**International Journal of Materials Science and Applications**
2021; 10(1): 18-24
http://www.sciencepublishinggroup.com/j/ijmsa
doi: 10.11648/j.ijmsa.20211001.14
ISSN: 2327-2635 (Print); ISSN: 2327-2643 (Online)

# The Use of Taguchi Method to Elaborate Good ZnO Thin Films by Sol Gel Associated to Dip Coating

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**To cite this article:**

Modou Pilor, Bouchaib Hartiti, Alle Dioum, Hicham Labrim, Youssef Arba, Amine Belafhaili, Mounia Tahri, Salah Fadili, Bassirou Ba, Philippe Thevenin. The Use of Taguchi Method to Elaborate Good ZnO Thin Films by Sol Gel Associated to Dip Coating. *International Journal of Materials Science and Applications*. Vol. 10, No. 1, 2021, pp. 18-24. doi: 10.11648/j.ijmsa.20211001.14

**Received**: February 16, 2021; **Accepted**: March 9, 2021; **Published**: March 17, 2021

**Abstract:** ZnO thin films have been prepared by dip coating sol gel method using Taguchi technique. The underlying principle was to make something as little as conceivable the measure of examination and make sense of the best conditions for developing ZnO thin films with great properties. We used a trial plan of L9, with three levels (high, medium, low) and four elements (annealing temperature, precursor concentration, dip coating speed, annealing time). For each paper three sol-gel arrangements were arranged, and test is rehashed three time. We have chosen to carry out the optimization based on the gap energy calculated from the transmittance of the films obtained. Each sample was characterized with spectrophotometer. This characterization allowed us to draw the transmittance curve and to deduce the gap energy of each deposited ZnO thin film. A signal to noise and an analysis of variance (ANOVA) were used to determine the optical and electrical properties. The film that we obtained with the optimal condition was exanimated by using the characterization methods like UV-visible spectroscopy, X-ray diffraction, SEM (Scanning Electron Microscopy) and EDS (Energy Dispersive Spectroscopy). With the legal statement under oath condition, ZnO thin film showed high crystal quality and the transmittance is a greater amount of 90%.

**Keywords:** Zinc Oxide (ZnO), Taguchi Method, ANOVA, Sol-gel, Dip Coating

## 1. Introduction

Zinc Oxide (ZnO) is a sort of component used for make electronic circuits with a wide direct band gap of 3.37eV. It has the characteristics of high exciton binding energy (~60meV), noxiousness free, high electron mobility, and amazing piezoelectric conduct [1]. It is used in gas sensors [2], photodiode [3], solar cell [4], optical modulator waveguides [5], photonics crystals [6], varistors [7] etc.

ZnO thin films can be prepared by different technique such as Chemical bath deposition [8], sol–gel joined spin coating [9], sol-gel process associated dip coating [10], spray pyrolysis [11], sputtering magnetron [12], electrodeposition [13], etc.

Among these methods, sol–gel is the most popular technique due to its simplicity and reliability of synthesis and reproducibility [14]. The basic component of this technique is to give an attractive thin film size and distribution at high return and low-cost preparation [15].

Consequently, the examinations using traditional trial strategies are inefficient. Therefore, advancement through test configuration is a successful method to limit the quantity of experiment, just as the expense of generation. Many plan of investigation approaches have been applied to improve the proficiency of synthesis procedures, for example Box–Behnken [16], Taguchi [17], D-optimal [18], Central Composite Design [19], etc.

Various research studies revealed that the Taguchi configuration is a perfect method in ZnO thin films synthesis [20-22]

The targets of this work are: (1) to apply the Taguchi robust design method on the enhancement of properties and to get the ZnO thin films by using ideal conditions and (2) to study the morphological, optical and structural properties of ZnO films deposited at the optimized conditions.

