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Optimization of ZnO Thin Films using Sol-Gel Dip Coating by Taguchi Method

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ABSTRACT

The purpose of this study was to use the Taguchi approach to examine as little as is practical to understand the ideal circumstances for producing thin films of ZnO with excellent attributes. Sol-gel dip coating was used to produce a thin layer of zinc oxide on a glass substrate while it remained at room temperature. An L9 trial plan of three levels high, medium, and low with four elements such as annealing temperature, speed of dip coating, the concentration of precursor, and annealing time was employed. The test was repeated three times, and three sol-gel configurations were made for each paper. We have decided to carry out the optimization using the estimated gap energy derived from the obtained film transmittance. Spectrophotometric analysis was used to characterize each sample. We were able to determine each ZnO thin film's gap energy that was deposited due to this characterization, which also enabled us to create the transmittance curve. To ascertain the electrical and optical characteristics, a signal-to-noise analysis and an analysis of variance (ANOVA) were utilized. In a formal declaration made under oath, the thin film of ZnO showed excellent crystal quality and more than 90% transmittance.

KEYWORDS: Zno thin film coating, Sol-gel dip coating.

1. INTRODUCTION

Silica glass, also known as fused quartz or quartz glass, finds various applications in streetlights due to its unique properties[1-4]. It is commonly used for lamp envelopes, offering high optical transparency and temperature resistance to protect the internal components from external elements. Silica glass used in streetlight applications can encounter significant challenges related to the accumulation of dust and water[5-8]. Dust particles settling on the surface of the glass over time can have a detrimental effect on its optical transparency, reducing light transmission and overall brightness. Superhydrophobic surfaces on silica glass are of great importance due to their numerous benefits and applications. These surfaces offer self-cleaning properties by repelling water and allowing it to roll off easily, carrying away dirt and contaminants. This reduces the need for frequent manual cleaning

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and maintenance, making them highly desirable for streetlights. This superhydrophobic nature can be observed by increasing the contact angle between the glass surface and the water particle[9-11]. By reducing water accumulation and light scattering, superhydrophobic coatings enhance the performance of silica glass in street light applications, ensuring efficient light transmission and uniform illumination. They also contribute to the durability of the glass, offering resistance against scratching, abrasion, and corrosion. Overall, superhydrophobic surfaces on silica glass provide enhanced functionality, reduced maintenance requirements, and improved performance, making them essential for streetlights[12-13].

In this work, superhydrophobic coatings are prepared on the silica glass surfaces by sol-gel method. The sol-gel method is a versatile and precise technique for preparing superhydrophobic coatings with numerous advantages[14-16]. It offers excellent control over the coating properties, allowing for customization based on specific requirements[17]. The method provides strong adhesion between the coating and the substrate, ensuring long-term durability and stability of the superhydrophobic surface[18-20]. Furthermore, sol-gel coatings can maintain the transparency of the underlying material, making them suitable for applications where optical clarity is crucial, such as silica glass[21-23]. The sol-gel process is relatively easy to apply, offering convenience and scalability. It is compatible with various coating techniques, making it adaptable to different production scales. The method also allows for the incorporation of additives, such as nanoparticles or catalysts, to introduce additional functionalities to the coating[24-27]. In this work, ZnO sol-gel has been prepared through the sol-gel route and the ZnO nanoparticles coating has been done by Dip-coating technique.

The goals of this research are to (1) use the Taguchi systematic design approach to improve properties and obtain ZnO thin films under ideal conditions; and (2) examine the optical, morphological, and structural characteristics of ZnO films coated under ideal circumstances.

2. EXPERIMENTATION AND METHODOLOGY

2.1 Materials used for Sol-Gel preparation:

1) **Zinc Acetate Dihydrate (ZAD):** Zinc acetate dihydrate ($Zn(CH_3COO)_2\cdot 2H_2O$) is a white crystalline solid used in pharmaceuticals as a source of zinc in dietary supplements and medications. It is also utilized in the textile industry as a mordant in dyeing processes and serves as a catalyst in chemical reactions. Extrapure Zinc Acetate Dihydrate of molecular weight of 219.49 was used for the experiments which is shown below.

Figure 4 – Amorphous Zinc Acetate Dihydrate used for experimentation

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2) **Isopropanol:** Isopropanol, also known as isopropyl alcohol or IPA, is a colorless liquid widely used as a solvent and cleaning agent. It is utilized for cleaning electronic components, as a disinfectant in pharmaceuticals and healthcare, and as a solvent in industries such as paints, inks, adhesives, and personal care products. Extrapure Isopropanol of molecular weight of 60.10 was used for the experiments which is shown in the figure.

