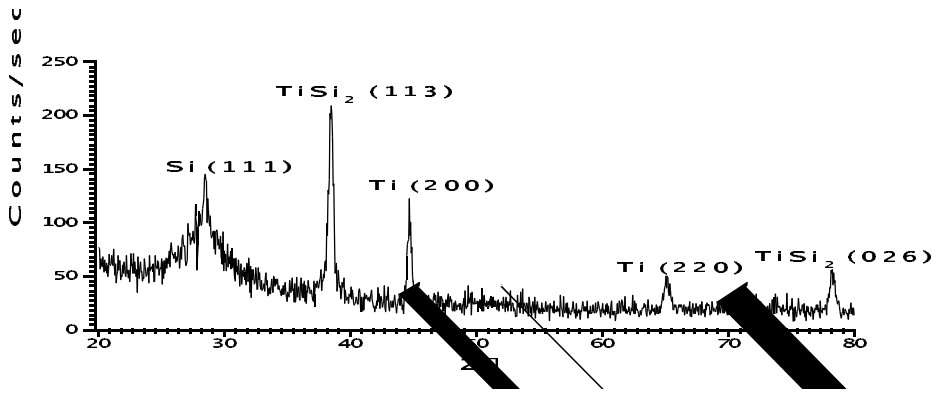
Fig. 1 : Reflectivity patterns of Ni/Si system I pristine, II pre annealed, III post annealed

Fig. 1 shows the XRR patterns of the Ni/Si samples. Annealing of the as-deposited specimen causes some change in the reflectivity pattern but the specimen treated with post annealing (after irradiation) shows the reduction in the heights of Bragg peaks as well as indications for the change of the interfacial structure.

The reflectivity patterns of pristine and irradiated specimens for Ti/Si system show that irradiation causes the shift in Bragg peaks. The reflectivity pattern after irradiation is attributed to the absence of sharp interface between the Ti and Si due to irradiation. This shows intermixing effects at the interface of Titanium and Silicon. Fig. 2 (a) and (b) are the GIXRD patterns for Ti/Si system that shows different crystalline peaks.



(a) Pristine specimen



(b) Irradiated specimen

Fig. 2 : GIXRD patterns of Ti/Si system

5.2.7 Mixing in Au/Si system by nuclear energy loss

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In the present work, we report the formation of silicide phases in the Au/Si system by ion beam mixing at room temperature. The samples (56nm Au on Si) were irradiated by 1 MeV Xe ions to fluence of $1x10^{15}$ and $5x10^{15}$ ions/cm^2. The ion energy was chosen in such a way that it deposits maximum energy at the interface. The Rutherford backscattering spectrometry (RBS) measurements were done on the pristine and irradiated samples to determine the composition of mixed region. Grazing incidence X-ray diffraction (GIXRD) measurements were performed which showed the formation of silicide phase (Au_2Si, Au_3Si, Au_5Si and Au_5Si_2). Scanning electron microscopy (SEM) measurements indicated the micron size crystallites in the irradiated samples.

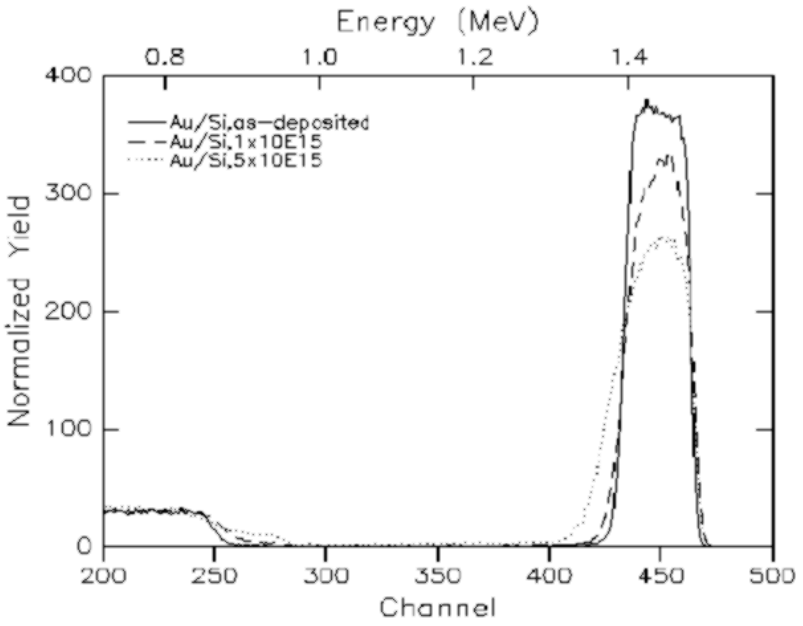
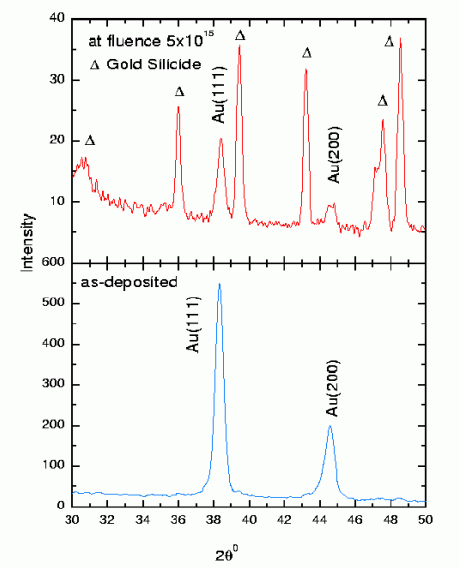
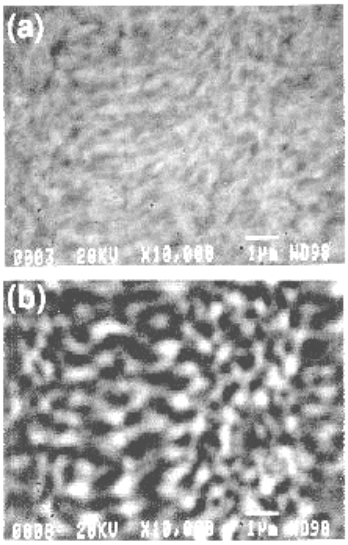


Fig. 1 : RBS of a thin (56nm) Au film on Si (100) substrate after being irradiated with 1 MeV Xe^+ ions to a fluence of $1x10^{15}$ and $5x10^{15}$ ions/cm^2 at RT

Fig. 1 shows the RBS spectra of the as-deposited sample and samples irradiated at room temperature (RT) at fluences of $1x10^{15}$ and $5x10^{15}$ ions/cm^2. It is seen from Fig. 1 that ion irradiation induces a progressive intermixing at the Au/Si. The reduction in the slope of the lower edge of the Au peak and front edge of silicon indicates that the mixing occurred in the Au/Si interface which increases with the fluence. The RBS spectra shows the large mixing at fluence $5x10^{15}$ ions/cm^2. Fig. 2 (a) shows the GIXRD pattern of the as-deposited sample in which only the peaks of Au(111) and Au(200) are seen. Fig 2 (b) shows the GIXRD of the irradiated sample at fluence $5x10^{15}$ ions/cm^2. It indicates the formation of gold silicide (Au_2Si, Au_3Si, Au_5Si and Au_5Si_2) and unreacted gold [1]. The formation of a phase depends on the mobility of the reacting species and the temperature during irradiation. In a solid state reaction and ion beam mixing both the reaction kinetics as well as thermodynamics driving forces play an active role during phase formation [2]. The silicide phase formation in the present work is obtained by irradiation at room temperature and without any post annealing, whereas in all the previous work, the Au silicide formation is reported either by irradiation at eutectic temperature [3] or by post irradiation annealing [4]. This may be due to the fact that in the present work the nuclear energy deposition (S_n) by elastic collision is around 5.7 keV/nm, which is optimized to be maximum at the interface. Whereas in all the previous work the S_n is lower and it is not optimized for the interface.

The surface morphology of the films was studied by SEM. Fig 3 (a) and 3 (b) shows the SEM micrograp of the irradiated films at fluences of $1x10^{15}$ and $5x10^{15}$ ions/cm^2. Fig. 3 (a) shows the mixture of Au and amorphous gold silicide phase and Fig. 3 (b) shows crystalline size of gold silicide around 0.5 micron. Two figures show that with the increase in the fluence amorphous phase changed to crystalline phase. The previous





work reported formation of triangular islands in Au/Si (111) system during thermal annealing at eutectic temperature. The size of this type of islands as obtained by [3] are around few microns. But in the present case, the crystalline size of gold silicide is less than 1 micron at RT.

Fig. 2 : (GIXRD) pattern of Au/Si system: (a) as-deposited (b) Xe^+ ions irradiated at a fluence of $5x10^{15}$ ions/cm^2

Fig. 3 : SEM micrograph of Au/Si system: (a) Xe^+ ions irradiated at fluence $1x10^{15}$ ions/cm^2 (b) irradiated at a fluence $5x10^{15}$ ions/cm^2

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5.2.8 UV-VIS and Photoluminescence (PL) Characterization of Ag Nanoparticles Doped Glasses prepared by Ion Implantation/Ion Exchange Methods

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Attempt has been made to prepare Ag doped glasses by ion implantation and ion exchange methods. The motivation for this work has been derived from the "caged" fluorescence observed from silver oxide nanoparticles coated glasses reported by Dickson et al [1]. These films could be sensitized by blue light, which presumably forms Ag_n clusters, those give rise to red luminescence on exposure to green light. This property has a potential application in the field of optical memory devices.

Here we report preparation of Ag_n clusters doped glasses by ion exchange method in combination with ion implantation technique.

Preparation of Ag Doped Glasses by Ion Exchange followed by Ion Irradiation:

In the present investigation soda lime glass slides were ion exchanged with fused salts of $AgNO_3$ and $NaNO_3$ (molar ratio 5 x 10^{-3}) [2] at 350° C for 30 minutes. Fig. 1 depicts absorption and emission spectra recorded on one such sample. As can be seen, absorption spectra for both the samples (Figure 1(A) and (B)) are featureless. The samples were irradiated with 100MeV Ag ions at a fluence of 1 x 10^{13} cm^{-2} from the Pelletron accelerator at NSC, New Delhi. Irradiation of these samples leads to a distinct peak at ca. 620 nm in the Photoluminescence (PL) spectrum (Fig. 1(C) and (D)). Controlled sample without any ion exchange but after irradiation did not show these features (spectra not shown). The PL at ca. 620nm in case of silver oxide films has been attributed before [3,4] to the formation of Ag_n clusters whose size is less than eight atoms. Thus, the present experiments suggest the formation of such Ag_n clusters in the glass matrix.

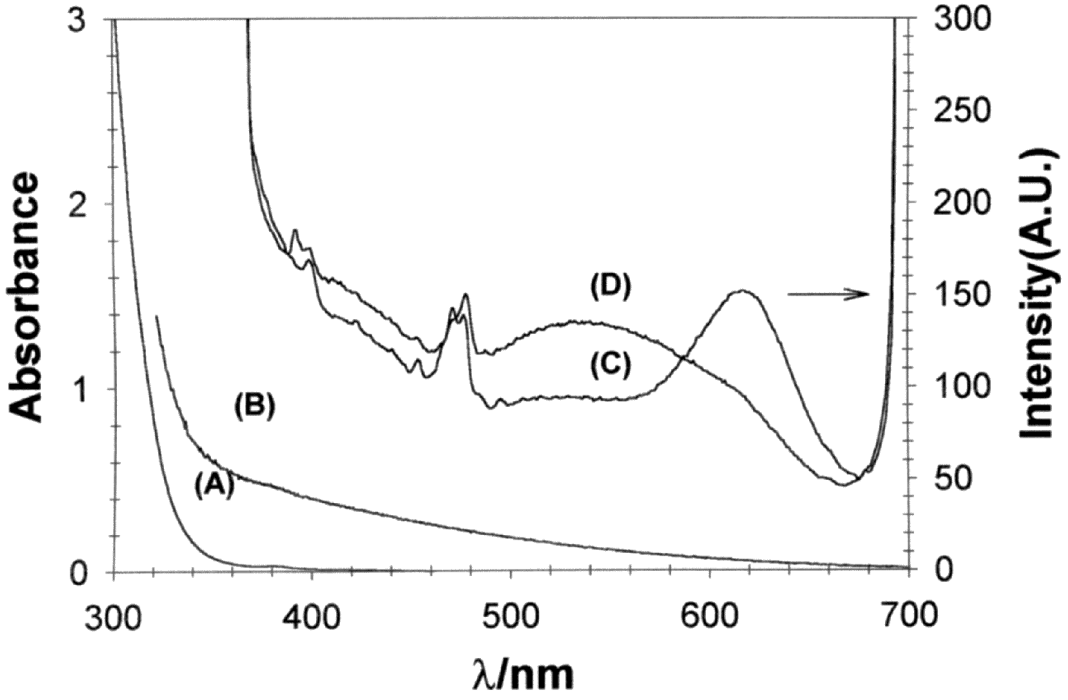

Fig. 1 : Absorption (A) and emission (D) spectra recorded for ion-exchanged sample.

Absorption (B) and emission (C) spectra recorded for ion-exchanged sample after Ag^+ ion irradiation at 100 MeV, 1 x 10^{13} cm^{-2} Fluence

Preparation of Ag/Ag_xO Doped SiO_2 Glasses by Ag Ion Implantation followed by Ag Ion Irradiation:

Ag^+ ions were implanted in quartz at 165 MeV or 100 MeV at a fluence of ~ 5 x 10^{14} cm^{-2}. Some of the samples were also implanted by O^+ ions at 30MeV (5 x 10^{14} cm^{-2} fluence). A few of the ion-implanted samples were irradiated by Ag^+ ions at 190 MeV or 130 MeV (5 x 10^{13} cm^{-2} fluence). This procedure is known to lead to the formation of metal nanoparticles in the glass matrix [5]. UV-visual absorption spectra recorded on the irradiated samples are found to be featureless and do not show the expected plasmon resonance at ca. 420 nm. It could be due to low density of Ag in the matrix. Fig. 2 shows PL spectra recorded on these samples. The O-implanted sample shows the distinct emission at ca. 620 nm associated with Ag_n clusters. Annealing the samples in nitrogen atmosphere at 460° C lead all the PL spectra to merge to the same feature, that is, they show characteristic Ag_n PL at ca. 620 nm.

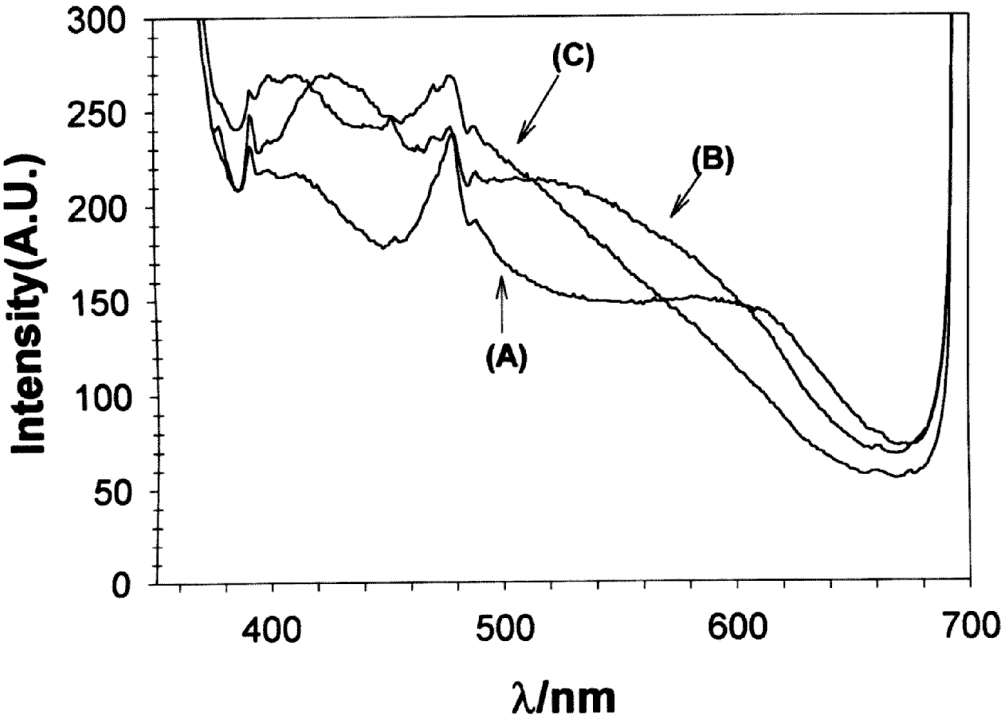

Fig. 2 : Emission spectra of ion implanted SiO_2 samples before annealing. (A) For O^+ implantation at 30 MeV, 5 x 10^{14} cm^{-2}, then Ag^+ implantation at 165 MeV, 5 x 10^{14} cm^{-2} followed by Ag^+ Irradiation at 190 MeV, 2.5 x 10^{13} cm^{-2} Fluence. (B) Ag^+ at 100 MeV, 5 x 10^{14} cm^{-2} Implantation followed by Ag^+ Irradiation at 130 MeV, 5 x 10^{13} cm^{-2} Fluence. (C) Ag^+ at 130 MeV, 5.8 x 10^{14} cm^{-2} Implantation

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5.2.9 Controlled Growth of Silicon nanocrystals in SiO_2 Matrix using MeV Ag Ion Irradiation

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Amongst various means of synthesizing nanoparticles, the swift heavy ion irradiation is found to be one of the efficient ways of generating monodispersed nanoparticles. Here we demonstrate the formation of silicon nanoparticles in a matrix of silicon oxide mainly by the phenomenon of electronic loss component (S_e). This component varies as a function of the material properties as well as the type of ions. Nanoparticles and nanowires of silicon embedded in a stable protective matrix and capable of emitting light at room temperature in visible range of wavelengths find importance[1]. This is due to the necessity of silicon based light emitting devices for optical communication since they are the only ones which are compatible with the already developed silicon based technology. In our earlier studies[2] we have shown that high energy Ni ion irradiation into silicon oxide leads to formation of nanoparticles of silicon with 2-5 nm diameter. It was shown that phase separation (Si-O to Si + O) alone is operative in these experiments. Present report emphasizes the role of varying the fluence of silver ions in the growth of nanoclusters of silicon with different sizes. The fluence dependent monodispersive phenomenon of generating controlled sizes of nanocrystalline silicon may find important applications in the optical devices since this may lead to the tunable optical emitters.

Films of silicon monoxide were deposited on p-type silicon (111) surface by vacuum evaporation at a pressure of 10^{-5} Torr. High purity Silicon monoxide powder was used as a source for the deposition of the films of SiOx on the substrates maintained at room temperature. These SiOx films were irradiated with 100 MeV and 150 MeV Ag ions at room temperature in vacuum (10^{-7} Torr). The fluence was varied between 10^{11} to 10^{13} ions/cm^2.

Amorphous SiOx films did not show any photoluminescence before irradiation. However after irradiation the films exhibited clear peaks in the photoluminescence. The

PL spectra for the samples irradiated with different fluences ranging from 1×10^{13} to 1×10^{11} ions/cm^2 were recorded. Two luminescence peaks in the visible region are observed in each of these. One peak is centered around 350 nm and the other peak around 550 nm. It is observed that the position of the peak at 350 nm is unaffected by the fluence of irradiation, however the peak at 550 nm is seen to exhibit a small red shift with increasing fluence. It is necessary to make some comments about the origin of the PL spectra in order to understand the change in the intensity and the red shift. The peak at 550 nm should be correlated to the luminescence from Si nanoclusters embedded in the matrix of SiOx. The origin of PL in such systems, lies in the generation of carriers inside the nanocrystalline silicon and recombination via interface states[3]. Hence this peak can be attributed to the one arising from the interface states assisted recombination in Si nanocrystallites. The red shift, observed in this peak with increasing fluence, can be thought of arising from the irradiation assisted growth in the sizes of silicon nanocrystals, i.e. more and more phase separation. The PL peak observed at 350 nm is known to be one originating from a defect in silicon oxide associated with radiative recombinations[4]. The results are tabulated in Table 1.

Table 1

Fluence	Peak Position
1×10^{13}	561 nm
5×10^{12}	559 nm
1×10^{12}	550 nm
5×10^{11}	548 nm

Low angle X-ray diffraction pattern was recorded for samples irradiated with different fluences. It indicates the formation of silicon nanocrystallites in the film. The size of these crystallites was estimated using Debye Scherres formula. These are tabulate in Table 2. The crystallite size is seen to increase with increasing fluence in the beginning but again reduces at higher fluences. This may be expected as a result of amorphization.

Table 2

Fluence	Sizes in nm For 100 MeV Ag ions	Sizes in nm For 150 MeV Ag ions
1×10^{13}	3.11	2.35
5×10^{12}	2.44	3.40
1×10^{12}	2.28	3.28
5×10^{11}	2.44	2.35

The morphology of the film after irradiation was studied with the help of Transmission Electron Micrograph (TEM). Nanoparticulates with elliptical shapes are seen

which have higher density and much clarity in case of the films irradiated with 150 MeV as compared to those of 100 MeV.

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5.2.10 Effect of Swift Heavy Ion Irradiation on Doped Nanoparticles of Fe_2O_3/TiO_2 for Photoelectrochemical Splitting of Water

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It is well established that irradiation of solids with energetic particles leads to creation of wide variety of defect states therein [1,2]. Damage by energetic ion beams has been well studied over the past decades. Since irradiation can lead to a controlled introduction of defect states in the material system, it has been and being beneficially used to control the material properties.

Nanocrystalline materials consist of small grains. If the grain size of nanostructured material is comparable to the diffusion length of the defects to interfaces, then all the irradiation produced defects can readily annihilate in the interfacial regions, instead of formation of defect clusters. Consequently a substantial reduction of radiation damage can be expected in nanocrystalline materials compared to solids with conventional grain sizes, assuming that interface as grain boundaries in conventional materials.

In the present work we have examined the effect of swift heavy ion irradiation on the structural and photoelectrochemical properties of doped and undoped Iron Oxide and Copper Oxide thin films prepared by Spray-Pyrolysis. Although initially the study was planned on Fe_2O_3 and TiO_2. But, considering the attractive features of Copper Oxide as a semiconductor material for PEC study, the Copper Oxide was chosen as the study material in place of TiO_2 . Effect of irradiation on the stochiometry of the thin films has also been studied by Elastic Recoil Detection Analysis (ERDA).

