REALIZATION AND CALIBRATING OF A DIP COATER FOR THIN FILMS DEPOSITION

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Résumé

Dans ce travail, un système de trempage-tirage a été réalisé et calibré localement en laboratoire dans le but de préparer des couches minces par vois sol-gel. Ce système assure un mouvement de translation vers le bas et vers le haut avec une vitesse contrôlée. Les positions bas et haut sont fixées à l'aide de deux capteurs de proximité.

Un porte-échantillon est immergé dans une solution pendant un temps déterminé, puis retiré. La vitesse d'immersion, la vitesse de retrait et la durée d'immersion sont commandées facilement par l'utilisateur dans une large plage de choix. Ce dispositif de revêtement par trempage fonctionne en mode automatique, et peut être également utilisé manuellement. Les vitesses d'immersion et de retrait ont été identifiés par rapport aux fréquences du variateur de vitesse. La stabilité du système a été bien démontrée. La caractérisation par diffraction des rayons X des couches minces d'oxyde de zinc obtenu par ce dispositif présente trois pics forts de ZnO, (101), (002) et (100) respectivement. Ce système à faible coût a prouvé sa robustesse et sa fiabilité.

Mots clés *: Automatique; Convertisseur de fréquence; Contrôle du mouvement; trempage-tirage; Couches minces de ZnO*

Abstract

In this work a homemade dip coater was carried out and calibrated in the laboratory in order to prepare thin films using sol-gel process. This system allows a translation motion downwardly and upward with a controlled speed. The down and up positions are fixed using two proximity sensors.

A sample holder is immersed in a solution for a predetermined time and then is removed. The dipping speed, withdrawing speed and immersion time are controlled easily by the user in a wide range of choices. This dip coater works in automatic mode and it can be used manually. The dipping and withdrawing speeds were identified to frequencies of the converter. The stability of the system was well demonstrated. The XRD characterization of zinc oxide thin films obtained by using this dip coater shows three strong peaks of ZnO respectively (101), (002) and (100). This low cost system has proved its robustness and reliability.

Key words *: Automatic; Frequency converter; Motion control; Dip coating; ZnO thin films*

ملخص

في هذا العمل تم انجاز و معايرة نظام غمس و سحب محلي الصنع في المختبر من أجل إعداد طبقات رقيقة عن طريق عملية الصول جال. هذا النظام يسمح بالحركة إلى الأسفل وإلى الأعلى مع سرعة يمكن السيطرة عليها، الوضعيتين العليا و السفلى يتحكم بهما باستخدام اثنين من أجهزة الاستشعار عن القرب، سرعتي الغمس و السحب و مدة الغمس يتم التحكم بهم بسهولة من طرف المستعمل من خلال مجال كبير للاختيار.

يتم الغمس لمدة معينة، تم تتم عملية السحب، سرعتي الغمس و السحب متعلقتان بالتواتر الصادر من مغير السرعة.التوصيف باستعمال أشعة اكس للطبقات أكسيد الزنك المنجزة بهذا الجهاز أسفر عن ثلاث قمم عظمى قوية لأكسيد الزنك (101)، (002) و(100) على التوالي.هذا الجهاز القليل التكلفة أكد قوته و فعاليته.

كلمات مفتاحيه *: التحكم الآلي, محول التواتر, التحكم في الحركة, الغمس و السحب, أغشية أكسيد الزنك الرقيقة*

Tehnics for depositing thin films have increased dramatically over the past four decades. This development is mainly due to the need for new devices in the electronics technology. Rapid advances in electronic semiconductor devices and improved characteristics of thin films may not be possible without the development of new processes for depositing thin films. Techniques of thin films deposition is still under rapid changes that will lead to getting more complex and advanced electronic devices in the future. Several methods have been used to prepare thin films such as pulsed laser deposition [1] magnetron sputtering[2] chemical vapor deposition[3], chemical spray pyrolysis [4], the sol-gel process[5,6] ... etc..

The economic impact of these techniques has always been the interest of electronic and optical devices manufacturers; it will be profitable to have thin films of good quality with the lowest possible cost. The sol-gel deposition method (gelling solution) refers to a simple and low-cost process using chemical precursors that can produce thin film at room temperature and atmospheric pressure, with higher purity and good homogeneity. The method is used widely for the fabrication of metal oxides, such as ZnO [6], SnO_2 [7] and TiO_2 [8]. The advantage of this method is that it responds to the demand of low cost, this is one of the most versatile techniques for preparing thin films [9]. The two best-known methods for thin films preparation with control of the deposited thickness are dip coating and spin coating. Both techniques are used at room temperature and atmospheric pressure.

