Local Electronic Structure of MgO: Impact of Deposition Time and Ion Irradiation

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Abstract

Present work investigates local electronic structure of MgO films under irradiation of 100 MeV oxygen ions. MgO thin films of different thicknesses are grown using radio frequency sputtering. Near edge X-ray absorption fine structure (NEXAFS), measurements in surface sensitive, total electron yield (TEY) and bulk sensitive, total fluorescence yield (TFY) modes were performed at both O K-edge and Mg K-edges for pristine and irradiated counterpart. TFY measurements at both Mg K-edge and O K-edges for MgO thin films reflect the spectral features associated with hybridization among Mg²⁺ and O²⁻ ions. These spectral features are analogues to that of bulk MgO. TEY mode Mg K and O K-edge NEXAFS spectra exhibit slightly reduced spectral features, which may be due to weakening of Mg-O hybridization at surface or some possible contamination of environmental species. The local electronic structure of these films under irradiation modifies slightly.

Keywords- Irradiation, Local electronic structure, Sputtering, Deposition time.

1. Introduction

Swift heavy ion irradiation is an important tool to modify the characteristics of materials by transferring its energy to the lattice of target materials (Avasthi, 2009; Hofsäss et al., 2011; Singh et al., 2012). Depending upon the amount of energy loss, defects like vacancies or columnar are expected inside the target. Though the effects on the physical characteristics by energetic ion irradiation are investigated by numbers of researchers in the field (Krasheninnikov and Nordlund, 2010; Thomé, 2016; Costantini et al., 2018),

however, the investigation of the effect on local electronic structure are hardly reported. In our opinion, investigations related to local electronic structure are extremely important as these ion-matter interactions are pronounced for inducing changes at local levels (Avasthi, 2009; Krasheninnikov and Nordlund, 2010; Hofsäss et al., 2011; Singh et al., 2012; Thomé, 2016; Costantini et al., 2018). Ion matter interaction in these materials induces thermal spikes which causes modification of lattice disorder (Lazanu et al., 2011; Crespillo et al., 2017; Agulló-López et al., 2016). This may cause modification of local environment around metal ions in materials (Sinha et al., 2021). Thus, techniques, which can detect changes occurring at microscopic scale effectively, are desirable. In most of studies, which discuss about the ion-matter interaction the direct information of changes, occurring at local level are ignored. Thus, present work focuses on the investigation of local electronic structure of irradiated materials. Effect of intermediate annealing on the local electronic structure and dielectric properties of zinc ferrite was investigated using these measurements (Singh et al., 2013). Local electronic structure of polycrystalline CdTe is sensitive to CdCl₂ treatment and air exposure (Berg et al., 2018). Local electronic structure of MgO thin films is also sensitive to thermal annealing (Singh et al., 2018a), deposition time (Singh et al., 2017), prolonged annealing (Singh et al., 2018b) and ion implantation (Singh et al., 2018c; Singh et al., 2021). These studies envisage that near edge X-ray absorption fine structure (NEXAFS) spectroscopy is an effective technique to investigate any change related to local electronic structure in various materials including MgO, which is a well-known insulating oxide for its defect dependent characteristics (Kumar et al., 2021; Bhakta et al., 2023; Bishnoi et al., 2024).

Though, MgO seems to be a simple system but it's growth faces challenges in thin film form because of its high band-gap (Nourozi et al., 2019). This leads to use of numerous deposition methods for MgO in order to overcome these challenges. Some of these methods are chemical bath deposition (Tezal et al., 2022), chemical spray pyrolysis technology, electron beam evaporation technique, radio frequency (RF) magnetron sputtering, chemical vapor deposition (Al-Rikabi et al., 2003) and pulsed laser deposition (Ismail et al., 2019). Among these deposition methods, RF sputtering is considered suitable and effective approach for commercial large-scale production, easy control of thin film growth and stoichiometry (Chen et al., 2015; Garg et al., 2024). Thus, present work investigates the local electronic structure of RF sputtering grown MgO thin films under swift heavy ion irradiation using near edge X-ray absorption fine structure (NEXAFS) spectroscopy.

