Advanced Tablet Coating Technique: Dip Coating Technique

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Abstract

The dip coating technique is a widely used method in thin film deposition, particularly in industries like electronics, optics, and materials science. This process involves immersing a substrate material into a liquid solution or suspension (the coating material) and then slowly withdrawing it at a controlled speed. As the substrate is pulled out, a thin film of the coating material adheres to its surface due to capillary action, forming a uniform layer upon subsequent drying or curing. Factors like viscosity, surface tension, and concentration of the coating solution, as well as withdrawal rate and drying techniques, are carefully controlled to achieve the desired film characteristics. This technique offers advantages such as simplicity, cost- effectiveness, and scalability for large-scale production. However, challenges related to controlling film thickness, achieving uniformity, and handling high-viscosity coatings exist and require precise process optimization(1). In this process, the cores are held in a suitable device (e.g. baskets) and dipped into the coating solution and then dried carefully to avoid sticking to each other. For better or heavier coats, the dipping and drying process may need to be repeated multiple times one after the other. There are several dipping arrangements available, some of which include small suction tubes that hold the tablets apart until the drying process is complete, after which you can add more tablets or start the recoating cycle(2). The coating techniques used in tablet production greatly influence the aesthetic qualities such as colour, texture, mouthfeel, and taste masking. However, these techniques have certain limitations and drawbacks. To overcome these limitations, Tablet in Tablet is considered one of the best alternatives. The objective of this study is to comprehensively review the formulation, characterization, and challenges associated with the development of Tablet in Tablet dosage form. Currently, there are only a few patents filed or granted on this topic, including the Tablet in Tablet of cyclophosphamide and capecitabine. Our focus is to provide the rationale behind the development of such dosage forms (3).

Keywords: dip coating, techniques, mechanism, polymers, surface modification, thermal spray, characterization, functional features.

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Introduction

Dip coating is a commonly utilized method in the field of surface coating and material fabrication. It entails submerging a substrate into a liquid solution or dispersion, such as a polymer solution or a suspension of nanoparticles, and then gradually removing it at a regulated pace. This procedure results in the formation of a thin film or coating on the surface of the substrate, as the liquid adheres and forms a uniform layer during withdrawal. Due to its simplicity, cost-effectiveness, and ability to produce precise coatings of controlled thickness, this technique is employed in various industries, including electronics, optics, and biomedicine. The underlying principle of this technique revolves around the interaction between surface tension, viscosity, and solvent evaporation. Factors such as the speed of withdrawal, concentration of the solution, properties of the substrate, and drying conditions significantly influence the quality and thickness of the coating(4). Tablet coating is a process used in pharmaceutical manufacturing to apply a layer of coating material onto a tablet core. This technique offers various advantages such as improved appearance, taste masking, protection of the active ingredients from degradation, and controlled release of the drug.

The process involves several steps:

Preparation of Coating Solution: The coating material, typically a polymer or a blend of polymers, plasticizers, pigments, and other additives, is dissolved or dispersed in a solvent or a mixture of solvents to form a coating solution.

Coating Application: Tablet coating can be performed using different methods

Pan Coating: Tablets are placed in a rotating pan, and the coating solution is sprayed onto them using spraying nozzles while the pan rotates. The tablets are constantly tumbled to ensure uniform coating.

Fluidized Bed Coating: Tablets are suspended and moved within a chamber using a stream of air, while the coating solution is sprayed onto them. This method allows for a more controlledand even coating.

Drying: After the coating is applied, the tablets undergo a drying phase to remove the solvent from the coating solution. This can be achieved through hot air drying or other methods to ensure a solid and uniform coating.

Curing and Polishing: Some coatings may require further curing or polishing steps to enhance their properties, such as durability or appearance (5).

Dip coating is considered the oldest commercially used wet chemical thin film deposition method. The first patent for this process was granted to Jenaer Glaswerk Schott & Gen. in 1939 for sol-gel derived silica films. Currently, sol-gel or more generally CSD derived coatings are being extensively researched for a wide range of applications including ferroelectrics, dielectrics, sensors and actuators, membranes, superconducting layers, protective coatings, passivation layers, etc. The process can be divided into three crucial technical stages:

1. Immersion & dwell time: The substrate is submerged into the precursor solution at a constant speed, followed by a specific dwell time to ensure sufficient interaction between the substrate and the coating solution for complete wetting.

