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PREPARATION OF THIN FILMS BY SILAR AND SPIN COATING METHOD

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Abstract: *There is great interest in the synthesis of chalcogenide metal thin films by using various deposition techniques. Thin films are the cheapest semiconductor materials, and could be used in gas sensors, solar cells, photodetectors, and other optoelectronic devices. In this work, there are two different deposition techniques, namely spin coating and successive ionic layer adsorption and reaction (SILAR) were used to produce nanostructured thin films. This article gives a short review of some advantages and limitations of these techniques. Lastly, we also review some experimental findings according to literature review.*

Keywords: *Spin coating, SILAR technique, Thin films, Semiconductor, Solar cells.*

INTRODUCTION

What is thin film? It could be defined as thin layer deposited onto substrate (with the film thickness less than 1000 nm). These materials have ranged from several manometers-micrometres in thickness as reported by many researchers [1, 2]. The application of thin films including photodetectors [3, 4], waveguides applications [5, 6], solar cells [7-10], gas sensor [11, 12] and optoelectronics devices. Currently, thin film technology is playing an important role to enhance the properties of the materials [13, 14]. Up-to-date, there are several deposition techniques such as chemical bath deposition technique [15-18], thermal evaporation method [19, 20], spray pyrolysis technique [21, 22], sputtering method [23, 24] and electro deposition method [25-27] as proposed by many scientists to produce various types of thin films.

In this work, spin coating method and successive ionic layer adsorption and reaction (SILAR) technique were discussed. A brief survey on the disadvantage, advantage of these deposition methods and results will be reported.

LITERATURE SURVEY

Spin coating technique

This method is used for developing uniform coatings of organic materials on different types of flat surface. A spin coater machine is used for this process. In 1958, this method was first designated by Meyerhofer and Emslie. Currently, spin coating method could be considered as one of the popular

deposition techniques because of fast, cheap and simple method for generating homogeneous samples. Moreover, this method also has great advantage to develop extremely uniform layers of samples on different substrates. The thickness of film was mainly controlled by several conditions such as spin speed and viscosity photoresists. A well-defined thickness by using liquid precursor is achieved through spin coating method. Spin coater (or spinner) is the instrument that is used for spin coating process [28]. The basic principle of spin coating method is shown in Figure 1. There are four key stages (fluid dispense, spin-up, stable fluid out flow, evaporation) are used in order to prepare thin film by using spin coating method. In this process, firstly, the substrate rotates while distribute the liquid onto the sample. Secondly, desired quantity of a solution is putting on the substrate while, substrate is rotating in order to spread the solution on the substrate. The speed of a rotor and quantity of solution have great impact on the properties of the thin films [29, 30]. The whole processes of developing thin films are shown in figure 1. The advantages and limitations of this technique were listed in Table 1.

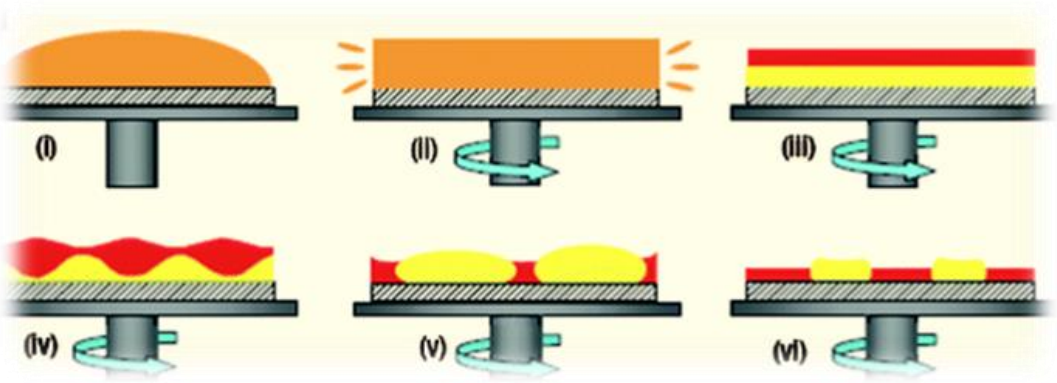

Fig 1: Principle of Spin Coating [29].

