

5.2 MATERIALS SCIENCE

There have been a large number of experiments in materials science with energetic ion beams on the problems mainly related to electronic sputtering, ion beam mixing, nanostructuring of the materials, surface modifications, materials modifications, ion beam induced epitaxial crystallization etc.

There were five on-line ERDA facility experiments on (i) hydrogen content measurement in Niobium used in LINAC resonator cavities, (ii) loss of oxygen in Indium oxide due to SHI irradiation, (iii) loss of oxygen in zinc oxide having different grain size, (iv) composition analysis of pristine and low energy ion irradiated glass and (v) nitrogen loss in iron nitride, aluminum nitride and nickel nitride. The irradiation chamber and in-situ XRD facility in beamhall II were used in 3 user experiments this year. Enhanced hydrogenation in SHI irradiated Pd-Pr layer was observed by ERDA. The evolution of F and CF gases were monitored by on-line QMA in irradiation of teflon.

Several ion beam mixing experiments were performed. From the ion beam mixing experiments performed at IUAC this year and previous years, an overall picture appears that ion beam mixing occurs if the ion beam creates track in one of the two systems/layers. However, if the heat of mixing of the system is not favorable, the ion beam mixing does not take place even if both the components of the bi-layer have track creation by ion-beam as observed in Fe/Bi case. Surface studies of the SHI irradiated ultra thin Au films were performed and is under investigation.

Experiments on damage cross section of fullerene C_{70} by swift heavy ions were performed. Surface plasmon tuning of the Ag- C_{60} nanocomposite thin films was demonstrated by ion irradiation. Metal film on silica was irradiated by low and high energy ions to get plasmonic properties. The enhancement in PL was observed in PbS nanoparticles embedded in conducting polymer matrix due to SHI irradiation. SHI irradiation studies were performed on nanoclay-PVDF nanocomposite.

The amorphous TiO_2 film was shown to get transformed to nanocrystalline as a result of swift heavy ion irradiation. Phase transformation from amorphous TiO_2 to a mixed rutile and anatase phase was observed by ion irradiation. The nanostructures appears at the surface of semiconducting oxide thin film of SnO_2 after swift heavy ion irradiation. There were indications of elongation of ZnS nanoparticles by high energy heavy ion irradiation. SHI irradiation induced recrystallization of Ge implanted in Si was studied and the work is in progress.

Nanostructures at surface of InP were created by 1.5 keV atoms and 500 keV Ar ions. The shape was circular or ripples depending on the angle of incidence. $Si_{1-x}Ge_x$ nanostructures embedded in silica matrix were synthesized by atom beam co-sputtering followed by annealing. Room temperature ferromagnetic behavior was observed in Ni implanted ZnO thin

films. Low energy He and H ion irradiation studies were performed to investigate blistering phenomenon.

SHI induced modifications were studied in NiO, Al₂O₃, YBCO, borosilicate glass, metal polymer nanocomposite, polypropylene, conducting polymers, PMMA etc. Studies on SHI irradiated nanostructured titanates of Ba and Sr were performed for photochemical splitting of water. SHI irradiated polymer was used for graft monomers on the irradiated polymers. A large number of experiments on the straggling and energy loss measurements were performed in mylar, polypropylene, polycarbonate and kapton. The electronic circuits for devices in spacecraft were tested for radiation damage, to simulate the space radiation. The electrical characterization studies of the pristine and irradiated high speed NPN power transitions and bipolar junction transistors were carried out.

Apart from a school and a workshop in the area of ion beams in materials science at IUAC, an Indo French conference on Nanostructuring by ion beams was organized, jointly by IUAC Delhi and CSNSM Orsay, fully financed by Indo French Centre for Promotion of Advanced Research.

5.2.1 Enhanced hydrogenation in SHI irradiated Pd-Pr layers

Shubhra Kala¹, B. R. Mehta¹, S. A. Khan² and D. K. Avasthi²

¹Department of Physics, IIT, Delhi, New Delhi

²Inter-University Accelerator Center, New Delhi

Hydrogen-induced reversible metal-semiconductor transition between dihydride and trihydride states in rare earth (RE) metals is unique in terms of simultaneous changes in optical and electrical properties. This phenomenon in RE known as switchable mirror effect can be utilized in a number of technological applications, such as, hydrogen storage and solid state display devices.[1] In general, these applications require faster and increased H incorporation during loading followed by complete or maximum removal during deloading. Surface/subsurface sites, defects, lattice plane edges and corners are preferred H active sites and are known to enhance the hydrogen interaction as compared to the conventional interstitial sites.[2] Irradiation with swift heavy ion (SHI) has recently emerged as a new technique for nanometer structuring as also for modifying nanostructuring owing to controlled and localized modification of materials.[3]

In the present work, the effect of post-deposition SHI on the structural and hydrogenation characteristics of Pd-Pr thin films, prepared by vacuum evaporation, has been investigated. Pd overlayer is essentially required to protect the Pr layer against oxidation and to catalyze hydrogen absorption and desorption kinetics. Swift heavy ion irradiation was carried out by 120 MeV Ag⁺¹⁰ ions using a 15 UD Pelletron tandem accelerator. Five samples were irradiated

with ion fluences of 1×10^{10} , 1×10^{11} , 1×10^{12} , 5×10^{12} and 1×10^{13} ions/cm² and one sample was kept un-irradiated. Hydrogenation properties of these samples were investigated by acquiring hydrogen concentration profiles by elastic recoil detection analysis.

During hydrogen loading, on acquiring PrH₃ state, a large enhancement in the H stoichiometry from 3.64% to 17.81% is observed with the increase in ion fluence from 1.0×10^{10} to 1.0×10^{13} ions/cm², with respect to the un-irradiated sample. On subsequent deloading, 31% H gets desorbed from SHI irradiated samples in comparison to only 12% in case of un-irradiated sample. Hydrogen active layer thickness, which is participating in H loading/deloading process, also increases on SHI irradiation. Creation of defects inside the samples on SHI irradiation has been indicated by X-ray diffraction studies and further confirmed by TEM analysis. TEM studies revealed that the damage zones is confined to about 5-6 nm in diameter, therefore, can be associated with the passage of a single ion and thus, corresponds to a latent track. As, H stoichiometry of ~ 2 is observed in samples irradiated with an ion dose $\geq 10^{11}$ ions/cm², therefore, a small number of ion tracks (about 1%) are sufficient for complete removal of incorporated H, corresponding to PrH₃ to PrH₂ transformation. The hydrogen active defect sites along ion tracks provide efficient and two-way transport routes for H diffusion, both during loading and deloading, resulting in increased incorporation, complete removal and larger active layer thickness. On contrary to this, in un-irradiated sample, small amount of H incorporation during loading and inefficient H removal during deloading is due to crystallite interlocking.[4]

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5.2.2 SHI Induced Effect on FeN/Si System

Renu Dhunna¹, Chhagan Lal¹, S.A. Khan² and I.P. Jain¹

¹Centre for Non-Conventional Energy Resources, University of Rajasthan, Jaipur

²Inter University Accelerator Centre, New Delhi

The iron nitride thin film (80nm) was deposited on Si (100) by reactive ion beam sputtering. Total gas flow rate was kept 3 sccm and the ratio of argon and nitrogen gas was kept 2.4:0.6. During the deposition overall pressure of argon and nitrogen was 1.2×10^{-4} torr. Thin films of FeN were irradiated by Au⁺⁸ at 1 pna current with 100 MeV energy from the Pelletron accelerator of Inter University Accelerator Centre, New Delhi.

In order to identify the formation of new phases induced in FeN/Si system by Au ion irradiation, GIXRD spectra were accumulated. GIXRD analysis of as deposited sample indicated the amorphous nature of the film. After the irradiation of this system by Au ions at 3×10^{12} ion/cm² fluence a broad maximum around $2\theta \approx 42.8^\circ$ is obtained which also shows amorphous nature of the film.

On-line ERDA technique with a large area ΔE -E detector telescope is employed to monitor the change of nitrogen content of the films. For FeN/Si system, the total accumulated fluence of projectile ions was of the order of 1×10^{13} ions/cm². After the irradiation of FeN film, nitrogen content is not depleted as shown in figure 1. A possible explanation of this is that the volume density of activated nitrogen radical is never large enough, that significant number of N₂ molecules could be generated. Alternatively, the nitrogen radicals may be re-incorporated more readily. Iron content of the film remains also same within the experimental error ($\pm 10\%$).

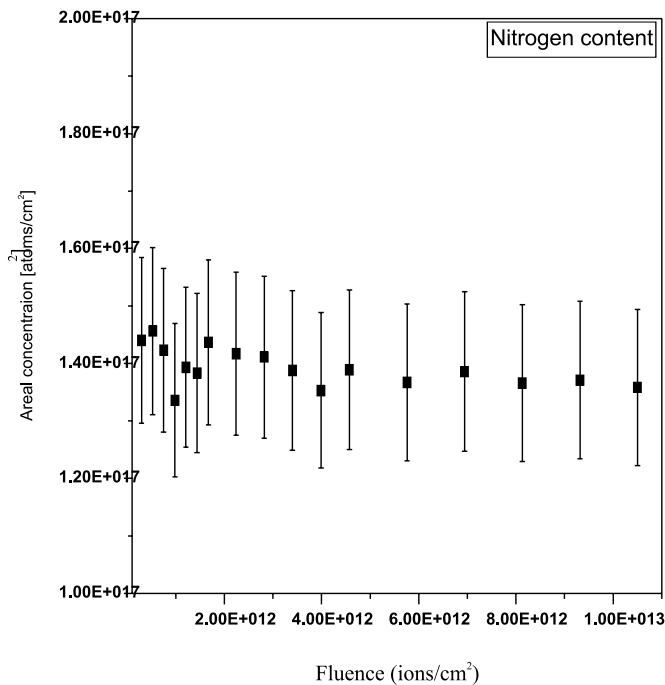


Fig. 1. Nitrogen content versus fluence

The magnetic properties of the samples were characterized by MOKE at room temperature. Measurements in the longitudinal geometry were performed with the magnetic field applied in the sample plane either in the plane of incidence (parallel magnetization). The coercivity of as-deposited sample is found 22.32 Oe. After irradiation coercivity of FeN/Si system is obtained 17.85 Oe. Swift heavy ion irradiation results in significant atomic rearrangements. The observed effects include defects creation or annihilation, track formation, intermixing or sputtering due to which coercivity of irradiated sample decreases.

5.2.3 Swift heavy ion induced surface modifications of gold thin films

P. Dash¹, P. Mallick¹, H. Rath¹, Jai Prakash², A. Tripathi², D.K. Avasthi², P.V. Satyam³ and N.C.Mishra¹

¹Department of Physics, Utkal University, Bhubaneswar

²Inter-University Acceleration Centre, New Delhi

³Institute of Physics, Bhubaneswar

Current studies have indicated that in metallic thin films and multilayers, the effects of electronic energy loss of swift heavy ions (SHI) are significantly enhanced as compared to bulk materials. Gupta and Avasthi [1] found an enhanced electronically mediated sputtering in Au thin films compared to the sputtering mediated through nuclear energy loss. Satpati et al [2] found higher sputtering in nano dispersed Au target than in continuous Au thin films when bombarded with 100 MeV Au ions. In the present study, we probe into the surface modification of ultra-thin Au films under the impact 58 MeV Ni and 107 MeV Ag ions, where confinement of the energy deposited by the energetic ions is expected to produce enhanced sputtering yield. The energies of the ions were so chosen to avoid the velocity effect in ion-matter interaction.

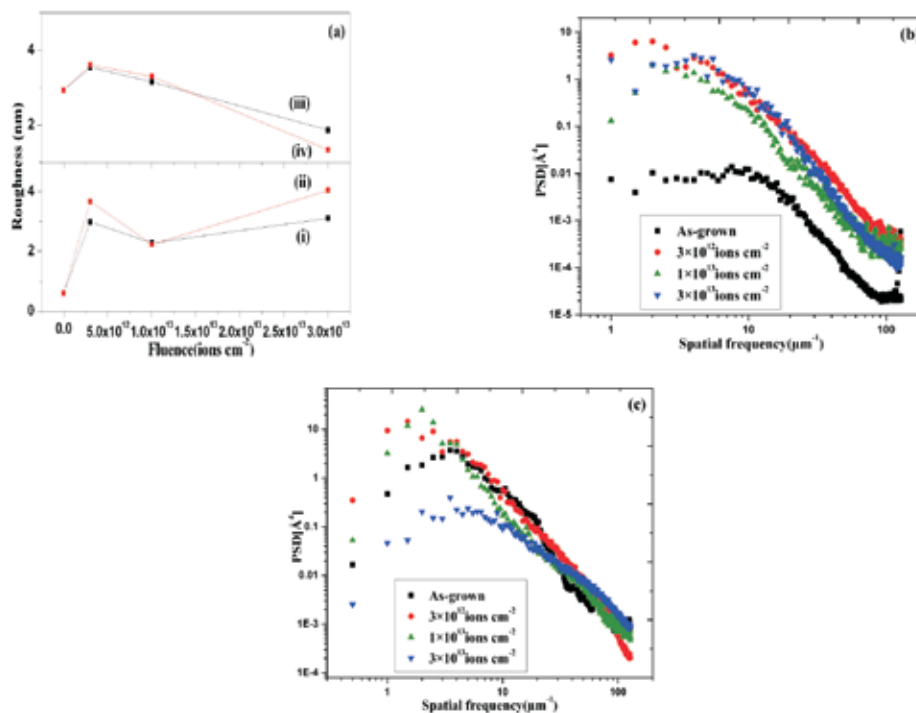


Fig.1. (a) Roughness variation of 10 nm and 20 nm Au film with fluence for (i) and (iii) 58 MeV Ni (ii) and (iv) 107 MeV Ag respectively. (b) and (c) Plots of PSD function versus spatial frequency q of 10nm and 20 nm Au surfaces for as grown as well as irradiated at different fluences by 58 MeV Ni ion respectively

The surface roughness as estimated from the AFM analysis did not show monotonic variation with irradiation fluence for both the film thicknesses (Fig. 1). The nature of variation however is same for the two ion beams, but the extent of variation was higher for Ag ions with higher S_e than that for Ni ions. The increase of roughness at low fluences in both the films seems to arise due to sputtering by individual ions. At high fluences, overlap of damaged zones smooth the surface and reduce the roughness. At the highest fluence, roughness in 10 nm film increases while that in 20 nm film continue to decrease. This widely different behavior in the two cases points to the importance of the film thickness and confinement of energy deposited by SHI in the films. Power spectral density analysis of the AFM images provides a scaling exponent δ in the range 2-3.71 for Ni and Ag ion irradiated 10 and 20 nm thick Au films. These δ values showed that surface morphology of the gold irradiated samples is governed by the combined effect of evaporation-recondensation and diffusion dominated processes.

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5.2.4 Role of melting temperature in intermixing of miscible metal/metal bilayers induced by swift heavy ions

A. Gupta¹, R. S. Chauhan¹, D. C. Agarwal¹, S. Kumar², S. A. Khan³, S. Mohapatra³, A. Tripathi³, T. Som⁴ and D. K. Avasthi³

¹Department of Physics, R. B. S. College, Agra

²Department of Physics, CITM, Aravali hills, Sector-43, Faridabad

³Inter-University Accelerator Centre, New Delhi

⁴Institute of Physics, Sachivalaya Marg, Bhubaneswar

Ion irradiation is considered to be a tool for the creation and the tailoring of new materials with novel properties because of the high energy density deposited per incident ion and the capability of such violent process to drive the solid far from the equilibrium [1]. In metals damage creation occurs when the electronic energy loss, exceeds a certain threshold value, which may vary from metal to metal. Thus, SHI induced defect production and atomic motion in metals has stimulated great interest to understand the interfacial mixing across the metal/metal interfaces to produce novel materials and phases [2, 3]. Recently, Wang *et al.* [in Ni/Ti] and Kumar *et al.* [in Ti/Au] have reported mixing across the interfaces of a metallic bilayer systems [2, 3] due to inelastic thermal spike [4], consisting of one S_e -sensitive and another S_e -insensitive metals, by heat exchange along the ion track. In both the

cases, the melting temperature of S_e -insensitive material was lower than that of S_e -sensitive material. These studies motivated us to study SHI induced interface mixing in a metallic bilayer system where the S_e -insensitive material has a higher melting temperature than that of the corresponding S_e -sensitive layer. This would help to check for the applicability of the hypothesis of heat exchange across the interface along the ion track under the present configuration (Ti/W bilayer system).

Bilayer samples were prepared in an ultra high vacuum chamber by using electron beam evaporation technique. Thicknesses of 50 nm Ti and 49 nm W films were sequentially deposited on a Si (100) substrate without breaking the vacuum. The samples were irradiated by 120 MeV Au ions at different fluences ranging from 2.5×10^{12} to 2.5×10^{13} ions cm^{-2} at room temperature (RT) using the 15 UD Pelletron accelerator at Inter-University Accelerator Centre (IUAC), New Delhi. The thickness and the composition of the pristine and the irradiated samples were analyzed by means of RBS using 1.35 MeV He^+ ions at Institute of Physics, Bhubaneswar.

Typical RBS spectra of Ti/W bilayer sample before and after irradiation with 120 MeV Au ions at different fluences are shown in Figure 1. The spectra were simulated with the help of the 'RUMP' simulation code. The concentration profiles were fitted with a Gaussian error function and the variances were determined. It is observed that variance remain constant with increasing ion fluence (as shown in figure 2), which indicates that no significant mixing takes place in case of Ti/W bilayer system. This is accompanied by a continuous reduction in the W-peak area with increasing ion fluence, while the Ti-peak area remains same within the statistical limit. This could be attributed to SHI-induced sputtering of the partially oxidised W thin film.

In absence of any mixing across the Ti and W interface, which is otherwise a thermodynamically miscible system, we make a comparison with the similar type of Ti/Au bilayer system where Ti (melting temperature 1941K) is S_e -sensitive and Au (melting temperature 1337 K) is S_e -insensitive. It was shown that mixing took place due to lower melting point of Au than Ti and the energy transfer from Ti to Au during transient thermal spike [3].

In the case of Ti/W system, thermal spike calculation for W shows that the spike temperature reaches up to 2027 K for a 1 nm cylindrical shell, which is 1688 K lower than its melting temperature of 3695 K. Hence, an excess thermal energy, equivalent to a temperature of 1688 K is required to melt W. Due to the large temperature gradient at the interface the heat energy is transferred from Ti to W, but this energy is insufficient to melt W because melting temperature of S_e -insensitive material W is higher than the melting point of S_e -sensitive material Ti. Hence the intermixing does not occur at the interface in Ti/W system. We conclude that mixing between the S_e -sensitive and the S_e -insensitive materials occurs if the energy transferred by S_e -sensitive material across the interface is sufficient to melt the

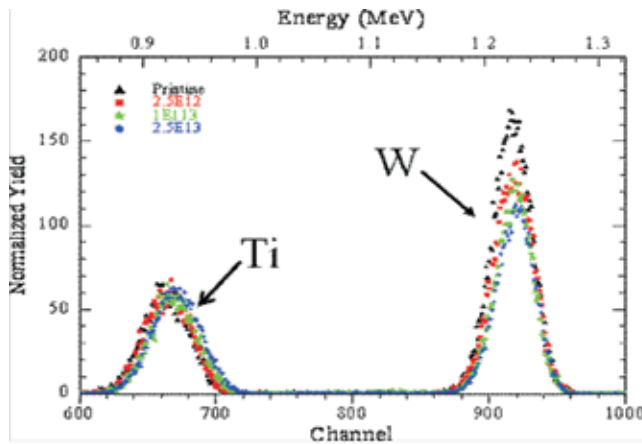


Fig. 1. RBS spectra of Ti/W film on Si (100) substrate after being irradiated with 120 MeV Au ions of different fluences at RT

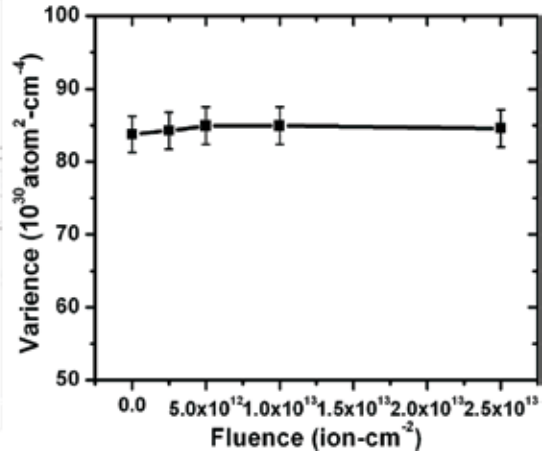


Fig. 2. Variation of variance with ion fluence

S_c -insensitive material and mixing does not occur if the melting point of the S_c -insensitive material is larger than the melting point of the S_c -sensitive material.

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5.2.5 Swift Heavy Ion Induced Modification in Bi/Te Bilayer System

Th.Diana¹, D.K.Avasthi² and H.Nandakumar Sarma¹

¹Department of Physics, Manipur University, Imphal

²Inter-University Accelerator Centre, New Delhi

Bismuth telluride compounds find wide application in thermoelectric applications like thermoelectric generators and coolers due to its high figure of merit ZT . Due to miniaturization of electronic devices, bismuth telluride thin films have attracted considerable attention. Bismuth telluride thin films have been prepared by various methods like flash evaporation[1], sputtering[2], molecular beam epitaxy[3], vacuum evaporation etc. However, it has not yet been prepared and studied using SHI far to our knowledge. Thus, it is of great interest to investigate the possibility of mixing in the Bi/Te bilayer system by SHI. It is observed from

the previous works that the ion beam mixing occurs if the ion beam creates track in one of the layers. Track formation is seen in bismuth [4]. In order to study SHI induced mixing in bilayer, we have deposited Bi and Te thin films over well-cleaned glass substrate by thermal evaporation at room temperature. The samples are irradiated with 100 MeV Ag⁷⁺ ions under the fluences 10¹², 3x10¹², 10¹³, 3x10¹³, 10¹⁴ using 15 UD pelletron at IUAC, New Delhi.

Irradiated as well as pristine samples are characterized by XRD technique with CuK α -radiation (1.54Å) using Bruker D8 advance XRD in the scan range of 15-60° and scan speed of 0.5 deg/min at IUAC, New Delhi. The results show that the sample has crystalline phase with peaks corresponding to that of bismuth telluride Bi₂Te₃. Some oxides are also indicated. It is observed that after irradiation the absorption decreases in the visible region. Further we need to take Rutherford Backscattering Spectroscopy for the compositional thin film analysis.

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5.2.6 Modifications of optical and microstructural properties of silver-fullerene C₆₀ nanocomposite by swift heavy ions

R. Singhal¹, D. C. Agarwal¹, Y. K. Mishra², S. Mohapatra³, A. K. Chawla⁴, J. C. Pivin⁵, R. Chandra⁴ and D. K. Avasthi¹

¹Inter University Accelerator Centre, New Delhi

²Functional Nanomaterials, Institute for Materials Science Christian-Albrechts-University Kaiserstraße 2, 24143 Kiel, Germany

³University School of Basic and Applied Sciences, GGS Indraprastha Univ., Delhi

⁴Institute Instrumentation Centre, IIT, Roorkee

⁵CSNSM, IN2P3-CNRS, Batiment 108, F-91405 Orsay Campus, France

The optical and microstructural properties of silver-fullerene C₆₀ nanocomposite and their modifications induced by swift heavy ion irradiation are reported.

Thin films of Ag-C₆₀ nanocomposite were deposited by evaporating Ag and C₆₀ simultaneously from two crucibles. Films were grown on different substrates such as glass, Si and carbon coated Cu grids in a high vacuum chamber. These nanocomposite films on glass substrate and TEM grids were irradiated with 120 MeV Ag ions delivered by 15 UD Pelletron

accelerator at Inter University Accelerator Centre, New Delhi in material science beam line. In the case of 120 MeV Ag ions, the electronic (S_e) and nuclear (S_n) energy losses in fullerene C_{60} are $\sim 1.0 \times 10^3$ and 3.8 eV/\AA respectively and the range of Ag ions in C_{60} is $\sim 18.2 \mu\text{m}$ as calculated by SRIM 2003 programme.

The Ag atomic fraction and film thickness, estimated by Rutherford backscattering spectroscopy were found to be 28 % and 55 nm. Figure 1 shows the bright field images of pristine and 120 MeV Ag ions irradiated films at different fluences. Almost spherical particles can easily be seen in both, the pristine and irradiated films. The average particle size $\langle D \rangle$ was found to be 6 nm for pristine sample and 8, 9 and 10 nm for the films irradiated at the fluences of 6×10^{12} , 1×10^{13} and 3×10^{13} ions/cm² respectively. The absorption spectra of these films are shown in figure 2. With increasing fluence, SPR peak is blue shifted from 522 to 473 nm with a little decrease in FWHM. According to Mie theory, a growth of Ag NPs from 6 nm to 10 nm can give a red shift to the SPR wavelength by only 2-3 nm whereas in present case, SPR is blue shifted ($\sim 49 \text{ nm}$) with ion irradiation. It is due the transformation of fullerene C_{60} to amorphous carbon with ion irradiation. In the Raman spectra, at and above a fluence of 3×10^{12} ions/cm², vibrational Raman modes of C_{60} are almost vanished and two broad bands at 1380 and 1560 cm⁻¹ start appearing which are D and G bands of amorphous carbon. The transformation of fullerene C_{60} matrix into amorphous carbon network changes the dielectric function of the matrix which causes blue shift to the SPR wavelength.