2. Deposition & Drainage: By pulling the substrate upwards at a constant speed, a thin layer of precursor solution is carried along, resulting in film deposition. Any excess liquid will drain from the surface.

3. Evaporation: The solvent evaporates from the fluid, leading to the formation of the as- deposited thin film. This process can be expedited by applying heat during drying. Subsequently, the coating may undergo further heat treatment to eliminate residual organics and induce crystallization of the functional oxides. Although the dip coating process can be used with various types of precursor solutions such as sol-gel, MOD, and hybrid, the use of sol-gel solutions provides the greatest potential for modifying film properties by altering the size and structure of the inorganic species in the sol, along with the solvent(s). While sol-gel chemistry has been briefly mentioned in other chapters of this book, this section provides a comprehensive review of the corresponding aspects. Following this, the classical dip coating process is examined in relation to sol-gel derived coatings. This includes the deposition of inorganic sols, considering factors such as time scales, sol structure, and capillary pressure, and their impact on properties like refractive index, surface area, and pore size of the deposited film. Lastly, advanced dip coating techniques such as angular dependent dip coating and the

evaporation-induced self-assembly (EISA-process) are introduced, which allow for the rapid production of patterned porous or nanocomposite thin film materials (6).

A comprehensive examination of recent advancements in the processing and growth of metal-oxide thin films for electronic and photonic devices is presented in this review. The focus is specifically on precise solution-based technological coatings for electronics and optics, as well as new concepts for oxide material growth in various systems such as electrochemical, catalytic, energy storage and conversion, information technology, semiconductor device processing, and related devices. The review compiles and compares the nature of soluble precursor solutions and their relationship to the film formation process using different solution coating techniques. It emphasizes the advantages of precursor design in creating complex oxides for devices. The versatility of solution-processable oxides and functional material coatings makes it crucial to capture the progress made in oxide deposition for plastic electronics, see-through and wearable devices, and high-fidelity thin film transistors on curved or flexible displays. Solution processing, even for oxides, provides control over composition, thickness, optical constants, porosity, doping, tunable optical absorbance/transmission, band structure engineering, 3D-substrate coating, complex composite oxide formation, and multi- layered oxide systems. These achievements are more challenging to attain using chemical vapour deposition (CVD) or atomic layer deposition (ALD) processes. The limitations of solution processing for certain technologies are also discussed, along with insights into the future of solution-based processing of metal-oxide materials for electronics, photonics, and other technologies (7). There is a wide range of coating methods available due to the diverse applications and needs in various fields. These methods involve numerous on-line/off-line parameters, resulting in different outcomes in terms of material microstructure, effectiveness, suitability, and durability. However, coating methods are particularly useful in specific applications, especially for corrosion and wear protection. Corrosion can lead to a decrease in the mechanical properties of materials, and the release of corrosion products can have detrimental effects in different applications. It is important to investigate the deposition mechanisms of coating materials to understand their advantages and disadvantages for specific applications. Among the many available processes, physical vapour deposition (PVD), chemical vapour deposition (CVD), micro-arc oxidation (MAO), sol-gel, thermal spray, and polymer coatings are considered the most effective and applicable. Each method offers different deposition techniques, materials, second phases, thicknesses, and densities, which need to be carefully considered for factors such as mechanical stability, corrosion properties, biocompatibility (in biomedical applications), and enhancement of material behaviour. Despite the benefits provided by coating processes, they also have disadvantages that can affect their reliability. Negative thermal effects, such as distortion, cracks, and delamination, as well as the destructive effects of loose atmospheric protection (such as the penetration of inclusions and contaminations into the substrate) and the properties of coating materials (such as melting point, availability in different forms, and biocompatibility) are the most crucial drawbacks (8). Thin liquid films are frequently applied onto flat or cylindrical surfaces through a dip-coating process. This method is extensively utilized in industrial settings due to its simplicity and ability to handle large quantities. A smooth and wettable substrate, whether flat or curved, is steadily pulled out from a liquid reservoir at a constant speed. By adjusting the composition of the liquid, layered coatings can be precisely deposited using this approach. However, the main challenge lies in achieving a flawless coating without any imperfections such as blisters, holes, cracks, or uneven material buildup, particularly when operating at higher withdrawal speeds; The wetting properties of surfaces can be customized with submicron precision using photolithography or micro contact printing. This is achieved by forming self-assembled monolayers of molecules like alkylsilanes or alkylthiols, which can transform a hydrophilic surface into a hydrophobic one. For instance, the contact angle of water can significantly increase from 0° to approximately 110°. By patterning this hydrophobic monolayer, it becomes feasible to accurately distribute ultrasmall volumes of liquid in the nanoliter to femtoliter range. This can be accomplished by dip coating the substrate into a liquid bath or slot. Unlike conventional dispensing techniques such as ink jetting or microsyringe delivery, achieving such precise volume distributions is challenging or even unattainable. This article delves into the process of dip coating chemically micropatterned surfaces. The thickness of the liquid coating that adheres to the hydrophilic portions is heavily influenced by factors such as viscosity, surface tension, density, pattern size, geometry, and orientation. As our focus is on "printing" liquid micropatterns onto a secondary target surface, it is crucial to achieve pattern fidelity between the designed chemical pattern and the formed liquid microstructures. Additionally, we aim for a uniform coating thickness across structures of varying sizes and shapes. Alongside our experimental investigations, we have developed a hydrodynamic model to determine the maximum film height deposited on vertically oriented hydrophilic strips on a hydrophobic plane. This model differs in two key ways from the traditional dip coating analysis on homogeneous surfaces and has shown remarkable agreement with experimental results (9).

