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Characteristics of ZnO:Er nano thin films produced different thickness using different solvent by sol-gel method

Sol-Gel yöntemiyle farklı çözücüler kullanılarak farklı kaplama kalınlıklarında üretilen ZnO:Er nano ince filmlerin karakterizasyonu

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Characteristics of ZnO:Er Nano Thin Films Produced Different Thickness Using Different Solvent By Sol-Gel Method

Highlight

- *Er doped ZnO based semiconducting nano thin films are produced by the sol-gel technique.*
- *XRD has been used to determine phase and lattice parameters*
- *The resistivity measurement for electrical properties and transmittance measurement for optic properties have been carried out.*

Graphical Abstract

All undoped and doped ZnO thin films are oriented along the c–axis (002 plane). The intensity of (002) peak is increased with increasing number of dip. This is related to the increment of grain size. Because the intensity of the diffracted beam from bigger grains is increased, the grain size of undoped ZnO with 20 dipped sample is the biggest among the prepared samples. Therefore, the intensity of scattered rays will be more.

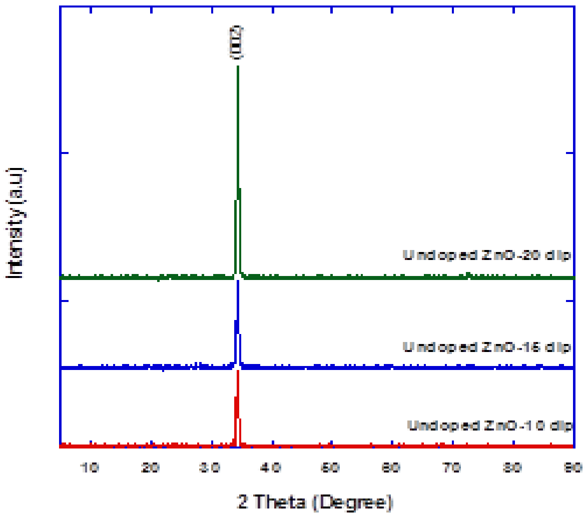

Figure . XRD patterns of different dipped thin films

Aim

The aim of this study is the production of Er doped ZnO based semiconducting nano thin films by the sol-gel technique using dip coating method which is widely used method for preparing nano size materials and the preparation of thin films at different coating thickness using different solvent.

Design & Methodology

$Zn_{1-x}Er_xO$ *semiconductor materials are prepared in different compositions (x=0.0, 0.01, 0.03 and 0.05) and in different dipping numbers (10, 15 and 20) using different solvents.*

Originality

Originality of this study is the production of ZnO based semiconducting nano thin films at different coating thickness using different solvent.

Findings

The intensity of (002) peaks which determines the orientation of the structure has decreased with increasing number of coating. The grain size has decreased with the increase in the number of bottoms and the addition of Er. Examination of electrical properties showed that the resistivity values decreased with the increase of the Er doping to the undoped ZnO sample. Optical transmittance values have generally increased with increasing coating thickness.

Conclusion

In this study, the structural, electrical and optical properties of ZnO-based thin films produced with different coating thicknesses and different Er dopings are examined and comparisons were made for each material.

Declaration of Ethical Standards

The authors of this article declare that the materials and methods used in this study do not require ethical committee permission and/or legal-special permission.

Sol-Gel Yöntemiyle Farklı Çözücüler Kullanılarak Farklı Kaplama Kalınlıklarında Üretilen ZnO:Er Nano İnce Filmlerin Karakterizasyonu

Araştırma Makalesi / Research Article

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ÖZ

Bu çalışmada, Er katkılı ZnO tabanlı yarıiletken nano ince filmler, nano boyutlu malzemelerin hazırlanmasında yaygın olarak kullanılan daldırma kaplama yöntemi kullanılarak sol-jel tekniği ile üretilmiştir. $Zn_{1-x}Er_x O$ ince filmler farklı çözücüler kullanılarak farklı kaplama kalınlıklarında hazırlanmıştır. Er katkısının ve film kalınlığının ZnO yarıiletken nano ince filmlerin yapısal, elektrik ve optik özellikleri üzerindeki etkisi ayrıntılı olarak incelenmiş ve sonuçlar aynı koşullarda hazırlanan katkısız numune ile karşılaştırılmıştır. Yarıiletken ince filmlerin faz ve kafes parametrelerini belirlemek için X ışını kırınım analizi (XRD) kullanılmış ve mikroyapı özellikleri için tarama elektron mikroskobu (SEM) ölçümleri yapılmıştır. Elektriksel özellikler için özdirenç ölçümü ve optik özellikler için geçirgenlik ölçümü yapılmıştır.

Anahtar Kelimeler: ZnO, ince film, sol-gel yöntemi, yarıiletken.

Characteristics of ZnO:Er Nano Thin Films Produced Different Thickness Using Different Solvent By Sol-Gel Method

ABSTRACT

In this study, *Er* doped *ZnO* based semiconducting nano thin films are produced by the sol-gel technique using dip coating method which is widely used method for preparing nano size materials. $Zn_{1-x}Er_x O$ thin films are prepared different coating thickness using different solvent. The effect of the Er doping and film thickness on structural, electric and optic properties of the ZnO semiconducting nano thin films are investigated in detail and compared with undoped sample which prepared in same conditions. X-ray diffraction analysis (XRD) has been used to determine phase and lattice parameters of the semiconducting thin films and scanning electron microscope (SEM) measurements are made for microstructure properties. The resistivity measurement for electrical properties and transmittance measurement for optic properties have been carried out.

