Effect of 120 MeV Ag ion irradiation on the structural and electrical properties of NiO/ZnO heterojunction

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Abstract

NiO/ZnO heterojunction deposited on Si(100) substrate by evaporation method were irradiated with 120 MeV Ag ions. The effects of ion irradiation on various properties of the heterojunction were studied. Crystallinity of NiO layer improved due to irradiation at the fluence of $3 \times 10^{11}$ ions cm$^{-2}$ as revealed by the increase in grazing incidence X-ray diffraction (GIXRD) peak intensity. Further irradiation reduced the intensity of GIXRD peaks but could not suppress it completely even at the highest fluence ($1 \times 10^{13}$ ions cm$^{-2}$), where the whole bilayer is expected to be covered with ion tracks. This observation indicated that the columnar region around the ion path is not completely amorphized, but its crystallinity is reduced. Evolution of the area under GIXRD peaks with ion fluence gave radius of this modified columnar region as $\sim 4.9$ nm. The peaks of ZnO were not clearly evident in the GIXRD pattern. However a weak peak due to ZnO could be seen in the Raman spectra, which also did not completely vanish even at the highest fluence of irradiation. The bilayer sample irradiated at a fluence of $1 \times 10^{12}$ ions cm$^{-2}$ showed the highest switching ratio, which could be due to the thermal spike induced diffusion of ions across the NiO/ZnO junction.

1. Introduction

Zinc oxide (ZnO) is a preferred material for optoelectronic applications owing to its wide band gap [1, 2], large exciton binding energy [3], high electron mobility [4], corrosion resistance [5] and high chemical stability [6]. ZnO based transparent conducting oxide (TCO) in thin film form find many applications, for example LEDs [7], laser diodes [8], solar cells [9], storage devices [10], spintronics [11], gas sensors [12] etc. For UV photo detector applications, p-n junctions with ZnO as n-type semiconductor have been investigated extensively [13, 14]. The appearance of n-type conductivity in ZnO is due to the presence of oxygen vacancies and zinc interstitials [15]. Among different p-type materials that have been considered to form p-n junction with ZnO, p-type NiO stands out due to its band alignment with ZnO [16]. The vacancy at Ni$^{2+}$ sites in NiO make this material p-type [17, 18]. This being a wide band gap (3.6 to 4.0 eV) semiconductor [19] like ZnO (3.37 eV), also find applications as transparent conductive oxide films [20], anode material [21], electrochromic devices [22] etc. In comparison to ZnO based nano sensors, NiO/ZnO heterojunction exhibit superior gas sensitivity and many other promising applications [23].

Ohta et al. [24] reported preparation of transparent NiO/ZnO heterojunction for the first time. Since then these junctions have been prepared using varieties of methods like sol-gel spin coating [25], chemical bath deposition [26], hydrothermal method [27], pulsed laser deposition (PLD) [28]. It is well established that the structure, microstructure and hence the properties of the films can be engineered either during the time of
deposition by tuning various deposition parameters or by various post deposition modification techniques such as thermal annealing and ion irradiation methods. In recent years, swift heavy ion (SHI) irradiation has been considered as one of the unique tool for modification and engineering of materials [29].

In this paper, we report 120 MeV Ag ion irradiation induced effect on the structural, and electrical properties of NiO/ZnO heterostructure. Our study indicated that the sample irradiated at a fluence of $1 \times 10^{12}$ ions cm$^{-2}$ shows highest switching ratio. This is shown to be due to thermal spike induced diffusion of ions across the NiO/Zn junction of the sample.

2. Experimental details

Zinc layer of $\sim 70$ nm was deposited on Si(100) substrate by thermal evaporation method. Since the melting temperature of Zn is low ($419.5$ °C), therefore we chose the resistive thermal evaporation method which is one of the excellent low-thermal budget deposition technique. Pellets of Zn were used as the target material and kept in Al$_2$O$_3$ coated Mo boats. During deposition, the chamber pressure was maintained at $\sim 2.5 \times 10^{-7}$ kPa and the rate of deposition of the films was $\sim 0.2$ nm s$^{-1}$. The asdeposited thin films of Zn were further annealed at 400 °C for 1 h in ambient atmosphere for ZnO formation. Subsequently, NiO film of $\sim 100$ nm was deposited on the ZnO layer by e-beam evaporation following the procedure described elsewhere [30]. NiO in the form of pressed pellet was taken as target and is evaporated at a rate of $\sim 0.1$ nm s$^{-1}$. The vacuum during deposition was $\sim 2.5 \times 10^{-7}$ kPa.

