



Gazi University

**Journal of Science**

PART A: ENGINEERING AND INNOVATION

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## Influence of the Hot Water Parameter on the Structural and Optical Properties of SILAR-Deposited ZnO Samples

Salih AKYÜREKLİ<sup>1</sup> Tuğba ÇORLU<sup>1</sup> Irmak KARADUMAN ER<sup>2\*</sup> Selim ACAR<sup>3</sup> <sup>1</sup>Innovative Technologies Application and Research Center, Suleyman Demirel University, Isparta, Türkiye<sup>2</sup>Department of Medical Services and Techniques, Eldivan Medical Services Vocational School, Çankırı Karatekin University, Çankırı, Türkiye<sup>3</sup>Department of Physics, Faculty of Science, Gazi University, Ankara, Türkiye

Keywords	Abstract
ZnO SILAR Morphological Characterization The Temperature of the Hot Water	In this study, ZnO thin films were grown by the Successive Ionic Layer Adsorption and Reaction (SILAR) method. The SILAR method is a chemical solution-based method consisting of 4 steps: solution, hot water, air and deionized water. Our main goal is to examine the changes in SILAR method production by changing the hot water parameter from these steps. It is widely known that chemical synthesis methods and their relative parameters have a crucial effect on the size of the produced thin films, surface area/volume ratio, porosity as well as defects in the film which in turn affect the morphology. All parameters were kept the same and changes were made in the hot water step, in addition to the classic 90°C hot water step, constant temperature ultrasonic cleaner at 40 °C, an ultrasonic cleaner at room temperature, and an ultrasonic breaker at room temperature are used instead of the hot water step. For this purpose, alternative devices such as ultrasonic cleaner and ultrasonic breaker were used to break the unwanted weak bonds at lower temperatures during production. The structural, morphological, optical and electrical properties were characterized and the results were investigated in detail.

Cite
Akyürekli, S., Çorlu, T., Karaduman Er, I. & Acar, S. (2022). Influence of the hot water parameter on the structural and optical properties of SILAR-deposited ZnO samples, <i>GU J Sci, Part A</i> , 9(4), 439-451.

Author ID	Article Process
S. Akyürekli, 0000-0001-6005-667X	<b>Submission Date</b> 26.09.2022
T. Çorlu, 0000-0001-5828-207X	<b>Revision Date</b> 12.10.2022
I. Karaduman Er, 0000-0003-3786-3865	<b>Accepted Date</b> 03.11.2022
S. Acar, 0000-0003-4014-7800	<b>Published Date</b> 31.12.2022

### 1. INTRODUCTION

Zinc oxide (ZnO) material is one of the most promising candidates due to its inexpensive, abundance, chemical and mechanical stability, and optical and electrical properties among metal oxide materials (Irani et al., 2016; Vaizoğullar, 2018). One of the most significant reasons for the wide application area of ZnO material is that the direct band gap of ZnO is 3.37 eV and the excitation binding energy value is 60 meV (Şahin et al., 2022). Due to these properties, ZnO has lots of applications. Some of these are rubber, ceramic, pharmaceutical and electronic industry, medical applications, and many others. Great interest in ZnO nanoparticles is used by biological sciences such as an antibacterial, antifungal, and antifouling agent. The safeness of ZnO molecules and their compatible nature with the human body made them appropriate materials for biological applications. ZnO nanoparticles have diameters less than 100 nanometers.

The effect of defects on the performance of optoelectronic devices is a well-known issue. Defects can act as non-namely recombination centers in optoelectronic circuit devices such as light-emitting diode and laser. Thus, it can reduce the efficiency of the circuit device and even make light production impossible. In this case, the defects can act as centers that reduce carrier mobility and that prevent the circuit device from operating at high frequencies. Particle size and crystal morphology play an important role in making better circuits. For this reason, many thin-film growth methods have been developed. These are physical vapor deposition, ion sputtering, solvothermal method, hydrothermal methods, sol-gel methods, spray pyrolysis, chemical bath

deposition, and SILAR (Successive Ionic Layer Adsorption and Reaction), etc. Among these methods, SILAR is one of the fast and most affordable methods because it does not require any sophisticated substrate (Çorlu et al., 2018). Parameters can be controlled and changeable according to the main purpose. SILAR method is based on sequential ionic layer adsorption and reaction (Çorlu et al., 2018). The originality in this method is the adsorption (deposition) of one substance onto another substance on the substrate (Şahin et al., 2022). Changing the molarity of the solution used in the SILAR cycle, increasing or decreasing the hot water temperature, changing the thickness, differences in the waiting time in the solution, etc. parameters lead to different structural, optical and electrical properties in samples grown with SILAR (Karaduman Er et al., 2021).

The spooling operation can be performed in a single row or with multiple consecutive repetitions (Radhi Devi et al., 2020). The removal of unwanted substances that may occur on the surface is achieved by rinsing with the step of deionized water. In general, the quality of the films can be changed by changing the structural, optical and electrical properties of the films produced by changing one or more parameters from these steps. Şahin et al. (2022) have studied the effect of thymus syriacus plant extract on the physical properties of ZnO nanoparticles synthesized by SILAR method and studied their properties with this changing process. Gahramanli et al. (2021) have investigated role of temperature in the growth and formation of Cd<sub>x</sub>Zn<sub>1-x</sub>S/PVA nanocomposites through SILAR method. The main purpose of this study is to discuss the results obtained by changing the hot water parameter from SILAR parameters and also the effect of the vibration on this parameter. Structural, optical and electrical changes will be shown on the effect of changing this step on production and its difference from the traditional production method. For this purpose, the structural, morphological, optical and electrical properties of the produced ZnO thin films were investigated and The most suitable parameter for advanced industrial or technological applications was determined.

## 2. MATERIAL AND METHOD

([Zn(NH<sub>3</sub>)<sub>4</sub>]<sup>2+</sup>) zinc-ammonia mixture was used for the production of ZnO on the cleaned glass substrates. In order to prepare the zinc-ammonia mixture, firstly, 1,363 g of ZnCl<sub>2</sub> (99% Sigma Aldrich) material was weighed on a precision balance. The weighed material was placed in a flask and 100 mL of distilled water was placed on it and mixed in a magnetic stirrer for 30 minutes. Thus, 0.1 M ZnCl<sub>2</sub> (pH≈5.5) solution was obtained. The prepared solution was mixed with 25-28% (2 mL) NH<sub>3</sub> at [18:2] ratios to make it ready for growth.

