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5.2.19 Ion implantation studies on Ga_2O_3

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Ion implantation is a versatile technology in the semiconductor industry that facilitates selective area doping by exercising more control over dopant concentration compared to bulk doping. It is advantageous in the way that the ions can be incorporated into the material at relatively lower temperatures. Nevertheless, the defects created on ion implantation can degrade the material quality and therefore defect engineering in ion implantation is a part to be looked upon. Gallium oxide is an ultrawide bandgap semiconductor with high potential for power electronic applications, owing to its large bandgap, moderate electron mobility and high breakdown electric field. In addition, the availability of low-cost melt-grown substrates is attracting the attention of the research community to accelerate the developments in Ga_2O_3technology. Implantation studies on Ga_2O_3 are gaining attention due to its efficient doping capability and its significance is being realized through various studies.

In this work, Ga_2O_3 bulk and thin-film samples are implanted with Ge and Si ions using the Negative Ion Beam Facility (NIBF) at Inter University Accelerator Center (IUAC), Delhi for the fluences ranging from 10^{13} to 10^{15} cm^{-2}. The implantation doses and energy were selected to acquire the desired dopant profile which is simulated using the Stopping Range of Ions in Matter (SRIM) software. In order to evaluate the structural changes and damages, the bulk single crystals were first characterized by X-ray diffraction and Raman spectroscopy and the results are being analyzed. The spectra of the implanted samples are compared with the unimplanted substrate used as a reference which is depicted in Figures 1 and 2. Further characterization will be carried out on the samples to understand the defects created upon the implantation of Ga_2O_3. This includes photoluminescence and temperature-dependent Raman studies. The electronic and electrical properties of the crystal will be performed to know the effectiveness of implantation doping in the samples.

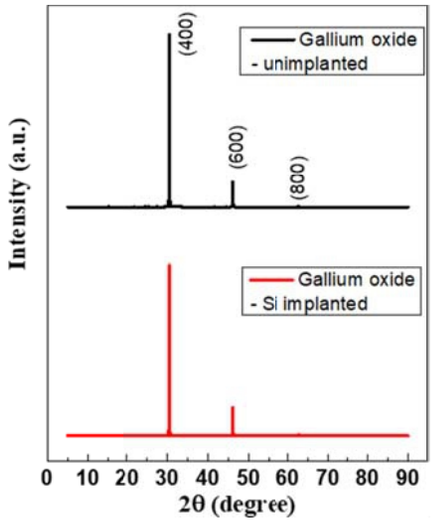

Figure 1. XRD of unimplanted and Si implanted Ga_2O_3

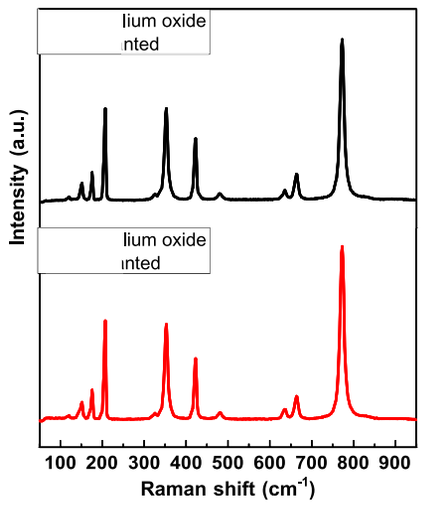

Figure 2. Raman spectra of unimplanted and Si implanted Ga_2O_3

5.2.20 Structural Phase Transitions induced by Swift Heavy Ion Irradiations in Lead Free Ferroelectric Perovskite Oxides

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Ferroelectric ceramic oxides have attracted the attention of the scientific community due to their excellent dielectric, ferroelectric and piezoelectric properties, which made them potential candidate for fabrication for actuators, sensors and various memory devices. However, in consideration of environmental concern, an extensive research on lead free ferroelectric ceramics with significant piezoelectric and dielectric properties has been of recent demand.

