

Thin Film Deposition: Solution Based Approach

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Abstract

The wet chemical processing opens the way to deposit thin film with the versatility and ease for a variety of materials. Liquid film deposition involves the application of a liquid precursor on a substrate which is then converted to the required coating material in a subsequent post-treatment step. Different non-vacuum solution based deposition techniques have been developed to grow thin films with high efficiency and functionality. Spin coating is one of an effective technique for thin film fabrication due to low cost, uniformity, less hazardous, and capability of easy scaling up. The typical process involves depositing a small amount of a fluid onto the center of a substrate and then spinning the substrate at high speed. Dip coating is another simple, cost effective route with feasibility to scale-up for commercial production. The dip coating process can be divided into three important technical stages, immersion, withdrawal and evaporation. The coating may be subjected to further heat treatment in order to burn out residual compounds and induce crystallization of the functional oxides. Spray coating is a promising technique to grow thin film in research and industry to prepare thin and thick films. It is an easy approach to fabricate thin film with uniform distribution at small scale from a few nanometers to micrometers in thickness. Inkjet printing is the emerging promising technique to develop large-scale, and flexible thin films. The inkjet printing process allow easy customization to grow variety of complex structures.

Keywords: thin film deposition, spin coating, dip coating, spray coating, inkjet printing

1. Introduction

This chapter includes the details of solution based routes to deposit thin films which includes spray coating, dip coating, spin coating and inkjet printing processing. The contribution of different experimental parameters such as solution viscosity, surface tension, droplet size, substrate material & temperature, nature of solution are discussed briefly.

2. Spin coating

Spin coating is a quick and common route to deposit thin films on substrates with primary advantage of ease to produce very uniform films. The solution of a specific material is spun at high speeds, the centripetal force and the surface tension of the liquid together create an even covering on the substrate. The excessive solvent

is evaporated, and spin coating results in a thin film ranging from a few nanometers to a few microns in thickness. Spin coating technique is used to coat small substrates from a few mm square to a metre or more in diameter. The key advantage of spin coating technique is the simplicity and relative ease to set up the process, coupled with the thinness and uniformity.

Spin coating consists of three major stages, solution dispensing, rotation dominated thinning and solvent evaporation as shown in **Figure 1**. The rotation pulls the liquid (solution) coating into an even covering and then evaporates to leave the desired material on the substrate in an even distribution. The high spin speeds and the high airflow leads to fast drying, which in turn results in high consistency at both macroscopic and nano length scales. However, the fast drying times lead to lower performance for certain processes, which requires time to self-assemble and/or crystallize.

Spin coating also relatively low throughput process due to an inherently batch (single substrate) process compared to roll-to-roll processes. Despite these drawbacks, spin coating is usually the starting point and benchmark for most academic and industrial processes that require a thin and uniform coating. Spin coating process can be broadly divided into 4 main stages.

Deposition: The first step involves the solution dispensing onto the substrate surface. The centrifugal force pulls the solution across substrate surface, if the substrate is already spinning (dynamic spin deposition) or is set on spin after deposition (static spin deposition).

Spin up: Once the solution is translated to the substrate surface, the rotation disc is speed up to the required rotation speed, either immediately or by a gradual speed up steps. In this stage, the solution initially spins at a different rate than the substrate speed, but eventually the two rotation speeds match up once the drag force balances rotational accelerations leads to the formation of thin fluid layer.

Spin off: During spin off stage, the solution begins to turn into thin film dominated by viscous forces. As the solution is flung off the substrate, the thin film changes color due to interference effects and it indicates completion of dry process.

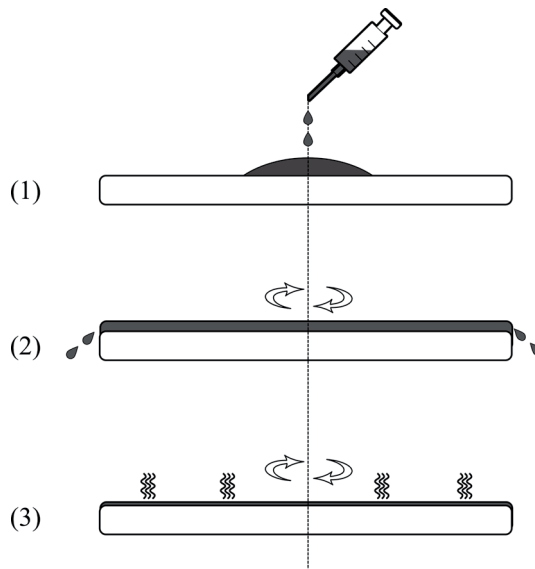


Figure 1.
Schematic of spin coating.

