In this work, the effects of swift heavy ion (SHI) irradiation on the structural, morphological, optical and electrical properties of pristine and Ni-doped $BiFeO_3$ (BFO) thin films have been investigated. The targets of pristine and Ni-doped BFO with compositions $BiFe_{1-x}Ni_xO_3$ (x = 0, 0.01, 0.03 and 0.05) have been synthesized via the sol-gel method [*for details, see Mater. Res. Express 5 (2018) 065506*]. Also, the influence of Ni-doping at Fe site of $BiFeO_3$ on the structural, dielectric and multiferroic properties was investigated [*for details, see J. Appl. Phys. 124 (2018) 164105*]. The thin films with composition of $BiFe_{1-x}Ni_xO_3$ (x = 0, 0.01, 0.03 and 0.05) were deposited on the n-type single crystal Si(100) substrates through pulsed laser deposition (PLD) technique. Further, irradiation on the thin films was performed using 15 UD Pelletron tandem accelerator at Inter University Accelerator Centre (IUAC), New Delhi with 120 MeV Ag^{9+} ions at different ion fluences, i.e., 1×10^{13} ions/cm^2, 5×10^{13} ions/cm^2 and 1×10^{14} ions/cm^2 with current of 1pnA (particle nanoampere). All the films were fixed to the target ladder placed inside the high vacuum (~10^{-6} torr) chamber during irradiation. Irradiation was performed in the direction perpendicular to the sample surface. The ion beam was focused to a spot of 10 mm diameter and then scanned over an area of 1×1 cm^2 using magnetic scanner to cover the complete sample surface for uniform irradiation. In order to investigate the effects of SHI irradiation on the aforementioned properties, all the films have been characterized by various analytical techniques, such as X-ray diffraction (XRD), Raman spectroscopy, atomic force microscopy (AFM), scanning electron microscopy (SEM), energy dispersive X-ray spectroscopy (EDS), UV-visible diffuse reflectance spectroscopy (DRS) and two-probe resistivity setup.

The effect of the irradiation on the structure and phase of the thin films have been examined by the XRD and Raman data analyses that indicate the polycrystalline growth of the films and partial amorphization after irradiation. Raman spectra of unirradiated thin films of $BiFe_{1-x}Ni_xO_3$ (x = 0, 0.01, 0.03 and 0.05) display quite different characteristics as compared to all the irradiated thin films at all the fluences and most of the Raman modes of the irradiated thin films disappear that indicate the partial amorphized nature of the thin films. SEM micrographs exhibit that the grains are not uniformly distributed and it has been observed that when the ion fluence rise, the grain size rises. The EDS spectra exhibit peaks corresponding to all the required elements in the sample, i.e., Bi, Fe and O, that confirm the purity of $BiFeO_3$ thin film. The AFM images of the thin films display uniform distribution of spherical grains throughout the surface. For the pristine $BiFeO_3$ thin film, the average grain size and rms roughness is found to be 43.52 nm and 5.88 nm, respectively. The obtained value of E_g is found to be 2.29 eV for pristine $BiFeO_3$ thin film and decreases for the irradiated thin films as ion fluence increases that possibly be because of the occurrence of defect induced energy levels near to the conduction band. All the thin films display rectifying I-V behavior with hysteresis, which is suppressed for SHI irradiated thin films as the fluence increases. Hence, it is concluded that by selecting appropriate amount of doping, fluence and energy during SHI irradiation, it is possible to alter the characteristics of $BiFeO_3$ films such as I-V and energy band gap, which could be highly useful for potential device applications.

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5.2.43 Nanostructures from ion irradiation of metal films: aluminium and indium on silicon

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Earlier reports from the literature and our group have demonstrated that ion irradiation induces similar morphological changes on metal films in a widely varying range of ion energies from keV to the MeV, based on variations in the total fluence [1,3]. We have undertaken a systematic series of experiments to investigate the self organised morphological and structural changes of a range of metal films grown on single crystal silicon substrates using ions in the keV and SHI in the MeV energy ranges. Nanostructure formation is driven by energy minimization [4] and is governed by the initial film thickness, substrate material, and ion fluence. The metals we have chosen are Al, In, for which SRIM calculations have indicated large variations in energy transfer in the films.