Dip Coating

Dip coating is a widely utilized technique in various industries for manufacturing purposes. It has proven to be a valuable method for creating thin films through the use of a specialized dip coater in research and development. When optimized, dip coating allows for the production of remarkably uniform films, with the ability to easily control factors such as film thickness. A notable advantage of dip coating, when compared to other processing techniques, is its straightforward design. It is a cost-effective solution that requires minimal setup and maintenance. Additionally, it has the capability to generate films with an exceptional level of uniformity and nanometer-scale roughness.

This guide serves as a practical primer on dip coating, providing an overview of key aspects such as the formation of the wet film, the evolution of thickness, the dynamics of drying, and the influence of processing conditions on the various stages of dip coating and the resulting final film thickness. Additionally, it offers explanations for the most frequently encountered problems that may arise during the process.

Dip Coating Theory

Withdrawal and Film Formation

It is easy to understand the withdrawal step of the dip coating process as the result of multiple sets of forces interacting. These forces can be classified as either entraining forces or draining forces. The liquid is drawn back towards the bath by draining forces as they strive to remove it from the substrate. Entraining forces, on the other hand, are those that help keep fluid on the substrate. The thickness of the wet film coated onto the substrate is determined by the balance between these sets of forces. Four zones can be distinguished in the wet film production during the withdrawal stage (shown in the image below).

These four areas are:

1. The static meniscus, in which the hydrostatic equilibrium determines the meniscus'sshape

2. The area surrounding the stagnation point is known as the dynamic meniscus. The equilibrium between the entraining and draining forces is known as the stagnation point.

3. Where the wet film has attained a specific thickness (h0), known as the constant thicknesszone

4. The area where the wet film starts, known as the wetting zone.

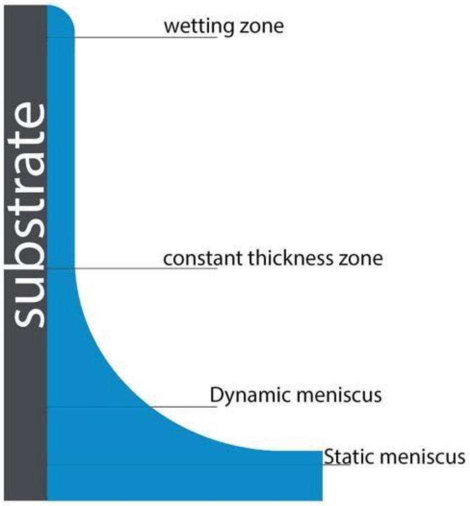


Fig.no.1 The dip coating formation involves four distinct region. These are the static meniscus, the dynamic meniscus, the constant thickness zone, and the wetting zone.