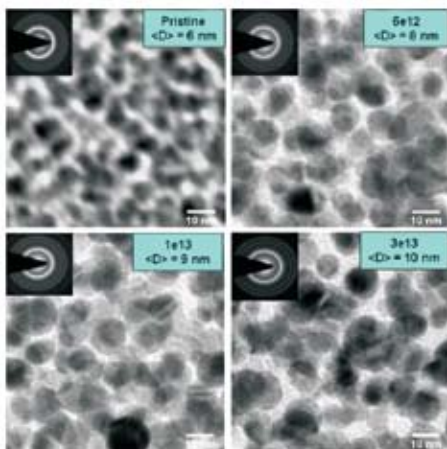


Fig.1. Bright field images of pristine and 120 MeV Ag ions irradiated films at the fluences of 6×10^{12} , 1×10^{13} and 3×10^{13} ions/cm²

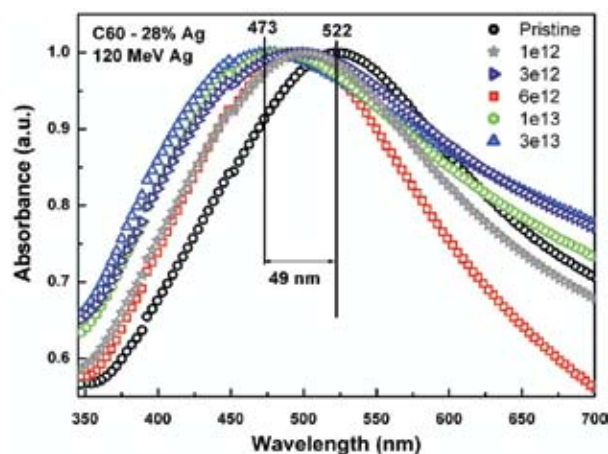


Fig.2. Absorption spectra of pristine and 120 MeV Ag ion irradiated films of C_{60} -Ag nanocomposite showing a blue shift ($\sim 49 \text{ nm}$) in the SPR wavelength of Ag nanoparticles

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5.2.7 Investigation of MOS capacitor embedding Au nanoparticles

Y. S. Katharria, R. Singhal, Y. K. Mishra, D. C. Agarwal and D. K. Avasthi

Inter-University Accelerator Centre, New Delhi

High density of states around the Fermi level, a wide variety of available work functions and smaller energy perturbations due to carrier confinement make metal nanoparticles a better candidate for charge storage as compared to their semiconductor counterparts. Owing to these advantages, MOS structures embedding Pt, Au, Ni, Co, Al, W, Ru etc. are being extensively explored for their charge retention property [1]. In the present work, we investigated charge trapping behavior of MOS structure containing Au nanoparticles.

Au-SiO₂ films with 100 nm thickness were grown on n-type Si (100) substrates using atom beam co-sputtering method. Rutherford backscattering spectrometry showed Au content of 10at.% in these films. A 40 nm thick film of SiO₂ were grown to have a control gate oxide to enable charge retention in the Au nanoparticles. Thermal evaporation was used to get Au gate contact of 2 mm diameter above SiO₂ layer and an Al Ohmic contact on Si side. A Boonton 7200 capacitance meter working at 1 MHz was used to investigate capacitance-voltage (C-V) characteristics of the samples. Current-voltage (I-V) measurements were done using Keithley 2400 source meter unit.

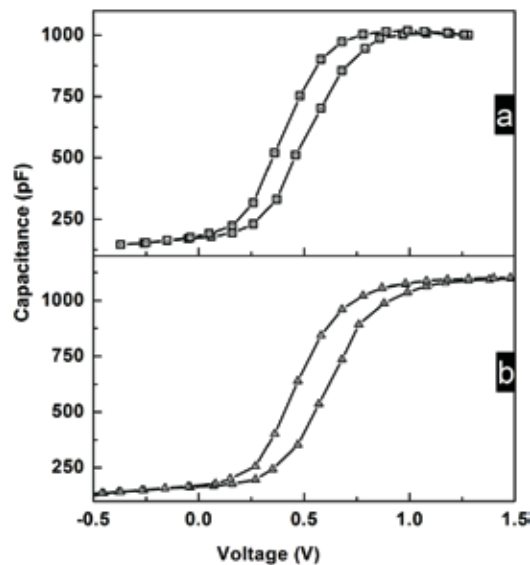


Fig.1. C-V characteristics of MOS capacitor containing Au nanoparticles with voltage sweep of (a) -0.2 to 1.3 V, and (b) -2 to 2 V.

Fig.1 shows the CV characteristics of the MOS capacitor containing Au nanoparticles. The voltage is first swept from inversion/deep depletion to accumulation region (from -ve gate bias towards +ve) of the MOS and then back to the inversion/deep depletion region. The

MOS devices show accumulation typically at gate voltage $V_G > 1$ V and a hysteresis as the voltage is swept back to obtain inversion region. The hysteresis corresponds to trap densities of $3.4 \times 10^9 \text{ cm}^{-2}$ and $4.9 \times 10^9 \text{ cm}^{-2}$ for the voltage sweeps defined in Fig.1(a) and (b).

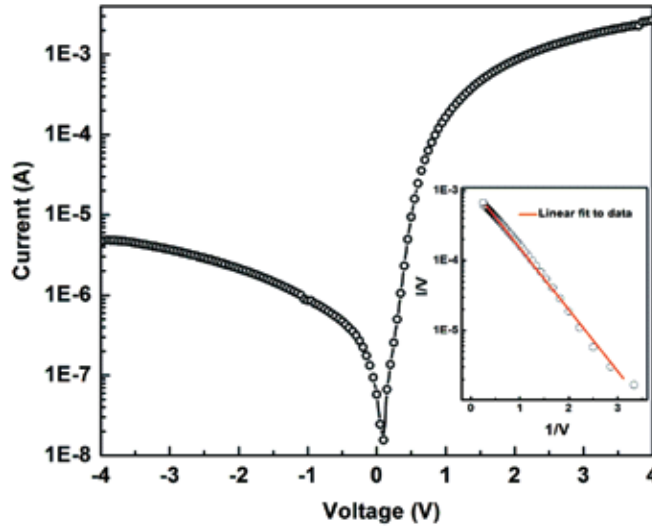


Fig.2. I-V characteristics of MOS capacitor. Inset shows of a plot of $\ln(I/V) - 1/V$ for +ve gate voltages.

I-V characteristics of the MOS capacitors voltage sweep from -4 to +4V are shown in Fig.2. High density of defects inside the gate oxide layer could be responsible for the high value of leakage current density. A linear behavior of $\ln(I/V)$ with $1/V$ under +ve gate bias implies that the current transport occurs predominantly through direct tunneling mechanism of substrate electrons into the nanoparticles.

In summary, we have shown that MOS capacitor fabricated with co-evaporated Au-SiO₂ can also be used for charge retention.

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5.2.8 Swift heavy ion modification of noble metal nanoparticles in fused silica glass prepared using low energy ion beam

Ranjana S. Varma¹, D. C. Kothari¹, P. Kumar², D. Kanjilal², S. S. Santra³ and R. G. Thomas³

¹Department of Physics, University of Mumbai, Mumbai 400 098

²Inter-University Accelerator Centre, New Delhi

³Bhabha Atomic Research Centre, Trombay, Mumbai

Metallic nanoparticle embedded in a transparent dielectric medium have the potential for applications in areas such as nonlinear optical devices, fast optical switching devices and optical sensor [1]. In this work, Instead of direct annealing, we have used Swift heavy ion irradiation to observe the annealing behavior along the ion track. The purpose of this paper is to report the predominant effect of electronic excitations in the nucleation and growth by performing irradiation with 120 MeV Ag^{9+} ions of noble metal particles M/ SiO_2 films (where M= Ag, Au or Cu) beam mixed with low energy. Thin metal film (Ag, or Au, or Cu) of 15 nm is deposited on fused silica glasses using thermal evaporation technique. These samples were low energy (LE) beam mixed using 100 keV Ar^+ ions at a current of about 1 μA to avoid sample heating and sputtering during ion irradiation to nominal doses: 1×10^{13} , 1×10^{16} ion/ cm^2 . All these samples were irradiated using swift heavy ions (SHI) 120 MeV Ag^{9+} ions at the Pelletron accelerator at the Nuclear Science Centre (NSC) in New Delhi.

RBS spectra were recorded for LE irradiation followed by SHI irradiated of noble metal M/ SiO_2 films (M=Ag, Au, and Cu respectively) deposited on fused silica glasses and the un-irradiated samples. The diffusion length calculated for Ag, Au, and Cu are 23, 14, 34 nm respectively, after LE irradiation followed by SHI irradiation. RBS data shows that LE irradiation followed by SHI irradiated causes the loss of 58.6 %, 53.0 %, and 12.1 % of the entire Ag, Au, and Cu contents in the samples respectively, because of sputtering of metal from the surface of sample. The maximum metal atomic concentration occurs near the interface of the film. The metal concentration in fused silica glasses for samples irradiated with LE followed by SHI irradiation obtained after simulation using RUMP code [33], are 15, 27 and 23 (at. %) for Ag, Au and Cu respectively. The average size of the nano-hillocks obtained from AFM analysis for sample irradiated with 120 MeV Ag^{9+} ion at a fluence of 5×10^{12} ion/ cm^2 and pre-irradiated with 100 keV Ar^+ ion at 1×10^{16} ion/ cm^2 is about 200 nm for Ag and Au film, and about 160 nm for Cu film.

The optical absorption spectra were recorded for Ag/ SiO_2 , Au/ SiO_2 and Cu/ SiO_2 samples after LE beam mixing at a fluence of 1×10^{16} ion/ cm^2 followed by SHI irradiation at a fluence of 5×10^{12} ion/ cm^2 . It is seen that the sample displays higher intensity SPR peak after SHI irradiation. The LE beam mixed sample at a fluence of 1×10^{13} ion/ cm^2 does not show any SPR peak before and after SHI irradiation at a dose of 5×10^{12} ion/ cm^2 (not shown here), only peak due to SHI created defect is observed. The averaged particle size was calculated using Mie's theory [2]. Table 1 gives a summary of the SPR wavelengths and deduced sizes of metal nanoparticles. A shift towards lower wavelength corresponds to the decrease in the size of nanoparticles [2], whereas we have observed increase in the size which is possibly due to the elongation Parallel to the incident light, as observed by Penninkhof [3]. The thermal spike created during SHI irradiation results in plastic flow of silica, which induces an in plane stress perpendicular to the ion beam direction [4]. The in-plane stress acting on metal nanoparticle results in its elongation along the direction of ion beam.

Table 1: The size of the Ag, Au, Cu nanoparticle calculated using Mie's theory

Samples	Peak position		Radius using Mie's theory	
	LE beam mixing	LE beam mixing followed by SHI irradiation	LE beam mixing	LE beam mixing followed by SHI irradiation
Ag:SiO ₂	423.2	401.5	1.58	2.17
Au:SiO ₂	577.7	567.3	1.88	2.45
Cu:SiO ₂	624.4	610.7	9.83	10.38

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5.2.9 Study of formation of Cu nanoparticle using Swift Heavy Ions

Ranjana S. Varma¹, D. C. Kothari¹, R. J. Choudhari², Ravi Kumar², R. Tewari³ and G. K. Dey³

¹ Department of Physics, University of Mumbai, Mumbai

² Inter-University Accelerator Centre, New Delhi

³ Bhabha Atomic Research Centre, Trombay, Mumbai

Composite materials consisting of nanometer-sized metallic particles embedded in insulating matrices are interesting for optoelectronics due to their non-linear optical properties [1]. In the present work, combined use of keV and MeV ion beams is made to form nanoparticles in fused silica glass. Low energy ion beams are used to create nucleation sites and high energy ion beams are used to possibly induce electronic energy loss mixing. The sequence of process being: low energy irradiation of fused silica substrate, Cu thin film coating, SHI irradiation and annealing. Fused Silica substrates were irradiated using 30 keV argon ions at fluences of 1×10^{17} ion/cm². 10 nm Cu films were deposited on the low energy irradiated fused silica. These thin films coated fused silica samples were then irradiated using 120 MeV Ag⁹⁺ ions at fluences ranging from 4×10^{13} to 1×10^{14} ion/cm². The size of Cu nanoparticles estimated from optical absorption spectra and from GAXRD measurements correlate well and the average size is ~ 7.8 nm. Fig. 2 shows the transmission

electron microscopy image of 1200 °K annealed sample irradiated at a fluence of 1×10^{14} ion/cm². The average size of the nanoparticle from TEM is 8 nm.

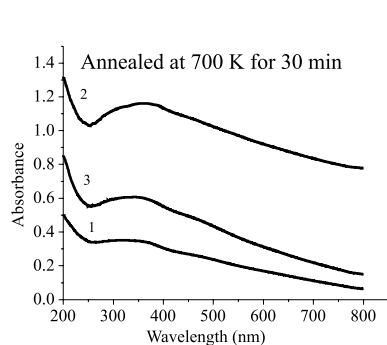


Fig. 1. UV-Vis spectra of SHI irradiated samples after annealing in Ar atm. at 700 °K, for different fluences (1) 4×10^{13} ion/cm² (2) 6×10^{13} ion/cm² (3) 1×10^{14} ion/cm²

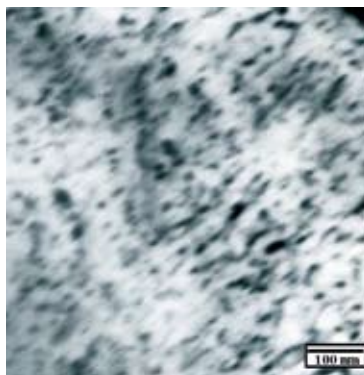
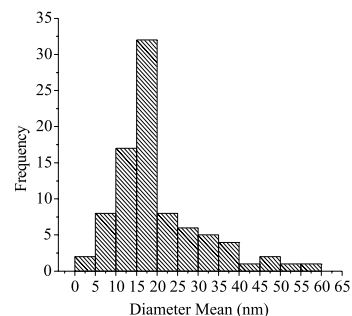


Fig. 2. TEM image of the sample SHI irradiated at a fluence of 1×10^{14} ion/cm² and annealed at 1200 °K for 30 min in Ar atmosphere and Size distribution of Cu nanoparticles obtained from the TEM image.



The study suggests that irradiation induced defects provide nucleation sites for the formation of nanoparticles, latent tracks provide fast diffusion channels for Cu and, annealing provides required energy for the diffusion.

5.2.10 Surface patterning on Indium Phosphide with low energy bombardment : an evolution from nanodots to nanoripples

I. Sulania¹, A.Tripathi¹, D. Kabiraj¹, S.Varma², M. Lequeux³ and D.K.Avasthi¹

¹Inter University Accelerator Centre, New Delhi

²Institute Of Physics, Bhubaneshwar

³Grenoble University, France

The bombardment of surfaces with low energy ions can lead to the development of various micron and nano sized surface structures. From literature, we know that by controlling the angle of incidence, there will be different shapes of structures obtained. These structures include pits, cones, dots, ripples etc. The phenomena responsible for production of such features are sputtering, redeposition of sputtered material and surface diffusion.

In the present study, commercially available (100) oriented Indium Phosphide (InP) samples with a thickness of ~ 0.5 mm have been taken and bombarded with 1.5 keV Argon atoms for a fixed fluence of 8×10^{16} atoms/cm². We varied the angle of incidence of the atom

beam, from normal incidence to 76° , with respect to surface normal. The bombarded surface shows the nanostructures as analysed by Atomic Force Microscopy (AFM). For normal to small increase in the incident angle of the beam, nanodots pattern observed and after a critical angle of incidence, ripple patterns are observed. The evolution of nanostructures from dots to ripples has been analysed in terms of their size, shape and roughness by means of AFM imaging. XPS has been used to study the effect of angle of incidence on the sputtering of InP.

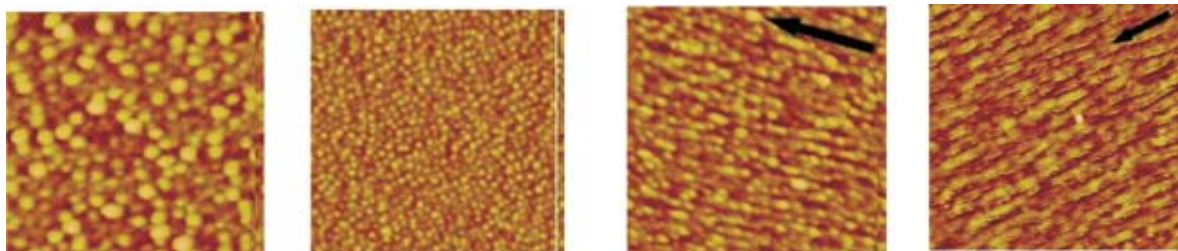


Fig.1. AFM micrographs for incidence angle of 0, 23, 45 and 63 degrees (from left to right

5.2.11 Formation of ion bombardment induced surface nanostructures on InP(100)

I. Sulania¹, A.Tripathi¹, D. Kabiraj¹, P.Kumar¹, S.Verma² and D.K.Avasthi¹

¹Inter University Accelerator Centre, New Delhi

²Institute of physics, Bhubaneswar

The bombardment of surfaces with moderate energy ions can lead to the development of various micron and nano sized surface structures. These structures include pits, cones, dots, ripples etc. The phenomena responsible for production of such features are sputtering, ion reflection, redeposition of sputtered material and surface diffusion. The nanostructure formation on semiconductor surfaces has attracted much attention due to their potential application in low-dimensional devices, such as ordered quantum nanodots for optoelectronics and quantum devices. We report the formation of well ordered nanodots on Indium Phosphide (InP) using 500 keV Ar ion irradiation at 30 degree with respect to the surface normal. This energy has been chosen as the nuclear energy loss dominates over electronic energy loss.

The structural properties of these nanodots have been investigated using Atomic Force Microscope (AFM). AFM studies reveals that the average sizes of the InP nanodots vary from 80 nm to 100 nm. These nanodots are circular in shape. We have studied the effect of ion fluence on the density of these dots. With increase in fluence, the dots are becoming more and more dense irrespective of their sizes. The ordering and homogeneity of these nanodots increases with ion fluence, leading to well ordered dots. As In and P have different sputtering rates due to their masses. P being a lighter element, is sputtered more than In. XPS has been used to see the effect of ion fluence on the sputtering of InP, as the fluence is increased the surface is becoming more and more In rich. The structures are more dense on the surface as

the fluence is increased, the reason for this is as InP is bombarded more and more, more of the In comes on the surface to form the nanodots on the nucleation sites created by the ion beam due to agglomeration.

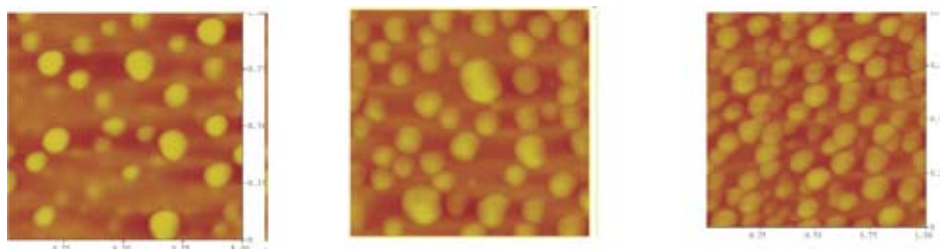


Fig.1. AFM micrograph: $1\mu\text{m}\times 1\mu\text{m}$: 1×10^{15} ions/cm², 1×10^{16} ions/cm² and 1×10^{17} ions/cm²

5.2.12 Synthesis of $\text{Si}_{1-x}\text{Ge}_x$ nanoparticles using atom beam sputtering and irradiation effects

Kapil U. Joshi¹, D. Kabiraj², A. M. Narsale¹, J. C. Pivin³, D. K. Avasthi², D. Kanjilal², T. K. Gundurao⁴, T. N. Warang¹ and D. C. Kothari¹

¹Department of Physics, University of Mumbai, Mumbai

²Inter-University Accelerator Centre, New Delhi

³CSNSM, IN2P3-CNRS, Batiment 108, F-91405 Orsay Campus, France.

⁴SAIF, Indian Institute of Technology (IIT) Bombay, Powai, Mumbai

$\text{Si}_{1-x}\text{Ge}_x$ alloys nanoparticles have attracted much interest as their band gaps are variable between those of nc-Si and nc-Ge via so called “band-structure engineering”. Takeoka et. al. [1] succeeded in fabricating the $\text{Si}_{1-x}\text{Ge}_x$ nanoparticles embedded in SiO_2 film where Blueshift of photoluminescence peak frequency with the decrease in particle size was observed. We have deposited Si/Ge/ SiO_2 composite films using atom beam sputtering method at IUAC (New Delhi) [2]. The main advantage of atom beam sputtering technique is that the charging of target is minimum as atoms are neutral. RBS analysis of three sets of as-deposited films show (Si: Ge: O) content in atomic % as (24: 42: 33), (47: 13: 40) and (46: 10: 43), respily. The as-deposited films were annealed using conventional furnace annealing (CFA) and rapid thermal annealing (RTA). The as-deposited films were also SHI (Swift Heavy Ion) irradiated using 100 MeV Ni ions and 180 MeV Ag ions to study the effect of electronic energy loss (S_e) variation. The films were characterized using GXR D, UV-vis spectra, Raman, FTIR spectroscopy. Particle size of nanocrystalline SiGe was evaluated using Scherrer formula [3] as ~ 4.7 nm for CFA films and is higher in the range 12-31 nm in RTA films and 3-80 nm in SHI irradiated films at different fluences. Fig. 1 show GXR D plot of three sets of films CFA. The films were polycrystalline with cubic diamond structure. GXR D of RTA and SHI irradiated films show peaks corresponding to different SiGe compositions. UV-visible spectra show the red shift in absorption edge as the annealing temperature is increased for CFA, RTA (except: set-1) and SHI irradiated films. Fig. 2 shows Tauc’s plot for set-1 films RTA showing two absorption edges possibly due to nc-Ge (bulk E_g at 0.7 eV) and nc-SiGe (higher E_g than either

Si or Ge). FTIR spectra reveal the phase separation of SiGe nanoparticles from SiO_x matrix, upon annealing. Raman spectra in fig. 3 show that SiGe phase is formed with c-Ge as core and c-SiGe as shell in the SiO_2 matrix. Curve-3 (red) shows that the Si-Si peak is weak and diminishes with increasing annealing temperature. This indicates that our assumption that the system consists of relaxed Ge core surrounded by SiGe shell is true [4]. Rapid thermal annealed samples of other sets show same red shift in absorption band gap as the annealing temperature is increased.

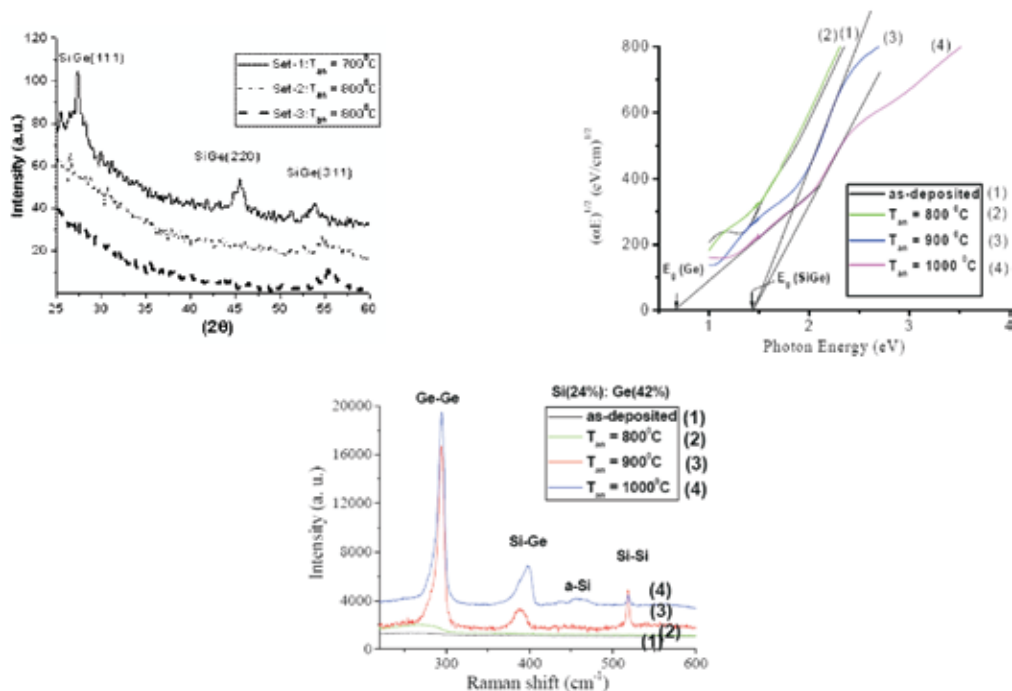


Fig.1. GXR D plots of the samples annealed at 700 °C (set-1) and at 800 °C (for set-2, 3)

Fig. 2 Plot of $(\alpha E)^{1/2}$ vs E obtained from UV-vis spectra for as-deposited and samples RTA at $T_{an} = 800, 900, 1000$ °C as shown by curve (1), (2), (3) and (4). Extrapolated lines for curve -3 and curve-4 gives two intercept on energy axis as band gaps

Fig. 3 Raman spectra of set-1 as-deposited and annealed samples for $T_{an} = 800, 900, 1000$ °C shown by curve (1), (2), (3) and (4) respectively

As-deposited films were irradiated using SHI shows that the band gap is decreasing with increasing fluence. In all annealing treatments CFA and RTA as well as in SHI irradiation, absorption edge is red shifted with increase in annealing temperatures and irradiation fluence (in SHI). We have compared the results of the set-3 films irradiated by 100 MeV Ni and 180 MeV Ag ion beams with different fluences for S_e variation. The nature of the absorption band gap variation with fluence is similar i.e. the band gap is red shifted with increase in fluence. But it is observed that the absorption band gap decreases rapidly as the S_e value is increased from 0.769 keV/A (for 100 MeV Ni ion beam) to 1.35 keV/A (for 180 MeV Ag ion beam).

In all the annealing treatments, the atom beam sputter deposition in conjunction with RTA has an advantage over other treatments.

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5.2.13 Ion beam induced anisotropic deformation of Si chevrons

Rupali Nagar¹, C Patzig², B Rauschenbach², V Sathe³, D Kanjilal⁴, B R Mehta¹ and J P Singh¹

¹Department of Physics, Indian Institute of Technology, Delhi

²Leibniz-Institut für Oberflächenmodifizierung e.V. Leipzig Permoserstraße 15 D-04103, Leipzig, Germany

³UGC-DAE, CSR, University Campus, Khandwa Road, Indore

⁴Inter University Accelerator Centre, New Delhi

The unification of electrical and mechanical expertise at the sub-micron scale has led to nano-/micro- electromechanical systems (N/MEMS) [1]. Size miniaturization and enhanced performance are the key motivations in the on-going research in these fields. A successful implementation of nanostructure-based coatings/films as practical devices requires rigorous tests for their reliability and performance. The durability of a device, its robustness to the wear and tear under operating conditions, yield strength, and fracture toughness can be gauged by performing various mechanical tests on the specimens. Mechanical properties such as stiffness, hardness, and Young's modulus of columnar glancing angle deposition (GLAD) grown films are a measure of the robustness of nanostructures. Recently, it was reported that intense electronic excitations induced by 100 MeV Ag⁺⁸ ion irradiation of Cr nanorods can cause a four-fold increase in their hardness as studied by nanoindentation [2]. Strengthening by grain size reduction has been cited as the reason for the observed increase in the hardness.