Thin films of Fe_2O_3 and CuO were irradiated with 170 MeV Au^{13+} ions with the fluence values of 1 x 10^{12}, 5 x 10^{12} and 1 x 10^{13} ions/cm^2 using 15 UD Pelletron accelerator at Nuclear Science Centre, New Delhi. The ion beam was made to scan over an area of 10 x 10 mm^2 using magnetic scanner in order to achieve the fluence uniformity across the sample area. For ERDA study, the film sample were kept tilted at angle of 10° with respect to the beam direction and the recoils generated by the incident ion beam were recorded.

Using Ga/In eutectic, silver paint and copper wire, ohmic electrical contact was generated, for PEC measurements, on the uncoated area of substrate, which was covered by aluminium foil during film deposition. This area was later covered by non-transparent and non-conducting epoxy-resin (Hysol, Singapore). The Photoelectrochemical measurements involved monitoring current-voltage (I-V) characteristic, under darkness and illumination, using a three electrode system in a glass cell fitted with pyrex glass window to facilitate the transmittance of light to the photoelectrode surface. The thin film electrode (working electrode, surface area of 1 cm^2) was used in conjuction with platinum and saturated calomel electrodes (SCE), which were used as counter and reference electrodes, respectively. PEC measurements were performed using a potentiostat (Model ECDA-001, Con-Serv Enterprises) and a light source (250 W-Xe Lamp, Bentham). The intensity of light was fixed at 0.16 W cm^{-2}. The electrolyte used was 1M NaOH (pH 13).

The phase identification has been studied using X-Ray Powder Diffractometer (X'PERT Model, Philips, with graphite monochromator) using Cu-Kα wavelength before and after irradiation. The XRD pattern of thin film at 1 x 10^{13} ions/cm^2 shows diffuse nature, which is indicative of presence of amorphous material. The appearance of crystalline peaks over amorphous background exhibit that the material has no change in crystal structure after irradiation. On irradiation the peak intensities are decreased and the peak width are increased, which clearly show that the point / cluster of defects are produced, causing certain amount of amorphization in the material. Some X-ray peaks shift toward higher 2 θ. One small component in this structure shows a downward shift of 2 θ. This presumably corresponds to the ion induced defect track regions. A shift toward higher 2 θ implies vertical compression or in-plane expansion.

From XRD patterns the average grain/particle size has also been determined using Scherer's equation. It has been observed that on irradiation at low fluences i.e. 1 x 10^{12} the particle / grain size has been decreased by ~30% (average) but at higher fluence the particle size increases. The relative concentration of Fe : O remains same during irradiation for fluences ranging from 7 x10^{11} to 1 x 10^{13} ions/cm^2 . We can infer from this that no depletion of oxygen is taking place during irradiation.

We have also measured the effect of irradiation on the photo-electrochemical properties of thin films. On irradiation at fluence 1 x 10^{12} ions/cm^2 a significant rise in photocurrent density can be observed. However at higher fluence (1 x 10^{13} ions/cm^2), the photocurrent density decreases. Thus on irradiation of thin films at low fluences the photocatalytic activity increases. Which might be due to point defects created which are

probably allowing the film to absorb photons more efficiently. However at higher fluence the probable fusion of particles/grains might be taking place along with the partial amorhization as has been observed in the XRD patterns. This is evident from the observed drop in photocurrent. Finally from this study, it can concluded that photocatalytic properties of material may be enhanced by the irradiation at optimized conditions.

We extend our thanks to Prof. N. C. Mishra , Dept. of Physics, Utkal University, Bhubhneshwar for the discussions. The authors also thank Dr. Umesh Tiwari, Solid State Physics Laboratory, Delhi for XRD analysis of thin films.

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5.2.11 Ion Beam Induced Modification of Lattice Strains in $In_{0.1}Ga_{0.9}As/GaAs$ system

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Strained Layer Superlattices (SLS) are layered structures of different materials having small lattice mismatch (~ 0.1 to 2.0%). This mismatch is accommodated by biaxial (compressive and tensile) strain in the layers. SLS have potential device applications for high performance detectors, high speed and high frequency digital and analog circuits. The strain accommodation in SLS is limited by the thickness of the epilayer and beyond a critical thickness of the layer, the system starts relaxing giving rise to misfit dislocations. Typical thicknesses below this critical value are about a few hundred angstroms for lattice mismatches ~1%. The presence of defects and dislocations deteriorates the performance of the devices and thus it is important to characterize the strain and strain-relieving mechanisms. The thickness and composition of the epilayer decides th bandgap of SLS, which can be tuned by ion irradiation. Swift Heavy Ion (SHI) irradiation is more suitable for this purpose because the process of ion beam mixing can be confined to a narrow region at the interface. We have carried out a systematic study of SHI irradiation on MBE grown $In_{0.9}Ga_{0.9}As/GaAs$ samples and their subsequent characterization by High Resolution XRD. The effect of ion flu-

ence on the initial strain/layer thickness has been investigated. Four samples with same composition but different InGaAs layer thickness have been studied in this work. These have been irradiated with 150 MeV Ag ions with fluences from 5 x 10^{12} to 2 x 10^{13} ions/cm^2.

In the HRXRD study, strain measurements are performed by determining the angular shift in the layer peak position with respect to that of the substrate. Figure 1 shows the HRXRD on one of the pristine and irradiated samples. The compressive strain in all these samples is found to be decreasing with increase in fluence, as shown in figure 2. Also evident from this figure is the fact that the indium composition is decreasing with fluence. This is in agreement with our previous results, where a lattice matched structure ($In_{0.53}Ga_{0.47}As/InP$) has been irradiated by 130 MeV Ag ions and studied by RBS/Channeling [1]; ion irradiation seemed to have induced a finite tensile strain in this structure.

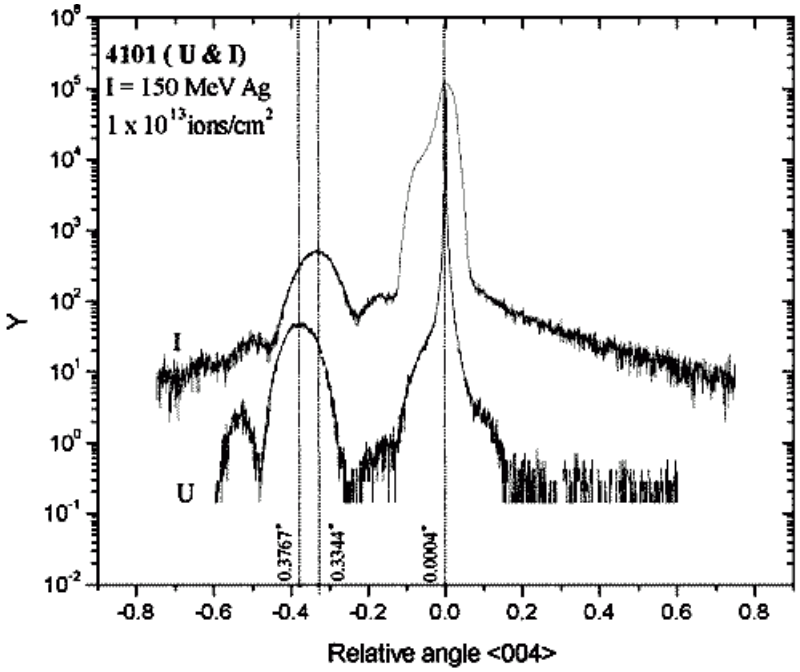

Fig. 1 : High Resolution XRD of 400 Å $In_{0.1}Ga_{0.9}As/GaAs$

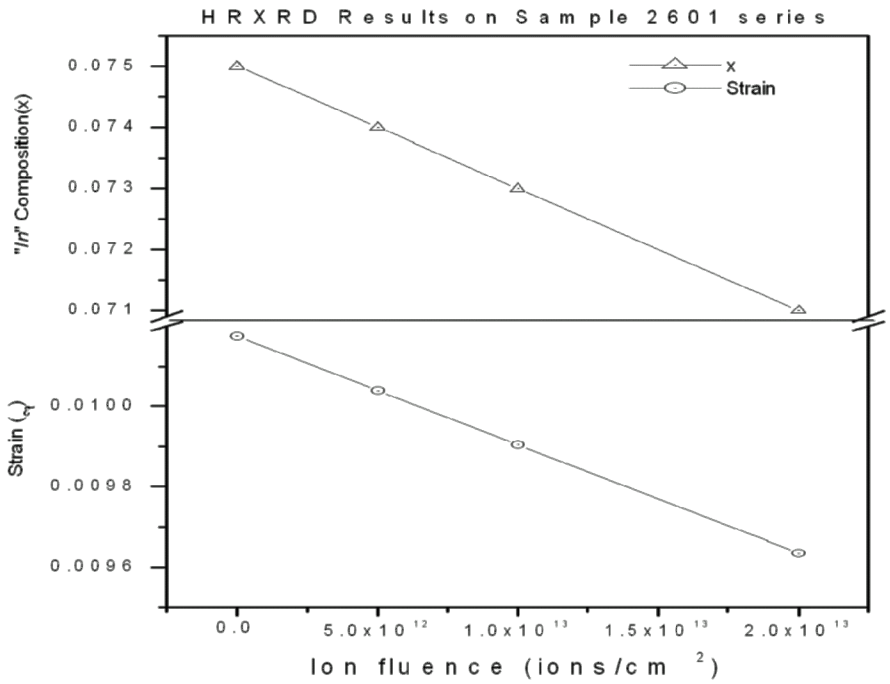

Fig. 2 : Variation of In composition and strain with fluence

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5.2.12 Swift heavy ion induced strain in a lattice matched superlattice

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Ion beam mixing experiments have been performed to introduce strain in an initially lattice matched ($In0_{.53}Ga_{0.47}As$ / InP) multilayer system. HRXRD has been employed to study these mixing effects. This experiment is a part of our series of experiments to study ion beam based methods to measure and engineer the bandgap/strain of III-V compound semiconductor multilayers for the integration of optoelectronic circuits.

Ten periods of [$In_{.53}Ga_{.47}As$(15nm) /InP(15nm)] were grown on an InP substrate using MOCVD facility at Warsaw, Poland. This sample was then cut in to three parts with one part kept unirradiated (*P523U*). The other parts were irradiated by 130 MeV Ag ions delivered from 16MV Pelletron accelerator at Nuclear Science Centre, New Delhi with two different fluences 5 x 10^{12} ions/cm^2 (*P523I*) and 5 x 10^{13} ions/cm^2 (*P523I2*). Then each of these three samples was again cut in to two parts and one part was annealed (Rapid Thermal Annealing (RTA)) at 450ºC for 90 sec. in N_2 atmosphere using the RTA facility at Dresden, Germany. Annealed samples are referred with a tag "RTA" at the end of the sample name. All these samples were characterized by HRXRD near the reflection <006> (InP) at ROBL Material Research Station, Grenoble. The wavelength of radiation was ~ 0.15 nm.

In our earlier work [1] we studied the low fluence sample (i.e P523 & P523I) using RBS/Channeling measurements. RBS spectra showed ion beam induced inter diffusion of In, Ga, As & P across the interfaces and RBS/C showed that the irradiation can induce finite tensile strain. Good reduction in RBS/C spectra of irradiated sample indicates that the sample structure was not spoiled by irradiation. However we could not determine the exact value of strains in that experiment. Here these values are measured very accurately using HRXRD. Effects of annealing (RTA) have also been studied here.

Fig. 1 shows the HRXRD spectra of low fluence samples. These spectra are displaced (Shifted upwards) on the intensity axis for clarity. The inset shows the complete interference pattern of P523U. Very high number of satellite orders in the HRXRD spectra shown in these figures, indicate that the interfaces are very sharp and the boundaries are almost rectangular in shape. Also indicated fact is that these interfaces remain considerably sharp even after irradiation. Broadening in the substrate peak is due to the implantation damage in the substrate region (about 13μ deep), which could be annealed out up to some extent by RTA. Otherwise the basic structure of the lattice is invariant under irradiation and annealing process. The reduction in the intensity of satellite peaks shows the inter-diffusion of elements across the interfaces. Superlattice period and strain values are obtained using the simple HRXRD formulae based on Bragg's law [1]. Measured strain values showed the dependence of strain on the ion fluence. For a detailed understanding we have analyzed the HRXRD data of high dose samples using a computer code RADS Mercury. The analysis suggests that the diffusion process will form a thin $In_xGa_{1-x}As_yP_{1-y}$ on the top of every layer. This effect is observed even in the pristine sample but the effect is more in irradiated and/or annealed samples. The mixing effects are more prominent for ion beam processed (high fluence) and annealed sample as expected. Simple irradiation changes the strain but the interface quality could be improved after annealing as it can be observed by the asymmetry in the substrate peak. However annealing alone doesn't change the strain value significantly. Hence it is shown that the irradiation can induce a tensile strain in an initially lattice matched system without much loss of the crystalline/interface quality of the samples.

Fig. 1 : HRXRD spectra of low dose samples zoomed around the substrate peak, maintaining the order given in label box; inset: complete interference pattern of P523U

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5.2.13 Lattice Strain Measurements using Automated High Energy Channeling Facility at NSC

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Ion channeling experiments along the off-normal (<110> for <100> growth) directions are sensitive to lattice strains because there exists a small misalignment in the channeling directions at the interface. Hence one can estimate the strain value by mapping the off normal axis using the channeling angular scans. The difference between the positions of the channeling dips obtained from layer and the substrate around the off normal axis is a measure of the lattice strain. The FWHM of such an angular scan is directly related to the channeling critical angle, which varies inversely as square root of incident energy. Hence the angular scans will be sharp in high energy channeling there by the strain resolution and the sensitivity of the method are improved. If the energy is too low then the results are mislead by steering effect due to the broad critical angles. Hence high energy channeling measurements are recommended for strain measurements. Keeping these points in view, recently we have developed an automated high energy channeling facility at NSC, New Delhi 1[1]. Here we present some recent results to show the performance of this facility. Table 1 shows the sample Identity (ID), specifications, growth technique, and the measured strain values.

40 MeV Si ions delivered from the 15 MV Pelletron accelerator of NSC have been used as incident beam in high energy channeling experiments performed using the recently developed automated high energy channeling facility at NSC. High energy channeling experiments have been performed on single strained layer samples grown at SSPL. RBS / channeling on these samples were performed using *3.5 MeV He* ions at *IOP*, Bhubaneswar (IOPB) and 4.5 *MeV He* ions at Alabama (Alabama) for comparison.

Fig. 1 shows the channeling angular scans measured along the off-normal axial direction of 4101 at NSC. A clear angular shift in the <110> angular dip obtained from the epi-layer is observed when compared to that of the substrate. Reasonably good reduction in the channeled yield has been observed from this data. It indicates the good crystalline quality of the samples on one hand and the performance of the automated high energy channeling facility on the other hand. Fig. 2 shows the similar scans measured at Alabama on the same sample. Corresponding strain values are in Table 1. Although the angular shift is same in both the experiments the shift is clearer in Fig. 1 when compared to that of Fig. 2. It is because of two reasons: one is obviously the small critical angle and the other is the good resolution of goniometer and the efficiency of the automation program. Measured strain value is given in Table 1 along with the result of high energy channeling experiment performed at NSC. In this case the sample has been exposed to radiation for considerably long time during the alignment process and hence the measured strain value is less than that of the low energy result. In this connection it is to note that the channeling measurements have been performed on fresh spot in the case of 4101.

Fig. 1 : Channeling angular scans around the <110> axis of 4101 obtained using the automated high energy channeling facility at NSC (Beam: 40 MeV Si)

Table 1 : Sample specifications and measured strain values

Sample ID	Specification (Grown at SSPL using MBE)	Lattice Strain Measurements	
		40 MeV Si Channeling at NSC t	Low energy He Channeling t
2601	In 0.1Ga0.9As(250 A0)/ GaAs	0.4 (On irradiated spot)	0.503 (at IOPB)
4101	In 0.1Ga0.9As(400 A0)/ GaAs	0.56 (On fresh spot)	0.56154 (Alabama)

Fig. 2 : Channeling angular scans measured around both the normal (<100>) and off-normal (<110>) axial directions of 4101U at Alabama

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5.2.14 Investigations on High energy Sn Irradiation induced effects in GaN

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GaN is a wide band III-V compound semiconductor and has been used to produce blue light emitting diodes, lasers as well as high temperature and high power devices [1]. GaN based devices will have to operate in a radiation environment, and so it is of fundamental importance to achieve a detailed knowledge of radiation induced defects which have often been shown to affect the electrical and optical priorities of semiconductor materials [2]. In the present investigation GaN epitaxial films were grown by MOCVD with (0001) orientation and thickness in the range of 2-3 μm on 420 μm thick C-oriented sapphire substrates. The GaN was unintentionally doped n-type with a carrier density of $4x10^{16}$ cm^{-2} as determined by conductivity and hall effect measurements. The samples were degreased with organic solvents and etched with NH_4OH : H_2O (1:1) for 10 seconds to remove the native oxides. The samples were irradiated at room temperature with Tin (Sn) ions at 75 MeV to fluences of 10^{11}, $5x10^{11,}$ 10^{12}, 5 x 10^{12}, 10 ^{13}, 5x 10^{13} and 10^{14} using 15 UD accelerator. Using the Monte Carlo simulation program

TRIM 95, it was found that the projected range of the 75 MeV Sn in GaN is 13055 nm and straggling is 1090 nm. The induced effect of high-energy irradiation in GaN epitaxial layers have been analysed by Raman scattering. The behaviour of Raman shift, FWHM and area of GaN modes with Sn irradiation for E_2 (high) and A_1 (LO) Raman modes of GaN layer have been observed [3]. New peaks have been observed at fluences 10^{12}, 5 x 10^{12}, 5 x 10^{13}, 10^{14} cm^{-2}. Optical absorption spectra have also been recorded in which it was found that the band gap value gradually decreases from 3.4eV to 2.9eV with increase in irradiation fluence. X-ray rocking curve (XRC), Atomic force Microscopy (AFM) and Scanning Electron Microscopy (SEM) analysis have been carried out on as grown and Sn irradiated GaN samples. The FWHM of (0002) plane rocking curve increases with increase of ion fluence. The increase in FWHM indicates the degradation of the crystalline quality of the GaN epitaxial layers due to high-energy irradiation. The irradiation induced modification of the surface roughness (R_{ms}) was studied with AFM and it was found that there was an decrease in R_{ms} value with increase of ion dose. It is clear that Sn ion irradiation makes the GaN surface relatively rough when compared to as-grown one. SEM photograph shows the interface damage of the GaN epitaxial layers due to irradiation effects.

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5.2.15 Irradiation effect on dielectric properties of $NiMn_{0.05}Ti_x(Zn^{2+}, Mg^{2+})_xFe_{1.95-2x}O_4$ ferrites thin films

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Swift heavy ion (SHI) induced modifications on dielectric properties of $NiMn_{0.05-}Ti_x(Zn^{2+},Mg^{2+})_xFe_{1.95-2x}O_4$ for x = 0.0, 0.2 thin films were studied. Thin films of $NiMn_{0.05-}Ti_x(Zn^{2+},Mg^{2+})_xFe_{1.95-2x}O_4$ for x = 0.0, 0.2 were grown on Si substrate using RF magnetron sputtering. X-ray diffraction patterns of all the films are well fitted with spinel structure of these ferrite materials and matches well with the bulk target materials. The dielectric measurement of the prepared ferrite films was made using HP 4284A LCR meter as a function of frequency from 1kHz to 1MHz. The well characterized thin films were irradiated with 190 MeV Ag ion for three different fluence values $5x10^{10}$, $5x10^{11}$ and $1x10^{12}$ ions/cm^2 using 15UD Pelletron at Nuclear science Centre, New Delhi. The dielectric measurements were performed on the irradiated films for the comparative studies on irradiated and unirradiated films. The dielectric constant for pristine films decreases with the increase in the frequency

of the applied field, which is in well agreement with Koops model. It is observed that even-though the dispersion in the dielectric constant ε with the frequency is same as the pristine sample the ε decreases with fluence as compared to the pristine film. This decrease in dielectric constant can be explained on the basis of the decrease in the number of ferrous sites, which are available for polarization and the flow of space charge carriers which could be correlated to the production of the defects created in the films due to the irradiation. For 20% Zn substituted film, $NiMn_{0.05}Ti_{0.2}Zn_{0.2}Fe_{1.75}O_4$, again the decrease in ε with frequency can be well explained by Koop's theory. However, for 190 MeV Ag ion irradiated thin films an anomalous behavior is observed as compared to the unirradiated film. The dielectric constant ε shows an increase in magnitude with increase in the ion fluence below 20kHz, whereas above 20kHz, it decreases from that of the unirradiated film. Further study is going on to understand this anomalous behavior.

All the films show an increase in the dielectric loss tanδ with increase in the fluence. It is observed that after irradiation ε' decreases and tanδ increases with the frequency which implies that the imaginary part ε'' of the dielectric constant increases on irradiation of the films. It is also observed from the figure that all the films show a resonance peak in the dielectric loss curve, which was not present in the unirradiated films. Also it has been found that the resonance peak shift towards the lower frequency with increase in the fluence.