In the present study, Aluminium and Indium thin films were deposited on silicon (100) substrates via thermal evaporation technique in the Target Lab of IUAC. Thereafter, Al thin films were subjected to irradiation with 100 keV Ar^+ and 100 MeV Si^{7+} and 100 MeV O^{7+} beams with different ion fluences, using the LEIBF and the Pelletron facility at IUAC. XRD analysis shows the enhanced crystallinity for SHI irradiated samples, while for low energy ion crystallinity of the films decreases with increased ion fluence. SEM analysis revealed the formation of circular craters on the film surface with varying diameters, from a few hundred nm to 1 micron, while in the case of high energy ions, changes in surface morphology take place, resulting in the narrowing of size distribution of nanostructures. Indium films were also irradiated by 100 MeV Si^{7+} and 100 MeV O^{7+} ion beams. From the XRD

analysis, it was found that the films irradiated by O^{7+} ion beams showed reactivity. For 100 keV Ar^{+} indium films analysis is underway. RBS analysis using the IUAC facility indicates that ion beam mixing doesn't occur in this system and sputtering is see in at highest fluences for Ar irradiated films, but, as expected, is not observed for the SHI irradiated films. The use of Target Lab, Pelletron and LEIBF beam time and analysis facilities (AFM, SEM, RBS) of IUAC are gratefully acknowledged.

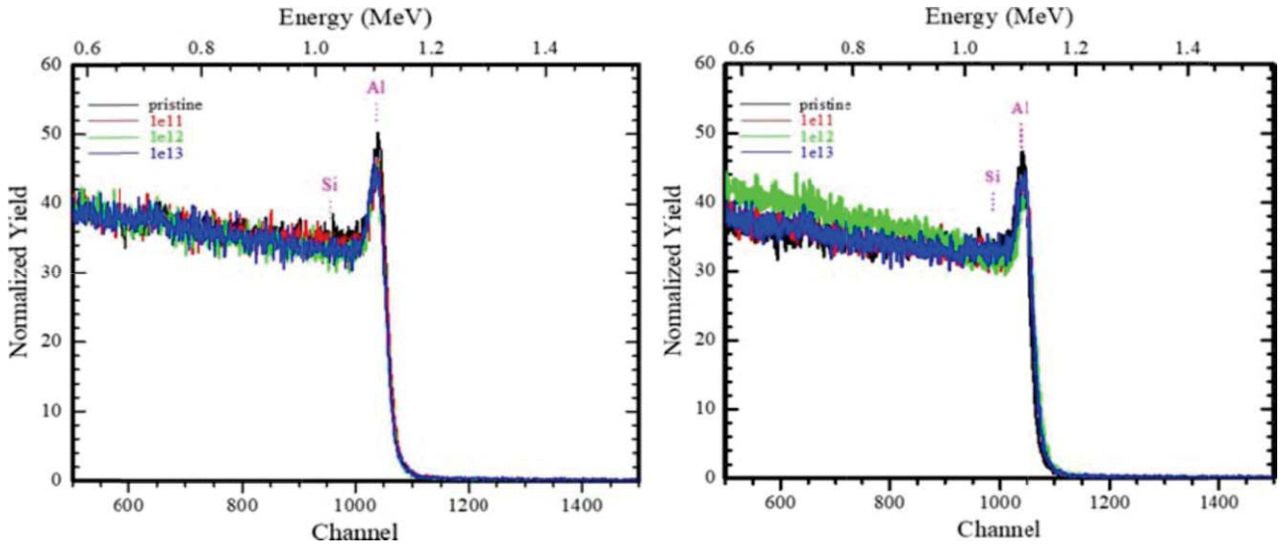

Figure 1. RBS spectra of 25 nm Aluminium films irradiated with a) 100 MeV O^{7+} ions and b) 100 MeV Si^{7+} ions.