2. Experiments

MgO films were deposited using RF sputtering method on Si (100) substrate for various deposition durations i.e. 25, 49, 81, 100 and 144 min. The sputtering power and substrate temperature was 40 W and 350°C, respectively. Deposited films were annealed at 700°C for 1 h (Singh et al., 2017; Singh et al., 2018a). Each film is cut into four pieces and three pieces were irradiated by 100 MeV O^{7+} using Pelletron accelerator at Inter University Accelerator Center (IUAC), New Delhi. The fluences of irradiation were kept as 1×10^{11} , 1×10^{12} and 1×10^{13} ions/cm².

All the characterization were performed at various beamlines of Pohang Accelerator Laboratory (PAL), South Korea (Singh et al., 2017). Pristine and irradiated films were characterized using X-ray diffraction (XRD) performed at 5A beamline. X-ray energy was fixed at 17 keV for these measurements. X-ray reflectivity (XRR) measurements was performed at 1D KIST beamline. NEXAFS were carried out at 10D XAS KIST beamline in total electron yield (TEY) and total fluorescence mode (TFY) mode (Kasrai et al., 1996; Singh et al., 2018d). Schematic of these modes of measurements are shown in **Figure 1**.

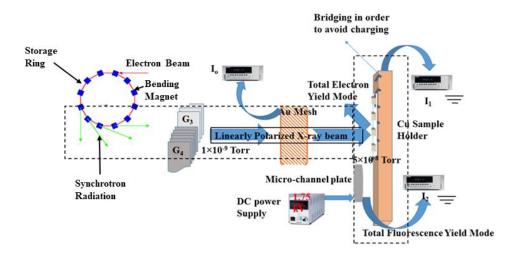


Figure 1. Procedure for measuring NEXAFS spectra in TEY and TFY mode (Reprinted with permission from Singh et al., 2018d).

3. Results and Discussion

Thickness of the films having deposition times of 25, 49, 81, 100 and 144 min are 12, 28, 32, 40 and 52 nm respectively. A schematic of these films based on the NEXAFS and XRR studies in our previous work is provided in **Figure 2** (Singh et al., 2017).

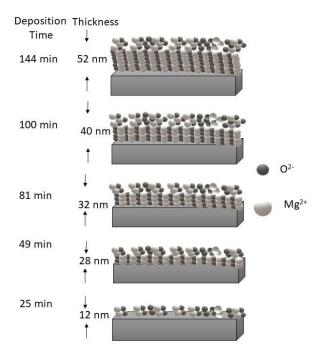


Figure 2. Thickness of MgO films for various deposition times as estimated from X-ray reflectivity measurements (Singh et al., 2017).

3.1 Mg K-edge Spectra for Irradiated Film

Figure 3(a) shows the TEY and TFY mode Mg K-edge spectra of pristine and irradiated counterparts of MgO film having deposition times of 25 min. Deconvolution shows the presence of minor feature corresponding to structure A_1 , however, structures A_2 , A_3 , A_4 and A_5 are well defined and distinguishable. With fluence of irradiation, there is no change of these structures revealing no change of local atomic structure of the film with irradiation. Structure A_1 has value of 1303.7 eV for TEY mode; however, this value is around 1303.6eV for TFY mode measurement (**Figure 3(b)**). In both the cases, this value increases linearly and attains values close to 1304.1 eV (**Figure 3(c)**).