Sol-gel process associated to dip coating consists of dipping a substrate into a chemical solution for a short time (a few seconds), then withdrawing it with a straight upward motion at defined speed. In This case, the Landau Levich relationship [10] gives the deposited thickness according to the deposition speed. Withdrawal speed is an important parameter that affects the porosity and thickness of the films and the morphology of the grains [11]. The thickness 140, 160 and 190 nm of films were obtained with withdrawing speeds 10, 15 and 20 cm/mn respectively [11].

1. MECHANICAL PART

This section of the system consists of three elements: an electric motor, a rotation-translation converting system and a sample holder. The electric motor is three-phase asynchronous type with associated mechanical reducer. The motor power is 180 w driving the system by converting electrical energy into a mechanical one (rotation).The reducer associated with the motor has a high reduction ratio. The speed at its output is:

n_S = 0,783 rpm at 50Hz which is still considered high. The mechanic reducer has also the role of stopping the motor in order to prevent a drive by inertia. Rotation-translation conversion system is provided by a rod-crank system, a pulley of 14 cm of diameter attached to the shaft of the reducer. The rotational disc movement of the pulley is transmitted to a metallic rod by means of a connecting rod. The fixing system between pulley connecting rod and metallic rod is achieved by bearings where two guides control the movement of the metallic rod making the movement rectilinear (figure.1). The translation movement amplitude of the metallic rod is related proportionally to the distance D between the center of the pulley disc and the fixation point of the connecting rod.

The movement of the metallic rod is secured in a controlled manner without vibration. The substrates are pinned on a sample holder attached at the bottom of the metal rod. The sample holder can hold four substrates at time. The electric motor is fixed to a cage whose upper face is reinforced by aluminum. Two parallel rods are used for guiding the principal rod. The system scheme is depicted in is illustrated by figure 1.

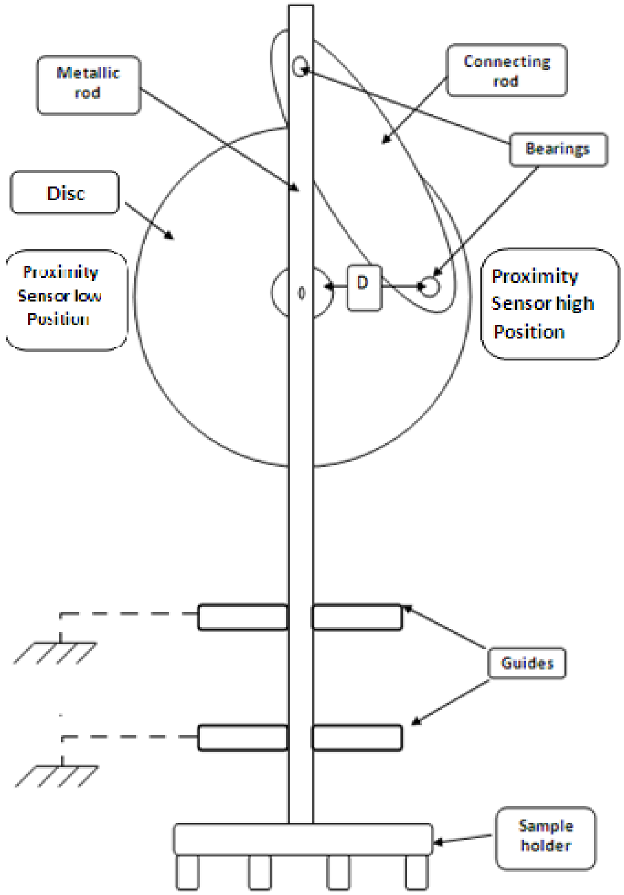

Figure 1 : Illustration of the mechanical system.

2. CONTROL PART

The control part consists of an electrical cabinet including a frequency converter, protection devices (for motor and user) and push buttons to control the system (wired automation).

The speed at the mechanic reducer output is: nS = 0,783 rpm at 50Hz which remains high relative to the desired speed, for this reason, we adopted a frequency converter (inverter), the inverter uses a single phase (230V, 50Hz) and transform it on three phases (200V, 0.5 to 200 Hz).This device permits the adjustment of the motor speed, the direction of its rotation and its thermo magnetic protection.

The converter allows us to vary the substrate speed of dipping and withdrawing by varying the motor speed according to the relationship:

$$Ns = 60\ f / p\ (rpm) \qquad (1)$$

Where p is the number of pole pairs of the motor, ns is the rotation speed and f the frequency. From the previous relation, we can change the motor speed by varying the frequency of the converter using a potentiometer or by entering data in the converter directly.

For the control circuit we have used control relays, a timer to change the immersion time, and two inductive proximity sensors to determinate the position of the sample holder (high position and low position). Using sensors, we can avoid the periodicity movement of rod-crank system.

The sequential steps of the system are shown in figure 2.