A SILAR cycle of the produced ZnO thin films is given in Figure 1. Each glass substrate was cleaned individually in propanol for 10 minutes. Solutions were prepared during the substrate cleaning process. The cleaned and prepared glass substrates were subjected to the classical SILAR cycle as the first growth method. The glass substrate was kept in the ([Zn(NH<sub>3</sub>)<sub>4</sub>]<sup>2+</sup>) solution, which was prepared before, for 15 seconds, respectively. The substrate, which was removed from the solution, was kept in distilled water at 90°C for 7s. The substrates, which were kept in air for 60 s after hot water, were then immersed in distilled water at room temperature and kept there for 30 s. Thus, a cycle is completed. After the first growing process, ultrasonic cleaner was chosen as the hot water parameter in the second growing method. The cleaned substrate was first immersed in the prepared solution and then in an ultrasonic cleaner containing water at 40°C temperature, and the subsequent stages were kept the same as the first growth process. In another growth process, the prepared glass substrate was immersed in an ultrasonic cleaner filled with room-temperature water as an alternative to the hot water parameter. Finally, the cleaned glass substrate was used in the magnification process at room temperature using an ultrasonic breaker. Thus, ZnO material was grown on glass substrates by using different methods as an alternative to the hot water parameter. This cycle was repeated forty times.

Each sample was named according to the production parameter. The sample grown using the 90°C hot water parameter was named ZnO1, the growth using the ultrasonic cleaner with 40°C water was named ZnO2, the growth made with an ultrasonic cleaner with room temperature water was named ZnO3, and the growth with a 1 pulse vibration ultrasonic breaker with room temperature water was named ZnO4, as given in Table 1.

XRD analysis were measured in the Bruker D8 Advance Twin-Twin XRD system using the Bragg-Brentano method in the angle range of 20°-80° (2θ). The surface morphological characterization using FEI QUANTA FEG 250 SEM device of ZnO thin films were observed. Elemental components were obtained by using energy dispersive X-ray spectrometry (EDAX) together with SEM analysis. Optical analyzes such as absorbance and

transmittance were performed using the UV-1800 SHIMADZU device. The surface morphology of all the thin films were investigated with using the ez-AFM Nanomagnetics device in  $2 \times 2 \mu\text{m}^2$  area. The electrical characterization was taken with a Keithley 2400 source meter by a computer.

The thickness was estimated by the weight difference method using this relation;

$$d = \frac{m}{\rho A} \quad (1)$$

with  $m$  is the film mass deposited on the substrate,  $\rho$  is the density of the ZnO material in the bulk form (ZnO;  $\rho=5.61 \text{ g/cm}^3$ ). The thickness of the ZnO thin films were found 375 nm, 390 nm, 400 nm and 415 nm for ZnO1, ZnO2, ZnO3 and ZnO4, respectively. It is thought that the molecules in the solution cannot sufficiently adhere to the surface due to the vibration effect and as a result, the thickness of the samples decreases. Even if samples are grown in the same cycles, changes in parameters can lead to changes in thickness (Abdulrahman et al., 2021).

