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A comprehensive study of spin coating as a thin film deposition technique and spin coating equipment

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Abstract. Description and theory of spin coating technique has been elaborately outlined and a spin coating machine designed and fabricated using affordable components. The system was easily built with interdisciplinary knowledge of mechanics, fluid mechanics and electronics. This equipment employs majorly three basic components and two circuit units in its operation. These include a high speed dc motor, a proximity sensor mounted at a distance of about 15 mm from a reflective metal attached to the spindle of the motor to detect every passage of the reflective metal at its front and generate pulses. The pulses are transmitted to a micro-controller which process them into rotational speed (revolution per minute) and displays it on a lead crystal display (LCD) which is also a component of the micro-controller. The circuit units are a dc power supply unit and a PWM motor speed controlling unit. The various components and circuit units of this equipment are housed in a metal casing made of an 18 gauge black metal sheet designed with a total area of 1, 529.2 cm^2. To illustrate the use of the spin-coating system, ZnO sol-gel films were prepared and characterized using SEM, XRD, UV-vis, FT-IR and RBS and the result agrees well with that obtained from standard equipment and a speed of up to 9000 RPM has been achieved.

Keywords: spin coating; proximity sensor; micro-controller, components; equipment, substrate

1. Introduction

Spin coating is a procedure used to apply uniform thin films to flat substrates. A typical process involves depositing a small puddle of a fluid resin onto the center of a substrate and then spinning the substrate at high speed (typically around 3000 rpm) (Mitzi *et al*. 2004). Centrifugal force will cause the resin to spread to, and eventually off, the edge of the substrate leaving a thin film of resin on the surface. Final film thickness and other properties will depend on the nature of the resin (viscosity, drying rate, percent solids, surface tension, etc.) and the parameters chosen for the spin process. Factors such as final rotational speed, acceleration, and fume exhaust contribute to how the properties of coated films are defined (Hellstrom 2007, Mitzi *et al*. 2004). A machine used for spin coating is called a spin coater, or simply spinner (Madou 2002, Mihi *et al*. 2006).

Rotation is continued while the fluid spins off the edges of the substrate, until the desired thickness of the film is achieved. The applied solvent is usually volatile, and simultaneously evaporates. So, the higher the angular speed of spinning, the thinner the film. The thickness of the

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film also depends on the concentration of the solution and the solvent (Hellstrom 2007).

Spin coating is widely used in micro-fabrication, where it can be used to create thin films with thicknesses below 10 nm (George *et al*. 2001, Chu *et al*. 2012, Hong and Han 2004). It is used intensively in photolithography, to deposit layers of photoresist about 1 micrometre thick (Hanaor *et al*. 2011, Wesam *et al*. 2009). Photoresist is typically spun at 20 to 80 revolutions per second for 30 to 60 seconds (Atthi *et al*. 2010, Sigma-Aldrich Co. 2004). Owing to the low values of thickness which can be achieved using spin coating methods, this method is often employed in the fabrication of transparent Titanium dioxide and Zinc oxide thin films on quartz or glass substrates, such thin film coatings may exhibit self-cleaning and self-sterilizing properties (Hanaor *et al*. 2011).

One of the most important factors in spin coating is repeatability. Subtle variations in the parameters that define the spin process can result in drastic variations in the coated film (Middleman and Hochberg 1993, Wang 2008). In spite of few studies regarding the spin coating method, the spin coating method has some merits, such as the easy control and handling of chemicals and substrates, and fabrication of thin film at faster rates and less vigour of high annealing temperatures and low cost (Kamaruddin *et al*. 2010, Ilican *et al*. 2008). This study will consider a comprehensive description of spin coating process and theory and a design and fabrication of a spin coating equipment at an affordable cost.

2. Description of spin coating process

There are four distinct stages to the spin coating process. These include:

2.1 A dispense stage

A typical spin process consists of a dispense step in which the resin fluid is deposited onto the substrate surface. Two common methods of dispense are: Static dispense, and Dynamic dispense. Static dispense is simply depositing a small puddle of fluid on or near the center of the substrate. This can range from 1 to 10 cc depending on the viscosity of the fluid and the size of the substrate to be coated (Hellstrom 2007, Middleman and Hochberg 1993). Higher viscosity and or larger substrates typically require a larger puddle to ensure full coverage of the substrate during the high speed spin step. Dynamic dispense is the process of dispensing while the substrate is turning at low speed. A speed of about 500 rpm is commonly used during this step of the process (Hellstrom 2007, Middleman and Hochberg 1993). This serves to spread the fluid over the substrate and can result in less waste of resin material since it is usually not necessary to deposit as much to wet the entire surface of the substrate. This is a particularly advantageous method when the fluid or substrate itself has poor wetting abilities and can eliminate voids that may otherwise form (Schubert and Dunkel 2003, http://www.holmarc.com/spincoatingmachine.html).