The as fabricated NiO/ZnO heterojunction samples were irradiated by 120 MeV Ag ions at various fluences at room temperature using the 15 UD Tandem Pelletron Accelerator at the Inter-University Accelerator Centre (IUAC), New Delhi. The samples were mounted on copper ladder in a high vacuum chamber ($\sim 10^{-7}$ kPa) for irradiation. The ion beam was magnetically scanned over an area of $1 \times 1 \text{cm}^2$ to ensure uniform irradiation on the sample surface. The ion flux was kept low ($\sim 3.5 \times 10^9$ ions cm$^{-2}$ s$^{-1}$) to avoid heating on the samples due to ion beam. Following the procedure as outlined in the literature [31–33], the increase in temperature at the sample surface was estimated to be less $\sim 3.4$ K. Hence we ruled out the beam heating effect for the observation modification. The irradiation fluence range was from $3 \times 10^{11}$ to $1 \times 10^{13}$ ions cm$^{-2}$. The penetration depth and energy loss parameters of 120 MeVAg ion have been calculated by using SRIM codes [34] for the NiO and the ZnO films.

The structural characterization of pristine and irradiated samples were carried out using a Bruker D8 (Model: AXS D8 Discover) diffractometer operated with voltage and current of 40 kV and 40 mA respectively in grazing incidence mode at room temperature. In order to investigate the irradiation induced structural modification, the incident angle of Cu K$\alpha$ X-radiation ($\lambda = 1.540$ Å) on the sample surface was fixed $\sim 0.5^\circ$ and all the grazing incidence x-ray diffraction (GIXRD) spectra were collected under identical scanning condition. Raman Spectroscopy of the samples were undertaken using Jobin Yvon Horiba Raman spectrometer (LABRAM-HR) having overall spectral resolution of 1 cm$^{-1}$. The source used is diode laser having the wavelength of 473 nm and power of 23 mW. The current density versus voltage (J–V) characteristics of the samples were undertaken using Keithley 2612 A source-meter in a two probe station with aluminium as contact material.

3. Results and discussion

The GIXRD pattern of pristine and 120 MeV Ag ion irradiated NiO/ZnO heterostructure at various fluences is presented in figure 1. As evident from this figure, the peaks (111), (200), (220) and (311) corresponds to fcc structure of NiO (JCPDS card No.75-0296). The peaks of ZnO however were not above the noise level. Irradiation with 120 MeV Ag ions had a complex effect on the intensity of the NiO peaks. The GIXRD peak intensity increased on the first fluence ($3 \times 10^{11}$ ions cm$^{-2}$) of irradiation, and then decreased on subsequent irradiation at higher fluences. The peaks however, did not vanish even at the highest fluence ($1 \times 10^{13}$ ions cm$^{-2}$), where cylindrical ion tracks are expected to overlap as discussed later. Similar observations have been reported earlier [32, 35–37]. The position of the peaks were not affected by irradiation.

The increase of the area under the XRD peaks in low fluence ($3 \times 10^{11}$ ions cm$^{-2}$) of irradiation as is seen in the present study indicated improvement of crystallinity. This is not a frequently observed phenomenon as SHI irradiation ordinarily leads to defect and disorder in the crystal structure and hence suppression of the XRD peak intensity. A few studies however have reported improvement of crystallinity upon irradiation in the low fluence regime [30,37–39]. This unusual effect of irradiation points to the applicability of one of the two frequently used models of ion–matter interaction: (i) Coulomb Explosion (CE) [40] and (ii) Thermal Spike (TS) [41–43].

Coulomb explosion model is based on the electrostatic repulsion between the ions created in the wake of the projectile ion before charge neutrality is restored. This repulsive force can induce disorder and amorphization to
the original crystalline structure of a target medium along the ion track. The improved crystalline due to 120 MeV Ag ion irradiation in the low fluence regime as observed in the present study therefore cannot be explained by the CE model. On the other hand, the thermal spike model predicts formation of amorphized/defected latent tracks if the thermal spike temperature in the track region exceeds the melting temperature of the medium. In the surrounding region however, the temperature falls off with increasing radius away from the track. The material surrounding the track region can thus experience a significant rise in temperature above the ambient. Such transient temperature rise in the surrounding region cannot melt the material since it is less than the melting temperature, but can have varied consequences depending upon the materials characteristics and ambient. Such transient temperature rise in the surrounding region cannot melt the material since it is less than the melting temperature, but can have varied consequences depending upon the materials characteristics and ambient [29, 30, 37].