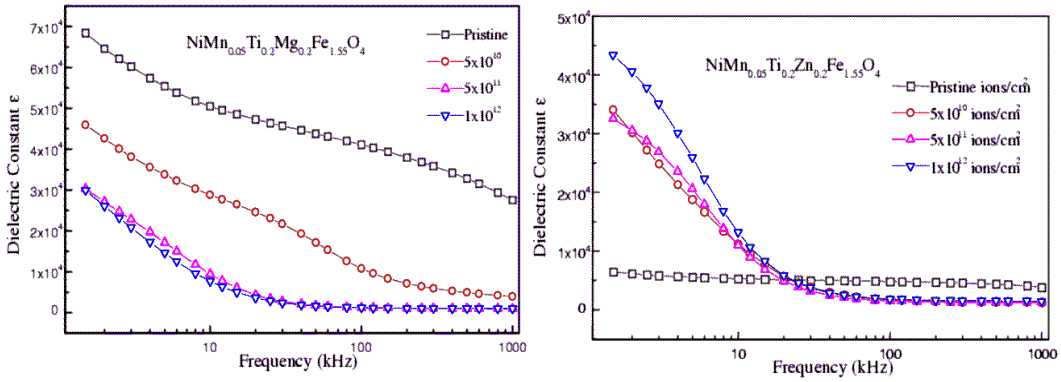

Fig. 1 : Variation of dielectric constant ε with frequency in $NiMn_{0.05}Ti_{0.2}(Zn^{2+},Mg^{2+})_{0.2}Fe_{1.95-2x}O_4$ for different fluences

Fig. 2 : Variation of dielectric loss tanδ with frequency in $NiMn_{0.05}Ti_{0.2}(Zn^{2+},Mg^{2+})_{0.2}Fe_{1.95-2x}O_4$ for different fluences

5.2.16 Mössbauer studies of 190 MeV Ag ion irradiated $NiMn_{0.05}Ti_xMg_xFe_{1.95-2x}O_4$ ferrite

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A series of samples of $NiMn_{0.05}Ti_xMg_xFe_{1.95-2x}O_4$ for x = 0.0, 0.2 were prepared by conventional solid state technique. The spinel cubic structure of this ferrite was confirmed by Powder X- Ray Diffraction technique. The Mössbauer absorbers were made using fine powder of the $NiMn_{0.05}Fe_{1.95}O_4$ and $NiMn_{0.05}Ti_{0.2}Mg_{0.2}Fe_{1.55}O_4$ with thickness of 20mg/cm^2. The Mössbauer absorbers were irradiated with a 190 MeV Ag ion beam with different fluence values in the range of 1×10^{11} to 1×10^{13} ions/cm^2 using the 15UD Pelletron Accelerator at NSC, New Delhi. The ^{57}Fe Mössbauer studies at room temperature has been performed on unirradiated and irradiated absorbers using a multichannel analyzer with constant acceleration drive system with ^{57}Co source in a palladium matrix. The Mössbauer spectra were analyzed by least square fitting NORMOS/SITE program developed by R.A. Brand.

All the Mössbauer spectra were well fitted with the two sextets for two occupancy sites of Fe in the spinel structure and the paramagnetic doublet. The Mössbauer spectra for unirradiated $NiMn_{0.05}Fe_{1.95}O_4$ and $NiMn_{0.05}Ti_{0.2}Mg_{0.2}Fe_{1.55}O_4$ is shown in Fig.1. It is observed that with the substitution of Ti^{4+} and Mg^{2+} in $NiMn_{0.05}Fe_{1.95}O_4$, the hyperfine field decrease which can be understood from Neel's molecular field theory. The occupancy ratio, B/A, of Fe^{3+} at tetrahedral and octahedral site decreases and isomer shift increase with substitution. The spectra for 190 MeV Ag ion irradiated $NiMn_{0.05}Fe_{1.95}O_4$ and $NiMn_{0.05}Ti_{0.2}Mg_{0.2}Fe_{1.55}O_4$ were also analyzed. The recorded Mössbauer spectrum for the pristine and the irradiated samples of $NiMn_{0.05}Ti_{0.2}Mg_{0.2}Fe_{1.55}O_4$ is shown in Fig.2. For both the series of samples no significant changes in hyperfine field, isomer-shift and the quadruple splitting values were observed, whereas the broadening of sextet is being observed with fluence. Also an increase in the isomer shift and the quadruple splitting value for the paramagnetic doublet is found to increase with the increase in the fluence. The peak widths are getting broadened with increase in ion fluence, and the paramagnetic contribution observed as doublet in the spectra is increasing which may be attributed to the increase in the amorphization and disorder induced in the material by the SHI irradiation. From the comparative studies of the pure and substituted systems on irradiation it is being concluded that the effect of the irradiation is similar on both the ferrite systems. The paramagnetized volume for the substituted ferrite is increased faster with the fluence than that of the pure Ni ferrite.

Fig. 1 : Mössbauer spectra for unirradiated samples of $NiMn_{0.05}Fe_{1.95}O_4$ and $NiMn_{0.05}Ti_{0.2}Mg_{0.2}Fe_{1.55}O_4$

Fig. 2 : Mössbauer spectra for unirradiated and 190 MeV irradiated $NiMn_{0.05}Ti_{0.2}Mg_{0.2}Fe_{1.55}O_4$.

5.2.17 Characterization of Zn and Mg Substituted Nickel Ferrite Thin Film using Elastic Recoil Detection Analysis

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Characterization of Zn and Mg substituted Ni ferrite thin films i.e. $NiMn_{0.05}Ti_{0.2}Mg_{0.2}Fe_{1.55}O_4$ and $NiMn_{0.05}Ti_{0.2}Zn_{0.2}Fe_{1.55}O_4$ was done using online elastic recoil detection analysis (ERDA) at the 16 UD Pelletron accelerators at NSC. The films were grown by RF magnetron sputtering of a bulk target of the desired stoichiometry, viz., $NiMn_{0.05}Ti_xA_xFe_{1.95-2x}O_4$ for A=Zn and Mg, on Si (100) substrate in argon atmosphere. The substrate temperature was kept at 650°C. To compensate the loss of oxygen during deposition, if any, the films were annealed in-situ in oxygen partial pressure of 200 torr at 700 °C. X-ray diffraction patterns of the as deposited films reveal the spinel structure of the films. However, our main emphasis is to see whether films retain the stoichiometry of the target material. To understand this we have performed ERDA studies of these films using 200 MeV Ag ion beam. Typical two-dimensional recoil spectrum for $NiMn_{0.05}Ti_{0.2}Zn_{0.2}Fe_{1.55}O_4$ is shown in Fig. 1. The Ni/Zn and Mn/Ti bands could not be separated from the present data.

From the analysis of our experimental results, we have observed that the films retain the proper stoichiometry as that of the target. From the stoichiometry of the film, the ratio N_{Fe} / N_O is found to be 0.387 which resemble the values obtained for both the irradiated samples with different fluences. These results indicate that the oxygen con-

tent and the film stoichiometry remain unchanged even upto the fluence of 1.3 x 10^{13} ions/cm^2.

5.2.18 Oxygen Content Meaurement and Compositional Analysis of 190 MeV Au^{14+} Ions Irradiated Li-Mg Ferrite Thin Films

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Ferrite thin films are gradually gaining their interest due to their application in recording media. Presently we have chosen $Li_{0.25}Mg_{0.5}Mn_{0.1}Fe_{2.15}O_4$ system, which is very well known in the microwave application. We aimed to study the changes in oxygen content and compositions of the elements in the film due to swift heavy ion irradiation. The ion energy used in this experiment is nearly 1MeV/amu which compels the ions to lose their energy by inelastic collisions with electrons. Thus as an effect of thermal spike model, the excess heat generated during irradiation can decrease the oxygen content of the film. This effect is of interest as for nanoparticles the heat conduction is not the same as that of bulk. To observe the above facts we have done online ERDA.

Nanocrystalline thin films of $Li_{0.25}Mg_{0.5}Mn_{0.1}Fe_{2.15}O_4$ were deposited on Si (100) substrate using high pressure R.F. magnetron sputtering technique at TIFR. After deposition annealing is done in oxygen atmosphere at 700°C and 800°C for 3 hours. From XRD pattern the crystallite size was calculated following Debye-Scherrer equation.

These films were mounted in a ladder inside the Goniometer chamber at NSC materials science beam line. Goniometer movement was used to bring the sample along the beam direction one by one. Different fluences were given to the films and online ERDA was performed. The beam spot was confined to approximately 1 x 1 mm^2 by the use of two double slits located before the chamber. A large angle position sensitive detector telescope (LAPSDT) was used. A collimated beam of 190 MeV Au ions was incident on the ferrite films. The detector with the entrance window, made up of 1.5 μm polypropylene foil in front of it, was placed at an angle of 45° with respect to the beam.

From the recoil spectrum, we observe the recoils of all the elements of the film like Li, Mg, O, Mn-Fe, Si. The elements Mn (Z=25) and Fe (Z=26) could not be separated from the present experiment. From the initial concentration of the elements of the sintered disc, used during R.F. magnetron sputtering, we know that the concentration of Mn is 0.1 and of Fe is 2.15. For each film the concentrations are determined in the identical way. The ratios of N_{Mg}/N_O, N_{Fe}/N_O and N_{Li}/N_O are determined with increasing fluence. Using the following relation we have done the quantitative determination of the elements

$$\frac{N_x}{N_O} = \left[\frac{Y_x}{Y_O}\right]\left[\frac{\sigma_O}{\sigma_x}\right]$$

x = Fe, Mg, Li ;
N = the number of atoms/cm^2 ;
Y = area under recoil spectrum to a fixed depth ;
σ = Rutherford recoil cross section.

The ratios obtained are given in the following table:

Films	$\frac{N_{Mg}}{N_O}$	$\frac{N_{Fe}}{N_O}$	$\frac{N_{Li}}{N_O}$
Ideal ratio	0.125	0.5375	0.0625
Annealed at 700°C	0.106±0.003	0.474±0.009	0.058±0.002
Annealed at 800°C	0.088±0.002	0.414±0.007	0.025±0.002

The oxygen content is calculated from the 2D recoil spectrum by taking the ratio of the areas of O and of Fe recoils. The relative counts (with standard deviation and error bar) are more or less constant with the fluence.

The experimental data and results show that the elemental concentrations remain nearly same with the fluence. Similar graph is observed for oxygen content of the films, during irradiation, with fluence. Oxygen concentration in the film annealed at 800° C is less as compared to the films annealed at 700° C. This difference may be due to the crystal growth process of the film annealed at 700°C during irradiation. Thus for the film annealed at 800°C some oxygen evaporation may be taking place.

5.2.19 In-situ Resistivity Measurement of Nanoferrite Li-Mg films Irradiated by 190 MeV Au^{14+} Ions

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To probe the swift heavy ion induced modifications in the $Li_{0.25}Mg_{0.5}Mn_{0.1}Fe_{2.15}O_4$ films, irradiated by 190 MeV Au^{14+}, an in-situ measurement of resistivity is carried out using two-probe method. The importance of this experiment lies in the fact that the three films of three different particle sizes are used here. For the three cases a variation in the resistivity with fluence is observed. The variation in the particle size is obtained with dif-

ferent annealing temperatures (700ºC, 800ºC and 900ºC) and the measurement is done from XRD peaks.

The film annealed at 900ºC has highest resistivity among the three films. This film when irradiated at 5×10^{11} ions/cm^2 shows a decrease in its value from 1.5×10^8 Ωcm to 2.5×10^6 Ωcm. Then the resistivity gradually decreases with increase in fluence. A similar decrease is also observed in the other films. But their natures are not exactly same as this film. Fig. 1 shows the nature of the resistivity with fluence. Our experimental results reveal the modifications in the electrical properties of the films. The grain size of these films being very small the SHI induced modifications are not exactly same as bulk. The basic condition used here is $S_e>S_{eth}$, which increases the probability of formation of amorphized latent tracks [1,2]. These latent tracks may generate shock waves, which may create permanent disorder in the system, which consequently decreases the resistivity. This decrease in resistivity may also occur due to the confinement of heat in the small volume of nanoparticles [3]. The confined heat is helping the electrons to move towards the conduction band by increasing their energy. These electrons may lose their energy in the localized stable bands below the conduction band. One more possibility is the bursting of the larger grains to finer grains. From the XRD spectrum we see broad peaks, which account for the above phenomenon. The XRD spectra also prove that the crystalline film has not changed to amorphous nature. Bursting may consequently migrate the high angle boundaries, which increases the overall conductivity of the system. The research work is still going on to explain all the curves in the fig. Thus our experimental results prove that nanoparticles are not radiation resistant.

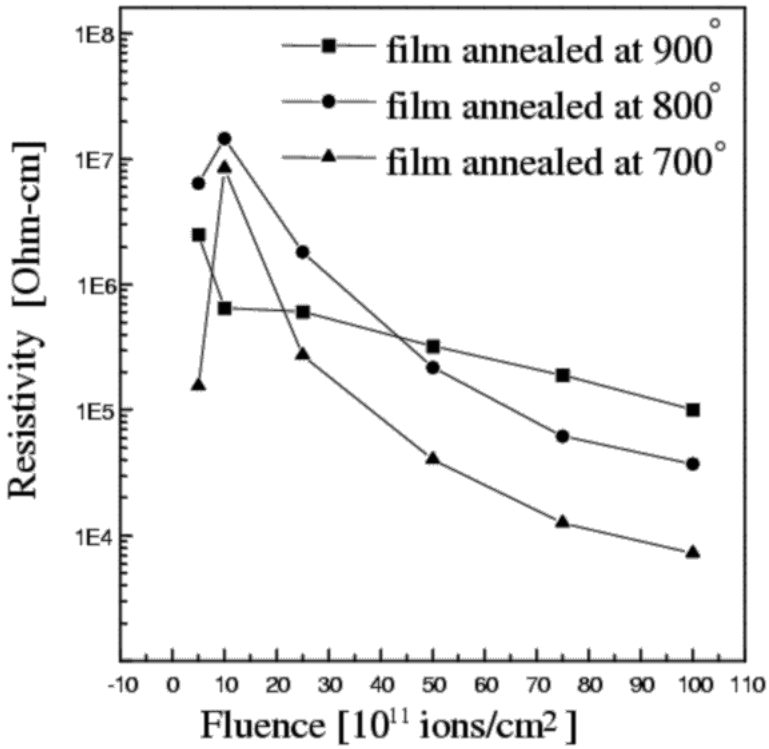

Fig. 1 : Resistivity vs fluencc when irradiated with 190 MeV Au^{14+} ions

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5.2.20 Mössbauer Studies on Irradiated Nanosized $Mg_{1.0}Mn_{0.1}Fe_{1.9-x}In_xO_4$ (x = 0, 0.3, 0.6, 0.9)

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Importance of nanoparticles lies in their fundamental properties like small volume, high surface to volume ratio, grain boundaries comparable to grains etc. Some works are already been done on Mössbauer studies on ferrite nanoparticles. The Mössbauer studies reflect the changes in magnetic properties, chemical environment and symmetry of the system studied. Here we have taken $Mg_{1.0}Mn_{0.1}Fe_{1.9-x}In_xO_4$ (x = 0, 0.3, 0.6, 0.9) nanoparticles which has already given the proof of its difference from bulk, which is out of the scope of this report. Presently we are interested to see the swift heavy ion induced modifications in these nanoparticles.

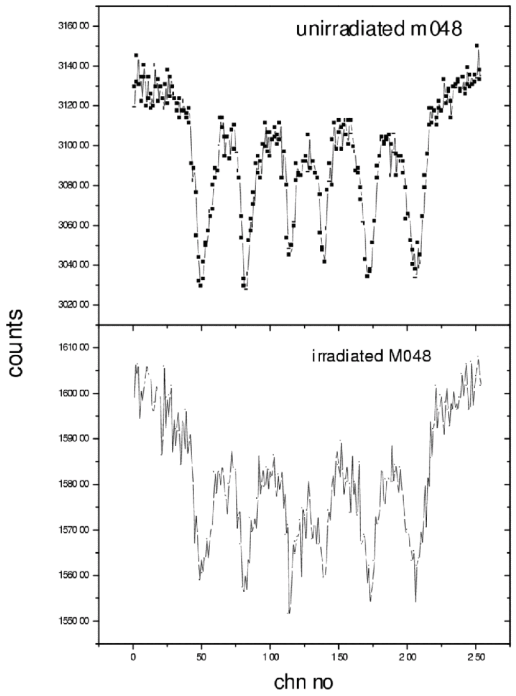

Fig. 1 : Mossbauer spectra of M048 irradiated with a fluence of 2.5 x 10^{12} ions/cm^2

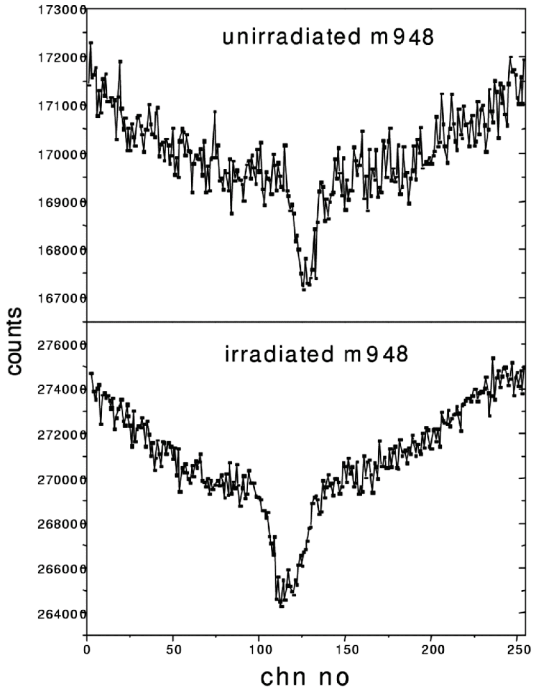

Fig. 2 : Mossbauer spectra of M948 irradiated with a fluence of 2.5 x 10^{12} ions/c

The sample preparation is done by standard ceramic technique and finally ball-milled for 22 hrs and 48 hrs to get nano order size. From XRD pattern the average grain size calculation is done using Debye Scherrer method. These powdered samples are irradiated with 190 MeV Au at the fluence of 2.5×10^{12} ions/cm^2. Mössbauer studies on this samples are done using a 5 mCi Co^{57} source placed in rhodium matrix.

In each case a change in the spectrum is observed. Here we present the spectra of two samples at x = 0 and x = 0.9. The detailed analysis and fitting are still going on but by comparing the spectra we can observe a difference in pristine and irradiated sample. For x = 0 (M048), the spectra is much more broadened after irradiation. This represents the splitting of the two sextets of A and B sites. So definitely there is a change in the hyperfine field of A site. The difference in peak intensities represents a rotation in the internal magnetic field that may take place due to irradiation at this fluence.

For x = 0.9 (M948), a much broadened doublet is obtained after irradiation. The position of the doublet shows an increase in the value of isomer shift and quadrupole splitting. This explains that for this sample the environment is much more affected due to irradiation. The irradiated spectrum is showing a tendency towards magnetic sextet with very less intensity.

5.2.21 Conversion Electron Mössbauer Spectroscopy Studies of Irradiated Li-Mg Ferrite Thin Film

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Many research works have been done to observe the swift heavy ion induced defects in insulators like garnets and ferrites [1,2,3]. In most of the cases the mechanism used to observe the magnetic effects is Mössbauer technique. This helps in reflecting the effects of heavy ions like rotation of magnetic field, track formation and induced magnetic properties. Zinc ferrite is a well-known ferrite showing the induced magnetic properties due to heavy ion irradiation. In the present case the film we have used is $Li_{0.25}Mg_{0.5}Mn_{0.1}Fe_{2.15}O_4$. Conversion Electron Mössbauer spectroscopy is an effective technique to observe the Fe environment for ferrite thin films. This film when irradiated with 190 MeV Au^{14+} at the fluence of 2.5×10^{12}- ions/cm^2 shows a transformation from magnetic sextet to paramagnetic doublet. This change is not a common behavior that has been observed till now. As it is mentioned earlier also that with 0.97 MeV/amu the tendency of the ions remain high to form the tracks so in this case also the track formation may be a reason. Previously for powdered $MgFe_2O_4$ [1] a rotation of internal magnetic field is observed with the same fluence. But in the present case the exact analysis is still going on. Fig. 1 below shows the difference observed in both the cases.

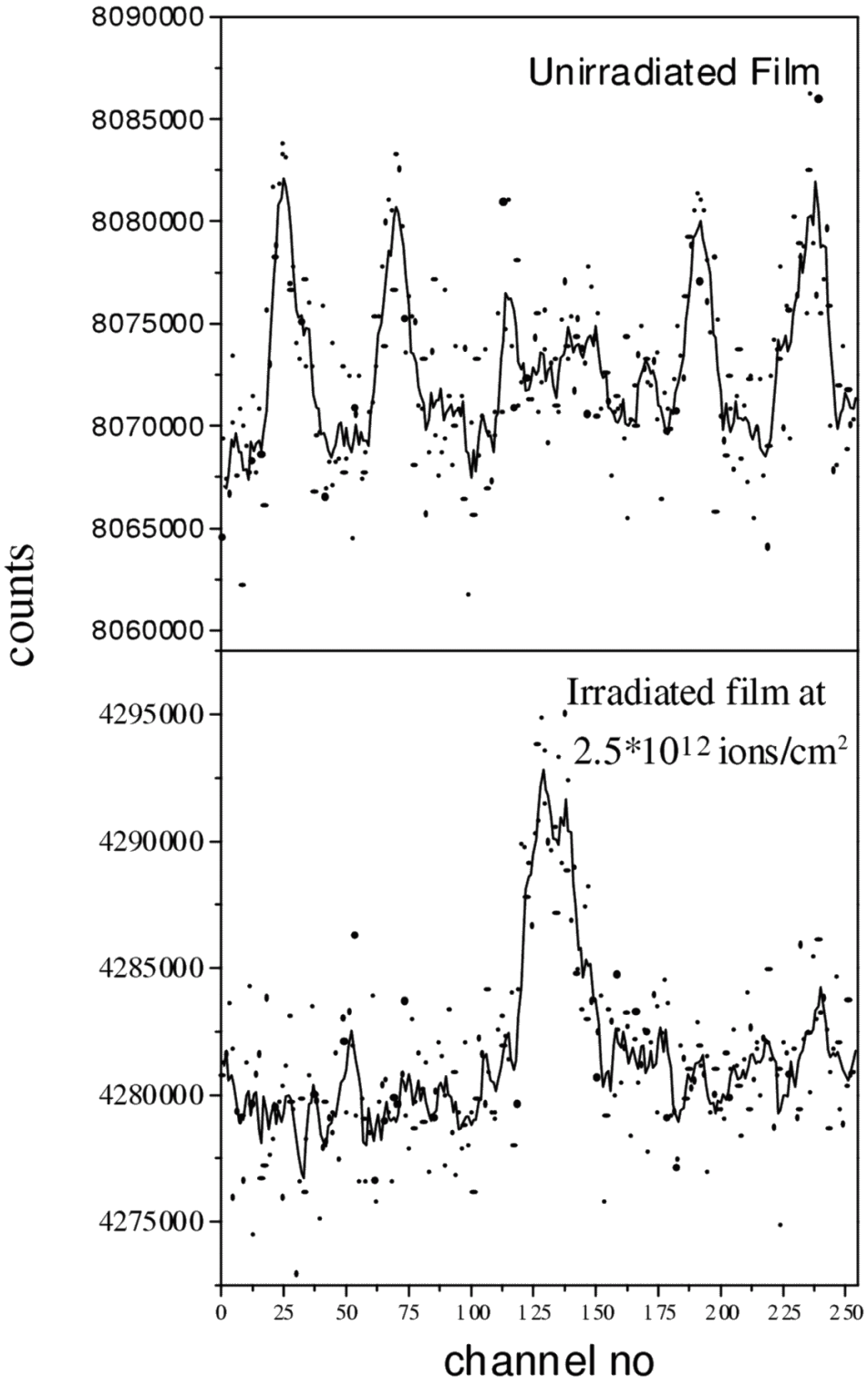

Fig. 1 : Conversion Electron Mössbauer Spectroscopy showing swift heavy ion induced defects in thin films

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5.2.22 Study of Swift Heavy Ion Beam Irradiation Effects On Oxygen Stoichiometry Of La-2125 Type Superconducting Thin Films By Elastic Recoil Detection Analysis

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$La_{2-x}Dy_xCa_yBa_2Cu_{4+y}O_z$, y = 2x; (La-2125) is a tetragonal mixed oxide superconducting system with P4 / mmm space group, showing maximum T_c ~ 75 K for x=0.5 [1]. Properties of mixed oxide superconductors are highly sensitive to oxygen content and ordering [2], hence it is very interesting to study the oxygen stoichiometric changes, if any, taking place due to irradiation of SHI on La–2125 compounds. Elastic Recoil Detection Analysis (ERDA) is a reliable technique to study the material composition by detecting recoiling elements in forward direction during irradiation of ions [3].