2.2 Substrate acceleration stage

This stage is usually characterized by aggressive fluid expulsion from the wafer surface by the rotational motion (Meyerhofer 1978, Mitzi *et al*. 2004). Because of the initial depth of fluid on the wafer surface, spiral vortices may briefly be present during this stage; these would form as a result of the twisting motion caused by the inertia that the top of the fluid layer exerts while the wafer

below rotates faster and faster (Shanshan *et al*. 2009). Eventually, the fluid is thin enough to be completely co-rotating with the wafer and any evidence of fluid thickness differences is gone. Ultimately, the wafer reaches its desired speed and the fluid is thin enough that the viscous shear drag exactly balances the rotational accelerations (Meyerhofer 1978, Mitzi *et al*. 2004).

Typical spin speeds for this stage range from 1500-6000 rpm, again depending on the properties of the fluid as well as the substrate (Souza *et al*. 2009). This step can take from 10 seconds to several minutes (Shanshan *et al*. 2009). The combination of spin speed and time selected for this stage will generally define the final film thickness (Meyerhofer 1978, Mitzi *et al*. 2004). In general, higher spin speeds and longer spin times create thinner films. The spin coating process involves a large number of variables that tend to cancel and average out during the spin process and it is best to allow sufficient time for this to occur (Meyerhofer 1978, Mitzi *et al*. 2004).

2.3 A stage of substrate spinning at a constant rate and fluid viscous forces dominate fluid thinning behaviour

This stage is characterized by gradual fluid thinning. Fluid thinning is generally quite uniform, though with solutions containing volatile solvents, it is often possible to see interference colours "spinning off", and doing so progressively more slowly as the coating thickness is reduced (Hanaor *et al*. 2011, Lin *et al*. 2007, Chibane *et al*. 2012). Edge effects are often seen because the fluid flows uniformly outward, but must form droplets at the edge to be flung off. Thus, depending on the surface tension, viscosity, and rotation rate among other factors, there may be a small bead of coating thickness difference around the rim of the final wafer (Hanaor *et al*. 2011, Meyerhofer 1978). Mathematical treatments of the flow behaviour show that if the liquid exhibits Newtonian viscosity (Newtonian viscosity implies that the shear rate is proportional to the shear stress) and if the fluid thickness is initially uniform across the wafer (albeit rather thick), then the fluid thickness profile at any following time will also be uniform leading to a uniform final coating (under ideal circumstances) (Middleman and Hochberg 1993, Emslie *et al*. 1958). In the spin coating process the interactions between substrate and solution layer are stronger than the interaction between solution surface layer and air. During the spin coating process the solvent evaporates, this leads to an increasing concentration and therefore increasing viscosity, which affects the pattern of formation (Suciu *et al*. 2011, Sevvanthi *et al*. 2012).

2.4 A stage of substrate spinning at a constant rate and solvent evaporation dominates the coating thinning behaviour

As the prior stage advances, the fluid thickness reaches a point where the viscosity effects yield only rather minor net fluid flow. At this point, the evaporation of any volatile solvent species will become the dominant process occurring in the coating. In fact, at this point the coating effectively "gels" because as these solvents are removed the viscosity of the remaining solution will likely rise effectively freezing the coating in place (Peeters and Remootere 2008, Meyerhofer 1978). (This behaviour was used in the seminal work of Meyerhofer where he quantified the coating thickness dependence on spin speed and viscosity and its relationship to the evaporation rate.) (Meyerhofer 1978).

After spinning is stopped many applications require a posterior heat treatment (as for "spin-on-glass" or sol-gel coatings). On the other hand, photoresists usually undergo other

processes, depending on the desired application/use (Habibi and Sardashti 2008, Suciu *et al*. 2011).

Clearly stages (iii) and (iv) describe two processes that must be occurring simultaneously throughout all times (viscous flow and evaporation). However, at an engineering level the viscous flow effects dominate early on while the evaporation processes dominate later (Meyerhofer 1978, Schubert and Dunkel 2003).