Columnar films consisting of five-armed silicon nanosprings were grown by glancing angle ion beam sputter deposition on flat Si(111) substrates pre-patterned with 370 nm silica (set A samples) and 510 nm diameter polystyrene spheres (set B samples). The details of the growth have been reported elsewhere [3]. Identical samples taken from each set A and B were subjected to 1.2 MeV Ar⁺⁸ ion beam at liquid nitrogen temperature. The samples were irradiated at different ion fluences ranging from 10¹⁵, 10¹⁶, and 10¹⁷ ions cm⁻².

The mechanical stiffness of individual chevrons was determined by AFM [Nanoscope IIIa, Veeco Instruments] based *force-distance (F-d) spectroscopy*. It was observed that for irradiated samples of set A, the increase in the stiffness is approximately 31% for fluence of 10^{15} ions cm^{-2} and 171 % for 10^{16} ions cm^{-2} . Similarly, for set B, an increase of 66 % for 10^{16} ions cm^{-2} and 253 % for 10^{17} ions cm^{-2} fluence is observed. Figure 1(a) summarizes the variation of average stiffness of the chevrons with the ion fluence for all the samples. It is observed that the stiffness of the chevrons increases with increasing ion fluence.

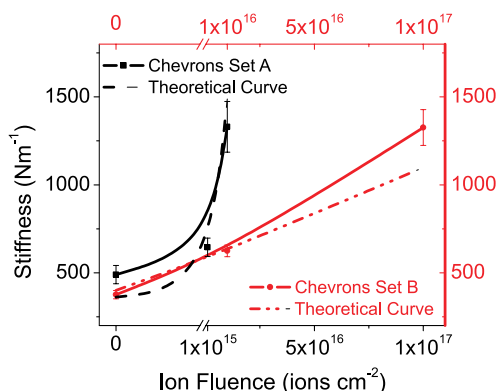


Fig.1(a). The variation in stiffness of Si chevrons of set A and B with ion fluence.

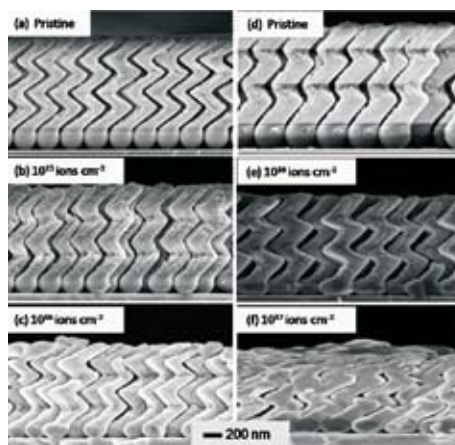


Figure 1(b) shows the cross-sectional SEM views of pristine and irradiated samples of set A (a to c) and B (b to d) respectively. The observed dimensional changes are in line with the ion hammering effect.

The SEM micrographs of irradiated samples exhibited *ion hammering effect* as shown in figure 1(b) [4]. To investigate the changes in the material properties post-irradiation, micro-Raman spectroscopy was performed on the chevron samples. The spectra for pristine samples have a TO-like band at 470 cm^{-1} characteristic of a-Si. Though the Raman line shift even for the sample irradiated at 10^{17} ions cm^{-2} is within 2 cm^{-1} towards lower wave numbers of the pristine sample, the Raman line width shows a monotonic increase with ion fluence. The full width at half maximum (FWHM) increases from 67 cm^{-1} for pristine sample to 83 cm^{-1} for 10^{16} ions cm^{-2} and increases further to 90 cm^{-1} for 10^{17} ions cm^{-2} indicating that the chevrons films progressively incorporate more and more disorder. Similarly, for set A the FWHM increases monotonically from 83 cm^{-1} for pristine sample to 94 cm^{-1} for sample irradiated at 10^{16} ions cm^{-2} . For both sets of samples this indicates that ion irradiation enriches the amorphous character in the Si chevrons.

The stiffness of the irradiated chevrons is thus expected to decrease as compared to pristine samples. The two effects namely, dimensional changes and amorphization of

chevrons counteract. Whereas, dimensional changes favor stiffer chevrons, higher degree of amorphization favors softer chevrons [5]. In the present study, it is found that post-irradiation the stiffness of the chevrons increases.

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5.2.14 Effect of SHI irradiation on nano semiconductor: conducting polymer composite

S Chowdhury¹, A M P Hussain¹, G A Ahmed², F Singh³, D K Avasthi³ and A Choudhury²

¹Dept. of Physics, Gauhati University, Guwahati, Assam

²Dept. of Physics, Tezpur University, Napaam, Tezpur, Assam

³Inter University Accelerator Centre, New Delhi

In this work composite of PbS nanocrystal and MEH-PPV was synthesized and subjected to energetic ion bombardment. The synthesis of PbS nanocrystals was performed following method described by Scholes and his group [1] and dispersed in toluene and mixed with a solution of MEH-PPV in known weight ratio. To carry out SHI irradiation, the polymer and nanocrystals solution was deposited on 1 cm x1 cm ITO substrate and subjected to 120 MeV Si⁺⁹ ion beam with approximate beam current of 0.5 pA. Structural and optical properties of as deposited and ion irradiated films were studied using XRD, TEM, UV-Vis and PL techniques.

Significant blue shift in UV-Vis absorption spectra reveals reduction of particle size which is also supported by XRD and TEM study. The PL investigation shows that photovoltaic property of the system initially decrease and then increases with applied ion fluence but after that the same material may be better used for luminescent devices that emit with wavelengths between 800 and 1000 nm.

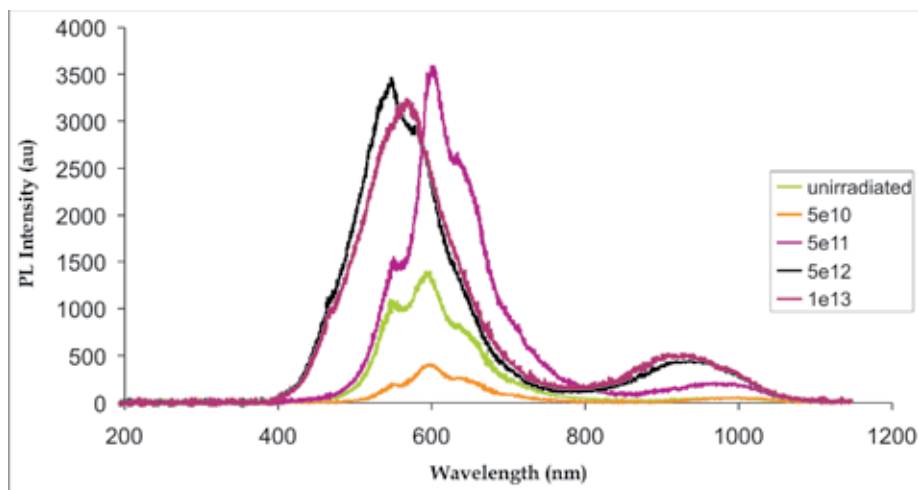


Fig. 1. PL spectra of nanocrystal: polymer composite before SHI irradiation and after irradiation at different fluences

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5.2.15 Optical Properties of Irradiated ZnS Embedded in PMMA Matrix

Sumit Kumar¹, Shweta Agrawal¹, M.Singh¹, F.Singh², P.K.Kulriya², D.K. Avasthi², and Y.K.Vijay¹

¹Department of Physics, University of Rajasthan, Jaipur

²Inter-University Accelerator Center, New Delhi

Swift heavy ion induced modifications in embedded polymer matrices have become an interesting area for scientific community due to changes in its structural, optical properties of the material. The structural and luminescence properties (Bhargava and Gallagher et al) of ZnS doped PMMA matrix has been studied by much research. Ion interaction with material is important due to nature of electronic and nuclear energy losses. In present work we have studied the changes in optical property of ZnS nanoparticle doped in PMMA matrix which has been irradiated by 100MeV Silicon beam at doses 10^{11} - 10^{12} ions/cm². Photoluminescence has been carried out to reveal surface emission.

The ZnS nanocrystal has been synthesized by chemical route method and doping of these nanocrystals in PMMA matrix has been performed by solution cast method [1]. Irradiation of these embedded matrix has been performed by Si⁺⁷ (100MeV) at fluence 10^{11} - 10^{12} ions/cm².

Characterization of these samples has been carried out using X-Ray diffraction (XRD), Optical absorbance and Photoluminescence. XRD measurements of these samples have been performed by using Bruker AXS D8 advance model PW 1600 powder x-Ray diffractometer. Optical absorption measurements have been performed using a Hitachi-330 spectrophotometer. The spectral range in this case was chosen from 300 to 800nm. Photoluminescence was carried out by He-Cd laser with excitation wavelength $\lambda=325\text{nm}$.

Fig 1 Shows the XRD pattern of ZnS nanoparticle in PMMA matrix. In which a peak at $2\theta = 10^\circ$ corresponds to amorphous PMMA and other peaks corresponds to ZnS. These peaks are associated to the presence of ZnS nanocrystals having hexagonal structure with in the PMMA matrix as compared with standard data by JCPDS software. After irradiation there is sharp reduction in peak intensity for the same samples, so it may conclude that the SHI penetrate the samples along the trajectory of the ion beam.

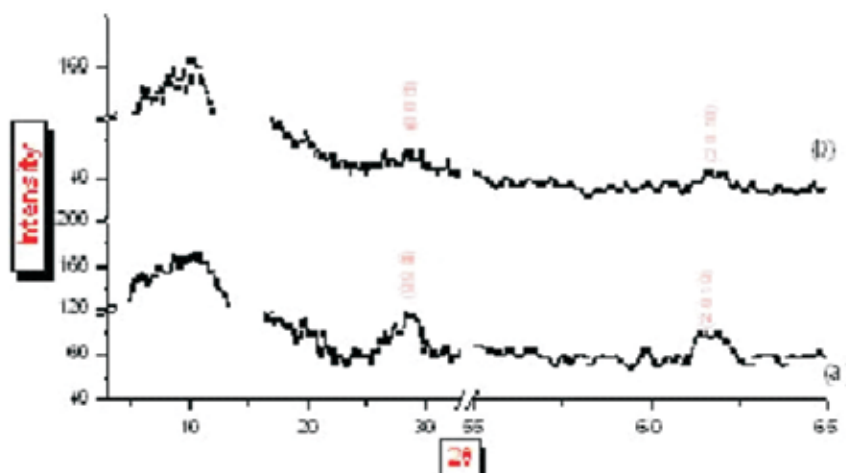


Fig.1. XRD pattern of ZnS nanoparticle in PMMA matrix

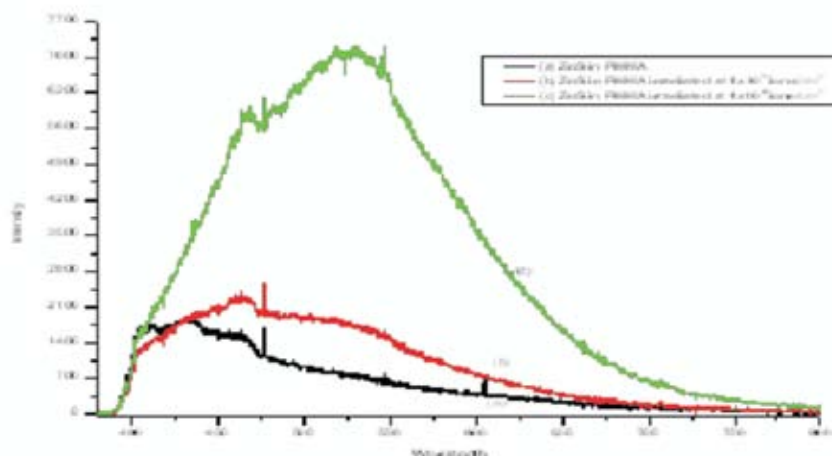


Fig. 2. Photoluminescence spectra of irradiated ZnS in PMMA different fluence (after irradiation there is an enhancement in peak intensity)

Detailed analysis is in progress.

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5.2.16 Ion beam induced blistering and exfoliation studies in semiconductors

U. Dadwal¹, R. Scholz², P. Kumar³, D. Kanjilal³, S. Christiansen², U. Goesele² and R. Singh¹

¹Department of Physics, Indian Institute of Technology, Delhi

²Max Planck Institute of Microstructure Physics, Weinberg 2, Halle, Germany

³Inter University Accelerator , New Delhi

The hydrogen and helium ions having energy of 100keV were implanted in various semiconductors namely AlN, GaN, InP, ZnO and Ge with fluence values ranging from 0.2×10^{17} to 4.0×10^{17} cm⁻². The implantation was done in order to investigate the blistering/exfoliation phenomenon in implanted samples for layer transfer applications[1,2]. All samples were implanted at room temperature with 100keV H⁺ or He⁺ ions with above mentioned fluence values at different substrate temperature of LN₂, RT, 100 and 300°C respectively. The implantation was performed at Low Energy Ion Beam Facility (LEIBF), Inter University Accelerator Centre, New Delhi. During implantation, the sample surface normal was inclined at ~7° relative to the incidence ion beam in order to avoid channeling effects[3]. After implantation the samples were cut into different small pieces and annealed at different temperature for different annealing time in air ambient box type furnace. In order to check the optically detectable surface blisters/exfoliation in the samples, we used Nomarski optical microscope. The microstructural characterization of the implantation induced damage in respective samples was performed by cross-sectional transmission electron microscopy (XTEM). The XTEM measurements were carried out using a Philips CM20T machine operated at 200 kV. The typical optical and XTEM pictures for one of the sample AlN are shown in below figures.

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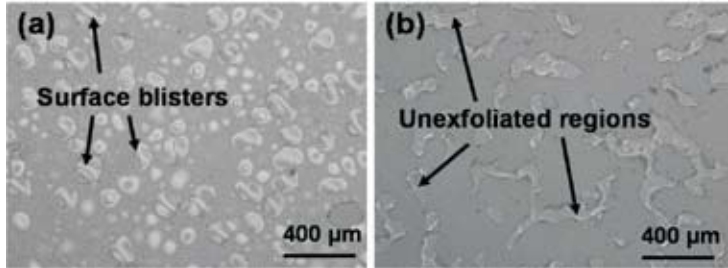


Fig. 1. Nomarski optical images of H^+ ion implanted AlN samples with implantation temperature of (a) RT (b) 300°C after post-implantation annealing at 800°C for 1 hr.

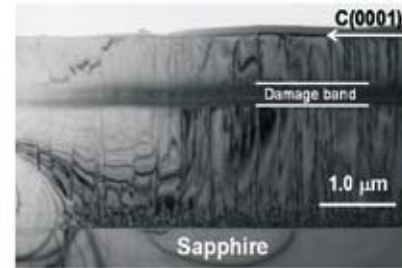


Fig. 2. XTEM image of 100 keV H^+ ion implanted AlN at 300°C implantation temperature in the as-implanted state.

5.2.17 Room temperature ferromagnetism in Ni implanted nanodimensional ZnO films

B. Pandey¹, S. Ghosh¹, P. Srivastava¹, P. Kumar² and D. Kanjilal²

¹Nanotech Laboratory, Indian Institute of Technology, Delhi

²Inter University Accelerator Centre, New Delhi

Diluted magnetic semiconductors (DMSs), the key materials required to realize future spintronics devices, have attracted considerable scientific attention [1]. Zinc oxide (ZnO) is one of the promising candidates for making DMS after doping it with different transition metals (Fe, Co, Ni etc.). Ni-doped ZnO is considered to be one of the promising candidates in this regard because of the possibility to combine optical transparency and ferromagnetism in the same matrix. Room Temperature Ferromagnetism (RT-FM) is found in Ni-doped/implanted ZnO samples in the recent past by different groups. In some reports, RT-FM is attributed to the presence of Ni nanoparticle and in other studies, RT-FM is shown to be intrinsic in nature. The objective of the present study is to explore the origin of RT-FM of the implanted films by varying incident Ni^{2+} ion fluence (ions/cm^2).

Using Low energy ion beam facility (LEIBF), Ni^{2+} ions of energy 200 keV were implanted in ZnO thin films deposited by vapor phase transport on Si substrate (ZnO/Si) [2,3] and by pulsed laser deposition on sapphire substrate (ZnO/Sapphire). The incident ion fluences were 6×10^{15} , 8×10^{15} and 2×10^{16} ions/cm^2 corresponding to Ni concentrations of $\sim 2\%$, $\sim 3\%$ and $\sim 7\%$ respectively. Structural, morphological, magnetic and electrical properties of the films were studied by glancing angle x-ray diffraction (GAXRD), Atomic Force Microscopy (AFM), Magnetometers (AGM and SQUID) and four probe method in van der Pauw geometry respectively.

It is found that the implanted ZnO/Si film is polycrystalline in nature after implantation. While implanted ZnO/Sapphire film is highly oriented in (002) direction and structure remains

almost same even after implantation. In both the cases, no pre- or post-heat treatment was carried out and no peaks corresponding to Ni or NiO is detected in the XRD spectra.

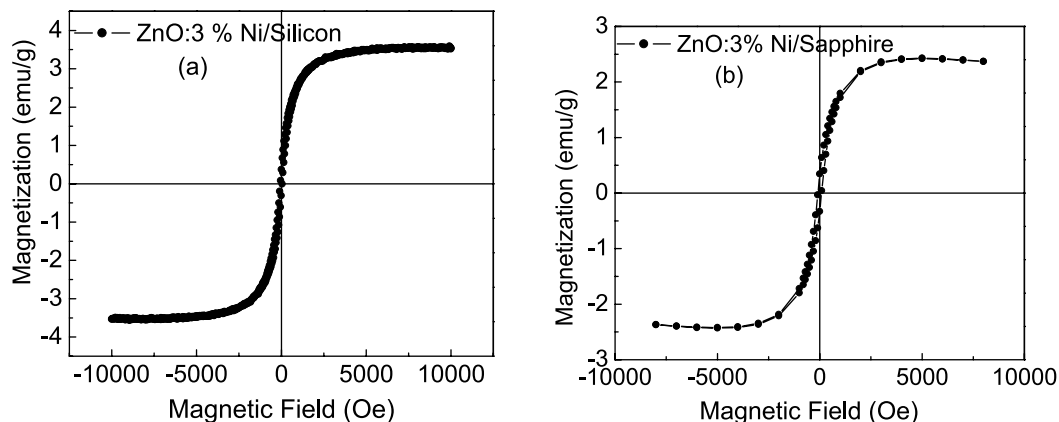


Fig.1. (a) Magnetograph (M-H) of 3 % Ni implanted ZnO/Silicon film; (b) Magnetograph (M-H) of 3 % Ni implanted ZnO/Sapphire film

Fig. 1 (a) and (b) show the M-H curve of ZnO/Si and ZnO/Sapphire films implanted with fluence 8×10^{15} ion/cm² (~ 3% Ni) respectively. It is found that in both the cases saturation magnetization (M_s) is the maximum in the films implanted at this fluence (8×10^{15} ions/cm²). In case of ZnO/Si film the magnetic properties depend on the crystallite size, number of oxygen vacancies and charge carrier concentration in the films. Also variation is correlated with the microstructural properties of the films [3]. While in case of ZnO/Sapphire films, the cause of change in magnetic properties with fluence is not understood yet. Based on the literature reports, the bound magnetic polaron may be the most likely cause of this change.

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5.2.18 Swift iron ion induced effects in gallium phosphide

S.K. Dubey¹, R.L. Dubey¹, V. Jadhav¹, A.D. Yadav¹ and D. Kanjilal²

¹Department of Physics, University of Mumbai, Mumbai

²Inter University Accelerator Centre, New Delhi

In the present work, $\langle 111 \rangle$ oriented polished surfaces of gallium phosphide single crystals were irradiated with 100 MeV $^{56}\text{Fe}^{9+}$ ions for various ion fluences varying from 1×10^{11} to $1 \times 10^{14} \text{ cm}^{-2}$ at room temperature. Three dimensional (400 nm x 400 nm) AFM micrographs of the non-irradiated and samples irradiated with various ion fluences were recorded. The image obtained from non-irradiated sample (Fig. 1(a)) showed the presence of ripples on the surface of gallium phosphide with root mean square surface roughness of 0.15 nm. AFM images obtained from the samples irradiated with various ion fluences showed the presence of hills separated with valleys. The average diameter of hills was found to be 19.7, 19.8, 20.7 and 22.6 nm for ion fluences 5×10^{12} , 1×10^{13} , 5×10^{13} and $1 \times 10^{14} \text{ cm}^{-2}$ respectively. The root mean square surface roughness of gallium phosphide surface increases up to ion fluence $5 \times 10^{13} \text{ cm}^{-2}$ whereas for ion fluence $1 \times 10^{14} \text{ cm}^{-2}$ root mean square surface roughnesses was found to decrease [1].

Figure 1 shows first-order Raman spectra of the non-irradiated and irradiated gallium phosphide samples with various ion fluences. The spectrum of non-irradiated sample shows two peaks at 365.05 cm^{-1} and 402.18 cm^{-1} , which correspond to the characteristic LO and TO Raman modes of crystalline gallium phosphide. The spectra obtained from the samples irradiated with various ion fluences showed decrease in the intensity of Raman peak with respect to ion fluence. The peak position also shifted towards the lower wave number. The decrease in the Raman peak intensity with increasing ion fluence indicates the increase of the defects concentration in the surface region of gallium phosphide. The shift of peak position indicates presence of stress in the irradiated samples. The stress (σ) in the irradiated samples was determined from the shift of the LO peak using the following relation [2-4]

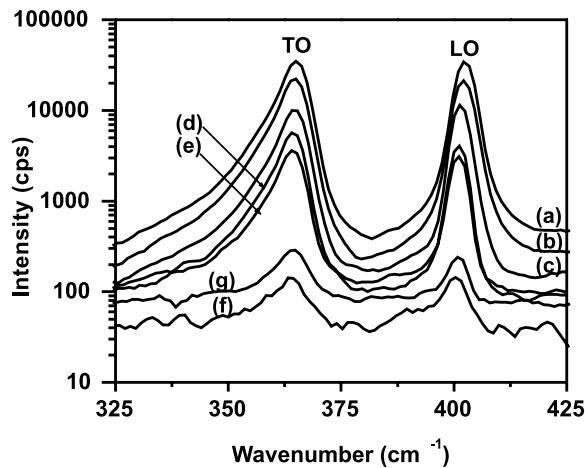


Fig. 1. First-order optical Raman spectra of gallium phosphide samples; (a) Non-irradiated and irradiated with 100 MeV $^{56}\text{Fe}^{9+}$ ions at ion fluences (b) 1×10^{11} , (c) 1×10^{12} , (d) 5×10^{12} , (e) 1×10^{13} , (f) 5×10^{13} and (g) $1 \times 10^{14} \text{ cm}^{-2}$

$$\sigma \text{ (MPa)} = -250 \Delta\omega \text{ (cm}^{-1}\text{)} \quad \text{-----} \quad (1)$$

where $\Delta\omega = \omega_s - \omega_0$. In this expression, ω_0 and ω_s denote the wave numbers of the LO peak in the non-irradiated and irradiated gallium phosphide sample respectively. The ratio of the area under the LO Raman peaks of non-irradiated sample (A_v) and irradiated samples (A_i) and the values of stress calculated using the above equation. It is seen from the figure that the ratio of area (A_i / A_v) decreases, whereas stress in the samples increases with ion fluence. The decrease in the area suggests the decrease in the crystallinity of the gallium phosphide after irradiation. The enhancement of stress with ion fluence is observed due to lattice mismatch.

These results indicate amorphization of gallium phosphide surfaces at higher fluences ($>1 \times 10^{13} \text{ cm}^{-2}$). The electron-phonon coupling strength was also estimated by measuring the normalized Raman intensity (I_R) of the 2LO phonon with respect to that of the LO phonon. It was evident that the electron-phonon coupling strength reduces after irradiation.

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5.2.19 Effects of Swift Heavy Ion on optical properties of AlGaIn/GaN MQWs

N Sathish¹, G Devaraju¹, N Srinivasa Rao¹, V Saikiran¹, A P Pathak^{*1}, S A Khan² and D K Avasthi², E. Trave³ and P. Mazzoldi³

¹School of Physics, University of Hyderabad, Hyderabad

²Inter University Accelerator Centre, New Delhi

³Dip. Fisica "G. Galilei", Università di Padova, via Marzolo 8, 35131 Padova, Italy

AlGaIn/GaN Multi Quantum Wells (MQW) have numerous optoelectronic applications including semiconductor photodiodes. Quantum well interdiffusion

technology has become increasingly important in the drive towards fabrication of photonic integrated circuits due to its versatile band gap tuning process. Recently we have demonstrated strain modification in lattice matched heterostructures using SHI and extensively strain relaxed MQWs have also been studied [1]. In this study, we are presenting our new results concerning high energy irradiation on AlGaIn/GaN MQWs characterized by PL.