Keywords: ZnO, thin film, sol-gel method, semiconductor.

1. INTRODUCTION

Technology and science are an integral part of life for humans. In this context, it is necessary and sufficient to use science to implement developing and advancing technology. As people's living conditions varied and increased, new requirements emerged, and these requirements brought new technological pursuits. In most of the major breakthroughs that are performed, the importance of materials used in developed products has been very great and this case has led to the emergence of material technology. As the requirements for technological devices increased day by day, the expected features of the used materials increased as well. As a result of all these requirements, scientists have improved their study at the point of producing technological materials that are constantly evolving and able to respond to the needs of time. Therefore, every new material and technique developed are of great importance [1-5].

Rapid advances in today's technology have increased expectations in the properties and performance of materials to be used. Materials that cannot be produced by classical production methods (coating etc.) and increase in the need has led to the emergence of new production techniques [6,7]. The sol-gel method,

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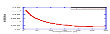
commonly used in thin film production, is very important for nano-sized material production among these methods [8,9].

Similar studies in the literature are as follows. ZnO and ZnO:TiO_2 thin films are prepared on glass using sol-gel spraying coating technique by Firdaus et al. in their study. The performance and properties of nanostructured ZnO and ZnO:TiO2 thin films at different thicknesses are investigated. The effect of thickness on electrical and optical properties is identified using the 2-point probe KEITHLEY 2400 welding meter and UV-Vis spectrophotometer, respectively. According to current-voltage measurements, nanocomposite ZnO:TiO2 thin films have higher conductivity than nanostructured ZnO thin films. Optical properties show that the band gap for nanocomposite ZnO:TiO2 decreases with increasing thickness [10].

Chien et al. investigate the properties of Zn//Al /Zn multilayer structures formed on glass in different thicknesses in their study, ZnO films are deposited using cathodic arc plasma technique at low temperature (<75ºC). Microstructure, optical and electrical properties are examined and discussed. The multilayer films showed high (002) peak orientation. For ZnO (50 nm)/Al (10 nm)/ZnO (50 nm) multilayer film, an average transmittance of about 70% in the visible region and the lowest resistance of about $4.02x10^{-4}$ Ω cm can be achieved [11].

In this study, ZnO-based thin films, which are called materials with thickness below 1 µm, were produced using the sol-gel method. In today's technology, they are widely used in technological applications due to their various positive properties [12,16]. The goal of the present study is the investigation of processing, characterization, and the effect of doping concentration and film thickness on the structural, electrical and optical properties of $Zn_{1-x}Er_xO$ nano thin films.

2. EXPERIMENTAL DETAILS

 semiconductor materials were prepared in different compositions (x=0.0, 0.01, 0.03 and 0.05) and in different dipping numbers (10, 15 and 20) using different solvents (monoethanolamine, diethanolamine and triethanolamine). Zinc acetate dihydrate ($C_4H_6O_4Zn$-$2H_2O$) and Erbium 2-4 pentanedionate (($Er(OOCCH_3)_3$-$4H_2O$)) were mixed with appropriate amounts of methanol, monoethanolamine, diethanolamine and triethanolamine solvents at room temperature for 8 hours in magnetic stirrer until a transparent solution was formed. After this process, the glass substrates are dipped into these prepared solutions and pull into the vertical furnace at 400 ºC for 5 minutes. This process are repeated according to numbers of diping number. At the end, the all coated nano thin films have been sintered at 400 ºC for 30 minutes. The coated nano thin films have been characterized by structural, electical and optical and the results are given below.

3. RESULTS AND DISCUSSION

3.1. XRD Measurements

Different solvents such as monoethanolamine, diethanolamine and triethanolamine have been used during preparation of samples. XRD measurements are performed to determine whether the materials are in the desired ZnO structure [17-19]. The XRD analysis of the $Zn_{0.95}Er_{0.05}O$ thin films at the highest doping rate with different solvents are given in Fig 1. As seen from this figure, ZnO structure was completely formed in the samples used as solver monoethanolamine (MEA) and however, the similar result could not be obtained for other solvers (DEA and TEA). After this result, this study is only focused on undoped and Er doped with different compositions (x=0.0, 0.01, 0.03 and 0.05) and different dipping numbers (10, 15 and 20 bottom) with MEA solver.