Figure 5 – Isopropanol used for experimentation

3) **Monoethanolamine (MEA):** Monoethanolamine (MEA) plays a crucial role in the sol-gel technique, a method used to produce thin films or coatings. In sol-gel processing, MEA is commonly used as a stabilizer and pH adjuster. It helps to control the hydrolysis and condensation reactions of precursor materials, such as metal alkoxides, during the gelation process. MEA acts as a complexing agent, promoting the formation of stable sols and preventing premature gelation. Its presence also allows for better control over the viscosity and rheological properties of the sol-gel solution, facilitating the application and coating processes. 99% pure Monoethanolamine of molecular weight 61.08 was used for the experiments.

Figure 6 –Monoethanolamine used for experimentation

4) **Acetone:** Acetone is commonly used as a cleaning agent due to its excellent solvent properties. It is a colorless liquid that readily dissolves a wide range of substances, including oils, greases, and organic residues. As a cleaning agent, acetone is often used to remove contaminants from various materials, including glass, metals, plastics, and electronic components. It can effectively dissolve adhesives, paint, ink, and other stubborn substances, making it useful in industrial, laboratory, and household cleaning applications. Extrapure Acetone of molecular weight of 58.08 was used for the experiments which is shown below.

Figure 7 – Acetone used to clean the glass substrates

2.2 ZnO Sol Preparation:

This solution serves as the precursor for the deposition of a thin film of zinc oxide onto glass substrates. To create the sol-gel solution, zinc acetate dihydrate (ZAD) with a purity of 98% from Sigma Aldrich was chosen as the zinc source. It was dissolved in 250 mL of isopropanol (ISOP), a common organic solvent. Isopropanol acts as a solvent for dissolving the ZAD and facilitates the formation of a homogeneous solution[28-30]. In addition to isopropanol, monoethanolamine (MEA) was introduced into the solution. MEA serves a dual purpose in this process: it acts as both a solvent and a stabilizer[31-33]. By adding MEA in a 1:1 ratio with respect to ZAD, the viscosity of the solution can be controlled, ensuring ease of handling and coating. Furthermore, MEA helps stabilize the sol-gel solution, preventing particle agglomeration or precipitation, and promoting uniform film formation[34].

Once the ZAD, isopropanol, and MEA were combined, the sol-gel solution was subjected to stirring under controlled conditions. The Magnetic stirrer with a hot plate was used to stir the solution under constant temperature. The stirring was carried out at a temperature of 75°C for a duration of 1 hour and 30 minutes. This step is crucial for achieving complete dissolution of the ZAD and promoting the reaction between the precursor and the solvent. Continuous stirring ensures thorough mixing and the formation of a homogeneous sol-gel solution.

2.3 Dip Coating and Annealing:

With the sol-gel solution prepared, the next step involved depositing the ZnO film onto glass substrates. Prior to deposition, the glass substrates were thoroughly cleaned using distilled water and acetone to remove any impurities or contaminants that could interfere with film formation. Cleaning the substrates ensures good adhesion and a clean surface for the film to grow on[35-37]. The deposition technique employed in this process is known as dip coating. It involves immersing the cleaned glass substrate into the sol-gel solution and then slowly withdrawing it at a controlled rate. As the substrate is withdrawn, a thin film of the sol-gel solution adheres to its surface. The withdrawal speed plays a critical role in determining the thickness of the resulting film. After each coating layer, the film was subjected to a drying step. The coated glass substrates were placed in a heater and heated at a temperature of 150°C for a duration of 10 minutes. This drying process served two purposes. Firstly, it facilitated the removal of the solvent (isopropanol) from the film, allowing it to solidify. Secondly, it evaporated any remaining liquid, ensuring that only a solid ZnO film remained on the substrate[38-42].

Figure 9 – Muffle Furnace used for Annealing

Finally, the pre-annealing step was performed to further enhance the properties of

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the ZnO film. The coated glass substrates were annealed at a temperature of 150°C for a duration of 10 minutes. Pre-annealing helps improve the film's crystallinity, promoting the formation of well-defined crystal structures. Additionally, it can enhance the adhesion of the film to the substrate. This operation is repeated for 10 times and dense coating of ZnO was obtained on the glass surfaces. After the pre-annealing process, annealing heat treatment was performed for all the specimens at different temperatures like 450 ^{0}C, 500 ^{0}C and 550 ^{0}C. For the annealing process, the Muffle furnace was used to heat the specimens.