Table 1: Advantages and limitations of Spin Coating:

Advantages	**Limitations**
➢ It is simple and easy method	➢ Photoresist costs and size of substrates affected on the working of spin coaters.
➢ It is low cost and fast technique as compared to other techniques.	➢ This method has less material efficiency.
➢ Spin processing is mostly used in the semiconductor industry, where thin films with thickness of less than 11 nm (are created for many applications).	➢ This technique is considered as a slow developing technique because only single substrate can be used at a time.
➢ Most substrates used are photo masks, wafers, micro scope slides, or even small pieces.	➢ In some nano-technologies, it working outcomes become low due to fast drying processes [29].
➢ This method is also used for cleaning or etching.	
➢ Film thickness is varying through changing spin speed.	

➢ It is used in conductive oxide, antireflection coatings and flat screen display coatings [29, 30].	

Uma and co-workers [31] used the spin coating method for the development of TiO_2 thin films. Structural, optical and mechanical properties of samples were characterized through scanning electron microscope, UV-Visible Spectrophotometer, energy dispersive X-ray fluorescence analysis (EDXRF), X-ray diffraction (XRD) and micro hardness unit. The optical properties were studied using UV-Visible spectrophotometer, and revealed that band gap energy is 3.61 eV. Non-crystalline behaviour has been investigated through XRD. Scanning electron microscope (SEM) results showed that surface of thin films is smooth and crack free. Moreover, hardening index number was found 0.53.

Porosity and structure of the Fe-doped TiO_2 thin films were studied through scanning electron microscope and X-ray diffraction as reported by Bilalodin and co-workers [32]. The ankerite structure was identified through XRD patterns. Moreover, 0.6 g/L concentration of iron doping transformed the anatase crystal phase into rutile phase as indicated in XRD studies. SEM showed that the grain size decreases (100 to 83 nm) with increasing iron dopant.

ZnO and ZnO:TiO_2 thin films were successfully produced (glass slide as substrate) as described by Firdaus and co-workers [33] through using spin coating method. Filed Emission Scanning Electron Microscopy (FESEM) analysis revealed ZnO films are less homogeneous with porous, and thickness in the range of 896 to 4884 nm, while ZnO:TiO_2 films have smooth surface, less porous with thickness in the range of 890 to 3919 nm. They described the different band gap values could be obtained under different samples. The obtained band gap (ZnO films) is in the range of 2.81 to 2.98 eV. However, band gap value of ZnO:TiO_2 films become reduce from 2.97 to 2.71 eV as film thickness increases. Composite ZnO:TiO_2 has greater value of conductivity as compared to ZnO films as analysed using 2-point probe meter.

Spin coating technique was applied to synthesize pure TiO_2 and cobalt-doped titanium dioxide thin films as proposed by Asli and co-workers [34]. The as-deposited films are amorphous phase as supported in XRD data. However, titania anatase structure could be observed at 500 °C (annealing temperature) for 1 day. Also, XRD patterns show that several diffraction peaks such as (101), (200) and (211) plane could be detected in annealed films. The band gap values were reduced (3.44 to 3.25 eV) with increasing cobalt concentration from 0.055 to 0.244. They also conclude that the resistivity of Co-doped TiO_2 films was reduced, indicating these materials could be employed in photovoltaic applications.