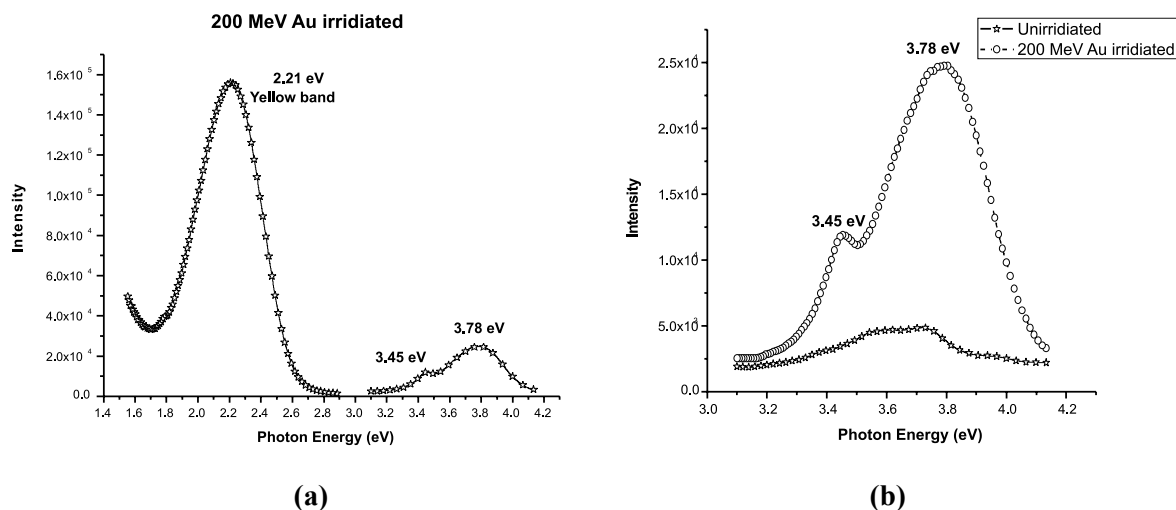


Fig.1(a) PL spectra of irradiated sample recorded at RT, Shows Yellows luminescence (b) GaN band edge luminescence of Unirradiated and irradiated samples, shows after irradiation GaN luminescence intensity increased by one order.

AlGaIn/GaN MQWs were grown on Sapphire substrate by MOCVD at IEMT Warsaw, Poland. The irradiation was performed at room temperature. The samples were irradiated with 200 MeV Au⁸⁺ ions at a fluence of 5x10¹¹ ions/cm² scanning over 1cm x1cm from IUAC, New Delhi 15MV Pelletron accelerator. Pristine and irradiated samples were studied by PL. Photo luminescence studies were carried out at room temperature (295K). PL was excited with Xe UV lamp (265nm) and detected with HAMAMATSU N2-cooled detector.

Photoluminescence of unirradiated sample shows weak A and I2 transitions from GaN bulk crystal because most of the light is absorbed in the multilayer, so that very little signal is observed from the GaN bulk layer. Luminescence around ~3.7 eV coming from the QWs, shows that GaN layers are confined between AlGaIn layers. Also we did not observe any yellow luminescence ~ 2.1 eV in the unirradiated sample. After irradiation we observed very strong yellow luminescence at ~2.1 eV (see fig 1(a)), which are generally observed due to the deep levels like Ga vacancy or oxygen related defects. In the irradiated sample free exciton structures vanish and only one excitation I2 (~ 3.45 eV) is observed , which may be due to the bound exciton to neutral donors, commonly observed in undoped n type GaN's. QWs luminescence intensity has increased by two orders. This shows that the confinement effects are enhanced in irradiated samples [2].

The Optical properties of AlGaN/GaN MQWs before and after irradiation have been analyzed using PL. This study shows that SHI increase the confinement effects in the MQWs and intensity of the active layer of the MQWs luminescence is increased by two orders. This may be due to induced strain in the GaN and AlGaN layers. However, the unwanted yellow luminescence also has been introduced by the SHI which may be due to the point defects like Ga vacancies created during irradiation. Of course these may be annealed out by RTA.

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5.2.20 SHI irradiation induced recrystallization of Ge implanted in Si

N Srinivasa Rao¹, N Sathish¹, G Devaraju¹, A P Pathak¹, D Kabiraj², S A Khan², P K Kulriya², F Singh², B K Panigrahi³, K G M Nair³ and D K Avasthi²

¹School of Physics, Univ. of Hyderabad, Central University (P.O), Hyderabad

²Inter University Accelerator Centre, New Delhi

³Mat. Sc. Division, Indira Gandhi Centre for Atomic Research, Kalpakkam

In recent years, Ge implanted in Si and SiO₂ matrices has attracted great interest from both basic and applied point of view. It has been observed that ion beams are very useful in synthesis, characterization and modification of nanostructured materials. Recrystallization of amorphous films can be done by using swift heavy ion beams. The present study concentrates on the structural characterization of ion irradiated Ge implanted Si. We characterized films by using Raman, XRD and AFM.

In the present experiment, 400 keV Ge⁺ ions were implanted with a fluence of 5x10¹⁶ ions/cm² into a cleaned p-type Si(001) substrate. During implantation the substrate was maintained at 573 K. The as-implanted sample was irradiated with 100 MeV Au⁸⁺ ions with a fluence of 1x10¹³ ions/cm² to investigate the behavior of high fluence Ge implanted in Si.

X-ray diffraction spectra of pristine and irradiated samples are shown in Fig. 1 at fixed fluence. In Ge implanted Si, due to high Ge fluence the as-implanted sample shows the amorphous nature. However, Au ion irradiation results in crystallization as indicated by the Ge (311) at 53.746^o and Si (311) at 55.84^o peaks in the spectra. The appearance of Si peak would indicate the crystallized top layer. The large width of the Si peak would implies existence of defects in the layer. In Raman, the appearance of Ge mode along with SiGe mode indicates the recrystallization of the amorphized layer. The mode around 150 cm⁻¹ may be attributed to residual damages in the top Si layer which is consistent with the broad Si peak in the X-ray spectra.

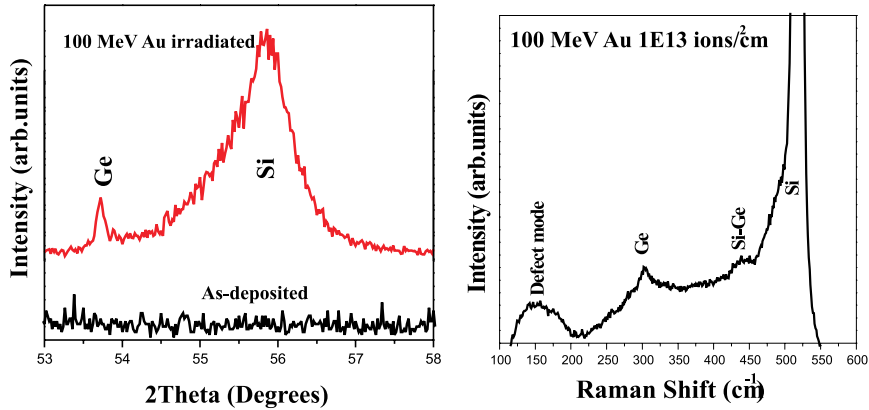


fig. 1. GIXRD and Raman of Ge implanted Si irradiated with 100 MeV Au ions

The AFM image after Au ion irradiation indicates a smooth surface with an rms roughness of about 0.3 nm. This indicates that the roughness of the sample is not affected appreciably by ion irradiation. The energy deposited by annealing and SHI irradiation helps to release the strain between the grains in the films and results in a further improvement in the crystalline quality of the film. The recrystallization mechanism can be explained on the basis of thermal spike model.

5.2.21 Band gap modification with Swift Heavy Ion Irradiation in MQWs

G.Devaraju¹, N.Sathish¹, N.Srinivasa Rao¹, A.P.Pathak¹, S.A.Saif³, D.K.Avasthi² and B.M.Arora³

¹School of Physics, University of Hyderabad, Central University (P.O), Hyderabad

²Inter University Accelerator Centre, New Delhi

³Tata Institute of Fundamental Research, Homibhabha Road, Colaba, Mumbai

InGaAs/InP Multi Quantum Wells (MQW) have numerous optoelectronic applications including semiconductor photodiodes. Quantum well interdiffusion technology has become increasingly important in the drive towards fabrication of photonic integrated circuits due to its versatile band gap tuning process. To achieve spatial tuning of band gap in these samples, Swift Heavy Ion (SHI) beam irradiation has been used.

Highly tensile strained 15 multi quantum wells of InGaAs/InP were grown on InP semi insulating substrate. Such grown MQWs were then irradiated with 100 MeV gold ions for varied fluencies using 15 MV Pelletron accelerator, IUAC, New Delhi. The irradiated samples had been subjected to rapid thermal annealing at 700°C for 60 sec to anneal out defects; as a consequence it improves luminescence properties. Different characterization techniques such as HRXRD and PL have been employed to investigate strain, interfacial changes and

luminescence properties. Eventually, effect of irradiation-induced strain has been measured and compared with pristine sample. Tailoring of band gap was achieved as a function of fluence.

Fig. 1 shows a comparison of 002 HRXRD symmetric scans for as-grown, irradiated and subsequent annealed samples. This shows a clear shift of zeroth order peak to the higher angle side. This implies an increase in the average tensile strain of 0.0360% in MQW-I1 to 0.079% upon annealing at 700°C with respect to the pristine sample. As a function of fluence the interface mixing induced disorder is observed from the vanishing satellite peaks [1].

We also notice that the band gap has changed as a function of fluence. This study reveals that by irradiation with different fluences at fixed energy, the band gap may be tailored, due to tensile strain induced by interfacial mixing. We also found that in the LT-PL spectra, the peak shifts by 45nm and 82nm for fluences 5×10^{12} ions/cm² and 1×10^{13} respectively.

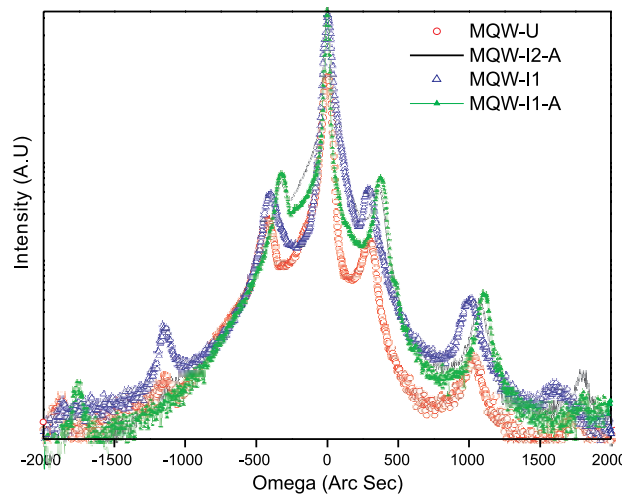


Fig..1. HRXRD (004) scans of MQW-U, MQW-I2 and MQW-I2-A samples

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5.2.22 Nanoring formation and elongation of ZnS in SiO₂ by 100 MeV Ni ion beam irradiation

Shiv P Patel¹, Lokendra Kumar¹, A. Tripathi², V.V. Sivakumar², I. Sulania² and D. Kanjilal²

¹Department of Physics, University of Allahabad, Allahabad

²Inter-University Accelerator Centre, New Delhi

II-VI compound semiconductors are promising materials in current research due to their interesting opto-electronic properties. Among them, ZnS has been extensively investigated as luminescent material having various applications in opto-electronic and display devices. ZnS is a direct wide band gap (3.91 eV) compound semiconductor with a high index of refraction and high transmittance in the visible range; it is one of the more importance materials in photonics [1-3].

Thin films of ZnS-SiO₂ were prepared by rf co-sputtering method. The thickness of the films were 170 nm measured by ellipsometry method. The samples were irradiated at room temperature with 100 MeV Ni ions and the vacuum of irradiation chamber was of the order of 10⁻⁶ torr during all experiment.

The films were irradiated with an ion beam of normal to sample surface at different fluences (Φ) 5 x 10¹¹, 1 x 10¹², 5 x 10¹², 1x 10¹³, and 5 x 10¹³ ions/cm². The pristine sample does not show any rings like structure on the surface of the film. On the other hand, the irradiated samples shows the rings like structures on the surface. AFM image of irradiated sample with fluence 5 x 10¹¹ ions/cm² revealed the elongation of particle perpendicular to the beam direction [4,5] but not rings formation. As fluence increases, at fluence 5 x 10¹² ions/cm², agglomeration of particles takes place which results the formation of rings like structure. At fluence 1 x 10¹³ ions/cm², the formation of rings has been completed. Further, increase in fluence results the elongation of rings structure perpendicular to the beam direction.

In conclusion, we have prepared thin films of ZnS-SiO₂ by rf co-sputtering and created rings like structure on surface of the samples by swift heavy ion irradiation. Ion beam irradiation also leads the elongation of the nanoparticles perpendicular to the beam direction.

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5.2.23 Structural and optical investigations in low energy ion beam irradiated ZnO

S. Chattopadhyay¹, S. Dutta¹, D. Jana¹, S. Chattopadhyay², A. Sarkar³, P. Kumar⁴ and D. Kanjilal⁴

¹Department of Physics, University of Calcutta, 92 A.P.C. Road, Kolkata

²Department of Physics, Taki Government College, Taki 743429

³Department of Physics, Bangabasi Morning College, 19 R.C. Sarani, Kolkata

⁴Inter University Accelerator Center, New Delhi

ZnO has wide device applications due to some of its unique properties like large band gap (~3.3 eV), large exciton binding energy at room temperature (~ 60 meV), high optical gain (300 cm^{-1}), very short luminescence life time etc. [1] which are required for various optoelectronic [2] and magnetooptical [3] devices like ultraviolet (UV) light-emitting diodes (LEDs), blue luminescent devices, UV lasers [4–6] etc. It is now well known that defects are playing an important role in structural, electrical and optical property modification in ZnO [7]. An understanding of the nature of defects and their effects on the physical properties is very much important on the point of technological application. Photoluminescence (PL) is one of the most useful experimental techniques to study defects in II-VI compounds like ZnO [8]. There are few reports available showing modification of PL emission by irradiation of swift heavy ion [9, 10] and electron beam [8]. But we have not find any report of study on the modification of PL emission due to low energy ion beam irradiation. Even after a lot of efforts, the correlation of defect centers with PL properties is a less understood topic till date. Understanding of amorphisation, if any [11], due to defects and the recovery of defect centers with subsequent annealing will be studied from room temperature PL measurements.

In this report we have studied the role of low energy ion beam irradiation on structural and optical properties of polycrystalline ZnO. Polycrystalline ZnO (99.99%, Sigma-Aldrich, Germany) samples are palletized and sintered at $500 \text{ }^\circ\text{C}$ for 4 hours. These samples are irradiated with 1.2 MeV Ar^{8+} ion beam with different fluence of 1.7×10^{14} , 1×10^{15} , 5×10^{15} , 1×10^{16} and 5×10^{16} ions/ cm^2 using low energy ion beam facility at Inter-University Acceleration Centre (IUAC), New Delhi.

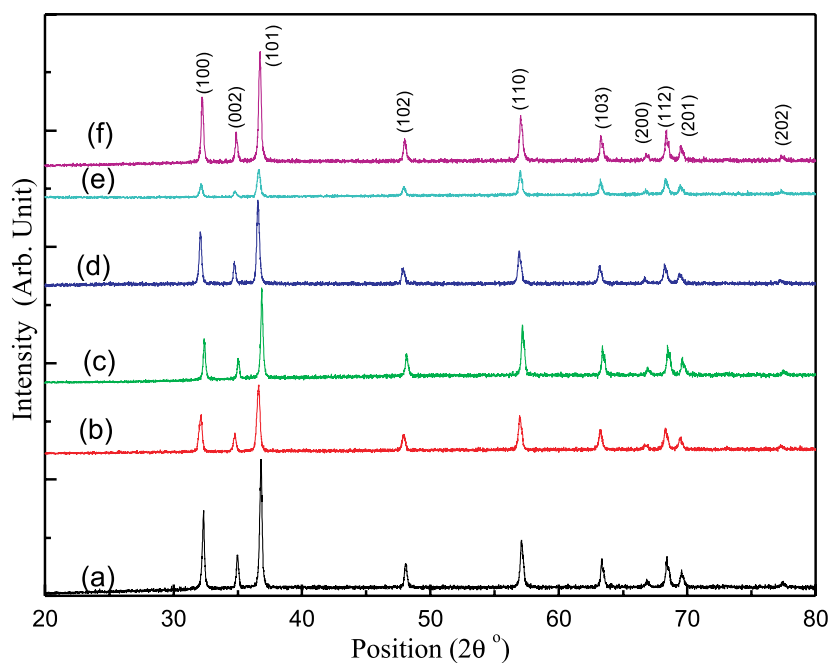


Fig. 1. XRD spectra of ZnO samples (a) Un-irradiated and irradiated with fluence (b) 1.7×10^{14} (c) 1×10^{15} (d) 5×10^{15} (e) 1×10^{16} and (f) 5×10^{16} ions/ cm^2 .

Room temperature XRD of all the samples has been taken. From XRD data a degradation of crystallinity up to the fluence of 1×10^{16} ions/cm² has been found as shown in figure 1. But improvement of crystalline quality has been observed upon irradiation with fluence of 5×10^{16} ions/cm² (figure 1). The photoluminescence studies of these samples are under progress.

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5.2.24 Studies on high energy ion-irradiation in Silicon and GaAs

Shramana Mishra¹, D. Kabiraj², Anushree Roy¹ and S. Ghosh³

¹Department of Physics and Meteorology, IIT, Kharagpur

²Inter-university Accelerator Center, New Delhi

³Department of Physics, Jawaharlal Nehru University, New Delhi

High energy ion irradiation is a material engineering processes, in which the mass of the ions used in the beam and the ion fluence play an important role in determining the changes in the structural, electrical and optical properties of the target materials. Raman spectroscopy is a unique non-destructible tool for studying the structural phases, size of the micro-domains and crystallinity of the material.

In this project we would like to study the effect of high energy ion irradiation on silicon and GaAs via Raman spectroscopy and other supporting characterization techniques, like x-ray diffraction, electrical measurements etc. Ion irradiation on target materials was carried out at Inter-university Accelerator Center, New Delhi in November 2008. Target materials used were n and p type silicon wafers of different carrier concentration, undoped GaAs and n-type

GaAs (carrier concentration $\sim 10^{18} \text{ cm}^{-3}$) wafers. Ion beams used were 100MeV oxygen (O^{7+}) and 110MeV silicon (Si^{8+}). The beam current was $\sim 3 \text{ pA}$ for O^{7+} and $\sim 2.5 \text{ pA}$ for Si^{8+} . Ion fluence range was from 5×10^{11} - 10^{14} ions/ cm^2 as the maximum structural change is expected in this range. Lithium (Li^{3+}) ion irradiation (50MeV, 1pA) on undoped and chromium doped semi-insulating GaAs were carried out previously.

Raman spectra of the undoped GaAs showed Raman longitudinal optical (LO) phonon and transverse optical (TO) phonon modes and other weak second-order features. The spectral profiles of irradiated samples were found to be significantly different compared to that of the virgin sample. It was observed that on irradiation, there was an asymmetric broadening of the LO phonon mode together with the increase in intensity of the amorphous mode of GaAs. Both originate from the continuous random network structure in the material. From the Raman line-shape analysis and the ratio of the intensities of various Raman modes, it can be observed that with the increase in ion fluence, there is an increase in amorphous fraction with respect to the crystalline component for pure GaAs wafers. However, for Li^{3+} irradiated chromium doped GaAs (GaAs:Cr) wafers an opposite trend was observed. Raman measurements reveal that high energy Li^{3+} irradiation results in local annealing via a decrease in disorder/defect related states in this material till an optimum irradiation dose is reached. For further investigations, temperature dependent Raman measurements were carried out. From the analysis of the temperature dependent measurements, it was observed that there was an increase in anharmonicity in the Raman modes with fluence for undoped GaAs wafers, however for the GaAs:Cr wafers, there is slight decrease in anharmonicity with fluence. These results will be reported shortly. Room temperature Raman measurements of the n-type GaAs showed the presence of the LO phonon-plasmon coupled mode. Further studies on this material are under progress.

5.2.25 The Effect of Ion Beam on PVDF/Layered Silicate Nanocomposites

Vimal Kumar Tiwari¹, Pralay Maiti¹, D.K. Avasthi² and P. K. Kulriya²

¹School of Mat.Sc. and Technology, Institute of Technology, BHU, Varanasi

²Inter University Accelerator Centre, New Delhi

Poly(vinylidene fluoride) (*PVDF*) is known to have high chemical stability towards acids and bases, mechanically strong, insulator and ferroelectric material. *PVDF* forms a wide variety of crystalline structures, among them β -form exhibits piezo- and pyroelectric properties. Therefore, it has versatile technological importance including sensors and actuators. The nanocomposites of PVDF and organically modified layered silicate nanoclay were prepared by melt extrusion technique with appropriate amount of nanoclay into the matrix polymer. Thin sample of approx 30 μm thickness were prepared by compressing polymer/nanocomposites melt in between two cover slips. Thin polymer samples including nanocomposites were irradiated with Si^{7+} ion having energy 80 MeV at a 0.5 pA current under different fluences.

In last brief report, we have shown the effect of irradiation on nanostructure and structure of PVDF and PVDF nanocomposites with increasing order of fluences. We have also reported comparison study with AFM and FTIR analysis in last brief report. The SHI destroy the spherulitic morphology of PVDF. But the NCs does not change much even after the high fluence of SHI irradiation. So it is clear that SHI have very little effect on the NC film surfaces, whereas PVDF surfaces get damaged by SHI bombardment.

Figure 1 shows the heat of crystallization (ΔH_c) as a function of the fluence during cooling in DSC. Pure PVDF shows very low ΔH_c values at higher fluence, which confirms the nonavailability of crystallizable polymer chains as a result of SHI-induced degradation. The NCs show a slight decrease in the ΔH values of the entire fluence range studied here.

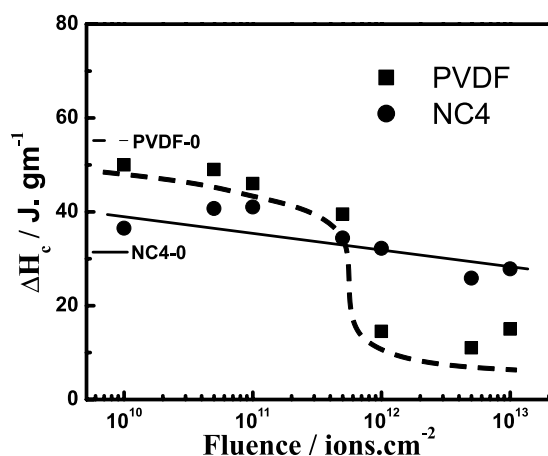


Fig.1. Heat of crystallization against fluence for pure *PVDF* and nanocomposite (*NC4*). The solid and the dash lines are guide to the eye. The small horizontal solid and dashed lines are the value for the corresponding pristine samples.

The SHI beyond 10^{12} ions/cm² fluence degrade pure *PVDF* while the dispersion of few weight percentages of nanoclay in nanocomposite protects the matrix from degradation. *PVDF* in layered silicate forms intercalated nanostructure and the extent of intercalation increases with increasing fluence. Pure *PVDF* almost amorphizes under *SHI* irradiation, whereas the crystallinity has slightly been reduced in nanocomposites even after high fluence *SHI* irradiation, as evident from optical images, *XRD*, *DSC* and *FTIR* analyses. The nanoclay acts as nucleating agent as obvious from the higher temperature of crystallization and *SHI* irradiation promote segregation of the layered silicate during bombardment and, hence, giving two temperatures of crystallization due to pure *PVDF* and nanoclay-induced crystallization. The surfaces, including spherulitic morphology, get damage pure *PVDF* after *SHI* irradiation, but it is unaffected in *NCs*.

5.2.26 Low energy ion modification of metal/polymer composite films prepared by sputtering method

N.L.Singh¹, Dolly Singh¹, Chaitali Gavade¹, V.V. Siva Kumar², P. Kumar² and D.Kanjilal²

¹Physics Department, M.S.University of Baroda, Vadodara

²Inter University Accelerator Centre (IUAC), New Delhi

Metallized polymers are widely used for different applications like packaging, microelectronic devices, optical filters, magnetic data storing devices etc [1]. In such applications, a good adhesion between the polymer and the metal is required. The connectivity between the metal and polymer phases controls the properties of the composite. Present work deals with the low energy irradiation effect on structural and optical properties of Ni dispersed polyimide film. Such material is useful in the radiative environment because PI is highly stable polymer in radiative environment. The major degradation processes in ion irradiated polymers are excitation and ionization, free radicals formation, macromolecular chain scission and cross linking and outgassing of volatile degradation product [2]. Other observed changes are connected with decarbonylation, i.e. with transformation of imide into amide group, aromatic ring opening leading to production of unsaturated bonds and carbonization and graphitization [3].

Thin film was formed by RF sputtering, in which simultaneous sputtering of polymer and Ni was done. The assembly contains a target holder acting as cathode and a substrate holder acting as an anode. Substrates are kept on the anode at a distance of 5 cm from the cathode. The cathode is connected to RF power supply. The anode and the rest of the system are properly grounded electrically insulated height adjustable table. The process has done at room temperature at the pressure of 1×10^{-6} torr. . Different concentrations of Ni viz .5%, 10% and 15% were dispersed by simultaneous sputtering of polymer and Ni foil. Thin film of metal dispersed polymer was formed on glass substrate. These films were irradiated with 100KeV Ar⁺ ion beam with the total ion fluences varies from 10^{14} - 10^{16} ions/cm².

Optical study of the pure and doped samples was carried out by means of UV-Vis spectra. It is observed that the band gap decreases slightly at higher concentration but no significant change has been observed with fluence. It reveals that PI is highly radiation resistant material.

Structural characterization of pristine and irradiated films was done by X-ray diffraction. The crystalline size was observed to decrease upon irradiation.

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5.2.27 Dielectric Response of Makrofol–N PC Irradiated with 100 MeV Si⁸⁺ Ions

S. Asad Ali¹, Rajesh Kumar², R.G. Sonakawade⁴, and Rajendra Prasad^{1,3}

¹Department of Applied Physics, Z. H. College of Engg, & Tech., AMU, Aligarh

²University School of Basic & Appl. Sciences, G.G. S. I. University, Delhi

³Vivekananda College of Technology and Management Aligarh

⁴Inter-University Accelerator Centre, New Delhi

Makrofol-N, a bisphenol A polycarbonate (PC) is widely used for ion track recording and to prepare track etched membranes as micro filters. When SHI penetrates a solid, the material along the trajectory of the ion beam is modified. Potential charge carriers may be formed by splitting of covalent, atomic or molecular bonds under the influence of the energetic ions[1]. Dielectric response of a material provides information about the orientational and translational adjustment of mobile charges present in the dielectric medium in response to an applied electric field.