Two films of $La_{1.5}Dy_{0.5}Ca_1Ba_2Cu_5O_z$ (film1) and $La_{1.7}Dy_{0.3}Ca_{0.6}Ba_2Cu_{4.6}O_z$ (film2), deposited on $LaAlO_3$ single crystal substrates using Pulsed Laser Deposition technique, were irradiated by 200 MeV Ag^{+15} ions with tilt angle of 30°. The vacuum in the experimental chamber was ~ 10^{-6} mbar. The recoils from the films were detected in Large Area Position Sensitive Detector Telescope at an angle of 55° with the beam line. The pressure of Isobutene in the detector was kept around 21 mbar. The data was recorded, event-by-event, in list mode in computer using 'Freedom' software so that the fluence dependent study can be performed off-line.

The atoms from the films as well as from the substrates are recoiled during irradiation with Ag ions. Here, oxygen is recoiled from the films and also from the substrates. It is necessary to identify the oxygen atoms recoiled from the films to determine oxygen stoichiometry. The spectra of recoiled oxygen due to only thin films are identified by depth profiling using SRIM98 software. After ion fluence ~ 10^{13} to 10^{14} ions / cm^2, the ratio of O to Ca recoils were 11.3 ± 0.89 and 18.47 ± 1.75 in film 1 and film 2, respectively, with acceptable statistical errors [Table 1]. These ratios are comparable to the ratio of oxygen and calcium contents in stoichiometry of respective films which

proves that there is no change in oxygen content is occurring due to irradiation. To confirm the results of constant oxygen content in films after irradiation, counts of Al recoils from single crystal substrates were chosen as second relative reference to see the change in oxygen stoichiometry. The calculated ratio of oxygen recoils of films to Al recoils of substrates is verified to be nearly constant with acceptable statistical error after every event of irradiation at the same spot.

The ERDA results show that the oxygen content in the La-2125 type films matches with the oxygen in stoichiometry of the samples and confirms good quality of the oxide films. The ratio of counts of oxygen to aluminum recoils of the films is similar at different fluences within statistical errors. It clearly shows that, within statistical errors, the oxygen stoichiometry in film1 do not change due to 200 MeV Ag ions irradiation dose ~ 10^{12} ions/cm^2. In case of film2, ratio of counts of O to Ca recoils show that the same oxygen stoichiometry is maintained after the ion fluence of ~10^{13} ions/cm^2 and ratio of O to Al recoils shows constant oxygen content within statistical errors but needs more investigations and experiment to obtain accurate results.

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5.2.23 Low temperature resistivity study in 50 MeV Li^{3+} irradiated $La_{0.7}Pb_{0.3}MnO_3$

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$La_{0.7}Pb_{0.3}MnO_3$ sample has been prepared by usual solid state reaction technique. High-purity (each of purity 99.99 %) La_2O_3, PbO and MnO_2 with appropriate stoichiometric proportions were mixed and ground. The well-mixed oxides were then pre-heated, reground, sintered and annealed at appropriate condition step by step.

Three pellets of this sample have been irradiated with three different fluences (6.5×10^{13}, 1.5×10^{14} and 6.5×10^{14} ions/cm^2) by 50 MeV Li^{3+} beam at 15UD Pelletron accelerator of Nuclear Science Centre(NSC), New Delhi. Detailed SRIM calculation [1] has been done to optimize beam energy and the thickness of the pellets. The 50 MeV Li ion irradiation creates the point/clusters of defects.

The resistivity of all the samples (one unirradiated and three irradiated samples of different fluences) has been measured by the conventional four-probe technique in the temperature range 80K-400K in presence of magnetic field varying from 0 to 1.5 T.

The details of radiation damage effect on this system above Curie temperature has been given in the proceedings of ICNTS 21 held at New Delhi last year. Presently studies on irradiation induced changes in transport properties of this system are going on. There exist a few theoretical models that predict the temperature dependence of resistivity in low temperature regime. In the present work our investigation reveals that the low temperature resistivity data is best fitted by the equation $\rho = \rho_0 + \rho_{2.5} T^{2.5}$ for all the samples and in absence and presence of magnetic field. Here ρ_0 is the resistivity originating from domain, grain boundary and other temperature independent scattering phenomena, $\rho_2 T^2$ term corresponds to the electron-electron scattering process [2], $\rho_{2.5} T^{2.5}$ is the contribution of electron-magnon scattering mechanism [2]. In unirradiated sample Fig. 1 clearly shows the dominating feature of electron-magnon scattering ($\rho_{2.5} T^{2.5}$) over electron-phonon scattering ($\rho_{4.5} T^{4.5}$) process [3]. Table 1 shows the values ρ_0 and $\rho_{2.5}$ of obtained from ρ vs. $T^{2.5}$ plotting. Further studies are going on for the better understanding of the influence of radiation damage on different scattering mechanisms governing its resistivity.

Table 1 : Resistivity of $La_{0.7}Pb_{0.3}MnO_3$ at different magnetic fields

Fluence (ions/cm^2)	Below T_{im}			
	ρ_0 (Ohm-mm)		$\rho_{2.5}$ (Ohm-mm K$^{-2.5}$)	
	0 T	1.5 T	0 T	1.5 T
0	2.25	1.85	2.57 10^{-6}	2.75 10^{-6}
6.5 x 10^{13}	0.37	0.29	0.52 10^{-6}	0.55 10^{-6}
1.5 x 10^{14}	1.42	1.12	2.30 10^{-6}	2.37 10^{-6}
6.5 x 10^{14}	2.95	2.19	4.84 10^{-6}	5.47 10^{-6}

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5.2.24 Effect of 100MeV Oxygen Ion Irradiation on Hole Doped CMR Material

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Energetic ions passing through solid trigger a wide range of process and cause permanent changes, which are particularly interesting to both solid-state physicists and material scientists. It is well known that irradiation of solids with energetic particles leads to creation of a variety of defect states therein. The defect states and a grain boundary have dramatic consequence for the magneto-transport in manganites with an advanced feature of enhancement in low field CMR. In this experiment we are trying to study the effect of swift heavy ion irradiation effect on $La_{0.7}Ca_{0.3}MnO_3$ system.

Thin films of $La_{0.7}Ca_{0.3}MnO_3$ for SHI irradiation were prepared by pulsed laser deposition technique. KrF pulsed excimer laser with wavelength of 248 nm and pulse width of 20 nsec was used for the deposition. Energy density of 2 J/cm^2 and repetition rate of 10 Hz was maintained during the deposition. Films were deposited on single crystal $LaAlO_2$ (100) substrate, which was maintained at 650^{O}C during deposition. Also the oxygen pressure of 200mT was maintained during deposition and film was slow cooled in oxygen atmosphere. Thickness of the films was around 3000 A^O. The films were characterized by XRD and then four-probe resistivity measurement was done at low temperature. These characterized films were used for SHI irradiation with 100 MeV oxygen ion with fluence values of 10^{11}, 10^{12}, 10^{13} and 10^{14} ions/cm^2. The irradiation was carried at Nuclear Science Center, New Delhi.

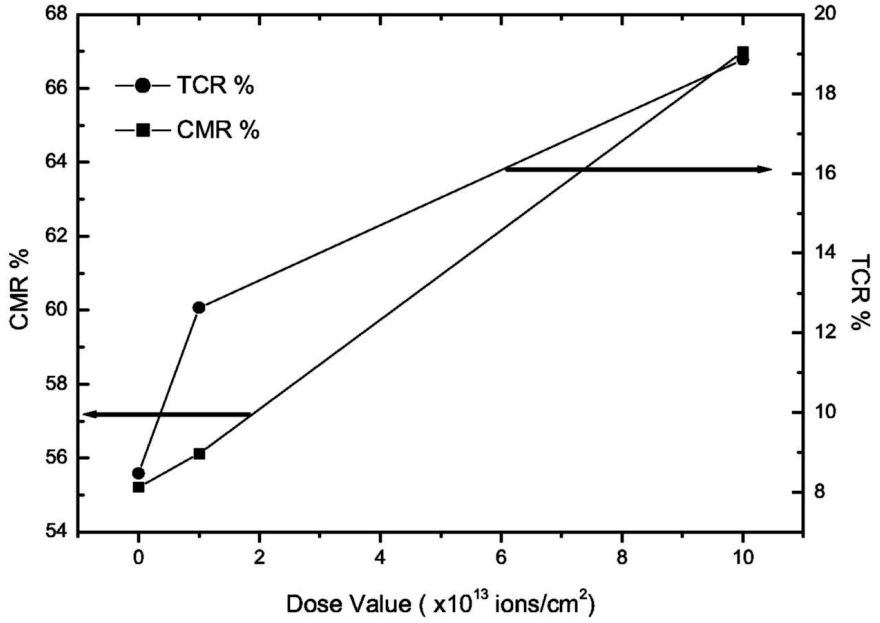

Fig. 1 : CMR and TCR values for irradiated samples of $La_{0.7}Ca_{0.3}MnO_3$

After irradiation these films were characterized by XRD and low temperature four probe resistivity measurement. The resistivity is measured in presence of 1 T magnetic field and in absence of magnetic field. Colossal Magnetoresistance (CMR %) and Temperature Coefficient of Resistance (TCR %) are calculated using these measurements.

The maximum CMR and TCR values are shown in Fig. 1. We are in a process of further investigations. We will also be doing the study of morphological changes using AFM.

5.2.25 Effect of 50 MeV Li^{3+} Ion Induced Irradiation on Single Crystals of Hexagonal Ferrite and Rare Earth Orthoferrite

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Hexagonal ferrite and rare earth orthoferrite belong to a family of magnetic material with rich array of magnetic properties [1]. M- type Hexaferrite of the type (Ba/ Sr) $(Fe/ Ga/ Al)_{12}O_{19}$ have shown promising results for use in integrated mm-wave devices due to their large magneto crystalline anisotropy [2]. Hexagonal ferrites ($SrFe_{12}O_{19}$ or $BaFe_{12}O_{19}$) are widely known as technical materials having applications in a number of electronic and / or magnetic devices. Whereas rare earth orthoferrite possess extremely high velocities of the domain wall motion and it is expected that they find applications in communication techniques, in optical internet, in sensors of magnetic fields and electrical currents, mechanical quantities etc.

Swift Heavy Ions (SHI) irradiation induced modification in materials have been widely studied and the study of irradiation induced effects become important because of their application in material technology. When energetic ion penetrates through the condensed medium it loses its kinetic energy in two modes : (i) Nuclear and (ii) Electronic stopping. The electronic energy loss is dominant at high energy (MeV) and are responsible for reversible and irreversible excitation in the electronic subsystem of the material near the surface region which may increase the mobility of defect forming atoms near the surface. In order to study the irradiation induced defects in bulk sample, uniform irradiation distribution of the projectile is required. Since monoenergetic irradiation induced effects provide the information of irradiation from a particular site at which the irradiation takes place. Keeping it in mind the concept of beam moderator for uniform distribution of projectile ion is developed. The beam moderator is able to provide uniform irradiation throughout the bulk sample.

The materials of hexagonal ferrites in the form of pure $SrFe_{12}O_{19}$ and substituted by Ga-In ($SrGa_xIn_yFe_{12-(x+y)}$ where x = 5, 7, 9 and y = 0.8, 1.3 and 1) and rare earth orthoferrites ($REFeO_3$ where RE = Ho, Dy, Er, Y, Yb) in the form of single

crystals were irradiated with 50 MeV Li ions at 15-UD Pelletron facility at NSC, New Delhi at different fluence of $1x10^{12}$, $1x10^{13}$, $5x10^{13}$, $1x10^{14}$ and $6x10^{13}$ ions/cm^2.

Mechanical characteristics were measured on these materials using Vicker's microhardness tester mhp-100 attached to optical microscope. Neophot-2 of Carl Zeiss, Germany whereas dielectric characteristics were carried using HP 4192A Impedance Analyzer. The values of dielectric constant at room temperature for unirradiated pure $SrFe_{12}O_{19}$ at different frequencies of 1KHz, 10KHz, 100KHz and IMHz comes out to be 11482.9, 8663.4, 2453.5 and 80 respectively whereas the respective values at same frequencies for irradiated $SrFe_{12}O_{19}$ comes out to be 12564.9, 11460.16, 5684.5, 241.28 respectively. The dielectric constant for pure strontium hexaferrite shows a remarkable increase in the values of irradiated as compared to unirradiated samples. The data regarding changes on other substituted hexaferrite (Ga-In) and rare earth orthoferrite shows similar behaviour and analysis on large number of materials is in progress. Further investigations are being carried out to elaborate on the extent of structural and magnetic modification caused by the electronic energy loss in these type of magnetic materials.

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5.2.26 Interesting aspects of Li^{3+} irradiated Bi-HTSC

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Irradiation by 55 MeV Li^{3+} beam has been carried out on $Bi_2Sr_2Ca_1Cu_2O_{8+\delta}$ (or Bi-2212) sample. The resulting changes has been studied from precise resistivity measurements down to superconducting transition (T_{c0}) with close temperature intervals (~10K/h with 0.1K difference) near T_{c0}. Preliminary analysis of the resistivity data shows interesting behaviour in the normal as well as near to superconducting state. The normal state resistivity above $2T_{c0}$ fits very well (Fig. 1) with the 2D-VRH model [1] even with the presence of radiation induced defects. Very near to T_{c0} a genuine critical fluctuation re-

gime [2] in the paraconductivity data has been found. The value of the exponent (~1) is unaltered due to irradiation.

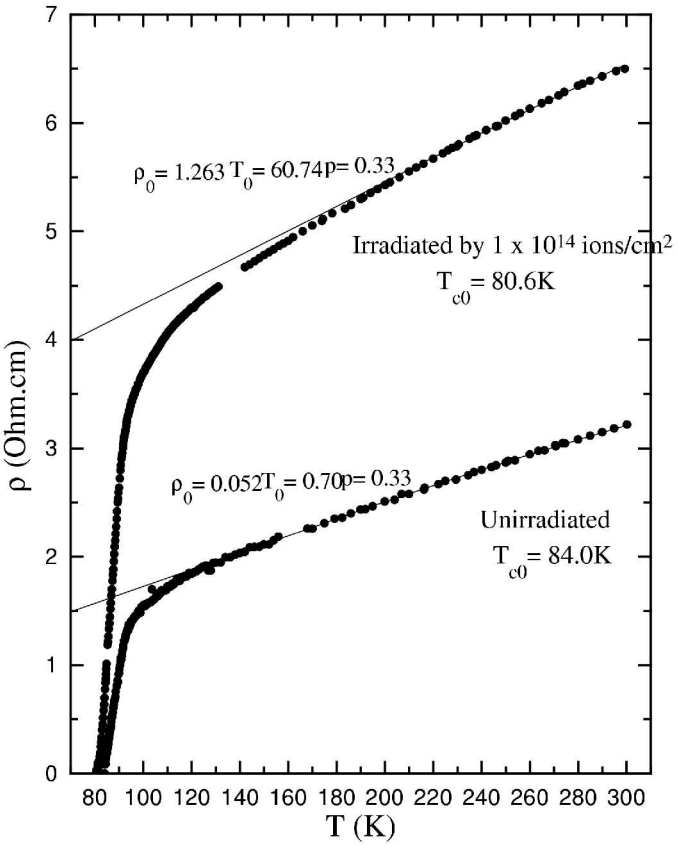

Fig. 1 : Dependence of resistivity ρ with temperature for pristine and irradiated samples of Bi-HTSC

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5.2.27 Swift Heavy Ion Induced Modifications in Ferroelectric Thin Films and Relaxors

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(1) 50 MeV Li^{3+} ion induced modifications

The ferroelectric $Pb(Zr_{0.52}Ti_{0.48})O_3$ (PZT) and $SrBi_2Ta_2O_9$ (SBT) thin films were prepared by laser ablation technique. The ferroelectric hysteresis, capacitance - voltage, dielectric analysis and dc leakage current were measured before and after Li^{3+} ion (50 MeV; 1 X 10^{14} ions/cm^2) irradiation. There was a considerable degradation in the ferroelectric properties. The irradiated PZT thin films exhibited slim hysteresis loops with a pinching at the zero coercive field and near zero remnant polarization. The room temperature dielectric constant, the dielectric loss, the remnant polarization decreased and the phase transition temperature of the irradiated PZT thin film decreased significantly from 355 ^oC to 235 ^oC and the SBT thin film exhibited a broad, diffuse phase transition with a marginal decrease in the phase transition temperature. After the irradiation, the PZT thin films showed a double butterfly loop, indicating an antiferroelectric behavior, with a large decrease in the switching voltages and capacitance. The degraded ferroelectric properties of the irradiated PZT film were partially regained after annealing at a temperature of 400^oC for 10 minutes. This may be attributed to the thermal annealing of the defects generated during the irradiation. In SBT thin films, antiferroelectric C - V characteristics were observed with a decrease in the capacitance value and ferroelectricity could not be regained after annealing. The annealed irradiated SBT thin films partially regained their original dc leakage current characteristics. The PZT film showed the characteristic ohmic region up to 3 V and it did not show significant change in the leakage behavior after irradiation. The SBT film showed a linear region up to 11 V, which is a characteristic of ohmic behavior, after irradiation the linear region was increased to 12 V with a significant increase in the current density.

(2) 70 MeV Oxygen ion induced modifications

The ferroelectric PZT, SBT and $Pb(LaTi)O_3$ (PLT) thin films were prepared by laser ablation technique. The ferroelectric hysteresis, capacitance - voltage, dielectric analysis and dc leakage current were measured before and after oxygen ion (70 MeV; 5 x 10^{11} to 1 x 10^{13} ions/cm^2) irradiation. The properties of the PZT films degraded after irradiation. The XRD showed the presence of amorphous material after irradiation. The dielectric constant decreased with the increase of irradiation fluence. However, the irradiation with fluence 1 x 10^{12} ions/cm^2 showed lesser effects than that of 1 x 10^{13} ions/cm^2, which showed a maximum degradation in the properties. The hysteresis and C – V curves showed a typical antiferroelectric nature after irradiation. The remnant polarization and the coercive fields were observed to decrease with fluence. The degraded properties could be partially restored by annealing at 400°C for 10 minutes. Similar results were also observed for the SBT, SBTN, and PLT thin films. The SBT films exhibited least degradation after irradiation. These results can be explained by the radiation theory based on the pinning of ferroelectric domains by radiation induced defects. The trapping of charges

by the radiation-induced defects can pin the ferroelectric domains and there by reduce the remnant polarization and in turn the dielectric constant.

5.2.28 Influence of High Energy Ion Irradiation on Field Emission Characteristics of CVD Diamond Thin Films

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The CVD diamond has novel and interesting features [1]. The negative electron affinity (NEA) due to the hydrogenated surface is thought to be responsible for its potential candidature as Cold Cathode material in Field Emission Displays [2]. As a result over the past few years, this field has gained considerable attention of the researchers world wide.

The field emission characteristic of a material is observed to depend on the 'physical structure' of its emitting surface, in addition to the work function. There are various physical and chemical means of surface conditioning/modification so as to obtain better field emission properties. Hence we though to study the influence of ion beam irradiation on the field emission characteristics of the CVD diamond films. In case of CDV diamond, it has been observed that field emission is dependent on ration of sp^2 to sp^3 components present in the film [3]. The sp^2 to sp^3 ratio can be varied by changing the concentrations of hydrogen and methane gasses introduced in the CVD reactor. Therefore we have deposited the CVD diamond films with different sp^2 to sp^3 ratios.

It is a well known fact that diamond thin film should contains a certain amount of hydrogen because diamonds are grown under hydrogen circumstances [4-5]. In order to reveal the amount of hydrogen present in the CVD diamond films prepared under different experimental conditions, we have also measured the H-content in these films by Elastic Recoil Detection Analysis (ERDA) with 100 MeV ^{107}Ag ions.

In the present work the diamond thin films were synthesized on Silicon (111) substrate by Hot Filament Chemical Vapour Deposition system indigenously fabricated in the laboratory. Methane (CH_4) and Hydrogen (H_2) were used as source gasses. The filament temperature was kept at about ~ 2200 ^{0}C and the substrate at about ~ 950 ^{0}C. The distance between substrate and filament was 8 mm. The diamond thin films were synthesised at different methane concentrations 1.0%, 1.5%, 2.0%, for 2 hour and 4 hour deposition periods. The CVD diamond thin films were irradiated with 100 MeV ^{107}Ag ions at different fluence 3 x 10^{11}, 1 x 10^{12}, 3 x 10^{12}, 1 x 10^{13} ions/cm^2 using NSC Pelletron over the area 1 cm^2.