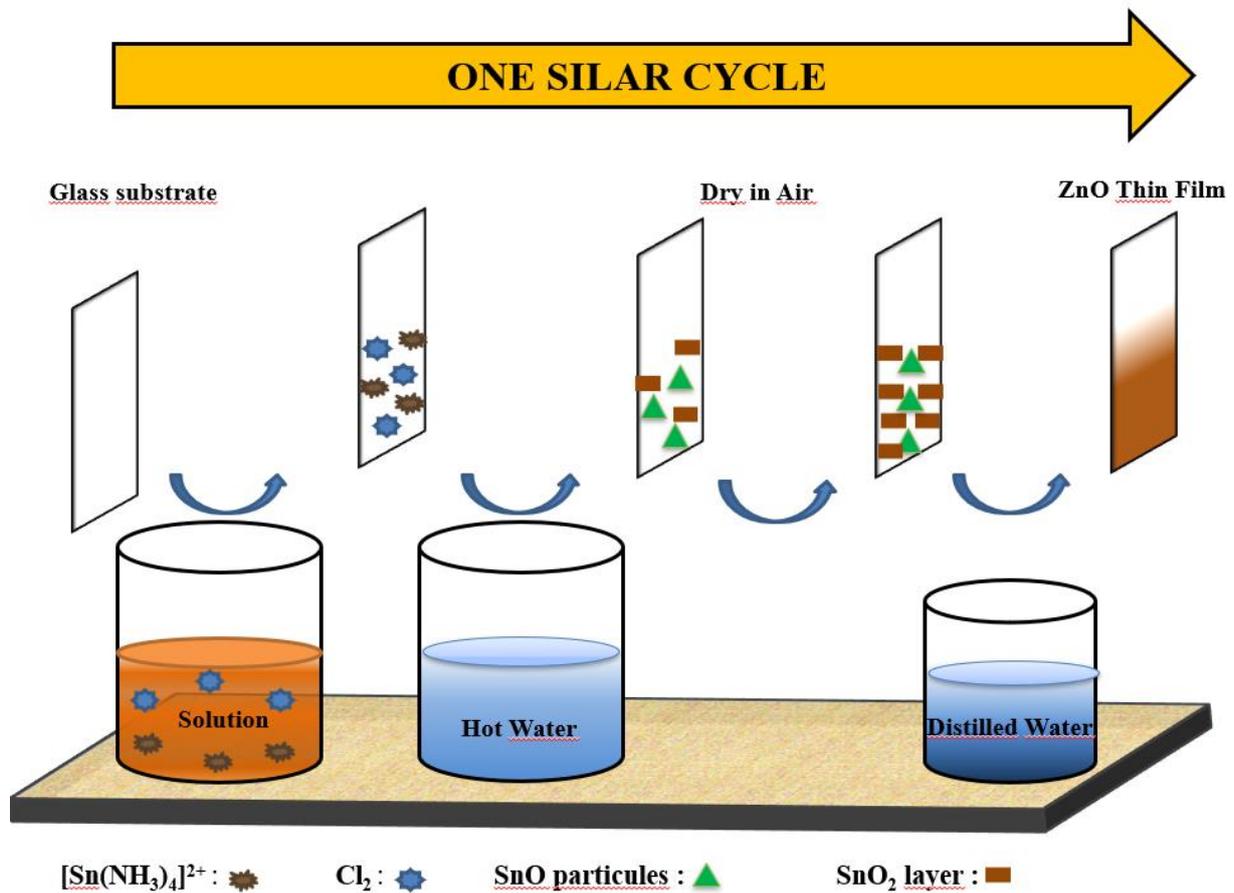


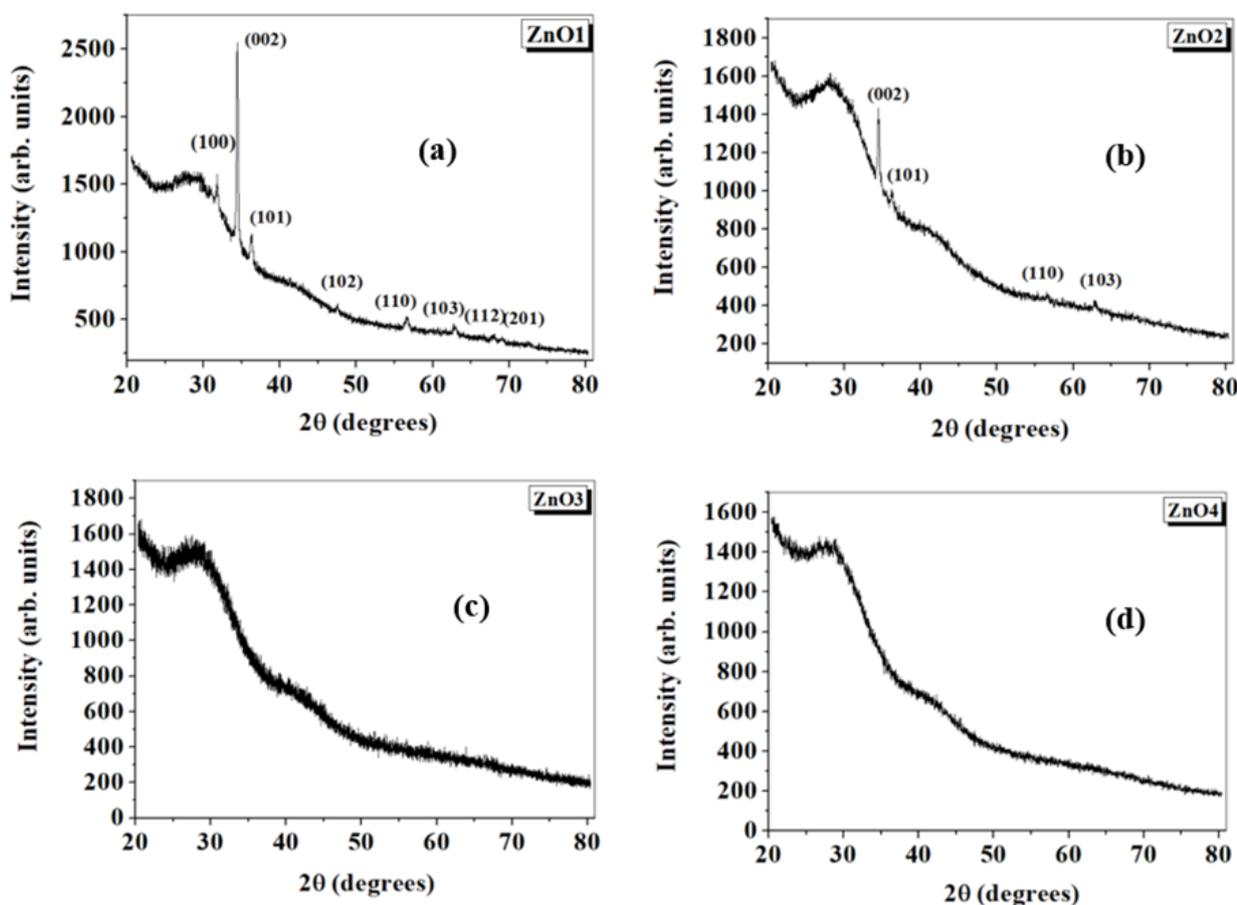
Figure 1. The schematic diagram of SILAR cycle

Table 1. Renaming of each sample according to the production parameter

Producing Parameter	Sample Code
the 90°C hot water	ZnO1
the ultrasonic cleaner with 40°C water	ZnO2
with an ultrasonic cleaner with room temperature water	ZnO3
with a 1 pulse vibration ultrasonic breaker with room temperature water	ZnO4

### 3. RESULTS AND DISCUSSION

Figure 2 shows the pattern of XRD analysis of all samples. It was observed that (100), (002) and (101) zinc peaks were dominant in ZnO1 and ZnO2 thin films and the peaks overlapped with a wurtzite structure (JPDS Card No: 01-079-0208) (Gujar et al., 2008; Yıldırım & Ateş, 2010). The water parameter was utilized to deposit nanoparticles on the substrate. Each alternative parameter affected the thickness and crystallinity of the produced films and therefore the peak intensities in the XRD patterns changed. Since the ZnO material is very thinly coated in ZnO3 and ZnO4 thin films, no peak is observed in the XRD pattern (Raidou et al., 2014). Raidou et al. (2014) have changed the rinsing period of ZnO films to 20 s, 30 s and 40 s. They looked at the changes in the XRD peaks with the change of time and seen that the crystallinity of the XRD peaks increased with increasing time. The enhancement of the rinsing period was provided more thermal energy (Raidou et al., 2014). There are several factors that increase the reaction rate during thin film production. High temperature causes situations such as increasing chemical reactivity and changing the reaction path in production. This is why good quality films are produced using the temperature parameter. The transition phase from the prepared solution to the thin film form consists of two processes; nucleation and growth. Thin film thickness occurs when small molecules come together to form larger particles during the nucleation stage. In this research, the use of an ultrasonic breaker formed an obstacle for the molecules to come together, so that the desired thickness of the sample did not occur (Rayathulhan et al., 2017).



**Figure 2.** The pattern of XRD analysis of ZnO thin films; a) ZnO1, b) ZnO2, c) ZnO3 and d) ZnO4

Figure 3 depicts the SEM analysis of ZnO thin films. It was observed that the ZnO1 material coated on the glass was thicker and more homogeneous than the other magnifications. Although similar homogeneity was seen in the ZnO2 thin film, the material grew thinner on the surface than the ZnO1 thin film. The surface homogeneity and adhesion of the material on the surface decreased gradually in ZnO3 and ZnO4 thin films, respectively.