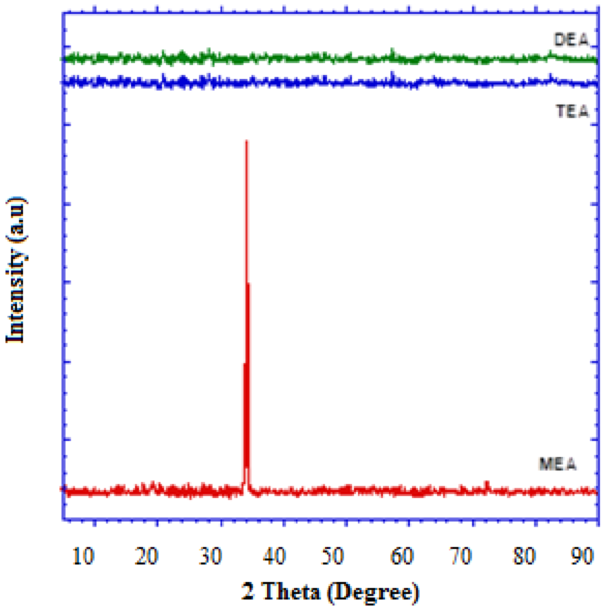


Figure 1. XRD patterns of undoped ZnO thin films

XRD analysis of ZnErO thin films in different dopings (x = 0.01 ,0.03 and 0.05) with various dipped: Fig. 2, 3 and 4 shows the XRD graphs of thin films prepared by 10, 15 and 20 dip with different doping ratio, respectively. The hegzagonal ZnO stuctures have been observed to form for all doping ratios as well as no Er phase has been detected. This is an indication that the additive materials (Er) have entered the structure [20]. Another important result is that the orientations of prepared thin films are in one direction. These orientations are along the (002) planes for all doping ratios. We also see graph of Fig 2.b that the peak position of (002) plane shifted lower angle with increasing doping ratio. From here, *c* lattice parameters are calculated and given in Table 1. With rising doping ratio of Er, *c* lattice parameter is partially increased. This is an expected result because the ionic radius of Zn^{+2} (0.74 Å) is smaller than the ionic radius of Er^{+3} (0.88 Å). Obtaining information about grain sizes using XRD graphs is useful for structural analysis, the grain size of the samples is calculated by Scherrer's formula given as

$$D = 0{,}94\lambda / \beta \cos\theta \qquad (1)$$

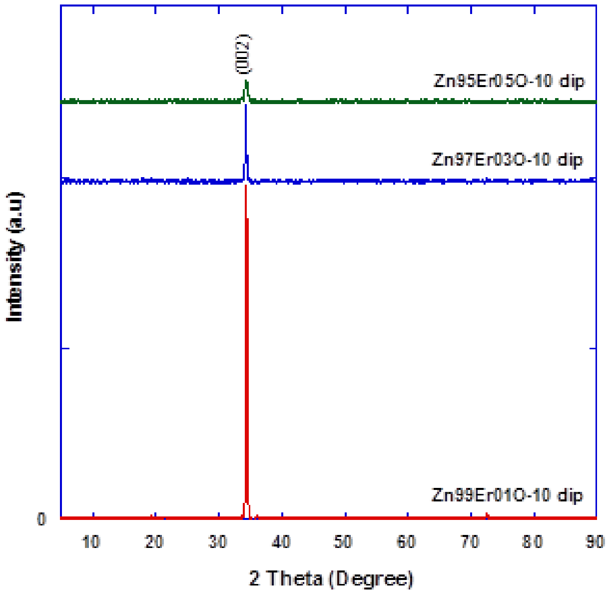


Figure 2. XRD patterns of 10 dipped thin films

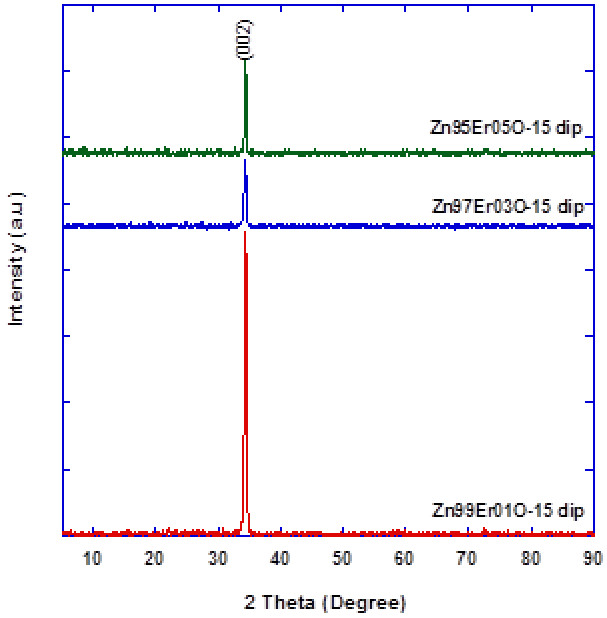


Figure 3. XRD patterns of 15 dipped thin films

Here, *D*, *λ*, *β* and *θ* represent the grain size (nm), X-Ray wavelength (15.418 nm), the full width half maximum (FWHM) value for the (002) peak and the Bragg angle [21,22], respectively and given in Table 1. Er doping reduces the grain size. Grain size decreased with the increase of the dipping number. This situation can be interpreted that with the increasing the dipping number, a more rigid structure has been formed. Doping materials are placed in both inter-grain and intra-grain. From this point on, with the increase in the dip number, the gap between the grains fills more and compresses the structure and reduces the grain size. Therefore, we can say that this is an expected result by increasing the number of dips.

XRD analysis of undoped ZnO thin films with various dipped: The XRD graphs of the undoped ZnO thin films with different numbers of dipped are given in Fig 5. The intensity of (002) peak is increased with increasing number of dip. This is related to the increment of grain size (Table 1). Because the intensity of the diffracted beam from bigger grains is increased, the grain size of undoped ZnO with 20 dipped sample is the biggest among the prepared samples. Therefore, the intensity of scattered rays will be more.