The field emission measurements were performed in all metal UHV chamber evacuated at ~ $1x10^{-8}$ torr. For the field emission studies a diode configuration was used with a phosphor coated conducting glass (circular tin oxide glass plate of 60 mm diameter) as an anode and CVD diamond film as a cathode. The sample was mounted on a stainless steel disc of same diameter as that of anode. The distance between anode and cathode was kept was at about ~ 1mm.

The field emission current-voltage (I-V) characteristics of the CVD diamond films show that in each case, the emission current drawn from the irradiated films is higher than that of as prepared films. The emission current is also found to increase with the ion fluence. The values of turn-on voltage, defined to draw 0.1 μA current, are tabulated in Table 1. The turn-on voltage is observed to be fluence dependent and has lower value for irradiated films than that of as prepared films, in each case.

Table 1 : Turn on Voltages of CVD Diamond thin films recorded at pressure 1 x 10^{-8} torr

Sr. No.	Deposition Time	Methane Concentration	Turn on Voltage (Volt)		
			As it is	Low Fluence 3 x 10^{11} ions/cm^2	High Fluence 3 x 10^{13} ions/cm^2
1)	2 Hr	1.0 %	1300	900	800
2)	2 Hr	1.5 %	1000	400	560
3)	2Hr	2.0	1000	560	880

Thus it may be concluded from the I-V characteristics and values of turn-on voltages, ion beam irradiation improves the field emission behavior of the CVD diamond films.

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5.2.29 Phase Transformations in Thin Films of C_{60} Irradiated with Swift Heavy Ions

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The present work is an attempt to study the structural transformations due to irradiation by ions of varying S_e values at various fluences. Thin films of C_{60} with a thickness of 230 nm, were deposited on quartz and float glass (FG) substrates at Nuclear Science Centre, New Delhi (NSC). This thickness is very small compared to the ranges of the ions used. The films were irradiated with swift heavy ions (SHI) Au (100 MeV), Ni (110 MeV) and O (70 MeV). The changes induced in the films are monitored as follows.

Fig. 1 shows a plot of resistivity vs. fluence. These results are from in-situ conductivity measurements. Samples showed a marked increase in the conductivity after some critical fluences (10^{12} and 10^{13} ions/cm^2 for $^{197}Au^{3+}$ and $^{58}Ni^{10+}$ ions respectively). Irradiation with $^{16}O^{6+}$ ions showed no change in conductivity in a fluence range of 10^{10} - 10^{14} ions/cm^2. Optical absorption measurements were performed on pristine and irradiated C_{60} films, using U3300 Hitachi spectrophotometer at NSC. The optical data, as well as temperature-dependent 4-probe conductivity measurements, were used to determine band gap. The pristine material is a high band gap semiconductor while the high fluence irradiated one becomes a narrow gap semiconductor.

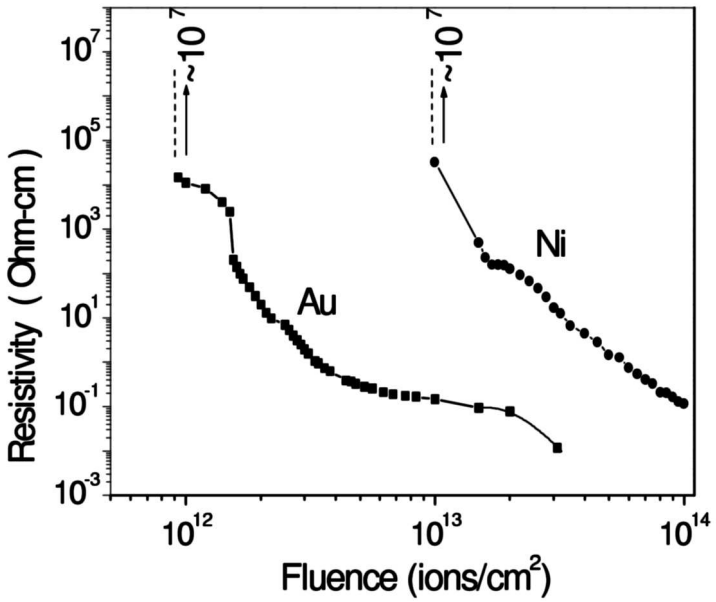

Fig. 1 : Resistivity vs Fluence for Ni irradiated C_{60} film on quartz substrate, showing onset of conductivity at flence of 10^{13}

Raman spectra of the pristine and irradiated C_{60} films were recorded using Raman spectrometer, Renishaw, Model 1000 at Indian Diamond Institute, Surat, Gujarat, at room temperature with Ar ion laser excitation 5145Å. The Raman spectrum of pristine C_{60} shows the characteristic modes [1] of C_{60} molecule -- at 1420 cm^{-1} (H_g mode), 1465 cm^{-1} (A_g) and 1570 cm^{-1} (H_g). With increase in fluence, asymmetry is seen to develop around the prominent peak (1465cm^{-1}). This asymmetry-producing peak (1458 cm^{-1}) has been identified [2] as one due to the dimerized / polymerized C_{60}. With increasing fluence, this peak increases, optimizes, then declines, ultimately vanishing at some sufficiently high fluence [3]. This peak, as well as those of $C_{60,}$ vanish almost at the same fluence. At high fluences (~ 10^{14}), two broad features, around 1380 cm^{-1} (D peak) and 1565 cm^{-1} (G peak) emerge, indicating formation of amorphous carbon (a-C) [4]. Fig. 2 shows these Raman peaks. For O ion irradiation D and G peaks were not observed even at a fluence as high as 10^{14}. O ion with a lower S_e, is expected to transform C_{60} into a-C at fluence values higher than this. Further work, at much higher fluences is planned to study this aspect in detail.

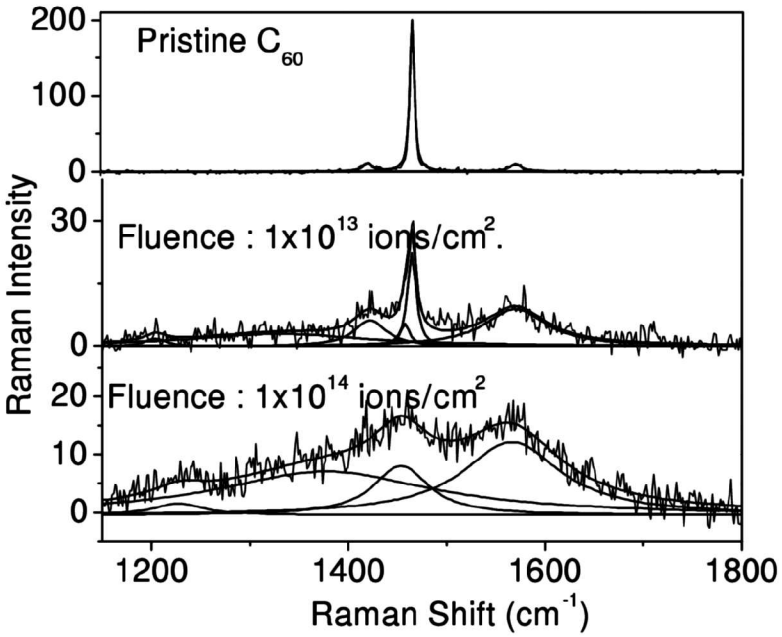

Fig. 2 : Raman spectra of pristine C_{60} compared with irradiated samples at high fluences showing D and G peaks of a-C

There is strong correlation between S_e value and the fluences at which the various phases come into existence or optimize. Lower the S_e , higher is the fluence required for onset of conductivity. Similarly, for closing of band gap and optimum dimer formation, higher fluence is required for ions with lower S_e.

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5.2.30 Physical and Chemical Response of 50 MeV Li^{3+} Ion Irradiated Blended Polymer

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The polymeric blends in the recent years are accepted as engineering plastic materials due to their better mechanical and electrical properties. Exposure of polymeric material to high energy radiation significantly changes its characteristics[1]. In the present work, samples of polyethylene teraphthalate (PET) blended with polyvinyl chloride (PVC) were irradiated using 50 MeV Li^{3+} ions at different fluences 10^{12}-10^{14}ions/cm^2 at Nuclear Science Centre, New Delhi. The thickness of the blended polymer was 460 μm. The effects of irradiation was studied with respect to their electrical, thermal and chemical behaviour by electrical frequency response using LCR bridge, thermal gravimetric analysis, differential scanning calorimetry method and FTIR spectroscopy.

Fig. 1 shows the behaviour of dielectric loss (tan δ) with temperature at different frequencies. It is observed that tanδ increases with increase of temperature at all frequencies. The dielectric loss is due to the perturbation of phonon system by an electric field, the energy transformed to the phonon is dissipated in the form of heat. The growth in tanδ and decrease in resistivity is brought about by an increase both in the conduction of residual current and the conduction of the absorption current [2]. The T_g is observed at 60^0C, which is at lower temperature corresponding to the T_g of pure PVC and PET. When polymer is heated, the movement of the large segments of the main chain sets in becoming a maximum at T_g, at which the loss tangent maximum occurs corresponding to the relaxation peak of the thermally stimulated discharge [3].

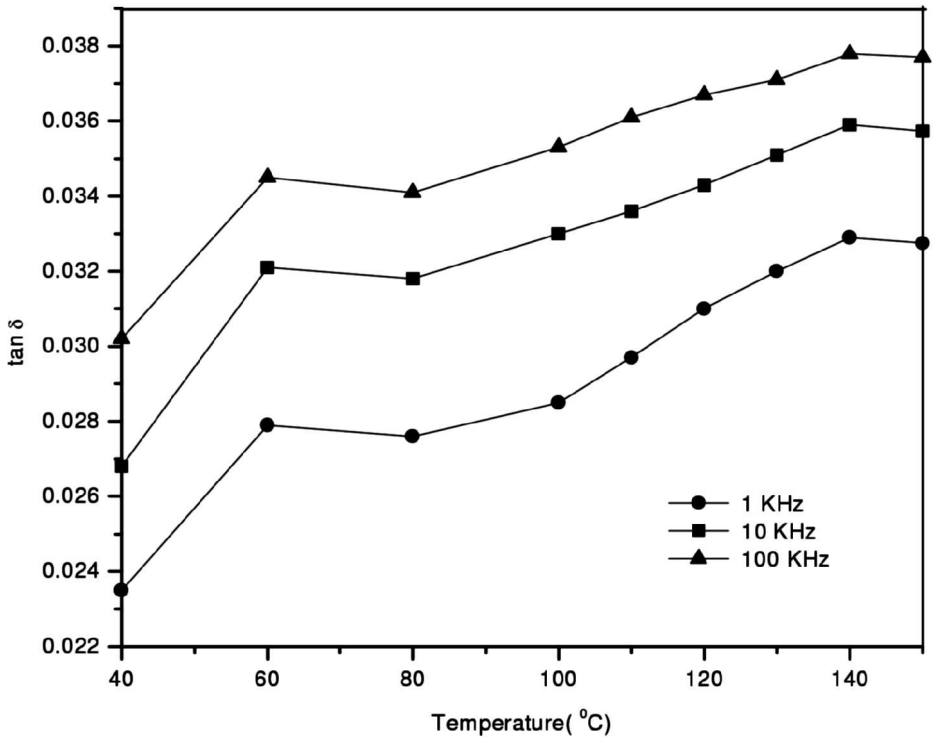

Fig. 1 : Dielectric loss (tan δ) in blended polymer with temperature at different frequencies

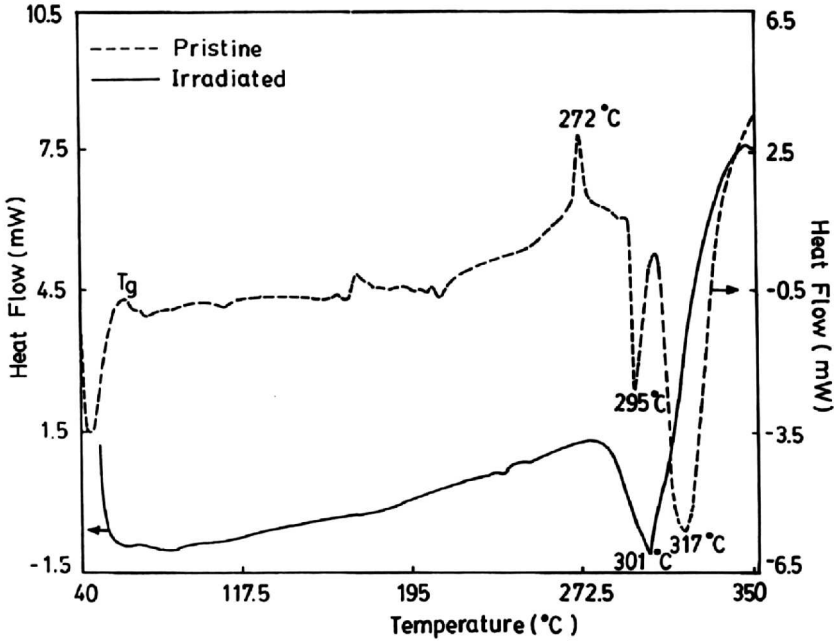

Fig. 2 : DSC thermograms of pristine and irradiated blended samples

DSC thermograms of pristine and irradiated blended samples are shown in Fig. 2. The endotherms denoting the softening temperature appeared at 295 ^{0}C and 317 ^{0}C for

PET and PVC respectively. The irradiated polymer gives a single endotherm at around 301 ^{0}C, which is the composite softening temperature of the blended polymer. A small peak at 272 ^{0}C is probably due to the oxidation of blended polymer. Due to irradiation, homogeneity of composition increases and endotherm peaks undergone a change.

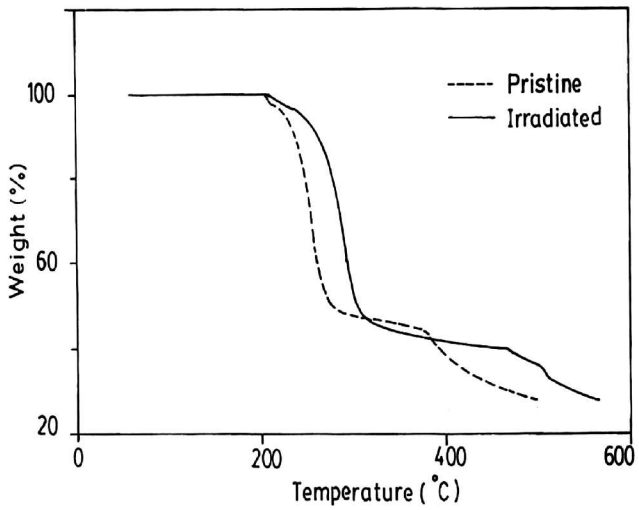

Fig. 3 : TGA thermograms of pristine and irradiated blended samples

Fig. 3 shows the TGA thermograms of pristine and irradiated blended samples. It is evident from the figure that there is a marginal improvement in the thermal stability due to Li^{3+} ion irradiation. It is observed from the TGA thermogram that irradiated sample shows lower weight loss than pristine in the temperature range of 200^{0}C and 400^{0}C. TGA analysis reveals a two step decomposition patterns for pristine and irradiated samples.

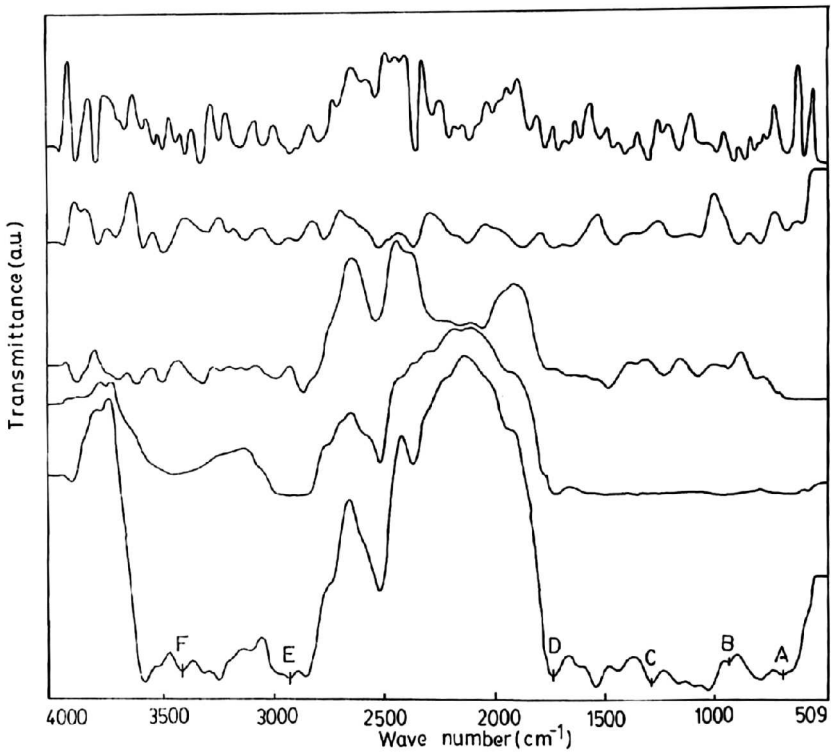

Fig. 4 : FTIR spectra of pristine and irradiated samples at different fluences

FTIR spectra of pristine and irradiated samples at different fluences are shown in Fig. 4. The absorption bands as obtained from the spectrum of pristine blend are identified as (A) 600-800 cm^{-1} (C-Cl stretching vibration) (B) 970 cm^{-1} (C-O-C antisymmetric vibration) (C) 1247 cm^{-1} (C=C stretching of phenyl ring) (D) 1720 cm^{-1} (C=O stretching vibration) (E) 2800-3000 cm^{-1} (C-H stretching in alkanes) (F) 3000-3700 cm^{-1} (OH stretching vibration). The simultaneous change of all IR lines indicates that irradiation affected regions are distributed randomly all over the polymer molecules. The change in most of the characteristic peak positions denotes the deformation of the structure. The absorbance or transmittance value of particular functional group changed. The transmittance of the bands decreased showing that degradation is the dominant phenomena resulting from irradiation.

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5.2.31 Effects of High Energy (MeV) Ion Beam Irradiation on Polyethylene Teraphthalate(PET)

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Ion irradiation of polymeric materials induces irreversible changes in their macromolecular structure and it can be used to change in a controlled way the physical properties of thin films or to modify the near surface characteristics of a bulk polymer [1]. Many experiments aimed at increasing the electrical conductivity of polymers by ion irradiation have been studied [2,3] with regard to the practical utilization of conducting polymers. In the present study, polyethylene teraphthalate (PET) films having thicknesses 230 μm were irradiated with 50 MeV Li^{3+} ions at different fluences 10^{12}-10^{14} ions/cm^2 at Nuclear Science Centre, New Delhi.

When a highly energetic charged ion strikes a polymer target, it loses its energy by electronic and nuclear stopping. The SRIM code calculations indicate that 99.95% of energy lost by 50 MeV Li^{3+} ion in 230 μm thick PET is electronic in nature. The ion irradiation of PET films leads to chain scission and as a result there are changes in the dielectric properties. The structural and electrical properties of irradiated PET films were studied using FTIR spectroscopy and variable frequency LCR meter.

It is observed that the resistivity of PET films decreases rapidly as frequency increases. It is also observed that the resistivity decreases as fluence increases. An ac field of sufficiently high frequency applied to a polymer may cause a net polarization which is out of phase with the field. This results in ac conductivity.

The dielectric loss (tanδ) is found to increase with the increase of temperature. The value of tanδ also increases with increase in the fluence. The dielectric loss is due to the perturbation of phonon system by an electric field, the energy transferred to the phonon is dissipated in the form of heat. The growth in tanδ and decrease in resistivity is brought about by an increase both in the conduction of residual current and the conduction of the absorption current [4].

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5.2.32 Study of Chemical Modification Induced by 120 MeV Silicon Ions in Polypropylene

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Polypropylene film was irradiated with 120 MeV $^{28}Si^{9+}$ in the ion fluence range of 10^{12}-10^{13} ions/cm^2. The spectral changes owing to ion bombardment were investigated by fourier-transform infra-red spectroscopy. The isotactic nature of PPy remains unaffected. A band was observed at 1640 cm^{-1} and its intensity was found to increase with ion fluence, confirmed the formation of unsaturated linkage. Increase in chain length of hydrocarbon due to addition of more CH_2 group in the polymer was also confirmed by observing the increase of absorbance with ion fluence of CH_2 wagging vibration band at 1304 cm^{-1}.

Polypropylene (PPy) is a well known polymer which is used in packaging industries, for manufacturing of disposable medical products. A large fraction of these products are now sterilized by radiation [1]. Polypropylene was chosen for the present study to investigate further the influence of heavier ion as the earlier studies were restricted to lighter ions.

Films of size (2 x 8) cm^2 were cut from commercially available sheets of isotactic polypropylene -[-CH_2-$CH(CH_3)$]$_n$-, having density 0.9 gm/cm^2 and thickness 6 μm and a stack of PPy containing 12 films was exposed to 120 MeV silicon ions in the material science irradiation facility of 15 UD Pelletron of the Nuclear Science Centre in New Delhi .The thickness of film was kept such that it was less than ion projected range so as to avoid any complexity due to ion implantation. The ion projected range was obtained as 90 μm using the range energy prescription of Ziegler et.al [2]. The irradiation was carried out at room temperature and under vacuum (10^{-6} Torr) for time periods ranging from 120 to 1200 seconds. The total number of ions falling on the target was estimated to be in the range of 10^{12} to 10^{13} ions depending on the time of bombardments. The ion beam was scanned using magnetic scanning system, so that an area of 1x1 cm^2 was uniformly irradiated. The beam current was kept low and was monitored intermittently with faraday cup.

The pristine and irradiated samples were characterised by Perkin- Elmer spectrometer RX1 FTIR system. IR spectra were recorded in the range 4000 - 400 cm^{-1}. These measurements were carried out nearly two months after the irradiation of the foils, hence the reported results represent the stationary state of the irradiated foils where the meta-

stable defects (if any) are expected to have got annealed and the radiation enhanced oxidation (if any) would have got completed.