The wet film thickness in this area is determined by the flow of solution and the dynamic meniscus. It is crucial to comprehend the physics underlying the dynamic meniscus' curvature and the stagnation point's thickness.

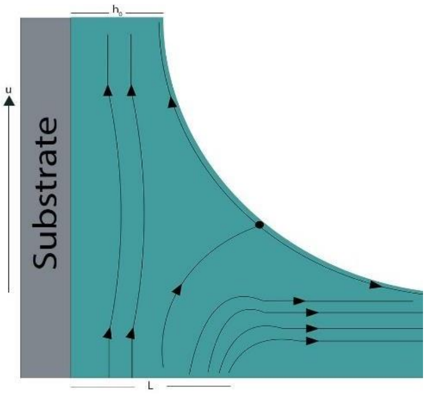


Fig.no.2 The flow of solution is determined by the balance of entraining and draining forces. At the dynamic meniscus, the entraining forces being to impact solution

flow until they are the dominant force.

The dynamic meniscus region is depicted in the figure below. It is in the boundary layer (L) where the static and dynamic meniscus meet. The viscous flow forces affect the solution's motion in this area. The draining forces are much larger than the viscous forces beyond the boundary layer. The meniscus in this area is determined by the equilibrium between hydrostatic and capillary pressure. When the entraining and draining forces are in equal balance, a stagnation point is reached. The film thickness is determined by the balance of these forces. The viscous flow, draining, and capillary regimes are the three coating regimes that are distinguished by the forces that control the behaviour of the coating.

Viscous Flow and Drainage Regime

The viscous flow regime is the first coating regime. High speeds and viscous solutions cause this to happen. In this case, gravitational attraction and viscous forces dominate the coating. In this case, the thickness of the liquid layer can be given by Equation 1.

$$h_0 = c(\frac{\eta U_0}{\rho g})^{\frac{1}{2}}$$

Equation 1. The equation for calculating the wet film thickness for dip coating while in the viscous flow regime.

The viscous forces on the solution as the substrate is removed in this case make up the entraining force. This is determined by the substrate's rate of withdrawal from the solution (U0) and viscosity (η). The gravitational constant (g) and the solution's density (ρ) determine the draining force, which is gravity. The dynamic meniscus's curvature and the constant (c) are connected. This constant is a feature of the solution itself and has a close relationship with its rheological characteristics. Generally speaking, this constant is 0.8 for Newtonian liquids.

This approximation is not valid in most cases because the withdrawal speeds or viscosity of the solutions used are too low. The viscous force decreases with a reduction in these two factors. The solution's surface-tension-driven movement then also affects the equilibrium between the entraining and draining forces. The coating is considered to be within the drainage regime in these circumstances. When surface tension is taken into account, the relationship between the wet film thickness and the substrate withdrawal speed is represented by the Landau-Levich equation (see Equation 2).

$$h_0 = c\frac{(\eta U_0)^{\frac{2}{3}}}{\gamma_{LV}^{\frac{1}{6}}(\rho g)^{\frac{1}{2}}}$$

Equation 2. The Landau-Levich equation is a modified form of the viscous flow equation, which takes account of surface-tension driven flow.

The Landau-Levich formula holds true up until extremely low withdrawal speeds are taken into account. A third regime of coating takes place when the speed is lowered to less than about 0.1mm.s-1. The capillary regime is the name given to this regime. The rate of solution entrained onto the substrate (via viscous flow) is less than the rate of evaporation in the capillary regime. Thus, an understanding of the capillary regime requires an understanding of the drying dynamics.

Drying Dynamics

Three stages are usually involved in the drying process of dip coating:

1 The drying front during coating
2 The time of constant rate
3 The period of declining rates

The most basic drying phases are the ongoing rate period takes place both during and after coating in the constant thickness zone. Here, the solvent evaporation occurs uniformly throughout the wet film at its surfaces. The falling rate period and the constant rate period are the most basic drying stages. The ongoing this only doesn't apply where the drying front is located, which is at the substrate's edges.

Until a gel-like film forms, the majority of the solvent will gradually be extracted from the wet film. The period of declining rates starts at this point. The little solvent that remains is trapped in the gel during the falling rate period, and the solvent's diffusion towards the surface controlsthe rate of evaporation.