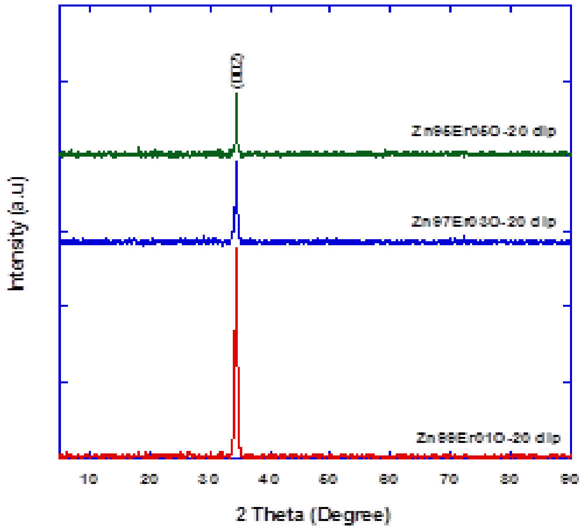


Figure 4. XRD patterns of 20 dipped thin films

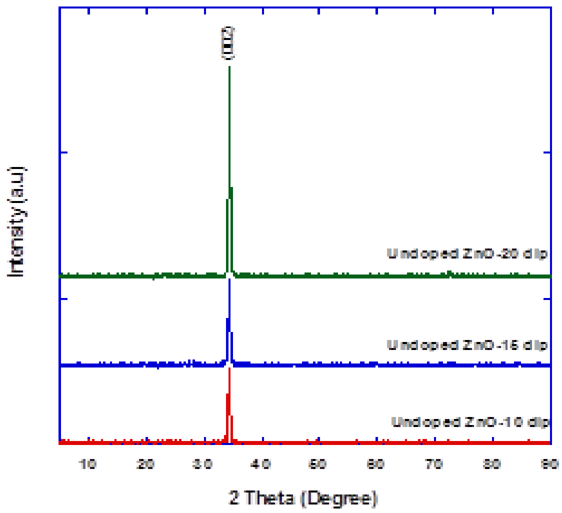


Figure 5. XRD patterns of different dipped thin films

All undoped and doped ZnO thin films are oriented along the c–axis. Therefore, only the *c* lattice parameter is calculated. *c* lattice parameters of doped thin films at all dips are very close to each other and a little higher than undoped ZnO thin films.

Table 1. *c* lattice parameters and grain size of 10, 15 and 20 dipped thin films

	Samples	**c (002)**	**Grain Size (nm)**
10 dipped	Undoped ZnO	5.20	20.54
	$Zn_{0.99}Er_{0.01}O$	5.21	21.44
	$Zn_{0.97}Er_{0.03}O$	5.22	18.18
	$Zn_{0.95}Er_{0.05}O$	5.22	17.90
15 dipped	Undoped ZnO	5.20	22.72
	$Zn_{0.99}Er_{0.01}O$	5.21	22.35
	$Zn_{0.97}Er_{0.03}O$	5.21	18.16
	$Zn_{0.95}Er_{0.05}O$	5.22	18.60
20 dipped	Undoped ZnO	5.20	26.81
	$Zn_{0.99}Er_{0.01}O$	5.21	20.58
	$Zn_{0.97}Er_{0.03}O$	5.21	17.96
	$Zn_{0.95}Er_{0.05}O$	5.22	15.06

3.2. SEM Measurements

The SEM images of the some prepared thin films are shown in Figure 6 and Figure 7. As can be seen from the figures, it is clear that grain sizes are reduced both by the doping of Er and by the increase in the number of dips. This result also supports the grain size results calculated with XRD data. The average grain size of the films is 20.02 nm. Increasing Er doping leads to smaller grains which shows that Er inhibits growth of the ZnO lattice. The reduction in grain size with Er doping might be due to pinning and dragging effects of the dopant at and between the grain boundaries, respectively. These two effects can also be seen to be in action in the observed mobility decrease in the grain boundaries.