The FTIR spectra of the pristine and the irradiated PPy are shown in Fig. 1. The symmetric and asymmetric stretching, scissoring or bending and wagging of CH_3 and CH_2 group frequencies were observed in the pristine as well as all the irradiated PPy samples. The peak position in the pristine and the irradiated PPy samples remained almost constant indicating that isotactic nature of the polymer was not disturbed by silicon ion irradiation even at highest ion fluence. The asymmetric stretching vibration of CH_3 and CH_2 groups are observed at 2960 and 2919 cm^{-1} respectively and the absorbance of the band increases with increasing ion fluence. The formation of unsaturated linkage in the ion beam irradiated PPy in confirmed by observing stretching band at around 1640 cm^{-1}. The methylene (CH_2) wagging vibration observed at 1304 cm^{-1} is of low intensity, the increase in absobance at 1304 cm^{-1} in the irradiated PPy accounted for in part by the formation of additional CH_2 group via. formation induced cross-linking mechanism [3]. Moreover the band at 1305 cm^{-1} is a temperature sensitive band and is directly related to the degree of crystallnity of polymer and thus increase in the absorbance of this band indicates the increase in crytallnity of PPy [4]. The symmetric scissoring vibration of CH_3 and CH_2 were observed at 1376 and 1458 cm^{-1} respectively. The bands 3/1 helix structure of PPy are found in pristine as well in irradiated PPy at 1167 and 973 cm^{-1}, showing that the helix structure has not been affected by ion irradiation.

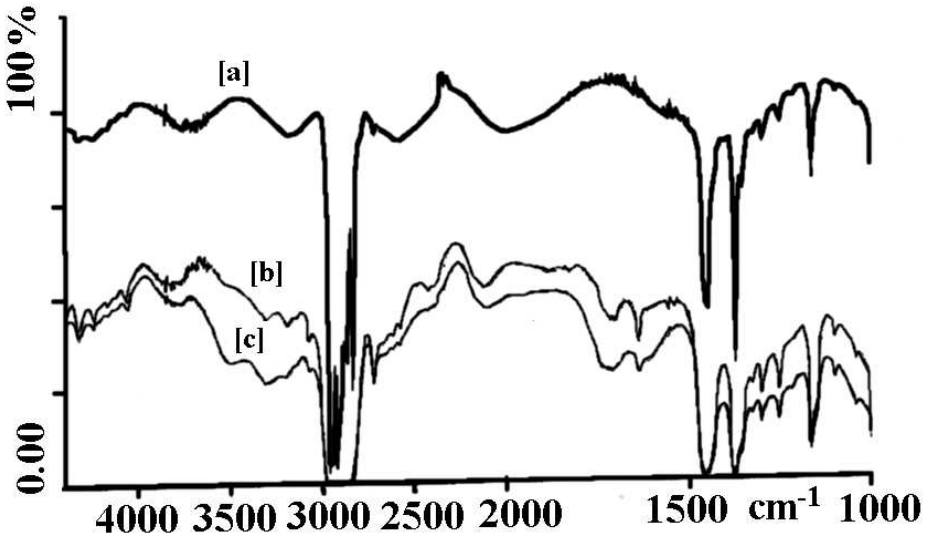

Fig. 1 : FT IR spectra of pristine and irradiated polypropylene films.
[a] Pristine [b] Irradiated with total ion fluence of 2.37 x 10^{12} ions/cm^2

It is evident from FT IR spectral analysis that the isotactic nature of PPy has not been destroyed due to silicon ion irradiation. The symmetric and asymmetric stretching, scissoring or bending and wagging of CH_3 and CH_2 group are observed both in pristine as well as in irradiated PPy. The increase in the absorbance of CH_2 wagging vibration due to irradiation indicates an increase in chain length of hydrocarbon due to some cross-linking

induced by ion irradiation, it also reflects an increase in the crystallnity of PPy. The stretching band at 1640 cm^{-1} indicates formation of unsaturated linkage in polymer induced by ion irradiation.

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5.2.33 Optical Study of Poly (Ethyleneterphthalate) Irradiated with Different Energies of Swift Heavy Ions of Silicon

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Thin films of poly (ethylenethererphtalate) were exposed to different energies of swift heavy ions of silicon. In general it was observed that there was an increase in the magnitude of absorption with the decrease in the energy and the increase in the fluence of the swift heavy ions of silicon respectively.

It is now a well established fact that irradiation of polymers with swift heavy ions leads to a wide variety of property changes (1-6). Poly (ethylenethererphtalate) generally known as PET has been found to have a number of commercial applications and is known for its superior mechanical strength, heat and chemical resistance, dimensional stability and flex-crack resistance. It has also been found to have exceptional flatness, coefficient of friction and optical clarity. It has already been investigated (7,8) for the changes in its thermal and chemical properties as a result of heavy ion exposure to swift heavy ions like argon, lead, dysprosium and helium. In the present work an attempt has been made to study the changes in the optical properties of PET by exposing thin films of it to different energies of silicon ions at two different fluences.

Thin films of 12 micron thick poly (ethylenethererphtalate) sold commercially as HOSTAPHAN were obtained from Hoechest A.G, Germany. A stack of eight films were put together so as to make a target assembly of 96 microns thickness. This target as-

sembly was found to be thick enough to stop all the ions including the swiftest used in the present work. The range of 120 MeV swift heavy ion in PET as calculated from the TRIM prescription (9) is 54.6 microns. The stack was exposed to two different fluences of 120 MeV silicon ions namely 1.23 x 10^{12} and 1.25 x 10^{13} ions. The irradiations were carried out at room temperature and under vacuum (10^{-6} torr) for time periods ranging from 60 to 600 sec. The ion beam was defocused using a magnetic scanning system, so that an area of 1.0 x 1.0 cm^2 was uniformly irradiated. The beam current was kept low to suppress thermal decomposition and was monitored intermittently with a Faraday cup. The foils from the stack in which visible changes could be observed after irradiation were separated and their UV-VIS measurements were carried out using a Perkin Elmer spectrophotometer.

The absorption spectrum in the range of 300 to 800 nm of the foils investigated in the present work is shown in Figure 1. The samples denoted by the terms V1L, V2L, V3L and V4L represent the foils irradiated with a total ion fluence of 1.2 x 10^{12} ions and energies 120 MeV, 95 MeV, 72 MeV and 45 MeV respectively. On the other hand the samples denoted by the terms V1H, V2H, V3H and V4H represent the films irradiated with a total ion fluence of 1.25 x 10^{13} ions and energies 120 MeV, 95 MeV, 72 MeV and 45 MeV respectively. The energy with which the individual foils were irradiated was calculated using the TRIM prescription (9) taking into account the thickness of each foil separately.

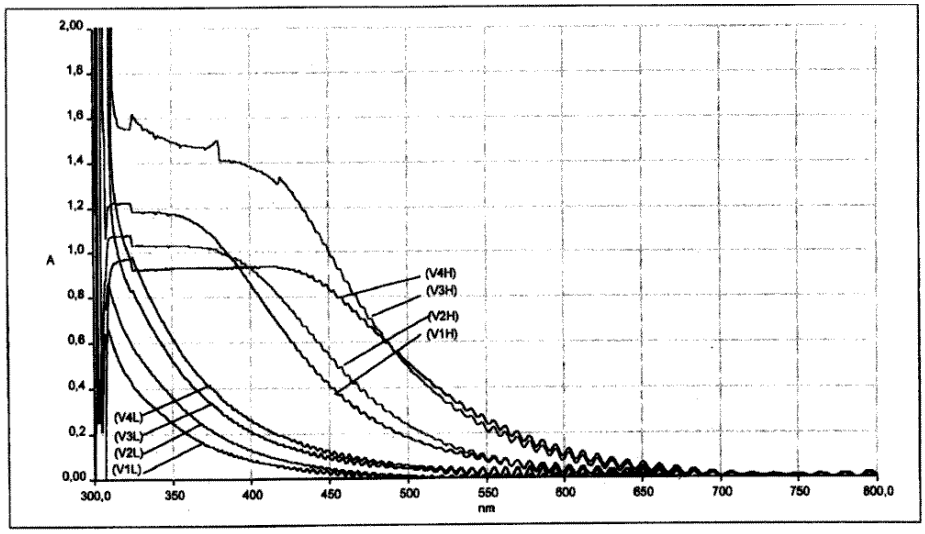

Fig. 1 : Plot of absorption as a function of wavelength for PET films exposed to Si beams of different fluence

The absorption spectra revealed that the absorption magnitude in both case, whether one considers the foils exposed to lower ion fluence or one considers the foils exposed to higher fluence, that there is a general increase in the absorption value with the decrease in the energy of the swift heavy silicon ions. This observed effect perhaps has its origin in the relative higher rate of energy loss the heavy ion of interest has to undergo in

the polymer medium as it slows down. It is further observed that there is a general shift of the absorption edge towards the red end of the spectrum with the increase in the total ion fluence, when one compares foils of similar energy which is perhaps indicative of the increase in the conjugation length. Therefore in conclusion it can be said that the optical studies carried out in the present work indicate that the optical absorption does get influenced to a good extent with the decrease in the energy and the increase in the fluence of swift heavy silicon ions.

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5.2.34 Effect of Si^{28} Ion-Beam Irradiation on Natural Rubber and Carboxylated Styrene Butadiene Rubber Latex Blends

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Ion beam irradiation of polymeric material induces irreversible changes in their macromolecular structure. Just like electron and gamma radiations ion beam radiation can be used to change the properties of thin films of polymers. The major phenomena associated with the interaction of ion with polymer film includes chain scission, chain aggregation, double bonds and molecular emission [1]. All these effects mainly depend on the target parameters like composition, molecular weight, temperature etc. and on ion beam parameters like energy, mass and fluence [2].

The effect of low and high fluences of ion beam irradiation on polymers have attracted large interest in the last decade and have been investigated in many laboratories.

Lee et al [3] have reported the surface smoothness, hardness and wear resistance of polymers by multiple ion implantation. Fink and co-workers [4] characterised the optical properties of ion beam irradiated polymers. Recently Rajulu et al [5-6] have investigated the chemical modifications occurred in PMMA/PVC blend films on ^{28}Si ion beam irradiation. Very recently, Hyong et al [7] observed that the interface adhesion of the two immiscible polymer blend systems can be improved by ion beam irradiation. Shariff et al [8] studied the influence of ion irradiation on the free volume controlled diffusion process in polycarbonate.

The present work is to investigate the effect of ion beam irradiation on natural rubber (NR) and carboxylated styrene butadiene rubber (XSBR) latices and their blends. Till date no systematic studies have been conducted in ion- beam irradiation of latex films. There are lot of works on electron beam and gamma ray irradiation studies in latices. The structural modifications occurred in the system is being characterised by UV-Visible spectroscopy, FTIR spectroscopy, and scanning electron micrographs.

The chemical modifications occurred in the NR/XSBR latex blends and individual latices on ion beam irradiation were analysed by our research group. It was observed that the colour of the sample changes to dark brown in accordance with increase in fluence in all cases. The colour change was more predominant in blends with higher ratio of XSBR latex. SEM of the irradiated samples was performed. The 70/30 NR/XSBR blend shows crack initiation at lower fluences and continues at higher fluences. For the 50/50 and 30/70 NR/XSBR latex blends the surface is smoother at higher fluences. This can be attributable in terms of the interphase reaction occurred in the system on irradiation.

FTIR analysis revealed that the chemical structure of the blends was varied on irradiation. The peak intensities were decreased with increase in fluence, but peak position was not changed. The intensity change was predominant at higher fluences. This is because of the chemical degradation occurred in the system at higher doses.

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5.2.35 Dielectric Behaviour of 100 MeV Ni^{58} Ion Irradiated Kapton-H Polyimide Film

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The high-energy heavy ion irradiation effects in kapton-H polyimide have drawn considerable interest owing to its possible application as conducting polymer and in the development of optical devices [1-3]. However, the dielectric relaxation behaviour of this polymer irradiated with energetic heavy ions has not been reported much. In the present communication we are reporting the temperature dependent dielectric constant / loss behaviour of kapton-H polyimide irradiated by high energy Ni^{58} ion (fluence: $1.9x10^{11}$ ions/cm^2) which were made from room temperature (35^0C) to 240^0C using a precision LCR meter (Hitester) at frequencies ranging from 100 Hz to 5 MHz.

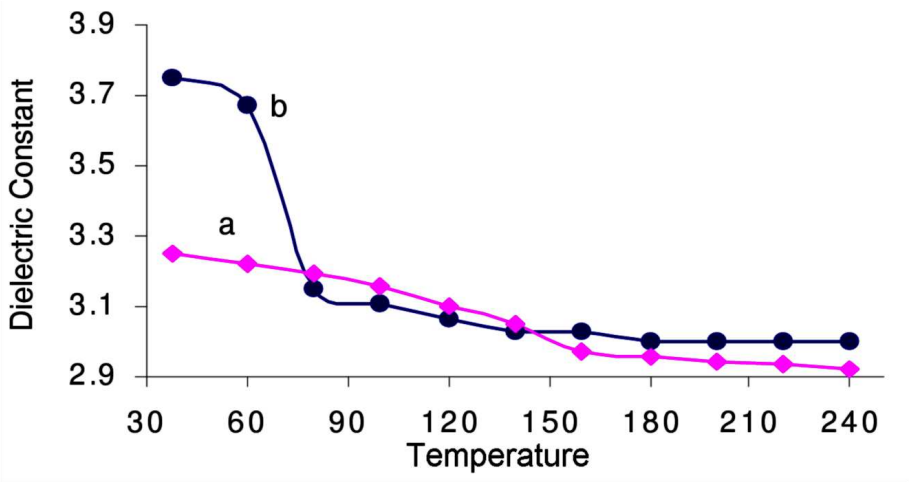

Fig. 1 : Variation in dielectric constant with temperature (Freq: 5 MHz) for (a) pristine and (b) irradiated kapton-H samples

Fig. 1 illustrates the representative temperature dependence of dielectric constant (ε′) of pristine and irradiated kapton-H. Fig. 2 illustrates the variation of dielectric loss (ε″) with temperature for kapton-H at 5 MHz and Fig. 3 illustrates the variation of dielectric loss (ε″) with frequency in irradiated kapton-H at 60^0C and 120^0C. The ε′ - T curve follows chair like model with three distinct temperature regions. The variation in ε′at low temperature region is ascribed to absorbed water (γ-relaxation). An increase in water absorption capacity in kapton-H due to irradiation has been shown by Shrinet et al [4], followed by dipolar (β-relaxation) in intermediate temperature. It appears that in this temperature region 60^0C-140^0C, the factors responsible for increasing or decreasing dielectric constant with temperature are

simultaneously operative. TSDC results have shown that this region mainly belongs to dipolar relaxation and to some extent to shallow energy traps [5]. An increase in dielectric constant with increase in fluence (results not shown) is in conformity with the formation of free radicals due to irradiation. The high frequency ε″ - T curve for pristine sample do not give any significant loss peak in high temperature range [6]. This confirms our conclusion that free radical formation is the major phenomena in irradiated sample. The free radical formation and appearance of new phases try to increase the ε′ value in high temperature region compensating the decrease in ε′ with T owing to polar nature of the polymer. The formation of free radicals due to high-energy irradiation has been reported earlier [3]. The appearance of these relaxations appears in the form of different dielectric loss maxima around 60^0, 120^0, 180^0C in ε″ -T curve. The frequency dependence dielectric loss (ε″) curves show the presence of smaller as well as large segmental groups.

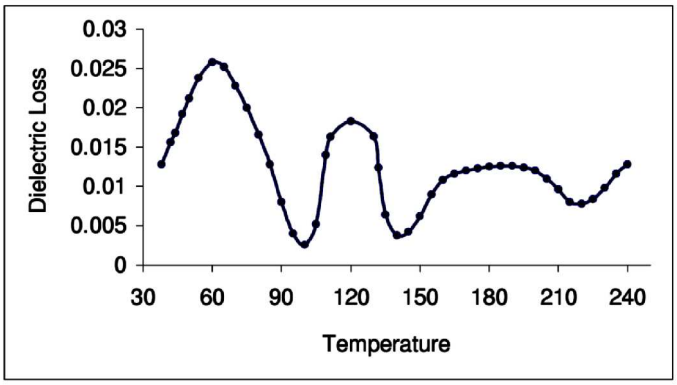

Fig. 2 : Variation of dielectric loss with temperature for irradiated kapton-H at 5MHz

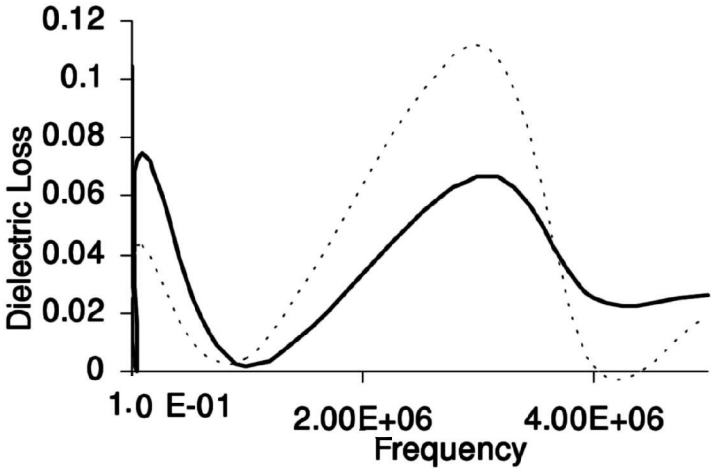

Fig. 3 : Variation of dielectric loss with frequency in irradiated kapton-H at (a) 60^0C (b) 120^0C

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5.2.36 Photo Conduction in 75 MeV Oxygen Ion Irradiated Kapton-H Polyimide

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Photoconduction in Kapton-H has been investigated for 75MeV oxygen ion irradiated samples [Fluences: 1.9×10^{11}, 1.9×10^{12}, 1.9×10^{13}]. Dark and photocurrents were measured in the temperature range of 30^0 to 250^0C at different fields ranging from 20 to 600kV/cm under steady state condition. The steady state condition is obtained in 10 minutes time after the application of field. The effect of operating temperature, field and fluence on the steady state current (I_s) has been illustrated in Figs. 1-3. An enhancement in the dark as well as photoconduction takes place with operating temperature and field. For the same parameters (field and temperature) significant increase in the conduction is observed when the samples are illuminated with light. This shows the excellent photo conducting property of kapton-H. An increase in photo conductivity with increase in fluence (Fig. 2) also indicates that there is some improvement in the photo conducting properties of kapton-H after irradiation. The photoconduction behaviour of kapton-H polyimide has been attributed to the photo generated charge carriers in the material [1,2]. The ketonic and the phenylene groups present in the structure of kapton-H are mainly responsible for governing the photoconduction mechanism. Modification in the charge trapping character of these groups [3] after irradiation is evident from the fluence dependent steady state conduction.

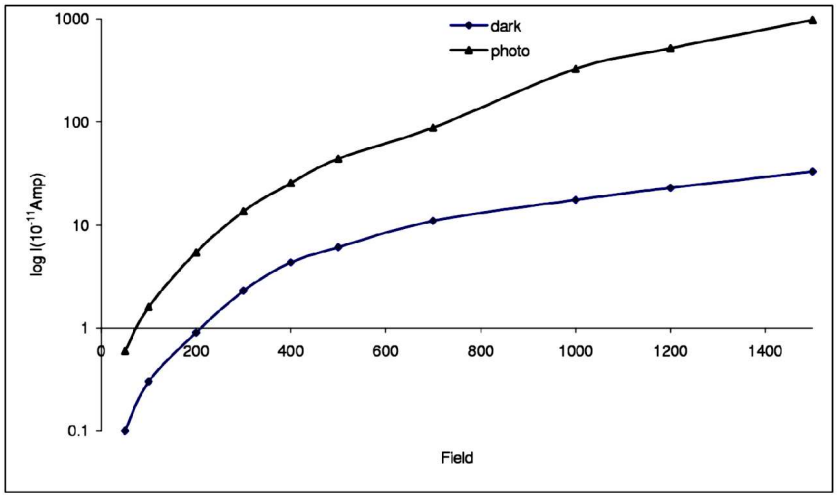

Fig. 1 : Variation of current with voltage for 75 MeV Oxygen irradiated Kapton-H

polyimide sample at 90^{0}C

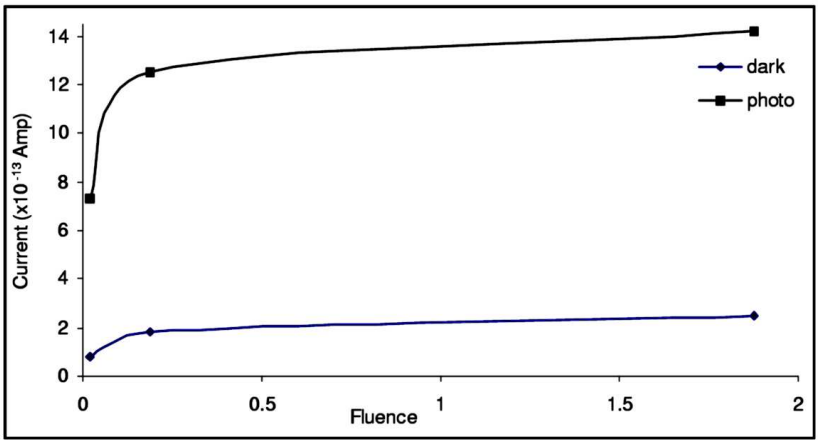

Fig. 2 : Variation of current with fluence for 75 MeV Oxygen irradiated Kapton-H polyimide sample at 30^{0}C, 500Volts

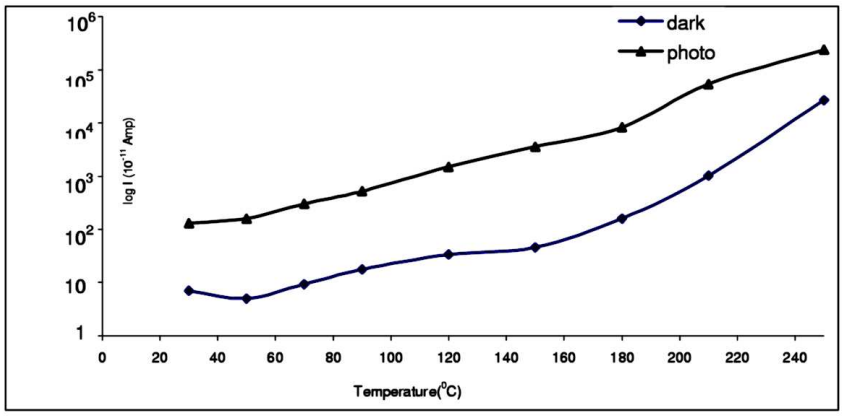

Fig. 3 : Variation of current with temperature for 75 MeV Oxygen irradiated Kapton-H polyimide sample at 1200Volts

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5.2.37 Investigation of Swift Heavy Ion Irradiation Effects on Ionic Conduction in P(VDF-HFP)-$LiClO_4$ Polymer Electrolytes

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Ionically conducting polymer electrolytes might replace the liquid electrolyte currently in use thereby enabling the fabrication of flexible, compact, laminated solid state structures free from leaks and available in various geometries [1]. It is generally believed that in polymeric materials the ionic conduction is a property of the amorphous phase and ion association, ion-polymer chain interactions and local relaxations of the polymer strongly influence the ionic conductivity [2]. Ion beam modification of polymers has been found to produce useful improvement in their physical and chemical properties [3,4]. In case of high energy ion beam irradiation of polymers, the electronic energy loss of incident particle is released into radiative decay, production of new reactive species (radicals, gases) and defects (unsaturation, scissions, cross-links) [5].