The figure below illustrates the more intricate drying stage that takes place at the drying front. The interface where the wet film and substrate meet is where the drying front is most noticeable; this is the wetting zone. Here, evaporation happens much more quickly due to the larger surface-area-to-volume ratio, which causes a wet film with a higher concentration to form. Surface-tension driven effects cause the solution to be drawn from the surrounding areas as a result. The solution will be subjected to a capillary force once the drying front solution has formed a dry film. The result is a thickening of the deposited film as the solution wicks into the dry film.

During the capillary regime of coating, solution is drawn into the drying film due to the capillary force. The rate at which the drying front retreats is much slower than the formation of the constant thickness zone for sufficiently fast withdrawal speeds. As a result, the constant rate period controls the drying dynamics, and the initial wet film thickness will determine the final film thickness. The drying front dominates the drying dynamics at slow withdrawal speeds, i.e., when the drying front recedes more quickly than the rate of withdrawal.

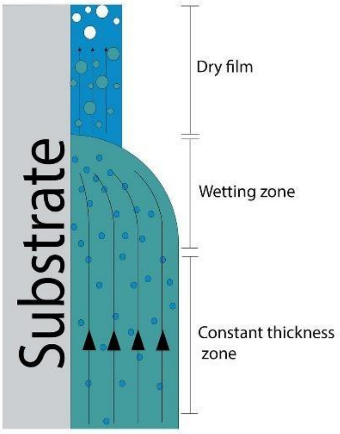


Fig.no.3 The dynamics of drying in dip coating are controlled by the formation ofconcentration gradient and capillary action of the dry film.

Capillary Regime

It does not take the wet film thickness into account in the capillary regime. This is due to the fact that at these coating speeds, a constant thickness zone is never really reached. The final thickness in the capillary regime is thus influenced by three factors: (i) the rate of withdrawal;

(ii) the characteristics of the solution; and (iii) the solvent's rate of evaporation. The dry film thickness is therefore given by Equation 3.

$$h_f = k\frac{E}{LU_0}, k = \frac{cM}{\alpha\rho}$$

Equation 3. The Capillary flow equation is dependent upon both the speed and the evaporation rate of the solvent. A constant (k) called the 'materials proportion constant' is used for the dry film properties.

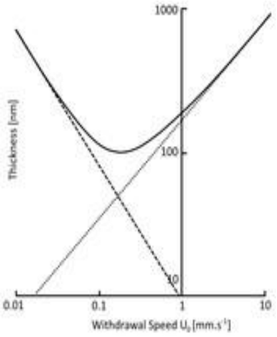
The final dry film thickness (hf) is determined in this case by the evaporation rate (E), the materials proportion constant (K), the width of the coated film (L), and the withdrawal rate (U0). K is the sum of the properties of the solute, solution, and dry film. The solute's density (ρ), molar weight (M), porosity (α) of the deposited film, and total concentration (c) in solutionall contribute to the constant.

The solute concentration, solute density, and material molecular weight are examples of properties that directly and simply affect the thickness of the dry film. The porosity value, on the other hand, is much more intricate. The porosity affects the drying dynamics in addition to changing the film's density relative to the raw material. As previously stated, capillary action will draw the wet film into the dry film at the drying front, or the point of contact between the two films. This is also significantly influenced by the film's porosity, which determines the rate at which the solution is drawn into the dry film as well as how far and how quickly it does so.

Film Thickness vs Withdrawal Speed

It is necessary to apply both the capillary regime equation and the Landau-Levich equation to determine the dry film thickness as a function of withdrawal speed. An example withdrawal speed vs. film thickness graph for the dip coating process is displayed in the figure below. When these two coating regimes transition, a minimum thickness for dip coating is reached. The various coating regimes take over as the withdrawal speed moves away from the minimum in either direction coating speeds. This happens when coating thickness is at its lowest possiblepoint.

Either the capillary regime equation alone or the Landau-Levich equation can provide the thickness curve for both high and low speeds. Nevertheless, neither formula by itself can provide precise coating thickness values over a range of coating speeds. This happens when coating thickness is at its lowest possible point. An equation that unifies the capillary regime equation and the Landau-Levich equation is required to compute the minimum thickness. We'lltalk about this in the section below.