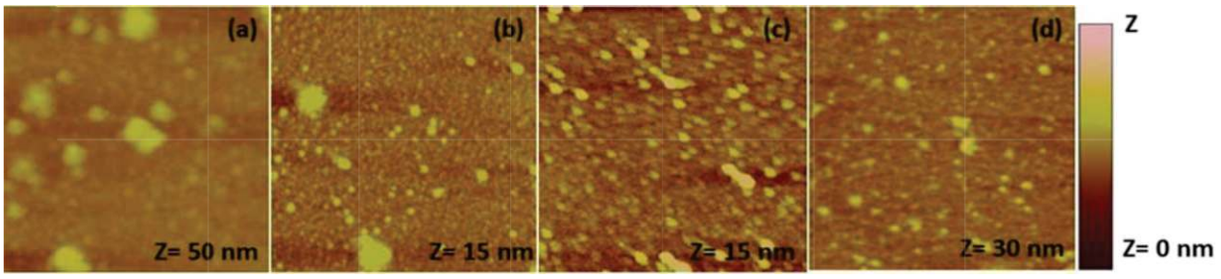

Figure 2. AFM images of 25 nm Aluminium films a) pristine film and 100 MeV Si^{7+}irradiated films at fluence of b) 1x 10^{11}, c)1x 10^{12} and d) 1x10^{13} ions/cm^{-2}.

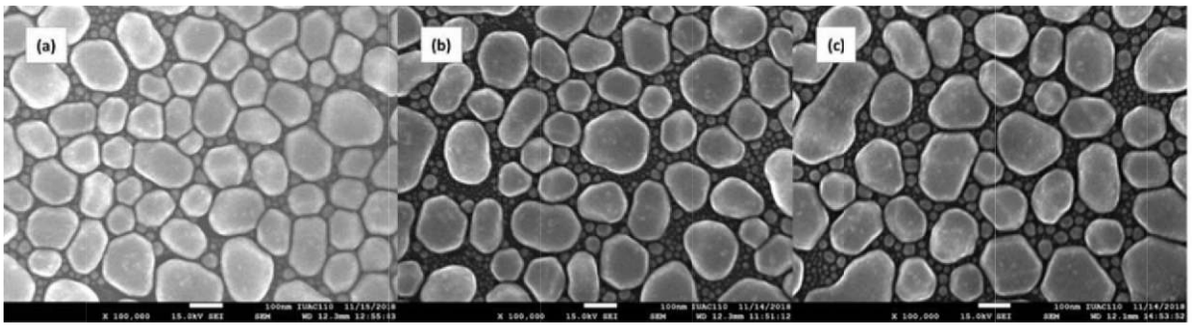

Figure 3: SEM micrographs of 25 nm Indium a) unirradiated film and 100k times magnified 100 MeV O^{7+} irradiated films with fluence b) 1x 10^{11} and c) 1x 10^{13} ions/cm^{-2}.

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5.2.44 Localized thermal spike driven morphology and electronic structure transformation in swift heavy ion irradiated TiO_2 nanorods

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Titanium dioxide (TiO_2) remains one of the most prominent members of the metal oxide family owing to its high chemical and thermal stability, excellent photoactivity, biocompatibility and availability in the form of three different polymorphs (anatase, brookite and rutile).1 Particularly, highly aligned and vertically oriented TiO_2 nanorods hydrothermally grown on fluorine doped tin oxide (FTO) coated glass substrate have shown strong potential in applications such as electrode material with transparent conducting window for solar cells,2 water splitting,3 bio-photoelectrochemical reactions,4 UV photodetectors,5 *etc.* In addition, the single-crystalline nature of each nanorod provides a long-range periodicity throughout the individual nanorod structure, which is a suitable platform for the study of irradiation effects. Moreover, a small lateral dimension (few tens of nanometer) of the nanorods provides limited space for thermal energy transfer and atomic displacement in the radial direction. Therefore, it is interesting to explore the effect of swift heavy ion (SHI) irradiation-induced localized energy transfer in the rutile TiO_2 nanorods.

