Production of PbO thin film using Silar method and electronic and interface properties of Pb/PbO/p-Si MIS contacts

E. Erdem^{a,}, S. Asubay^b, O. Gullu^{c,*}

^aBatman University, School of Graduate Studies, Branch of Physics, Batman, Turkey

^bDicle University, Faculty of Sciences, Department of Physics, Diyarbakir, Turkey ^cBatman University, Faculty of Sciences and Arts, Department of Physics, Batman, Turkey

In this paper, using SILAR thin film technique, which is low cost and is easy to control, the lead oxide (PbO) thin film was grown onto both microscope glass and inorganic semiconductor silicon (Si) wafer after appropriate chemical cleaning processes. The coating stage was performed by keeping the solution at 80 °C. After forming a PbO thin film on the silicon semiconductor, Pb metal was evaporated onto its upper surface. Optical, morphological and structural properties of the PbO thin film formed on glass were investigated. The electrical and interface characteristics of the Pb/PbO/p-Si MIS Schottky diode were investigated in the dark by using current-voltage (I-V), capacity-voltage (C-V) and conductance-voltage (G-V) in 10kHz-2MHz frequency range and capacity-frequency (C-f) characteristics in 1kHz-10MHz frequency range.

(Received October 12, 2021; Accepted January 25, 2022)

Keywords: Diode, MIS, PbO, Schottky, SILAR

1. Introduction

Thin films, which are within the scope of nanotechnology and form the basis of electronic device technologies, have a wide range of research and studies today. The performance provided by thin films, which have a high usage area, is the main parameter. The achieved performance is directly related to the type of material used and various production techniques [1].

When the semiconductors in thin-film technology are examined from a technological and scientific point of view, we can see that metal oxides cover an important material class among semiconductors. In this field, recent studies and findings have focused on metal oxide thin films [2]. Metal oxide semiconductors are technologically significant materials that yield many application areas [3].

Due to their performance in the fields of use in engineering sciences, metal oxides have provided a significant area of use in physics, chemistry, and materials science. Nanostructured metal oxides are used in many areas such as semiconductor devices, sensors, piezoelectricity, fuel cells, microelectronic circuits, batteries, surface passivation, and catalyst applications for resistance to abrasion, pollution removal, polymer UV resistance and durability, and magnetic drug carrier in cancer treatment. One of the metal oxides that have a lots of usage areas is lead oxide (PbO). Lead oxide has a great usage area in the automotive industry as an electroactive element in lead-acid batteries. In recent years, it has been observed that when lead oxides, formed by traditional methods, are brought to the nanoscale, they provide advantages such as more energy capacity and longer cycle life. For this reason, lead oxide battery applications with economical advantages and high reliability gain importance. Lead oxide is active in many and different fields, such as the production of pigment, lead glass, glaze, ceramics, cathode ray tube glass production, the development of the electromechanical properties of ceramics, and its use as an active catalyst in chemical reactions, as well as in energy applications [4].

^{*} Corresponding author: omergullu@gmail.com https://doi.org/10.15251/JOR.2022.181.44

In this study, lead oxide (PbO) was grown on microscope glass with the SILAR (Successive Ionic Layer Adsorption and Reaction) method, a thin film production technique, and its structural, morphological, and optical properties were examined. In addition, the current-voltage (I-V), capacitance-voltage (C-V), conductance-voltage (G-V), capacitance-frequency (C-f) measurements of the produced Pb/PbO/p-Si MIS (Metal-Interlayer- Semiconductor) Schottky diode were performed depending on the frequency at room temperature and in the dark.

2. Experimental Procedures

2.1. The materials used in the study and the preparation stages

Our scientific research is a bidirectional study on glass and silicon crystals. In the first phase of this study, a glass substrate was used. To remove organic and heavy metal contaminations on the glass substrate surface, it was kept in deionized water diluted with 10% hydrofluoric acid (HF) in an ultrasonic bath for 5 minutes. Afterwards, it was rinsed in deionized water. The rinsing process was repeated three or four times, the substrate did not need to be dried since the study would occur in a liquid environment.