$Na_{0.5}K_{0.5}NbO_3$ (KNN) is considered to be one of the good lead free ferroelectric ceramics because it exhibits a very high curie temperature (T_C = 420 ^{0}C), good dielectric and ferroelectric properties [1]. However due to the presence of volatile element in KNN, it is difficult to densify it properly leading to the deterioration of the physical properties. Fabrication of solid solution is one of the effective strategies to overcome these drawbacks. So in this work the solid solution of KNN with $BiScO_3$ i.e., (1-*x*) $Na_{0.5}K_{0.5}NbO_3$–*x* $BiScO_3$ ((1-x) KNN–*x* BS) is considered. $BiScO_3$ belongs to rhombohedral structure with curie temperature (T_C = 480 ^{0}C) and at room temperature it has different symmetry than KNN which may gives rise to get the MPB [2]. The preliminary structural analysis, ferroelectric phase transition using temperature dependent dielectric properties of (1-x) KNN–*x*BS were investigated.

The binary solid solution (1-*x*) KNN-*x* BS ($0 \leq x \leq 0.05$) were prepared by conventional solid state reaction route. Reagent grade oxide and carbonate powders of Nb_2O_5, Bi_2O_3, Sc_2O_3, K_2CO_3, and Na_2CO_3 were used as starting raw materials [1,2]. Before weighing, all these powders were preheated in a furnace at 200 ^{0}C for 2 hours in order to remove any moisture absorbed by the alkaline carbonates. Then the stoichiometric amount of powders was ground for 3 h in mortal under both dry mixing followed by wet mixing. Then the grounded powder was calcined at 875 ^{0}C for 6 hours at a heating rate of 5 ^{0}/minutes. After that, the calcined powder was mixed with 3 wt% PVA to prepare the pellets with a diameter of 10mm under a pressure of 6× 107 kg/m^2. The pellets were then sintered at 1125-1150 ^{0}C for 3 hours.

Figure 1 (a) shows the room temperature x-ray diffraction (XRD) patterns of (1-*x*) KNN-*x* BS ceramics. As observed from these patterns, all samples show a pure perovskite phase without any secondary phase which are in accordance with the literature reported earlier [1,2]. In order to study the phase transition behaviour of (1-*x*) KNN-*x* BS, we have carried out dielectric measurement from room temperature to 500 ^{0}C for selected frequencies and shown in Figure 1 (b) and (c). From Figure 1 (b) it can be observed that the first small anomaly around 200 ^{0}C corresponds to orthorhombic to tetragonal phase transition, while another sharp peak around 410 ^{0}C corresponds to tetragonal to cubic phase transition suggesting the ferroelectric to paraelectric phase transition [2]. Similar behavior is also observed in the temperature dependent dielectric loss curve. From Figure (c) it can be seen that with increase in BS concentration the curie temperature (T_C) decreases with respect to pristine KNN (T_C = 410 ^{0}C) and the room temperature dielectric constant also increases. The measurement and analysis on microstructural, Vibrational (using Raman Spectroscopy), Ferroelectric and piezoelectric properties are under progress. The synthesis and characterization of another series i.e., $Na_{0.5}K_{0.5}NbO_3$–x$BaSnO_3$ ((1-x) KNN–*x*BSn) is also underway.

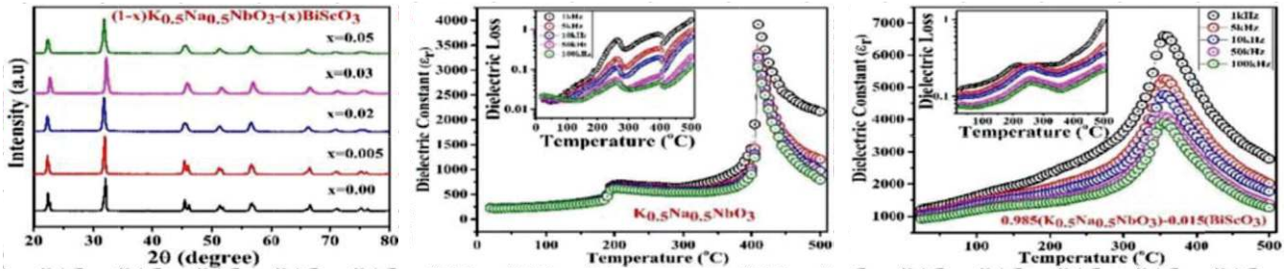

Figure 1 (a) Room temperature XRD pattern of (1-x) KNN-xBS ($0 \leq x \leq 0.05$), (b) & (c) Dielectric constant of both KNN and (1-x) KNN-xBS (x=0.015) (inset figure shows their respective dielectric loss.)