Fig. 1 illustrates the dielectric response of 100 MeV Si⁸⁺ ion irradiated Makrofol–N PC samples along with that of pristine samples. Significant changes have been observed after irradiation. It is evident that dielectric constant increases with the fluence and at a particular frequency it does not show any change with the frequency in the frequency range of 300-1000 kHz This happens at all the ion fluences. The variation of dielectric loss presented in Fig.2

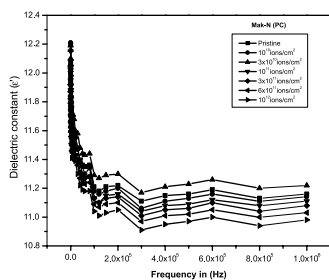


Fig. 1

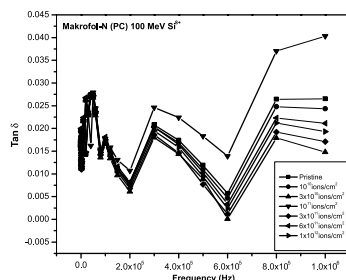


Fig. 2

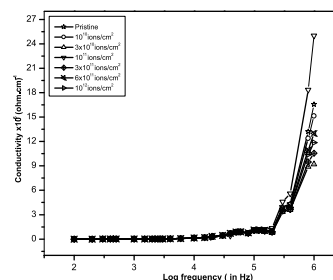


Fig. 3

Fig. 3 shows a sharp increase in A. C. conductivity in pristine as well as irradiated samples with frequency. Due to irradiation the increase in conductivity with fluence at a given frequency may be attributed to scissioning of polymer chains, resulting in an increase of free radicals, unsaturation, etc.

shows random behavior. It increases up to about 70 kHz followed by a decrease up to 200 kHz. The variations with ion fluences are visible only at higher frequencies in the range 600-1000 kHz. This indicates a small change in the dissipation factor of the polymer with ion irradiation. It may thus be concluded that irradiation changes the dielectric constant without affecting the dielectric losses only slightly.

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5.2.28 PALS and DBS Studies of 50 MeV Li³⁺ Ion Irradiated Polystyrene Films

S. Asad Ali¹, Rajesh Kumar², P.M.G. Nambissan³, Fouran Singh⁴ and Rajendra Prasad^{1,5}

¹Appl.Phys. Dept. Z. H. College of Engg. & Tech., AMU, Aligarh

²University School of Basic & Appl. Sciences, G.G.S.I. University, Delhi

³Saha Institute of Nuclear Physics, 1/AF, Bidhan Nagar, Kolkata

⁴Inter-University Accelerator Centre, New Delhi

⁵Vivekananda College of Technology and Management, Aligarh

Irradiation of polymeric materials with Swift Heavy Ions (SHI) results into the changes in their free volume properties[1] which can be characterized by Positron Lifetime Spectroscopy(PALS). Polystyrene films (1.5x1.5 cm²), an important and radiation resistant polymer having wide applications were exposed to 50 MeV Li³⁺ ion beam from 15 UD Pelletron accelerator at IUAC, New Delhi, India to the fluences of 5x10¹⁰, 5x10¹¹, 5x10¹² and 3.5x10¹³ ions/cm². PALS measurements were carried out using fast fast coincidence spectrometer of resolution 240 ps. Measurement of broadening of the spectrum of Doppler energy shifted annihilation γ -rays was also carried out using high resolution high purity Ge spectrometer and line shape parameter S was estimated[1].Life time spectra were analyzed using computer program Resolution and POSITRONFIT [2]. The mean lifetime τ_3 of ortho-positronium(o-Ps) is related to the size of the hole where the Ps atom is trapped. A spherical hole model [3] gives the mean free volume hole radius by following equation;

$$\tau_3 = \frac{1}{\lambda_3} = \frac{1}{2} \left[1 - \frac{R}{R_0} + \frac{1}{2\pi} \text{Sin} \left(\frac{2\pi R}{R_0} \right) \right]^{-1}$$

where $R_0 = R + \Delta R$ and $\Delta R = 1.66 \text{ \AA}$, ΔR is an empirical parameter, related to the penetration of Ps wave function into the bulk and has been determined by the fitting the experimental values of τ_3 obtained from the materials with known hole size [4]. The average free volume

V_f and fractional free volume F_v are given as $V_f = 4 / 3 \pi R^3$ and $F_v = C V_f I_3$ respectively where C is a structural constant evaluated from an independent isochronal experiment and is determined empirically to be ~ 0.0018 [5]. Table presents the data obtained from τ_3 for the values of R , V_h and F_v along with the S-parameter for the pristine and irradiated samples.

Table: The lifetime parameters of o-Ps and radius of free volume hole (R), free volume (V_f), fractional free volume (f_v) and S-parameter in Polystyrene (PS).

Fluence (ions/cm ²)	τ_3 (ns)	I_3 (%)	R (Å)	V_f (Å ³)	Fv	S-Parameter
Pristine	2.099±0.01	28.2±0.3	2.945	106.970	5.43	0.4585
5x10 ¹⁰	2.098±0.01	28.6±0.3	2.943	106.752	5.45	0.4565
5x10 ¹¹	2.096±0.01	28.4±0.3	2.942	106.644	5.45	0.4593
5x10 ¹²	2.109±0.01	27.4±0.3	2.953	107.844	5.32	0.4597
3.5x10 ¹³	2.056±0.01	24.9±0.3	2.906	102.776	4.44	0.4581

Free volume values change slowly with ion fluence with a decrease at the highest fluence of 3.5x10¹³ ions/cm². The decrease in τ_3 implies some shrinking of inner and inter-chains of free volume holes (i.e. the impact structure was attained). o-Ps intensity I_3 remains constant up to 5x10¹¹ ions/cm² and then decreases regularly. This decrease in I_3 that reflects a decrease in the number of free volume holes may be interpreted on the process of cross-linking. The cross-linking is known to be most effective process in polystyrene as bombarded with high-energy particles [6] due to free radical recombination and various degradation possibly resulting in change of amorphousity towards crystallinity. An increase in crystallinity for the polystyrene composite is effectively observed in the irradiation process where the decrease in I_3 with dose is thus ascribed to this effect [4]. The value of S-parameter shows a minor change with increasing fluence.

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5.2.29 Structural and mechanical properties of ion irradiated PMMA films

Manasvi Dixit¹, Kananbala Sharma¹, N.S. Saxena¹, P. K. Kulriya² and D.K. Avasthi²

¹Department of Physics, University of Rajasthan, Jaipur

²Nuclear Science Centre, New Delhi

Swift heavy ions (SHI) irradiations play a very significant role for the modification of the properties of materials. High energy ion irradiation of polymers leads to remarkable changes in their physical and chemical properties [1]. Permanent modifications in the molecular weight distribution and solubility [2], electrical [3], optical [4] and mechanical properties [5] of polymers occur. Crystallinity is also affected by ion irradiation and exhibit a decrease in crystallinity at high fluence irradiation. The crystallinity plays a crucial role in almost all polymer properties such as mechanical, optical, electrical and even thermal properties.

In this present study of PMMA films were exposed a beam of 50MeV of Li^{3+} ions with an ion fluence ranging from 10^{10} to 10^{12} ions/cm² 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 100 μm thick PMMA is electronic in nature. The modifications have been characterized by XRD analysis and FTIR Spectroscopy. The mechanical properties have also been done of pristine and irradiated PMMA through Dynamical mechanical analyzer (DMA).

The XRD spectra of the polymer films before and after SHI irradiation are presented in Fig.1. The diffraction pattern of virgin PMMA sample indicates that this polymer is amorphous in nature and shows prominent X-ray peak at $2\theta = 13.1^\circ$. Upon SHI irradiation the intensity of this peak increases upto 10^{10} ions/cm² fluence and after this fluence, a decrease in intensity has been observed i.e. the degree of crystallinity of the PMMA films increases with the increase in ion fluence upto 10^{10} ions/cm² due to systematic alignment of polymer chains by chain folding.

FTIR spectra of pristine and irradiated polymer films are presented in Fig.2. The IR analysis revealed that the chemical structure of the PMMA was varied on irradiation. The

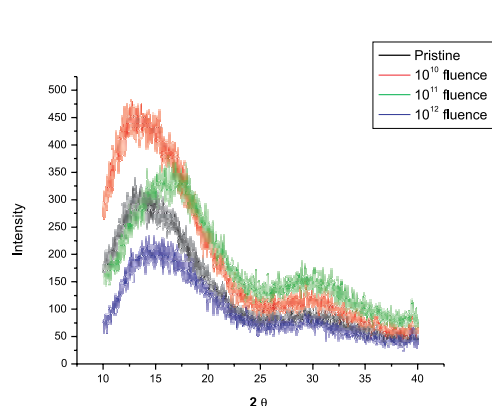


Fig.1. XRD pattern of pristine and irradiated PMMA polymer film

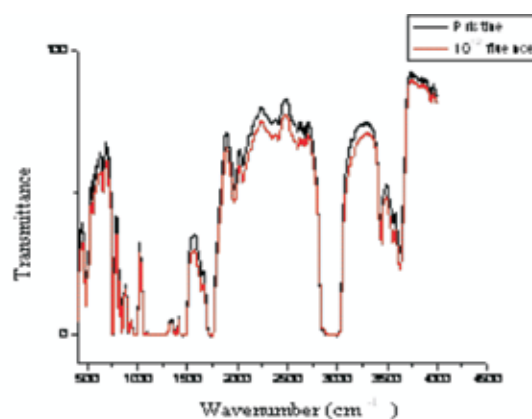


Fig.2. FTIR spectra of pristine and irradiated PMMA polymer film

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 fluences. The stress-strain behaviour of pristine and irradiated PMMA films at room temperature was also studied.

The mechanical properties like ultimate tensile strength, elongation at break and fracture energy of pristine and irradiated PMMA films are listed in table 1. From this table it is concluded that all the mechanical properties of PMMA increases upto 10^{10} ions/cm² fluence and then falls rapidly with the increase of fluence. This can be easily established by observing the appreciable increase in crystallinity of PMMA film, upto 10^{10} ions/cm² fluence.

Table 1: Mechanical properties of pristine and irradiated PMMA films

Sample	Ultimate tensile strength (MPa)	Elongation at break (%)	Fracture energy (J x 10 ⁻³)
Pristine PMMA	5.63	0.76	0.26
10 ¹⁰ fluence	24.48	2.05	3.26
10 ¹¹ fluence	17.59	1.08	1.01
10 ¹² fluence	12.89	1.01	0.58

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5.2.30 Effect of Li⁺ ion irradiation on electrical characteristics of thin film polymers

P.N. Gupta¹, G.K. Prajapati¹, R. Roshan¹ and R. Kumar²

¹Physics Department, Banaras Hindu University, Varanasi

²Inter University Accelerator Centre, New Delhi

Ion conducting polymers with their excellent processibility and potential commercial applications in electrochemical devices have made them an important class of materials for research purposes. Various methods like plasticization, addition of acids, salts and fillers

can be employed to modify the properties of polymeric systems in desirable manner. Ion irradiation of sufficient energy and appropriate doses has also been found to substantially modify the properties of polymers [1, 2].

In the present study thin film of polyvinyl alcohol having thickness ranging from 50 μ m to 200 μ m were irradiated with 50MeV Li³⁺ ions for five different ion fluences viz. 5e10, 1e11, 5e11, 1e12 and 5e12 ions/cm² at IUAC, New Delhi. The frequency dependent capacitance and dielectric loss for the samples were recorded at room temperature in wide frequency range from 75 kHz – 5 MHz using Agilent Precision LCR meter (model no. 4285A) and RS-232 interfacing with a PC using LABVIEW program. The variation of conductivity with frequency and ion fluence for pure PVA, PVA: H₃PO₄, PVA-PEG-Al₂O₃-NaI and PVA-PEG-Al₂O₃-KI samples have been studied.

Fig.1 shows the variation of conductivity with ion fluences for PVA-PEG-Al₂O₃-X (X=NaI, KI) along with PVA and PVA-H₃PO₄. It is clear from the figure that with the increase in ion fluence, conductivity first increases for NaI and KI systems while it decreases for pure PVA and PVA-H₃PO₄. This behaviour may be attributed to the fact that at lower fluences, main chain scission occurs which increases the amorphicity leading to increase in conductivity for salt systems while for pure and acid doped systems, crosslinking of main chains and broken fragments become dominant leading to decrease in conductivity [3]. At higher ion fluences, the variation in conductivity for the systems may be the result of combined effect of main chain scission and crosslinking. Fig.2 shows the variation of conductivity with frequency for irradiated PVA-H₃PO₄ sample at room temperature. Conductivity increases with increasing frequency and a relaxation is observed around 15 MHz for all irradiated samples.

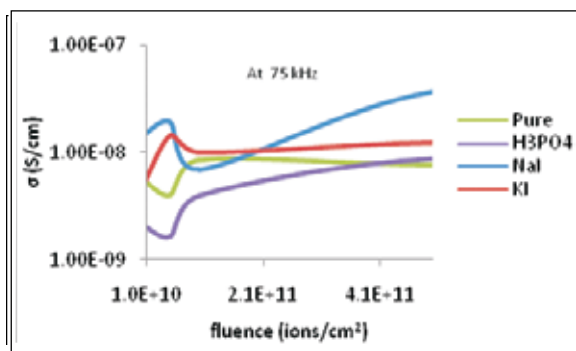


Fig. 1. Variation of conductivity vs. ion fluence

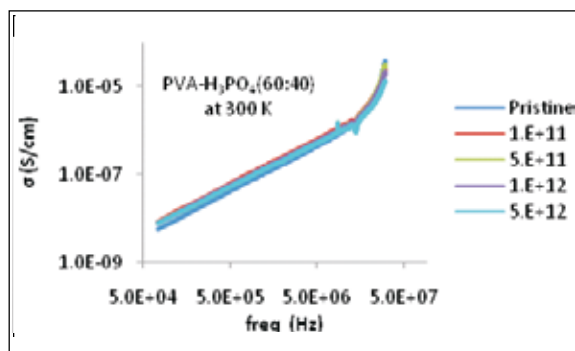


Fig. 2. Variation of conductivity vs. frequency

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5.2.31 Effect of Nitrogen Ion Implantation on the Structural and Optical Behavior of CR-39, Polycarbonate and Kapton Polymers

Tanu Sharma¹, Suman Mahendia¹, Sanjeev Aggarwal¹, Shyam Kumar¹, P. Kumar² and D. Kanjilal²

¹Department of Physics, Kurukshetra University, Kurukshetra

²Inter University Accelerator Centre, New Delhi

The samples of CR-39, Polycarbonate and Kapton were implanted to 100 keV N⁺ ions at doses varying from 10¹⁵ to 10¹⁷ ions/cm², utilizing the Low Energy Ion Beam Facility (LEIBF) at Inter University Accelerator Centre (IUAC), New Delhi. The implantation induced changes in the optical properties of these samples have been studied through UV-Visible absorption spectroscopy, while the structural changes have been revealed through RAMAN and GXRD techniques. The optical band gap of these polymers has been found to be reduced considerably with increasing implanted dose. The extent of lowering of optical band gap has been found to be maximum in CR-39 and minimum in Kapton with increasing ion dose. The changes in the structural behavior of these polymers as a result of implantation indicated clearly the formation of a three dimensional carbonaceous network consisting of disordered bonds in the implanted region of these samples. The final objective of our study is to correlate the change in optical and structural properties of these polymers as a result of implantation.

5.2.32 Statistical fluctuations in energy loss for swift heavy ions in thick polymeric absorbers

Pratibha¹, Neetu¹, Vishal Sharma², P.K. Diwan³, Shyam Kumar¹, S.A. Khan⁴ and D.K. Avasthi⁴

¹Department of Physics, Kurukshetra University, Kurukshetra, Haryana

²Department of Physics, Lovely Professional University, Phagwara

³Department of Physics, U.I.E.T., Kurukshetra University, Kurukshetra, Haryana

⁴Inter University Accelerator Centre, New Delhi

Both energy loss and straggling have been measured in thick polymeric foils for Li, C, O, Si and S ions at different energies, using Inter University Accelerator Centre (IUAC), New Delhi. These measurements have been performed in two different runs. In these runs, we performed the measurements at low energy and hence try to touch the Bragg's peak region. This is very crucial region because of the fluctuations of charge state of the incident ions. Hardly any such measurements have been compared with the corresponding theoretical/ semi-empirical formulations. Some new trends in energy loss straggling in thick foils have been observed. In the light of the present experimental data, we are in the process of modifying

Bethe-Livingston theory for Collisional energy loss straggling by introducing correlation effects.

5.2.33 Study of Charge Response of Polythylene Terephthalate (PET) Using 2.7 MeV/n ^{56}Fe and 3.9 MeV/n ^{32}S ions

S. Dey¹, A. Maulik¹, S. Raha^{1,2}, S.Saha³, S.K.Saha^{1,2}, D. Syam⁴

¹Centre for Astroparticle Physics and Space Science, Bose Institute, Kolkata

²Department of Physics, Bose Institute, Kolkata

³Saha Institute of Nuclear physics, Kolkata

⁴Department of Physics, Barasat Government College, Kolkata

The search for exotic particles (eg. Strangelets) in cosmic rays is an active field of research. An ideal choice of detectors for investigating such rare events at very high mountain altitudes is solid state nuclear track detectors (SSNTDs). We are using a commercially available plastic (PET) as SSNTD. A sensitive charge response characteristic is a prerequisite for any detector. Previously we had studied the charge response of the PET detector with exposure to ^{16}O and ^{238}U ions [1]. In order to better understand the charge response characteristic of the PET detectors, further studies were conducted by irradiating those detectors with 150 MeV ^{56}Fe & 125 MeV ^{32}S ion beams from the pelletron accelerator at IUAC, New Delhi.

5 cm × 5 cm pieces of PET as well as CR-39 were mounted on aluminium holders which in turn were mounted on top of two movable arms inside the GPSC facility. The energies with which the ions were incident on the detectors after scattering from the target (250 $\mu\text{g}/\text{cm}^2$ gold foil) was varied by placing the two arms at various angles with respect to the incoming beam. The beam current was ~ 1 pA for both Fe and S beams.

Fig 1(a) and 1(b) show the calibration curves obtained for Fe and S respectively.

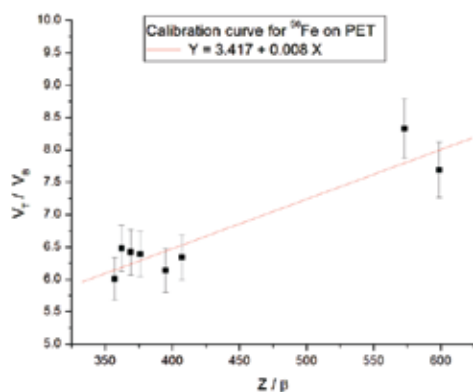


Fig. 1(a)

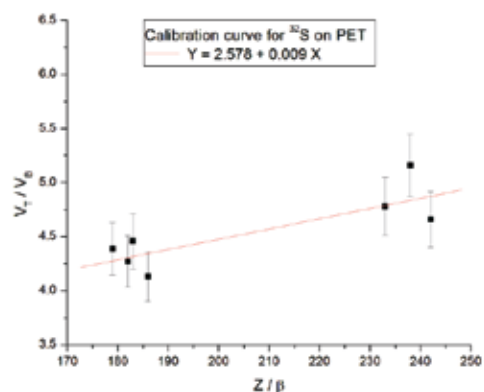


Fig. 1(b)

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5.2.34 Synthesis and Characterization of Track Etched Membranes from Teflon-FEP Films by Swift Heavy Ions Induced Graft Copolymerization

Inderjeet Kaur¹, Sunita Rattan², Nitika Gupta¹, D.K Avasthi³, A Tripathi³ and S.A.Khan³

¹Himachal Pradesh University, Department Of Chemistry, Shimla

²Amity School Of Engineering And Technology, Bijwasan, New Delhi

³Inter- University Accelerator Centre, New Delhi

Recently ion beam surface modification of polymers has shown great potential for improving surface properties of polymeric materials. The polymeric films irradiated with SHIs can be chemically etched to obtain nano or micro porous membranes with latent tracks and the grafting in these tracks by different monomers will enhance the functional properties of the membranes.

Teflon-FEP and polyvinylidene fluoride films have been irradiated with silicon ions using low fluences ranging between 6×10^5 and 1×10^6 ions/cm². To utilize the optimum conditions of grafting reactions, grafting of polar monomers viz. 4-vinyl pyridine (4-VP) and methacrylic acid (MAAc) on to pristine Teflon-FEP film by preirradiation method was carried with varying reactions conditions. Maximum percentage of grafting (Pg) of 4-VP (13.66 %) was obtained at an optimum total dose of 58.3 kGy at [4-VP] = 1.9 moles/L while MAAc produced 66.40% of maximum grafting at optimum conditions of 39.74 kGy total dose using [MAAc] = 2.5 moles/L in 10 ml of water. These optimum conditions obtained were then used to graft 4-VP and MAAc onto Teflon-FEP film and polyvinylidene fluoride irradiated at different fluence with Swift heavy ions (SHIs).

It was observed that better grafting of 4-VP was obtained in the SHI irradiated Teflon-FEP films while MAAc produced lower grafting as compared to the pristine FEP film. Maximum P_g (17.85%) of 4-VP was obtained at fluence 3×10^5 while in case of MAAc maximum P_g (39.53 %) was obtained at fluence 3×10^{11} .

The grafted films were characterized by FTIR spectroscopy, atomic force microscopy and UV-visible spectroscopy. Ion uptake studies from electrolytic aqueous solutions of sodium chloride were carried out by measuring the conductance of the solution before and after the treatment with the grafted films. The results showed that these membranes have affinity for Na⁺ ions and Cl⁻ ions and hence can be used in desalination processes for the separation of ions Thus these modified films can be used as potential materials

for separation of a variety of mixtures and are further being investigated for their use in membrane technology.

5.2.35 Mechanism of charge transport in 100 MeV swift heavy ions (silver (Ag^{8+})) beam irradiated polypyrrole

Amarjeet Kaur¹, Anju¹ and D.K. Avasthi²

¹Department of Physics and Astrophysics, University of Delhi, Delhi

²Inter University Accelerator Center, New Delhi

The fully undoped films of polypyrrole were irradiated with different fluences as 10^{10} , 10^{11} and 10^{12} ions/cm² of 100 MeV silver (Ag^{8+}) ions, where polypyrrole films were prepared by electrochemical polymerization technique [1]. In order to see the effect of radiation, comparative study between irradiated and unirradiated samples has been performed by using various techniques such as surface electron microscopy (SEM), atomic force microscopy (AFM), X-ray diffraction (XRD). An increase in conductivity with increase in fluence has been explained in light of reordering of polymer chains. The temperature dependence of dc conductivity of irradiated as well as unirradiated samples has been investigated in 77-300K. The observed conduction behavior of all the samples has been explained in terms of Mott's variable range hopping model [2] involving a single phonon process.

It has been observed by SEM and AFM images that the surface structure becomes smooth and polymer chains get more organized due to irradiation. Therefore, the conductivity increases by two orders. The room temperature conductivity of pristine sample is 6.5×10^{-7} while for the sample irradiated with highest fluence of 10^{12} ions/cm² is 9.4×10^{-5} . The increased degree of crystallinity leads to higher values of conductivity due to decreased scattering of charge carriers. The conductivity of all the irradiated samples remain stable under atmospheric conditions, for more than one year whereas the conductivity of unirradiated samples, changes by $\pm 15\%$, when kept under atmospheric conditions, over a period of time. The conductivity here can be assumed to consist of two components, namely σ_B and σ_H such that the total dc conductivity $\sigma_{dc} = \sigma_B + \sigma_H$. The intrachain conductivity σ_B can be described by the mechanism for band conduction model where as σ_H is contribution due to charge transport between the chains. With the organization of the polymer chains under the influence of silver ion irradiation the contribution of σ_H increases. However, the conductivity of doped conducting polymers decay over the period of time. In present investigations, by irradiating the polymers, the structural modification of the polymers is done so as to increase the conductivity, without using any dopant ion and there is no decay in conductivity over a period time, which is major achievement. To date no such report is available in which the charge transport in swift heavy ion irradiated conducting polymer has been discussed.

The variation of dc conductivity as a function of $1000/T$ (Fig.(a)) and $T^{-1/4}$ (Fig.(b)) in the temperature range 77-300K has been plotted for irradiated and unirradiated samples. No crossover of dependence of conductivity from $T^{-1/2}$ to $T^{-1/4}$ is observed. Therefore results have been analyzed in the light of Mott's variable range hopping (VRH) model [3-6]. In this model the dc conductivity shows the temperature dependence of type T^n where $n = 1/1+d$, where d is the dimensionality. Therefore, Mott's model suggests $n=1/2$ for one dimensional hopping, $n = 1/3$ for two dimensional hopping and $n = 1/4$ for three dimensional hopping. It was evident that linear dependence of conductivity data in the entire temperature range of measurement exists only for $T^{-1/4}$. So, 3D VRH seems to be dominant. Although crystallinity (results not shown), increases with fluence but over all charge transport mechanism has not been affected by it. The observed temperature dependence of activation energy rules out band conduction, in unirradiated as well as irradiated samples. Therefore, it further supports that hopping conduction may be the dominant mechanism of charge transport. In the present situation, it can be stated that each time the charge carrier moves between the polymer chain, the charge carrier just below the Fermi level jumps to a state just above it with energy W and transfers from one chain to other (adjacent chain) of which wave function overlaps that of the first chain.

The dc conductivity measurement showed that the conductivity has increased by about two orders of magnitude which are attributed to better organization and reordering of chains, due to ion impact. The observed temperature dependent dc conductivity for unirradiated as well as irradiated samples cannot be explained by the band conduction. However, same is well represented by 3D Mott's VRH model involving single phonon processes

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5.2.36 SHI induced effect on metal dispersed polymer composites

N.L.Singh¹, Sejal Shah¹, Anjum Qureshi¹, Dolly Singh¹, F.Singh² and D.K.Avasthi²

¹Physics Department, M.S.University of Baroda, Vadodara

²Inter University Accelerator Centre, New Delhi

Polymer composites were prepared by doping metals (Ni, Ag, Cu) and organometallic compounds (ferric oxalate, Palladium acetylacetonate, Ni-Dimethylglyoxime) in PMMA by chemical method. The films of thickness $\sim 100 \mu\text{m}$ were prepared by casting method. The films were irradiated with 120 MeV Ni^{10+} ion beam at the fluences of 1×10^{11} and 5×10^{12} ions/ cm^2 using 15 UD Pelletron facility at Inter University Accelerator Centre, New Delhi. Physical and chemical properties of pristine and irradiated samples were characterized by means of various measurement techniques.