# 2. Experimental Part

## *2.1. Preparation of Solutions*

The solution of ZnO sol–gel was prepared by dissolved zinc acetate dehydrate (ZAD) $Zn(CH_3COO)_2 2H_2O$ (Sigma Aldrich 98%) in 30 mL of isopropanol (ISOP) $C_3H_8O$ (Merck) and monoethanolamine (MEA) like solvent and stabilizer respectively. The acquired sol-gel solution was then stirred under 75°C during 1h 30. The ratio MEA/ZAD is maintained 1:1. A homogeneous solution was obtained [23]. The solution was dip coated on glass substrates cleaned with distilled water and acetone. The pre-annealing temperature and time was fixed to 150°C and 10 mn, respectively. After each layer, the film was dried at 150◦C for 10 mn in a heater to vanish the dissolvable [24]. This operation is repeated 10 times (Figure 1). Each these samples were characterized by using UV–visible spectrophotometer in the range of 200–800 nm. The sample prepared with an optimal combination of parameters has been characterized by using XRD with CuKα radiation (λ=0,154 nm) in angle 2 theta ranging from 10 to 100°at 0.02° scanning rate to investigate the structural properties, EDS and SEM to study the morphological and compositional properties.

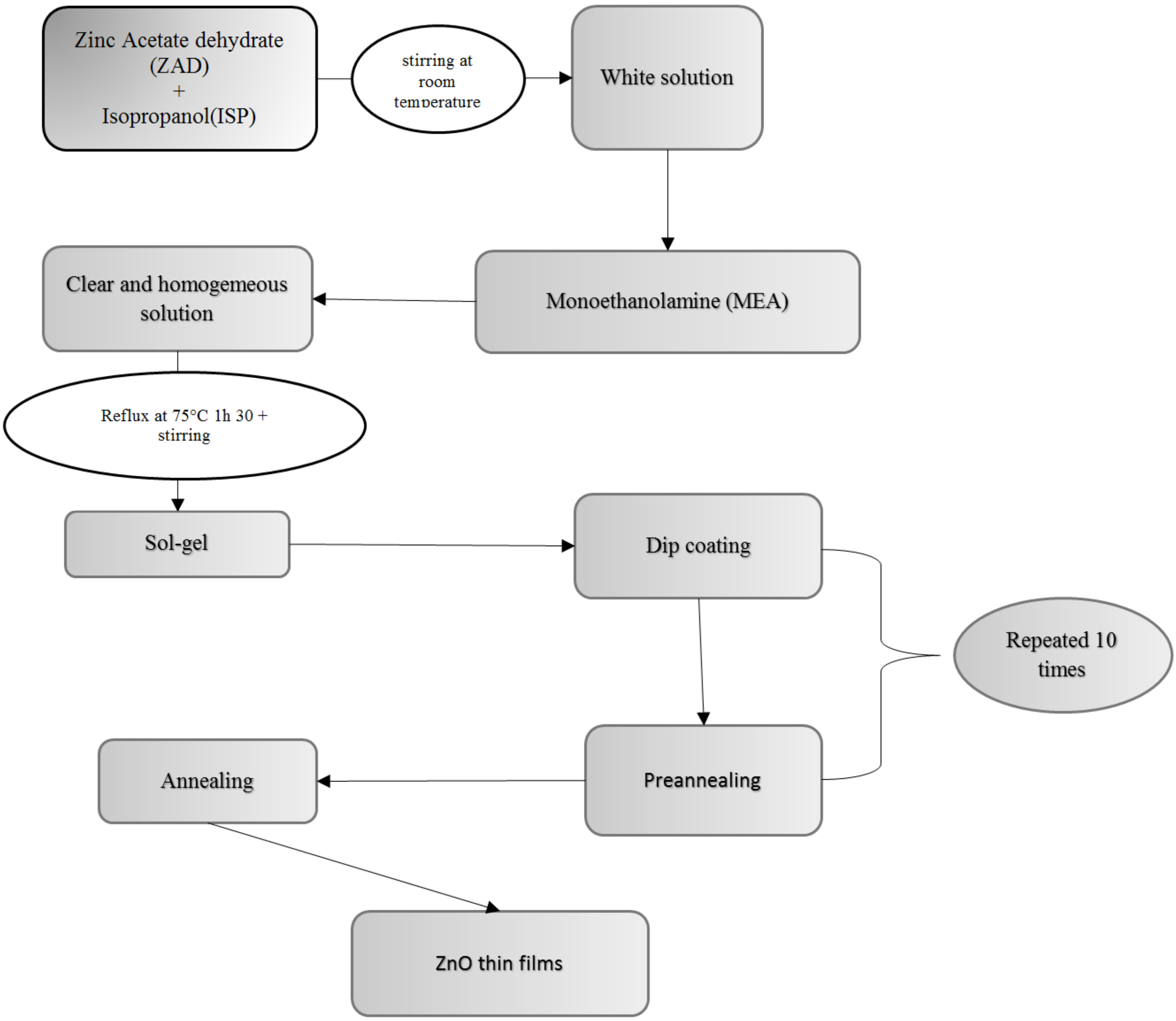


***Figure 1.*** *Preparation of sol-gel and thin films of ZnO.*

### *2.2. Design of Taguchi Approach*

Taguchi method is developed by Genichi Taguchi to improve the mechanical quality creation. This method studies two factors, the noise factor and the control factor. The difference accordingly is controlled by using (S/N) ratio and ANOVA is used to guess the error variance and figure out the role of each factor. [25]

We picked some factors ready to have significant influence on requiring reaction that we should improve as much as possible. Then, we have selected the appropriate experimental design to achieve the experiments.

L9 Taguchi table with orthogonal organized row was used to get the best limit conditions that give the optical band gap made by sol–gel method connected with dip-coating process. The optical band gap energy is a significant rule in photovoltaic devices. The limits are changed to decide the ability to change conditions for a best optical band gap of ZnO layer. Table 1 points to the different factors and their levels for the detailed ZnO thin films. Table 2 shows the combinations between these factors. Each experiment was repeated three times. The optimization of the best limits that gives a helpful band gap is based on the following equation (1) [26]