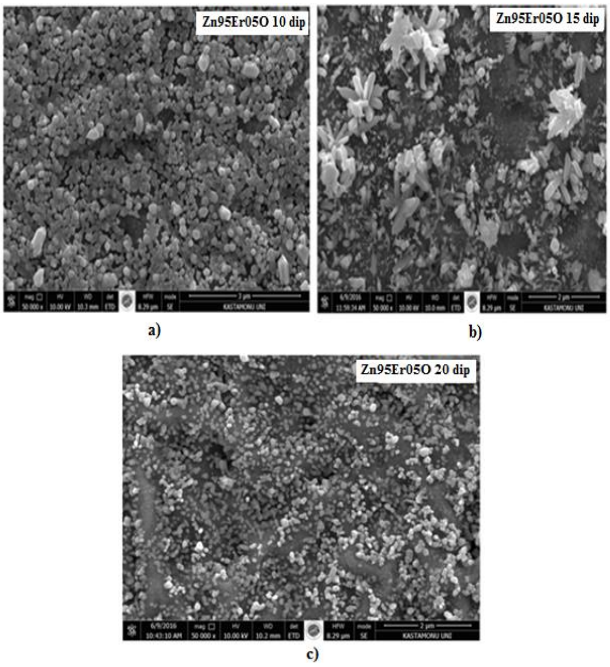


Figure 6. SEM images of different thicknesses of Zn95Er05O thin films

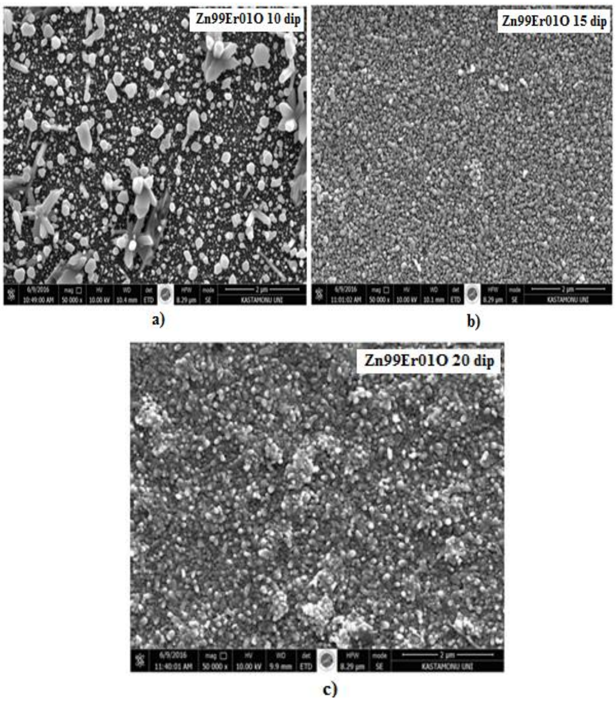


Figure 7. SEM images of different thicknesses of Zn99Er01O thin films

3.3. Electrical Resistivity (ρ-T) Measurements

Electrical measurements of Er-doped semiconductor nano thin films coated at different depths (different thicknesses) were performed using He cooled closed circuit cryostat system. Figure 8-11 present the *ρ-T* measurements of the undoped and Er doped ZnO thin film with various number of dipping. The resistivity of 10 dipped samples is higher than 15 dipped samples, however, it is less than 20 dipped samples in the series of undoped. Resistivity of 20-dip coated samples has significantly increased. These changing in the resistivity can be interpreted that Er atoms which are doped at small dips (10 and 15 dips) are probably placed in interface of the ZnO lattice. These atoms may create more oxygen voids and this may cause more lattice defects. A further oxygen gap is a factor that increases the conductivity of films. Er atoms in the 20 dipped $Zn_{1-x}Er_xO$ samples which has a tighter structure, are most probably placed in ZnO lattice instead of interface and therefore, the oxygen gaps are decreased [23]. The resistivity values of Zn95Er05O and Zn99Er01O samples are reduced by doping ratio, however, these values increased with the increase in the bottom number compared to the coating thickness.

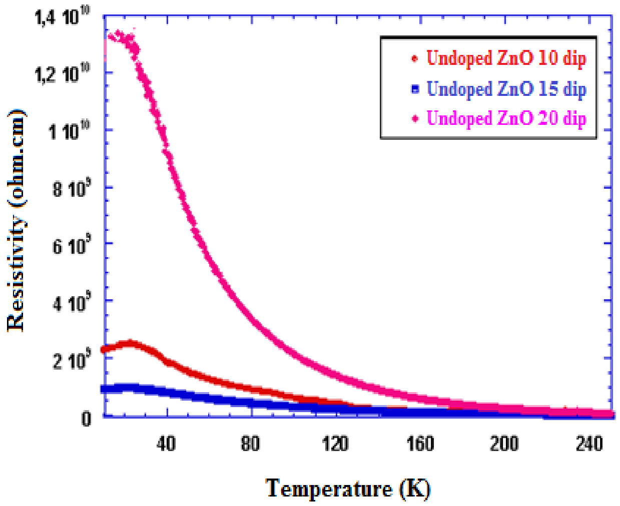


Figure 8. Resistivity as a function of temperature curves of undoped samples

The resistivity values vary depending on the Er^{+3} ions acting as free electron supplier donors and replacing Zn^{+2} within the ZnO crystal within the conductor. It is known that higher electron concentration causes lower resistivity and affects electron mobility. Er doped ZnO thin films have a smaller particle size and this leads to an increment in electron scattering, higher resistivity and lower mobility. Point and surface imperfections, different scattering centers (scattering from lattice and impurities) and free carrier concentration play an important role in the resistivity of films [24].

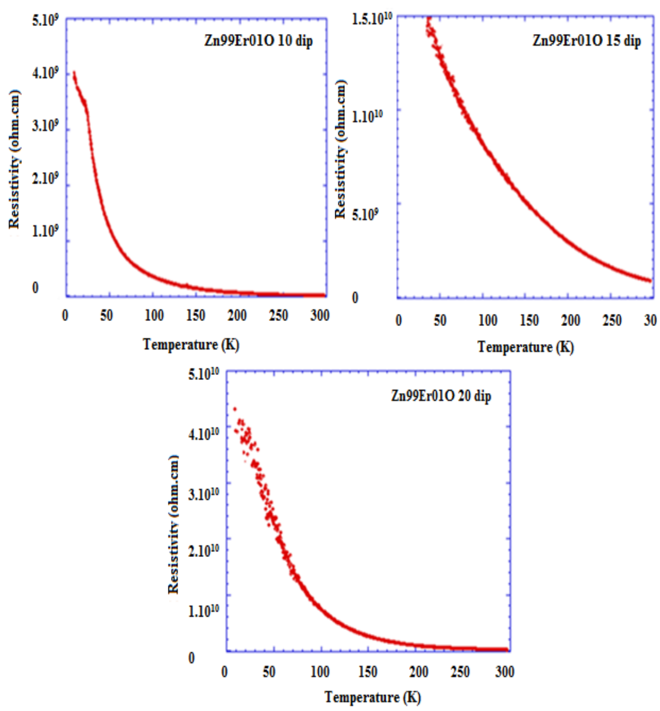


Figure 9. Resistivity as a function of temperature curves 10, 15 and 20 dipped thin films