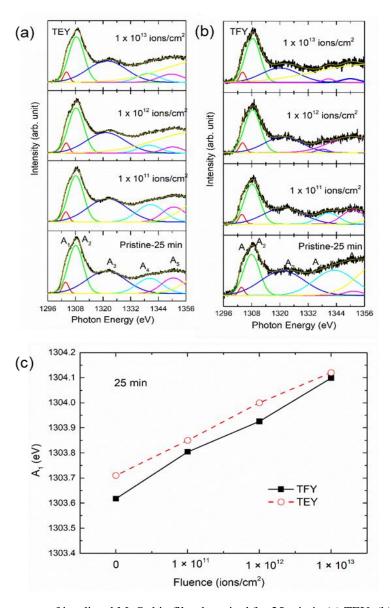


Figure 3. Mg K-edge spectra of irradiated MgO thin film deposited for 25 min in (a) TEY, (b) TFY Mode and (c) Shows variation of spectral feature A_1 with fluence of irradiation.

TEY and TFY mode Mg K-edge spectra for films having deposition time of 49 min are shown in **Figure 4(a)**. In this case, well defined structures A_1 , A_2 , A_3 , A_4 and A_5 are observed in the spectra of all films except the film irradiated at fluence of 1×10^{11} ions/cm². This behavior of these spectra is similar for both the modes. **Figure 4(b)** shows the values of structure A_1 for at different fluences. In case of pristine film, this value is 1304.4 eV for both the modes and shifts towards lower values upto the fluence of 1×10^{12} ions/cm². The value of A_1 increases thereafter (**Figure 4(c)**). For deposition time of 81 min, splitting between A_1 and A_2 is not influenced by the fluence of irradiation as is the case for deposition time of 49 min (**Figure 5(a)**). In this case, structures A_1 , A_2 , A_3 , A_4 and A_5 are well defined and distinguishable in the spectra of pristine and irradiated when measured in TEY and TFY mode (**Figure 5(b)**). In this case, behavior of A_1 with fluence of irradiation is opposite to that observed for deposition time of 49 min (**Figure 5(c)**).

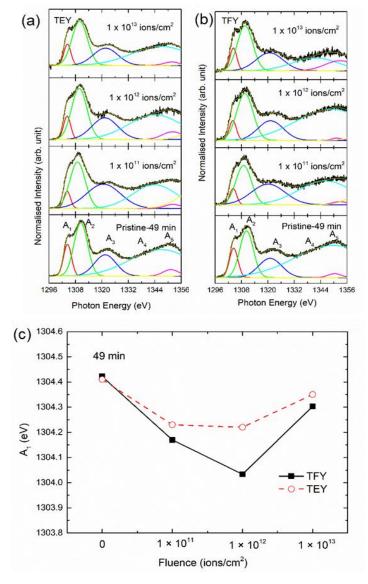


Figure 4. Mg K-edge Spectra of irradiated MgO thin film deposited for 49 min in (a) TEY, (b) TFY Mode and (c) Shows variation of spectral feature A₁ with fluence of irradiation.

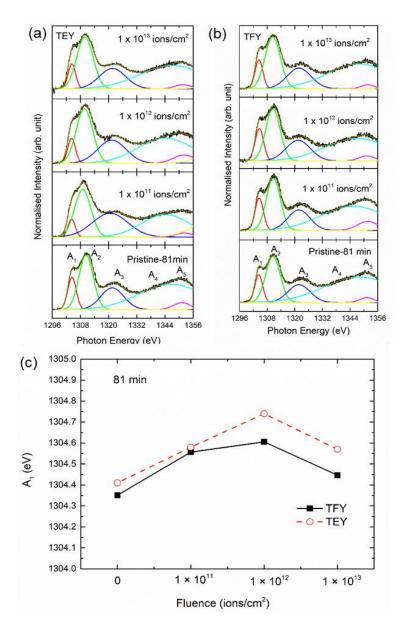


Figure 5. Mg K-edge Spectra of irradiated MgO thin film deposited for 81 min in (a) TEY, (b) TFY Mode and (c) Shows variation of spectral feature A₁ with fluence of irradiation.

Behavior of structures A_1 , A_2 , A_3 , A_4 and A_5 appear in Mg K-edge spectra of pristine and irradiated counterparts of films having deposition times of 100 min (**Figure 6(a)** & **6(b)**) is analogues to that of 81 min deposition duration. Moreover, variation of structure A_1 with fluence of irradiation is similar to that of 81 min. In this case, too A_1 observed in TEY mode is slightly higher than that of TFY mode values (**Figure 6(c)**).