In certain cases, the edge effects are observed on the substrate due to formation of droplets at the edge and to be thrown off.

Evaporation: As fluid outflow ceased, the thinning process is dominated by evaporation of the solvent. The rate of solvent evaporation depends on the solvent vapor pressure, volatility, and ambient temperature.

All above processes are repeated several times to research desired film thickness. The solution casting and drying stages of are an integral and crucial part of the spin coating process, which contributes to the key process such as stacking/crystallization, phase separation and aggregation. Precise control of these processes is critical as the characteristics of deposited thin films not only depend upon morphology (thickness, uniformity) but also on deposition processing. In general, the spin speed speeds of >1000 rpm is recommended for industrial processing to ensure the high uniformity. However, spin speeds down to 200 rpm can be employed for laboratory scale deposition which might slow down the drying process but allow additional time for self-assembly.

In case of slow spinning, the solvent begins to evaporate as solvent is dispensed across the substrate and produces internal currents resulted in formation of highly ordered well assembled thin films by slowing down the evaporation rate. But, the coating across the surface is usually highly uneven with a typical “coffee staining” effect. However, it is possible to obtain a high level of nanoscale order by spinning at very lower speeds. Slow drop casting without rotation is a good way to deposit highly ordered thin films at the nanoscale but at the expense of uniformity.

In general, the thickness of a spin coated thin film is proportional to the inverse of the spin speed squared. However, the relation does not always apply and use to predict the film thickness without experimental data. Usually a test film is grown and measured. By using the data point(s) obtained from test film, spin thickness curve can be plotted with a reasonable accuracy. The spin speed can then be adjusted to obtain the right film thickness. The exact thickness of a deposited thin film is subjected to many factors as the material concentration and solvent evaporation rate which in turn depends upon the solvent viscosity, vapor pressure, temperature and local humidity etc. Therefore, the spin thickness curves for specific solution is commonly determined empirically [1].

The following parameters play very crucial role during spin coating process of thin film deposition.

Spin Speed: The spin speeds is one of the important parameter as it defines the range of thicknesses that can be achieved from a given solution. The speed affects the degree of centrifugal force applied to the liquid fluid as well as the velocity and characteristic turbulence of the air immediately above it. As the substrate starts spin, the dispensed solution flows radially, due to the action of centrifugal force, and the excess fluid spin off the substrate. The fluid continues thinning until the centrifugal force equals the viscosity. Although spin coating process is divided in three stages, however, they are not always very distinctly separate in time. In general, spin coating produces a uniform thin film relatively easily from about 1000 rpm upwards. A good quality thin film can be deposited at 500 or 600 rpm with precise control on spin speed. In general, the spin coating rpm might span a factor of ten (e.g. from 600 rpm to 6000 rpm) which in turn cause variation in film thickness. For instance, a solution which gives a film thickness of 10 nm at 6000 rpm may give a thickness of around 32 nm at 600 rpm. The dependence of thin film thickness upon the square root of spin speed has its own merits and demerits. The spin coating allows a precise control on the thickness of film but that can be achieved in a relatively narrow range for a given solution. The thickness of

thin film for a given material/solvent combination also depends upon the maximum concentration of the material dissolved in the solvent. The thickness of $>1\ \mu\text{m}$ can be achieved for high solubility materials (100 mg/ml or higher). However, for less soluble materials (a few mg/ml), the maximum thickness might be limited to 20 nm or so and the thickness of a film is approximately linearly dependent upon the concentration of the material in the solution, however increase in the material concentrations affects the viscosity of the solution and thus a non-linear relationship develops. Films deposited at slow spin speed exhibit more edge-on orientation as compared to films at fast spin speed which exhibit more plane-on orientation, and the orientation varies smoothly between these limits [2].