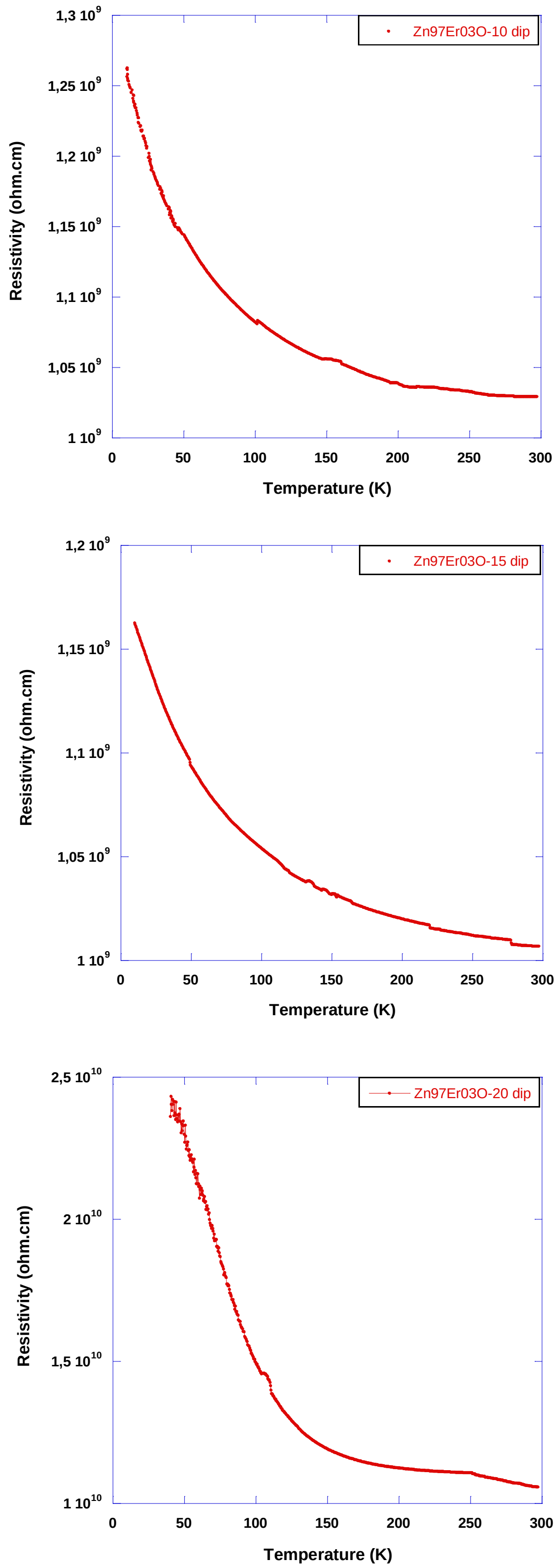


Figure 10. Resistivity as a function of temperature curves 10, 15 and 20 dipped $Zn_{97}Er_{03}O$ thin films

