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M. Nabil

mohammed_diab35@yahoo.com

Benha University

S. S. Fouad

Ain Shams University

G. L. Katona

University of Debrecen

E. Baradács

University of Debrecen

Janos J. Tomán

University of Debrecen

G. Vecsei

University of Debrecen

Z. Erdélyi

University of Debrecen

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Morphological Evolution and Optical Modeling of ZnO/Mn/ZnO Multilayer Thin films via Mn Interlayer Engineering

M.Nabil[a*], S.S.Fouad[b*], G.L.Katona[c], E. Baradács[c,d] , Janos J. Tomán[c], G. Vecsei[c], Z.Erdélyi[c]

[a]*Department of Basic Engineering Sciences, Faculty of Engineering (Shoubra), Benha University, Benha, Egypt.*
[b]*Department of Physics, Faculty of Education, Ain Shams University, Cairo11566, Egypt.*
[c]*Department of Solid-State Physics, Faculty of Science and Technology, University of Debrecen P.O. Box400, Debrecen-4002, Hungary.*
[d]*Department of Environmental Physics, University of Debrecen, Poroszlay u.6, Debrecen-4026, Hungary.*

Corresponding author: - ***M.Nabil*** ***E.mail :- mohammed_diab35@yahoo.com***
S.S.Fouad ***E.mail :- profsu95@gmail.com***

Abstract.

The influence of Mn on the structural and optical characterization of ZnO/Mn/ZnO multilayer thin films has been systematically investigated. Thin film samples with Mn layer thickness between 0-80nm were prepared by using a combination of atomic layer deposition (ALD) for ZnO, and direct current magnetron sputtering (DC) for Mn interlayer. The addition of Mn between the ZnO layers alters the electronic structure and defect distribution of the system, leading to observable changes in optical response. The thickness, morphology and optical response of the thin films under study were examined via X-Ray reflectivity (XRR), focused on the ion beam (FIB) cross-sectioning, scanning electron microscope (SEM). XRR analysis revealed an increase in surface roughness of the films and Mn layer density with higher Mn thickness, indicating improved Mn layer compactness, while FIB and SEM analysis confirmed the thickness and showed relatively sharp interfaces. From the measured transmittance and reflectance (T& R) spectra, a decrease in the refractive index *n* and the valence and surface electron energy loss functions (*VELF* and *SELF*), while an increase in the extinction coefficient *k,* dielectric constants (ε_1 and ε_2) ,and optical and electrical conductivity *(σ_{opt})* and *(σ_{elec})*), highlighting the tunability of the optical properties with *Mn* interlayer thickness. The comprehensive analysis of the optical parameters derived confirms that controlled *Mn* interlayer provides an effective strategy for tuning the electronic and optical properties of ZnO/Mn/ZnO multilayers, making them promising candidates for advanced optoelectronic and photonic applications.

Keywords: *ZnO/Mn/ZnO multilayer, Transition metal, Dielectric function, Transport properties.*

1. Introduction

ZnO-based multilayer structure has attracted significant attention due to their remarkable optical transparency, wide band gap, and versatility in optoelectronic applications **[1-5].** The incorporation of transition metal interlayers has emerged as an effective strategy to tailor the structural and optical behavior of ZnO-based multilayer systems **[6-10]**.

Among various transition metals, manganese (Mn) plays a crucial role in modifying the morphology and optical response of ZnO due to its multiple oxidation states and strong interaction with ZnO layers. The variation of Mn layer thickness within ZnO/Mn/ZnO system plays a decisive role in controlling grain size, surface roughness and defect density which directly influences the optical constants and band gap energy **[11-14]**.

Therefore, doping ZnO with Mn provides a strategy for engineering its optical characteristics without compromising its structural integrity. Understanding the correlation between Mn content (layer thickness), morphological evolution and optical response is essential for optimizing these structures for advanced photonics, light-harvesting, sensing devices, and optoelectronic applications. The optical constants, refractive index (n) and extinction coefficient (k), extracted from transmittance (T) and reflectance (R) measurements, were used to determine the complex dielectric function of the prepared films **[15-18]**. The dielectric response provides direct insight into changes in the electronic structure induced by dopant incorporation **[19,20].** From the dielectric function, the optical and electrical conductivity, the valence and surface electron energy loss functions (VELF and SELF) were evaluated, allowing their sensitivity to Mn incorporation to be accurately assessed **[21-22]**. In addition, high quality, uniform and precise Mn incorporation into the ZnO/Mn/ZnO multilayer structure, can be achieved through optimized atomic layer deposition (ALD) and DC magnetron sputtering **[23-30]**.

Despite the significant progress achieved in ZnO-based multilayer thin films, a systematic understanding of how Mn interlayer thickness influences the structural characteristics and the resulting optical constants and derived optical parameters remain limited. In particular, comprehensive studies that correlate structural evaluation with dielectric, optical conductivity, energy-loss functions and related optical parameters in ZnO/Mn/ZnO multilayer structures are still scarce. Therefore, the present work systematically investigates ZnO/Mn/ZnO multilayer thin films with different Mn interlayer thickness were deposited by atomic layer deposition and DC magnetron sputtering. Their structural ,morphological, and optical properties were systematically investigated, and the *n, k* dielectric constants ε_1 and ε_2 and consequently optical and electrical conductivity (σ_{opt}) and (σ_{elec}) and (VELF and SELF) were evaluated to provide a deeper understanding of the effect of Mn interlayer thickness on the optical behavior of the multilayer system.

The precise control over layer thickness enables a clear correlation between Mn addition and the observed modifications in *n, k* dielectric constants ε_1 and ε_2 and consequently optical and electrical conductivity (σ_{opt}) and (σ_{elec}) and (VELF and SELF) behavior.

2. Experimental procedure.

We have prepared glass/ZnO(80nm)/Mn (X nm)/ZnO(80nm) samples with X=0, 20, 40, 60, 80 nm. The samples were prepared by atomic layer deposition, ALD (Beneq TFS-200) and by DC magnetron sputtering. First, 80 nm ZnO was grown on the glass substrate by ALD. Subsequently, the sample was removed from ALD and loaded into the DC magnetron sputtering system to prepare the Mn layer (except for the sample without Mn layer). Afterwards the samples were reloaded into ALD and another 80 nm ZnO layer was deposited on top. Please note that the ZnO layer of the sample without Mn layer was also prepared in two steps. The Mn layer was prepared using Ar plasma with a work pressure of $6 \cdot 10^{-1}$ Pa, the deposition rate was 0.1nm/s. The base pressure of the chamber was $6 \cdot 10^{-5}$ Pa. The ZnO layers were prepared at 200 ºC using water (H_2O) and diethylzinc (DEZ) precursors. The H_2O and DEZ pulses were 300 ms long, while the purge time was 3 s in all cases. In total, 444 cycles were used to grow 80 nm ZnO. Further details can be found in ref. **[30]. Fig.1** illustrates the schematic diagram showing the step-by-step preparation workflow.