To prepare Pb/PbO/p-Si Schottky diodes, p-type Si substrate with (100) orientation, 400 μ m thickness, 1-10 Ω .cm resistivity was used. To get rid of the native oxide layer on the crystal, the shiny surface of the crystal was cleaned by deionized water diluted with 10% HF with the help of cotton, and then the crystal was rinsed in deionized water for 5 minutes and made ready for coating.

2.2. Preparation of experiment solution

During the experimental studies, high purity lead acetate trihydrate salt (Pb(CH₃COO)₂ · $3H_2O$) purchased from Sigma-Aldrich was used. To prepare the solution, lead acetate trihydrate (molecular weight 380 g/mol) as starting material, was dissolved in 50 ml of deionized water. After the solution was stirred for 10 minutes with a magnetic stirrer, 5 ml of ammonium hydroxide (NH₄OH) was added and stirring was continued (the solution turned to yellow). In the second step of solution preparation, 5 ml of hydrogen peroxide was added to 50 ml of deionized water to prepare 2% hydrogen peroxide (H₂O₂). Therewithal, 50 ml of deionized water was added to two separate beakers, and then the chemical coating process was started. All of the starting solutions are prepared with deionized water. Deionized water and lead acetate trihydrate salt for the solutions, the stirring process was carried out for a constant 10 minutes for each solution. The lead acetate solution was prepared as 0.1 M.

After preparing the solutions, the materials were kept in a heater at 80 $^{\circ}$ C for 10 minutes so that the dissolution could occur and the materials used in the coating were at the same temperature. Then the coating stage was started, the temperature was not changed during the coating.

2.3.Film coating

After preparing the solution to be coated, the coating process was first put on the glass substrate. The solutions placed in four separate beakers were arranged side by side as lead acetate trihydrate solution with ammonium hydroxide added on the far left, deionized water next to it, hydrogen peroxide solution next to deionized water, and deionized water on the far right.

A SILAR growth method for the formation of PbO thin films includes the following steps:

1- First, the cleaned glass substrate is immersed in the beaker containing lead ions and liquid ammonia for 15 seconds, so that the lead complex ions are absorbed onto the glass substrate.

2- The substrate is then immersed in the beaker containing deionized water for 10 seconds so that the loosely coupled lead complex ions are separated from the glass substrate surface.

3- The substrate is then immersed for 15 seconds into a beaker containing a 2% hydrogen peroxide solution (5ml) dissolved in 50ml water, where lead complex ions react with H_2O_2 to form lead oxide.

4- Finally, the substrate is immersed in a beaker containing deionized water for 10 seconds to separate loosely coupled PbO ions.

Coatings were obtained after 25 rounds of cycles. The films were annealed at 300 °C for 30 minutes to remove any hydroxide phase present. The thickness of the obtained film was calculated as $5.1 \,\mu$ m from SEM (Scanning Electron Microscopy) measurements.

The crystal structure and surface morphology of the films were analyzed with the help of XRD and SEM-EDS devices. The optical properties of the films were measured with a UV optical spectrometer.

2.4. Preparation of Pb/PbO/p-Si Schottky diode

At this stage, the p-Si crystal, whose properties were specified in the previous sections, was first subjected to the classical chemical cleaning process. Then, high purity Al metal was coated on the matte surface of the p-Si crystal in the PVD system for ohmic contact. At this stage, it was heat-treated at 570 °C for 3 minutes in a nitrogen gas atmosphere in the furnace for ohmic contact formation. Then, the shiny surface of the p-Si/Al substrate was coated with PbO using the 25-round SILAR deposition method, the same as the treatment on glass. Since we did not want the Al-coated matte surface of the crystal to be coated, the matte surface was cleaned with cotton every 5 cycles. After coating, it was annealed at 300 °C for 30 minutes under normal atmospheric conditions. In the last stage, with the help of a tungsten filament obtained by a physical evaporation method, under ~ $4.09.10^{-5}$ torr pressure and through which ~30A current is passed, obtained by vacuum system, highly pure (~99.99%) lead (Pb) is applied to the PbO coated surface of the substrate. A thin Pb layer was formed by evaporating the metal. In this way, Pb/PbO/p-Si/Al MIS diode structure was obtained.

3. Research Findings and Discussion

3.1. X-ray diffraction (XRD) measurements of PbO film

X-ray analysis of the PbO thin film obtained on the glass substrate using the SILAR technique was made by the Rigaku Ultima IV X-ray diffractometer. The X-ray diffraction pattern of the PbO thin film grown on a glass substrate is given in Figure 1.