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

Where ΔEg is the difference between the best value (3.37eV) 3 [27] and experimental value of the band gap calculated from the transmittance spectra given by the experiments.

### *2.3. Levels, Control Factors and Orthogonal Array*

The experiments were done with 4 control factors. These elements are grouping concentration of $Zn(CH_3COO)_2\,2H_2O$, annealing temperature, annealing time and dip-coating speed. The levels of ZAD concentration were set on 0,25 mol/L, 0,50 mol/L, 0,75mol/L, annealing temperature on 450°C, 500°C and 550°C, annealing time on 60 mn, 90 mn, 120 mn, dip coating speed on 30 mm/mn, 40 mm/mn, 50 mm/mn. A L9 orthogonal organized row for 4 factors (each at 3 levels) was listed in Table 1. [28]

***Table 1.*** *Factors and levels.*

| | Low level | Medium level | High level |
|---|---|---|---|
| Annealing temperature (°C) | 450 | 500 | 550 |
| Precursor concentration (mol/L) | 0,25 | 0,50 | 0,75 |
| Annealing time (mn) | 60 | 90 | 120 |
| Dip coating speed (mm/mn) | 30 | 40 | 50 |

***Table 2.*** *L9 taguchi, Experimental values and the S/N ratio.*

| Experiments | Control factors | | | | Sample 1 | Sample 2 | Sample 3 | S/N ratio |
|---|---|---|---|---|---|---|---|---|
| | A | B | C | D | | | | |
| 1 | A1 | B1 | C1 | D1 | 0,17 | 0,27 | 0,19 | 13,37 |
| 2 | A1 | B2 | C2 | D2 | 0,22 | 0,12 | 0,20 | 14,65 |
| 3 | A1 | B3 | C3 | D3 | 0,03 | 0,17 | 0,14 | 17,83 |
| 4 | A2 | B1 | C2 | D3 | 0,13 | 0,17 | 0,10 | 17,30 |
| 5 | A2 | B2 | C3 | D1 | 0,27 | 0,13 | 0,22 | 13,36 |
| 6 | A2 | B3 | C1 | D2 | 0,22 | 0,03 | 0,15 | 15,18 |
| 7 | A3 | B1 | C3 | D2 | 0,13 | 0,08 | 0,05 | 20,65 |
| 8 | A3 | B2 | C1 | D3 | 0,07 | 0,17 | 0,17 | 16,71 |
| 9 | A3 | B3 | C2 | D1 | 0,03 | 0,07 | 0,22 | 17,43 |

## 3. Results and Discussions

To make the S/N ratio analysis we used ‘’the lower is better’’ according to the equation (2) to measure the thin films qualities. [29].