3. Theory of spin coating

3.1 Spin speed

Spin speed is one of the most important factors in spin coating. The speed of the substrate in rpm affects the degree of radial (centrifugal) force applied to the liquid resin as well as the velocity and characteristic turbulence of the air immediately above it (Hellstrom 2007, Supekar *et al*. 2013)). In particular, the high-speed spin step generally defines the final film thickness. Relatively minor variations of ±50 rpm at this stage can cause a resulting thickness change of 10% (Hellstrom 2007, Mihi *et al*. 2006). Film thickness is largely a balance between the force applied to shear the fluid resin towards the edge of the substrate and the drying rate which affects the viscosity of the resin. As the resin dries, the viscosity increases until the radial force of the spin process can no longer appreciably move the resin over the surface. At this point, the film thickness will not decrease significantly with increased spin time (Peeters and Remootere 2008, Emslie *et al*. 1958, Lin *et al*. 2007).

When the centrifugal and viscous forces are in balance, the equation below must be satisfied (Emslie *et al*. 1958)

$$-\eta \frac{\partial^2 v}{\partial z^2} = \rho \omega^2 r \tag{1}$$

Where *z* and *r* define a cylindrical coordinate system aligned with the axis of substrate rotation, *v* is the fluid velocity in revolution per second in the radial direction (a function of depth), and ρ is the fluid density in grams per centimeter cube, ω is the rotation rate in radians per second, while η is the viscosity in poise. With appropriate flow and velocity boundary conditions, and considering a film that is initially uniform, the film thickness as a function of time, *h(t)*, is given by (Emslie *et al*. 1958, Peeters and Remootere 2008)

$$h = \frac{h_o}{\sqrt{1 + 4Kh_o^2 t}} \tag{2}$$

where h_o is the film thickness at time zero (but not physically meaningful because of the first stage of unstable solution expulsion at early time), and *K* is a system constant defined by

$$K = \frac{\rho \omega^2}{3\eta} \tag{3}$$

These equations are strictly valid only when K is constant. However, for spin coating of sol-gel or other complex solutions this may not hold during all stages of spinning. Both viscosity and density are expected to increase as evaporation progresses, so caution must be used when applying these equations. In the early stages of fluid thinning (before evaporation becomes important), the thinning rate would be defined by (Emslie *et al*. 1958, Hanaor *et al*. 2011, Middleman and Hochberg 1993)

$$\frac{dh}{dt} = -2Kh^3 \tag{4}$$

At longer times, solvent evaporation becomes an important contribution. Meyerhofer was the first to estimate the effect of this on final coating thickness. A quite reasonable approximation is that evaporation is a constant throughout spinning, as long as the rotation rate is held constant (see below). Therefore, he simply added a constant evaporation term to the equation above. So, the governing differential equation became (Hanaor *et al*. 2011, Meyerhofer 1978, Schwartz and Roy 2004)

$$\frac{dh}{dt} = -2Kh^3 - e \tag{5}$$

where "e" is the evaporation rate [ml/s/cm^2] (this is effectively the contribution to the interface velocity that is driven by the evaporation process alone).

Instead of solving this equation explicitly, Meyerhofer assumed that early stages were entirely flow dominated, while later stages would be entirely evaporation dominated. He set the transition point at the condition where the evaporation rate and the viscous flow rate became equal. This can be thought of as the fluid-dynamical "set" point of the coating process. When these assumptions are made, the final coating thickness, h_f, is predicted by

$$h_f = C_o\left(\frac{e}{2(1-C_o)K}\right)^{\frac{1}{3}} \tag{6}$$

where C_o is the solids concentration in the solution (Hanaor *et al*. 2011, Schwartz and Roy 2004). When the physically applicable dependence of the evaporation rate on spin-speed was factored in, this was successful in matching the regular exponents for the dependence of final film thickness with spin speed. Research has shown that the evaporation rate should be constant over the entire substrate and depend on rotation rate according to

$$e = C\sqrt{\omega} \tag{7}$$

where the proportionality constant, C, must be determined for the specific experimental conditions (Hanaor *et al*. 2011, Meyerhofer 1978, Hellstrom 2007). This square root dependence arises from the rate-limiting-step being diffusion through a vapour boundary layer above the spinning disk. It should be noted that this results when airflow above the spinning substrate is laminar (Hellstrom 2007; Birnie 2013).