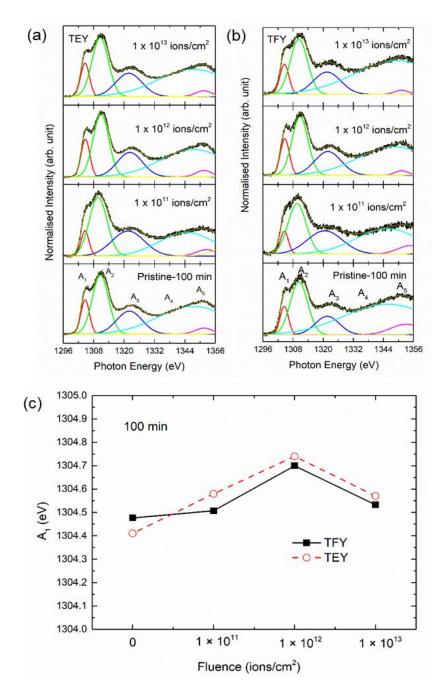


Figure 6. Mg K-edge spectra of irradiated MgO thin film deposited for 100 min in (a) TEY, (b) TFY Mode and (c) shows variation of spectral feature A₁ with fluence of irradiation.

Figure 7 shows the distinguishable and well-defined structural features A_1 , A_2 , A_3 , A_4 and A_4 in TEY (**Figure 7(a)**) and TFY (**Figure 7(b)**) mode Mg K-edge spectra of pristine and irradiation counterpart for deposition duration of 144 min. A_1 structure has almost similar values for pristine and irradiated counterpart (fluence 1×10^{11} ions/cm²) for both mode (**Figure 7(c)**). This value slightly increases for fluence of 1×10^{12} ions/cm² and remains almost same on further increasing fluence in both modes. Thus, behavior of Mg K-

edge spectra in influenced by both fluence of irradiation and deposition time. These effects are further discussed by estimating $\delta A_1 A_2$ for these samples.

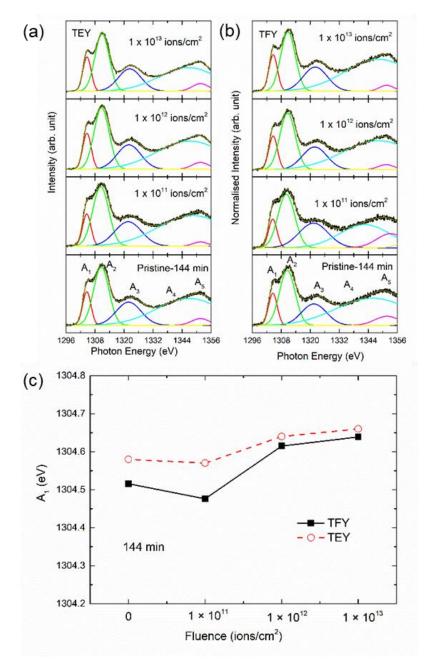


Figure 7. Mg K-edge spectra of irradiated MgO thin film deposited for 144 min in (a) TEY, (b) TFY Mode and (c) shows variation of spectral feature A₁ with fluence of irradiation.

3.1.1 Behavior of $\delta A_1 A_2$ with Fluence of Irradiation

Figure 8 shows the variation of $\delta A_1 A_2$ with fluence of irradiation. In both TEY and TFY mode, $\delta A_1 A_2$ values are almost same within experimental errors. In case of 25 min, $\delta A_1 A_2$ is ~4.2, which is less than that

of perfect MgO crystals revealing that these films are disordered and there is no improvement with irradiation. In case of higher deposition time, value of $\delta A_1 A_2$ is around 5.5 eV, which close to that of standard MgO system showing that films are ordered at these deposition durations i.e. 49 min, 81 min, 100 min and 144 min. The one common behavior observed under irradiation is that $\delta A_1 A_2$ values reduce for fluence of 1×10^{11} ions/cm² compared to pristine counterpart and increases on further increasing fluence. For deposition duration of 49, 81 and 100 min, the value of $\delta A_1 A_2$ appears to decrease slightly at fluence of 1×10^{13} ions/cm². Thus, these films for these deposition durations appear to exhibit fluence dependent oscillatory behavior. However, in case of deposition duration of 144 min, $\delta A_1 A_2$ value increases further at this fluence.