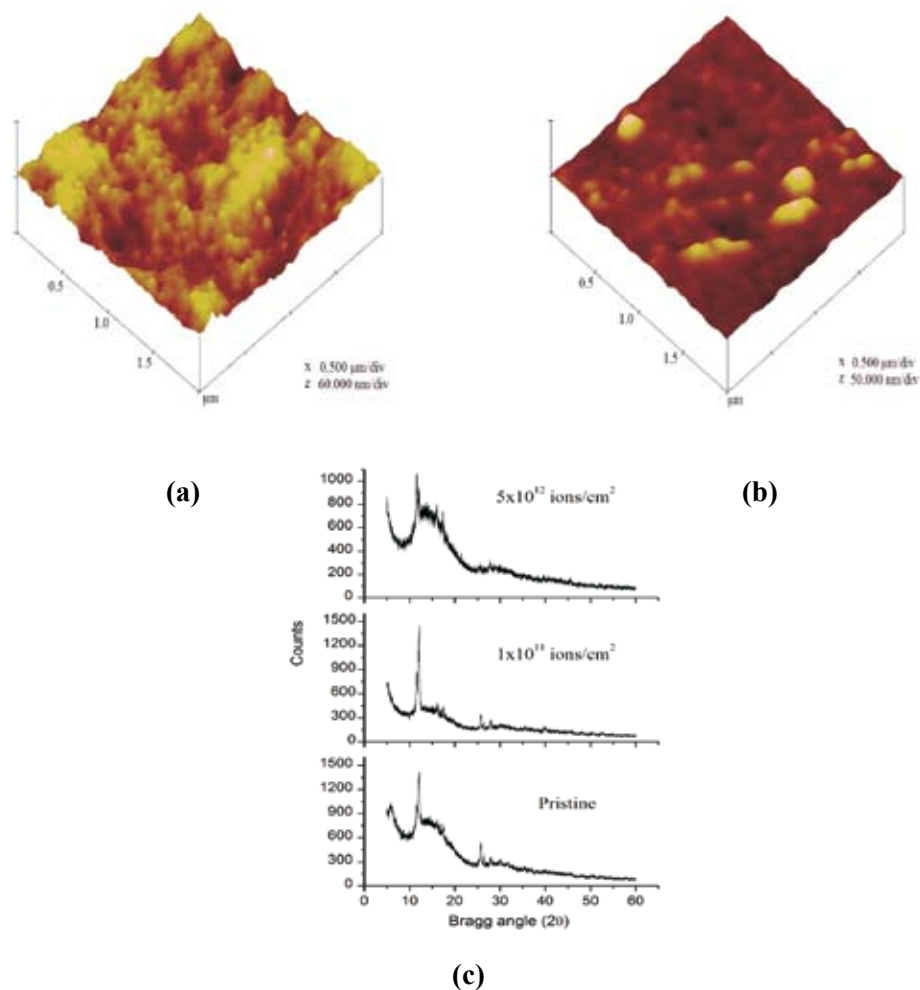


Fig. 1. AFM images of (a) pristine and (b) irradiated (5×10^{12} ions/ cm^2) films of PMMA+ 15% Fo composites, (c) XRD spectrum of pristine and irradiated PMMA+10%Pd(acac)

The surface morphology of pristine and irradiated films of Fo dispersed PMMA films was studied in terms of surface roughness by AFM on $2 \times 2 \mu\text{m}^2$ area. The average roughness values of 15% Fo dispersed PMMA films were obtained to be 24 nm and 33 nm for pristine and irradiated films respectively. SHI on polymers leads, in general, to an increase in surface roughness due to large sputtering effects.

XRD analysis of pristine and irradiated Pd(acac) dispersed PMMA was done. Results show that crystallite size decreases slightly upon irradiation. No considerable change in the peak position is observed which reveals that the lattice parameters do not change significantly. The decrease in intensity and broadening of the peak after irradiation indicate a decrease in crystallinity and evolution of the composite towards a more disordered state. The samples are also analyzed for its dielectric properties and surface morphology and the results have been published.

5.2.37 Effect of SHI on magnetic properties of NiO nanoparticles doped PMMA composites

N.L.Singh¹, Chaitali Gavade¹, Dolly Singh¹, Sejal Shah¹, Anjum Qureshi¹, Pawan Kularia², D.K.Avasthi² and Alok Banerjee³

¹Department of Physics, M. S. University of Baroda, Vadodara

²Inter University Accelerator Centre, New Delhi

³UGC-DAE, Consortium for Scientific Research, Khandawa road, Indore

Polymers have traditionally been considered as excellent host matrices for composite materials. Inclusion of magnetic nanoparticles in polymers have been shown promise in various potential applications like spin polarized devices, carriers for drug delivery, magnetic recording media, high frequency applications etc. Another useful aspect of polymer host matrices is that they provide for possibilities of mediating nanocomposites including dipolar, exchange (isotropic and anisotropic), superexchange and magnetoelastic interactions [1].

PMMA was synthesized by solution polymerization technique. We also synthesized NiO nanoparticles by chemical route [2]. These particles were dispersed in PMMA by acetone solvent. The composite films of different concentrations (2%, 6% and 10%) of NiO powder in PMMA were prepared by casting methods. These films were irradiated with 50 MeV Li³⁺ ions at a fluence of 10¹² ions/cm².

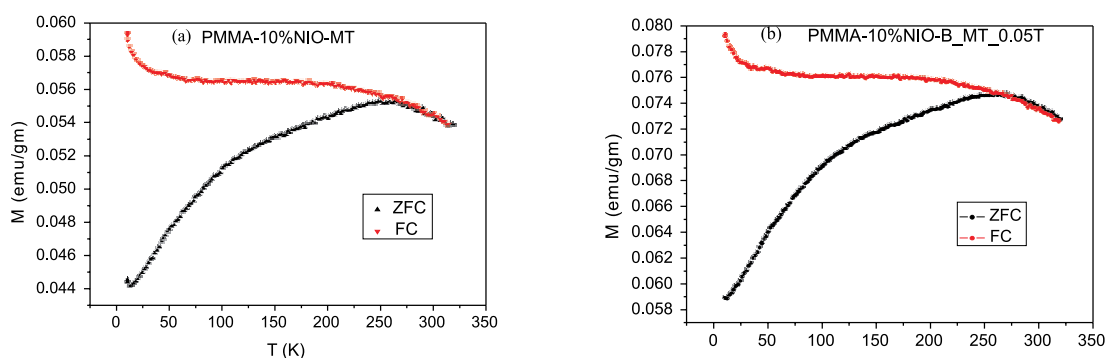


Fig. 1. Panel shows the FC-ZFC magnetization for (a) pristine and (b) irradiated samples

The Quantum Design VSM was used to measure dc magnetization. It was done using field-cooled-zero-field-cooled (FC-ZFC) susceptibility. For the zero field cooled (ZFC) measurements the sample was cooled down from room temperature to 10 K in the absence of an external magnetic field and the magnetic data were acquired during the warming run in a constant external field. In the field cooled (FC) measurements the samples were initially cooled down to 10 K in the presence of a magnetic field and the FC data were recorded during the warm up cycle in the same magnetic field. FC-ZFC measurements were done from 10-320 K at 0.05 T applied field. We have compared FC-ZFC curves for nanocomposites. None of these curves show any characteristic sharp change in magnetization associated with the well established ferromagnetic to super paramagnetic transition in single domain nanoparticles. This indicates that the particles (mostly in clusters) in the polymer matrix are predominantly ferromagnetic at room temperature [3, 4].

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5.2.38 Effect of swift heavy ion irradiation on the optical and electrical characteristics of polypropylene/TiO₂ Composite

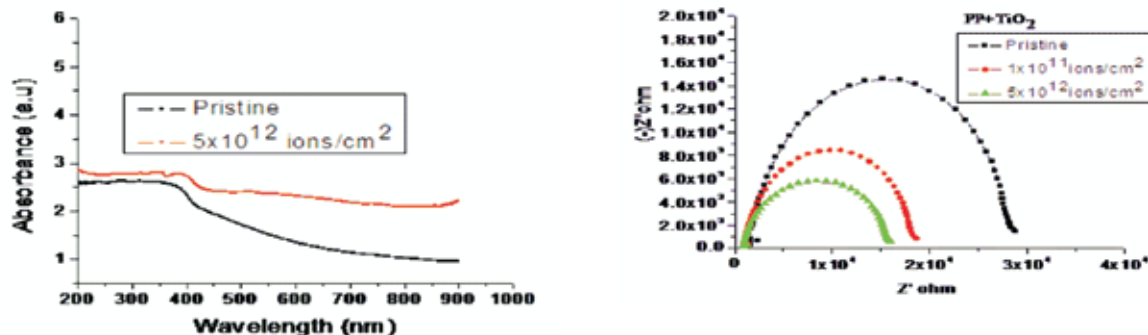
N.L.Singh¹, Dolly Singh¹, Anjum Qureshi¹, Sejal Shah¹, Pawan Kularia², Ambuj Tripathi² and D.K.Avasthi²

¹Department of Physics, M. S. University of Baroda, Vadodara

²Inter University Accelerator Centre, New Delhi

Irradiation of polymeric materials has established as one of the most acceptable approach to alter polymer properties significantly. The growing interest in the ion irradiation of polymer materials is due to the possibility of verification of the technological parameters as for the fundamental reasons and also considering some possible applications to device fabrication. Thus optical, mechanical, electrical and chemical properties can be selectively modified using ion bombardment. Ion irradiation of polymers is followed by the processes of macromolecular destruction, cross-linking, free radicals formation, carbonization and oxidation. Therefore, the understanding of certain structural rearrangements influence on the suitable electrical, optical etc. properties of the polymers opens a way to design devices with required parameters [1].

Polypropylene/TiO₂ composites of master batch in composition of 35% PP and 65% TiO₂ pellet were supplied by Dizayn group of Company, Turkey. These samples were irradiated with 140 MeV Ag¹¹⁺ ions at the Inter University Accelerator Centre (IUAC), New Delhi.



Optical and electrical characterization of pristine and irradiated films was carried out by means of UV- visible and Impedance gain phase analyzer respectively.

UV-visible absorption analysis indicates a decrease in optical band gap from 2.67 eV to 2.43 eV upon irradiation at the fluence of 5×10^{12} ions/cm². The geometrical shapes of the complex impedance plane plots indicate that the composite material is electrically equivalent to RC networks that reduce pure resistance [2]. It was observed that the diameter of the semicircles decreases as ion fluence increases. It indicates that the samples become more conductive behavior upon irradiation.

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5.2.39 Nanomaterial Filled SHI Induced Ion Tracks and their Application in Nanodevices

Nabanita Dutta¹, G A Ahmed¹, D Mohanta¹, A Choudhury¹ and F Simgh²

¹Tezpur university , Napaam, Assam

²Inter-University Accelerator Center, New Delhi

In recent years, there has been growing interest in swift heavy ion track technology [1-3]. Track-etch membranes offer distinct advantages over conventional membranes due to their precisely defined structures. Fabrication of nano particles have been tried in different matrices to investigate if it leads to changing physical, optical, electrical and

magnetic properties. Mostly used polymer matrix to be listed out is polyvinyl alcohol (PVA)[4].

Swift heavy ion (SHI) technology was used to create definite and ordered pores in PVA film. For that certain damage zones on polymer film was created by SHI irradiation. SHI irradiation has been performed in GPSC. Si ion was used of energy 100MeV. Fluence was used in the range of 1×10^5 to 7.5×10^6 . This low fluence was attained using gold foil scattering. Finally these damage zones were etched out using a suitable etchant NaOH.

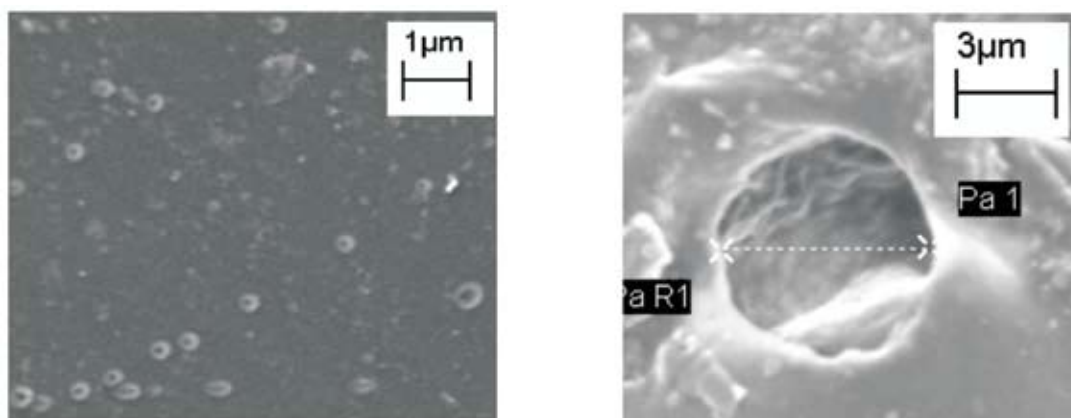


Fig. 1. SEM images of etched iontracks

From scanning electron micrographs creation of voids of almost spherical shape was confirmed. Although in SEM micrographs, voids of around 1 to 3 μm is observed yet voids of size 20 to 40 nm are also present. The diameter of the etched track depends on etching time and concentration of the etchant. The solvent used for dissolving the etchant has got small effect on it. But due to technical limitation of the instrument photographs of the voids of these much diameter could not be taken out. These voids are filled with PbS. For that the etched irradiated polymer films are washed two three times after etching and then dipped in a 1mM PbCl₂ solution and washed again and then the films are treated with Na₂S solution. In these process the Pb⁺⁺ ions were diffused in to the voids and then finally reacted with sulphur and formation of PbS nanoparticles occurred.

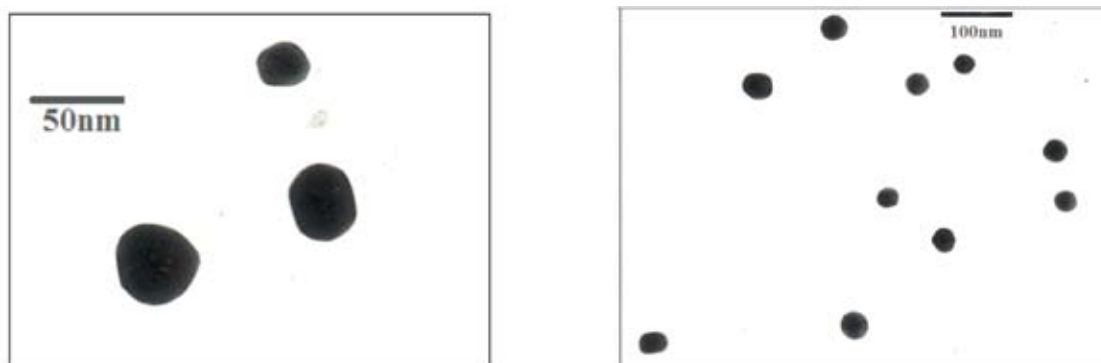


Fig. 2. TEM images of PbS filled ion etched ion tracks.

PbS nanoparticle with narrow size distribution along with uniform dispersity is obtained in this process. The advantage of using PVA films is that PVA can be again dissolve in distilled water and free standing PbS nanoparticles can be produced in this way.

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5.2.40 Magnetic Studies of SHI Irradiated Nanostructured Zinc Ferrite

Jitendra Pal Singh¹, R. C. Srivastava¹, H. M. Agrawal¹ and Ravi Kumar²

¹Department of Physics, G. B. Pant Univ. of Ag. & Tech., Pantnagar, Uttarakhand

²Inter University Accelerator Centre, New Delhi

Swift heavy ion (SHI) beam is widely used for the modification of magnetic nanostructures. For this purpose, nanostructured zinc ferrite was synthesized by using the nitrates of appropriate cations and citric acid [1]. The precursor materials in the present study were sintered at 500°C and 1000°C for 1 hr (accordingly the samples are coded as ZF500 and ZF1000). These samples were irradiated by 100 MeV oxygen beam with fluence of 1×10^{13} ions/cm² and 5×10^{13} ions/cm². The electronic stopping (S_e), nuclear stopping (S_n), and the projected range (R_p) values for oxygen beam, calculated by using the SRIM code are : $S_e = 1.09$ keV/nm, $S_n = 0.618$ eV/nm and $R_p = 65$ μ m. The value of threshold electronic stopping for producing the columnar defect in zinc ferrite is ~ 13 keV/nm[2]. This reveals that only point/cluster defects are expected after irradiation. The crystallographic phase and particle size of the pristine and irradiated samples were determined by the XRD. Further, magnetic measurements at room temperature and 10K were carried out in order to observe the change in magnetization after irradiation.

Fig. 1 shows the XRD spectra of the pristine and irradiated samples of system ZF500 and ZF1000. All the samples contain the peaks corresponding to the cubic spinel phase, beside this presence of ZnO-phase was observed after irradiation. The average particle size for the system ZF500 is 16, 10, 10 nm for the fluence of 0 (pristine), 1×10^{13} ions/cm² and 5×10^{13} ions/cm² respectively while for system ZF1000 these values are 62 nm, 46 nm and 41 nm. Similar type of reduction in particle size behavior was also observed in Ni-Zn and Mn-Zn ferrite irradiated by 190 MeV Ag¹⁵⁺ ion beams [3]. The magnetization vs applied magnetic field curves for the pristine and irradiated samples of both the systems was recorded. The

nature of M-H plots for the pristine and irradiated samples reveal that the samples are superparamagnetic even after irradiation with 100 MeV oxygen beam at RT. The observed changes have been attributed to the generation of defects and destruction of crystal structure i.e. formation of ZnO phase, which results degradation in the intrinsic parameters [4]. The increment in the magnetization for the system at 10K may be attributed to the formation of paramagnetic centers due to splitting of particles by SHIs [5].

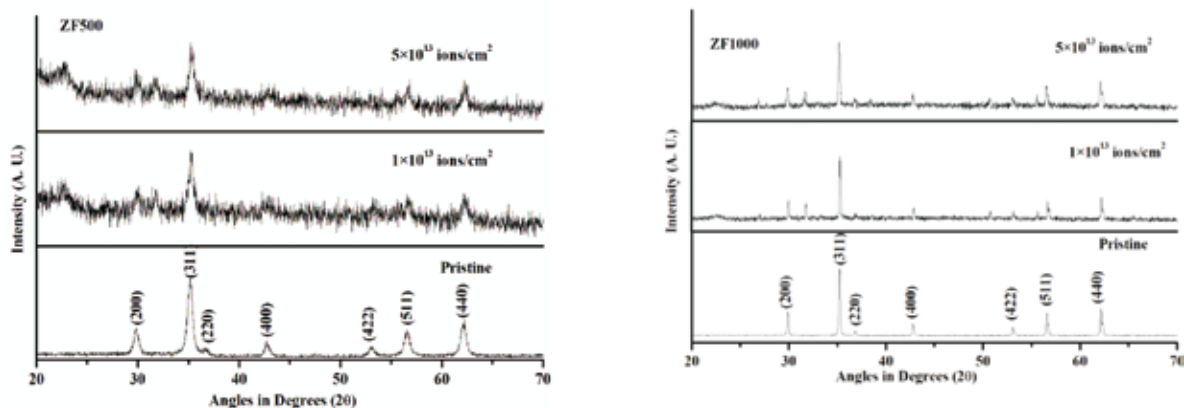


Fig1. XRD spectra of pristine and irradiated samples (a) ZF500 and (b) ZF1000

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5.2.41 Studies on the Effects of Swift Heavy Ion Irradiation on Nanostructured Titanates of Barium and Strontium in Photoelectrochemical Splitting of Water

Anjana Solanki¹, Jaya Shrivastava¹, Monika Gupta¹, Vidhika Sharma¹, Pushpendra¹, Pawan Kulriya², Saif A. Khan², Ambuj Tripathi², D.K.Avasthi², Vibha.R.Satsangi³, Rohit Shrivastav¹ and Sahab Dass¹

¹Department of Chemistry, Dayalbagh Educational Institute, Dayalbagh, Agra

²Department of Physics & Comp.Sc., Dayalbagh Educational Institute, Dayalbagh, Agra

³Inter University Accelerator Center, New Delhi

Development of photocatalysts which can efficiently split water into hydrogen and oxygen under sunlight irradiation has extensively been explored. In search for efficient photocatalysts for water splitting, the effect of various nanostructured mixed oxides are being investigated. Among them, Barium strontium titanate (BST) proves to be an interesting candidate due to its high dielectric constant, good thermal and chemical stability and high capacity of charge storage. Swift heavy ion (SHI) irradiation plays a vital role in the field of modifications of the properties of films, foils and surface of bulk solids. It penetrates deep into the materials and produces a long and narrow disordered zone along its trajectory [1]. The effect of ion beam on the materials depends on the ion energy, fluence, temperature and ion species [2]. The energy loss by high energy ions in the target mainly via inelastic collisions results in the excitation of the target electrons, which is associated with modification in the crystallographic, optical, electrical and morphological properties [3,4]. These changes are expected to affect photoelectrochemical behavior of the semiconducting materials for hydrogen production using metal oxide semiconductor. The changes in the property of semiconductor materials on account of SHI irradiation have been, for the first time studied by our group in DEI, in PEC hydrogen generation and interesting results have been obtained.

Thin films of BST were prepared on ITO coated glass substrate by sol-gel spin coating. Structural and optical properties of the deposited films were characterized by XRD and UV-Vis spectra. The X-ray diffraction profiles of BST (60/40) thin films represent the perovskite cubic elementary cell having the particle size/grain size of 51 nm (Debye Scherrer's calculation). These thin films were irradiated with 120 MeV Ag⁹⁺ and 120 MeV Si⁹⁺ ions at fluence ranging from 1x10¹² to 2x10¹³ ions/cm². The irradiated and unirradiated samples were converted into the photoelectrode using silver paste and copper wire for PEC measurements. Photoelectrochemical measurements were carried out using NaOH (13 pH) as electrolyte and a light source. A significant photoresponse of the unirradiated film (0.19 mAcm⁻²) was observed when illuminated with 150 W UV-Vis source in PEC cell. The results on the effects of irradiation on these films in terms of XRD, AFM, SEM, UV-Vis absorption and PEC studies, are awaited as the transient defects caused due to irradiation anneal with time which may in the present case affect the stability/performance of the electrode in the PEC cell [5]. The irradiated samples, therefore, have been kept for more than 60 days, after which the XRD, AFM, SEM, UV-Vis Spectroscopy and PEC studies, would be undertaken.

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5.2.42 SHI Induced Effects on the Multiferroic Behaviour of Bi-Substituted Co_2MnO_4 Thin Films

N.E.Rajeevan¹, Ravi Kumar², D.K.Shukla³, R.J.Choudhary⁴, P.P.Pradyumnan¹

¹Department of Physics, University of Calicut, Kerala

²Materials Science Division, IUAC, New Delhi

³Department of Physics, Aligarh Muslim University, Aligarh

⁴UGC DAE Consortium for Scientific Research, Indore

Multiferroic thin films - reveal a range of interesting phenomena and have stimulated the exploration of new logical devices [1-2]. Optimization of the multiferroic properties of thin films envisages a wide variety of smart devices such as multiple memory devices, electric field controlled ferromagnetic resonance devices, transducers etc. It has been well established that structural, electrical and magnetic properties of spinel oxide films are extremely sensitive to the external pressure or stress/strain which can be induced through different routes like substituting cations of different ionic radii, creating cation-oxygen vacancies/defects by swift heavy ion (SHI) irradiation [3] etc. We report the effect of SHI irradiation on the structural and multiferroic properties of thin films of Bi- substituted Co_2MnO_4 prepared by Pulsed Laser Deposition (PLD). Thin films of spinel $\text{Bi}_x\text{Co}_{2-x}\text{MnO}_4$ with varying Bi content ($x = 0.0, 0.1$ and 0.3) were grown by PLD technique under optimal conditions from single phased targets [4] on quartz and LaAlO_3 (LAO) substrates. These films were irradiated at room temperature with 200 MeV Ag^{15+} ions with the fluence values of 1×10^{11} , 5×10^{11} and 1×10^{12} ions/cm². XRD analysis revealed that the polycrystalline $\text{Bi}_x\text{Co}_{2-x}\text{MnO}_4$ films grown on quartz and LAO substrates are oriented and exhibited single phase cubic structure with low strain before and after the irradiation, which was confirmed by Raman scattering studies. Observed hardening of the Raman modes with irradiation is indicative of the slight contraction in the unit cell. The increasing background towards the higher wave numbers of the Raman spectra, retained even after the irradiation of the $\text{Bi}_x\text{Co}_{2-x}\text{MnO}_4$ films on all the substrates is suggestive of the strong electron-phonon coupling. Improvement in dielectric properties of the films was observed up to the fluence of 5×10^{11} ions/cm². Dc magnetization hysteresis loop study, showed that the $\text{Bi}_x\text{Co}_{2-x}\text{MnO}_4$ films exhibited ferrimagnetic ordering below the transition temperature $T_c \sim 185$ K and the saturation magnetization was found to increase with the irradiation at optimal fluence value 5×10^{11} ions/cm². This is due to the improved texturing and crystallinity of the film. The tunable ferroelectric and ferrimagnetic nature exhibited by the $\text{Bi}_x\text{Co}_{2-x}\text{MnO}_4$ thin films is supposed to assure a position in multiferroic device applications.

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5.2.43 Swift heavy ions induced modifications in hydroxyapatite thin films

K. Elayaraja¹, P. Rajesh², S. P. Parthiban¹, M.I. A. Joshy¹, R.V. Suganthi¹, R. Vani¹, V. Sarath Chandra¹, P. K. Kulriya³, I. Sulania³, F. Singh³, K. Asokan³, H. K. Varma² and S. Narayana Kalkura¹

¹Crystal Growth Centre, Anna University, Chennai 600025

²Biomedical Technology Wing, Sree Chitra Tirunal Institute for Medical Sciences and Technology, Poojappura, Thiruvananthapuram

³Inter-University Accelerator Center, New Delhi

Synthetically prepared hydroxyapatite [HAp, Ca₁₀(PO₄)₆(OH)₂] is the most suitable ceramic material for hard tissue replacement implants as it directly bonds with bone tissues [1]. The surface properties of implant materials, such as surface roughness, chemical composition, electrical charge, porosity and wettability play an important role in binding with living cells [2]. Ion beam irradiation is known to be a powerful tool for surface modification of thin films [3-5], as it provides selective surface modification without affecting the bulk properties and reproducibility, further it improves osseointegration [6].