Spin Coating Duration: In most standard spin coating processes, the substrate is kept spinning until the film is fully dried, which mainly depends upon the boiling point and vapor pressure of the solvent at ambient conditions. In general, 30 seconds are usually an adequate time for most of solvents. However, the drying may take considerably longer for higher boiling point solvents therefore solvents are most commonly used either as additives or with additional drying steps. The spin coating process involves a large number of variables that tend to cancel and average out during the spin process and allows sufficient time to manipulate deposition process. By combining the effects of fluid flow and solvent evaporation to leave a very thin layer of coating material on the substrate [3]. During evaporation process while spinning, the viscosity increases until equals the centrifugal force and spin time cannot move the solution anymore. At this point, the film thickness does not change significantly with increased spin time. This step can take from 10 seconds to 1 minute.

Solution Dispensing: Another important aspect is dispensing of the solution on substrate either in a static dispense deposition or a dynamic dispense deposition process. In a static dispense procedure, the solution is translated to the substrate while it is stationary. The entire substrate is covered with solution before rotation begins. The spinning is then started and raised up to required speed as fast as possible. The static dispense procedure usually covers the entire substrate (or at least all of the active part of the substrate) prior to initiate the spinning. The major challenge for a static dispense is the evaporation of solvent in the solution before the spin coating process begins. This issue is more pressing in case of high vapor pressure solvents. Therefore, the static dispense process gives lower substrate-to-substrate film uniformity as compared to dynamic dispense.

In a dynamic dispense, the substrate is first set on spinning and allowed to acquire desired speed and then the solution is dispensed at the center of the substrate. The centripetal force pulls the liquid away from the middle of substrate across the entire area before dries up. The dynamic dispense is more suitable due to precise controlling and better substrate-to-substrate variation. In this process, the solvent has less time to evaporate before the spinning start. Therefore, the ramp speed and dispense time is less critical as the substrate has been reached to the desired rpm. The dynamic dispense requires less solution in general although depends upon the wetting properties of the surface.

The major setback of the dynamic dispense is the incomplete substrate coverage in case of low spin speeds below 1000 rpm and viscous solutions due to insufficient centripetal force to pull the liquid across the surface. The lower rotation speed enhances the probability that the solution may be dispensed before the substrate has completed a full rotation. For the majority of spin coating above 1000 rpm, a dynamic dispense is recommended as standard process. A dynamic dispense process can be performed at speeds all the way down to around 500 rpm. However it becomes more difficult to get complete surface coverage.

The nature of solution: In spin coating process, the choice of the source compound and solvent must satisfy a number of essential conditions such as stable in air at room temperature, less or no toxic. In spin coating process different parameters of solution as viscosity, concentration, density, surface tension, evaporation rate of the solvent determines the spin speed and spin time to get desired thickness.

The nature of substrate: The nature of substrate material is also important parameter as it contributes towards the good coverage of substrate surface and quality of deposited thin film. Generally the substrate is chemical resistance to the coating solution and a heat resistance to withstand the subsequent curing or heat treatment. In certain cases, a surfactant is introduced into the formulation to facilitate complete substrate coverage during dispense or modifications are applied to the substrate to ensure good wetting. For instance, the substrate surface is changed from hydrophobic to hydrophilic depending on the nature of solution [1].

3. Dip coating

The dip coating is a facile, simple, the low cost and the high quality coating processing used for industrial as well as laboratory applications. The dip coating is commonly used for optical coatings such as in the production of automotive rear mirrors and large area antireflective coating for solar control glasses [4]. The dip coating process involves immersing of a substrate into the solution of coating materials and then withdraw the solution. The process can be defined as deposition of aqueous-based liquid phase onto the surface of substrate using a solution. Generally, the required material is dissolved in solutions and directly coated on the substrate surface, then the sedimentary (solvent) wet coating is evaporated to get dry film. The dip-coating process involves complex chemical and physical multi-variable parameters. The film thickness and morphology depends on immersion time, withdrawal speed, dip-coating cycles, density and viscosity, surface tension, substrate surface and evaporation conditions of coating solutions. Photo-assisted dip-coating is used to control the evaporation process of coated solution and the irradiation effect facilitate the film disposition. To increase the uniformity and thickness of films, the multi-layered dip-coating is applied [5].