Poly (vinylidene fluoride-co-hexafluoropropylene) (PVDF-HFP) as host polymer, lithium perchlorate ($LiClO_4$) as salt were used without further treatment to prepare mechanically stable 20-30 μm thick polymer electrolyte films. Films were prepared by varying the polymer-salt ratio. Appropriate weights of (PVDF-HFP) and $LiClO_4$ were dissolved in tetrahydrofuran and stirred continuously until the mixture took a homogeneous viscous liquid appearance. The solutions of different compositions were cast on to glass plates and allowed to evaporate the solvent at room temperature.

Polymer electrolyte samples were irradiated at GPSC beam line with Si ion beam of energy 100 MeV at six different fluences of 5×10^{10}, 10^{11}, 3×10^{11}, 7×10^{11}, 10^{12} and 3×10^{12} ions/cm^2 respectively. Ionic conductivities of polymer electrolytes were evaluated from the complex impedance plots using a Hioki 3532-50 LCR Hitester in the frequency range 42 Hz to 5 MHz. A symmetric copper electrode system was used for ionic conductivity measurements.

Fig. 1 shows the conductivity (ln σ) versus temperature inverse plots of (PVDF-HFP) based polymer electrolytes. The figure shows that the ionic conduction in the polymer electrolyte system obeys the VTF (Vogel-Tamman-Fulcher) relation, which describes the transport properties in a viscous matrix [6]. Fig. 1c is the plot for unirradiated sample, Fig. 1a (fluence 10^{11} ions/cm^2) and fig.1b (fluence $5x10^{10}$ ions/cm^2) are the plots for irradiated samples. The figure shows that the electrical conductivity is enhanced by about an order of magnitude upon irradiation. Moreover enhancement in conductivity is more for higher fluence. Maximum conductivity of irradiated (PVDF-HFP)-$LiClO_4$ electrolyte at 100°C is found to be 8.3×10^{-5} S/cm which is an order of magnitude higher than the conductivity of unirradiated sample at the same temperature ($8.0x10^{-6}$ S/cm).

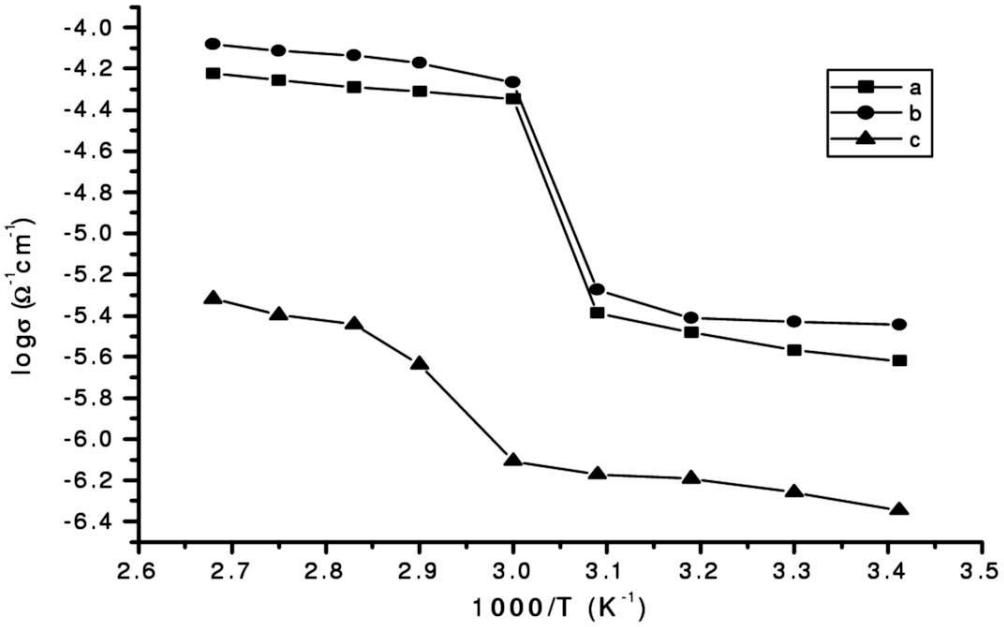

Fig. 1 : Temperature dependence of ionic conductivity of (PVDF-HFP)-LiClO4 (1:0.5) electrolyte at different fluences (a) 10^{11} ions/cm^2 (b) 5 x 10^{10} ions/cm^2 and (c) unirradiated electrolyte sample

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5.2.38 Gas permeability studies of 100 MeV Si^{8+} ion irradiated polymers

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In present work, the aim was to develop the polymeric membrane filters using Swift Heavy Ion (SHI) irradiation. SHI, when passes through polymeric media results the production of latent tracks surrounded by halos [1]. If, thickness of the membrane is quite lower than the stopping range of projectile, the transmitted tracks are being created. Chemical etching is used to dissolve the damage area. Because of transmitted tracks, the shape of tracks is changed in cones from both sides.

In our experiment we used Si^{8+} ion beam of 100 MeV as projectile and polycarbonate as target material. The membranes were irradiated at the fluence 10^6-10^8 ions cm^{-2} using rotating flywheel attachment [2] in GPSC chamber. The irradiated membrane was characterized by gas permeation using permeability cell [3] for hydrogen and carbon dioxide. The 6N NaOH was used as etchant at 60(±2) °C. The irradiated membranes were etched in the steps of 1 or 2 minutes. The permeability has been measured after every etching.

The permeability was found to increase with increasing etching time as the damaged area is etched. In transmitted tracks, as the cones of both sides meet at their vertex due to increasing etching time, a rapid enhancement in gas permeability was observed. The time at which it was observed is also depended on the fluence. The permeability of H_2 and CO_2 increases with etching time, whereas, the permselectivity of H_2 over CO_2 increases and reached up maximum and on further etching, it was reduced. This is due to the large opening of tracks at their vertexes.

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5.2.39 Luminescence in Sapphire Induced by Swift Heavy Ion Irradiation

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Most of the research studies for production of color centers in sapphire have been carried out using low energy (keV range) ion beam where effect of nuclear energy loss is important [1]. There is a lack of research in SHI induced radiation color centers in sapphire where electronic energy loss plays a major role [2]. Sapphire is highly resistant to ion irradiation. The threshold value of S_e for formation of extended defects or discontinuous tracks has been found to be 18 keV/nm [3]. In this report the role of electronic energy loss and nuclear energy loss for generation of color centers are analyzed. Single crystals of sapphire (α-Al_2O_3) of size (10mmx10mmx1mm) were irradiated at room temperature with 190 MeV and 100 MeV Ag ion beam using 15UD Pelletron tandem accelerator at Nuclear Science Centre, New Delhi. The samples were irradiated at varying fluence from $1x10^{11}$ to $1x10^{13}$ ions/cm^2. The pristine and irradiated sapphires were characterized by photoluminescence (PL) using Mechelle-900 Spectrograph under 2.8 eV He-Cd laser excitation and by ultra violate/ visible optical absorption techniques.

Optical absorption spectrum of irradiated sapphire crystal is shown in Fig. 1. For irradiated sapphire, absorption occurs at 6.1 eV and 4.8 eV. They are attributed to F (oxygen vacancy with two electrons) and F^+ (oxygen vacancy with one electron) center respectively. Peak appearing at 2.8 eV (Fig.1b) corresponds to F_2^{2+} (two oxygen vacancies with two electrons) type defect [4].

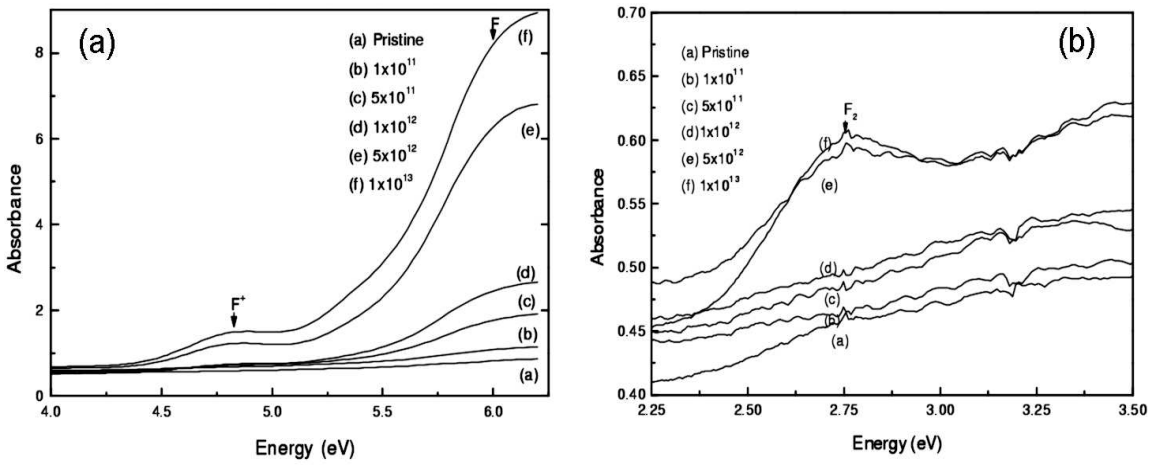

Fig. 1 : UV-VIS absorption spectra of 190 MeV Ag irradiated sapphire

Photoluminescence measurement was carried out for irradiated and pristine sapphire using 2.8 eV excitation of He-Cd laser. PL peak is observed at 2.25 eV (Fig. 2). Observed PL spectrum at 2.25 eV corresponds to F_2^{2+} center [4,5]. Intensity of PL spectrum increases rapidly after a fluence of $5x10^{12}$ ions/cm^2. These F_2^{2+} centers can be considered as aggregate of defects like F^+ centers.

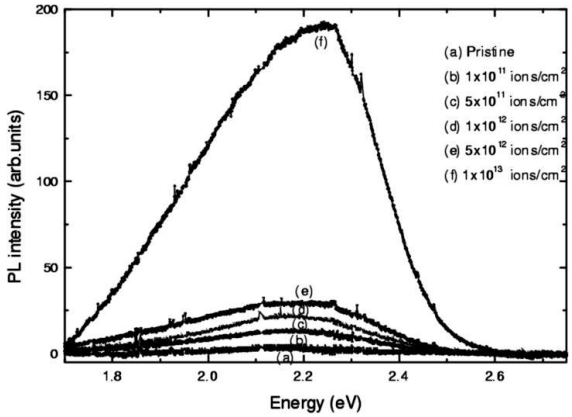

Fig. 2 : Photoluminescence spectra of pristine and 190 MeV Ag irradiated sapphire

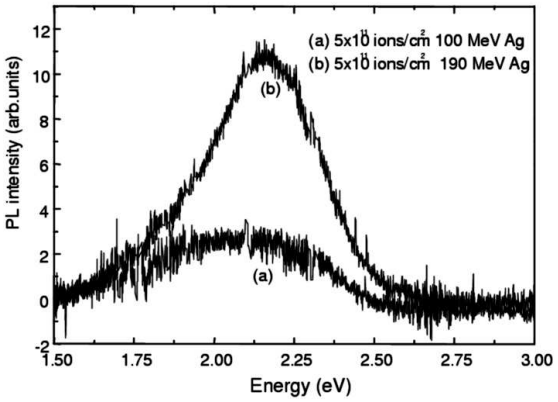

Fig. 3 : PL spectra of sapphire irradiated using 190 MeV and 100 MeV Ag ion

To observe the energy loss dependent behaviour of defects, we have also irradiated α-Al_2O_3 using 100 MeV Ag ion beam. Change in PL spectrum for sapphire irradiated at $5x10^{11}$ ions/cm^2 using 100 MeV and 190 MeV Ag ion beam is shown in Fig. 3. PL spectrum of low intensity is ob-

served for samples irradiated at 100 MeV, while peak intensity is very high for sapphire irradiated with 190 MeV Ag ion (Fig. 3). Comparing the energy loss of silver ion beam at two fluences, it is found that there is not much change in electronic energy loss between 100 MeV Ag ion beam and 190 MeV Ag ion beam. S_e is 20 keV/nm for 100 MeV Ag and S_e =23 KeV/nm for 190 MeV Ag ion irradiation. S_e values for both 190 MeV and 100 MeV Ag ion exceed the threshold value for damage creation (18 keV/nm). However, nuclear energy loss (S_n) at 100 MeV is twice that at 190 MeV (S_n is 0.1 keV/nm for 100 MeV Ag and S_n is 0.05 keV/nm for 190 MeV Ag). Although S_n for 100 MeV is much higher than that for 190 MeV Ag, the PL spectra show that 190 MeV Ag beam having higher S_e produces much more F_2^{2+} centers. Inelastic collision of ion with electrons of sapphire (S_e) seems to be the dominant factor for production of color centers in sapphire.

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5.2.40 Latent track creation in fused silica by 200 MeV silver beam

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In this study, we report estimation of track radius from UV/Visible absorption spectra of SiO_2 irradiated by 200 MeV Ag ions which agrees well with that determined using transmission electron microscopy (TEM). Tracks could be created efficiently due to the fact that the S_e value of 200 MeV Ag ion beam on SiO_2 is 14 keV/nm which is much higher than the S_e^{th} value (1.8 KeV/nm) [1]. Optical grade fused silica (SiO_2) were irradiated at room temperature at varying fluence from $5x10^{10}$ to $1x10^{13}$ ions/cm^2 of 200 MeV Ag ion beam using 15UD Pelletron tandem accelerator at Nuclear Science Centre. Samples are irradiated at varying fluences from $5x10^{10}$ ions/cm^2 to $1x10^{13}$ ions/cm^2. UV-Visible absorption spectra of the irradiated and pristine silica were taken using Hitachi 3300 UV/Visible spectrophotometer. High Resolution TEM (HRTEM) studies were carried out using 200 keV transmission electron microscope (JEOL 2010) having point to point resolution of 0.19 nm, for morphology and structure analysis of SiO_2. The bright-field images obtained from 200 keV JEOL 2010 transmission electron microscope, show a distribution of almost circular areas in contrast to the surrounding matrix. The dark circular area at the center shows the amorphous character

of the latent core in the amorphous matrix. From TEM, radius of latent track is found to be R ~ 6.0 ± 0.3 nm.

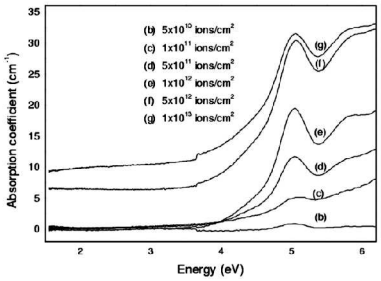

Fig. 1 : UV-VIS absorption spectra of irradiated SiO_2

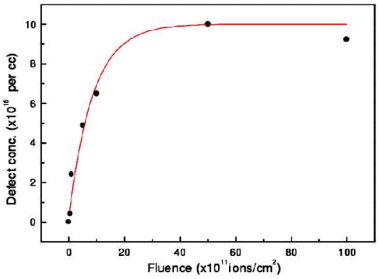

Fig. 2 : Variation of defect concentration with fluence

UV-VIS absorption studies show peaks (Fig. 1) at 5eV and 5.8 eV which increase with fluence. These peaks correspond to defects like E' centers, oxygen deficient centers and non-bridging oxygen hole centers [2]. The absorption spectra are corrected by subtracting the absorption coefficient of unirradiated specimen. The number densities of defect centers are calculated through $N\ [cm^{-3}] = 7.2 \times 10^{15}\ F/f$ where $F = H \times \Delta E$ and f is the oscillator strength. Here ΔE is the full width at half maximum and H is the peak height. The number densities of defect centers are plotted with fluence (Fig. 2).

Variation of defect concentration with fluence shows saturation at higher fluence beyond 1×10^{12} ions/cm^2 having Poisson relation type behaviour. Using Poisson relation, latent track radius was estimated to be 5 nm. These results agree well with the track radius obtained from TEM studies. This value of track radius agrees with value of track radius (~ 5.2±0.8 nm) calculated for S_e~14 keV/nm from Rutherford Back Scattering Channeling (RBS-C) analysis and TEM studies [3,4].

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5.2.41 On-Line Ionoluminescence, in-situ Photoluminescence and off-line Thermoluminescence Studies in Kyanite

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Natural and synthetic minerals find important place in the field of Archeology and Geology as some of these are found to be potential dosimetric and thermoluminescence dating materials [1]. Ionoluminescence (IL), also known as ion beam induced luminescence (IBIL) is a phenomenon of emission of light when energetic ions interacting with solid. It provides information about the chemical form of elements present in the sample. Also, it allows the detection of rare earth elements in the host materials with a minimum detection level of a few ppm. In material sciences, the IL methods can be applied to study the intrinsic and extrinsic luminescence phenomena and is capable of microcharacterizing various inorganic materials.

In the present work on-line IL, in-situ photoluminescence (PL) and off-line thermoluminescence (TL) due to 100 MeV Ni^{+8} swift heavy ion irradiation have been studied and the results obtained are presented here.

Online IL of kyanite single crystals irradiated with 100 MeV Ni ions for fluences in the range 0.75-3.00 x 10^{11} ions/cm^2 was recorded. A pair of IL bands with peaks at about 775 and 875nm along with a pair of sharp peaks in the range 680-720 nm are recorded. Also, similar results are obtained in the case of pelletized kyanite samples irradiated for the same amount of ion fluence. It is observed that the IL intensity is found to be very large during early stage of ion beam bombardment, where as in the later stages it diminishes with increase of ion fluence. The pair of sharp peaks observed in the range 680-720nm are attributed to luminescence centers activated by Fe^{3+} ions [2].

The PL of kyanite single crystals irradiated with Ni ions in the fluence range $1x10^{11}$-$5x10^{13}$ ions/cm^2 was recorded by 442 nm laser beam excitation. A pair of sharp and intense emission with peaks at 688.7 and 705.9 nm along with a broad PL emission in the region 700-900nm are observed. The PL intensity in all the samples studied in the present work is found to decrease with increase of ion fluence. The decrease in PL is attributed to Al-O and Si-O bands [3].

TL of crystalline as well as pelletized kyanite bombarded with 100 MeV Ni ions with the fluence in the range $1x10^{11}$-$5x10^{13}$ ions/cm^2 have been studied at room temperature. Two TL glows - a weak one with peak at ~ 425K and another well resolved glow with peak at ~ 505K are recorded in crystalline form of kyanite. In the case of pelletized kyanite samples, two TL glows – a weak one with peak at ~ 450K and another well resolved and intense glow with peak at ~ 525K are observed. It is found that as the fluence of Ni^+ ion increases, the TL intensity increases. However, when the fluence was

increased beyond $5x10^{12}$ ions/cm^2, the TL intensity was found to be decreased. The decrease in TL intensity may be attributed to amorphization of the material. The reduced in TL intensity and shift in glow peaks are attributed to physical structure of the sample [4].

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5.2.42 Swift Heavy Ion Beam Induced Luminescence Studies of Color Centers in PSL and FED Phosphors

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In recent years imaging plates incorporating X-ray phosphors have gained interest in the fields of medical X-ray diagnostics and diffraction in two dimensional detectors [1]. X-ray information can be stored in the form of locally trapped electron-hole pairs thereby creating a latent image, which is recovered by scanning the film with a laser beam pixel by pixel. During the optical excitation trapped electrons are released and subsequently recombine with holes causing the emission of light (Photo Stimulated Luminescence).

BaFBr is an efficient X-ray storage phosphor [2] being useful in the fabrication of Image Intensifying screens when europium is incorporated in it. The process involved is retrieval of hidden image by means of photo stimulation. The mechanism is associated with the process of radiative recombination of de-trapped electrons and holes. It is due to this reason; several people tried to understand the trapping mechanism in these BaFCl type materials through photo luminescence and thermally stimulated luminescence studies. Thermally stimulated luminescence is caused due to release of electrons from color centers and subsequent recombination with holes. The generation of color centers in these PSL phosphors is possible by ion beam irradiation. In order to understand the luminescence mechanism in these PSL phosphors, the authors have undertaken a systematic study of thermally stimulated luminescence of ion beam irradiated mixed crystals of

$BaFCl_{1-x}Br_x$. Effect of irradiation, effect of composition and effect of annealing time were considered.

Field Emission display (FED) phosphors are essential to providing high intensity; full color flat panel displays that are readable in sunlight. Portable computers are increasing the demand for FED's. Some day FED's may replace CRT's. In view of their importance, studies of luminescence processes in ion beam irradiated Sr2CeO4 FED phosphors (a newly synthesized blue emission phosphor) are undertaken.

(i) Photo Stimulable Luminescent Phosphors

BaFCl, BaFBr, $BaFCl_{1-x}Br_x$ single crystals have been grown by flux method adopting slow cooling technique. The structure of the crystals has been confirmed by X-ray diffraction measurements while the purity of the samples was estimated by spectro chemical methods. The samples have been irradiated with 100 MeV Si ion beam. A conventional thermo luminescence setup has been used to record the thermally stimulated luminescence. Work is in progress for the analysis of the ion-irradiated samples.