Fig. 1. X-ray diffraction pattern of PbO thin film grown on glass using the SILAR method

When the diffraction pattern was analyzed, it was observed that the PbO thin film produced was in the form of PbO and Pb_2O_3 crystal phases at the peaks indicated in the figure, and it was compatible with the literature [5].

3.2. SEM-EDS analysis of PbO film

Scanning Electron Microscope (SEM) images and Energy Dispersive Spectrometry (EDS) spectrum of the PbO thin film obtained using the SILAR method were taken with the help of the JEOL brand SEM device.

The Scanning Electron Microscope (SEM) image of the PbO film is given in Figure 2. In this image, it was determined that leaf-like nanostructures were formed on the PbO surface and this shape was consistent with similar results in the literature [6].



Fig. 2. SEM image of PbO (Lead Oxide) film

The EDS spectrum of the PbO thin film is given in Figure 3. When the EDS plot was analyzed, the presence of lead and oxygen in the thin film formation was obviously seen.



Fig. 3. EDS spectrum of the lead oxide film.

3.3. Optical measurements of the film (UV-Visible and PL -Photoluminescence)

The bandgap value of the obtained PbO thin film was calculated with the following equation.

$$Ah\nu = C(h\nu - E_g)^m \tag{1}$$

In this equation, m is an exponential constant and takes the value $\frac{1}{2}$ in allowed direct passes. E_g is the bandgap energy, v is the frequency, h is the Planck's constant, C is the proportionality constant, and A is the absorption coefficient [2].

The absorption spectrum of the PbO film was measured in the wavelength range of 200 nm to 1100 nm using the Shimadzu 3600 UV-Vis spectrophotometer, and a graph as shown in Figure 4 appeared. This absorption spectrum is in agreement with previous studies for PbO.



Fig. 4. The absorption-wavelength plot of the obtained PbO thin films.

In the equation (1) of the absorption-wavelength graph of the obtained PbO thin films, the m value represents 1/2 direct allowed transitions, and m=1/2 is taken as $(Ahv)^2$ -hv graph. This graph is given in Figure 5. In the given figure, the extension of the linear part of the graph for $(Ahv)^2 = 0$ was found, and it was determined that the forbidden energy band gap of PbO was $E_g=2.96 \text{ eV}$. This value is in agreement with the values given for PbO in the literatüre.



Fig. 5. Variation of the obtained film $(\alpha hv)^2$ *with respect to the photon energy (hv).*

PL measurement of PbO film was obtained by using the Perkin Elmer LS 55 device. The PL spectrum of the PbO thin film is given in Figure 6. PL is affected from the chemical structure, defects in the crystal, optical stimulation states of the materails [7]. The peak occurring around 630 nm for PbO in this measurement is in agreement with the results in the literatüre.



Fig. 6. PL spectrum of the obtained PbO film.

3.4. Electrical characterization of Pb/PbO/p-Si Schottky diode

3.4.1. Current-voltage characteristics of the produced diode

Current-voltage measurements of the produced Pb/PbO/p-Si/Al MIS diode were taken at room temperature and in the dark by help of the KEITHLEY 4200-SCS device.

The current-voltage characteristic of the diode is calculated with the help of the equations given below.

$$I = I_{o} \left[exp \left(\frac{qV_{D}}{nkT} \right) - 1 \right]$$
⁽²⁾

In the case of $qV_D >> nkT$ in this equation, the term 1 can be neglected alongside the exponential term.

The new version of the equation takes the form [8];

$$I = I_{o} \left[exp\left(\frac{qV_{D}}{nkT}\right) \right]$$
(3)

If the natural logarithm of both sides of (3) is taken and then the derivative with respect to V_D , the new equation for the ideality factor is given as;

$$n = \left(\frac{q}{kT}\right) \frac{dV}{d\ln(I)} \tag{4}$$

In the above equations; *n* is the ideality factor, V is the voltage of the diode, q is the electron charge, k is the Boltzmann constant (8.625×10^{-5} eV/K), T is the temperature in Kelvin. I_o is the reverse bias saturation current, and given as;

$$I_{o} = AA^{*}T^{2} \exp\left(-\frac{q\Phi_{B}}{kT}\right)$$
(5)