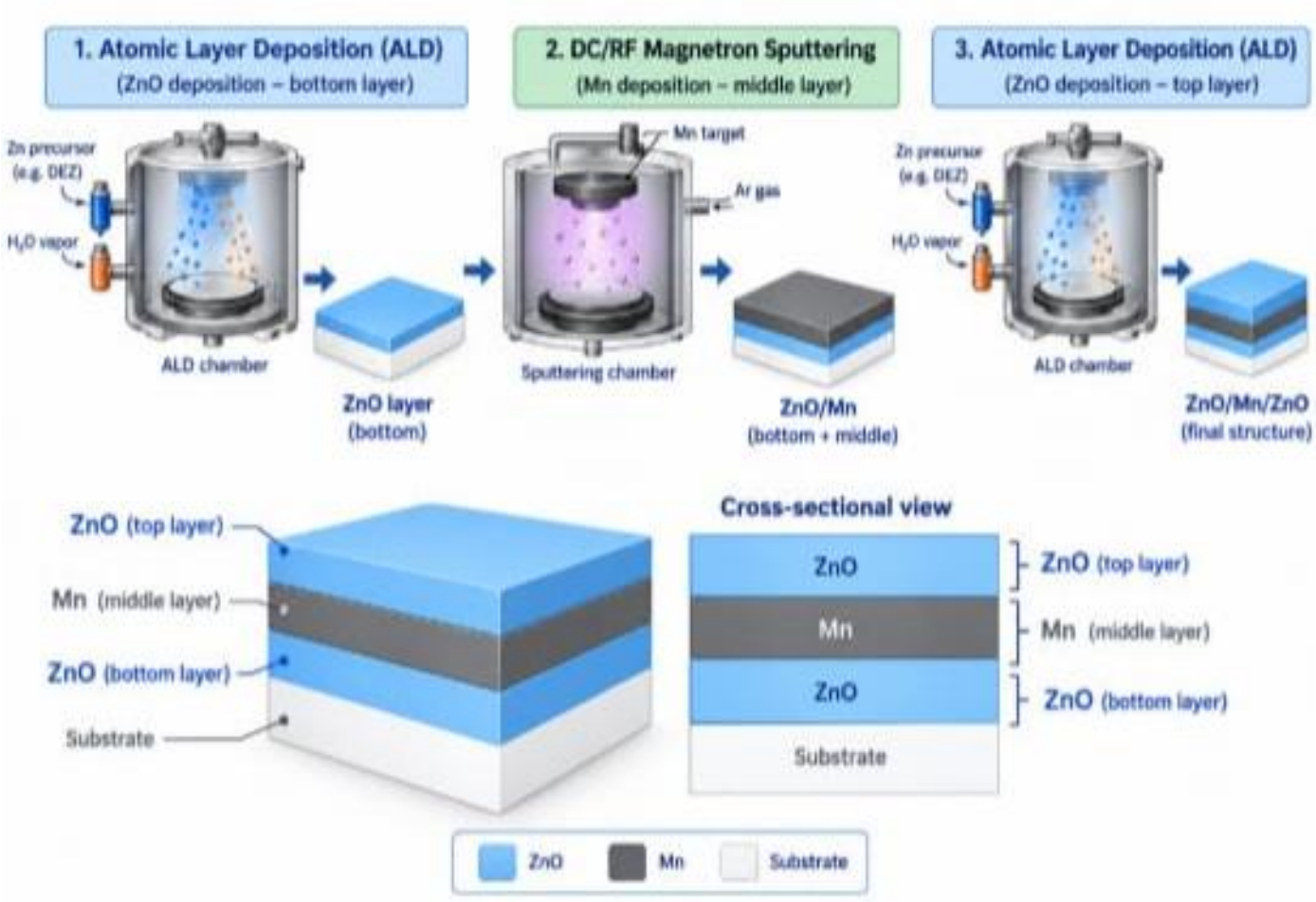


Fig. 1: *Schematic drawing for the steps used for preparing the ZnO/Mn/ZnO with different Mn interlayers.*

Grazing incidence X-ray diffraction (GIXRD) measurements were performed using a Rigaku Smartlab Diffractometer in the range of 25 – 80°. X-ray reflectivity (XRR) measurements were also performed to obtain layer thickness, density and roughness information. Rigaku SmartLab Studio II software was used to evaluate (fit) the XRR and GIXRD results. Focused Ion Beam Scanning Electron Microscopy (FIB-SEM) measurements using a Thermo Scientific Scios 2 DualBeam apparatus was also used to check thickness and continuity of the films. A SemiLab SE-2000 spectroscopic ellipsometer was also used to determine the thickness of the prepared films. The transmittance and reflectance spectra were recorded by using a double beam spectrophotometer (UV-3101PC Shimadzu). The thin film samples in this study will be labelled as ZnO, $ZM_{20}Z$, $ZM_{40}Z$, $ZM_{60}Z$ and $ZM_{80}Z$ corresponding to the nominal glass/ZnO(80nm)/Mn (X nm)/ZnO(80nm) structure with X=0, 20, 40, 60, 80 nm, respectively.

3.Results and discussion.

3.1 Morphological Evaluation in ZnO induced by Mn Addition Via XRR, FIB and SEM.

The GIXRD patterns were recorded to determine the crystallinity of the samples, providing insight into the effect of the Mn interlayer on the film structure. **Fig.2** presents the XRD pattern of the ZnO/Mn/ZnO multilayer films and pure ZnO.

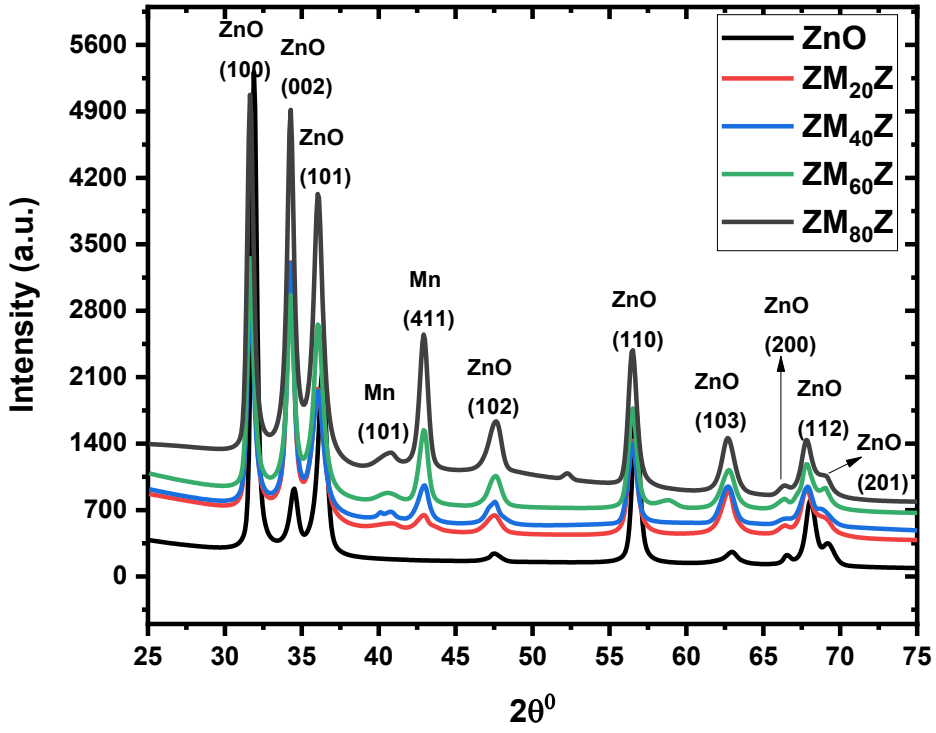


Fig.2 *The XRD patterns of the five different samples of pure ZnO & ZnO/Mn/ZnO system, marking the Mn diffraction peaks.*

Additional diffraction peaks appearing between 2θ = 45°-50° and 60°-65°, are not characteristic in pure ZnO wurtzite phase, and indicate the contribution of Mn interlayer. XRR curves were obtained to provide information about the layer thickness, surface/interface roughness and density of each layer in the ZnO/Mn/ZnO structure compared to that of pure ZnO, giving insight into the influence of Mn interlayer. **Fig.3** shows the obtained reflectivity curves together with their fitness. The fitting parameters are summarized in **Table.1**.