Dip coating consists of four basic steps as immersion, dwelling, withdrawal, and drying as shown in **Figure 2**. In first stage, the substrate is immersed into a solution to be deposited until it is completely covered with liquid. The substrate is then

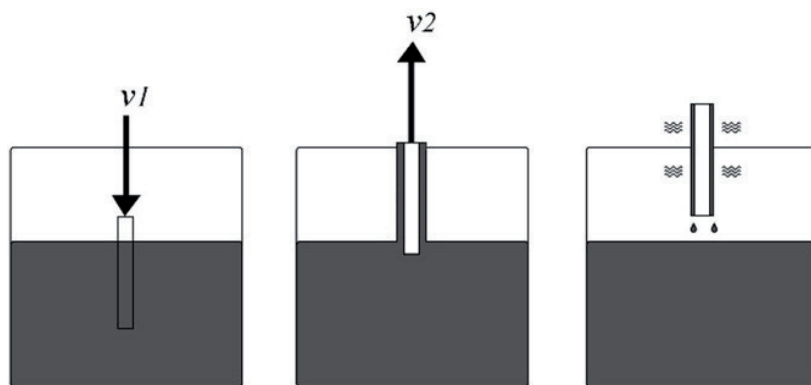


Figure 2.
Schematic of dip coating in lab.

withdrawn after a short interval. During the withdrawal process, a thin layer of the solution residues on the substrate surface. Once the substrate is fully withdrawn, the solution from the deposited film starts to evaporate and leaves behind a dry film. The deposited material undergoes a chemical or physical change.

The withdrawal and drying stages are critical stages to determine the properties of the deposited film. The withdrawal stage involves the interaction of different sets of forces. These forces are categorized into two categories, entraining forces and draining forces. Draining forces draw the liquid (solution) back to the bath from substrate surface. On the other hand, the entraining forces keeps the solution onto the substrate surface. The balance between these two sets of forces determines wet film thickness coated onto the substrate. During the withdrawal stage, the formation of the wet film can be broken into four regions as illustrated in **Figure 3**.

In the static meniscus region, the shape of the meniscus is determined by the balance of the hydrostatic and capillary pressures. Whereas the dynamic meniscus region occurs around the stagnation point where the entraining forces and draining forces are in equilibrium. In constant thickness zone, the wet film achieves a given thickness. The dynamic meniscus and the flow of solution in this region determine the wet film thickness. The transition between the dynamic and static meniscus happens within the boundary layer. Beyond the boundary layer, the draining forces are significantly higher as compared to the viscous forces and the balance between the capillary and hydrostatic pressure governs the meniscus shape.

Dip coating typically has three different stages for drying, drying front during coating, the falling rate period and the constant rate period. The simplest drying stages are the constant and the falling rate periods. The constant rate period occurs within the constant thickness zone which involves the evaporation of solvent at the surfaces of wet film across whole area. The only exception is the edges of the substrate, where the drying front occurs. Over the time after deposition, most of the solvents is evaporated from the wet film and left a gel-like film. This is when the

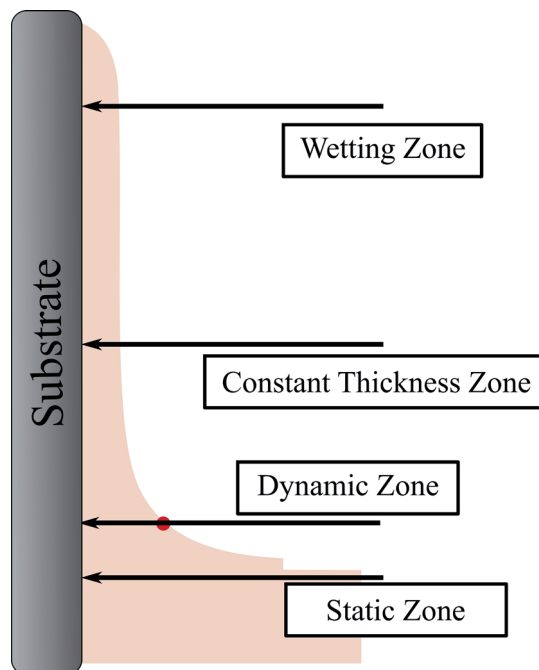


Figure 3.
Different regions during dip coating film formation.

falling rate period starts. In falling rate period, small amount of solvent left trapped within the gel material and the evaporation is determined by the diffusion of solvent towards the surface. The drying front appears at the interface between the wet film and substrate. Due to the large surface to volume ration, the evaporation occurs much faster at the surface which leads to the formation of a wet film with higher concentration. The more complex drying stage occurs at the drying front.