Both the capillary regime equation and the Landau-Levich equation can be used to determine the film thickness. At the crossover between the two regions, both equation must be taken intoaccount.

Minimum Film Thickness Equations

The equations controlling the thickness during the capillary regime and the drainage regime must be combined to find the minimum thickness. In order to relate the wet film thickness to the dry film thickness, we must first modify Equation 2. The'materials proportion' constant can be added to the equation to accomplish this.

Equation 4 is obtained by adding the two equations. In this case, the drainage regime (Equation 2) is related to the second term in the bracket, which is related to the capillary regime (Equation 3). A universal solution constant (D) has been created by rolling up the constants found in Equation 2.

$$h_f = k\left(\frac{E}{LU_0} + DU_0^{\frac{2}{3}}\right)$$

Equation 4. The equation for film thickness gives information on the final dry film thickness and includes the contribution from the capillary and drainage regimes.

Differentiating the thickness with respect to withdrawal rate allows one to get the smallest film thickness from this equation. Equation 5 can be obtained by setting this derivative to (the gradient at the minimum's inflection point)

$$U_{min} = \left(\frac{2DL}{3E}\right)^{-\frac{3}{5}}$$

Equation 5. The minimum thickness for dip coating can be found by taking the differential of the thickness equation and determining where the slope of the graph becomes zero.

Even though the film thickness attained is roughly estimated by this equation, a number of important factors have not been taken into account. These include surface air flow, fluctuating evaporation rates, gradients of viscosity and concentration, thermal gradients, Marangoni flow, and other variables that are subject to change.

Changing Withdrawal Speed

There are two basic forces that define the meniscus:Viscosity-based drag due to gravity

Surface tensions separating the solution and substrate

The solution (hereafter referred to as ink) in the meniscus will either pull up with the substrate to form a film or fall back into the reservoir during substrate withdrawal, as illustrated in the figure below. At the drying line, the meniscus ends. At this stage, the solvent has completely evaporated or drained, leaving behind a solid film. The speed of the drying line and the withdrawal speed are equal. The meniscus essentially "moves down" the substrate as it withdraws, remaining close to the substrate-reservoir boundary. The capillary region (low speed) and the draining region (high speed) are the two primary regions of dip coating that aredefined by withdrawal speed.

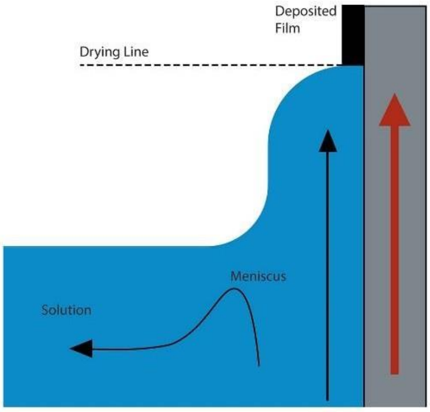


Fig.no.4 As the substrate is withdrawn, the liquid either falls back into the reservoirdue to gravitational forces, or is drawn up with the substrate due to surface forces.

Withdrawal rates in the draining region exceed 1 mm/s. The solvent isn't evaporating as quickly as the drying line is. Here, the Landau-Levich model states that the ink properties and the withdrawal speed control the film thickness. Here, evaporation rate is not a major consideration.

Withdrawal rates in the capillary region are typically less than 0.1 mm/s. The fact that the evaporation rate is greater than the drying line's "movement" is crucial in this situation. Here, the solvent evaporates as soon as the ink is fed into the meniscus' upper portion. After that, more ink is drawn up the substrate by surface forces, and the solvent evaporates once more. Capillary feeding is this. Consequently, in this area, the film is thicker the slower thewithdrawal speed.

Between these lies a region that a combination of the above models can accurately model. As seen in the figure below, this region creates the thinnest film and defines a V-shaped dependence of thickness on withdrawal speed. When working in the capillary region, it is best to work with higher withdrawal speeds as it can lead to some of the issues mentioned in this guide. To obtain a uniform coating, low withdrawal speeds might be required if the coating inkis highly diluted (10).