The SHI irradiation induced structural phase transition (on KNN and Optimized (1-*x*) KNN-*x* BS/BSn) will be investigated for different radiation dose rates, and irradiation environments. So the structural phase transition will be studied using *in situ* XRD measurement by irradiating with SHI of different fluence at different temperatures. The Rietveld refinement technique will be used for the structural analysis. The mechanism for the phase transition will also be correlated along with the Raman spectroscopic technique. Correlation of the dielectric, ferroelectric and piezoelectric properties with the change in the crystal structure due to ion irradiations will also be performed.

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5.2.21 Structural, morphological and electrical properties of Co and Ti co-doped α-Fe_2O_3 system

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Hematite (α- Fe_2O_3) and transition metal (TM) doped hematite (α- Fe_2O_3) are getting importance for development of low power consuming spintronics devices. The work is proposed to co-dope Co (divalent) and Ti (tetravalent) atoms into hematite structure and study their structural phase stability, morphology, electrical and magnetic properties in the bulk and thin-film form of the material. The experimental facilities at IUAC, New Delhi will be used to prepare thin films of Fe_2O_3 and Co/Ti co-doped Fe_2O_3, study of defect induced properties by ion beam implantation and irradiation. The materials will be characterized GIXRD, FE-SEM and EDX, and RBS. The electrical conductivity will be studied.

The target materials of compositions α-Fe_2O_3, $Fe_{1.4}Co_{0.3}Ti_{0.3}O_3$ and $Fe_{1.8}Co_{0.1}Ti_{0.1}O_3$ were prepared by mechanical alloying of oxide powders. The pellets were heated at 700º C -7h for α-Fe_2O_3 and 1000º C-6h for Co/Ti co-doped samples. The thin films were grown on Al_2O_3, P-Si (100) and Quartz substrates using thermal evaporation method at IUAC. The Thermal evaporation chamber (Excel instruments, Germany) was maintained at high vacuum (~10-7 mbar). The substrate temperature was maintained at 700° C. Deposition current and rate was maintained around (80-84) Å and (1- 1.5) Å/s, respectively. The FE-SEM images of target materials were recorded at different resolutions 20kx- 200kx and elemental compositions were determined using EDX. The temperature (250 K- 400 K) dependence of electrical conductivity were measured for the bulk sample of $Fe_{1.8}Co_{0.1}Ti_{0.1}O_3$. The conductivity of the bulk samples varied in the range of 10-7 (S/m) to 10-2 (S/m) depending on heat treatment of the samples in air or vacuum.