We have investigated the effect of 100 MeV Ag ion beam irradiation on the vertically oriented TiO_2 nanorods using the 15UD accelerator at Inter-University Accelerator Centre, New Delhi, India. The fluences used for irradiation were 1×10^{12}, 5×10^{12}, 1×10^{13} and 5×10^{13} ions/cm^2. The influence of SHI irradiation on the morphology, structural, electronic and optical properties was investigated via various microscopic and spectroscopic characterization techniques such as FESEM, High-resolution TEM, XRD, Raman spectroscopy, XPS, UV-VIS and photoluminescence measurements. The studies show that the nanorods undergo a significant morphological and structural transformation through bending and melting induced by a localized thermal spike, particularly at the highest fluence (5×10^{13} ions/cm^2). HRTEM analysis shows tracks with a lateral dimension of ~10 nm along the ion trajectory. Thermal spike simulation confirms the melting in TiO_2 nanorods within a cylindrical region having a radius of ~5.5 nm, supporting the TEM observation. Re-solidification during the rapid quenching of the SHI-induced thermal spike within a time interval of ~0.5 picoseconds is responsible for the deformation of the crystalline structure, as also observed in the XRD and Raman data. XPS analysis reveals the presence of ion beam-induced vacancies in the lattice, and the resulting strain is manifested by the shift in XRD and Raman peaks. An interesting feature of the irradiation is the transition from a direct to an indirect bandgap at the highest fluence (5×10^{13} ions/cm^2), which is also supported by the electronic band structure calculation using DFT. The results are published in Nanoscale Advances.6 Further, gas sensing experiments conducted with 1% H_2 gas show a higher sensing response for the nanorods irradiated at 5×10^{12} ions/cm^2 compared to the pristine sample. More interestingly, an *n* to *p*-type transition in conductivity is observed for the sample irradiated at 5×10^{13} ions/cm^2. For in detailed understanding, further measurements and analysis are under process.

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5.2.45 Interface modification of Fe/Cr/Al magnetic multilayer by Swift Heavy Ion Irradiation

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Among the wide range of intermetallic compounds, a combination of superior magnetic and mechanical properties makes iron-aluminides [Fe-Al] fascinating for various applications [1]. The physical and mechanical properties of Fe-Al systems can further be improved by adding Cr in Fe-Al intermetallic compounds [2]. The inclusion of anti-ferromagnetic Cr, even in small quantities in Fe-Al alloy, is capable of significantly modifying the magnetic properties. Thus, the constituents of Fe-Cr-Al alloys make it an interesting system to study various types of magnetic interactions since individual components exhibiting ferromagnetism and anti-ferromagnetism are present together. In such an intermetallic compound of metal alloys in the form of thin films, intermixed interface changes its structure and consequently magnetic properties. Thus, it is pivotal to understand a variety of interesting magnetic phenomena at interfaces such as improved spin and orbital magnetic moments or enhancement of the PMA.

In the present work, $[Fe/Cr/Al]_{x10}$ multilayer having different thicknesses $[Fe(2.5\ nm)/Cr(1.5\ nm)/Al(1.5\ nm)]_{x10}$ and $[Fe(1.2\ nm)/Cr(1.0\ nm)/Al(1.0\ nm)]_{x10}$ with different thickness, designated as ML1 and ML2, respectively, were deposited using electron beam evaporation on Si(111) substrate in an UHV environment with the base pressure $\sim 3\times10^{-9}$ Torr. The ML1 and ML2 multilayer samples (10mm × 10mm dimension) were irradiated with 120 MeV Ag^{9+} ions at room temperature (RT) at 15UD Pelletron Accelerator available at Inter University Accelerator Centre (IUAC), New Delhi. The SHIs induced modifications in structural and magnetic properties were examined with X-ray reflectivity (XRR), magneto-optic Kerr effect (MOKE), and X-ray absorption fine structure (XAFS) to reveal interface diffusivity, including variation in magnetic coercivity, in-plane anisotropy, and local structure. The height of 1st order Bragg peak of XRR is used to determine the diffusion length as a function of ion fluence, and it is evident that the lower thickness of $[Fe/Cr/Al]_{x10}$ multilayer possesses higher interface mixing as compared to samples with higher thickness. MOKE results confirm that both multilayers are soft magnetic in nature and show different behavior of in-plane anisotropy. With the SHIs irradiation, coercivity exhibits opposite trends for different thicknesses of Fe/Cr/Al multilayers due to different interface mixing. Fe K-edge XAFS is used for quantitative analysis of the Fe-Cr and Fe- Al phases as a function of multilayer thickness and ion fluence. The inelastic thermal spike (i-TS) model with size effect is used to explain the observed thickness-dependent intermixing due to the SHIs irradiation. The lattice temperature evaluation calculated by i-TS model shows longer spike duration in thinner film resulting in higher mixing [3].