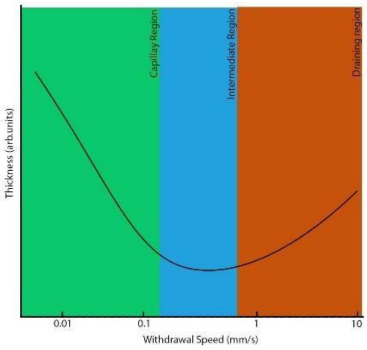


Fig.no.5 Dependence of film thickness changes on withdrawal speed.

. Reliable Coating Methods

Coating processes are used in a variety of industries, from the aerospace and automotive sectors to the tiny biomedical devices and implants inside the human body, to protect a particular part or area of a structure exposed to harsh and corrosive environments.

1. Physical Vapour Deposition (PVD) Coating.

The PVD process is well-known for providing thin protective films and corrosion and wear resistance to materials exposed to corrosive media. Its applications span from industrial parts to decorative objects. This method has the advantage of allowing the coating layers' mechanical, corrosion-resistant, and aesthetic properties to be changed as needed. PVD is a high vacuum process that generally involves the transfer of solid or liquid materials to a vapour phase, followed by the condensation of metal vapour, which forms a dense and solid film. The two most well-known forms of PVD are evaporation and sputtering. Multilayered coatings are always required because PVD produces thin coating layers, and material selection needs to be done with great care. In addition to their aesthetic uses, a lot of PVD-coated parts are used in high-wear applications where they abrade the surface and remove the coating layer. This phenomenon causes the parts' resistance to corrosion to decrease, increasing their susceptibility to corrosive media. Figure 1 represents a schematic view of different types of electron beam PVD machines. With this technique, a physical evaporation process dominates the growth of the coating. A variety of supply units, including electron beams, heating wires, laser beams, molecular beams, etc., can provide the thermal energy required for evaporation. The source material's atoms, which may be solid or liquid, are heated to the point of evaporation bythis thermal energy.

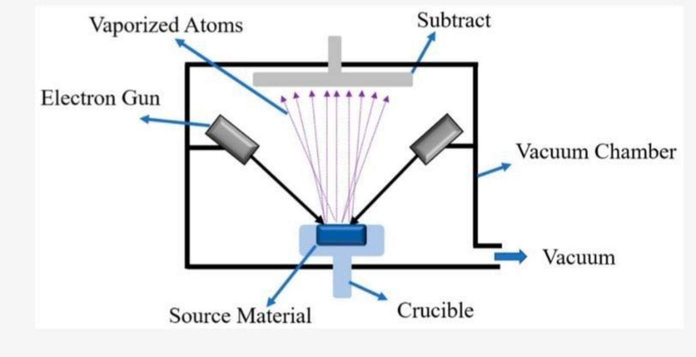


Fig.no.6 Schematic view of a physical vapour deposition (PVD) machine using electron beam as the heat source.

2. Chemical Vapour Deposition (CVD) Coating

CVD is another name for vapour deposition. The semiconductor industry uses this process extensively because it produces a strong, high-quality, and highly resistant coating layer on any substrate while subjecting it to a high vacuum. Mechanical parts that are in constant contact and require protection from wear and corrosion can be treated with CVD. In this procedure, a set of volatile material precursors would be introduced to the substrate, also referred to as a wafer, and a chemical reaction would result in the creation of a deposition layer on the material's surface. Nonetheless, certain byproducts of these chemical reactions may persist in the chamber despite being eliminated by the vacuum pump's continuous airflow.. A schematic of the CVD setup is shown in Figure 2. The heaters maintain a temperature high enough to promote the chemical reaction between the vaporized materials and substrate, and the vaporized CVD materials are pumped from the right side.