Several theoretical formulas were established to predict the thickness of deposited films, such as the Landau-Levich theory, via the following equation:

$$t_1 = 0.944 C_a^{1/6} \left(\frac{\eta U}{\rho g} \right)^{1/2} \quad (1)$$

where C_a is the capillary number and given by $C_a = \frac{\eta U}{\delta}$; η , δ , and ρ denote viscosity, surface tension, and the density of coating solutions, respectively; U is the withdrawal speed and g is the gravitational acceleration constant [5].

4. Spray coating

In recent years, spray coating has emerged as a viable approach for low-cost deposition of solution-processed thin films. Spray coating is a large-area, high-throughput, inexpensive, and industrially scalable process that can be used to create thin films of material which conform to the shape of the substrate. Spray coating involves ejecting fine liquid particles of smart materials by a jet stream of carrier gas onto the substrate as illustrated in **Figure 4**. Spray coating is a contact-free approach suitable for any substrate material and is particularly appropriate for low temperature processing [6]. The dynamics of spray droplet impingement on a substrate surface is a complex fluid mechanics problem subjected to different details, such as spreading, splashing, rebounding, coalescence and interaction with other droplets, drying phenomena, wetting/dewetting, and etc. Substrate properties such as roughness, permeability and surface energy also contribute significantly towards the droplet spreading and surface wetting. Spray coating on a permeable and rough surface hinders droplet spreading and increases the chance of splashing. Solution absorption by the substrate may also slow down droplet spreading. Therefore, droplet impact dynamics, such as droplet size and velocity, requires adjustment to enhance spreading and surface coverage on rough and permeable surface. The

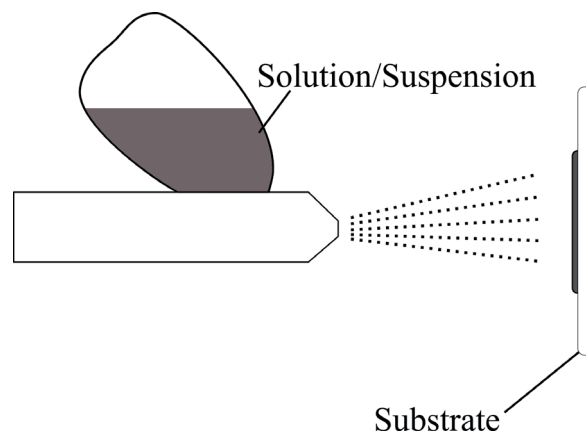


Figure 4.
Schematic of spray coating.

functionality of deposited thin film has a direct dependence on structure, morphology, roughness, and integrity of the stacked thin solid films [7].

The atomization process involves liquid breakup by the application of mechanical energy, which results in the production of a spray consist of micron-size drops. The solution characteristics such as liquid–vapor surface tension, viscosity, and density has direct influence on the atomization process. Atomization takes place when the dynamic pressure of an external force normally applied by a gas exceeds the internal pressure of the liquid droplet. These properties also affect morphological uniformity of deposited thin films. The increase in density hinder the movement of particulates and improve the morphological uniformity of a sprayed film [6].

Thin film formation by spray coating can be achieved by two different routes: one is by a drop-by-drop film formation approach, where the thin solid film is grown by impingement of a large number of individual droplets on the substrate surface that dry upon impact to cover the entire area. In second approach, the film is formed by immediate merging of impacted droplets and converted to a liquid film and to a solid film upon drying. The desired good quality solid film can be grown through drying of a liquid film if the spray process conditions such as the spray flow rate, and substrate temperature are optimized [7]. The influence of different experimental parameters on the thin film growth via spray coating is discussed as follow.

Effect of nozzle tip speed and solution precursor flow rate: The spray nozzle is not only deliver a mass of the solution to the substrate but also contributes towards the film characteristics as well by defining the number density, and size of the impinging droplets. The nozzle speed, nozzle height (distance from substrate), and flow rate (FR) (i.e., rate of liquid ejection from the spray nozzle) are closely related as they control the amount of liquid deposited per unit area [6, 7].