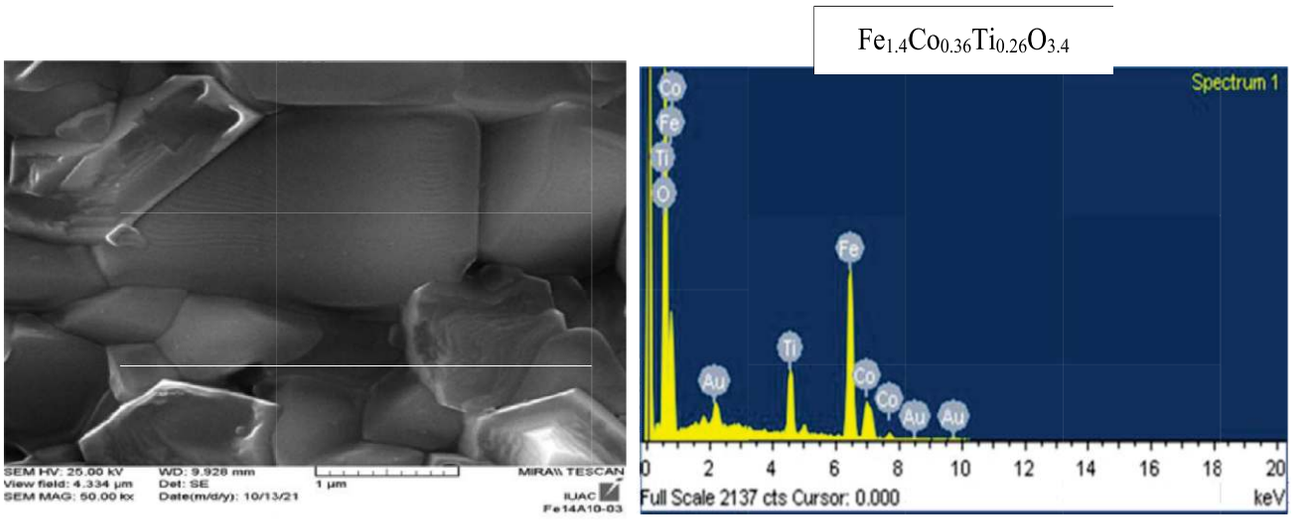

Fig: FE-SEM images and EDX data recorded for $Fe_{1.4}Co_{0.3}Ti_{0.3}O_3$ composition at IUAC, New Delhi using MIRA II LMH (TESCAN) and INCA PENTA FET3 (OXFORD) instruments.

GIXRD confirmed no typical crystalline structure in the as-grown films of the samples. The heat treatment of the as-grown films at 800 °C stabilized crystalline phase of hematite structure for $Fe1.8Co_{0.1}Ti_{0.1}O_3$ composition on all the substrates, whereas pure Fe_2O_3 films also crystallized after heat treatment (except on Alumina substrate) and $Fe_{1.4}Co_{0.3}Ti_{0.3}O_3$ films do not crystallized after heat treatment at 800 °C. Detailed analysis of the experimental data are in progress.

5.2.22 Role of swift heavy ion irradiation in device characteristics of manganite based thin films

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The targets of $LaMnO_3$ and $La_{0.7}A_{0.3}MnO_3$ have been prepared by using the conventional ceramic / solid state reaction method. The targets were characterized by X–ray diffraction (XRD) technique for their crystalline quality followed by their structural analysis using the Rietveld refinements. The PLD targets were utilized for the preparation of bilayered thin film devices using PLD method, in accordance with the proposed stoichiometries and proposed layer thicknesses. All devices were prepared in a set of four numbers to perform various fluence based SHI irradiation thereby to understand the effect of SHI irradiation on the device characteristics of proposed systems.

During the second year (i.e. next financial year; 2022–2023) of sanctioned UFR project, SHI irradiation will be performed followed by their characterizations and related analysis / publication process.

5.2.23 Anisotropic Magnetoelectric and Magnetotransport Properties of Manganite Based Thin Films

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During this project tenure, polycrystalline targets of $La_{0.90}Ca_{0.10}MnO_3$ (LCMO) and $La_{0.90}Sr_{0.10}MnO_3$ (LSMO) were prepared through various starting materials including La_2O_3, MnO_2, $CaCO_3$ and $SrCO_3$ powders of 99.9% purity (Sigma Aldrich make) by conventional solid state reaction (SSR) route. Thin films of these targets were fabricated on SNTO *(100)* single crystalline substrates using pulsed laser deposition (PLD) technique. Further, these films were irradiated with Ag^{+15} ions at an ion fluence of 1×10^{11} ions/cm^2, 1×10^{12} ions/cm^2 and 1×10^{13} ions/cm^2 using IUAC, New Delhi beam time facility during the same project.

For all the thin films, structural studies were carried out using X–ray diffraction (XRD; make: Philips; model: PW 3040/60 X'pert Pro) with Cu Kα radiation having λ = 1.54Å at room temperature. Frequency dependent electrical properties were performed by high precision LCR meter (make: Agilent; model: E4980A). Dielectric constant with varying frequency has been studied in the frequency range of 20 Hz to 2 MHz at room temperature. Moreover, the contribution of the charge carriers and relaxation time can be estimated by two theoretical models, namely, relaxation model and universal dielectric response (UDR) model. The frequency dependent ac conductivity was measured throughout the same frequency range. The hopping mechanism of the charge carriers in the presently studied pristine and irradiated films was evaluated by the theoretical fit of Jonscher's power law. Moreover, impedance behaviour of all the films was performed to recognize the grain and grain boundary contributions, separately, at different frequency ranges between 20 Hz and 2 MHZ.