2.4 Coating Process Flow:

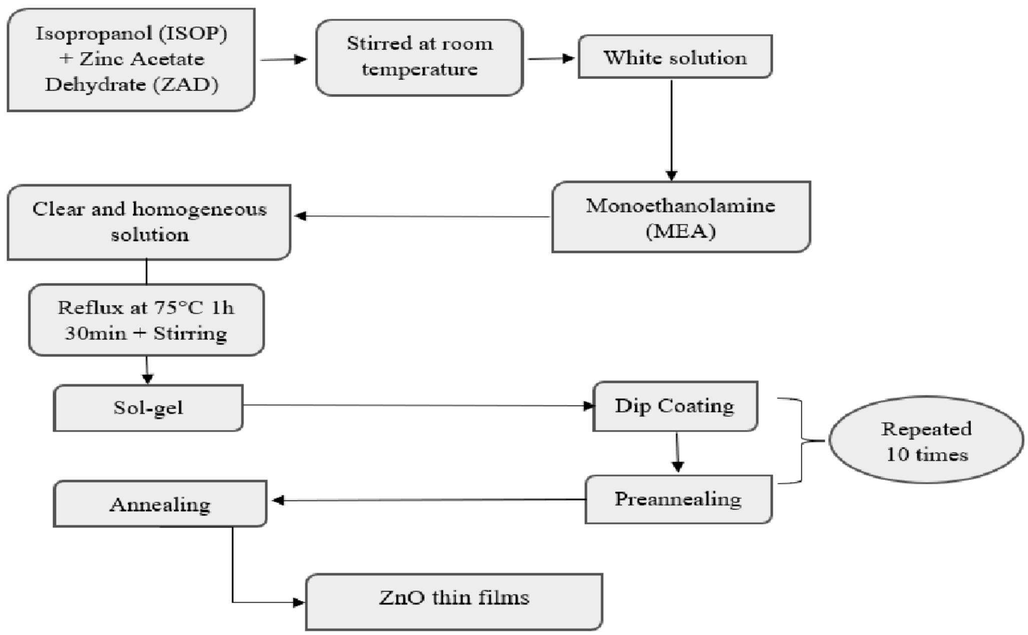

Figure 1: Preparation of thin ZnO films along with sol-gel

2.5 Taguchi Approach Design:

Genichi Taguchi created the Taguchi method to enhance the production of mechanical quality. The noise factor and the control factor are two elements that are examined by this procedure. As a result, the difference is controlled using the (S/N) ratio, and an ANOVA is performed to estimate the error variance and determine the contribution of each element. We chose a few variables that were poised to significantly affect the reaction we needed to optimize as much as possible. Then, in order to conduct the studies, we chose the best experimental design. The optimal limit conditions for the optical band gap produced by the dip-coating procedure in conjunction with the sol-gel approach were obtained using the L9 Taguchi table with the orthogonal ordered row.

In photovoltaic devices, the optical band gap energy is an important principle. To determine the ability to alter circumstances for the optimal optical band gap of the ZnO layer, the restrictions are altered.

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Table 1 lists the various elements and their weights for the specific ZnO thin films. The interactions between these parameters are displayed in Table 2. Three times each experiment was carried out. Based on the following equation, the best limitations that provide a useful band gap are optimized.

$$\Delta E = \left| E_{th} - E_{\exp} \right| \quad (1)$$

Where ΔE is the difference between the experimental band gap value and the best value (3.37eV) obtained from the transmittance spectra provided by the experiments.

Array of Orthogonal, Control Factors, and Levels:

Four control factors were used in the trials. These components include Zn (CH3COO)2 2H2O grouping concentration, annealing temperature, annealing time, and speed of dip-coating. ZAD concentrations of 0.25, 0.50, and 0.75 mol/L, annealing temperatures of 450, 500, and 550 °C, annealing times of 60, 90, and 120 mn, and dip coating speeds of 30, 40, and 50 mm/min were all established. Table 1 showed an L9 orthogonal arranged row with four elements (each at three levels).