Hydroxyapatite thin films were prepared by Pulsed Laser Deposition (PLD). The thin films were irradiated by 60 MeV Si⁵⁺ beam with four different fluences viz. 1×10¹¹, 1×10¹², 1×10¹³ & 1×10¹⁴ ion/cm². Similarly, 100 MeV Ag⁷⁺ beam with four different fluences viz. 1×10¹⁰, 1×10¹¹, 1×10¹² & 1×10¹³ ions/cm² were irradiated on the samples. The samples were characterized by glancing angle X-ray diffraction (GIXRD), atomic force microscopy (AFM) and photoluminescence spectroscopy (PL).

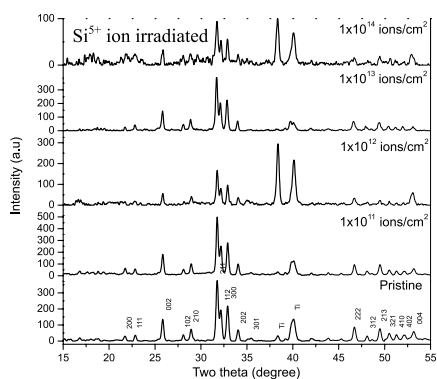


Fig. 1a. silicon (Si⁵⁺)

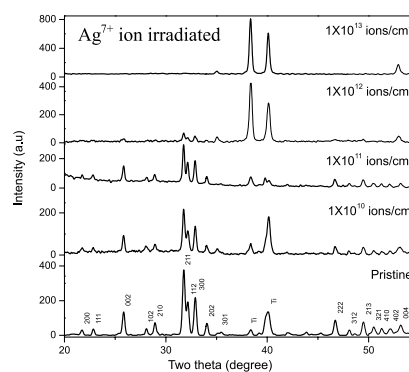


Fig. 1b. silver (Ag⁷⁺) ion

XRD spectra of the irradiated samples were as shown in Fig (1a & 1b). The pristine and irradiated samples showed the presence of pure phase of HAp. In the case of irradiated samples, the peak intensity corresponding to the plane (211) decreased as the fluence increases leading to lower crystallinity.

AFM revealed the variation in the particle size and roughness of the samples on irradiation (Table1). Irradiation induced pores (~ 190 nm) were found in all samples. Samples exhibited ionoluminescence. Presence of phosphorus in HAp is likely to be the origin of ionoluminescence. Irradiated samples exhibited very high intensity of luminescence in the visible range (450 - 750 nm) compared to the pristine samples.

Further characterization and analyses using Raman and bioactivity studies are under progress

Table 1

Fluence	Roughness (nm)(± 1)	Average particle size (nm) (± 1)	Pours (nm) (± 1)
Si⁵⁺ ions			
Pristine	49.67	138	-
1x10 ¹¹	60.30	157	160.
1x10 ¹²	68.53	192	191.87
1x10 ¹³	81.13	186	185.90
1x10 ¹⁴	50.40	133	133.00
Ag⁷⁺ ions			
1x10 ¹⁰	163.50	200	102.21
1x10 ¹¹	100.10	195	107.17
1x10 ¹²	50.12	190	111.17
1x10 ¹³	47.12	180	109.53

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5.2.44 Li ion beam irradiation induced transformations in Liquid Crystalline Materials

Rohit Verma^{1,2}, R. Dhar^{1,2} and Ambuj Tripathi³

¹Physics Department, Ewing Christian College, Allahabad

²Physics Department, University of Allahabad, Allahabad

³Inter University Accelerator Center, New Delhi

Devices, made up of liquid crystalline materials, are often used in high radiation environments such as nuclear installation centers and space applications. When these devices are used in such environments for a long time, different types and doses of irradiation affect these devices and often create the malfunctioning of these devices. Some of the earlier studies on the effect of different types and doses of irradiation suggest that generally the peak transition temperatures, transition enthalpies and transition entropies are lowered, while the conductivity of the materials are increased [1-3]. On the other hand different types of radiations are used to modify the various properties of different polymers (made up of long chain molecules same as LC molecules) [4-6]. For this reason we have irradiated some of the liquid crystalline materials (Cholesteryl Myristate (ChM), Cholesteryl Pelargonate (ChP), Hexyloxybenzoic acid (HOBA), Decyloxybenzoic Acid (DOBA) and Nonyloxybenzoic acid (NOBA)) by Li ion beam of different fluence and studied the radiation induced transformations in the physical properties of these compounds.

The experiment was performed by Li ion beam using current 3 nA. The pure and irradiated materials were characterized by Differential Scanning (DSC) Calorimetry and UV-Visible spectroscopy. The DSC Thermograms for the pure and irradiated (1e13 fluence of Li ion beam) DOBA at the scan rate of 5 °C/min were recorded. The thermodynamical analysis of the pure and irradiated samples suggests that all the peak transition temperatures and transition enthalpies are surprisingly increased for the irradiated DOBA as compared to pure DOBA. The ultra-violet spectra shows the variation of the absorbance (Arb. Unit) with wavelength (nm) of the pure and irradiated NOBA and DOBA respectively. We can clearly see that in the spectrum of the irradiated NOBA an additional peak at around 335 nm with the pure NOBA peak at 288 nm. Similarly in the spectrum of the irradiated DOBA we find an additional peak at around 337 nm with the pure NOBA peak at 290 nm. This additional peak clearly suggests the formation of new species in the irradiated samples.

We are in the process of studying the changes in the dielectric and electro-optical properties of these irradiated compounds.

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5.2.45 Effect of 200 MeV Ag¹⁵⁺ Ion Irradiation on Multiferroic BiMn₂O₅

D K Shukla¹, Ravi Kumar², S Mollah¹, P Thakur³, R J Chaudhary⁴, S K Sharma⁵, N B Brookes³ and M Knobel⁵

¹Department of Physics, Aligarh Muslim University, Aligarh

²Inter University Accelerator Centre, New Delhi

³European Synchrotron Radiation Facility, Grenoble Cedex, France

⁴UGC-DAE Consortium for Scientific Research, Indore 452001, India

⁵Instituto de Fisica Gleb Wataghin, Universidade Estadual de Campinas (UNICAMP), Campinas, 13.083-970, SP, Brazil

A modeled irradiation experiment has been performed using 200 MeV Ag⁺ ion as a projectile ion and Pulse Laser Deposition (PLD) grown thin film of magnetically frustrated multiferroic BiMn₂O₅ as a target. With increasing irradiation fluence, isotropic increase in the unit cell parameters and simultaneous decrease in grain size in primarily strained structure is observed, which may be the consequence of new higher energetic configuration

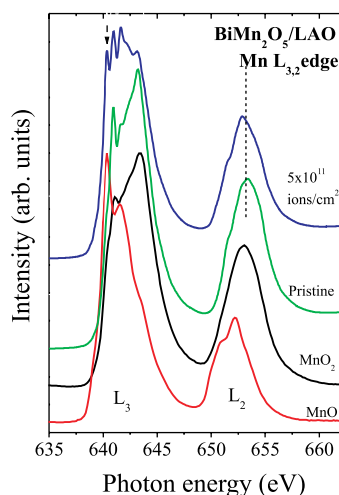


Fig. 1. Normalized Mn L_{3,2}- edge NEXAFS spectra of pristine BiMn₂O₅ thin film and irradiated at 5 x 10¹¹ ions/cm² along with the spectra of MnO and MnO₂. Arrow marked peak in irradiated plot clearly show the evolution of Mn²⁺ in Mn³⁺/Mn⁴⁺ network.

after irradiation. Interestingly, irradiation induced FM is observed in a primarily AFM character. Element specific observations using NEXAFS and XMCD show the evolution of Mn^{2+} in a network of Mn^{3+}/Mn^{4+} at the expense of Mn^{4+} . Calculations based on sum rule and orbital moment consideration using XMCD data show that spin moment is dominant at room temperature and at low temperature, the orbital moment dominates in irradiated films, whereas pristine film show the completely quenched orbital moment at low temperature. These findings spur that the swift heavy ion irradiation can be used to custom-tailor the properties of oxide multiferroics for technological application.

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5.2.46 Densification induced crack formation in SHI irradiated NiO thin film

P. Mallick¹, Chandana Rath², D.C. Agarwal³, R. Biswal⁴, D.K. Avasthi³, D. Kanjilal³, A. Tripathi³, P.V. Satyam⁵ and N.C. Mishra⁴

¹Department of Physics, North Orissa University, Baripada

²School of Material Science & Technology, IT, BHU, Varanasi

³ Inter-University Accelerator Center, New Delhi

⁴Department of Physics, Utkal University, Bhubaneswar

⁵Institute of Physics, Bhubaneswar

Antiferromagnetic Mott-Hubbard insulator NiO is an attractive material due to its wide variety of potential applications. The response of NiO to SHI irradiation was found to be different as compared to many oxide systems. The NiO thin film on Si (100) prepared by e-beam evaporation method [1] and subsequently annealed at 700°C were irradiated with 120 MeV Au ions at IUAC, New Delhi. The surface morphologies of the pristine as well as irradiated NiO thin films were studied by SEM and AFM. The pristine NiO films were crack free as judged from SEM micrograph (Fig. 1a). Cracks are seen to evolve in the films on 120 MeV Au ion irradiation at a fluence of 3×10^{12} ions cm^{-2} (Fig. 1b). The cracks widen with increase of irradiation fluence. Cracks have also been observed in NiO films on SiO_2 by 230 MeV Xe ion irradiation [2]. In this case, irradiation was done at very high fluences (1.7×10^{13} to 1.7×10^{14} ions cm^{-2}) at a tilt angle of 75° , while the sample was held at 80 K. We however observe initiation of crack formation at a fluence, which is an order of magnitude lower (Fig. 1b) than the above. Further the ion beam was incident perpendicular to films surface and

irradiation was done at RT in our case. The AFM micrograph of the film irradiated at the fluence of 3×10^{12} ions cm^{-2} (Fig. 1d) shows the growth of NiO grains as compared to that of pristine film (Fig. 1c). The observation of crack formation in the NiO film at the same fluence of irradiation where the grain growth takes place may be due to the consequence of the fusion of smaller grains into the bigger ones during the wake of SHI.

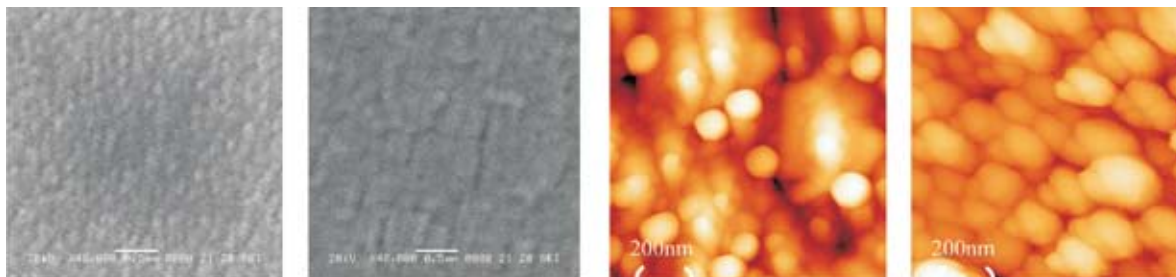


Fig. 1. SEM micrograph of 700 °C sintered NiO (a) pristine film, (b) the film irradiated at a fluence of 3×10^{12} ions. cm^{-2} , $1 \mu\text{m} \times 1 \mu\text{m}$ AFM image of (c) pristine film and (d) the film irradiated with 3×10^{12} ions. cm^{-2} .

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5.2.47 Effect of 150 MeV Ni^{11+} Ion Irradiation on Structural Properties of CuFe_2O_4 Ferrite Thin Films

D. Pathinettam Padiyan¹, M. Balaji¹, T. R. Raja sekaran¹, C. Sanjeeviraja², K. Asokan³ and D. Kanjilal³

¹Department of Physics, Manonmaniam Sundaranar University, Tirunelveli

²Department of Physics, Alagappa University, Karaikudi

³Inter University Accelerator Centre, New Delhi

CuFe_2O_4 nanomaterial was synthesised by Co–Precipitation method and thin films have been prepared using R. F. Magnetron sputtering system on Quartz substrates. A constant field of 200 W was used for coating and the substrate temperature was maintained at 200° C. The thickness of the films was varied by coating time (10 min, 20 min and 60 min) and the thickness was measured by optical method. The structural property of the pristine and irradiated films was confirmed by X-ray diffraction (XRD) using PANalytical X-Pert Pro Multipurpose diffraction system. The polycrystalline nature was noticed when the thin films were annealed at 1200° C for 8 hours. Thin films of various thicknesses were irradiated

with 150 MeV of Ni¹¹⁺ ions at the fluence of 10¹⁴ ions/cm² using 15 UD Pelletron at Inter University Accelerator Centre, New Delhi.

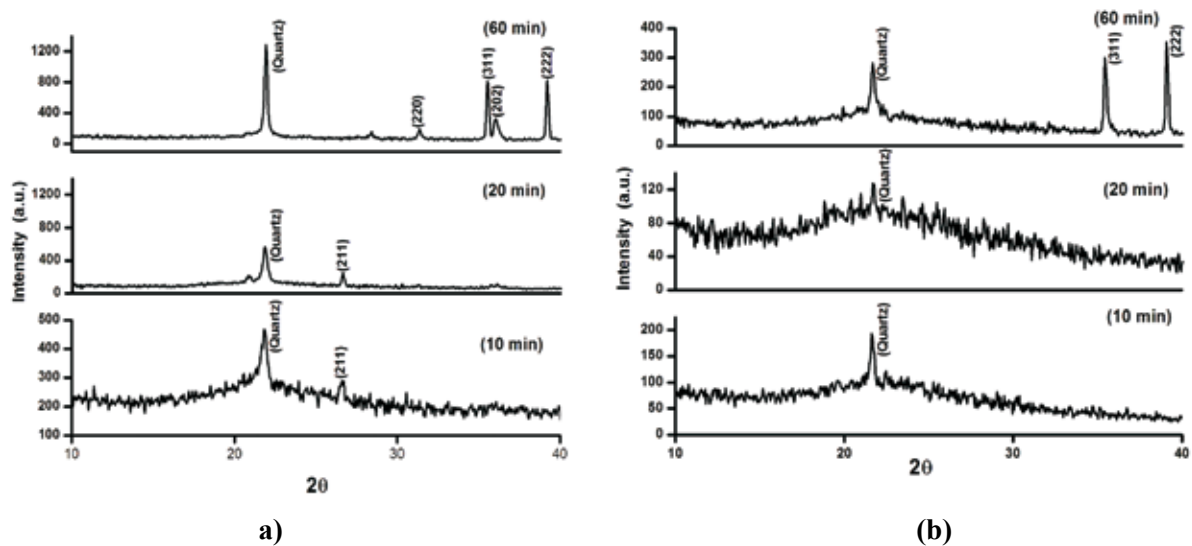


Fig.1. XRD pattern of (a) pristine CuFe₂O₄ and (b) after irradiation

From Fig. 1a, it is observed that the pristine films have the CuFe₂O₄ peaks and the peak at 21.6° is due to Quartz substrate. On irradiation of the 10 min coated and 20 min coated sample, the ferrite peak of (211) were obliterated and shown in fig. 1b. This can be ascribed to the amorphisation of the films on SHI irradiation. In the case of 60 min sample, only the two predominant peaks (311) and (222) are present after SHI irradiation and all the other X-ray peaks were destroyed by the irradiation process. The degree of amorphisation and other studies are in progress.

5.2.48 Swift heavy ion irradiation induced phase transformation of CoFe₂O₄ thin films

B. N. Dash¹, P. Dash², H. Rath², P. Mallick³, R. Biswal², P. Kulariya⁴, D.Kanjilal⁴, D.K.Avasthi⁴ and N.C. Mishra²

¹Department of Physics, S. V. M. College, Jagatsinghpur

²Department of Physics, Utkal University, Bhubaneswar

³Department of Physics, North Orissa University, Baripada

⁴Inter-University Accelerator Centre, New Delhi

Recently Cobalt ferrite (CoFe₂O₄) has attracted much attention as a promising candidate to form magnetoelectric coupling with BaTiO₃ due to its magnetostrictive property [1]. The magnetic character of the particles depends crucially on the size, shape and purity of this

material. Spin coated CoFe_2O_4 thin films on SiO_2/Si substrates followed by thermal annealing at 780°C for 1 hr were irradiated by swift heavy ion (SHI). Initial analysis of both XRD (Fig. 1) and Raman spectra (Fig. 2) indicated that SHI irradiation by 200 MeV Au ions result in the transformation of rhombohedral to cubic phase of CoFe_2O_4 at a fluence of 1×10^{11} ions cm^{-2} and films amorphize at higher fluence. Another interesting observation of the present study corresponds to the improvement of crystallinity in the irradiated films. The film irradiated at a fluence of 1×10^{11} ions cm^{-2} shows better crystallinity with cubic phase compared to the weak peak corresponding to the rhombohedral phase of pristine film as observed from the increased intensity of the XRD peak at this fluence. Raman study also confirm the XRD result and indicate that crystallinity improves at the intermediate fluence as observed from the increased intensity of peaks at 693.66 and 470.631 cm^{-1} , before being amorphized at the highest fluence.

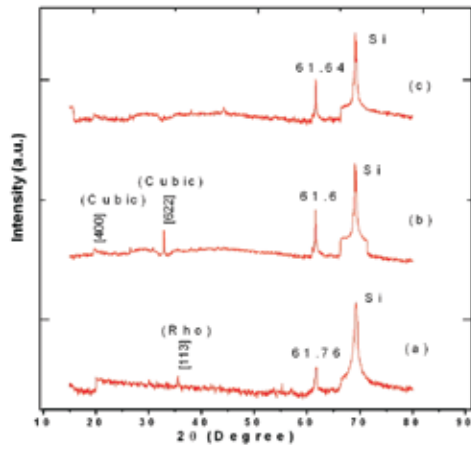


Fig. 1. XRD pattern of CFO films (a) pristine film, (b) films irradiated at 1×10^{11} ions cm^{-2} and (c) films irradiated at 1×10^{12} ions cm^{-2}

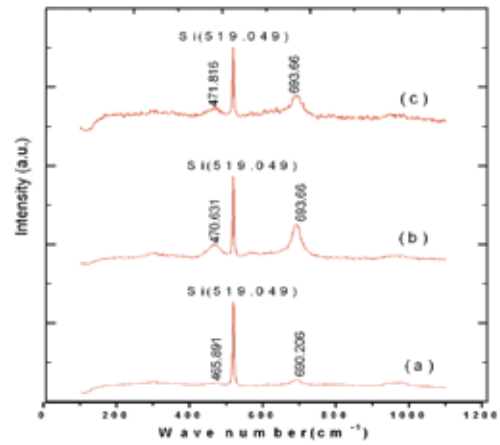


Fig. 2. Raman spectra of CFO films (a) pristine film, (b) films irradiated at 1×10^{11} ions cm^{-2} and (c) films irradiated at 1×10^{12} ions cm^{-2}

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5.2.49 SHI irradiation induced modification of BiFeO_3 thin films prepared by sol-gel method

B.N. Dash¹, P. Dash², H. Rath², P. Mallick³, R. Biswal², P. Kulariya⁴, D.Kanjilal⁴, D. K. Avasthi⁴ and N.C. Mishra²

¹Department of Physics, Salipur College, Salipur

²Department of Physics, Utkal University, Bhubaneswar

³Department of Physics, North Orissa University, Baripada

⁴Inter-University Accelerator Centre, New Delhi

BiFeO₃ (BFO) is a multiferroic material, which has gained importance due its stable ferroelectric and antiferromagnetic coupling at room temperature and due to the enhancement of its ferroelectric response in thin film form [1-3]. Both XRD (Fig.1) and Raman (Fig.2) studies of BFO thin films on ITO/glass substrates prepared by sol-gel method indicated that SHI irradiation by 200 MeV Au ions result in fragmentation of particles and progressive amorphization with increasing irradiation fluence.

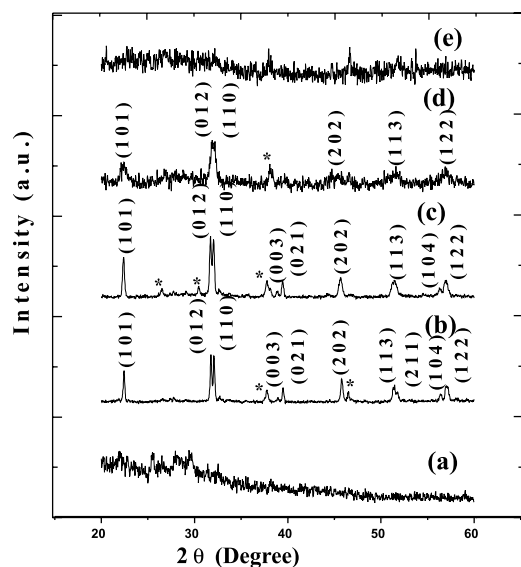


Fig. 1. XRD Pattern of BFO films (a) As-deposited film (b) films annealed at 550°C, (c) Au ion irradiated films at 1×10^{11} ions cm^{-2} , (d) Ag ion irradiated films at 5×10^{11} ions cm^{-2} , and (e) Au ion irradiated films at 1×10^{12} ions cm^{-2} respectively.

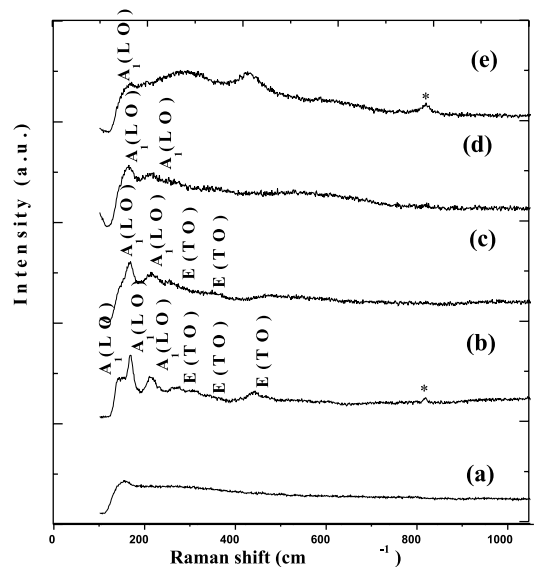


Fig. 2. Raman spectra of BFO films (a) As-deposited film (b) films annealed at 550°C, (c) Au ion irradiated films at 1×10^{11} ions cm^{-2} , (d) Ag ion irradiated films at 5×10^{11} ions cm^{-2} , and (e) Au ion irradiated films at 1×10^{12} ions cm^{-2} respectively.

The average crystallite size estimated from the XRD line width decreased from 38 nm (in pristine sample annealed at 550 °C) to 29 nm on irradiating these films by 200 MeV Au ions at 1×10^{11} ions cm^{-2} . Complete amorphization of the rhombohedral BiFeO₃ phase occurs at a fluence of 1×10^{12} ions cm^{-2} . Irradiation by another ion (200 MeV Ag) had the similar effect. For both the ions, the electronic energy loss exceeds the threshold electronic energy loss for creation of amorphized latent tracks in BiFeO₃. The electrical, magnetic and magnetoelectric characterization of these films is in progress to observe the effect of radial pressure around the latent tracks on these properties.

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5.2.50 Ion beam induced crystallization of alumina coatings deposited by e-beam evaporation

Priyanka Nayar¹, Atul Khanna¹ and D. Kabiraj²

¹Department of Applied Physics, Guru Nanak Dev University, Amritsar

²Inter-University Accelerator Centre, New Delhi

In this research project, we are studying the growth, characterization and effects of ion beam irradiation on the crystallinity, surface morphology, optical and electrical properties of alumina coatings deposited on glass, silicon and quartz substrates by e-beam evaporation technique. Alumina films of thicknesses of about 460 nm were prepared without any deliberate substrate heating. Films were characterized by X-ray diffraction, SEM and UV-visible absorption spectroscopic measurements. Electrical properties of films were measured by two-probe method and their room temperature resistivity was $\sim 10^7$ ohm-cm. Films on quartz substrates were crystalline δ -alumina phase.

The refractive indices of films on glass and quartz substrates were determined to be 1.64 ± 0.01 and 1.66 ± 0.01 respectively from the UV-visible absorption data. The optical absorption edge was 6.0 ± 0.1 eV.

The XRD pattern of film on quartz substrate shows sharp diffraction peaks at 45.7° and 63.7° which can be attributed to tetragonal δ -alumina phase.

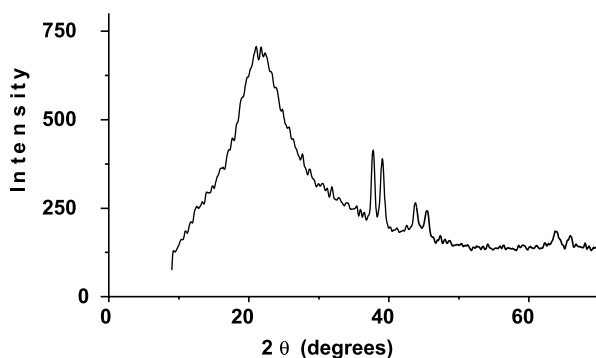


Fig.1. XRD pattern of alumina film

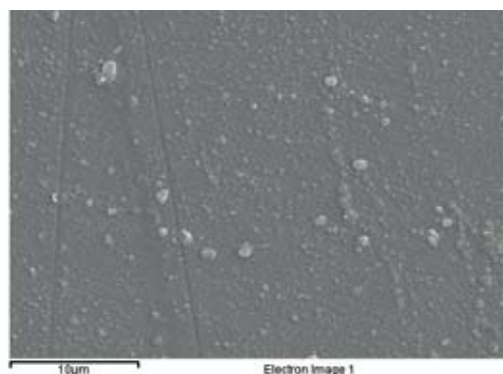


Fig.2. SEM image of alumina film

The SEM studies found that alumina films have a smooth surface morphology. Alumina coatings were irradiated by 84 MeV Ag⁶⁺ and 120 MeV Si⁹⁺ ions up to a maximum fluence of 10¹³ and 10¹⁴ ions cm⁻². The effects of ion irradiation on the structure and properties of coatings are presently being investigated.