Effect of Number of Spray Cycles: Increase in the number of spray cycles considerably enhance the surface coverage and increases the film thickness. However, the influence of the number of spray cycles on film roughness is subjected to the substrate temperature. The application of the second spray cycle results in a smoother film, as the second layer generally covers up the cavities, pinholes, and defects of the first layer. This hypothesis is correct for low substrate temperatures. However at high substrate temperature, droplets of the first cycle are dried quickly and left behind a layer or film with irregular structure or grains. The forthcoming droplets from second spray cycle on rough layer at high substrate temperature dry quickly before they can spread and cover up the defective site and non-uniformities produced by the first layer [7].

Effect of Substrate Temperature: In general, the effect of substrate temperature on film characteristics depends on physical properties and evaporation rate of the precursor solution. Fabrication of a thin film at a high substrate temperature is determined by the balance between the heat transfer, droplet dynamics, and mass diffusion. The solvents are usually evaporated quickly, once the solution droplets impact on a hot substrate surface. Thus, there is a short time for droplet to spread and coalescence hinder surface wetting and complete coverage. At high substrate temperature, the precursor solution viscosity increases rapidly and further hinders droplet spreading. Despite the disadvantages of high substrate temperatures, the high temperature treatment can reduce the coffee ring effect and enhance high supersaturation with fast precipitation, which in turn can lead to the formation of well distributed and uniformly textured layers from each individual droplet. Therefore, a moderate temperature range can form a compromise between these two opposing effects (adequate and efficient coalescence and merging of impacted droplets on the substrate and timely evaporation and precipitation) may enhance the quality of the film [7].

Effect of Substrate Type: Regardless the substrate temperature, the coverage of films on regular surface is poor, due to limited spreading on rough substrates surface which leads to the higher film roughness, and the lower thickness is compared to the films made at the same condition but on the smooth surface. A large amount of the deposited precursor solution is consumed to fill the surface roughness and results in much less surface and higher non-uniformity and thickness. Implementation of a second spray cycle, however, can improve the coverage of coatings on rough substrate surface [7].

Effect of Substrate Vibration: To boost droplet coalescence and wetting process, the precursor solution is sprayed on substrate placed on an ultrasonic-assisted vibrating surface. A significant improvement in surface coverage, and films uniformity is observed when a vibrating substrate is employed. Substrate vibration improves coalescence and spreading of the impinging solution droplets, before being completely dried out. The impact of droplets onto a vibrating surface also results in further droplet breakup into several smaller droplets [7].

Distance between the nozzle and substrate: Some solvent boiling point is too low (63°C), the spray solution can dry before reaching the. It results in a thin film with relatively large density of pinholes and thickness variations, and the interface defect. Therefore, a proper nozzle-to-substrate distance is of great importance. The distance is determined by the solution properties, temperature of substrate and droplet size [8].

5. Inkjet printing

Inkjet printing is a relatively novel process compared to industrial printing and other thin film coating technologies. Inkjet printing involves delivering of a small volume of a fluid material, typically in the picoliter to nanoliter range, onto the substrate surface [9]. Inkjet printers comprises of three basic parts, the motion stage, control systems attached with the print heads, and vision system. The printer heads are connected directly to the cartridge filled with solutions. Inkjet printing systems typically require three mechanical degrees of freedom, one rotational (θ stage) and two translational (X and Y stages) to generate 2D patterns align with previously printed patterns and realize facily stacked structures. The vision system is employed for substrate alignment and to observe ejected droplets in flight to monitor the droplets. Whereas, the control systems is used to optimize the stage and the printer head temperature, which can directly influence the substrate temperature and the dropping velocity, respectively [10].

Two different approaches are used to translate ink on the substrate surface: thermal and piezoelectric approach as depicted in **Figure 5**. The print cartridges consist of a series of tiny chambers and each chamber is attached with a heater over the nozzle. In case of thermal printing approach, a pulse of current is applied to the heater which leads to a rapid vaporization of the ink and build up pressure which push the droplet of ink out of nozzle [11]. The internal temperature creates a bubble in the cartridge and facilitates the single droplet formation of ink out of the nozzle via volume expansion. The negative pressure inside the cartridge after the droplet is ejected, draws new ink inside the reservoir [10]. In piezoelectric approach, the ink is ejected out from a nozzle through a sudden quasi-adiabatic reduction of the chamber volume via piezoelectric action. In the initial state, the ink in the printer head is in equilibrium state. Upon applying a voltage pulse signal to the piezoelectric element, the ink is ejected out due to the volume expansion inside the printer head. As the kinetic energy overcome the threshold, an in-flight droplet is generated and flights towards the target surface. In the sequentially applied opposite voltage, the ink in the printer head refills, and the whole process is repeated.