3.2 Acceleration

The acceleration of the substrate towards the final spin speed can also affect the coated film properties. Since the resin begins to dry during the first part of the spin cycle, it is important to accurately control acceleration. In some processes, 50% of the solvents in the resin will be lost to evaporation in the first few seconds of the process (Peeters and Remootere 2008, Lin *et al*. 2007).

Acceleration also plays a large role in the coat properties of patterned substrates. In many cases the substrate will retain topographical features from previous processes; it is therefore important to uniformly coat the resin over and through these features (Peeters and Remootere 2008, Hellstrom 2007).

While the spin process in general provides a radial (outward) force to the resin, it is the acceleration that provides a twisting force to the resin. This twisting aids in the dispersal of the resin around topography that might otherwise shadow portions of the substrate from the fluid (Hanaor *et al*. 2011, Peeters and Remootere 2008). In operation the spin motor accelerates (or decelerates) in a linear ramp to the final spin speed.

3.3 Fume exhaust

The drying rate of the resin fluid during the spin process is defined by the nature of the fluid itself (volatility of the solvent systems used) as well as by the air surrounding the substrate during the spin process (Oliveira *et al*. 2012, Middleman and Hochberg 1993). Just as a damp cloth will dry faster on a breezy dry day than during damp weather, the resin will dry depending on the ambient conditions around it (Ilican *et al*. 2008). It is well known that such factors as air temperature and humidity play a large role in determining coated film properties. It is also very important that the airflow and associated turbulence above the substrate itself be minimized, or at least held constant, during the spin process (Middleman and Hochberg 1993, Peeters and Remootere 2008).

All spin coaters employ a “closed bowl” design. While not actually an airtight environment, the exhaust lid allows only minimal exhaust during the spin process. Combined with the bottom exhaust port located beneath the spin chuck, the exhaust lid becomes part of a system to minimize unwanted random turbulence. There are two distinct advantages to this system: slowed drying of the fluid resin and minimized susceptibility to ambient humidity variations (Middleman and Hochberg 1993, Peeters and Remootere 2008).

The slower rate of drying offers the advantage of increased film thickness uniformity across the substrates (Middleman and Hochberg 1993, Peeters and Remootere 2008). The fluid dries out as it moves toward the edge of the substrate during the spin process. This can lead to radial thickness non-uniformities since the fluid viscosity changes with distance from the center of the substrate (Swati *et al*. 2006). By slowing the rate of drying, it is possible for the viscosity to remain more constant across the substrate (Middleman and Hochberg 1993, Peeters and Remootere 2008).

Drying rate and hence final film thickness is also affected by ambient humidity. Variations of only a few percent relative humidity can result in large changes in film thickness (Peeters and Remootere 2008). By spinning in a closed bowl the vapours of the solvents in the resin itself are retained in the bowl environment and tend to overshadow the effects of minor humidity variations (Aguilar and López 2011). At the end of the spin process, when the lid is lifted to remove the substrate, full exhaust is maintained to contain and remove solvent vapours (Peeters and

Remootere 2008).

Another advantage to this "closed bowl" design is the reduced susceptibility to variations in air flow around the spinning substrate (Shrestha *et al*. 2010). In a typical clean room, for instance, there is a constant downward flow of air at about 100 feet per minute (30m/min). Various factors affect the local properties of this air flow (Al-Juaid *et al*. 2012). Turbulence and eddy currents are common results of this high degree of air flow. Minor changes in the nature of the environment can create drastic alteration in the downward flow of air. By closing the bowl with a smooth lid surface, variations and turbulence caused by the presence of operators and other equipment are eliminated from the spin process (Mitzi *et al*. 2004).

4. Design of the spin coater

4.1 Materials/Components consideration

The Spin coater to be designed in this work consists of following basic component parts:

a. A high speed motor operated from a12 V-5 Ampere DC power source
b. A 12 V, 5 Ampere DC Power Supply
c. A pulse width modulation (PWM) motor speed controller
d. A proximity sensor to detect the rotation of the motor whose speed is being measured and
e. A microcontroller/alpha-numeric LCD module.

4.2 Design analysis

4.2.1 The DC power supply

The DC Power Supply to be considered must be able to deliver an output of 12 V, 5 A from the AC mains of (220-240) V_{rms}, 50 Hz (Nigeria line) (VRIOG steering committee 2009). The realization circuit is shown in Fig. 1.