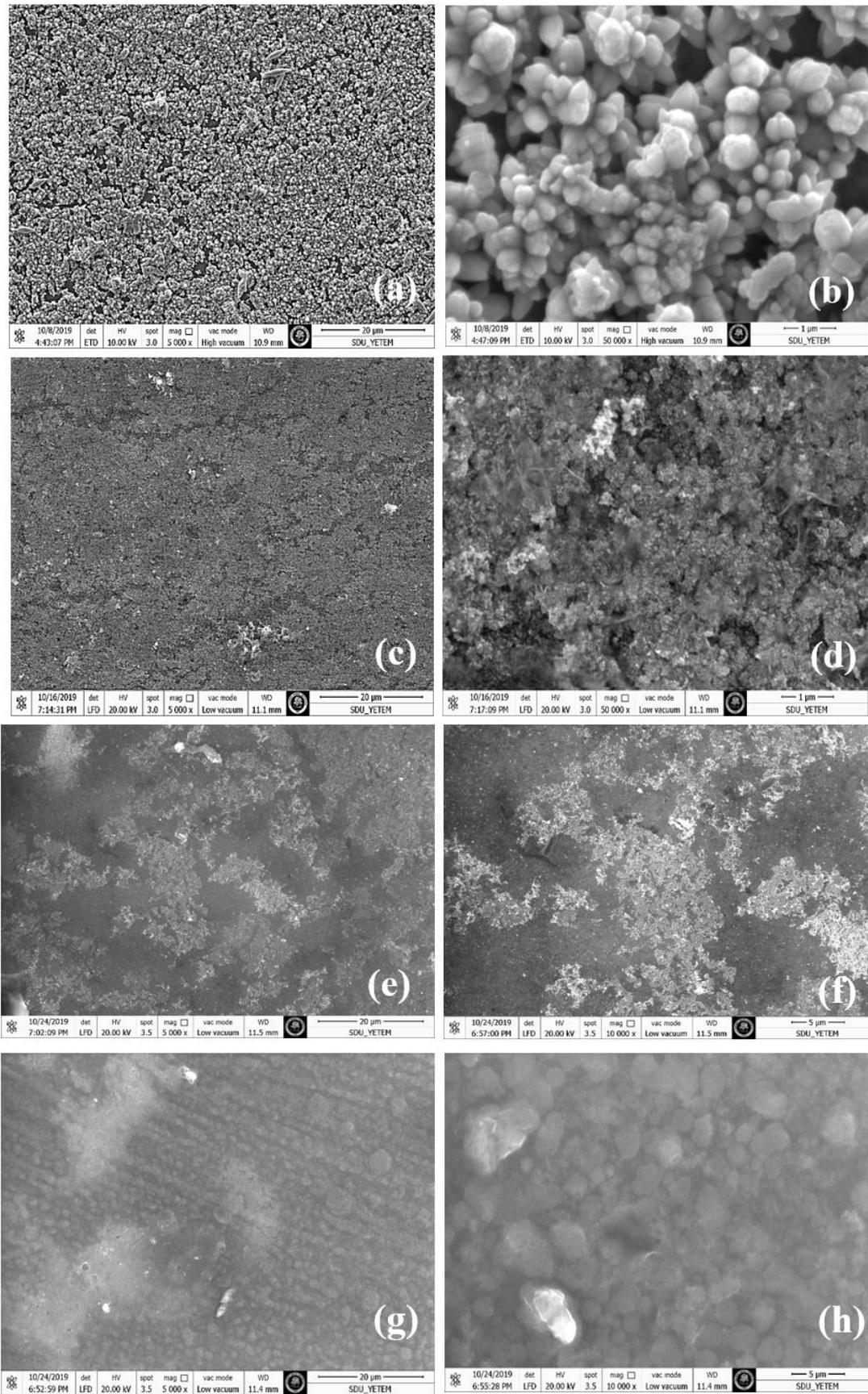
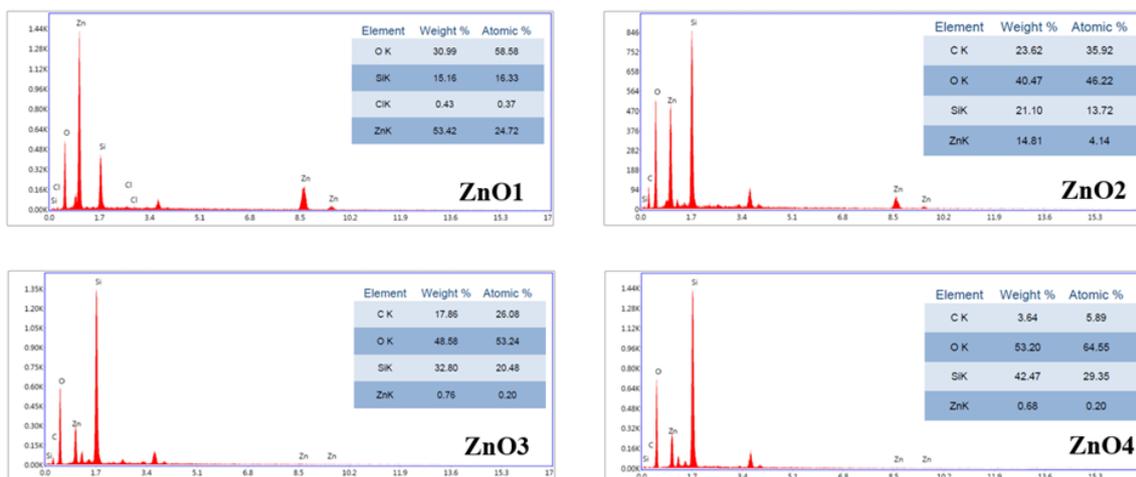


Figure 3. SEM analysis ZnO thin films; a) b) ZnO1, c) d) ZnO2, e) f) ZnO3 and g) h) ZnO4

EDAX analysis was taken to confirm the existence of desired elements in the thin films produced. EDAX spectra of all the synthesized samples are provided in Figure 4. It is important to note that all expected elements (Zn and O in samples) were detected along with the other impurities (Si and C). Changes were observed in the amount of elements depending on the differences used in the hot water parameter. Also, it is thought that the molecules in the solution cannot sufficiently adhere to the surface due to the vibration effect and as a result, the atomic ratio is decreased. Consequently, looking at the article as a whole, it is seen that the results compatible with the classical method are obtained in the sample produced with the help of an ultrasonic cleaner by changing the temperature parameter. It is understood from this that the Zn atoms of the samples produced at room temperature cannot sufficiently adhere to the sample. There are problems in the entry of atoms into the structure due to the low kinetic energy due to the low temperature of the water.