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

Where

i is the number of an experiment,

n the number of repeated experiments

Yi represents the primary response

***Table 3.*** *Mean of S/N ratio.*

| symbol | Level 1 | Level 2 | Level 3 | Optimal value |
|---|---|---|---|---|
| A | 15,28 | 15,28 | 18,29 | A3 |
| B | 17,11 | 14,93 | 16,81 | B1 |
| C | 15,11 | 16,46 | 17,28 | C3 |
| D | 14,72 | 16,82 | 17,31 | D3 |

Table 2 shows the experimental values of band gap energy and the S/N ratio that it was calculated by the equation (2). The mean of the S/N ratio for each level and the best value of each factor is shown in Table 3.

Figure 2 shows the mean of the S/N ratios of all levels of factors and permit us to figure out the best conditions. The best value of each factor goes along with the maximum point in the curve [30]. The best condition is A3, B1, C3 and D3

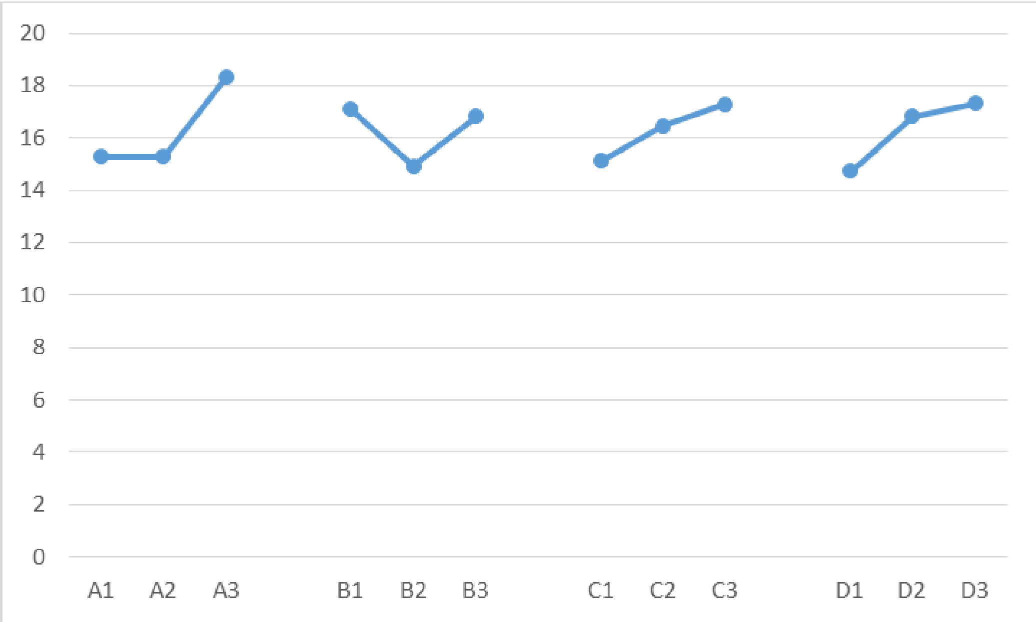


***Figure 2.** Graph of S/N ratio.*

As per to the Figure 2, the annealing temperature is the most famous and important among the four factors. Then again, different components have pretty much equivalent impact.

### *3.1. Description of the Films with Best Conditions*

From the results got by the analysis of variance, it was possible to decide the best conditions. The validation test was carried out by referring to the best conditions.