The input transformer, T has a primary rating of 220 V for the Nigerian domestic supply. The secondary must be cable of supplying 10 V AC RMS at 5 Amp. The supply is rectified by the bridge rectifier (BR), using four 1N4007 rectifier diodes. At the bridge rectifier, there is a voltage drop of 1.4 V because each diode uses 0.7 V when conducting and there are always two diodes conducting (Hewes 2011). The smoothing capacitor, C1 is determined by (Tyona and Akande 2008, Schuler 1999, VRIOG steering committee 2009)

where

C = smoothing capacitance in farads (F)

I_o = output current from the supply in amps (A) -2A

V_s = supply voltage in volts (V), this is the peak value of the unsmoothed DC, $8.6V_{RMS}$

f = frequency of the AC supply in hertz (Hz), 50 Hz in Nigeria.

Thus $C \cong 22000\,\mu F$.

The resistor R1 needs to be large enough to support the time constant, $\tau = RC$ of the filtering capacitor such that it does not discharge appreciably before the next half of the cycle resumes (Schuler 1999, Holt 1978). Hence R1 is chosen to be 27 kΩ.

Filtering or smoothing significantly increases the average DC voltage to almost the peak value (1.4 × RMS value); that is, the filtered output is 1.4 × 8.6 = 12.04 V DC (Tyona and Akande 2008,

Hewes 2011, Holt 1978).

4.2.2 The pulse width modulation (PWM) motor speed controller

The PWM motor speed controller is expected to vary the 12 V DC supply to the motor such that the speed of the motor becomes varied in steps. The realization circuit is shown in Fig. 2.

The heart of this system is the IC, CD4093 which is a quad 2 input NAND Schmitt trigger (Shinde *et al*. 2002). The system is operated from the 12 V DC power supply at a current level of 5A. The Schmitt triggers from four in 4093, that is U1a cable with cycle oscillator is adjustable. The U1b, U1c, U1d are buffer output from the oscillator to drive the switching MOSFET, Q1. The DC motor drives in accordance with the switching pulses from the oscillator. When R2 is varied, cycle varied and so is the motor speed (Ahmed and Alam 2013). Diodes D1 and D2 provides current paths between 4093 and R2, thus inhibiting current re-flux into the IC. Hence, 1N4004, a rectifier diode is chosen. R1 is a small value resistor which splits out current to R2, thus preventing overload current at low resistance. Hence, 1 kΩ is chosen.