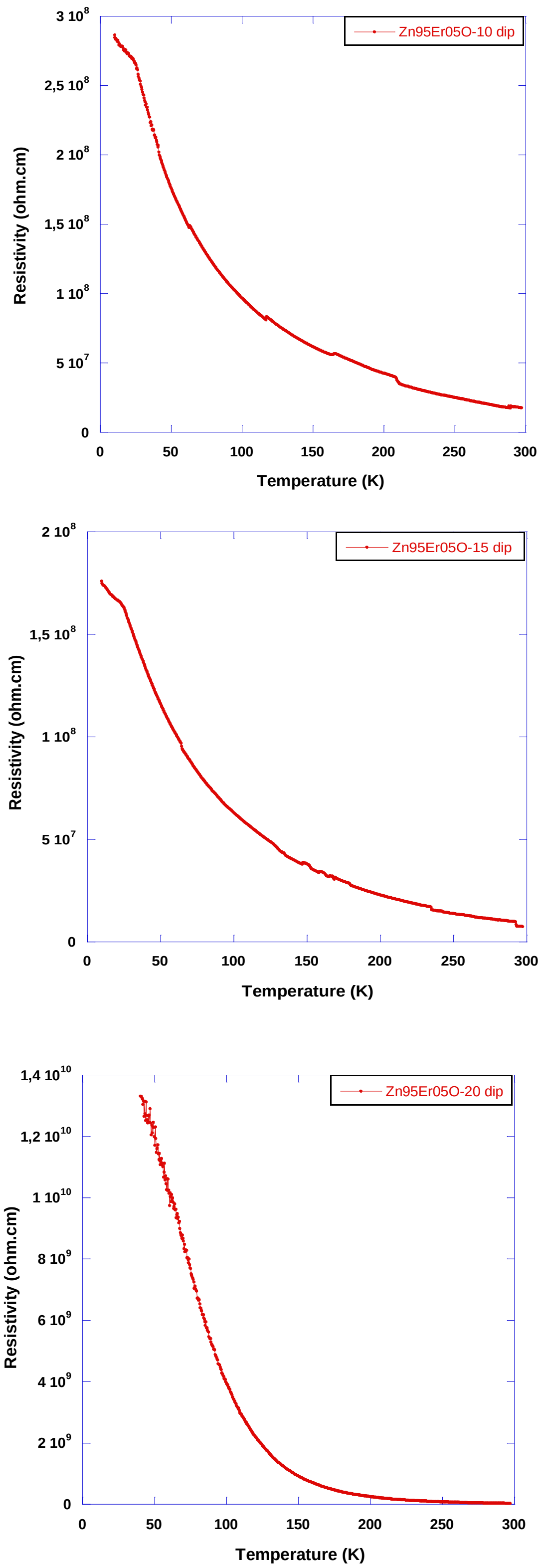


Figure 11. Resistivity as a function of temperature curves 10 , 15 and 20 dipped $Zn_{95}Er_{05}O$ thin films

3.4. Optical Measurements

Optical properties are determined with SHIMADZU Spectrophotometer which studies between UV and Vis region. Optical transmittance and reflection properties of thin films are investigated [25]. The measurements were made in the wavelength range of 190 – 1100 nm, which is the maximum measuring range of the device. 15 dipped undoped ZnO thin films showed higher transmittance in the visible area compared to other undoped samples (Figure 12). High transmittance is suitable and preferable for industrial applications as transparent electrode. However, the transmittance of 20 dipped undoped ZnO based thin films decreased. The observed weak transmittance is due to structural defects in the structure. The defect in the structure increases absorption in the visible area and this decrease transmittance. The increment of coating thickness according to high number of dipping, transmittance values have increased. This indicates that optically better materials are obtained with increasing the number of coatings. High transmittance is the result of the creation of a more precise structure [26,27].

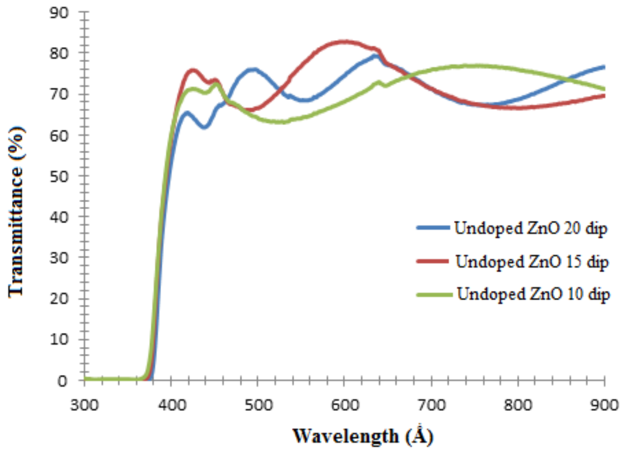


Figure 12. Optical transmittance spectra of undoped ZnO thin film

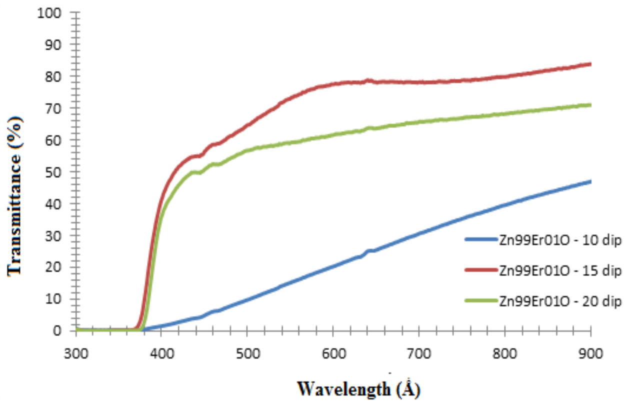


Figure 13. Optical transmittance spectra of Zn99Er01O thin film

It was observed that the transmittance value of thin films increased up to the doping ratio of x=0.03 and decreased after this doping ratio. Structural and lattice defects in Zn95Er05O sample, which has 0.05 doping ratio, have resulted in a decrease in transmittance values.

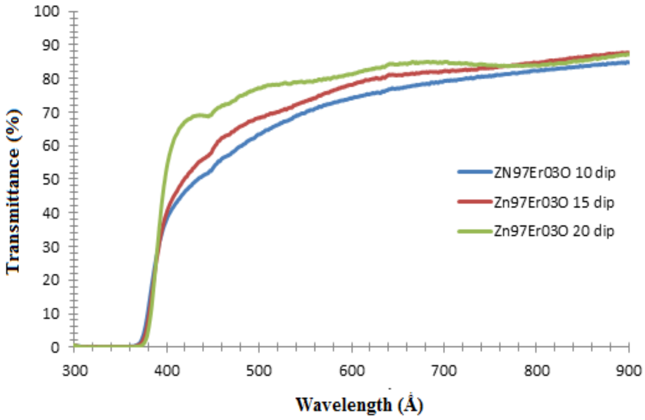


Figure 14. Optical transmittance spectra of Zn97Er03O thin film

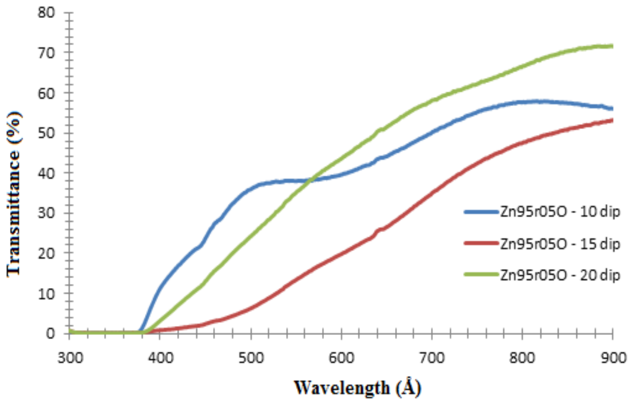


Figure 15. Optical transmittance spectra of Zn95Er05O thin film

In order to determine the energy gap of the films produced, the plots of the square of Kubelka-Munk function ($(\alpha h\upsilon)^2$) versus to energy (hυ) for ZnO and ZnErO thin films are given in Fig. 16.