We thus envisage that around the path of a single ion, two concentric cylindrical regions form: (i) The heavily disordered core of a few nm radius called the latent track as is conventionally seen [43, 44], (ii) A region surrounding this core where the temperature rise relaxes the strain generated during thin film growth [30] and hence improves crystallinity. In the low fluence regime, the second region of much larger radius than the core, overlaps leading to the observed improved crystallinity. At higher ion fluences (\( \geq 1 \times 10^{12} \) ions cm\(^{-2} \)), the highly disordered core regions, the latent tracks tend to overlap. As a consequence, the crystallinity is suppressed leading to the suppression of XRD peak intensity at high ion fluences [45].

The damage cross section can be estimated by fitting the fluence dependence of the area to Poisson’s equation [36, 46].

\[
A(\varphi t) = A_{\infty} + (1 - A_{\infty})e^{-\sigma_{fl} \varphi t}
\]  

(1)

where \( A(\varphi t) \) and \( A_{\infty} \) are the area under the XRD peak at an ion fluence (\( \varphi t \)) and saturation value of the area at high ion fluences (\( \varphi t \to \infty \)) respectively. Since the ZnO peaks were below the noise level at all ion fluences (figure 1), we considered the evolution of the area under three strong NiO peaks (111), (200) and (220) with ion fluence for extracting the radius of the core region. Equation (1) suggests exponential decrease of the area with ion fluence. We however found increase of the area at the first fluence (\( 3 \times 10^{11} \) ions cm\(^{-2} \)) of irradiation due to strain relaxation as discussed earlier. Irradiation at higher fluences beyond this value led to decrease of the area as predicted by equation (1). We therefore rescaled all the fluences taking the fluence \( 3 \times 10^{11} \) ions cm\(^{-2} \) as reference. The GIXRD peak areas at different fluences were normalized with respect to the area of the peaks at the fluence \( 3 \times 10^{11} \) ions cm\(^{-2} \). The normalized area of the (111), (200) and (220) peaks as a function of the rescaled fluence are shown in figure 2 (a). Figure 2 (b) gives the averaged normalized area of these three peaks. Fitting of figure 2 (b) to equation (1) gave the cross section, \( \sigma_{fl} \) of the core region as 7.68 \( \times \) 10\(^{-13} \) cm\(^{2} \). Assuming cylindrical geometry of the Ag ion track core, its radius, \( r \) is estimated from \( \sigma_{fl} = \pi r^{2} \) to be \( \sim 4.9 \) nm. Such a large track radius is not expected since the electronic energy loss \( S_{e} (\sim 27.62 \text{ keV nm}^{-1}) \) of 120 MeV Ag ions is less than the threshold electronic energy loss \( S_{\text{eth}} \) for track formation in NiO (\( \sim 30 \text{ keV nm}^{-1} \) [47]). These values however pertain to single crystals while the present study uses bilayers of NiO and ZnO thin films. Irradiation is expected to cause mixing at the interface of the bilayer system and that needs a lower value of \( S_{\text{eth}} \) than that of single crystals. For example an \( S_{\text{eth}} \) of \( \sim 12 \text{ keV nm}^{-1} \) has been reported for the occurrence of ion beam induced mixing at the interfaces of similar systems [48, 49]. We thus presume that the large track radius we

![Figure 1. Evolution of the GIXRD patterns of NiO/ZnO thin films with 120 MeV Ag ion irradiation at various fluences.](image)
observe pertains to the large interaction cross section of mixing at the interface of the bilayers rather than the creation of amorphized latent tracks.

Figure 3 shows the Raman spectra of NiO/ZnO bilayer irradiated at various Ag ion fluences. A band at \( \sim 304 \text{ cm}^{-1} \) corresponding to ZnO symmetric stretching vibration [50] appeared in the spectra at all fluences. This observation indicated that the ZnO layer is not completely amorphized even at the fluence of \( 1 \times 10^{13} \text{ ions cm}^{-2} \). This established the radiation resistance of ZnO as has been seen by many [51–53]. The Raman peaks corresponding to NiO phase however could not be extracted due to their overlap with the peaks of the silicon substrate.