**Figure 4.** EDAX analysis of ZnO thin films; a) ZnO1, b) ZnO2, c) ZnO3 and d) ZnO4

Figure 5 shows the AFM AFM images of ZnO1 (a), ZnO2 (b), ZnO3 (c) and ZnO4 (d). All samples were investigated with using the ez-AFM Nanomagnetics device in  $2 \times 2 \mu\text{m}^2$  area. It was observed that the films, which were given AFM images, respectively, had rough surfaces. With the change of the hot water parameter in the production step, some changes in the surface roughness of the films were observed (Méndez-Vilas et al., 2007). The surface roughness values of each thin film are given in Table 2. These values were found to be compatible with the results of SEM, EDAX and XRD analysis.

Although no clear growth was observed in the SEM and XRD analyses, it can be seen from the AFM results that the samples were formed very thin and the surface was completely amorphous. However, roughness values were obtained from the AFM results, which may indicate some growth in the samples. One pulse vibration will be so strong that the coating has not grown in the desired thickness and homogeneity. It can be attributed that the bonds of the chemicals are broken and there are difficulties in adhering to the surface. When the ZnO2 and ZnO3 samples are compared, the effect of temperature is clearly seen. Desired growth was not observed at room temperature. As the temperature increases, better adhesion to the surface is provided. The best example is ZnO1.

In optoelectronic systems, ZnO lasers are used distance and height measurement systems, medical communication and communication systems. The spectrum of light emitted in semiconductor lasers depends on the material used. They have a very high working speed. Therefore, ZnO is preferred more than other semiconductor types. Also, the electrical properties, atomic structure, and electronic band structure of semiconductors can be associated with the optical properties. Figure 6 shows the absorbance measurements of SILAR-deposited ZnO thin films. ZnO exhibits strong UV absorption spectra with the absorption peak ranging from 360 to 375 nm due to their excitonic transition (Kumar et al., 2014). This is in harmony with the semiconductor behavior (Kumar et al., 2014). ZnO film exhibits high transmittance ( $\sim 75\%$ ) in the visible region, as given in Figure 7. Transmission is inversely proportional to absorbance. As you explained above, the film thickness increases, which means more light is absorbed in the thick film. Therefore, the permeability is reduced.