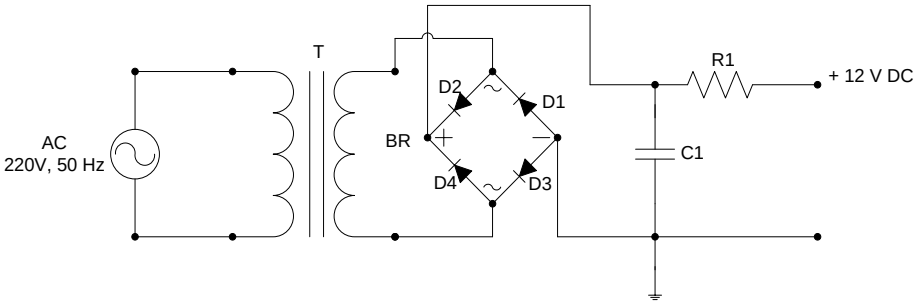

Fig. 1 Realization circuit of a DC Power Supply Circuit

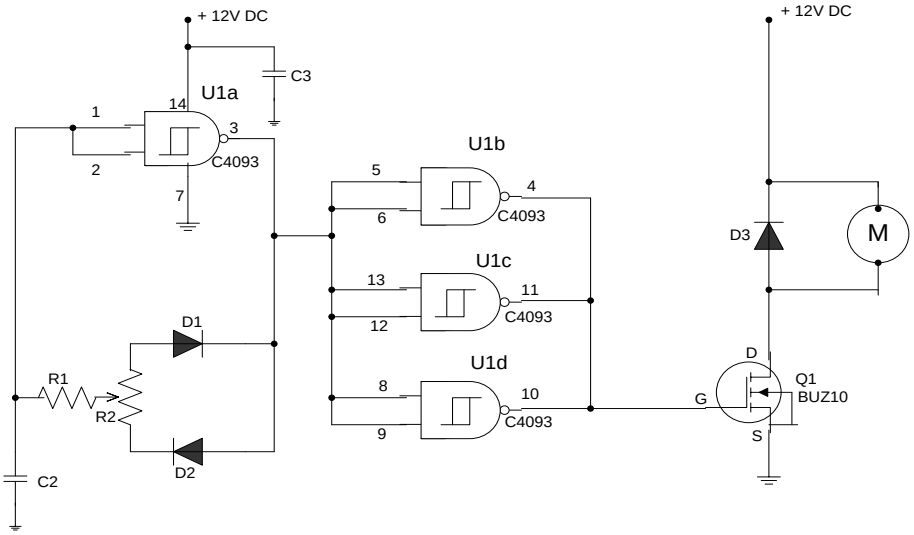

Fig. 2 Pulse width modulation (PWM) motor speed controller

4.2.3 The proximity sensor and the Microcontroller

The proximity sensor is mounted at a distance of about 10 mm to 20 mm from a reflective metal attached to the rotating spindle of the DC motor. The sensor detects the reflective element passing in front of it and thus gives an output pulse for each and every rotation of the motor. These pulses are fed to the microcontroller and counted. The counted pulses in the microcontroller are processed to rotational speed (revolutions). An LCD unit which is a component of the microcontroller display the speed in revolutions per minute (RPM).

5. Fabrication

5.1 Circuitries

The circuits, DC power supply and the PWM motor speed controller were constructed by mounting the components on a bread board (test board) as one circuit to confirm proper workability. After confirmation, the components are then transferred to a vero board and soldering is done to obtain firm positioning and electrical contacts. The component layout is shown in Fig. 3.

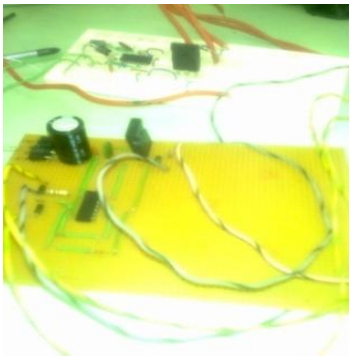

Fig. 3 Component layout

Fig. 4 Structure of the spin coater casing, (a) Side view and (b) front view

Fig. 5 Component assembly

5.2 Casing

The various components of this equipment are housed in a metal casing made of an 18 gauge black metal sheet designed with a total area of 1, 529.2 cm^2. A space of diameter 14 cm and 7 cm deep was designed on the top surface to house the bowl; this guaranteed the use of a 10 cm to 13 cm bowl. A top cover is designed with an area of 822.5 cm^2 to cover the bowl space to prevent dust and other unwanted particles from contaminating the bowl and the substrate (closed bowl system). The case structure is shown in Fig. 4.

5.3 Component assembly

The 14 cm-diameter bowl space determines the position of the motor which was positioned centrally. The proximity sensor was mounted on a holder and positioned 15 mm from the reflective metal attached to the rotating spindle of the motor. At the back of motor, the transformer was mounted while the micro-controller, the switch and the potentiometer spindle were positioned at the front. The circuit board was positioned beside the motor and connecting cables strategically arranged as illustrated in Fig. 5.

6. Test

The fabricated spin coating equipment in this work was adequately tested to ascertain its workability. The following tests were carried out:

i. The equipment was run continuously and repeatedly for 30 minutes and no over heating of electrical and electronic components was observed. This guarantees that the equipment can sustain a long experimental stress time.

ii. A trial film deposition of ZnO on glass substrate was carried out successfully and characterized using SEM, XRD, UV-vis, FT-IR and RBS and the result agrees well with that obtained from standard equipment.

After performing several tests of the device, initial conditions that can be recommended for a

good film deposition are the following:

- It must be assured that the plane of the disk that supports the substrates is perfectly leveled with the horizontal;
- The solution to be deposited should wet the substrate. If this does not happen, the solution will be thrown away from the substrate by centrifugal force without leaving any trace of material on the substrate. The flatness and/or roughness of the substrate are also a parameter that could affect the deposit;
- The solution that contains the material to be deposited should be uniform and homogeneous, free of lumps and air bubbles. The substrate must be clean and free of dust particles;
- There is an optimal relationship between spinning speed and concentration of the solution so that viscosity does not become an impediment to the flow of the solution on the substrate during spinning. It is not possible to establish a simple rule to determine these optimal conditions because there is a complex interplay between the material to be deposited, the substrate, the volatility of the solvent, spinning acceleration and speed, and even with turbulence of the surrounding air and conditions of atmospheric humidity (Aguilar and López 2011).

7. Conclusions

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