The optical band gap energy (Eg) values for undoped and Er doped ZnO thin films are calculated using the formula

$$\alpha h\nu = A(h\nu - E_g)^n \qquad (2)$$

where A is constant, α is the absorption coefficient, $h\nu$ is the discrete photon energy, E_g is the band gap energy, and *n* depends on the type of optical transmission in the band gap. n=1/2 is for direct band gap crystalline semiconductors. While the optical band gap energy is about 3.28 eV for undoped ZnO film, it is in the range of 3.23-3.28 eV for Er doped thin films as shown in Fig.16 and Table 2. Bandgap energy of the doped films is smaller than the undoped film. We can say that bandgap energy is increase with increasing both the Er doping and dip number. Expansion of this band gap is due to the decrease in the band tail width. Semiconductor materials coupled with a dopant cause the formation of the band tail in band gap.

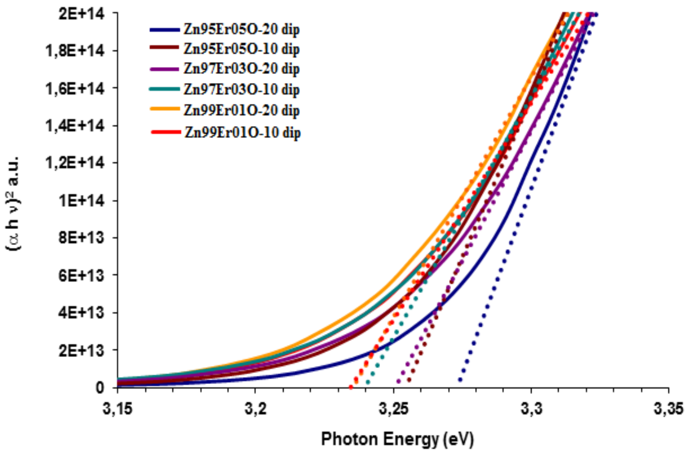


Figure 16. The plots of $(\alpha h\nu)^2$ vs (hν) for ZnErO thin films

Table 2. E_g values for ZnO and Er doped ZnO thin films

	Samples	Energy Gap (eV)
	Undoped ZnO	3.28
10 dipped	$Zn_{0.99}Er_{0.01}O$	3.23
	$Zn_{0.97}Er_{0.03}O$	3.25
	$Zn_{0.95}Er_{0.05}O$	3.25
15 dipped	$Zn_{0.99}Er_{0.01}O$	3.24
	$Zn_{0.97}Er_{0.03}O$	3.25
	$Zn_{0.95}Er_{0.05}O$	3.25
20 dipped	$Zn_{0.99}Er_{0.01}O$	3.24
	$Zn_{0.97}Er_{0.03}O$	3.26
	$Zn_{0.95}Er_{0.05}O$	3.28

3. CONCLUSIONS

In this study, the structural, electrical and optical properties of ZnO-based thin films produced with different coating thicknesses and different Er dopings were examined and comparisons were made for each material.

The following results were obtained:

- The intensity of (002) peaks which determines the orientation of the structure has decreased with increasing number of coating. Grain sizes are the smallest at 20 dipped samples between undoped and Er doped samples. All prepared thin films are oriented along the c-axis. c lattice parameters of all Er-doped thin films are close to each other in all number of coatings. These values are compared with the c lattice parameters of undoped sample, it is a little reduced. The ZnO hegzagonal structure has not changed.
- The grain sizes calculated from XRD data and SEM micrographs are compatible with each other. The grain size has decreased with the increase in the number of bottoms and the addition of Er.

- Examination of electrical properties showed that the resistivity values decreased with the increase of the Er doping to the undoped ZnO sample. When the highest (x = 0.05) and lowest (x = 0.03) doping ratios were compared, the resistivity values were decreased with Er additive.
- Optical transmittance values have generally increased with increasing coating thickness.

As a result, the grain sizes of 20-dipped ZnErO nano thin films are the smallest. The electrical conductivities are lower. Because they have high transmittance values in terms of optical properties, they are the best thin films in terms of transmittance in the produced films.

ACKNOWLEDGEMENT

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DECLARATION OF ETHICAL STANDARDS

The authors of this article declare that the materials and methods used in this study do not require ethical committee permission and/or legal-special permission.

AUTHORS' CONTRIBUTIONS

Ozgur Ozturk: Wrote the manuscript.

Elif Asikuzun: Wrote the manuscript.

Zeynep Banu Hacioglu: Performed the experiments and analyzed the results.

Serap Safran: Performed the experiments and analyzed the results.

CONFLICT OF INTEREST

There is no conflict of interest in this study.

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