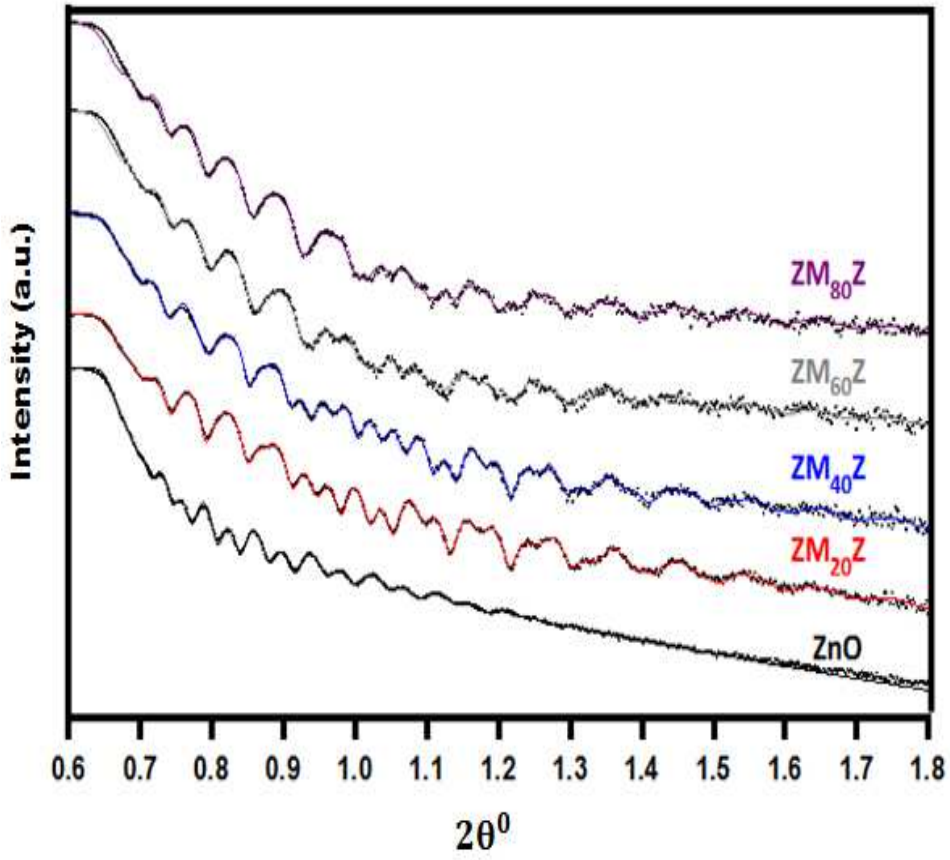


Fig.3 *Intensity curves of ZnO/Mn/ZnO multilayers with different thicknesses of Mn interlayers*

The results reveal that the Mn interlayer significantly influences the structural parameters. It can be seen that increasing the thickness of the Mn interlayer results in increasing Mn layer density.

Table.1 *summarizes the X-ray reflectivity (XRR) results including the layer thickness, surface roughness, and film density of the prepared structures in comparison with pure ZnO.*

Parameters / Samples		Thickness [nm]	Total film thickness [nm]	Roughness [nm]	Density [gm/Cm^3]
ZnO Pure		**159.5**	**159.5**	**3.6±0.5**	**5.4±0.1**
$ZM_{20}Z$	**ZnO top**	**79**	**181**	**3.0±0.5**	**5.6±0.1**
	Mn	**21**		**3.4±0.5**	**6.0±0.1**
	ZnO bottom	**81**		**2.3±0.5**	**5.7±0.1**
$ZM_{40}Z$	**ZnO top**	**82**	**199**	**3.5±0.5**	**5.7±0.1**
	Mn	**36**		**3.2±0.5**	**6.4±0.1**
	ZnO bottom	**81**		**2.1±0.5**	**5.6±0.1**
$ZM_{60}Z$	**ZnO top**	**87**	**224**	**3.8±0.5**	**5.5±0.1**
	Mn	**55.5**		**2.5±0.5**	**6.6±0.1**
	ZnO bottom	**81.5**		**2.0±0.5**	**5.8±0.1**
$ZM_{80}Z$	**ZnO top**	**88.5**	**239.5**	**3.9±0.5**	**5.5±0.1**
	Mn	**70**		**3.0±0.5**	**6.7±0.1**
	ZnO bottom	**81**		**2.0±0.5**	**5.5±0.1**

In order to elucidate how the Mn interlayer modifies the microstructure of the ZnO/Mn/ZnO, cross-sectional SEM images were taken after milling with focused ion beam (FIB). The FIB cross-sectional analysis shown in **Fig.4(a-e)** confirmed the uniformity of the multilayer structure and revealed clear interfaces between ZnO and Mn.

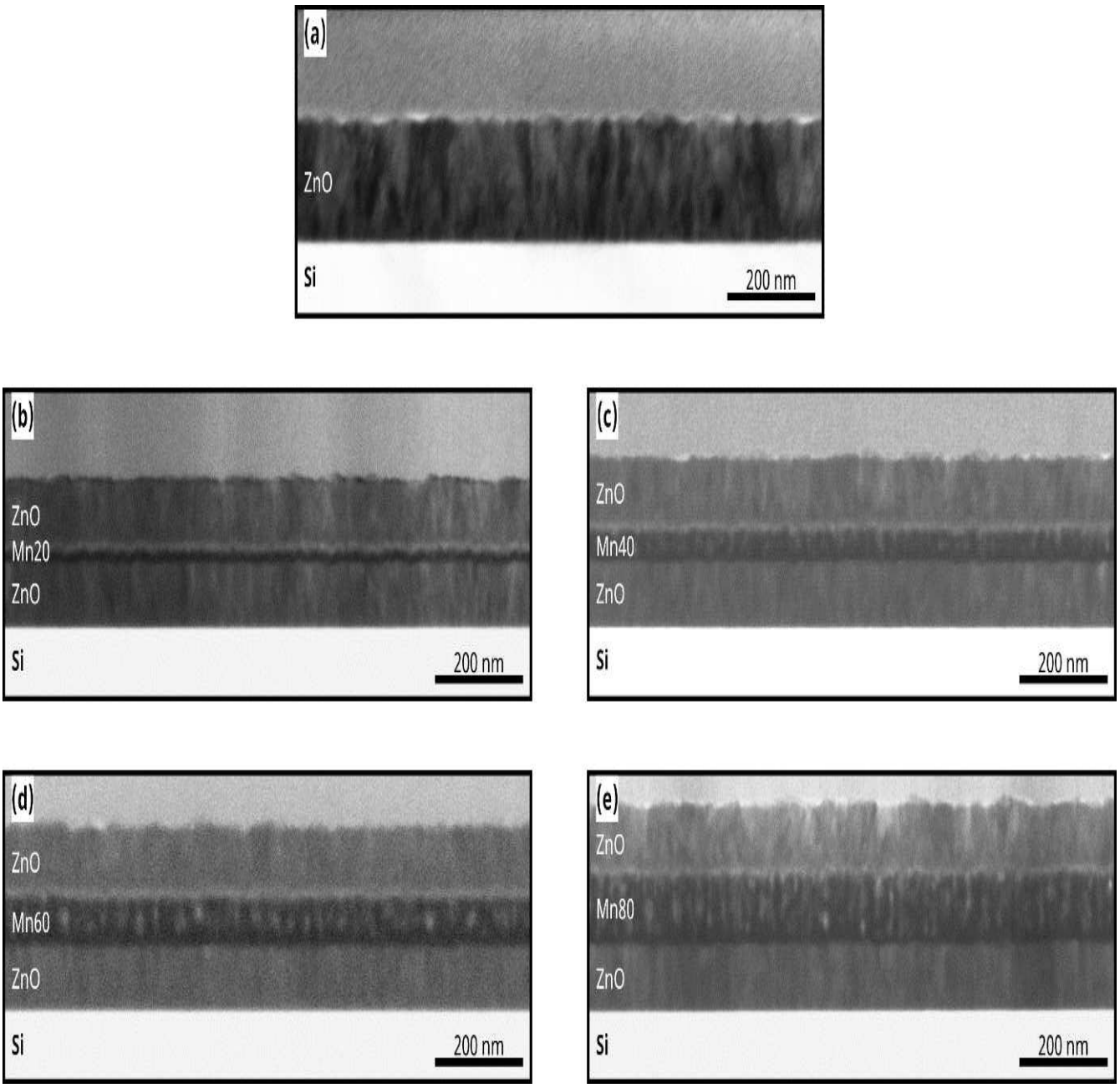


Fig.4(a-e) *FIB micrographs of pure ZnO and the four ZnO/Mn/ZnO samples, showing the difference morphology and layer structure after the addition of Mn layer.* *(a) ZnO, (b) $ZM_{20}Z$, (c) $ZM_{40}Z$, (d) $ZM_{60}Z$, (e) $ZM_{80}Z$.*