The resistive switching (RS) behaviour of the NiO/ZnO heterojunction was examined by applying the voltages with the sequence: 0 V \( \rightarrow \) 5 V \( \rightarrow \) 0 V \( \rightarrow \) −5 V \( \rightarrow \) 0 V. The J–V characteristic curve illustrated in figure 4 demonstrating the RS behaviour of pristine sample as well as the sample irradiated at the fluence of \( 1 \times 10^{12} \) and \( 1 \times 10^{13} \text{ ions cm}^{-2} \).

The bipolar resistive switching (BRS) behaviour is the change of resistance of the sample by reversing the electrical polarity of the applied electric field [54–56]. The NiO/ZnO heterojunction was initially at high resistance state (HRS). During the application of positive sweep from 0 to 5 V, the current increases gradually and the device switched to low resistance state (LRS). The device maintained the LRS during the sweep from 5 to −5 V and the same switched back to HRS during the voltage sweep back from −5 to 0 V. Clear current loop is not evident in the pristine sample during voltage cycle. The clear current loop with improved RS characteristics
is evident when the sample is irradiated at a fluence of $1 \times 10^{12}$ ions cm$^{-2}$. In this case, one will notice a cross point in the positive bias side around 0.2 V where the resistance state changed from LRS to HRS up to 0 V. The sample again retains LRS while sweeping the voltage from 0 to $-5$ V. Beyond this fluence of irradiation, the switching behaviour of the sample started decreasing. The sample showed much reduced RS behaviour with narrow current loop when irradiated at the highest fluence. The decrease of RS behaviour could be associated with ion irradiation induced disordering in the layers [57]. The switching mechanism can be explained by electroforming mechanism [54]. According to this mechanism, during set process the metal atoms are oxidized to form metal ions and then these ions start to accumulate at the electrodes. The device switches from HRS to LRS when the metallic (Zn/Ni) filaments develop a link between the electrodes and the same switches back to HRS state again when the filament ruptures [58].

In order to quantify the RS behaviour, we calculated the switching ratio i.e. ratio of current in LRS to corresponding HRS at 2 V for the heterojunction irradiated at different ion fluence (figure 5). The sample shows the highest switching ratio when the same irradiated at a fluence of $1 \times 10^{12}$ ions cm$^{-2}$. The switching ratio of the sample decreased beyond this ion fluence.

Sawa reported that the switching property of the film decreased due to increase of oxygen content of oxide layer in the process of oxidation whereas annealing increases the number of oxygen vacancies resulting good switching behavior [59]. Oxide samples are known to lose oxygen when subjected to SHI irradiation [60] due to
the generation of thermal spike [41, 42] during the passage of energetic ions through the sample. Even small quantities of oxygen vacancies could considerably influence the electrical properties [60] and thus also on the resistive switching. Diffusion of atoms across the interfacial region could also influence the switching characteristics [35]. Since the values of electronic energy loss ($S_{el}$) of 120 MeV Ag ions in NiO and ZnO are ~27.62 and 21.68 keV nm$^{-1}$ respectively as estimated from SRIM simulation program [34] far exceed the reported value of threshold electronic energy losses ($S_{el}^{th}$) (~12 keV nm$^{-1}$) for the occurrence of ion beam induced mixing at interfaces of similar types of systems [48, 49], we therefore believe that the materials melt along the ion path region during the passage of SHI due to the generation of thermal spike [41, 42]. Hence the ion beam induced mixing across the interface of the heterojunction is highly probable due to the high diffusivity ($\sim$10$^{-6}$–10$^{-9}$ m$^2$ s$^{-1}$ [61]) of Ni and Zn ions in the melted region along the ion path across the interface region. The occurrence of highest switching ratio for the sample irradiated at a fluence of 1 × 10$^{12}$ ions cm$^{-2}$ is shown to be due to diffusion of ions across the NiO/ZnO junction and the suppression of switching ratio at the fluence of $\geq$ 3 × 10$^{12}$ ions cm$^{-2}$ could possibly due to the ion irradiation induced disordering in the system [30].