### *3.2. Structural Properties*

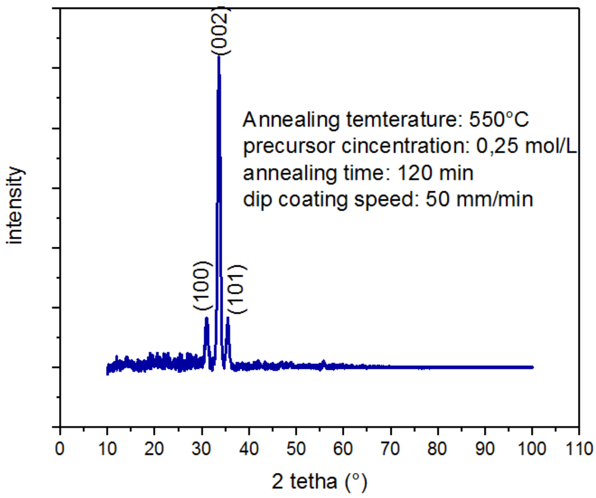


***Figure 3.** XRD pattern of ZnO thin film with best conditions.*

The structural properties were obtained through XRD technique. Figure 3 shows the description of the ZnO thin film with the best conditions.

It has showed that the deposited film is polycrystalline with a six-sided wurtzite pattern. The diffraction peak result with best conditions (space group: P63mc (186); a=3,05, c=5,30) are in agreement with JCPDS for ZnO (JCPDS036-1451). The good crystallinity is explained by the lowest surface energy density of the (002) plane of wurtzite crystal structure.

We can note that the film has a polycrystalline structure, with peaks positioned at 31.35, 34.13, and 35.60° locations, matching up to the planes (100), (002), and (101), respectively. As shown in Figure 1, ZnO thin film has peak (002) as the preferred direction of pointing. [31]

The interplanar distance *d* hkl is calculated from the Bragg formula (Equation 3)

$$2d_{hkl}\sin\theta = n\lambda \tag{3}$$

$\theta$diffraction angle

N diffraction order

$\lambda$wavelength

We calculated some parameters in the table 4 like the crystallite size (Dhkl), the dislocation (sigma), the lattice parameters a et c.

$$\frac{1}{d_{hkl}^2} = \frac{4}{3}\left(\frac{h^2 + hk + k^2}{a^2}\right) + \frac{l^2}{c^2} \tag{4}$$

$$D_{hkl} = 0,9\frac{\lambda}{\beta\cos\theta} \tag{5}$$

$$\partial = \frac{1}{D^2{}_{hkl}} \tag{6}$$

The wavelength of incident radiation is set to 0,154 nm and the lattice parameters (a and c) were calculated with the peaks (002) and (100).

***Table 4.*** *Crystallite size, lattice constants, interplanar distance and dislocation density of ZnO thin film with optimal conditions calculated from the intense XRD peaks (002).*

| experience | Interplanar distance $d_{hkl}$ (nm) | Crystallite size D (nm) | Dislocation density δ ($10^{-4}$) | Lattice parameter a=b | Lattice parameter c | c/a |
|---|---|---|---|---|---|---|
| 1 | 0,257 | 13,15 | 57,82 | 2,96 | 5,14 | 1,73 |
| 2 | 0,262 | 12,77 | 61,32 | 3,02 | 5,24 | 1,73 |
| 3 | 0,268 | 11,25 | 79,01 | 3,09 | 5,36 | 1,73 |
| 4 | 0,266 | 11,88 | 70,85 | 3,07 | 5,32 | 1,73 |
| 5 | 0,265 | 11,12 | 80,87 | 3,05 | 5,30 | 1,73 |
| 6 | 0,265 | 18,07 | 30,62 | 3,05 | 5,30 | 1,73 |
| 7 | 0,264 | 12,58 | 63,18 | 3,04 | 5,28 | 1,73 |
| 8 | 0,266 | 13,14 | 57,91 | 3,07 | 5,32 | 1,73 |
| 9 | 0,265 | 12,95 | 59,62 | 3,05 | 5,30 | 1,73 |

Table 4 shows different limits like the crystallite size (Dhkl), the dislocation (sigma), the lattice parameters a and c for a complete series of L9 Taguchi.

For each experiment the lattice limit is in agreement with JCPDS for ZnO (JCPDS036- 1451). The interplanar distance varies slightly.