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5.2.46 Effect of Ni and Co co-implantation on the structural, optical, and magnetic properties of ZnO

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To study the effect of co-doping of ions (Ni and Co) through direct implantation on the structural, optical, and magnetic properties of ZnO, thin films of pure ZnO have been deposited through sputtering on silicon wafer. A well-characterized ZnO thin films are implanted with Co and Ni subsequently using low energy negative ion implanter facility at Inter University Acceleration centre (IUAC), New Delhi, India. Four thin films are implanted with Ni at a fluence of 5.48×10^{16} ions/cm^2 and three of the Ni implanted films are subsequently implanted win Co in the fluence range of 1.6×10^{16} ions/cm^2 to 5.48×10^{16} ions/cm^2 and a single ZnO thin film is implanted with 5.48×10^{16} ions/cm^2. Four other films of ZnO are co-implanted with Ni and Co subsequently in the fluence range of 9.1×10^{15} ions/cm^2 to 3.65×10^{16} ions/cm^2. Further single porous ZnO film is implanted subsequently with a fluence of 9.1×10^{15}ions/cm^2, Ni and Co ions. The beam current is maintained at 1mA. To study the effect of ion implantation on the optical properties of the ZnO, reflectance measurement of the ions implanted thin films is done at room temperature at Rajiv Gandhi University, Arunachal Pradesh. To further investigate the effect of co-doping via ion implantation on the structural and magnetic properties, the characterization process is underway.

5.2.47 120 keV Ar ion beam irradiation of magnetron sputtered ZnO thin films

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The effect of low energy ion irradiation with Ar beam having 120 keV energy has been investigated on transparent ZnO thin films to study the modifications in structural and optical properties of ZnO. Here the synthesis of ZnO thin films has been done on quartz substrate by sputtering of the target of pure ZnO using the RF magnetron technique. Surface morphological properties have been observed by FESEM (Field Emission Scanning Electron Microscope) techniques and reveal the variation in the growth of surface grains. To observe the impact of ion irradiation on different characteristics of ZnO thin film various characterizations such as RBS (Rutherford Backscattering Spectrometry), Ultraviolet-visible spectroscopy, Raman Spectroscopy, XRD (X-Ray Diffraction) have also be performed. The XRD data shows that the pristine and irradiated samples show hexagonal wurtzite structure with significant intensities at (002) and (103) orientations. The absorption spectra obtained by UV-visible spectrophotometer reveals that with the increase in ion doses of Argon beam a slight shifting of peaks are observed.