	High level	Medium level	Low level
Annealing temperature (°C)	550	500	450
Annealing time (mn)	120	90	60
Speed of dip coating(mm/mn)	50	40	30
Precursor concentration (mol/L)	0.75	0.50	0.25

Table 1: Levels and Controlling Factors

3. RESULTS AND DISCUSSIONS

3.1 Taguchi Method Optimization:

According to equation (2), "the lower is better" was utilized to evaluate the thin films' properties when doing the S/N ratio analysis.

$$\frac{S}{N} = -10\log\left(\frac{1}{n}\sum_{1}^{n} Y_i^2\right) \quad (2)$$

Where i represents the number of an experiment, n represents the number of repeated experiments and

Yi symbolizes the primary response.

Experiments	Control Factors				Sample 1	Sample 2	Sample 3	S/N ratio
	W	X	Y	Z				
1	W1	X1	Y1	Z1	0.18	0.26	0.2	13.30993
2	W1	X2	Y2	Z2	0.21	0.11	0.19	15.1192
3	W1	X3	Y3	Z3	0.04	0.18	0.15	17.25073
4	W2	X1	Y2	Z3	0.14	0.18	0.11	16.70263
5	W2	X2	Y3	Z1	0.26	0.14	0.21	13.58857
6	W2	X3	Y1	Z2	0.23	0.04	0.16	15.73489
7	W3	X1	Y3	Z2	0.12	0.09	0.06	20.60481
8	W3	X2	Y1	Z3	0.08	0.18	0.18	16.24641
9	W3	X3	Y2	Z1	0.04	0.08	0.23	16.92504

Table 2: Experimental results with the S/N ratio, L9 Orthogonal Array using Taguchi method

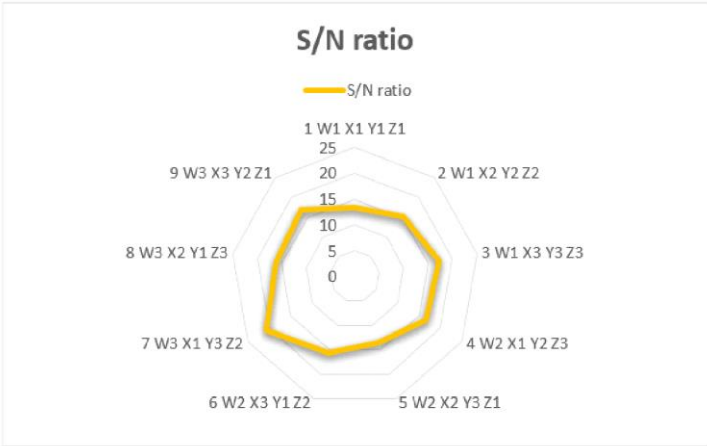

Figure 2: S/N ratio graph

Table 2 displays the computed values for the S/N ratio and band gap energy based on experimental data using equation (2). While Figure 2 provides the average S/N ratios for all levels of variables, allowing us to determine the ideal circumstances. The maximum point on the curve coincides with the best value for each factor. W3, X1, Y3, and Z2 are in the better position. The most well-known and significant of the four factors, as shown in Figure 2, is the annealing temperature. However, different factors have roughly comparable effects.

3.2 Surface and Cross-sectional Morphology:

Scanning Electron Microscopy (SEM) Test was conducted for the analysis of surface morphology and the cross sectional morphology of the obtained coated specimens. These obtained microstructures are analyzed by the SEM images shown below.