The crystallite size (D) was calculated using the Scherrer's equation by selecting the most intense diffraction peak (101) (002).

The c/a calculated value for each experiment is significantly equal to 1,73. This value is also in agreement with JCPDS for ZnO

### *3.3. Compositional and Morphological Properties*

Figure 4 shows a SEM image obtained from the manufactured thin film. It presents a group of elements that are the same in the surface. The study of the shapes elements showed that we have got hexagonal structure. This surface morphological is dotted with grains of different sizes.

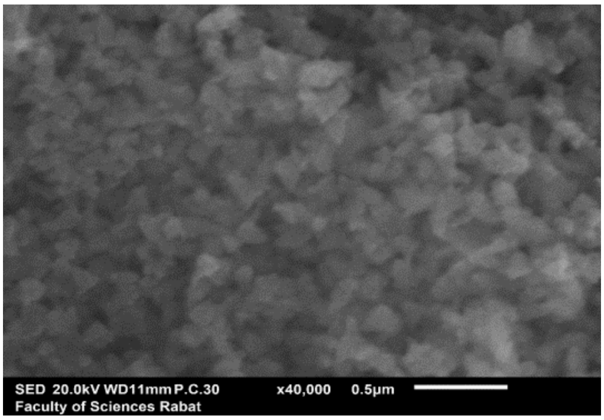


***Figure 4.*** *SEM image of the ZnO thin film with optimized conditions.*

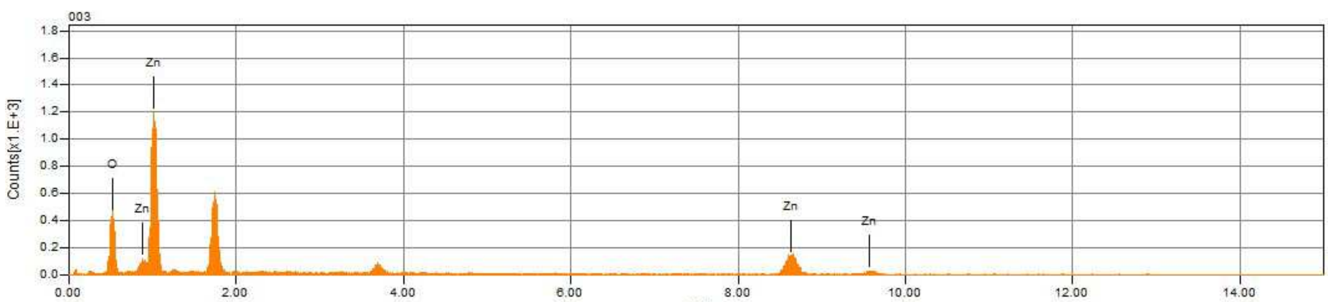


***Figure 5.*** *EDS of the ZnO thin film with optimized conditions.*

Figure 5 shows the EDS of the ZnO thin film with much-improved conditions. The peaks connected with the elements Zn and O are present. We can tell that other peaks of Mg, Al, Si and Ca are also present, but come from the supporting structure (substrate) on which the sample was deposited. Zinc and oxygen are in the proportions of 66,37% and 33,37% match up each pair items in order. [32]

***Table 5.*** *ZnO thin film composition deposited with best conditions.*

| Formula | mass% | Atom% | Sigma | Net | K ratio | Line |
|---|---|---|---|---|---|---|
| O | 30.08 | 55.25 | 0.19 | 11566 | 0.0322053 | K |
| Si | 20.83 | 21.79 | 0.24 | 23046 | 0.0230549 | K |
| Ca | 3.16 | 2.31 | 0.09 | 4076 | 0.0064003 | K |
| Zn | 45.94 | 20.65 | 0.55 | 12390 | 0.0848962 | K |
| Total | 100.00 | 100.00 | | | | |
| % ZnO | | | | | | |
| Formula | mass% | Atom% | Sigma | Net | K ratio | Line |
| O | 33.63 | 67.43 | 0.21 | 11566 | 0.0322053 | K |
| Zn | 66.37 | 32.57 | 0.80 | 12390 | 0.0848962 | K |
| Total | 100.00 | 100.00 | | | | |