The observation of cross point in RS characteristics as seen for the film irradiated at a fluence of 1 × 10$^{12}$ ions cm$^{-2}$ was also reported for Au/SrTiO$_3$/Ti memory cells by Sun et al[62]. The authors have seen that this cross point in RS behaviour disappeared at and above the second cycle of I-V sweeping. Since, the heterojunction irradiated at a fluence of 1 × 10$^{12}$ ions cm$^{-2}$ showed better switching ratio, we therefore studied cycle (upto 3rd cycle) dependent evolution of the RS behaviour of this heterojunction (figure 6) in order to see the stability of the device with cycle. As indicated from the figure, the value of current improved at the second cycle with the disappearance of cross point due to formation of stable loop [62]. Though the cross point is not there at the 3rd cycle but one sided current loop is evident which could be possibly due to the conversion of Al electrode to AlO$_x$ under the bias sweep [63].

The applicability of possible RS mechanisms like Ohmic or space charge limited conduction (SCLC) ($\log J$ versus $\log V$), Schottky emission ($\log J$ versus $V^{0.5}$), Poole-Frenkel emission ($\log (J/V)$ versus $V^{0.5}$) and Fowler-Nordheim tunneling ($\ln(I/V^2)$ versus $V^{-1}$) [64] to the present study have also been checked. Figure 7 shows the typical $\log J$ versus $\log V$ for the sample irradiated at a fluence of 1 × 10$^{12}$ ions cm$^{-2}$ along with the same for pristine sample as inset. As indicated from the figure, the $\log J$ versus $\log V$ data shows linear relation indicating the mechanism of electrical conduction either to be Ohmic or space charge limited conduction (SCLC). Generally, metal oxides exhibits Ohmic RS conduction at the low voltage regime due to the dominance of thermally generated carrier density over the injected current density [65] or space charge present in the sample [66]. The region showing Ohmic RS conduction is evident from the slope of $\log J$ versus $\log V$ curve around unity ($\sim$1.5) within that region [65]. Beyond that voltage, the curve shows a transition from Ohmic to SCLC with higher values of slope ($\sim$1.5 [64, 67]). The mechanism of SCLC is ascribed to the presence of some traps near the Fermi level of the layer [68]. As the voltage increases, the injected carriers start filling the traps [69] and the magnitude of current increases suddenly [70] once all the traps are filled. This increase in current resulted into higher slope in $\log J$ versus $\log V$.

The $\log J$ versus $\log V$ plot of pristine sample does not show much distinct curve (inset of figure 7) for HRS and LRS states as this sample does not show much switching ratio (figure 5). The pristine sample shows three distinct regionin its HRS and LRS with three different slope. The sample shows Ohmic conduction at low voltage
regime ($\leq 0.4 \text{ V}$) which switches to SCLC at higher voltage and continue to show SCLC upto 5 V. On the other hand, the sample irradiated at a fluence of $1 \times 10^{12} \text{ ions cm}^{-2}$ shows Ohmic conduction at low voltage regime ($\leq 0.7 \text{ V}$) in HRS which switches to SCLC at higher voltage and continue to show SCLC upto 5 V (figure 7). Though the slope of the three regions continuously decreased while coming from 5 to 0 V in LRS but the SCLC mechanism is retained. However, the J–V data does not fit to the other models as stated above.

4. Conclusion

We report the effect of 120 MeV Ag ions on the structural and electrical properties of NiO/ZnO heterostructure. Ion irradiation at a fluence of $3 \times 10^{11} \text{ ions cm}^{-2}$ led to improvement of crystallinity for NiO layers. Beyond this fluence, the XRD peak intensities of NiO showed monotonic decrease with ion fluence. Raman analysis confirmed that the ZnO phase is not amorphized due to irradiation. The occurrence of highest switching ratio for the sample irradiated at a fluence of $1 \times 10^{12} \text{ ions cm}^{-2}$ is shown to be due to diffusion of ions across the NiO/ZnO junction and the decrease of switching ratio at higher ion fluences is due to ion irradiation induced disordering in the system. The Ohmic conduction in low voltage regime and SCLC at higher voltage regime explain the possible mechanism for electrical conduction. The thermal spike model of ion-matter interaction is shown to be applicable for the observed modifications.

Acknowledgments

We acknowledge Dr V R Reddy, UGC-DAE CSR, Indore for providing GIXRD facility. We thank Mr A Gome and Mr A.K. Rathore of UGC-DAE CSR, Indore for their help in XRD and RAMAN measurements respectively. PKD and PM thank IUAC, New Delhi for providing financial support through project (UFR-58311) to carry out this work. The authors are thankful to the Pelletron group of IUAC, New Delhi, for providing a good quality scanned beam for irradiation.

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