From the FIB micrographs of pure ZnO and the four ZnO/Mn/ZnO samples, the images highlight changes in grain structure, interface sharpness and layer uniformity resulting from the addition of Mn layer to the ZnO multilayer architecture. The total thickness and individual layer dimension of the ZnO/Mn/ZnO stack were accurately determined, showing good agreement with the (XRR) results. Top-view SEM images of the sample surface were also taken as seen in **Fig.5(a-e)**.

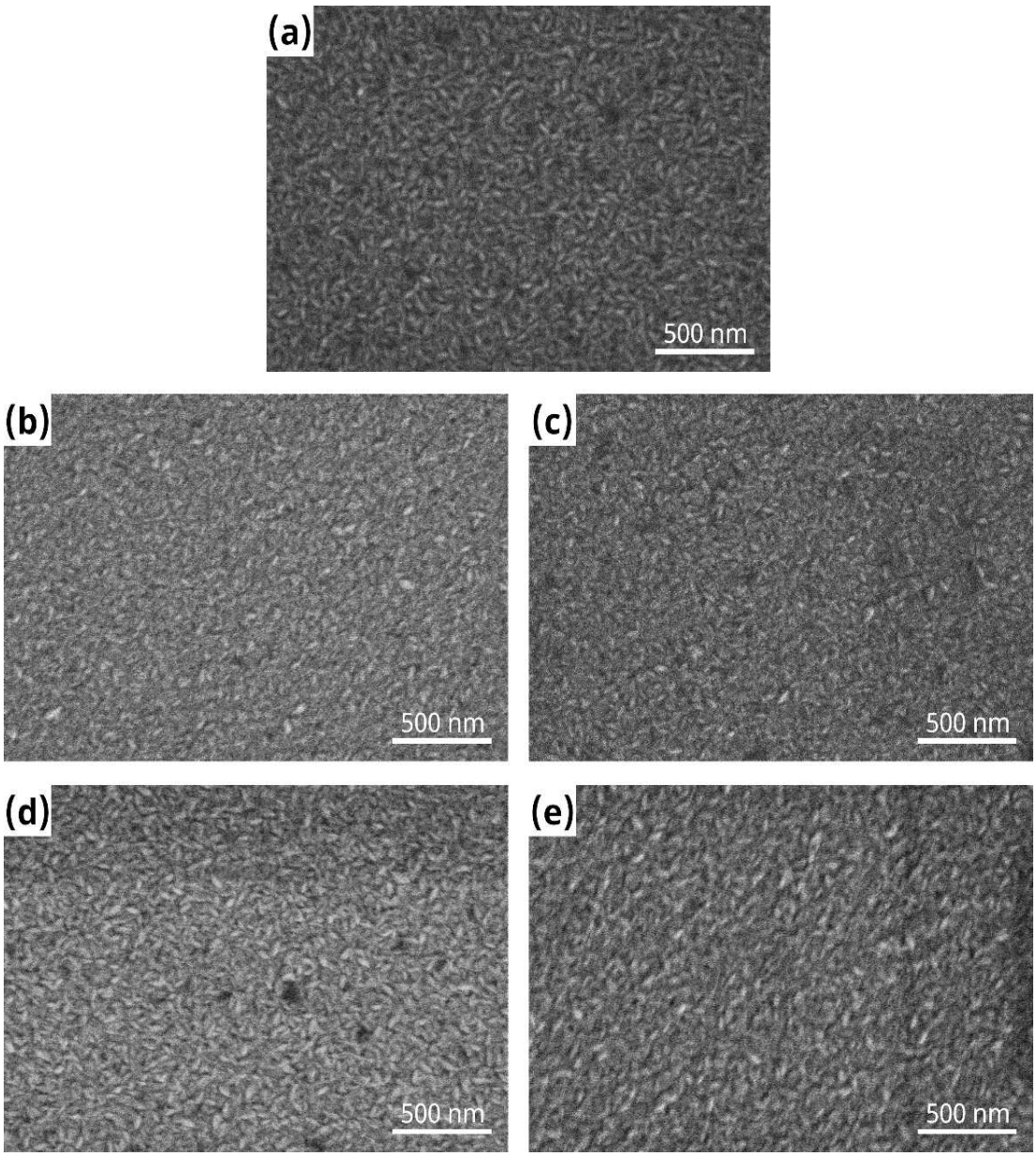


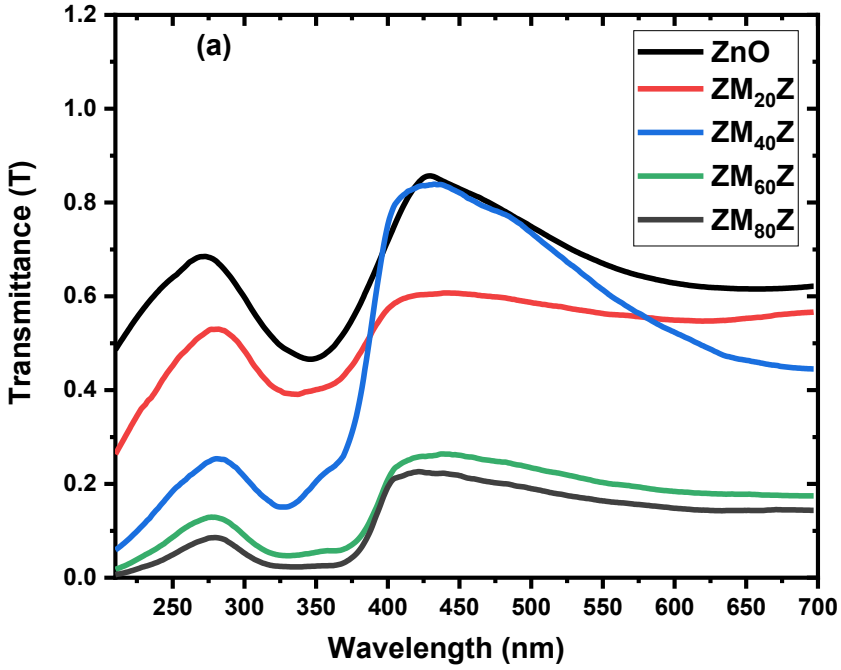
Fig.5 (a-e) *SEM images of pure ZnO and the four ZnO/Mn/ZnO samples, showing the variations in surface morphology after the addition of Mn interlayer. (a) ZnO, (b) $ZM_{20}Z$, (c) $ZM_{40}Z$, (d) $ZM_{60}Z$, (e) $ZM_{80}Z$.*

The SEM micrographs in **Fig.5(a-e)** highlight the structural changes and surface features associated with the addition of Mn layer. The progressive increase in Mn modifies the growth kinetics of the upper ZnO layer by introducing additional nucleation centers and interfacial strain. Thin Mn layers slightly increase surface roughness, whereas thicker Mn layers promote grain coalescence as observed in ZM_{60} *Z* and, $ZM_{80}Z$. The enhanced roughness seen in the SEM images is consistent with XRR results and contributes to the reduced optical transmittance "seen in **Fig.6(a)** "through increased light scattering. The increase in density and roughness with increasing Mn interlayer thickness that the Mn interlayer thickness indicates that the Mn

interlayer modifies the growth behavior of the ZnO layers, leading to a denser structure and a rougher surface, consistent with previous reports on transition -metal- modified ZnO films.