Taking the natural logarithm of both sides of this equation, it can be given as;

$$\Phi_{\rm B} = \frac{kT}{q} \ln \left(AA^* T^2 / I_0 \right) \tag{6}$$

In this expression, the area of the A diode (A= 0.00785 cm^2) is A* Richardson's constant and its value is A*= $32 \text{ AK}^- \text{ }^2\text{ cm}^- \text{ }^2$ for p-Si [8]. The saturation current density and Schottky barrier height can be calculated by equations (5) and (6), respectively.



Fig. 7. I-V graphs of Pb/p-Si reference without interfacial PbO layer and Pb/PbO/p-Si diode with PbO interface at room temperature.

To see the effect of the PbO thin film layer, the IV characteristics of the Pb/p-Si reference diode structure and the Pb/PbO/p-Si MIS structure were in the range (-2 V)-(+1 V) at room temperature and dark. They were measured in 0.02V steps and its graphs are shown in Figure 7. When the I-V graphs were examined, it was observed that the current decreased by more than 10 times in reverse and forward bias characteristics compared to the reference diode. This reduction is due to the formation of the natural oxide layer and the interfacial layer at the interface, as the PbO thin film layer exhibits an additional resistance effect between the Pb metal and p-Si.

The barrier height of the reference Pb/p-Si diode in Figure 7 was found to be $\Phi_B=0.701$ eV and the ideality factor n=1.68. The barrier height of the Pb/PbO/p-Si diode was calculated as $\Phi_B=0.780$ eV and the ideality factor n=2.06. These values are given in Table 1.

The fact that the ideality factor is greater than 1 in the produced diodes generally arises from the interfacial oxide layer, surface/interface states and series resistance (R_s) effect that may occur during production [8-11]. The barrier height of the Pb/PbO/p-Si MIS diode is approximately as 79 meV higher than that of the Pb/p-Si reference diode, which can be explained by the change in the charge distribution in the space charge region of the diode due to the presence of the PbO interfacial oxide layer.

In the above sections, it was stated that one of the reasons for the deviation of the diodes from the ideal state is the series resistance effect. The current through the diode under the influence of the series resistance (R_s);

$$I = I_{o} \exp\left(\frac{q(V - IR_{s})}{nkT}\right)$$
(7)

is expressed by this equation. One of the methods used to calculate values such as barrier height, ideality factor and series resistance of the diode is the Norde functions produced by Norde (1979) [12].

$$F(V) = \frac{V}{\gamma} - \frac{kT}{q} \left(\frac{I(V)}{AA^*T^2} \right)$$
(8)

Hereby, the value of γ is the first integer greater than the ideality factor. The I(V) value is the current value obtained from the I-V graph.

Ideality factor;

$$\mathbf{n} = \left(\frac{\mathbf{q}}{\mathbf{k}\mathbf{T}}\right) \frac{\mathrm{d}\mathbf{V}}{\mathrm{d}\ln(\mathbf{I})} \tag{9}$$

The barrier height expression calculated with the Norde method;

$$\Phi_{\rm B} = F(V_{\rm o}) + \frac{V_{\rm o}}{\gamma} - \frac{kT}{q}$$
(10)

In this equation, the expression $F(V_o)$ is the minimum F(V) value of the F-V graph. Series resistance expression given as;

$$R_s = \frac{kT(\gamma - \mathbf{n})}{qI} \tag{11}$$



Fig. 8. F(V)-V graphs of the Pb/PbO/p-Si MIS diode and Pb/p-Si reference diode.

F(V)-V graphs obtained from the Norde functions, the barrier height value of the reference Pb/p-Si diode is $\Phi_B=0.744 \text{ eV}$, the series resistance value is $R_s=6.282 \text{ k}\Omega$, the barrier height value of the Pb/PbO/p-Si diode is $\Phi_B=0.841 \text{ eV}$, the series resistance value is calculated as $R_s=2.726 \text{ k}\Omega$. These values are given in Table 1.