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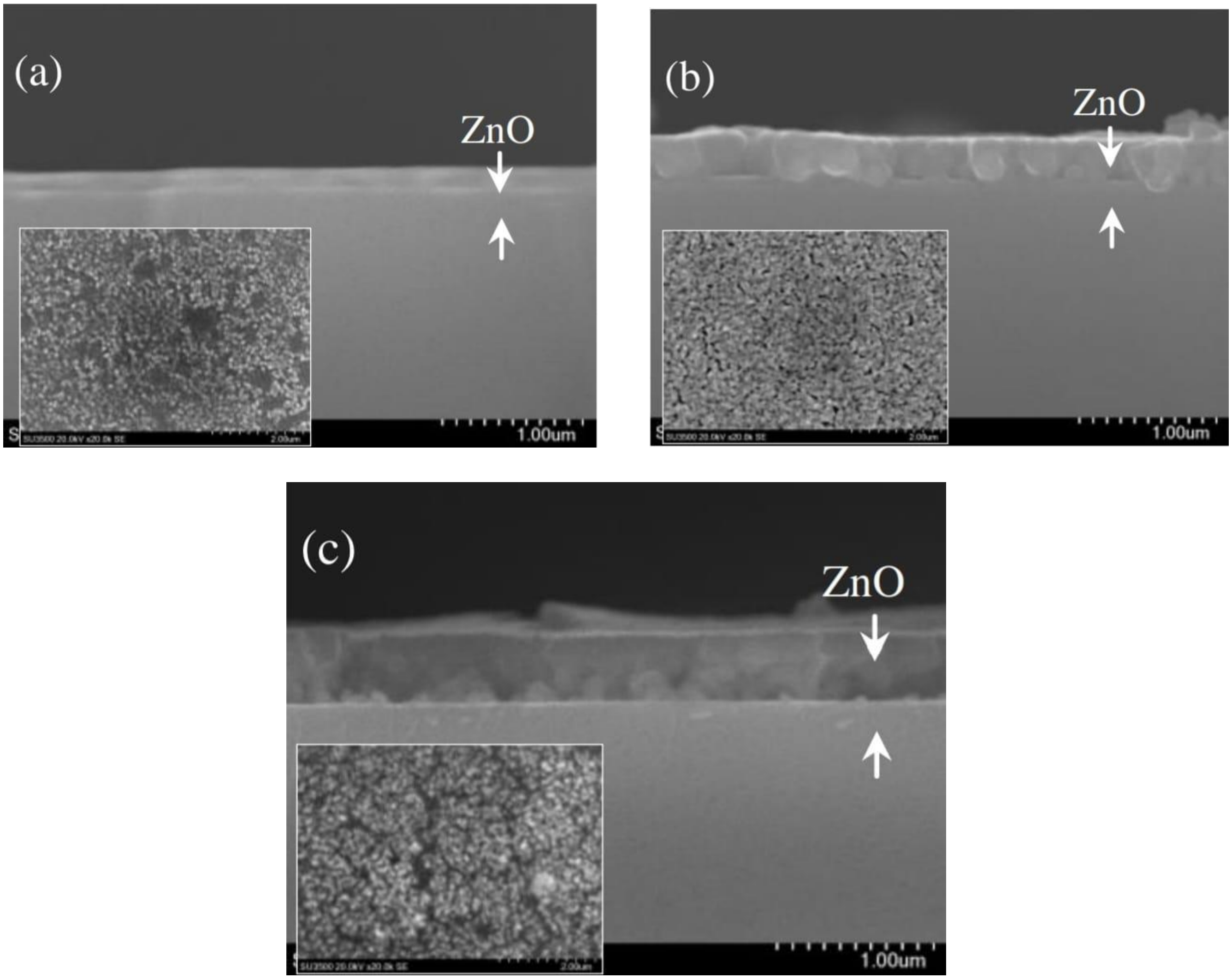

Figure 5 - Surface and cross sectional morphology of the specimens, 5(a) at precursor concentration of 0.5 M, 5(b) at precursor concentration of 0.7 M and 5(c) at precursor concentration of 0.9 M

The typical SEM images of surface and the cross sectional microstructure are shown in Figure 5. The average ZnO thin film thickness of the coated layer at a precursor concentration of 0.5 M was seen to be 135.6 nm, whereas the ZnO thin film deposition thickness was 329.7 nm at the precursor concentration of 0.7 M and the average thickness of the coating a 0.9 M concentration was 430.2 nm. This was analyzed with the help of cross-sectional microstructure images. From the surface microstructure images, we can observe that the grain size of the coating decreases as the precursor concentration increases. This was mainly because of the micro-densification effect. As the quantity of nuclei of metal centers increases because of the precursor concentration, the more dense ZnO film deposition is obtained. Therefore, the decreasing trend of grain size can be investigated when the precursor concentration increases from 0.5 M to 0.9 M.

4. CONCLUSION

ZnO thin films were deposited on the silica glass substrates uniformly and defect free specimens were obtained by employing Dip-Coating technique with suitable parameters. From the SEM images obtained, it was clearly noticed that the increase in the precursor concentration decreased the grain size of the ZnO thin films on the glass surface and the cross sectional thickness was also seen to be increased with the increase in precursor concentration from 0.5 M to 0.9 M. The optimum limitations for the production of ZnO thin films were obtained using the L9 Taguchi design.The examination of the S/N ratio allows us to confirm that the best parameters are W3 X1 Y3 Z2. The validation test uses this combination. In optoelectronic materials, the ZnO thin films can be employed as a window layer.

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