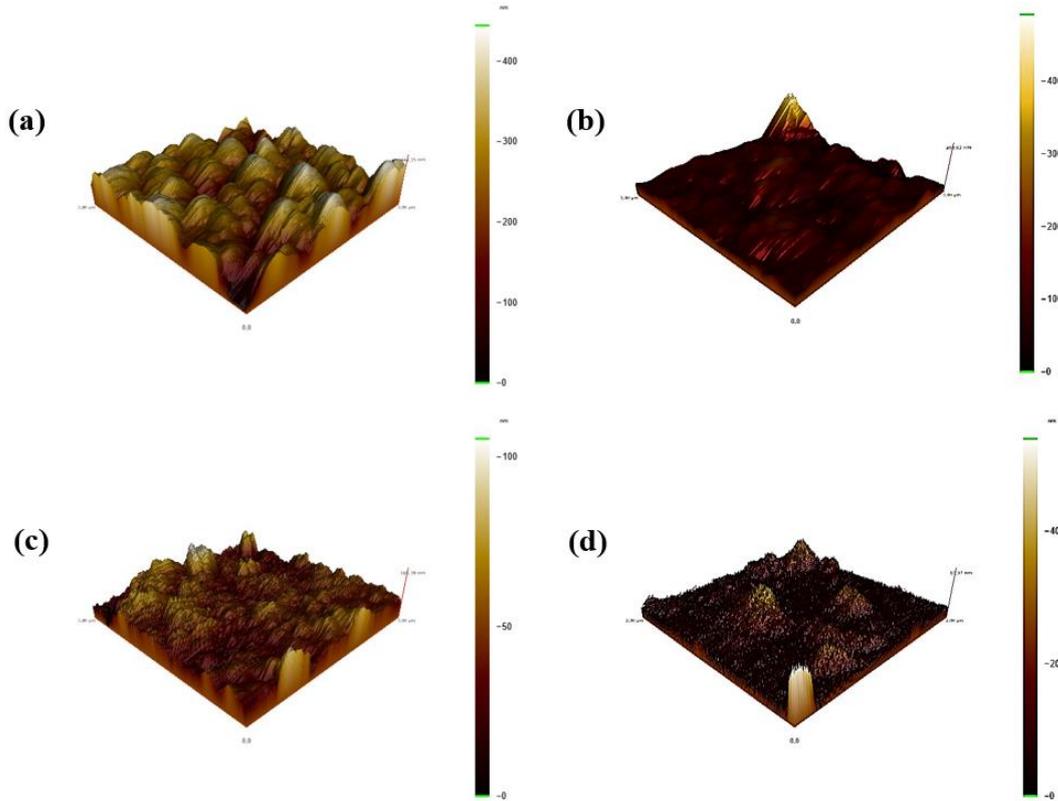


Figure 5. AFM images of a) ZnO1, b) ZnO2, c) ZnO3 and d) ZnO4

Table 2. Surface roughness values of each thin film

	ZnO1	ZnO2	ZnO3	ZnO4
Surface Roughness	46.58 nm	39.84 nm	20.32 nm	19.73 nm

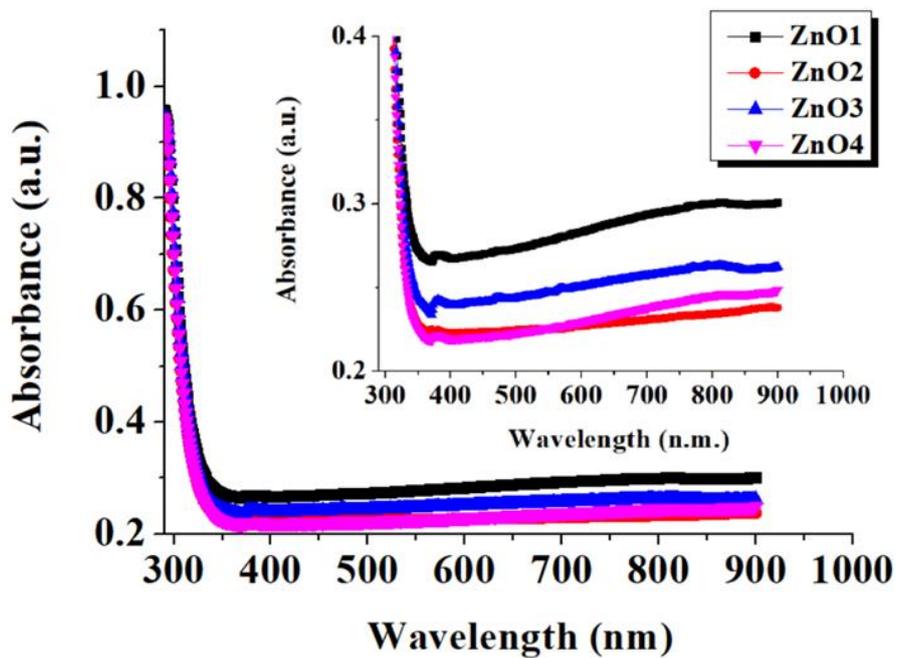


Figure 6. Absorbance spectrum of deposited films

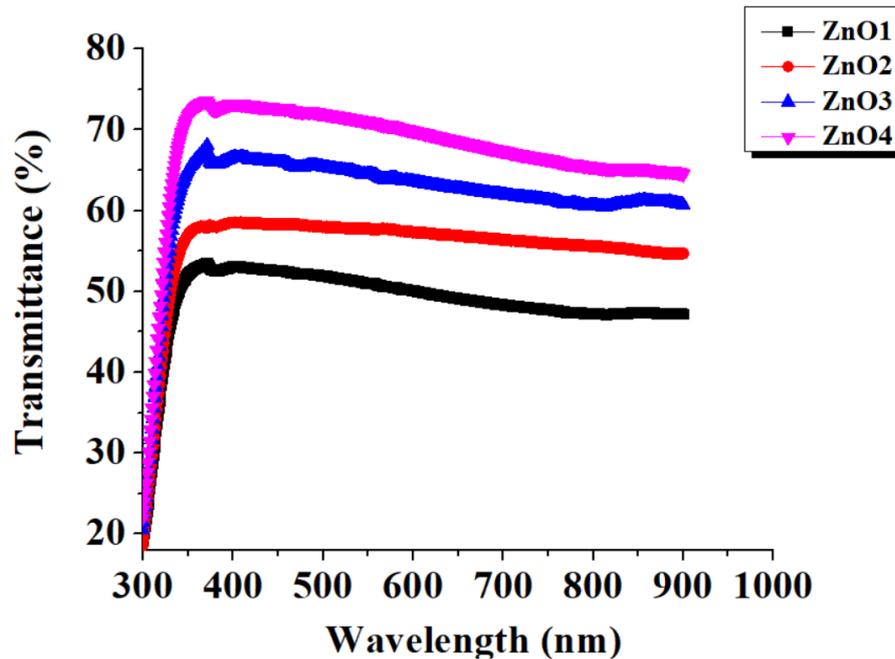


Figure 7. Transmittance spectrum of deposited films

Reflectance can be calculated from the below equation (Komaraiah et. al., 2016);

$$\text{Absorbance} + \text{Transmittance} + \text{Reflectance} = 1 \quad (2)$$

Figure 8 gives the Reflectance spectra of ZnO thin films. When the reflectance values are examined, it is seen that the samples are compatible with each other. As can be seen, there is a reflectance graph for the ZnO2 sample, but it is very low compared to other samples. The peak at 380 nm corresponds to the intrinsic transition of exciton from the ZnO conduction band to valence band. The electron transition from the localized level slightly below conduction band to valence band is associated with the peak at 392 nm, which has been reported by several authors (Fu et al., 1998; Komaraiah et. al., 2016).

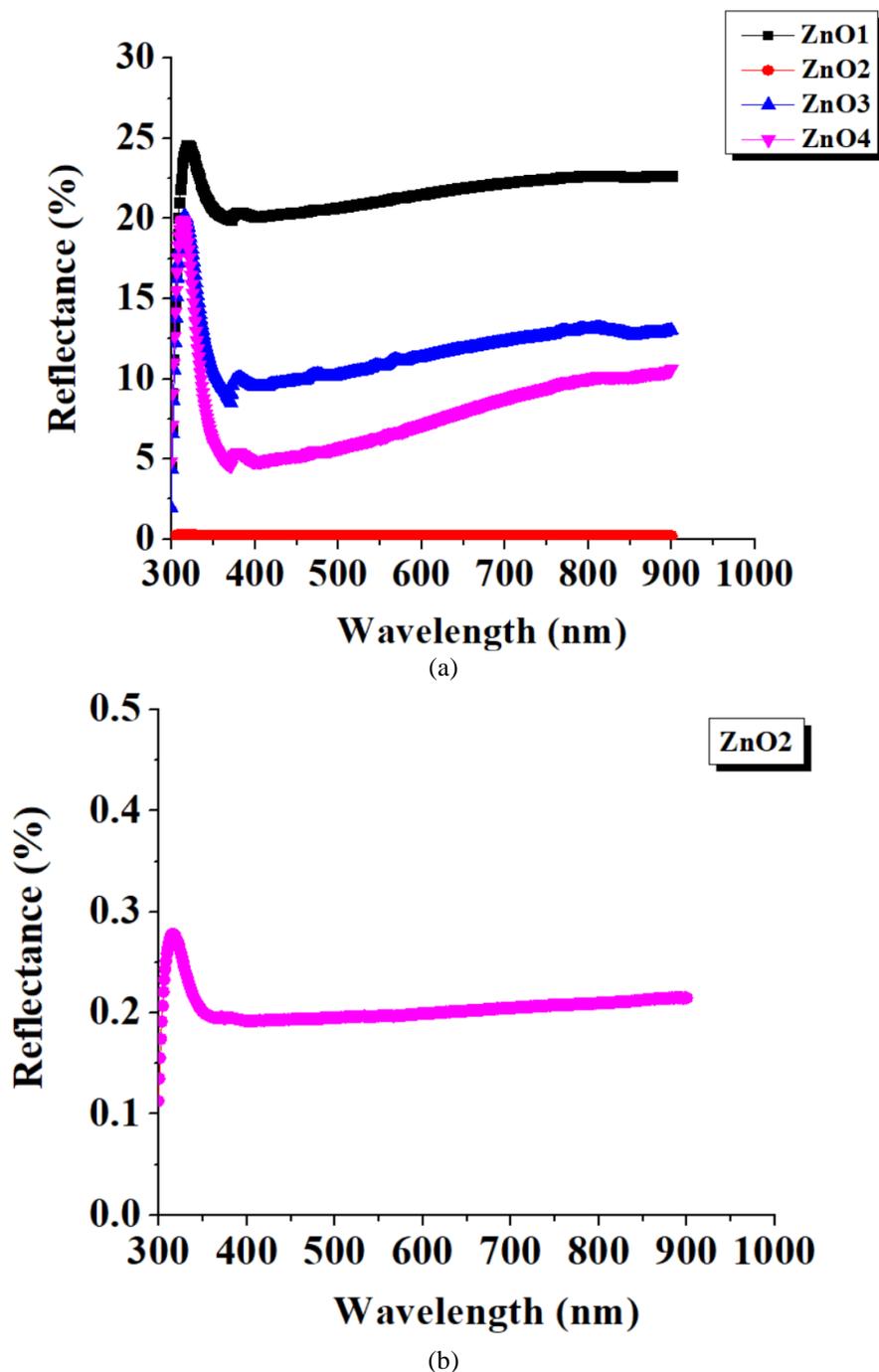
The variation of absorption coefficient ( $\alpha$ ) is showed in Figure 9 and the following equation is being used to compute this parameter (Komaraiah et. al., 2016);