### 3.4. Optical Properties

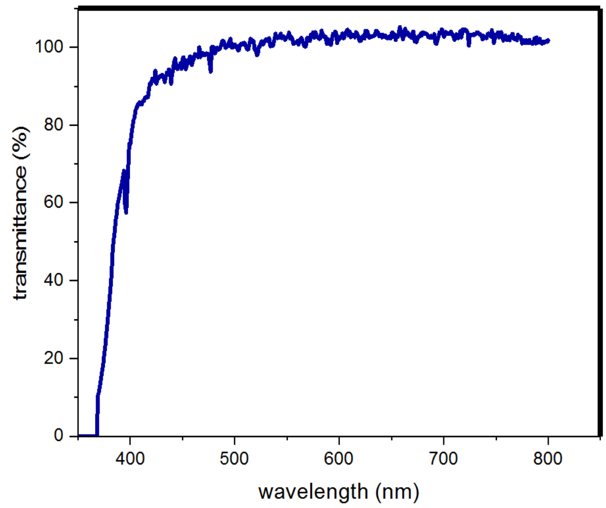


***Figure 6.*** *Transmittance of the ZnO thin film.*

The transmission curve is represented in figure 6, we can calculate the direct band gap by the equation (7), which represents the difference of $(\alpha h\upsilon)^2$ with (hυ)

$$(\alpha h\vartheta)^2 = B(h\vartheta - Eg) = f(h\vartheta) \quad (7)$$

B: constant

$h\vartheta$ : Energy of incident photons

Eg: Optical gap

The variations of $(\alpha h\vartheta)^2$ with energy (hυ) are represented on the Figure 7. The optical gap is figure out by prediction of numbers, based on what's known of the curve $(\alpha h\vartheta)^2$=f( $h\vartheta$ ) at the value $(\alpha h\vartheta)^2$=0. The bandwidth is figure out from this equation, and represented by the value of the optical gap, rated Eg which is close to 3.25 eV. [33]

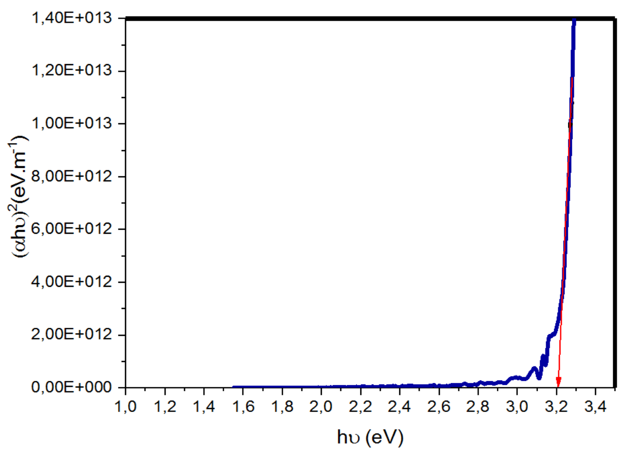


***Figure 7.*** *Determination of band gap energy.*

## 4. Conclusion

The L9 Tagushi design was used to get the best limits for the creation of ZnO thin films.

The S/N ratio analysis permit us to certify that the best limits are A1 B3 C4 D3. This combination is used to the validation test.

The structural, optical, morphological and compositional properties of sol-gel dip coated ZnO thin films by Taguchi method have been examined and it's closely to the reality can be found. The XRD spectra have showed that the films have the polycrystalline structure. The grain size of crystallites was in the range of 11-18 nm. We have got 3,25 on optical band gap and all the films are highly clear with more than 90% in the visible area. The ZnO thin films can be used as a window layer in optoelectronic materials.


## Acknowledgements

Prof. Bouchaib HARTITI, senior associate at ICTP (The Abdus Salam International Centre for Theoretical Physics), is very grateful to ICTP for financial support. Technical support from LMOPS (University of Lorraine, France) is also acknowledged.


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