All the above electrical measurements were also performed in current perpendicular to plane (CPP) geometry, where the direction of applied magnetic field has created a significant alteration in the electrical properties. In this context, anisotropic magneto dielectric measurements were performed to identify and understand the possible anisotropic magneto dielectric and magnetoelectric nature of the films under study. For the present study, the anisotropic magneto dielectric measurements were carried out in mainly two modes: (i) magnetic field applied perpendicular to the sample plane and (ii) magnetic field applied parallel to the sample plane. Based on the above mentioned characterization method, frequency dependent dielectric, ac conductivity and impedance have been analyzed for all the pristine and irradiated films. While the in-depth discussion of LCMO and LSMO are studied on the basis of morphological properties, theoretical fittings and understanding of possible anisotropic magneto dielectric and magnetoelectric properties. However, from the till–date quantitative studies on the same, it is confirmed that strain between the films and substrates is an important tool to control the anisotropic magneto dielectric and magnetoelectric parameters and properties which will be useful aspect of the present studies.

5.2.24 Radiation Damage in Polyethylene Terephthalate due to 1.75 MeV N^{5+} Ion – studied by XRD, UV-Vis Spectroscopy and FTIR Spectroscopy

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Lower energy ion irradiations are usually more suitable for investigating soft materials like polyethylene terephthalate (PET). We have studied effects of 1.75 MeV N^{5+} ion beams of fluences, ranging from 1×10^{11} to 5×10^{14} ions/cm^2 on structural, optical, and chemical properties of PET polymer. We used x-ray diffraction (XRD), UV–Visible spectroscopy, and Fourier transform infrared (FTIR) spectroscopy. The XRD patterns of PET samples (fig. 1) show that the crystallinity increases with ion irradiation at fluences of 4×10^{11} and 5×10^{12} ions/cm^2 in fig 2 [1]. Optical bandgap energy decreases more at the ion fluence of 5×10^{14} ions/cm^2. Absorption maxima shifted towards a higher wavelength value due to the formation of extended conjugation. Acetylenic (-C≡C-) group formation and free CO_2 group have been confirmed by FTIR spectroscopy. Interesting mechanism of the degradation product formation chemistry has been discussed [1].

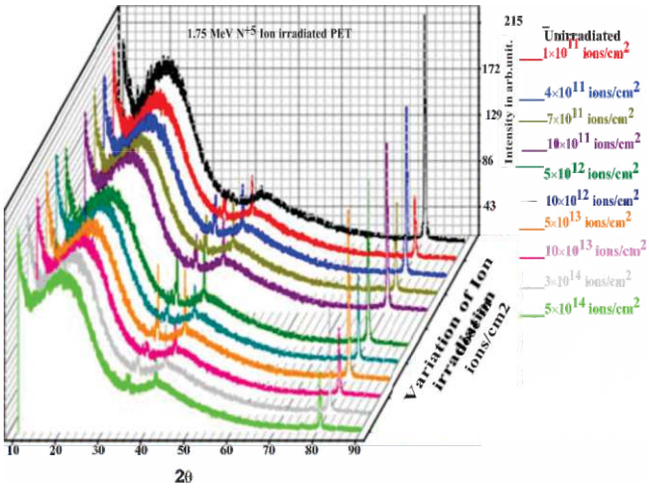

Fig. 1 XRD patterns of virgin PET sample and samples irradiated by 1.75 MeV N^{5+} ions to different ion fluences.