3.2 Correlation of Optical and Dielectric Response of Mn Interlayer in ZnO.

The optical behavior of Mn- ZnO system is strongly influenced by the modification of electronic structure and defect states introduced by the Mn ions **[31]**. The Mn interlayer between the ZnO layers generates additional energy levels within the band gap, increasing free-carrier concentration, and introduces lattice distortion. Therefore, studying the variation of transmittance (T) and reflectance (R) with Mn layer thickness provides important insight into how dopant-induced defects, band structure modifications, and morphological changes influence the optical response of ZnO/Mn/ZnO system. **Fig.6(a&b)** illustrates the effect of Mn interlayer on the (T) and (R) of the ZnO/Mn/ZnO system, showing a clear decrease in (T) and a corresponding increase in (R) as the Mn concentration increases.



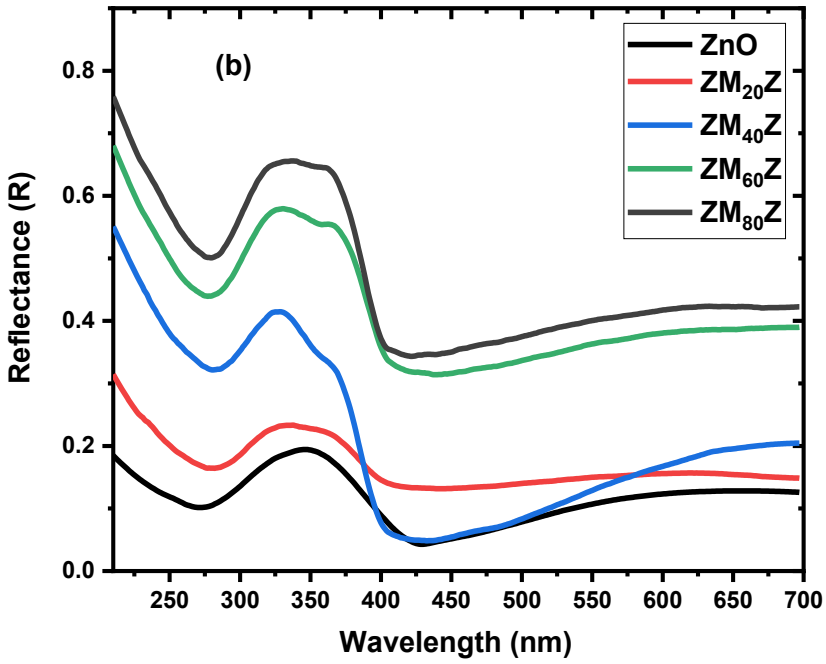


Fig.6 (a&b) *Transmittance and Reflectance for ZnO/Mn/ZnO thin film system.*

The transmittance exhibits a non-monotonic trend with increasing Mn interlayer thickness. It decreases at Mn20nm, reaches a maximum value at Mn40nm, and then decreases again at higher Mn thickness, indicating that the optical response is governed by the competition between absorption, reflection, and the evolution of the multilayer microstructure. The simultaneous increase in (R) and the decrease in (T) by increasing the Mn content is mainly due to higher defect levels and increase in surface roughness as seen in **Table.1**. The refractive index (*n*) and the extinction coefficient (*k),* play an important role in selecting the appropriate materials that are suitable for the fabrication of particular optoelectronic applications. The values of the refractive index *n*, and the extinction index or the absorption index *k* of ZnO, $ZM_{20}Z$, $ZM_{40}Z$, $ZM_{60}Z$ and $ZM_{80}Z$ thin films, can be calculated by using Murmann's exact equations for reflectance (R) and transmittance (T) by using and applying these equations **[32-36]:**

$$R = \frac{A\,e^{\beta} + B\,e^{-\beta} + 2\,C\cos\alpha + 4\,D\sin\alpha}{E\,e^{\beta} + F\,e^{-\beta} + 2\,G\cos\alpha + 4\,H\sin\alpha} \qquad (1)$$

$$T = \frac{16\,n_0 n_g\,(n^2 + K^2)}{E\,e^{\beta} + F\,e^{-\beta} + 2\,G\cos\alpha + 4\,H\sin\alpha} \qquad (2)$$

Where: -

$$A = [(n - n_0)^2 + k^2][(n + n_s)^2 + k^2]$$

$$B = [(n - n_s)^2 + k^2][(n + n_0)^2 + k^2]$$

$$C = (n^2 + k^2)(n_0^2 + n_s^2) - (n^2 + k^2)^2 - n_0^2 n_s^2 - 4n_0 n_s k^2$$

$$D = K(n_s - n_0)(n^2 + k^2 + n_0 n_s)$$

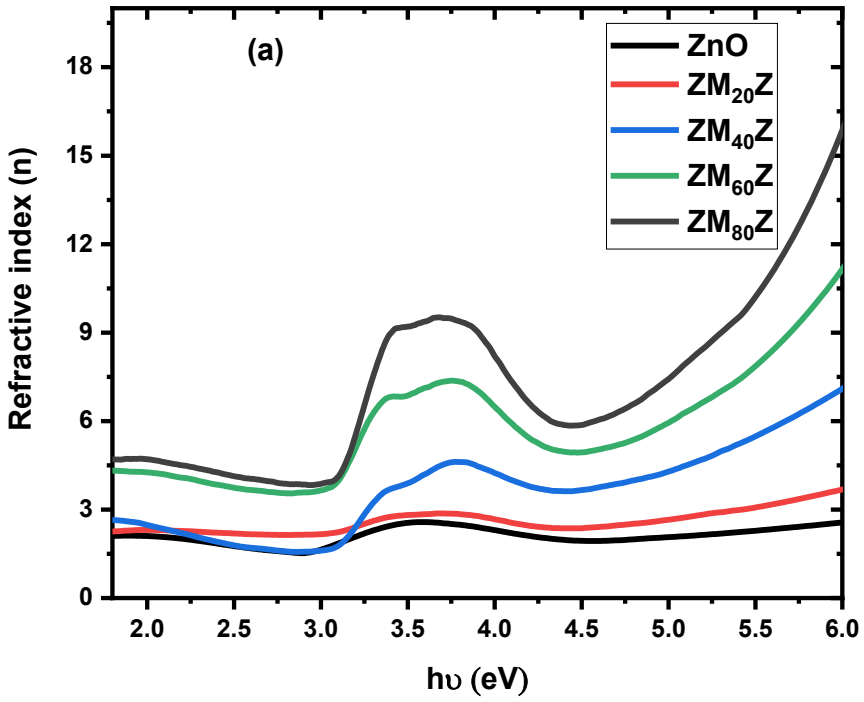
$$E = [(n + n_0)^2 + k^2][(n + n_s)^2 + k^2]$$

$$F = [(n - n_0)^2 + k^2][(n - n_s)^2 + k^2]$$

$$G = (n^2 + k^2)(n_0^2 + n_s^2) - (n^2 + k^2)^2 - n_0^2 n_s^2 - 4n_0 n_s k^2$$

$$H = K(n_s + n_0)(n^2 + k^2 - n_0 n_s)$$

Here, n_s is the refractive index of substrate and n_o is the refractive index of air. Murmann's exact eqs (1) and (2) are solved simultaneously to obtain the accurate values of n and k using a special iterative computer program. **Fig.7(a&b)** presents the variation of n and k to illustrate the effect of Mn interlayer on the ZnO/Mn/ZnO system.