5.2.48 Ion Induced Modification of Ferroelectric Polymer Composites for EMI Shielding

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In order to modify the EMI-SE and dielectric properties, we have synthesized pristine Polyvinylidene Fluoride (PVDF) and PVDF-Titanium dioxide (PVDF-TiO_2) nanocomposite thin films. The TiO_2 nanoparticles were synthesized by the Combustion method using Urea as a fuel. The PVDF and PVDF-TiO_2 nanocomposite thin films of different stoichiometric ratios were prepared using the solvent evaporation method [1]. The PVDF film, TiO_2 nanoparticles and PVDF nanocomposite thin films were characterized by XRD and FTIR spectroscopy. The XRD pattern of TiO_2 nanoparticles shows an anatase phase with crystalline particle of size 59.13nm as estimated using Debye- Scherer equation. The XRD graph of pure PVDF and PVDF nanocomposites shows the presence of phase and the crystallinity of the nanocomposites is enhanced due to the formation of cationic complexes with the functional group of PVDF, which confirms the intercalation of nTiO_2 with the PVDF polymer system. At room temperature, the FTIR spectra of thin films in the wavenumber range 4000-400cm^{-1} are obtained. The FTIR spectra reveal the unequal and equal stretching vibrations of the CH_2 group in the PVDF matrix and the continuous band absorption region between 1450 and 1000 cm^{-1} corresponds to fluorocarbon absorption (C–F). The full set of characteristic vibrational bands (480, 510, 840, 882, 1071, 1401 cm^{-1}) indicates that the PVDF in the polymer matrix is in the β-form [2].

The EMI SE of PVDF-nTiO_2 nanocomposite films is dependent on the frequency, mainly due to the absorption. On incorporation of 2 wt% nTiO_2 in the PVDF matrix, the EMI shielding value increases to a maximum of 35.31dB at a frequency of 4.58 GHz. The increase in wt% of the nanoparticles to the PVDF matrix has varied the EMI shielding of nanocomposite thin films. The EMI-SEwith frequency graph (Fig.1) shows up and down valleys in S-Band (2-4GHz) with an average variation of ~28% and ~35% around 2GHz and 4GHz, respectively and stable in C-Band (4-8GHz). Further, EMI-SE decreases with an increase in wt% of nTiO_2 fillers in the PVDF matrix. The dielectric constant (ε′) and dielectric loss (ε″) variations were studied in the frequency ranging from 10 Hz to 8 MHz at room temperature (30ºC) for pristine PVDF, and PVDF-nTiO2 with 2, 4, 6, 8 and 10 wt% nanocomposite thin films, respectively. The dielectric constant of PVDF-TiO_2 nanocomposite thin films decreases with an increase in the frequency (below VHF) and shows an anomaly at 10 wt% of nTiO_2 fillers in PVDF matrix (Fig.2). This may be due to addition of filler, which increases the density of the dipoles restricting the orientation polarization. The dielectric loss decreases in the low-frequency region and in high-frequency region, it follows a downward falling (Fig.3). The influence of restriction of orientation polarization at low frequencies causes the fall in dielectric loss values.

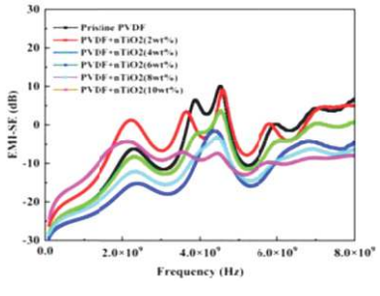

Fig.1: Variation of EMI-SE with Frequency for PVDF and nTiO_2

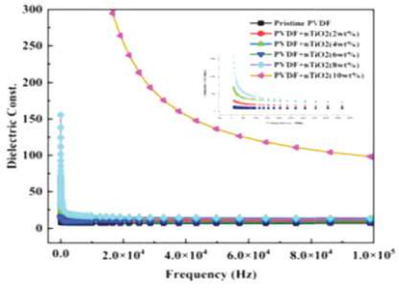

Fig.2: Dielectric Constant Variation with Frequency for PVDF and nTiO_2 composites.

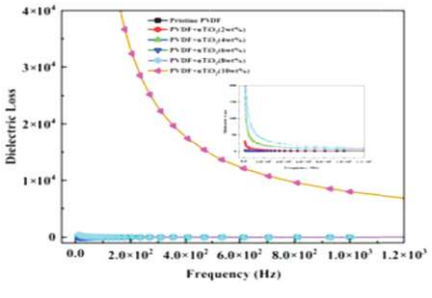

Fig.3: Dielectric Loss Variation with Frequency for PVDF and nTiO_2

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5.2.49 Formation of self-organized nano-dimensional structures on InP surfaces using ion irradiation and their wettability

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Introduction

Implantation of various surfaces with ion beams is one of the important tools for the fabrication of different kinds of self- organized nanopatterns on diverse substrate surfaces [1-2]. Beam parameters like ion energy, ion fluence and angle of incidence etcetera play a significant role in deciding the shape and size of the surface nano pattern (ripple or dots) [2-4]. These patterns have many practical applications.