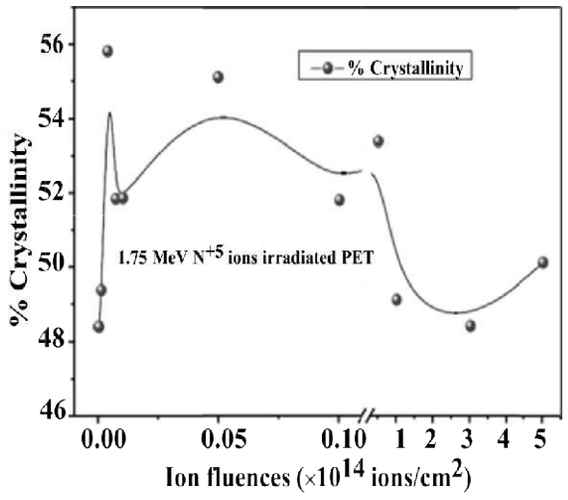

Fig. 2 Variation of crystallinity for various ion fluences exposure of our PET sample

Authors thanks Dr. Ambuj Tripathi (IUAC) and members of LEIBF facility at IUAC

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5.2.25 Influence of Swift Heavy Ion Irradiation on Charge Transport Properties of Manganite Based Thin Films

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To understand the effect of 100 MeV O^{+9} ions irradiation in modifying structural, microstructural, and transport properties of ~ 100 nm $La_{0.5}Gd_{0.2}Ca_{0.3}MnO_3$ (LGCMO) films grown on single crystalline (100) Nb:$SrTiO_3$ (SNTO) substrate by pulsed laser deposition (PLD) technique (LGCMO – Pristine) [1]. Here, the irradiation performed using 15 UD Tandem Accelerator on LGCMO films with different ion fluence ~ 5×10^{11} (LGCMO – 11), 5×10^{12} (LGCMO – 12) and 5×10^{13} ions/cm^2 (LGCMO – 13). The effect of manganite defects created through SHI irradiation at LGCMO–substrate (p–n) interface was investigated via various characterization techniques such as X–ray diffraction (XRD), Atomic force microscopy (AFM), and Keithley 2612 A sourcemeter. From XRD the decrease in structural strain is observed up to a fluence of LGCMO–12, while for LGCMO – 13 it gets enhanced due to the irradiation– induced modifications in the crystallinity for higher fluence films. The AFM shows improvement in average grain size up to LGCMO–12 but suppression in grain size with increased grain boundary density as well as rms surface roughness. To understand the transport properties across LGCMO–SNTO p–n interfaces for all films, current–voltage (I–V) characteristics show all the films exhibit non–linear behaviors. The LGCMO–PRI film exhibits backward diode–like behavior while the irradiated films up to LGCMO–12 show tunnel diode–like behavior and enhancement in conduction across LGCMO–SNTO interface. Higher ion fluence film (LGCMO–13) shows suppression in conduction across the p–n interface in the context of grain size, grain boundary, and interfacial strain, hence, the film exhibits the strongest backward diode characteristics among all the films.

In order to achieve the annihilation effect of irradiated LGCMO films, a 50 nm thick ZnO layer was grown on the surface of LGCMO/SNTO films using the chemical solution deposition (CSD) method under an oxygen environment with a controlled flow [1]. The XRD, AFM, and I–V measurements were performed again for annealed ZnO/LGCMO interfaces. In XRD patterns (recorded after annihilation) reveal the single–phase polycrystalline growth of the ZnO layer over the LGCMO/SNTO thin films surfaces. All the films show (ZnO–PRI to ZnO–13) positive value of tensile across the LGCMO/SNTO interface gets suppressed due to the annihilation effect which can provide a sharp interface. The effect of annihilation on the surface morphology of ZnO/LGCMO/SNTO films was studied by performing AFM measurements. Average grain size from AFM images is found to increase up to ZnO–12 while higher ZnO–13 shows a reduction in average grain size, after the annihilation process. The effect of annihilation on charge conduction across the ZnO/LGCMO p–n interface was studied by performing the I–V

characteristic using the four– probe method at room temperature. All the films exhibit non–linear I–V behavior and modifying conductivity under the annihilation effect due to the lattice strain which gets suppressed (i.e. improved interface) as well as due to an average grain size that gets increased for ZnO/LGCMO films after the annihilation process. In addition, I–V characteristics exhibits improved grain size resulting in the enhanced current across ZnO and LGCMO interface. Also, the reduced strain between LCGMO and SNTO materials are considered for enhancement in the current since reduced strain inserts comparatively lower disorder in the manganite layer.