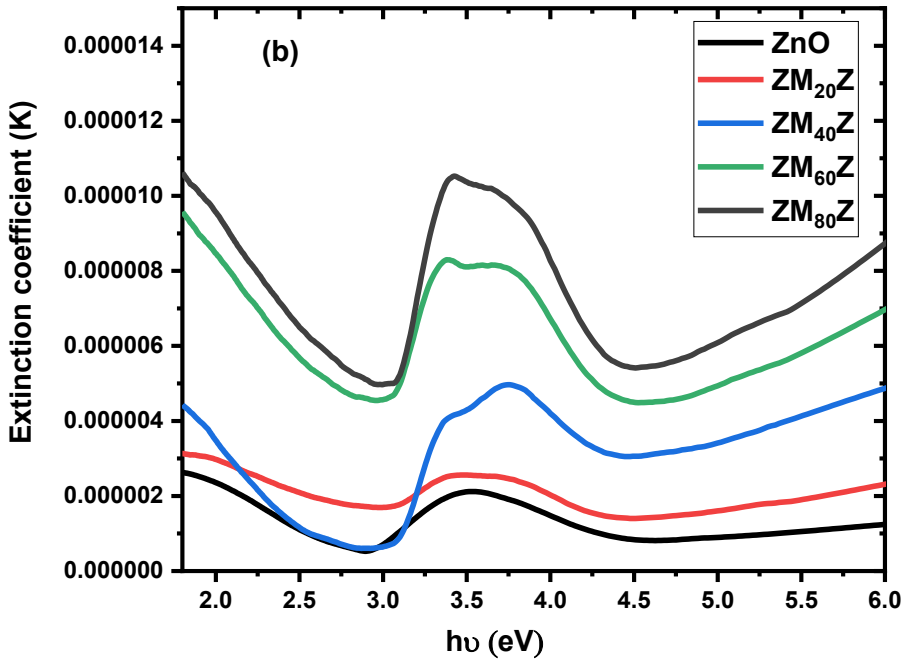


Fig.7 (a&b) *The refractive index and the extinction coefficient of the ZnO/Mn/ZnO system.*

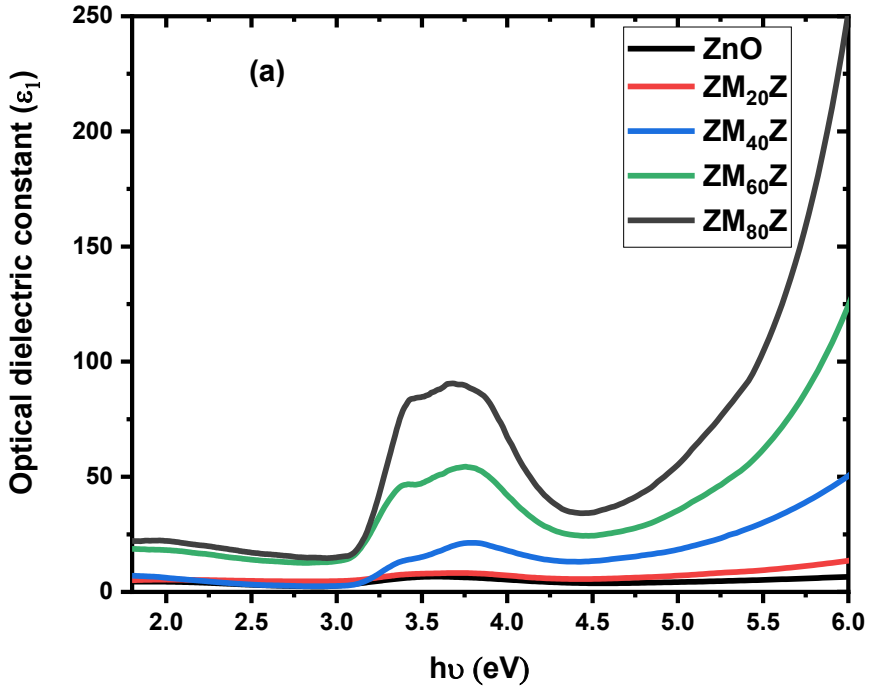
The observed increase in both *n* and *k* correlates with the measured transmittance and reflectance spectra. The reduction in (T) and the increase in (R) with the Mn content can be attributed to the combined effects of enhanced absorption and refractive index, demonstrating that Mn interlayer significantly modifies the optical performance of ZnO. The observed increase in both the refractive index (*n*) and the extinction coefficient (*k*) is consistent with the measured transmittance and reflectance spectra. The decrease in (T) and the increase in (R) with increasing Mn interlayer thickness are attributed to the combined effects of enhanced optical absorption and higher refractive index. The increase in *n* is associated with enhanced electronic polarizability whereas the increase in *k* reflects strong optical absorption induced by Mn interlayer. These results indicate that the Mn interlayer effectively modifies the optical response of the ZnO/Mn/ZnO multilayer system, leading to significant changes in its dielectric properties [**30**]. The noticeable modification in the optical constants' *n* and *k* with the addition of Mn layer between the ZnO layers directly influences the dielectric functions as well as the dielectric response of the material The real part of the dielectric constant (ε_1) associated with stored optical energy increases due to enhanced polarizability, while the imaginary part (ε_2), related to absorption and energy loss. This may also be attributed to the increase in absorption resulting

from defect states induced by Mn as mentioned earlier. The real and imaginary parts of the dielectric constants have been evaluated by the following formulas **[37]**.

$$\varepsilon_1 = n^2 - K^2 \qquad (3)$$

$$\varepsilon_2 = 2nk \qquad (4)$$

Fig.8(a&b) shows the distinction of the dielectric constants of ZnO/Mn/ZnO system with Mn (0, 20 , 40 , 60 and 80 nm) versus photon energy *hv*.



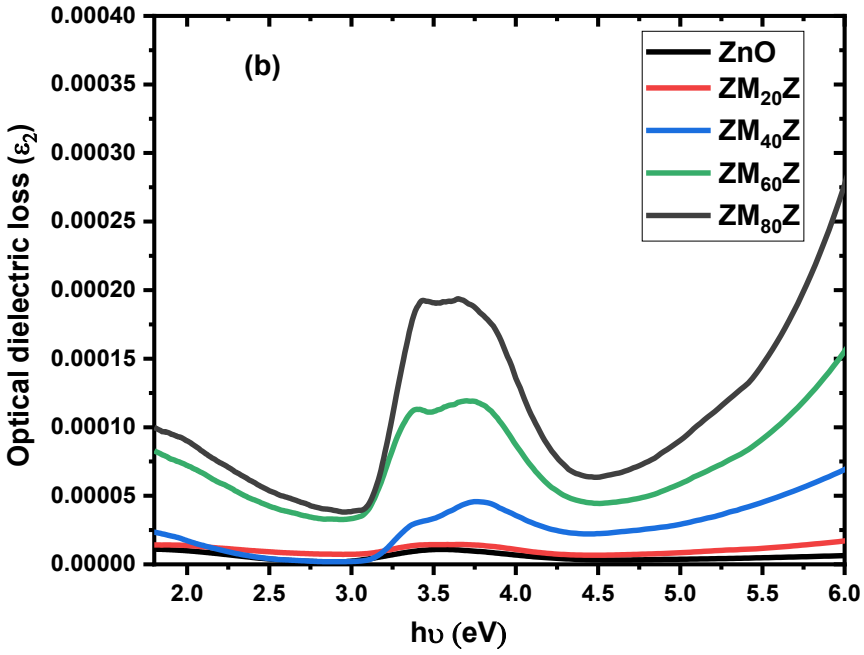


Fig.8 (a&b) *The dielectric constants ($\boldsymbol{\varepsilon_1}$ and $\boldsymbol{\varepsilon_2}$) of ZnO/Mn/ZnO system.*