Experimental Details:

InP samples have been irradiated with 50 keV Ar^+ ion beam at various ion fluence ranging from 2×10^{16} to 8×10^{16} ions/cm^2. The experiment was carried out in 90-degree beamline of Low Energy Ion Beam (LEIB) Facility, dedicated to Materials Science, in IUAC, New Delhi. Nuclear (Sn) and Electronic (Se) energy losses of 50 keV Ar^+ ions inside InP were calculated as 50.5 eV/Å and 19.7 eV/Å using SRIM software. The range of the Ar^+ ions inside InP is approximately 46.5nm. The pristine and irradiated samples were characterized with Atomic Force Microscope (AFM) and Contact Angle Measurement set-up at IUAC, New Delhi.

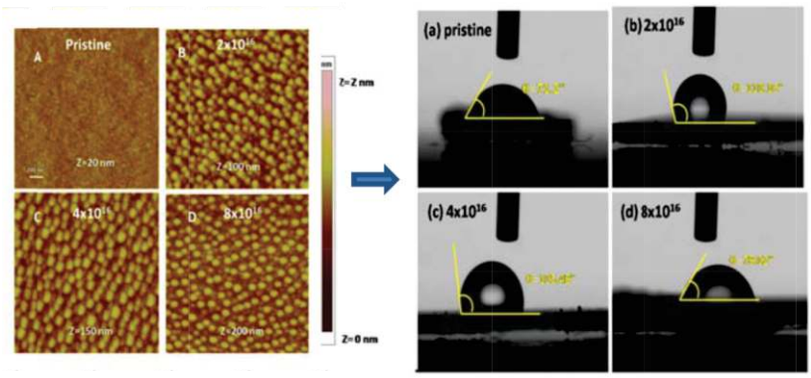

Figure 1. AFM micrographs (left side) of InP samples irradiated at various ion fluences with their corresponding contact angle (right) measurements

Results and Discussion

We observed the formation of regular nanodots on the surface of InP upon irradiation with Ar^+ ion beams. The distribution of size of dots is improved with ion fluence. The size of the dots vary from 70 nm to 90 nm. Due to an interplay between the roughening induced due to sputtering and smoothening due to surface diffusion, reorganization of adatoms occurs on the surface and thus, results in the formation of various surface pattern as a result of ion irradiation. RMS surface roughness increases from 0.4 nm to 10.3 nm with ion fluence and the surface becomes hydrophobic in nature at lower fluences due to trapped air between the nanodots which do not let water drop to seep in as explained in Cassie Baxter Formulism [5].

Conclusion: The nano-dimensional dots formation evolves with ion bombardment on InP surface resulting in change in contact angle of the water droplet with InP surface. Thus, wetting nature of any solid surface may be tuned by optimizing the ion beam parameters in a desired manner.

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5.2.50 SHI induced modifications in multifunctional properties of $BiFeO_3$ based heterostructures

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Swift Heavy Ion (SHI) is an intriguing method for creating or releasing strain in thin films. Because of the accompanying effects such as changes in surface morphology, magnetic anisotropy, columnar or point defect development, and material amorphization, it has been proven that SHI irradiation is responsible for the drastic changes in the electrical and magnetic properties of multiferroics. The multifunctional oxide $BiFeO_3$ (BFO) exhibits remarkable multiferroic properties that are appropriate for prospective application in nanoscale electronic devices such as random-access memory, Field effect transistors, capacitors, logic circuits, and so on. We reported earlier on irradiation-induced modification in the multiferroic characteristics of ~100nm BFO films, which showed an increase in structural strain up to fluence $5x10^{11}$ ions/cm^2 due to defect formation, resulting in improved resistance and polarization switching behaviour[1]. In addition, a focused study was conducted to understand the role of defects in analyzing the influence of electronic excitations induced by swift heavy ions on the ferroelectric properties of ~200nm BFO films grown by pulsed laser deposition. To create artificial defects, all the BFO/SNTO films were irradiated with 200 MeV Ag^{+15} ions by varying ion fluence. The XRD and AFM data reveal that ion irradiation causes structural defects and oxygen vacancies. Modifications in the local electronic structure at the O-K edge suggest that structural defects are responsible for the improvement in Bi-O and Fe-O hybridization and result in the ferroelectric behaviour of BFO Films [2].