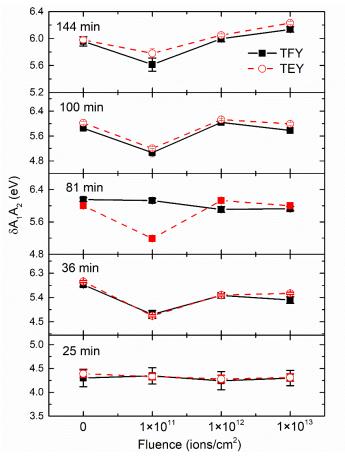


Figure 8. Variation of $\delta A_1 A_2$ with fluence of irradiation.

3.1.2 Thickness Dependent Behavior of $\delta A_1 A_2$ under Different Fluence Irradiation

As thickness dependent behavior is characteristics of thin films, hence, variation of $\delta A_1 A_2$ is also investigated as deposition times (**Figure 9**). In case of pristine films, $\delta A_1 A_2$ values increase exponentially as a function of deposition times. At 25 min, $\delta A_1 A_2$ values are in the range of 4.2 eV, however, these values are approximately 5.5 eV for rest of the deposition times. Under the fluence of 1×10^{11} ions/cm², behavior of $\delta A_1 A_2$ is linear and $\delta A_1 A_2$ values are in the range of 4.2 eV. It means that this fluence causes disorder in the films where crystalline order is already achieved. However, at fluence of 1×10^{12} and 1×10^{13} ions/cm², this variation is again exponential. This may be due to irradiation-induced crystallization in these films.

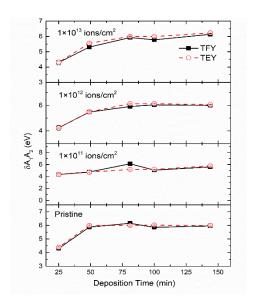


Figure 9. Variation of $\delta A_1 A_2$ with deposition times for different fluences.

3.2 O K-edge Spectra for Irradiated Film

Thus, these studies show that local electronic structure shows modification with fluence of irradiation as well as deposition time. Both the films exhibit spectral features B_1 , B_2 , B_3 , B_4 and B_5 centered at 536, 541, 548, 552 and 559 eV behavior of structures B_1 , B_2 , B_3 , B_4 and B_5 appear in O K-edge spectra of pristine and irradiated counterparts of films having deposition times of 81 and 100 min (**Figure 10**). Though shape of spectral feature remains almost same for pristine and irradiated counterpart but significant changes are observed in the pre-edge structure denoted by B_0 (Luches et al., 2004).

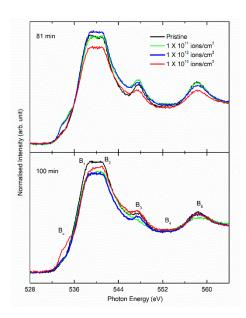


Figure 10. O K-edge spectra for thin film having deposition times of 81 and 100 min under different fluences of irradiation.

3.3 XRD Studies for Irradiated Films

Both the Mg and O K-edge measurements exhibit significant changes on local structural order with deposition time and fluence of irradiation. Thus, XRD studies are performed for pristine and irradiated counterparts of films having deposition times of 100 and 144 min. These films are selected, as films grown below this deposition times is amorphous in nature. XRD peak position corresponding to various planes at X-ray energy of 17 keV is shown in **Table 1** (Balakrishnan et al., 2020). **Figure 11** shows the XRD patterns of MgO thin films for deposition duration of 100 min at different fluences. XRD peak corresponding to (200) plane of rocksalt phase of MgO (**Table 1**) is maximum for fluence of 1×10^{12} ions/cm² and intensity reduces on further increasing fluence.