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5.2.26 Temperature dependent swift heavy ion induced effects in Cerium and Yttrium doped Zirconolite

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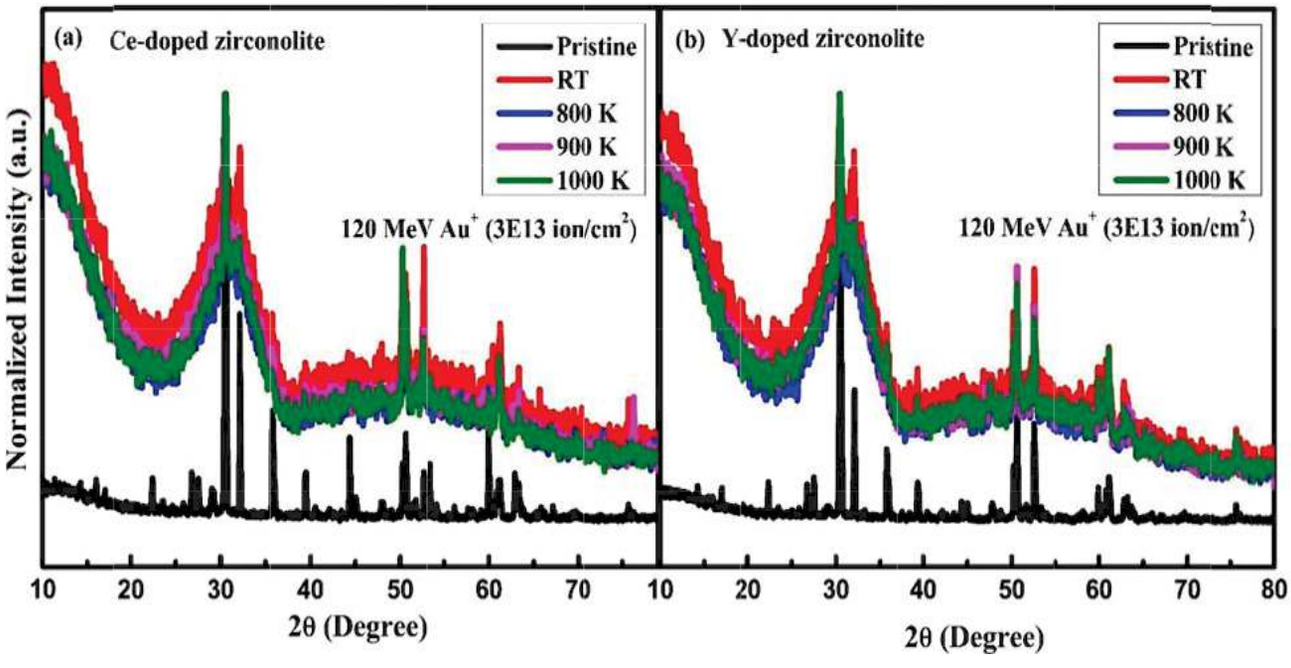
Zirconolite, a polyphase titanate ceramic has been considered as a stable host phase for immobilizing high level radioactive wastes (HLWs) [1]. Various studies have been performed to evaluate the radiation induced effects on zirconolite to check their long-term stability and durability. In most of the studies, zirconolite has been investigated for the induced effects of α-decay events irradiating by low energetic heavy ion beams at room temperature as well as high temperatures [2-4]. In present work, the swift heavy ion irradiation has been performed to understand its radiation tolerance and long-term stability under the effect of fission fragments. For said purpose, samples were prepared by solid state reaction method with 20% doping concentration of simulated oxides (CeO_2, and Y_2O_3). The prepared samples were found to be almost single phase 2M-zirconolite as characterized through XRD and Raman measurements [5]. It was concluded that the zirconolite can easily accommodate tri- and tetravalent wastes with its 20% concentration and retains its structural integrity [5]. To study the effects of the swift heavy ion (SHI) irradiation on the structural, physical, and chemical properties of the zirconolite compositions, samples were irradiated with 120 MeV Au^{9+} ions at room temperature, all the samples have been observed to undergo amorphization which is confirmed through disappearance of characteristic x-ray diffraction peaks and appearance of broad diffuse scattering bands.