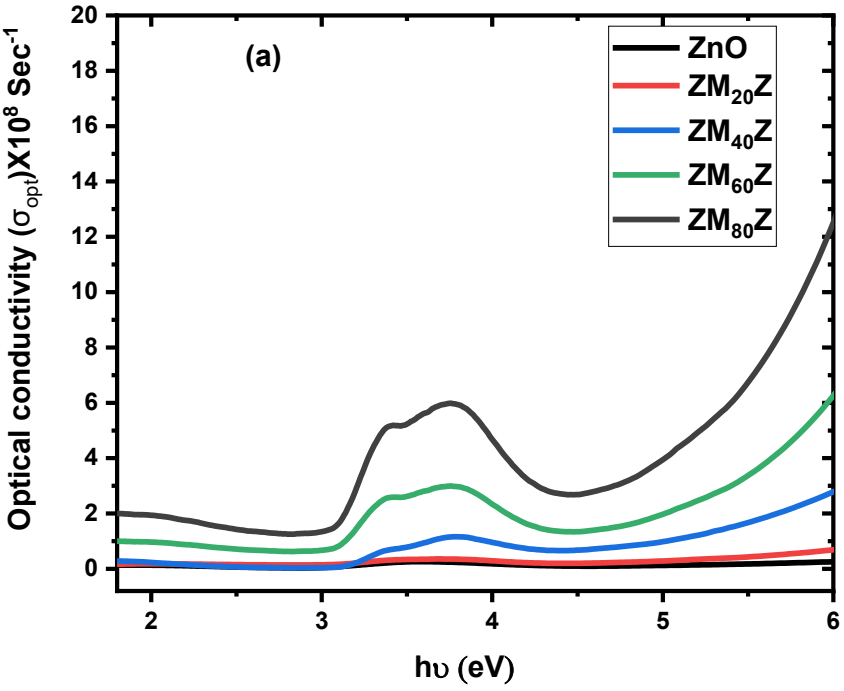
As observed, the optical dielectric constants ($\boldsymbol{\varepsilon_1}$ and $\boldsymbol{\varepsilon_2}$) exhibits a systematic increase with increasing Mn interlayer thickness over the entire investigated energy range. The ZnO sample shows the lowest dielectric response, while $ZM_{80}Z$ displays the highest values. The optical dielectric constant with Mn thickness, with $ZM_{60}Z$ and $ZM_{80}Z$ exhibiting a pronounced enhancement around 3.3-4.0 eV and higher photon energies', indicating strong electronic polarization and interband transition induced by the Mn interlayer. The increase in the refractive index and dielectric constants with increasing Mn interlayer thickness is not only associated with enhanced carrier density and electronic polarizability but also with the combined effects of the increased film density , and microstructural evolution ,,leading to the observed enhancement in the optical constants. This increase can be also attributed to the modification of the electronic structure and increased carrier density induced by Mn interlayers. Similar behavior has been reported for Mn-doped ZnO thin films, where transition metal doping enhances the optical response and dielectric response and dielectric polarization of ZnO **[38,39]**. The above feature originates from the strong optical transitions near the fundamental band edge. The optical conductivity (σ_{opt}), and electrical conductivity (σ_{elec}) are key parameters for understanding the influence of transition-metal on the electronic structure and charge transport mechanism of ZnO-based multilayer system. Optical conductivity provides direct inside into band structure while electrical conductivity reflects changes in carrier concentration. In ZnO/Mn/ZnO structures, Mn

interlayer plays a crucial role in tailoring these properties. Therefore, a combined analysis of (σ_{opt}) and (σ_{elec}) offers comprehensive framework for elucidating the impact of Mn content on the structural- electronic- optical correlation of the system under study. The evaluation of (σ_{opt}) and (σ_{elec}) in terms of dielectric parameters enables a consistent and physically meaningful interpretation of charge transport and optical transitions, particularly in multilayer semiconductor systems such as ZnO/Mn/ZnO. Using experimentally extracted optical constants *n* and *k,* the complex dielectric functions are calculated. The (σ_{opt}) and (σ_{elec}) are then determined from the dielectric response of the material according to the following equations **[40-41]:**