Mohd et al. prepared the TiO_2 thin films by using spin coating method. There are several techniques to characterize films such as X-ray diffraction, atomic force microscopy (AFM), thickness profiler, UV-Visible spectrophotometer, Field Emission Scanning Electron Microscopy (FESEM) and current-voltage measurement system as mentioned by them. The Titanium dioxide thin film that includes 0.4 g show the least roughness has been investigated from the atomic force microscopy analysis. Titanium dioxide thin film show optimum uniformity and roughness by adding 10 ml of acetic acid. Optical properties revealed that the transmittance was 80 %, indicating this films could be used for optoelectronic application. Anatase form of thin films was analysed through XRD. Current-voltage measurement system indicated the sensitivity of thin films for organic solvent which could increase the current value [35].

Sumana and co-workers [36] used the spin coating method to prepare the titanium dioxide thin film. The morphological, structural and topographical properties were investigated through X-ray diffraction, SEM and atomic force microscopy, respectively. X-ray diffraction analyses showed that crystallization happens for the films prepared at 400 °C and microwave radiation at 540 W. The formation of grains (with voids) could be seen in all samples as indicated in SEM studies. The atomic force microscopy revealed annealed films show higher surface roughness value and dielectric constant if compared to microwave exposed films.

Tahir and co-workers [28] investigated the nanocrystalline TiO_2 thin film by using the spin coating process. Anatase and rutile phase of TiO_2 were obtained when calcination temperature has ranged from 400 to 800 °C (through XRD pattern). Moreover, optical absorbance spectra revealed that band gap decreases with increasing calcination temperature. Electrical conductivity revealed that crystallite size increased as the electrical conductivity increased.

The Structural, morphological and optical studies of titanium dioxide thin film were carried out through X-ray diffraction, SEM and UV-Vis Spectrophotometer, respectively. The X-ray diffraction (XRD) revealed that particle size increases with increasing the annealing temperature. Uniform distribution of particles and spherical shape could be obtained in SEM images. Optical studies showed smaller band gap could be observed at higher temperature [37].

The transmittance spectra of the different samples conclude that surface roughness and film thickness chiefly depended on experimental conditions [36]. In another experiment, it is revealed that grain size decreases with increasing dopant revealed based on scanning electron microscopy analysis. Capacitance-voltage (C-V) characteristics show that dielectric value decreases with increasing annealing temperature [34]. Raman and Fourier Transform Infrared (FTIR) measurements confirmed that the anatase to rutile phase transition at 600 °C [38]. The influence of annealing on the properties of films was studied. They observed that density, crystallinity and band gap reduced with increasing annealing temperature [39]. Electrical conductivity was analysed through four point probes which showed that when crystalline size increased, the electrical conductivity increased [28] also. There are different phases such as crystalline and amorphous behaviours could be seen for the films prepared for 2000-5000 rpm runs. Development of crack free anatase films and thickness (can be controlled by spin rate) have been reported by them. However, it is very clear that spin rate will not affect the surface roughness based on atomic force microscopy [40].

SILAR DEPOSITION TECHNIQUE

Thin film deposition method consisted of physical technique and chemical technique. When material moved from a target source toward substrate to form layer, is called physical deposition method. Whereas, in chemical process precursors undergo chemical reaction at the surface of the substrate (chemical technique). One of the modern chemical deposition methods is successive ion layer adsorption and reaction (SILAR) method. Generally, this method consisted of two important processes, namely adsorption of ion onto the substrate and reaction of the adsorbed ion layer will be observed. During the deposition process, cleaned substrate will be dipped into different solutions (containing cationic and anionic precursors). Researchers explain that the adsorption of ions onto surface of substrate because of the presence of attraction force between ions in the solution and surface of the substrate [41]. Basically, film thickness, morphology and composition could be controlled through immersion-reaction cycle [42]. Table 2 shows the advantage of SILAR method. This method was first reported in 1985 by Ristov and co-workers [43].

Table 2: Advantage of SILAR method.