$$\alpha = \frac{2.303}{d} \cdot A \quad (3)$$

Where A is absorbance and d is film thickness. Extinction coefficient (k) analyse is some of the very important parameter in optical-based device design. The following equation is being used to compute this parameter (Tekin and Karaduman Er, 2022);

$$k = \frac{\alpha \lambda}{4\pi} \quad (4)$$

Figure 9 and Figure 10 show the absorption and extinction coefficient of thin films. It is believed that the absorption coefficient has exponential behavior due to random fluctuations of the interior fields in structural disorders.



**Figure 8. a) Reflectance spectrum of ZnO thin films b) Reflectance spectrum of ZnO<sub>2</sub> thin films**

Since band gap change is an important issue in optoelectronic applications, band gap change of thin films has been investigated. Figure 11 shows the plot of  $(\alpha h\nu)^2$  against  $h\nu$ . The nature of the transition was determined by using this relation, Tauc plot (Hammad et al, 2018);

$$\alpha = \frac{A(h\nu - E_g)^n}{h\nu} \quad (5)$$

where the symbols have their usual meanings. The optical band gap energy values of 3.39 eV, 3.40 eV, 3.43 eV and 3.45 eV were estimated ZnO1, ZnO2, ZnO3 and ZnO4, respectively. Variation is observed depending on the thickness and producing hot water parameters, affecting the optical band gap. When examined in the literature, an increase and decrease can be observed depending on the thickness, which is given by Zheng et al. (2022) and Hammad et al. (2018), respectively. Also, Shaba et al. (2021) have reported that synthesis

parameters affecting the properties of zinc oxide and its applications. Habibi et al. (2009) have reported that SILAR-deposited ZnO thin films were investigated by tuning the synthesis parameters and as a result of this, changes in the formation of structures were seen. Also, the quality of ZnO with the reaction time was enhanced in their study.