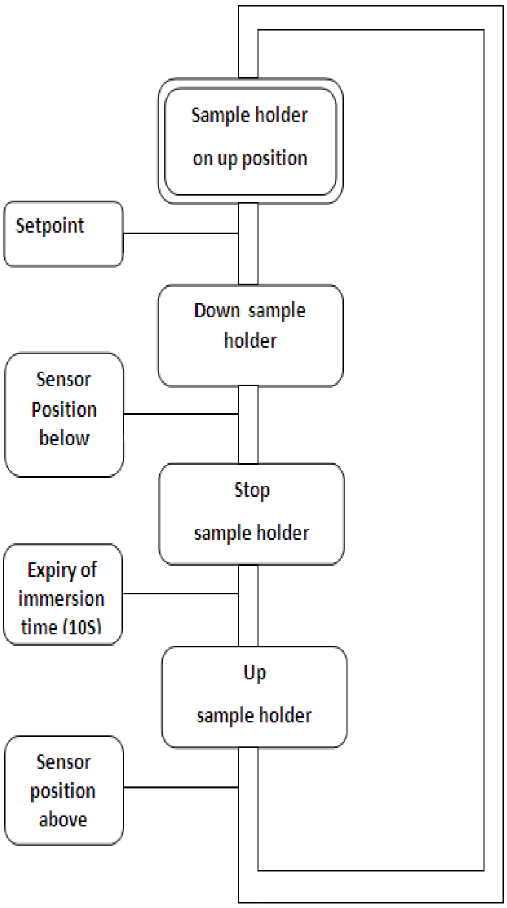

Figure 2 : The sequential steps of the system

3. TESTING AND APPLICATION

After the installation and automation of the system, we performed measurements on the speed and the stability of the system, after that we have used the home made system for deposition zinc oxide thin films on a glass substrate.

3.1. Speed measurement and stability

To measure the speed of translation of the metallic rod (movement of dipping and withdrawing), we used a stopwatch with two sensors which the distance between them is 5 cm (figure 3). At every time, we change the frequency of the inverter and we measure the time for which the metallic rod travels a distance of 5 cm, from this, we deduce the speed of translation.

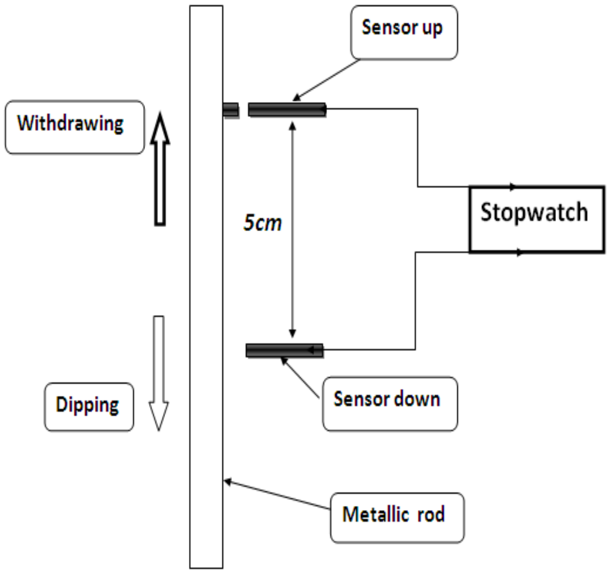

Figure 3 : System for measuring the speed

Sd and Sw are the speeds of dipping and withdrawing respectively (figure 4).. Sd was deduced when the rod travels from up sensor to down sensor and Sw was calculated when the rod travels from down sensor to up sensor.

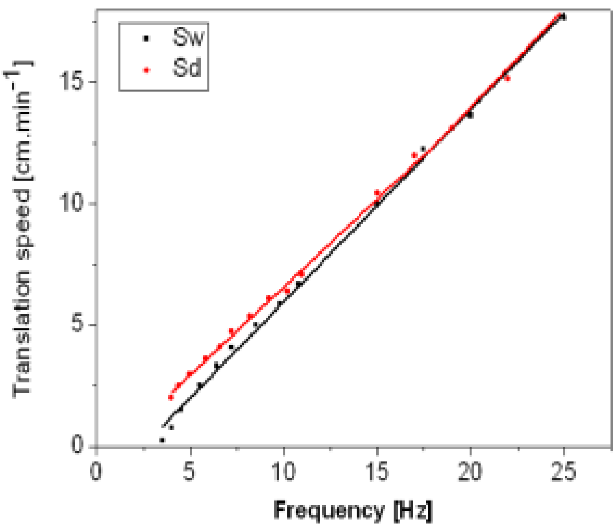

Figure 4 : Translation speed dipping (S_d) and withdrawing (S_w) depending on motor frequency.