The second method to find values such as barrier height, series resistance and ideality factor of a diode in series resistance is the Cheung method and this method was developed by Cheung (1986) [13].

$$\frac{\mathrm{d}\mathbf{v}}{\mathrm{d}(\ln I)} = \frac{\mathrm{n}\mathrm{k}\mathrm{T}}{\mathrm{q}} + \mathrm{I}.\,\mathrm{R}_{\mathrm{s}} \tag{12}$$

$$H(I) = n\phi_B + I.R_s$$
(13)

The Cheung equations are given as above.

Figure 9 shows the dv/d(lnI)-I graphs of the Pb/PbO/p-Si MIS diode and Pb/p-Si reference diodes. The series resistance of the Pb/Si reference diode was calculated as $R_s=1.178k\Omega$ from the slope of the linear lines in these graphs. According to Cheung's method, the ideality factor was calculated as n=1.70 by using the point where the linear line intersects the vertical axis. Similarly, the series resistance of the Pb/PbO/p-Si Schottky diode was calculated as $R_s=4.233k\Omega$ and the ideality factor n=1.97 according to the Cheung function. These values are given in Table 1.



Fig. 9. dV/d(lnI)-I graphs of Pb/PbO/p-Si MIS diode and Pb/p-Si reference diode.

The H(I)-(I) graphs obtained using the Cheung method are shown in Figure 10. From these graphs, the series resistance value of Pb/p-Si reference diode is calculated as R_s =1.259 k Ω , barrier height Φ_B =0.782 eV, while series resistance value of Pb/PbO/p-Si MIS diode as R_s =3.456 k Ω , barrier height Φ_B =0.809 eV. These parameters are given in Table 1.



Fig. 10. H(I)-I graphs of Pb/PbO/p-Si MIS diode and Pb/p-Si reference diode.

 Table 1. Diode parameters obtained from I-V, Cheung and Norde functions of Pb/p-Si

 reference diode and Pb/PbO/p-Si MIS diode.

	Cheung Function							Norde Fun	ction
	dV/d(lnI)-I		H(I)-I		I-V		$\mathbf{F}(\mathbf{V}_0)$ - \mathbf{V}_0		
Sample	$R_{s}(k\Omega)$	n	$\mathbf{R}_{s}\left(\mathbf{k}\Omega\right)$	$\phi_{B}\left(eV ight)$	n	$\phi_{B}(eV)$	I ₀ (A)	$R_{s}(k\Omega)$	$\phi_{B}(eV)$
Pb/p-Si	1.178	1.70	1.259	0.782	1.68	0.701	3.28x10 ⁻⁸	6.282	0.744
Pb/PbO/p-Si	4.233	1.97	3.456	0.809	2.06	0.780	1.91x10 ⁻⁹	2.726	0.841

3.4.2. Capacitance-voltage characteristics of the produced diode

The capacity of the metal-semiconductor rectifier contact in the literatüre,

$$C = A \left(\frac{\varepsilon_s \varepsilon_o N_a}{2}\right)^{\frac{1}{2}} \left(V_d - \frac{kT}{q}\right)^{\frac{1}{2}}$$
(14)

is given by the above equation. In this expression, $k=8.625 \times 10^{-5}$ eV/K (Boltzmann constant), \mathcal{E}_s dielectric constant of semiconductor ($\mathcal{E}_s=11.8$ for p-Si), \mathcal{E}_o dielectric constant of vacuum ($\mathcal{E}_o=8.85 \times 10^{-12}$ F/cm), q electron charge, V_d diffusion potential, N_a ionized acceptor density and T is the temperature in Kelvin [8]. The C-V measurements of the Pb/PbO/p-Si MIS diode are at room temperature (-2V)-(+2V) voltage range in 0.02V steps and respectively taken for 10 kHz, 20 kHz, 30 kHz, 40 kHz, 50 kHz, 60 kHz, 70 kHz, 80 kHz, 90 kHz, 100 kHz, 200 kHz, 300 kHz, 400 kHz, 500 kHz, 700 kHz, 800 kHz, 900 kHz, 1 MHz and 2 MHz frequency values and shown in Figure 11. It is seen that the capacity decreases as the frequency increases. This is explained by the inability of the interface traps to follow the applied signal and the corresponding decrease in C values.