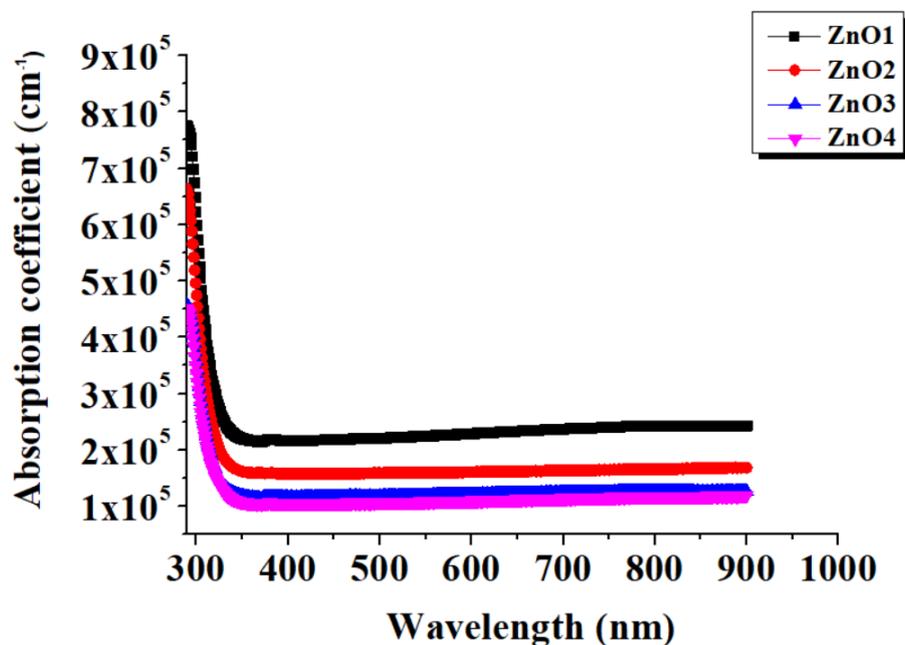


Figure 9. Absorption Coefficient of ZnO thin films

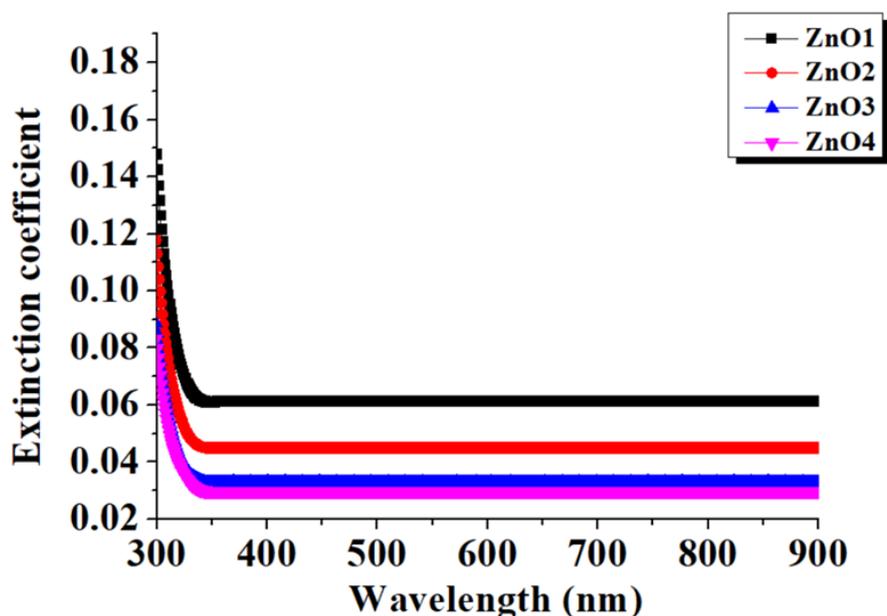


Figure 10. Extinction Coefficient of ZnO thin films

Figure 12 shows the current-voltage characteristics of SILAR-deposited ZnO thin films. All plots in Figure 12 indicate the ohmic behavior which is the nature of semiconductor ZnO thin films. ZnO4 is formed in a more amorphous structure, its electrical resistance is quite high compared to ZnO1, ZnO2 and ZnO3 samples. The resistance of growth samples was calculated as  $1.21 \times 10^5 \Omega$ ,  $1.33 \times 10^5 \Omega$ ,  $9.14 \times 10^5 \Omega$  and  $2.48 \times 10^9 \Omega$  for ZnO1, ZnO2, ZnO3 and ZnO4, respectively. The obtained resistance values are in good agreement with the previously

reported results (Gurav et al., 2010; Jambure et al., 2014). Table 3 shows the parameters of each thin film with morphological, optical and electrical results.

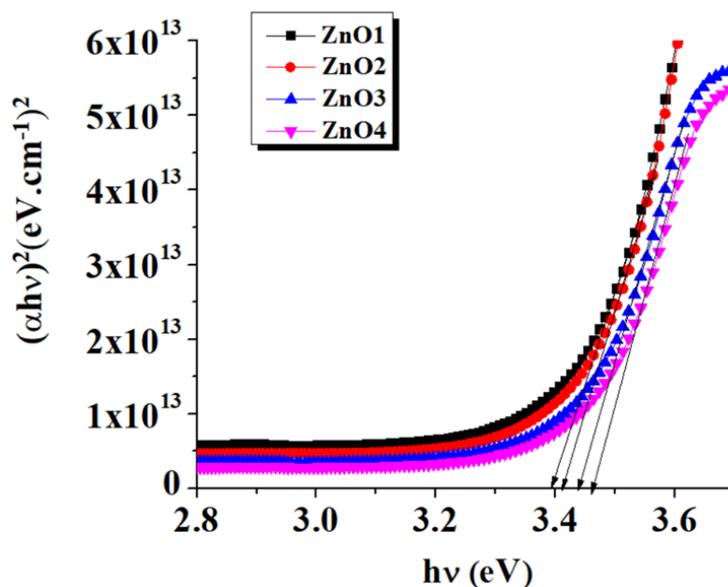


Figure 11. The plot of  $(\alpha hv)^2$  against  $h\nu$  of ZnO thin films

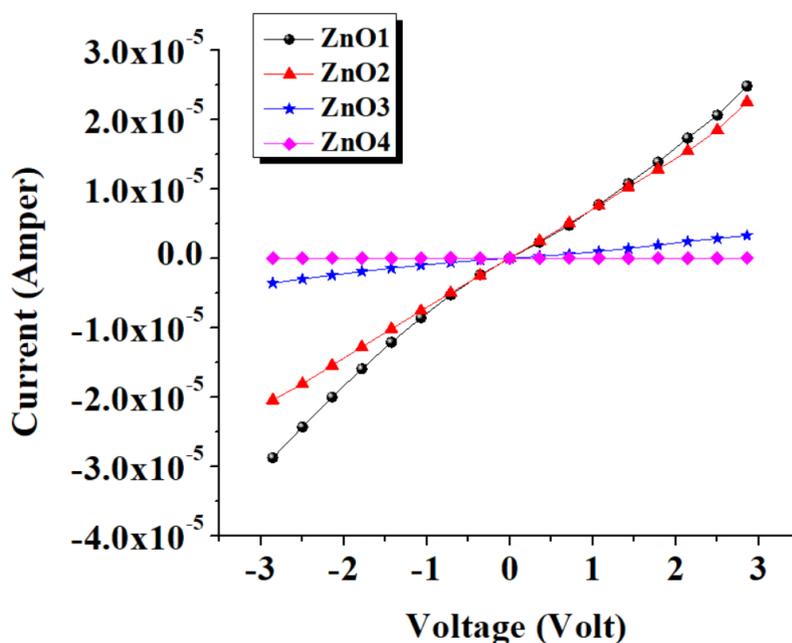


Figure 12. The I-V plots of ZnO thin films

Table 3. The parameters of each thin film with morphological, optical, electrical results

	ZnO1	ZnO2	ZnO3	ZnO4
Thickness (nm)	375	390	400	415
Optical Band Gap (eV)	3.39	3.40	3.43	3.45
Electrical Resistance ( $\Omega$ )	$1.21 \times 10^5$	$1.33 \times 10^5$	$9.14 \times 10^5$	$2.48 \times 10^9$

#### 4. CONCLUSION

In this work, ZnO material was grown by changing the second parameter of the classical SILAR method on glass substrates and the structural characterizations of the grown thin films were investigated. Adhesion effects of the material to be coated on the substrates were investigated by using hot water, ultrasonic cleaner and ultrasonic breaker, respectively, during magnification. It was observed that the dominant peak intensities also changed with the changed SILAR parameter in the thin films whose XRD patterns were examined. While the crystal phase orientation intensities were the highest in the ZnO1 thin film, the orientation intensity decreased in the ZnO2 thin film, and no peak was observed in the ZnO3 and ZnO4 thin films. Each value was found to be compatible. Optical analysis shows that thin films have high transmittance values, compared to absorbance and reflectance. Despite the fact that alternative devices such as ultrasonic cleaner and ultrasonic breaker were used to remove unwanted weak bonds at lower temperatures during production, The desired results could not be obtained and the samples could not adhere to the surface. It is thought that these vibrations cause excessive breakage and prevent the adhesion of Zn and O molecules. Different from the classical SILAR method production method, when the samples produced at 40 °C with the help of an ultrasonic cleaner are examined in detail, structural, optical and electrical results compatible with the literature have been obtained and a new approach has been brought to the SILAR method production.

#### CONFLICT OF INTEREST

The authors declare no conflict of interest.

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