The measurements were performed five times to check the reproducibility and stability of the manipulation (figure 5).

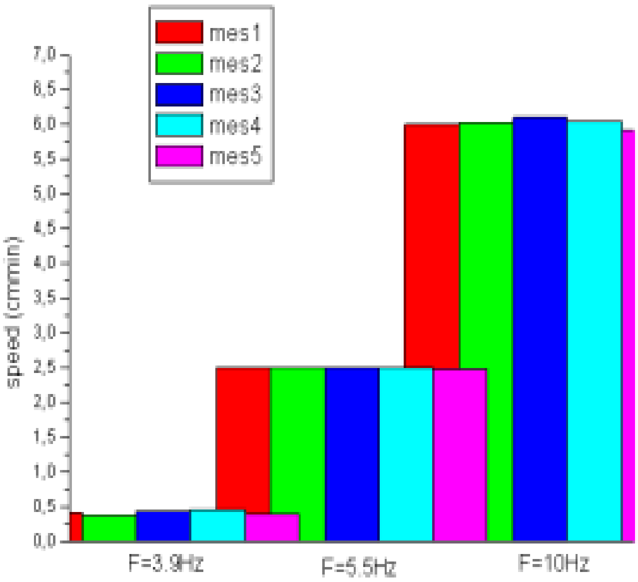

Figure 5 : Speed stability for three different frequency

We note that the speed of translation is proportional to the frequency applied on the motor in the range [4-25 Hz] and varies between [0.78 to 18 cm/min]. This speed range is effective for thin films deposition by dip coating method. We also notice that the speed of dipping is slightly higher than speed of withdrawing at the range [4-17.5 Hz], It can be justified during the decent gravity facilitates the training of the metallic rod down and the load torque of the system in dipping is lower than in withdrawing.

Note that the system is not stable from a frequency f = 3.9 Hz to lower values, the rod could stop before finishing the race. The inverter used is with constant U / f ratio (U voltage across the motor). For this type of drive, there is a lower limit frequency.

In our case, we found that below 3.9 Hz the translation speed is not reproducible because of the limitation of the frequency. The nominal torque operation which is defined as the intersection between the motor torque and the load torque decreases when the frequency decreases. The motor torque is close to zero and the force is not enough to remove the rod in a stable manner.

3.2. Application

ZnO sols were prepared using zinc acetate, 2-Methoxyethanol and monoethanolamine (MEA) as the solute, solvent and stabilizer respectively. The molar ratio of MEA to zinc acetate was kept at 1.0.and the Zn concentration was 0.5 mol/L. The resulting solution was stirred by a magnetic stirring at 70◦C for one hour. The solution were aged for 24 h, then ZnO thin films were coated by the dip coater realized in this work on glass substrate where the dip coating speed was 3 cm/mn and the emersion time was 10 seconds. After each coating, the sample was dried at 280 ◦C for 8 min, this procedure was repeated for ten times and then the substrate was annealed at 500◦C in ambient atmosphere for 2 hours. The crystal structure and orientation of the ZnO thin films prepared by the dip coater realized was investigated by X-ray diffraction (XRD) patterns (figure 6). XRD spectra of ZnO annealed thin film shows the strongest peaks for ZnO are (101), (002) and (100) Miller indices.

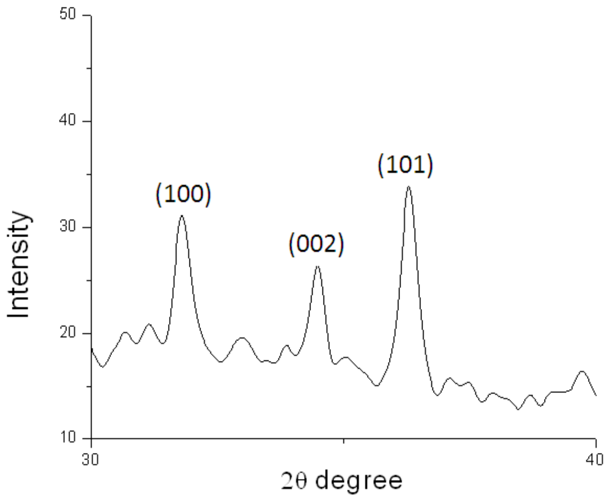

Figure 6 : Spectra of X-ray diffraction thin film of ZnO.

CONCLUSION

In present work, a system of thin films deposition was carried out and calibrated. This system is used for the deposition technique by sol-gel associated to dip coating. A mechanic system was controlled to dip and remove a glass substrate in a solution at a controlled speed. Speed variation is achieved by a frequency converter and an automated control system circuit, the withdrawing speed is stable above a frequency of 3.9 Hz, XRD characterization of a thin film based ZnO has proved the reliability of the dip coater.

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