• non-formation of precipitate in solution
• Save material costs
• does not require any capital and expensive instrument
• Does not need vacuum chamber
• low deposition temperature, energy saving
• good growth rate of films
• Thickness, particle size and morphology of the films can be easily controlled by changing the deposition cycle
• Stoichiometry of deposited material can be controlled by changing the concentration of precursor solution
• Thin films can be deposited onto any type of substrate

• Deposition on large area
• Simple and inexpensive method

Korosec and co-workers [44] synthesised nickel oxide/hydroxide thin film by dipping in nickel sulphate and lithium hydride solution following by heat treatment. Nickel hydroxide decomposes into nickel oxide at 220 °C and xerogel (325 °C). The presence of sulphate and carbonate ions could be observed in the samples as indicated in Fourier-transform infrared spectroscopy (FTIR) spectroscopy. Optimization of electrochromic responses is achieved for film obtained by 65 % thermal decomposition of nickel hydroxide. Nanoparticles with average diameter of 2-3 nm are embedded in amorphous matrix based on TEM analysis. Akaltun and co-workers [45] successfully deposited NiO thin film on glass substrate using SILAR routes. During the deposition process, nickel-ammonia complex ions (cationic precursor) and distilled water (anionic precursor) were used. The film thickness increases from 125 to 324 nm with increasing dipping cycle from 40 to 120 cycles. Crystallite size increases from 8.8 to 12.7 nm up-to 80 dipping cycles then decreases (9.3 nm) and band gap decreases from 3.85 to 3.64 eV. SEM study shows these films consist of uniform, smooth, dense and well-adherent morphology all over the substrate. They also highlight that 264 nm thick film shows better surface properties and the lowest resistivity value of 4.1 at 500 K if compared to other samples. Taskopru and co-worker [46] successfully deposited undoped NiO and Co-doped (1, 3, 5, 7 %) NiO by using SILAR method. Nickel nitrate and cobalt nitrate serve as cationic precursor during the deposition process. XRD study shows formation of polycrystalline cubic structure NiO thin film in basic conditions, exhibited (111) and (200) preferential orientation. Crystallite size increases from 13 to 28 nm upto 5 % doping level and, crystallite size decreases to 23 nm (more than 7 % doping level). Atomic percentage of cobalt is higher in all doped films according to energy dispersive X-ray analysis (EDAX) analysis. Optical studies found that all the films show high transparency in the visible region and band gap energy decreases from 3.71 to 3.30 eV due to Co-doping.

Gund and co-workers [47] investigated the effect of bath temperature and time on supercapacitive performance of SILAR-deposited NiO thin films. Nickel sulphate complexed with NH_4OH in pH 12 (as cationic precursor) at room temperature (reaction time varied 20, 30 and 40 seconds) and 2 % H_2O_2 solution serves as anionic precursor (temperature is varied from 318, 333 and 348 K). The structure of obtained samples were confirmed by XRD analysis and Raman spectroscopy. The films prepared at 333K for 30 seconds, showed the highest surface area (122.3m^2/g), a uniform and porous nano-flake structure [48]. These materials indicated specific capacitance value (674 F/g at scan rate of 5 mV/s), current density (0.5mA/cm^2) with 72.5 % capacity retention after 2000 cycles. The effect of dipping cycle on the supercapacitive performance of nickel oxide films have been described by Das and co-workers [49]. The films prepared for 40 cycles showed the highest specific capacitance value (1343 F/g at a scan rate of 2 mV/s), specific energy (64.38 Wh/kg), and specific power (2305 W/kg). Morphology investigation revealed the films prepared for 40 cycles have more electro active sites if compared to other dipping cycle.

CONCLUSIONS

There are several types of thin films such as TiO_2, Fe-doped TiO_2, ZnO have been successfully deposited by spin coating method. The band gap of TiO2 was about 3.6 eV. Films thicknesses (TiO_2) dependence on the spin rate. The ankerite structure was identified through XRD patterns and SEM showed that the grain size decreases with increasing iron dopant. SILAR method was used to prepare nickel oxide films. XRD study shows formation of polycrystalline cubic structure NiO thin films. The band gap values were observed in the range of 3.85 to 3.64 eV.

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