Equation (14) by arranging the equation;

$$C^{-2} = \frac{2(V_{\rm d} + V)}{\varepsilon_{\rm s}\varepsilon_{\rm o}qA^2N_{\rm a}}$$
(15)

the above equation is obtained. The V is the applied voltage. According to Equation 15, the graph of C⁻ ²-V in the reverse bias region should be linear and at the same time, $V_d = V$ for $C^{-2} = 0$. Therefore, the diffusion potential can be obtained from the C⁻ ²-V graph. C²-V graphs of Pb/PbO/p-Si MIS diode are shown in Figure 11. Diffusion potentials calculated at different frequencies at room temperature using this graph are available in Table 2.

However, if the derivative of the expression (15) according to V is taken,

$$\frac{d(C^{-2})}{dV} = \frac{2}{\varepsilon_s \varepsilon_o q A^2 N_a}$$
(16)

the above equation is obtained. If this expression is rearranged to N_a,

$$N_a = \frac{2}{\varepsilon_s \varepsilon_o q A^2 \frac{d(C^{-2})}{dV}}$$
(17)

the above correlation is obtained. Therefore, the carrier density can be calculated from the slope of the C⁻ ²-V graph. The carrier density values calculated from the slope of the C⁻ ²-V graph of the Pb/PbO/p-Si MIS diode are given in Table 2 depending on the frequency.

Fermi energy level of the MIS structure is given as:

$$E_{f} = kT ln(N_{v}/N_{a})$$
(18)

where N_v is the effective charge state density in the valence band of a p-type semiconductor in thermal equilibrium. Fermi energy level in the form of equation (18) is obtained. The E_f value can be calculated by substituting the N_a value reached by the slope of the C⁻ ²-V graph. In addition, the barrier height can be calculated by calculating the difference between the valence band and the Fermi level using the V_d value and the acceptor density obtained from the slope, by determining the point where the C⁻ ²-V graphs of the diode obtained at different frequencies cut the V axis. However, considering the energy-band diagram of the metal/semiconductor contact, the barrier height is given as;

$$\Phi_{\rm B} = E_{\rm f} + V_{\rm d} \tag{19}$$

In this way, Φ_B (barrier height), N_a (carrier density), V_d (diffusion potential) and E_f (Fermi energy level) values of the Pb/PbO/p-Si diode were calculated from the C⁻²-V curves and these values are given in Table 2.



Fig. 11. C-V and 1/C²-V graphs of Pb/PbO/p-Si Schottky MIS diode in the frequency range of 10 kHz-2 MHz.

Frequency (kHz)	<i>C-V</i>							
	$\Phi_B(eV)$	$N_a (\times 10^{15} cm^{-3})$	$V_d(V)$	$E_f(eV)$				
10	0.766	2.268	0.548	0.218				
20	0.870	2.144	0.651	0.22				
30	0.920	2.097	0.700	0.22				
40	0.946	2.078	0.725	0.22				
50	0.963	2.057	0.742	0.221				
60	0.973	2.048	0.752	0.221				
70	0.978	2.033	0.757	0.221				
80	0.994	2.06	0.773	0.221				
90	1.001	2.057	0.78	0.221				
100	1.008	2.074	0.788	0.22				
200	1.042	2.055	0.821	0.221				
300	1.061	2.048	0.84	0.221				
400	1.073	2.024	0.852	0.221				
500	1.082	1.999	0.861	0.221				
600	1.09	2.002	0.869	0.221				
700	1.097	1.954	0.875	0.222				
800	1.103	1.959	0.881	0.222				
900	1.108	1.973	0.886	0.222				
1000	1110	1.966	0.888	0.222				
2000	1.130	1.958	0.908	0.222				

 Table 2. Diode parameters obtained from the C⁻²-V curves extracted from the C-V measurements of the Pb/PbO/p-Si MIS diode.

3.4.3. Conductance-voltage characteristics of the produced diode

One of the conditions affecting the parameters obtained from the C-V calculations is the physical condition of the interface used. It is seen in Figure 12 that as the frequency increases, the conductance value increases in direct proportion to the voltage.



Fig. 12. Conductance-Voltage (G-V) graphs of Pb/PbO/p-Si diode.