$$\sigma_{opt.} = \omega\, \varepsilon_2\, \epsilon_0 \qquad (5)$$

$$\sigma_{elec.} = \omega\, \varepsilon_1\, \epsilon_0 \qquad (6)$$

The resulting conductivity spectra are presented in **Fig.9 (a&b).**



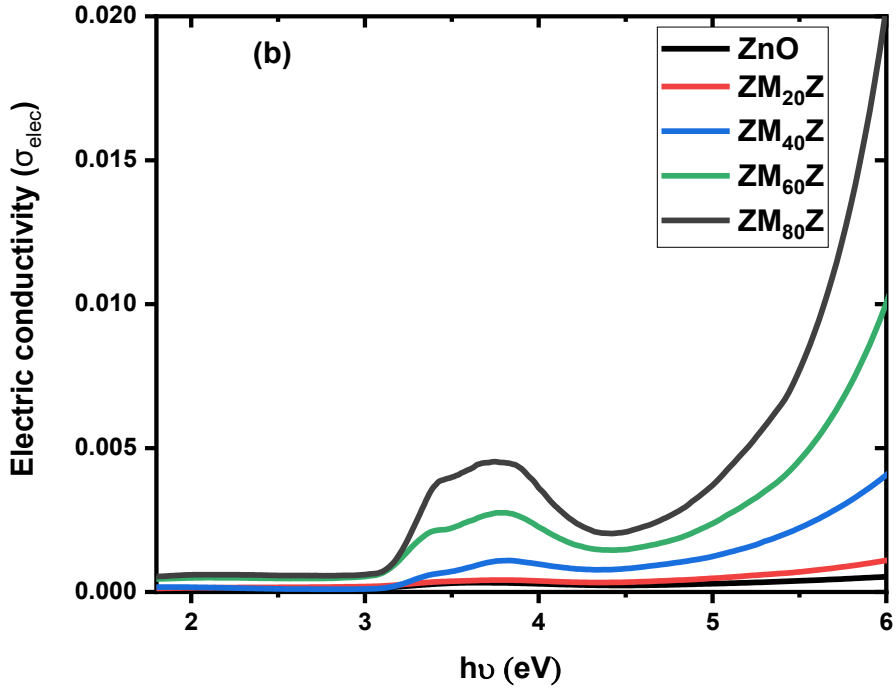


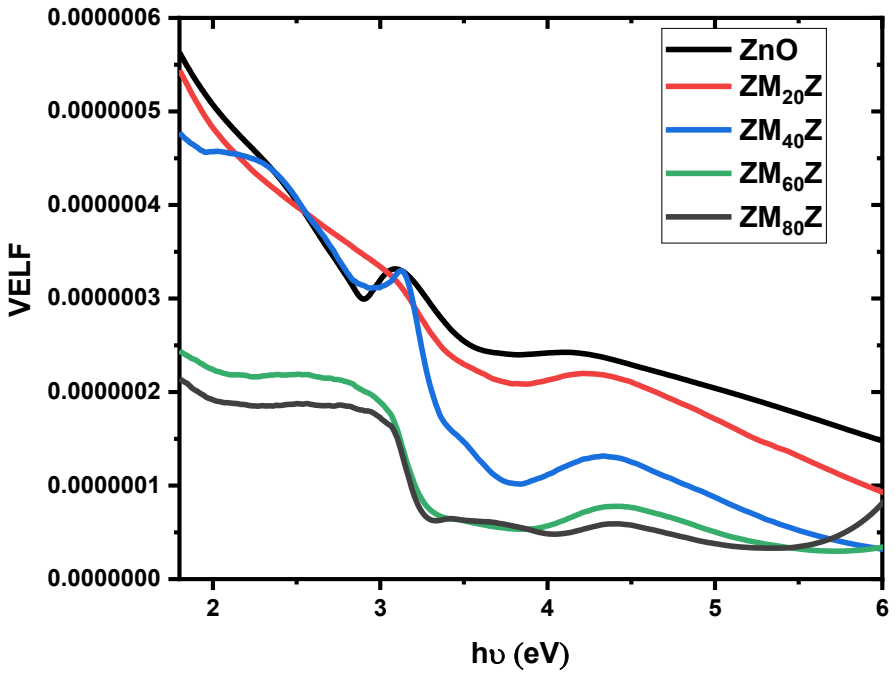
Fig.9 (a&b) *The optical and electrical conductivity of ZnO/Mn/ZnO system.*

The observed increase in optical and electrical conductivity with increasing Mn content is attributed to enhanced carrier density of the Mn layer (density increases from 6 g/cm^3 to 6.7 g/cm^3) as seen in **Table.1**, and the interaction arising from Mn layer interlayer into the layer structure. The broad hump observed in the optical and electrical conductivities spectra around 3.05 – 4.0 eV is associated with near-band -edge transitions of ZnO coupled with Mn-related charge transfer. We can also correlate the observed increase in σ_{opt} and σ_{elec}, with increasing Mn content in the ZnO/Mn/ZnO multilayer Furthermore, the possible development of preferred orientation in the top ZnO layer resulting from Mn interlayer between the ZnO layers leads to stronger optical transitions and higher σ_{opt}. The valence and surface electron energy loss functions (VELF and SELF) were evaluated to provide further insight into the electronic excitation and energy dissipation mechanisms beyond the conventional dielectric function. These parameters are particularly important for ZnO/Mn/ZnO multilayers because they provide complimentary information that cannot be obtained from the dielectric constants alone and are relevant for assessing the suitability of these multilayers in optoelectronic and photonic applications. Both quantities are derived from the complex dielectric constants as given in the following equations **[41]**:

$$VELF = \frac{\varepsilon_2}{\varepsilon_1^2 + \varepsilon_2^2} \quad (7)$$

$$SELF = \frac{\varepsilon_1}{(\varepsilon_1 + 1)^2 + \varepsilon_2^2} \quad (8)$$

In ZnO/Mn/ZnO multilayer structure, the addition of Mn alters the optical constants, and dielectric parameters, leading to noticeable changes in the energy loss spectra as seen in **Fig.10 (a&b)**



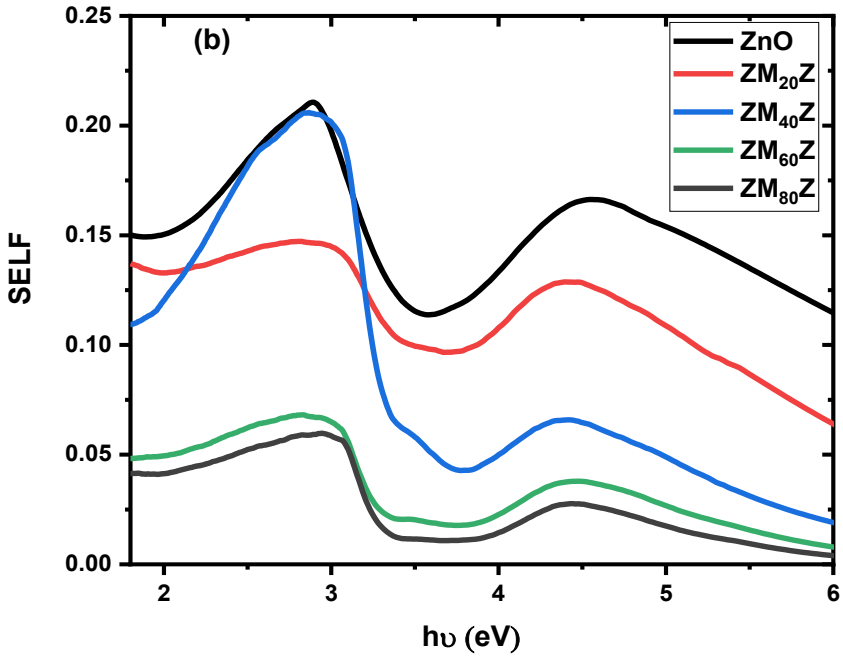


Fig10(a&b) *The energy loss spectra of the ZnO/Mn/ZnO thin film system.*

The VELF and SELF spectra show a systematic reduction in intensity with increasing Mn interlayer thickness, reflecting a suppression of bulk and surface plasmon excitations. With increasing Mn interlayer thickness, the VELF and SELF decrease systematically, whereas the n**,** ***k*** and the dielectric constants and both optical and electrical conductivities increase. This inverse behavior indicates that Mn–interlayer enhances the electronic polarizability and dielectric response of the ZnO/Mn/ZnO multilayer, facilitating stronger light-matter interaction and more efficient charge transport. The simultaneous increase in optical and electrical conductivity reflects enhanced photon absorption and carrier response, while the reduction in VELF and SELF suggests lower electron energy dissipation, confirming the beneficial role of Mn in improving the optical and electronic performance of the multilayer structure.

4.Conclusion.

Mn- ZnO thin film samples were prepared, and their structural, morphological, optical, and dielectric properties were systematically investigated. The structural and surface characteristics were examined using X-Ray reflectivity (XRR) and X-Ray Diffraction (XRD), scanning electron microscopy (SEM), and focused ion beam (FIB) analysis, which confirmed the formation of uniform thin films with well-defined surface morphology and layer thickness. The observed variation in the optical parameters with increasing Mn content in the ZnO/Mn/ZnO system

indicates significant modification of the electronic structure. The increase in (*n* and *k*) can be attributed to enhanced polarizability, increased defect states and improved light-matter interaction induced by Mn interlayer. The simultaneous increase in dielectric constants (ε_1 and ε_2), optical conductivity (σ_{opt}) and electrical conductivity (σ_{elec}), together with the reduction in VELF and SELF ,demonstrates that Mn interlayer modifies not only the optical response, but also the fundamental electronic interactions within the ZnO/Mn/ZnO multilayer. These correlated changes reveal a direct relationship between interfacial engineering and electronic structural evolution providing new insight into how Mn interlayer governs not only the optical constants but also the carrier dynamics and energy dissipation mechanism. Such a comprehensive correlation extends beyond the existing literature, providing a deeper understanding of the electronic structure evaluation beyond that reported in previous studies. Overall, these results demonstrate that Mn interlayer significantly influences electronic interactions and energy loss mechanism of the ZnO -based system, offering an effective route for tuning its structural, optical and electrical properties for potential optoelectronic and photonic applications.

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Author contributions.

M.Nabil:- Prepared all the figures, calculated the different parameters in the final form and responsible for the correspondence, **S.S. Fouad**, **G.L.Katona, E. Baradács**:- The idea and the writing and the revision. **Janos J. Tomán, G. Vecsei , Z.Erdélyi** - Prepared and supervising all the characteristics.

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All data generated or analyzed during this study are included in this published article.

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The authors declare that they have no conflict of interest.

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