To study the temperature dependent irradiation induced effect zirconolite compositions with the doping of simulated oxides, samples were irradiated with 120 MeV Au^{9+} ions at a fluence 3E13 ion/cm^2 at different temperatures 800K, 900K and 1000 K. Experiment of temperature dependent study of ion irradiation was done in high vacuum chamber of Materials Science Beam Hall II, using 15 UD Pelletron tandem accelerator at Inter University Accelerator Centre (IUAC), New Delhi. To investigate the structural changes in the matrices, we have carried out the wide angle and Grazing angle XRD measurements of the irradiated samples at IUAC, New Delhi.



The wide angle XRD patterns for both compositions (Ce-CZT and Y-CZT) irradiated with 120 MeV Au^{+} ions at different temperatures of 800K, 900K and 1000 K shown in Fig. 1(a) and (b) respectively, revealed that patterns for both compositions weere acquired the state of its amorphization similar to that of its room temperature stage. So, results suggest that critical temperature, at which a material remains crystalline for a particular value of ion fluence, is above 1000 K for Ce-CZT and Y-CZT under the induced effects of SHI irradiation. Other measurements and analysis are under process.

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5.2.27 Tunable Characteristics of Porous Silicon Optical Microcavities by Energetic N Ion Beams Interactions

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The present study demonstrates the tuning of optical characteristics of porous silicon (PSi) based microcavities by N ion beams interactions. These optical microcavities are prepared by using electrochemical etching of heavily doped *p+-type* Si. The PSi microcavities were exposed to N ions of 200 keV and 1 MeV at an optimized ion fluence of $1x10^{15}$ ions/cm^2. A significant red shifting of 32~60 nm resonance cavity mode was observed due to ion interaction. The experimental spectral data are in good agreement with the transfer matrix simulations. A substantial modification of PSi microcavity surface states is visualised through the Raman and X-ray photoelectron spectroscopies (XPS). The Raman spectral results show modifications from crystalline Si (c-Si) to nanostructured Si (n-Si) and subsequently to amorphous Si (a-Si). The XPS indicates the modification of Si–Si and Si–O bonds and the formation of new Si–N bonds implying the presence of Si_3N_4. These experimental observations along with analytical simulations and cavity modelling conclusively support the realization of cavity tunability and substantial modification in the optical field intensity and photon confinement within the spacer layer of the microcavity. These results suggest that ion beams are the effective tools to produce wider tunable optical properties in a microcavity with highly stable designer optical structures suitable for photonic applications. [2].

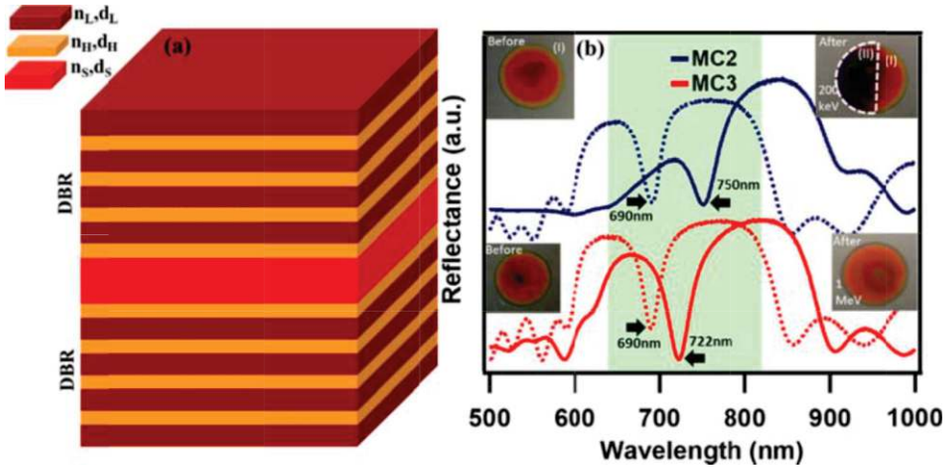

Figure 1. *(a) The 3-D schematic representation of the PSi-based microcavity (b) Experimental reflectance spectra of both PSi microcavities (MC2 and MC3) before and after N ion beam interactions. The color change before and after N ion beam interaction is shown in the conventional reflection images of both (MC2 and MC3) samples.*