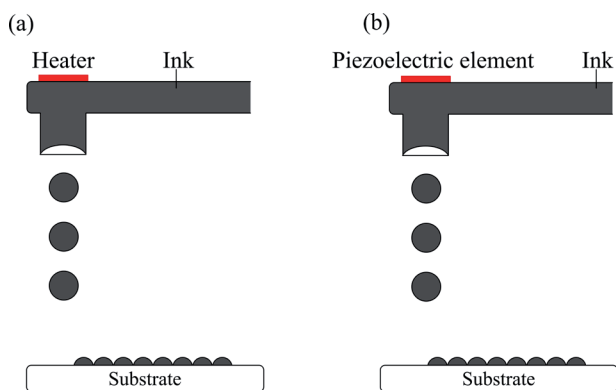


Figure 5. Schematic of inkjet printing (a) thermal printing. (b) Piezoelectric printing.

Several thousand droplets are delivered to substrate in every second and pinter head also moves at the same time. Thermal printing is simple in design and low cost approach, however, it is confined to vaporizable inks to form bubble. The elevated operating temperature makes it inappropriate for polymer-based printing. Therefore, piezoelectric printing is widely used than thermal printing [12].

Ink viscosity and surface tension are crucial parameters during inkjet printing process. The low value of viscosity is required to allow the ink to fill the chamber as well as nozzle and the high surface tension enough to hold the ink in the nozzle without dripping [13]. To create droplets via thermal nozzles, the inks must be heat-compatible and sensitive to the volume contraction/expansion depending on the temperature. The piezoelectric nozzles contain a piezoelectric film along the wall of a reservoir. The deformation of the film generates the mechanical volume expansion in response to applied voltage pulses and as result the ink is ejected in response to the pressure generated by the piezoelectric element [10]. Typical inks used with a piezoelectric printing require a viscosity of 0.5–40 cp and a surface tension of 20–70 dyne/cm as the piezoelectric transducer only generates limited power, and printing high viscosity ink is difficult [14]. The piezoelectric nozzles have relatively better resolution and require lower temperature, which enables more precise operation to deposit thin film and do not suffer from ink degradation concerns and temperature-sensitive solvent choice. However thermal nozzles are typically less expensive and widely used in commercial printers [10].

Stable drop formation without satellite droplets after ejection from the nozzles is also important to obtain well-defined printed patterns on a substrate. The droplet velocity and volume are strongly depend on the pulse width and amplitude. The size of the droplets increases linearly with the size of the nozzle. However, the fine droplets generates high resolution and high surface morphology of final printed film. Usually nozzle size, droplets shape and fly direction are determined by the manufacture company and these parameters depend on the composition of the ink, especially on the solvent. Surface tension and viscosity are the primary physical properties that determine the shape and droplet-tail of in-flight droplets, and satellite droplet formation [10].

6. Summary

Spin coating, dip coating and spray coating are very common techniques and widely used to deposit thin films in research laboratories as well as industries. The

spin coating is one of the important route for lab scale due high reproducibility and its suitability over a wide viscosity range. It is a quick and easy approach to grow uniform film at small scale from a few nanometers to a few micrometers in thickness. However, it is not suitable for scale up in industry due to its high material consumption and the restriction to large area. Dip coating is a commonly used method for mirror coating and dye processing as it can provide easy and fast deposition thin films over a large area. The key advantages is the large area processing and the thickness of film can be controlled by withdraw speed and viscosity of solution. Dip coating requires a high volume of coating solution and large tanks. Despite of long deposition life (several months) only about 20% of the solution can be used. The spray coating technique is able to access a broad spectrum of fluids, and offers the opportunity to tune the system to deposit any kinds of solution and obtain the desired film thickness. It is reproducible, and have great potential for large scale production. Inkjet printing is a powerful and cost-effective technique for deposition of liquid inks with high accuracy. The special characteristics offered by inkjet printing includes additive patterning, reducing materials consumption, non-contact deposition, low cost and capability of large area. Moreover, inkjet printing is capable of deposition a given material on a substrate that has pre-existing patterns, where contamination or damage of patterns would be induced with other deposition processes. However, to deposit materials on substrates, the solution or ink must be compatible with the print head and the viscosity should within a specific range.

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