The purpose of the proposed project is to investigate the effects of swift heavy ion (SHI) irradiation on the structural, optical, electrical and magnetic properties of Ca-doped $BiFeO_3$/ $LaNiO_3$ / $LaAlO_3$ heterostructures. We have deposited the Ca-doped $BiFeO_3$layer and $LaNiO_3$conducting buffer layer on single crystalline LAO (100) substrate using the pulsed laser deposition (PLD) technique. The distance between the target and the substrate was set at 5 cm using a 248nm KrF Excimer laser with 250mJ energy and a 5Hz repetition rate. The substrate temperature was kept at 700C during the deposition, and all films were deposited for 30 minutes. All proposed heterostructures were irradiated using a 15 UD Pelletron tandem accelerator at the Inter-University Accelerator Centre (IUAC) in New Delhi with 150 MeV Ag^{15+} ions at different ion fluencies i. e. 5×10^{10} ions/cm^2, 5×10^{11} ions/cm^2,1×10^{12} ions/cm^2 and 5×10^{12} ions/cm^{2}and also with 80 MeV O^{7+} ions at different ion fluencies i. e. 5×10^{10} ions/cm^2, 5×10^{11} ions/cm^2 and 5×10^{12} ions/cm^2 with current of ~1pnA. During irradiation, all of the films were attached to the target ladder, which was put inside a high vacuum (~10^{-6} torr) chamber. Irradiation was done perpendicular to the sample surface and scanned across 1×1 cm^2 area with a magnetic scanner to cover the whole sample surface for uniform irradiation.

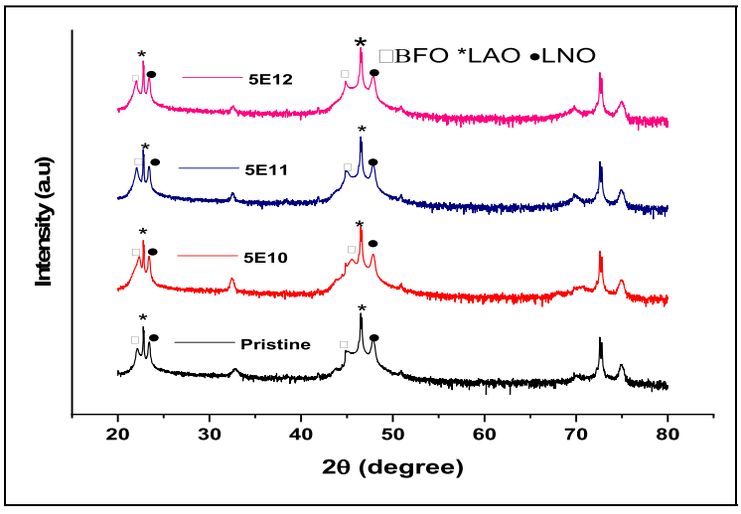

Figure1: XRD pattern of 150 MeV Ag ion irradiated BFO films and show that the ion irradiation causes structural defects and oxygen vacancies.

To explore the structural changes caused by irradiation, we have performed XRD measurements on irradiated heterostructures at room temperature as shown in Fig.1. Analysis of these data indicates the creation of O defects. All other measurements and analyses are under process.

REFERENCES:

[1] Ashish Ravalia, Megha Vagadia, P.S. Solanki, K. Asokan D.G. Kuberkar, Journal of Experimental Nanoscience10:**14**, 1057-1067(2015).