3.4.4. Capacitance-frequency (C-f) characteristics of the produced diode

C-f measurements of the Pb/PbO/p-Si diode were taken at a constant voltage values of 0.04 V in steps between 0 V and 0.72 V in the range of 1kHz-10MHz. As can be seen in Figure 13, the capacitance value decreases as the frequency increases.



Fig. 13. C-f measurements of Pb/PbO/p-Si diode at different voltage values.

This situation is attributed to the inability of the interface traps in the depletion region to follow the applied signal and the corresponding decrease in C capacitance values.

4. Conclusion

At the first stage of this study, the optical, structural and morphological properties of the PbO thin film formed on glass using the SILAR method were examined. When the X-ray

diffraction pattern of the structural analysis of the PbO thin film grown on the glass substrate using the SILAR technique was analyzed, it was seen that crystal phases in the form of PbO and Pb₂O₃ were formed in the structure. SEM-EDS analysis of the PbO thin film obtained using the SILAR method were performed. When the SEM image of the PbO film was analyzed, it was determined that leaf-like nanostructures were formed on the PbO surface in this image and this shape was consistent with similar results in the literature. In addition, when the EDS spectrum of the PbO thin film was analyzed, the presence of lead and oxygen was clearly seen in the formation of the thin film. The optical absorption spectrum of the PbO film was measured in the wavelength range of 200 nm to 1100 nm. It was determined that the bandgap of the forbidden energy of PbO belonging to the direct allowed transitions is $E_g=2.96$ eV. In addition, the PL measurement of the PbO film was taken and the peak, which appeared around 630 nm for PbO in the spectrum obtained, was found to be consistent with the results in the literature.

In the second stage of this study, the Pb/PbO/p-Si (MIS) diode was produced and the electrical and interfacial properties of this MIS structure were investigated. To see the effect of the PbO interfacial layer, the I-V characteristics of the Pb/p-Si reference diode and the Pb/PbO/p-Si MIS structure were measured, and as a result of the analysis of the I-V graphics, it was observed that the current decreased more than 10 times in the reverse and forward bias characteristics compared to the reference diode. The decrease is attributed to the fact that the PbO thin film layer exhibits an additional resistance effect between the Pb metal and the p-Si semiconductor, the natural oxide layer at the interface and the formation of the interfacial layer.

In our study, the PbO thin film used in the production of Schottky diodes was formed by the SILAR method, so our study is the first in the literature.

References

[1] S. Sonmezoglu, M. Koc, S. Akin, Institute of Science and Technology 28(5), 389 (2012).

[2] H. Cavusoglu, European Journal of Science and Technology 15, 412 (2019).

[3] H. Kung, 1989, Transition Metal Oxides: Surface Chemistry and Catalysis. Elsevier, Amsterdam; <u>https://doi.org/10.1016/S0167-2991(08)60923-4</u>

[4] B. Asik, 2012, Production and Characterization of Nano Structured Lead Oxide by Ultrasonic Spray Pyrolysis (USP) Method, Master Thesis, Istanbul Technical University, Department of Metallurgical and Materials Engineering, Istanbul, 1-7.

[5] S. Pasha, K. Chidambarama, N. Vijayan, W. Madhurai, Optoelectron. Adv. Mat. 6, 110 (2012).

[6] A. Gungor, Production and Characterization of Nano Dimensional Lead Oxide, Master Thesis, Mersin University Institute of Science and Technology, Mersin, 24 (2015).

[7] Z. Zhu, H. Yoshihara, K. Takebayashi, T. Yao, Journal of Crystal Growth 138, 619 (1994)' https://doi.org/10.1016/0022-0248(94)90879-6

[8] S. M. Sze, Physics of semiconductor devices (Second ed.), Wiley, New York, 1981.

[9] O. Gullu, DUMF Journal of Engineering 9(2), 689 (2018).

[10] O. Gullu, PhD Thesis, Ataturk University Institute of Science and Technology, Erzurum, 91 (2008).

[11] E. Senarslan, 2017, PhD Thesis, Ataturk University Institute of Science and Technology, Erzurum.

[12] H. Norde, Journal of Applied Physics 50(7), 5052 (1979).; <u>https://doi.org/10.1063/1.325607</u>
[13] S. K. Cheung, N. W. Cheung, Appl. Phys. Lett. 49, 85 (1986).; <u>https://doi.org/10.1063/1.97359</u>