(ii) Field Emission Display phosphors

Powder samples of blue emission display phosphors of Sr_2CeO_4 and $Sr_2CeO_4:Eu^{3+}$ were prepared using metal nitrates of strontium and cerium through Sol-gel route. Photo luminescence studies revealed emission lines at 452, 470 and 482nm. The 470nm blue emission belong to the host due d-f transition of cerium. The blue white emission band peaking at 482nm is due to ligand to metal charge transfer at cerium making it Ce^{4+} Incorporation of europium resulted in its characterstic emission at 593 and 610nm aloong with an enhancement of overall intensity by an order of 2.

Optically stimulated luminescence of ion (Swift heavy ion of Si, 100 MeV) beam irradiated Sr_2CeO_4 showed broad emission at 475, 580 and a sharp line at 890nm. In $Sr_2CeO_4:Eu^{3+}$ a strong emission around 552, 594 and 610nm was observed with a significant high intensity. This enhancement of luminescence intensity by an order of 2 suggests that the host lattice acts as a single host lattice for the generation of blue-white light and red lights under UV excitation.

Thermally stimulated luminescence of X-ray irradiated Sr_2CeO_4 and $Sr_2CeO_4:Eu^{3+}$ indicated glow peaks at 415, 470K and these glow peaks have been attributed to thermal ionization of F-aggregate and F centers due to oxygen ion vacancies in the material.

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5.2.43 Energy Loss of Heavy Ions in Gases

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The slowing down of energetic ions in multielemental target materials is a fascinating phenomenon. The energy loss of ions in compounds and mixtures is estimated by weighing the stopping powers of the individual elements by their relative concentrations in a linear combination given by Bragg rule. The Bragg additivity rule, however, is approximate and the stopping is known to depend on the physical state as well as in compound matter, also on the chemical structure of the target material [1,2] The present work aims, in particular, in elucidating the gas-solid effect by measuring the energy loss of ions in gaseous samples over a wide range of energies and compare these measurements with the stopping powers obtained indirectly by extracting them from the measured compound stopping powers in solid samples having the gaseous elements as constituents [3, 4].

The work concerns the measurement of energy loss of heavy ions namely C, F, Mg, Al, Si and Cu over the energy range of 6-37.8, 8.33-53.43, 9.83-62.99, 10.48-67.20, 10.73-68.81 and 15.28-97.95 MeV respectively in gaseous samples of H_2, O_2 and CH_4 employing ERDA technique. The experiment was conducted in GPSC using 15 MV UD Pelletron accelerator using 140 MeV Ag^{12+} as primary ion beam. The energy of the secondary ions was varied by changing the recoil angle over 30-70°. The targets namely LiF, C, Mg, Al, Si and Cu of thickness 8.8, 26.4, 17.4, 27.0, 23.2 and 63.1 μg/cm^2 respectively were prepared using e^- gun or resistive heat methods. A gas cell fitted inside with a surface barrier silicon detector in the rear and having a window of 2 μm thick PEN in the front was used. The energy loss measurements were made for the gas samples of H_2 at 25 mbar, or O_2 at 10 mbar or CH_4 at 25 mbar under a vacuum of $5x10^{-6}$ Torr. The measurements were performed in reflection geometry for the carbon recoil ions, and, in transmission geometry for the rest of the recoil ions. The analysis of the experimental data is in progress.

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5.2.44 Transverse Cooling of Channeled Ions

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A strong flux redistribution has been observed for a quasi-isotropic beam after its transmission through a thin single crystal [1]. This observation violated the general principle of reversibility. The given explanation was based on the electron capture and loss processes which occur during the ions journey through the crystal. The charge state of the ion is in dynamic equilibrium with several electron capture and loss cycles during its passage. The transverse energy of ion reduces when it captures an electron and the opposite happens on an electron loss. In certain conditions, we observe an enhancement of channeled ions flux. This happens when there is an increase in the density of the ions whose transverse energy got reduced after passing through the crystal. Also in some conditions there is an increase in the density of ions whose transverse energy got increased, in which case we observe reduction of the channeled ions flux. The former flux redistribution is termed as transverse cooling while the latter is termed as transverse heating. The conditions which determine the nature of redistribution include the energy (or velocity) of ions, nuclear charges of the crystal atoms and of the ions. Below a certain energy, for ions beyond certain atomic number, the enhancement pattern changes to reduction pattern. This energy is called the transition energy and scales as the square of the nuclear charge of ions. But certain group of ions show only cooling pattern for that same crystal.

Here we report an observation of only cooling patterns utilizing Si and O beams of NSC Pelletron. The experimental setup was almost similar to the ones used in the ref. [1, 2]. Instead of position sensitive gas ionization detector telescope, we used two-dimensional sensitive passivated ion -implanted junction silicon detector. This detector has an active area of 50×50 mm^2 providing an acceptance angle of $\pm 2.3°$ in both the directions. It was fixed on one of the two movable arms inside the general purpose scattering chamber (GPSC).

To produce an isotropic beam of ions, the primary beam was scattered from a Au self-supporting foil of thickness 250 μg/cm^2. The scattered ions after passing through 8.7 μm Si(001) crystal were detected by the above-mentioned detector. The surfaces of

Si crystal and the detector were made planar to each other with the help of a diode laser pointer. Also it was made sure that the centres of Au foil, Si crystal and the detector were collinear and at the level of the beam direction.

Our interest was to see the influence of velocity of the ions passing through the crystal on flux redistribution. The velocity was varied either by changing the energy of primary beam or by changing the scattering angle. Since the Si crystal and detector were fixed on a movable arm, we had the freedom of varying the scattering angle. Mean energy of scattered ions inside the crystal was taken as variable factor. With different combination of scattering angle and beam energy, we could vary the mean energy for Si from 21.3 MeV to 117.0 MeV. Similarly for oxygen, the mean energy could be changed from 23.1 MeV to 49.6 MeV.

A representative flux distribution obtained is shown in Fig. 1. The ratio of projection of spectrum on y-axis was generated and the peak to shoulder ratio was calculated for each case. This ratio was plotted against the velocity of ions (Fig. 2). It is observed that the ratio increases as the mean velocity of the transmitted ions decreases. The nature of variation is similar for both oxygen and silicon quasi-isotropic beam, although we could not vary the energy in case of oxygen as much as we could do for silicon. We got only enhancement pattern in the covered energy regions. In case of silicon we observed cooling pattern even at energies below the scaled transition energy (~15.6 MeV).

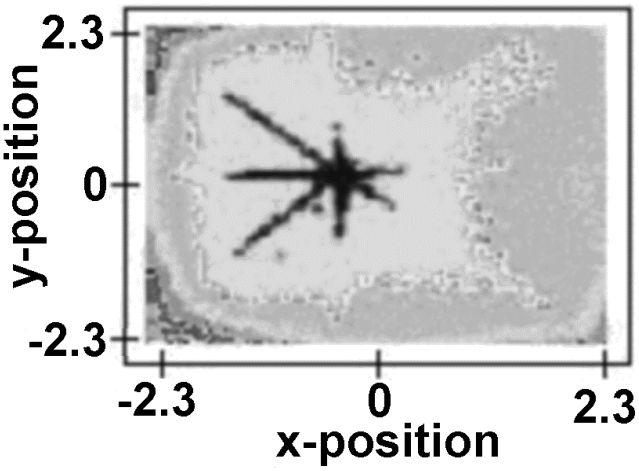

Fig. 1 : Two-dimensional Flux distribution of Si ions after transmission through a 8.7 μm thick Si(001) crystal. The average energy of Si ions in the crystal was about 21 MeV

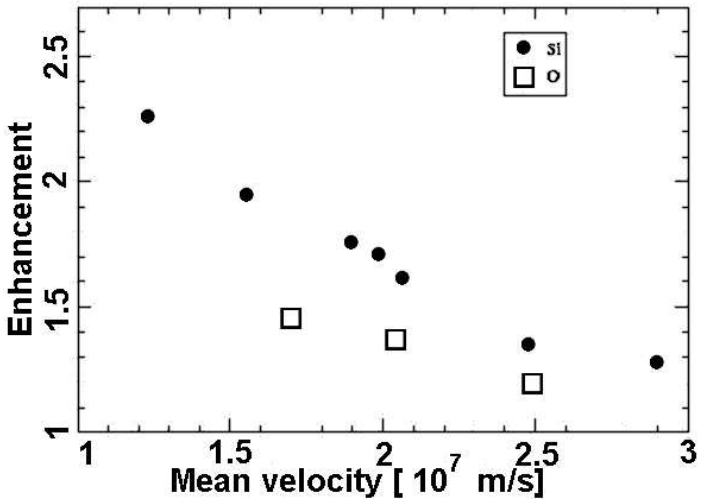

Fig. 2 : Variation of enhancement with mean velocity of ions transmitted through the Si crystal

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5.2.45 Possibility of Online observation of ion beam mixing using LAPSDT based ERDA at NSC – A very initial indication

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LAPSDT has been used to observe the online mixing in lattice matched InGaAs/InP superlattice ($In_{0.53}P_{0.57}As$/InP X 10 periods / InP Substrate). LAPSDT based ERDA experiment has been performed using 150 MeV ions at a recoil angle of 55° in Goniometer Chamber. The projection of *P* band of the ΔE-E spectrum for different ion fluence is shown in Fig. 1. A minimum fluence of 2.27 x 10^{12} ions/cm^2 was necessary to get sufficient statistics. Fig. 1a corresponds to this fluence and shows separate peaks corresponding to different layers. It should be noted that the sample contains *P* only in alternating layers which is reflected in fig. 1a. b, c & d correspond to higher fluences as shown in the respective figures. Higher fluences are chosen as multiples of the initial fluence so as to keep same statistics for all the spectra. Intermixing is clearly observed in these figures, the peaks in the *P* band are getting merged with increasing fluence. This is an indication that the online mixing experiments are possible with this facility. This type of online monitoring is necessary for achieving controlled modification. Moreover one can study

all intermediate fluences rather than some selected fluences as done in off-line measurements on mixing. This is a major advantage of ion beam characterization technique when compared to other off-line characterization techniques. In this work it is found that the mixing effect starts appearing from a fluence of 4.54 x 10^{12} ions/cm^2 in this system if irradiated by 150 MeV Ag ions. Possibility of online observation of ion beam mixing has been shown in this work.

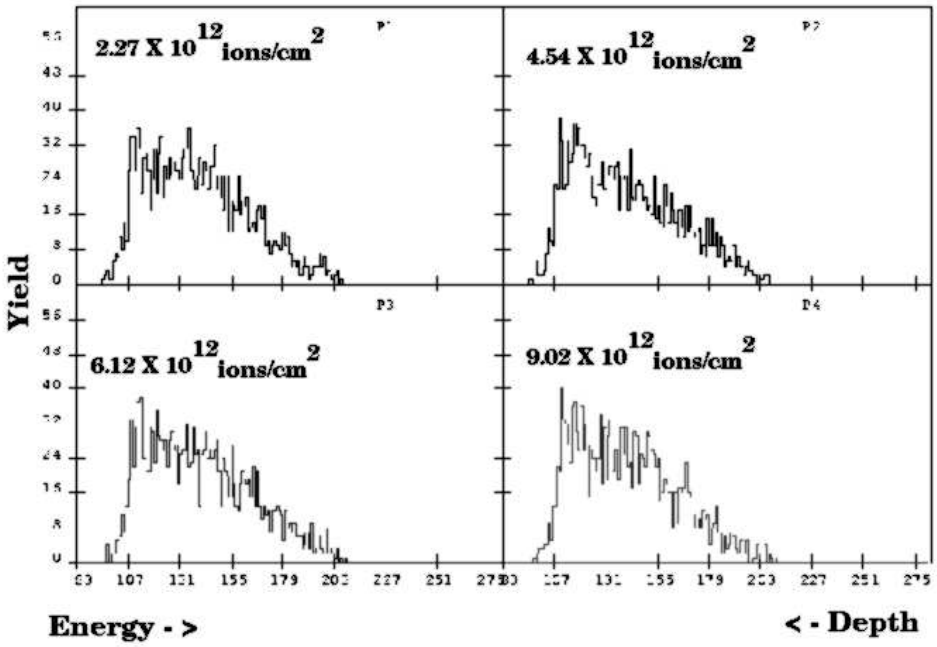

Fig. 1 : Online observation of ion beam mixing using ERDA – LAPSDT (projection of P band) (a) top left; (b) top right; (c) bottom left & (d) bottom right; Corresponding fluence is given in each figure

5.2.46 Heavy Ion Testing of VLSI Devices

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Background

The Space environment of LEO (Low Earth Orbit) and GEO (Geostationary Earth Orbit) contains high energy particulate radiation in the energy levels of a few MeV to hundreds of MeV. The components used in various subsystems of the spacecraft get exposed to these particulate radiation. Though most of the components are inherently tolerant to particulate radiation, the VLSI devices made of sub micron CMOS technology are very sensitive. The high energy particles cause Single Event effects like Single Event Upsets (SEU), Single Event Latchup (SEL) or Single Event Gate Rupture (SEGR) which results in malfunction/ catastrophic failure of the device.

Need for testing

The Spacecraft Subsystems use different VLSI devices such as Microprocessors, peripherals for microprocessors, Application Specific Integrated Circuits (ASIC), Field Programmable Gate Arrays (FPGA), High Density Memories etc in addition to other active devices. ASICs and FPGAs and the subsystems are interfaced with MIL – STD – 1553 Bus Interface configuration which uses 1553 Bus Controller / Remote Terminal Integrated Circuits. Some of the above mentioned devices are available in Radiation Hardened Version and tested at Source by the Manufacturer/ Defense Supply Center, Columbia/ Jet propulsion Lab (JPL)/other space customers. There are a few device types which are not available in Radiation Hardened Version but required to be used in spacecraft system. Such Components are subjected to Heavy Ion Irradiation and tested for its tolerance and suitability in space applications.

Device types tested

Four device types namely 1553 Bus Controllers of M/s DDC and M/s UTMC of USA, a Line Driver and a Line Receiver of M/s National Semiconductors were subjected to Heavy Ion Testing in Pelletron facility at Nuclear Science Centre, New Delhi.

Test facility and relevant details

Heavy Ion Testing was performed at GPSC Beam line in Pelletron. The details of Ions used, Test Setup, Test condition and the Results obtained are tabulated below. Functional Test Setup was made for on line monitoring during irradiation. Test patterns were generated such that it brings out the effects of the Radiation induced faults.

a. Details of Beam parameters

SPICIES	ENERGY	LET Induced in Si	FLUX	FLUENCE
$^{28}Si^{8+}$	110 MeV	11 MeV – cm^2 / mg	370 P / cm^2 / Sec	10^6 P / cm^2
$^{36}Cl^{9+}$	110 MeV	15 MeV – cm^2 / mg	255 P / cm^2 / Sec	10^6 P / cm^2
$^{48}Ti^{10+}$	120 MeV	22 MeV – cm^2 / mg	325 P / cm^2 / Sec	10^6 P / cm^2
$^{107}Ag^{8+}$	65 MeV	40 MeV – cm^2 / mg	9500 P / cm^2 / Sec	10^6 P / cm^2

b. Results summary

Sl. No	Device	Ion	Results	
			SEU	SEL

01	BU61580 Bus Controller (M/s DDC, USA)	$^{36}Cl^{9+}$	Large number of upsets & Functional Failure	Latched-up
		$^{28}Si^{9+}$	Large number of upsets & Functional Failure 1 Upset	No Latch-up
02	Summit 1553 Bus Controller (M/s UTMC, USA)	$^{107}Ag^{8+}$	2 Upsets	No Latch-up
		$^{48}Ti^{10+}$	No Upset	No Latch-up
03	Line Driver / Line Receiver M/s National Semiconductor	$^{107}Ag^{8+}$	No Upset	No Latch-up
04	Line Driver / Line Receiver M/s National Semiconductor	$^{48}Ti^{10+}$	No Upset	No Latch-up
05	Line Receiver M/s National Semiconductor	$^{107}Ag^{8+}$	No Upset	No Latch-up
06	Line Receiver M/s National Semiconductor	$^{48}Ti^{10+}$	No Upset	No Latch-up

c. Observations

127. BU 61580 testing resulted in Latchup and Functional failure for Cl and Large number of upsets & Functional failures for Si. Its LET_{th} for SEU is less than 11 MeV– cm^2/mg & for SEL it is less than 15 MeV–cm^2/mg.

128. 1553 Bus Controller Summit IC from UTMC, when subjected for Heavy Ion testing to Ag, resulted in two types of errors i.e.,

129. (a) Upsets in Configuration Registers
130. (b) Errors in Data Transferred

131. No Latch ups and upsets were observed for Line Driver and Line Receiver, when subjected for Heavy Ion Testing to Ag & Ti

Conclusions

Based on the heavy ion testing it was concluded that

132. BU61580 is not suitable for space applications

133.Summit 1553 controller and the line driver and receivers are suitable for space applications due to their high SEU LET and Latch up immunity.

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[5] Procedure Manual for Operating GPSC Chamber.

5.2.47 Electronic sputtering studies of LiF thin films

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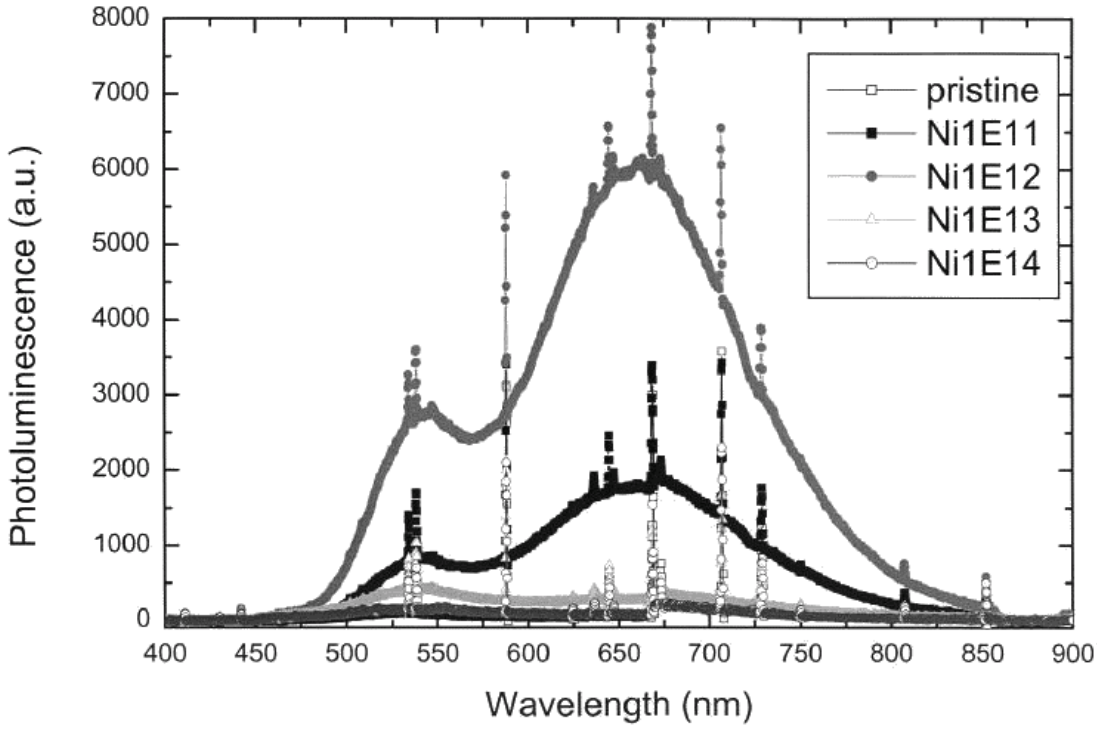
The electronic sputtering of LiF , an ionic insulator, is studied to understand the factors playing a role behind this phenomenon. We plan to understand SHI induced damage produced in LiF and to correlate the electronic sputtering process to the defects created by SHI. The velocity effect on sputter yield is observed in LiF due to bombardment of cluster projectiles with low energy where both S_e and S_n have significant role[1]. Angular distribution of sputtered particles from LiF has been recently studied by Toulemonde et al [2] showing that a jet like component of sputter yield is observed in the direction normal to the target. The creation of defects in LiF has been intensively studied and the primary defects observed in LiF due to impact of SHI [3,4]. At room temperature, the most significant types of defects are F centers (an electron on anion vacancy), F_2 centers and complementary hole centers. These defects were studied by techniques such as optical absorption spectroscopy, small angle x-ray scattering (SAXS) and chemical etching complemented by annealing experiments.

The thin films of LiF with different thickness are deposited by electron beam evaporation unit in the high vacuum deposition chamber of the target development laboratory. The samples studied are deposited on different substrates like Glass (insulator) and Si (semiconductor). In the first part of the experiment LiF samples were irradiated using 150 MeV Ag ions in the high vacuum chamber of the material science beam line. The sputtered species were collected on catcher. The catchers used were glass substrates on

which a Al film was deposited. The catchers were kept at angles of 0, 15, 20, 30, 40, 50, 60, 70, 80 and 90° and a 20 nm Al layer was deposited on the catcher foils afterwards to avoid the collected atoms from sputtering during ERDA study. The catchers are analyzed by on line Elastic Recoil Detection Analysis (ERDA) using 100 MeV Ag beam in the GPSC to find out angular distribution of sputter yield. A SSBD was also mounted at 45° inside the chamber to detect H and Li particles. The large area position sensitive telescope detector was used at 60^0 for the analysis and isobutane gas at a pressure of 13 mbar was used. In the two-dimensional ΔE-E spectrum, the mass bands corresponding to Li, C and F were clearly resolved. The detailed analysis of the spectra is underway.

The nature of radiation damage in LiF samples is studied by irradiating the samples with 150 MeV Ag and 90 MeV Ni beam at fluences from $1x10^{11}$ to $2x10^{13}$ ions/cm^2. The defects induced by ion beam are studied using optical absorption spectroscopy. The photoluminiscence characteristic of the films irradiated wih Ni beam has been studied and the spectra for the four cases is shown in Figure. Preliminary studies show that there is an enhancement in the photoluminiscence intensity due to F_2 color centres after irradiation by Ni beam. The maximum intensity is observed at a fluence of $3x10^{11}$ ions/cm^2. After this the peak intensity due to F_2 colour centres reduces, and disappears completely at fluences above $1x10^{13}$ ions/cm^2, possibly due to self annealing of the defects generated. Further studies are in progress.



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