Planes	JCPDS No. 87-0653 (λ=1.54148Å)	X-ray energy of 17 keV (λ=0.72 Å)	
(111)	36.96°	16.50°	
(002)	42.98°	18.79°	
(202)	62.36°	24.74°	
(113)	74.71°	27.10°	

Table 1. XRD peak positions of various planes at X-ray energy of 17 keV.

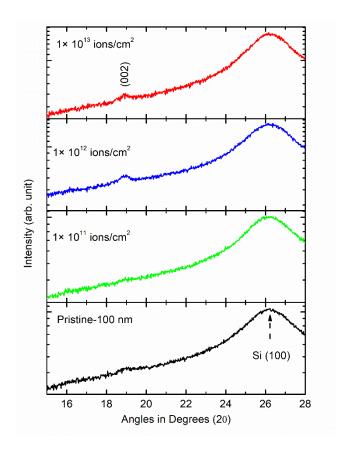


Figure 11. XRD patterns of MgO thin films having deposition time of 100 min irradiated at fluences of 1×10^{11} , 1×10^{12} and 1×10^{13} ions/cm² along with pristine counterparts.

Figure 12 shows the XRD patterns of MgO thin films for deposition duration of 144 min at different fluences. XRD peak corresponding to (200) plane of rock salt phase of MgO is maximum for fluence of

 1×10^{12} ions/cm² and intensity reduces on further increasing fluence. However, another peak corresponding to (113) plane of this phase also appears in the XRD pattern at fluence of 1×10^{12} ions/cm². Also, intensity of this plane is maximum at 1×10^{13} ions/cm² showing the changes in textured of the grown film (Mallick et al., 2008; Sharma et al., 2009).

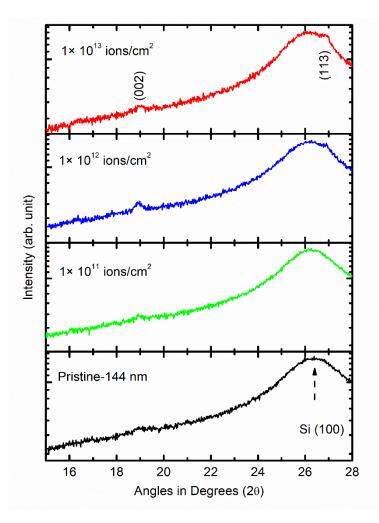


Figure 12. XRD patterns of MgO thin films having deposition times of 144 min irradiated at fluence of 1×10^{11} , 1×10^{12} and 1×10^{13} ions/cm² along with pristine counterparts.

Thus, all films exhibit irradiation induced changes in local atomic structure and crystalline order. These changes are associated with the energy deposited in these films induced by electronic excitations (**Table 2**). Dominance of electronic excitation can be seen from the S_e/S_n ratio, which is 10^3 (Dhyani et al., 2024).

Table 2. Parameters estimated from stopping and range of ions in the matter (SRIM) calculation (Ziegler et al., 2010).

Energy (MeV)	Ion	Stopping values (keV/µm)		Projected range	S_e/S_n
		Electronic (S _e)	Nuclear (S _n)	(µm)	
100	O ⁷⁺	5.1×10^{2}	2.8 ×10 ⁻¹	133	~103

4. Conclusions

Thus, we have successfully explored the local electronic structure of MgO thin films under heavy ion irradiation and investigation is concluded with following points.

- Local electronic structure of these films is sensitive to the deposition times.
- Mg K-edge spectra are able to provoke about the crystalline disorder/order.
- Irradiation induced effects are influenced by the fluence of irradiation.
- Irradiation induced effects are also sensitive to the thickness of the films.

Conflict of Interest

The authors confirm that there is no conflict of interest to declare for this publication.

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