5.2.51 Structure and dielectric study of Sr[(Mg_{0.32}Co_{0.02})Nb_{0.66}]O₃ thin film

Bhagwati Bishnoi¹, P.K.Mehta¹ and Ravi Kumar²

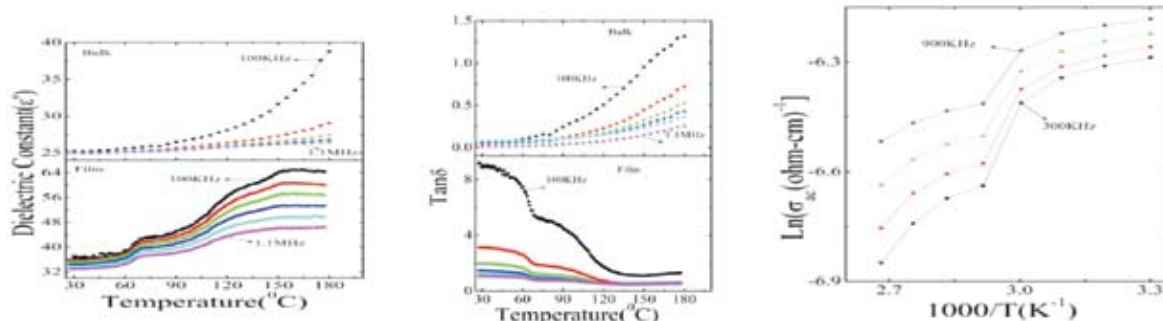
¹Department of physics, The M.S.University of Baroda, Vadodara

²Inter University Accelerator Centre, New Delhi

The progress in the communication applications of bulk has resulted in a demand for miniaturized components, which are a key factor for low power and integratable microwave devices. Ferroelectric materials and specifically ferroelectric/dielectric thin films have attracted a great deal of attention ranging from high charge storage memories, microwave communications, ferroelectric random access memories (FeRAM) to micro-electromechanical systems [MEMS] The integration of ferroelectric thin films onto the semiconductors in the recent years has given a boost to the development of memory as well as switching devices for a wide range of applications [1-2].

Single Phase Sr[(Mg_{0.32}Co_{0.02})Nb_{0.66}]O₃ SMCN, targets were prepared by using standard solid state reaction technique. 250nm-thick SMCN thin films were prepared by Pulsed Laser Deposition (PLD) technique using a pulsed KrF excimer laser (248nm, in wavelength, with a beam fluency of about 220mJ/cm² and a repetition rate 10Hz). The Structural analysis of the thin film is carried out by 3KW X-ray generator with Cu target θ -2 θ Goniometer. Dielectric properties were measured using a Agilent 4285A(LCR) bridge which has a frequency range of 75KHz-30MHz in temperature range 250K to 430K. The Structure and dielectric measurements on SMCN films are perhaps presented for the first time. Single phase monoclinic structure is maintained in SMCN film.

Dielectric measurements show the transition temperature(T_m) shifting toward lower temperature suggesting larger flexibility of dipole movement in films compared to bulk. Observed Dielectric constant ϵ' values in films are higher. Simultaneous increase in loss factor may have extrinsic factors responsible for it. The activation energy, calculated at 300 KHz, in low temp region is found to be 0.02eV, which corresponds to the shallow traps. At temperatures above 70°C it enhances to 0.1eV may correspond to either formation or migration of oxygen vacancies [3]. The observed two step activation energy values are found to be lower then that of our bulk pallets (0.123eV). Observed findings on SMCN samples in thin film form provide promising possibilities as alternative technology in memory devices, switching devices etc. The irradiation of film is carried out at IUAC (New Delhi). The beams used are (Ag) 200MeV and (O₂) 100MeV. The characterizations of irradiated films are under progress.



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5.2.52 Influence of 120 MeV Ag⁹⁺ ion Irradiation on the Structural and Photoelectrochemical Properties of Iron Oxide

Aadesh P. Singh¹, Saroj Kumari¹, Ambuj Tripathi³, P. Kulariya³, Rohit Shrivastav², Sahab Dass², Vibha R. Satsangi¹

¹Dept. of Physics & Comp.Sc., Dayalbagh Educational Institute, Dayalbagh, Agra

²Department of Chemistry, Dayalbagh Educational Institute, Dayalbagh, Agra

³Inter University Accelerator Centre, New Delhi

Metal oxide semiconductor in nanostructured form has great potential in the design of low-cost, environmental friendly solar-hydrogen production through photoelectrochemical (PEC) splitting of water. Presently, the solar-to-hydrogen conversion efficiency through PEC route is too low for the technology to be economically viable. Iron oxide (α -Fe₂O₃) has received considerable attention of researchers for its use in PEC cell for hydrogen production due to its suitable bandgap 2.0-2.2 eV, lying in the visible region of solar spectrum, where the sun emits the maximum energy.

Thin films of iron oxide were deposited on conducting glass substrates by spraying 0.15 M precursor solution, prepared by dissolving Fe(NO₃)₃·9H₂O in double-distilled water [1]. These films were irradiated with 120 MeV Ag⁹⁺ ions at the fluence values from 5×10¹¹ to 1×10¹³ ions/cm² at 1 pA.

X-ray diffraction (XRD) pattern of unirradiated and irradiated thin films sample confirmed the formation of single phase of hematite. From the XRD data, a decrease in average grain size from 28 to 17 nm was observed for samples irradiated up to fluence 5×10¹²

ions/cm², while at higher fluence of 1x10¹³ ions/cm², it was increased to 26 nm. Iron oxide thin films were additionally analyzed by Raman Spectroscopy to see any change in the phase. All peaks of unirradiated samples can be assigned to the α -Fe₂O₃ crystallographic phase. Irradiated samples of α -Fe₂O₃ exhibited an additional peak at 656 cm⁻¹ which suggest the partial transition of phase from α -Fe₂O₃ to Fe₃O₄ [2]. Increasing intensity of the peak at 656 cm⁻¹ with the increase in the fluence, indicates the presence of higher concentration of Fe₃O₄ in the irradiated samples. This phase transition in iron oxide from α -Fe₂O₃ to Fe₃O₄ may be due to the electronic energy loss, which is ~222 times greater as compared to nuclear energy loss.

For the photoelectrochemical measurements, all the unirradiated and irradiated iron oxide thin films were used as photoelectrode in PEC cell with 13 pH NaOH as electrolyte and current-voltage characteristic under darkness as well as under illumination were recorded. Figure 1 presents photocurrent density versus electrode potential curves for all the samples. The effect of irradiation of Ag⁹⁺ ions on iron oxide can be seen in terms of improving PEC response. On increasing the fluence from 5x10¹¹ to 1x10¹³ ions/cm², the photocurrent density was observed to increase continuously. Sample irradiated at fluence 1x10¹³ ions/cm² exhibited the maximum photocurrent density ~321 μ A/cm² at 0.95 V/SCE external bias, which is five times larger than the unirradiated sample.

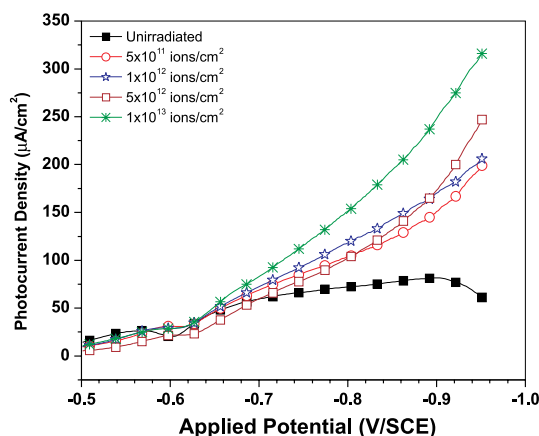


Fig. 1. Photocurrent density–potential characteristics of PEC cell with iron oxide thin films

Donor density as calculated from of Mott-Schottky curves was also observed to increase from 17.25x10¹⁹ to 71.41x10¹⁹ cm⁻³ on increasing the fluence. Resistivity for all the unirradiated and irradiated samples was also calculated from slope of the current-voltage characteristics under dark condition. The observed resistivity decreased continuously from 6.6x10⁵ Ω-cm for unirradiated to 4.3x10⁵ Ω-cm for sample irradiated at 1x10¹³ ions/cm². This increase in donor density and decrease in resistivity can be associated with the partial transformation of α -Fe₂O₃ to Fe₃O₄ phase as observed in Raman spectra. Regular decrease in resistivity and increase in donor density suggests that increasing fluence of irradiation increased the concentration of Fe₃O₄ in the samples of hematite, as Fe₃O₄ is highly conductive,

with conduction occurring via electron transfer from Fe^{2+} to Fe^{3+} sites [3]. A small inclusion of the Fe_3O_4 in $\alpha\text{-Fe}_2\text{O}_3$ is known to have large effect on the electrical properties [3-4].

Therefore, in conclusion, this study suggests that; (i) irradiation of 120 MeV Ag^{9+} ion on the $\alpha\text{-Fe}_2\text{O}_3$ is effective in improving the PEC performance of material (ii) the effect is associated with controlled phase transformation of $\alpha\text{-Fe}_2\text{O}_3$ to Fe_3O_4 and (iii) thin films irradiated at fluence 1×10^{13} ions/cm² showed maximum photocurrent density.

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5.2.53 The effect of 100 MeV Fluorine ion irradiation on interface trapped charge and oxide trapped charge of MOS devices

N. Pushpa¹, K. C. Praveen¹, A. P. Gnana Prakash¹, Ambuj Tripathi², Y. P. Prabhakara Rao³ and D. Revannasiddaiah¹

¹Dept. of Studies in Physics, University of Mysore, Manasagangotri, Mysore

²Inter University Accelerator Centre, New Delhi

³Bharath Electronics Limited (BEL), Jalahalli, Bangalore

The N-channel metal oxide semiconductor field effect transistors (MOSFETs) were exposed to 100 MeV F^{8+} ions in a dose ranging from 100 krad to 100 Mrad. The major radiation effects in MOS devices occur due to charge generation, transport and trapping in the SiO_2 . The degradation of MOS devices is mainly due the radiation induced interface (N_{it}) and oxide trapped charges (N_{ot}) at Si/ SiO_2 interface and eventually make the device malfunction. The subthreshold characteristics of 100 MeV F^{8+} ions irradiated MOSFET is shown in Figure 1. We observed that the threshold voltage (V_{TH}) of the irradiated MOSFET was found to decrease significantly after ion irradiation. The interface (N_{it}) and oxide trapped charge (N_{ot}) were estimated from the subthreshold measurements and were found to increase after irradiation (Figure 2). The densities of oxide-trapped (ΔN_{ot}) charge in irradiated MOSFETs were found to be higher than those of the interface trapped charge (ΔN_{it}). We compared the ion irradiated MOSFET results with Co-60 gamma irradiated devices. The net threshold voltage shift (ΔV_{th}) and contribution to that shift due to the interface traps (ΔV_{Nit}) and the trapped oxide charge (ΔV_{Not}) was calculated by the subthreshold measurements using

the technique proposed by McWhorter and Winokur [1]. The ΔN_{ot} is calculated using the standard expression $\Delta N_{ot} = \Delta V_{ot} Cox/q$ and the ΔN_{it} is calculated by the expression $\Delta N_{it} = \Delta V_{it} Cox/q$ where $q = 1.6 \times 10^{-19} C$. From the Figure 2 it can be seen that ΔN_{it} and ΔN_{ot} for Co-60 gamma irradiated MOSFETs increased about an order of magnitude while 100 MeV F^{8+} ions irradiated devices increased about two to three order of magnitude after a total dose of 100 Mrad.

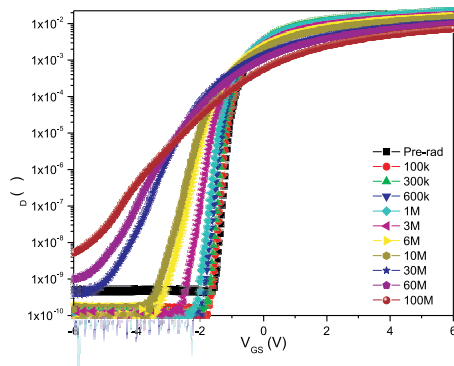


Fig. 1. I_D - V_{GS} characteristics of 100 MeV Fluorine ion irradiated N-channel MOSFET before and after irradiation (at $V_{DS} = 1$ V).

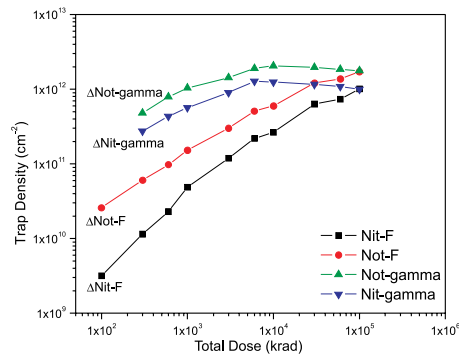


Fig. 2. Variation in ΔN_{it} and ΔN_{ot} after 100 MeV Fluorine ions and Co-60 gamma irradiation.

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5.2.54 The effect of 50 MeV Li^{3+} ion irradiation on generation-recombination centers in SiO_2

N. Pushpa¹, K. C. Praveen¹, A. P. Gnana Prakash¹, Ambuj Tripathi², Y. P. Prabhakara Rao³ and D. Revannasiddaiah¹

¹Dept. of Studies in Physics, University of Mysore, Manasagangotri, Mysore

²Inter University Accelerator Centre, New Delhi

³Bharath Electronics Limited (BEL), Jalahalli, Bangalore

In order to qualify bipolar devices and circuits for space and LHC applications, these devices and circuits need to be radiation hardened from 10's of krad to 100's of Mrad of total dose [1, 2]. When bipolar transistors are exposed to ionizing radiation, trapped oxide charge and interface states accumulate in the field oxide (SiO_2) that lies over the surface of the intrinsic base. This leads to an increase in surface recombination current in the emitter-base diode. Consequently, there is an increase in the base current of the device and the bipolar transistor suffers from a loss of dc current gain [3]. The NPN Si *RF* transistors were exposed to 50 MeV Li^{3+} ions in a dose ranging from 100 krad to 100 Mrad. The Gummel

characteristics, excess base current ($\Delta I_B = I_{Bpost} - I_{Bpre}$), dc current gain (h_{FE}) and collector-saturation current (I_{CSat}) of the Li ion irradiated transistors were studied before and after irradiation. Figure 1 illustrates the Gummel characteristics of Li^{3+} ion irradiated transistors. The base current (I_B) was found to increase significantly after ion irradiation as expected. The variation in current gain after 50 MeV Li^{3+} ions irradiation is shown in Figure 2 and it can be seen that the peak current gain changed from 120 to 10 after a total dose of 100 Mrad. The generation-recombination (G-R) centers in emitter-base spacer oxide and the bulk damage in the collector region of the transistors were responsible for the device degradation after 50 MeV Li^{3+} ions irradiation.

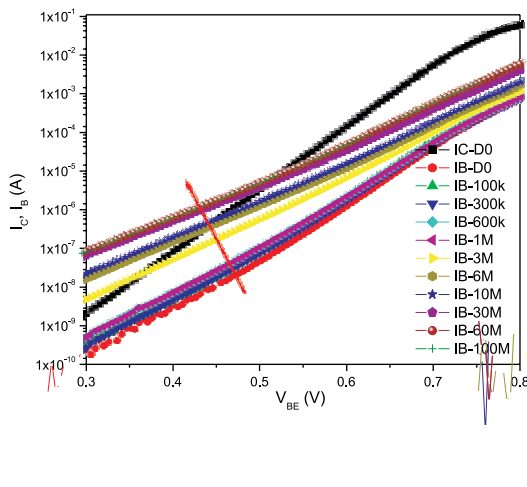


Fig. 1. Gummel characteristics of 50 MeV Li^{3+} ion irradiated transistor.

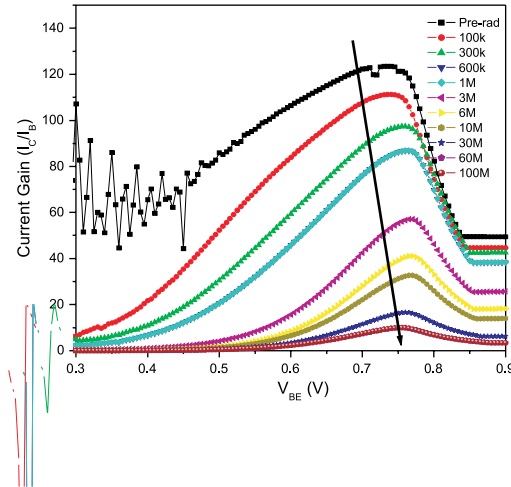


Fig. 2. The variation in DC current gain after 50 MeV Li^{3+} ion irradiation

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5.2.55 Effects of 50MeV Li^{3+} ion irradiation on 200 GHz SiGe Heterojunction Bipolar Transistors

K.C.Praveen¹, N.Pushpa¹, Y.P. Prabhakara Rao², Ambuj Tripathi³, Somya Gupta⁴, Navakanta Bhat⁴, John D Cressler⁵ and A. P. Gnana Prakash¹

¹Dept. of Studies in Physics, University of Mysore, Manasagangotri, Mysore

² Bharath Electronics Limited (BEL), Jalahalli Post, Bangalore

³ Inter-University Accelerator Center, New Delhi

⁴ Electrical Communication Engineering, Indian Institute of Science, Bangalore

⁵School of Electrical and Computer Engineering, Georgia Inst. Tech. Atlanta, GA 30332-USA

The 200 GHz SiGe HBTs were irradiated with 50 MeV Li^{3+} ions with the total doses of 300 krad and 600 krad. The various I-V characteristics of SiGe HBTs were studied before and after ion irradiation using computer interfaced Agilent 4155 Semiconductor Parameter Analyzer. Figure 1 illustrates the forward mode Gummel characteristics of Li^{3+} ion irradiated SiGe HBTs. The forward mode base current (I_B) of the ion irradiated SiGe HBTs increases slightly at low injection, as expected with increase in dose. The collector current remains unchanged after ion irradiation. The degradation in I_B is the result of increased generation of generation-recombination (G/R) traps in the emitter-base (EB) spacer oxide. These radiation induced G/R traps increase recombination current in EB diode, thus increases the I_B . The inverse mode Gummel characteristics of SiGe HBTs irradiated with 50 MeV Li^{3+} ions is shown in Figure 2. The existence of the radiation induced traps in Collector-Base (CB) space charge region was verified using the inverse-mode Gummel characteristics of the devices, in which the collector and the emitter terminals of the transistor are swapped.

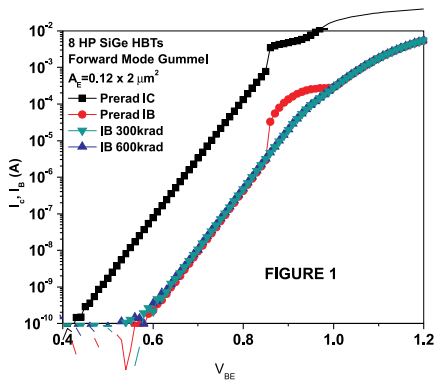


Fig.1. Forward mode Gummel characteristics

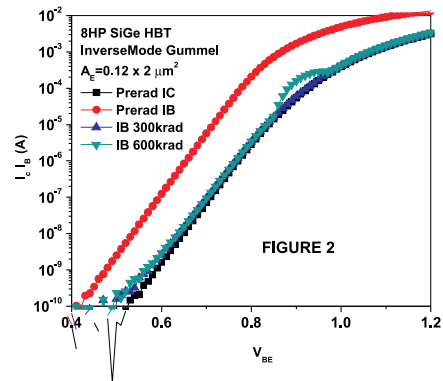


Fig. 3. Forward mode current gain

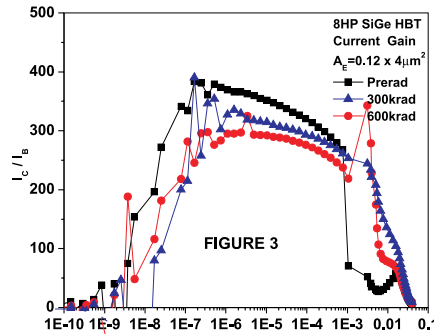


Fig.2. Inverse mode Gummel characteristics

In this case, the radiation induced traps in the CB junction now act as G/R centers in the inverse EB junction and can be easily identified for 600 krad of total dose. Figure 3 shows the degradation in forward mode current gain (β) with Li^{3+} ion doses. It is clear from the figure that β decreases for both the doses. Since I_B increases after irradiation and I_C remains constant, β decreases with increase in ion dose. We also studied neutral base recombination (NBR) and avalanche multiplication (M-1) of carriers in CB junction after Li^{3+} ion irradiation.

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5.2.56 Li Ion Induced Effects on Electrical and Defect Characteristics of npn Bipolar Power Transistors

C.M Dinesh¹, Ramani¹, M. C. Radhakrishna¹, S.A. Khan² and D. Kanjilal²

¹Department of Physics, Jnanabharati, Bangalore University, Bangalore

²Inter-University Accelerator Center, New Delhi

The effect of 50 MeV Li^{3+} ion irradiation on the electrical behavior of commercial BJT's is studied in this work. An attempt is made to account for the study of radiation induced gain degradation, capacitance, doping density and carrier removal rate. The Li-ion induced defect properties such as activation energy, trap concentration and capture cross section of deep levels are studied by C-V and DLTS measurements.

The transistors (2N3055 & 2N6688) are irradiated by 50 MeV Li^{3+} ion beam using 15 UD, 16 MV Tandem accelerator facility, available at Inter University Accelerator Centre (IUAC), New Delhi, India. The irradiation is performed in material science beam line having a typical pressure of 8×10^{-9} torr. The fluence is varied from 5×10^9 to 1×10^{13} ions cm^{-2} . $I_{CE}-V_{CE}$ characteristics are performed using Keithley 2400 source meters together with computer interface before and after the irradiation. Capacitance and DLTS spectra are studied for the transistor before and after exposure. The DLTS system (IMS-2000, M/s. Lab Equip, India) employed for the present study consists of a boxcar averager, a pulse generator, a thousand point digitizer, a voltage generator and a high speed capacitance meter.

Figure 1 (a) shows the variation of gain as a function of base-emitter voltage (from forward Gummel plots) before and after exposure to various fluences of 50 MeV Li^{3+} ions. The atomic displacements and vacancies produced upon irradiation in the bulk of the transistor reduce the minority carrier lifetime. A decrease in the minority carrier lifetime is reflected in

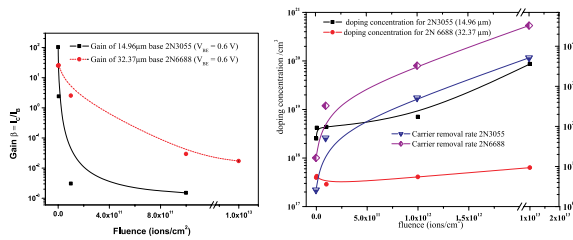


Figure 1. (a) Variation forward current gain of the transistors as a function of Li-ion fluences. (b) Variation of doping concentration and carrier removal rate with fluence. $ND = 2.52 \times 10^{18} \text{ m}^{-3}$ and $3.95 \times 10^{17} \text{ m}^{-3}$ for 2N 3055 and 2N 6688 respectively

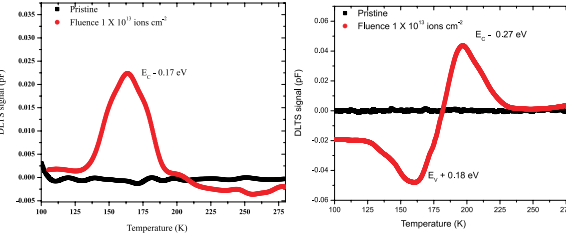


Figure 2. (a) DLTS spectra 50 MeV Li³⁺ ion irradiated 2N 3055 transistor for a fluence of $1 \times 10^{13} \text{ ions/cm}^2$. (b) DLTS spectra of 50 MeV Li³⁺ ion irradiated 2N 6688 transistor for a fluence of $1 \times 10^{13} \text{ ions/cm}^2$

the degradation of forward current gain of the transistor. The variation of current gain as a function of fluence is shown in figure 1 (a) [1-2].

The variation in the doping concentration for two types of transistors is shown in figure 1 (b). The doping concentration of larger base width transistor (2N 6688, 32.37 μm) does not change much with increasing fluence compared to smaller base width transistor (2N 3055, 14.96 μm). The doping concentration of 2N3055 transistor increases by about 100 times for the maximum ion fluence. This increase in doping concentration plays an important role in conduction of the device. From the C-V measurements, carrier removal rate (CRR) can be calculated from the changes in the space charge of the barriers. In figure, the variation of carrier removal rate with fluence is plotted for both types of the transistors. The carrier removal rate appears to vary almost linearly with ion fluence [3].

The deep level defects generated by irradiation of transistors by Li³⁺ ions are characterized using the DLTS technique. The DLTS spectrum is a plot of difference in capacitance (δC) versus temperature. Figure 2. (a) Exhibits the DLTS spectra of Li³⁺ ion-irradiated transistor (2N 3055) for a fluence of $1 \times 10^{13} \text{ ions/cm}^2$. The DLTS spectra for the other transistor 2N 6688 is shown in Figure 2. (b).

In the case of transistor 2N 3055, only one minority carrier deep level defect is observed in the DLTS spectra for the fluence $1 \times 10^{13} \text{ cm}^{-3}$. On the other hand, for the other transistor 2N 6688, one minority carrier and one majority carrier deep level defects are observed in the DLTS spectra.

Exposure of the transistors to Li-ions and subsequent characterization by I-V, C-V and DLTS measurements reveal that the defects and recombination centers generated in the emitter-base region as well as in the neutral base region are largely responsible for the

reduction in the collector current and increase in base current. The increase in the base current through multi-phonon recombination leads to degradation of the forward current gain of the transistors. In the case of smaller base width transistor (2N 3055) only one deep level defect is observed upon irradiation which is assigned to A-centre. On the other hand two defect levels (Di-vacancy and B-O complex) are observed for the larger base width transistor (2N 3688).

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