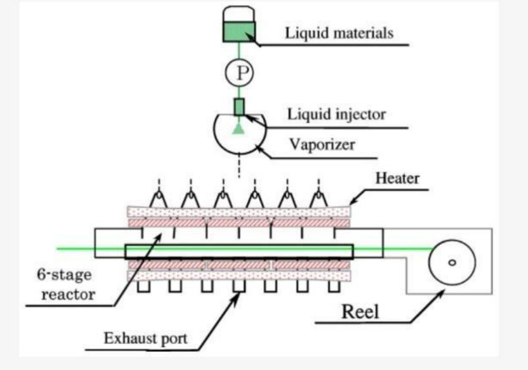


Fig.no.7 Schematic chemical vapour deposition (CVD) setup, mechanical parts, and operation mechanism

3. Micro-Arc Oxidation (MAO) Coating

In terms of the makeup of coating layers, the MAO process is regarded as a flexible coating method. The schematic of the process is illustrated in. In general, MAO creates micro- arcs as plasma channels by using a large voltage differential between the anode and cathode. Depending on the intensity of the micro-arcs, some of the substrate is melted when these arcs strike it. Concurrently, the pressure inside the plasma channels decreases, facilitating the coating materials' deposition on the substrate surface in the working electrolyte. Oxides are produced by a chemical reaction of oxidation brought on by the oxygen present in the electrolyte and are deposited on the substrate materials' surface. The ability to mix desired elements and compounds as a solute in the working electrolyte gives this process its versatility. Al, Mg, Ti, and their alloys are currently the materials that are coated with MAO the most frequently. The most crucial property of a layer treated with MAO is its high resistance to corrosion. Furthermore, this coating layer offers high bone ingrowth when formed on biomedical implants and fixations because of its porous structure. Advantages of MAO include a coating surface with excellent adhesion and hardness as well as varying porosity scales throughout the structure. It is the coating itself that has this kind of multistructural nature.

4. Electrodeposition Coating

Materials that use the deposition of metallic ions on a substrate are thought to be protected by electrodeposition. An ion transfer occurs in the unit cell as a result of the potential difference between the anode and cathode poles in this process. The submerged sample eventually develops a coating layer as a result of ions from the other electrode. Popular electrodeposition materials have been the subject of extensive research. Among the common metals that have been the subject of extensive research are Ni-P, Ni-P/Sn, Ni-P-W, Ag/Pd, Cu/Ag, Cu/Ni, Co/Ag, and Co/Pt. These studies show that the electrodeposited coatings greatly improve the substrate's ability to withstand corrosion. Furthermore, it has been demonstrated that this method holds promise for creating superhydrophobic polymeric coatings like polythiophene. Generally speaking, there are two types of electrodeposition processes: electrolytic deposition (ELD) and electrophoretic deposition (EPD), which are covered in more detail in the sections that follow.

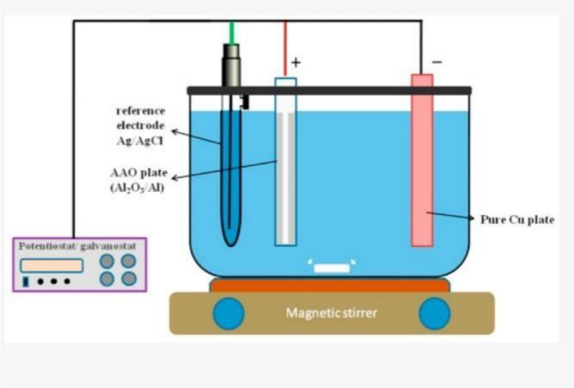


Fig.no.8 Schematic setup for electrode position of copper metal particles overaluminium oxide.

4.1. Electrolytic Deposition (ELD) Coating

Electrochemical layer deposition (ELD) is a technique used to create a uniformly thick, dense metallic coating on conductive substrates. Inside an electrochemical unit cell, substrate and deposition materials are chosen to serve as the cathode and anode, respectively. **Figure 5** illustrates a general overview of the process. Metallic ions migrate toward the working electrolyte and then toward the substrate when a potential difference between the anode and cathode poles is applied. The electrolyte must become super-saturated during the deposition phase, which is brought on by the circuit's charging current. With this method, the electrolyte's metallic ion concentration doesn't change while coating is happening. While the majority of applications for this technique are decorative and low-wear/corrosion, there have been reports of the development of other applications, including solid-oxide fuel cells, electronics, biomedical, optics, and high temperatures. In a process more akin to the MAO process, ceramic materials can be deposited on metallic substrates by further increasing the potential difference in electrolytic unit cells. When Ni-Co-Al2O3 was applied to steel pipes, Tian et al. observed a significant increase in the substrate's ability to corrode when exposed to oil sand slurry. When Ni-Co-SiC was applied to carbon steel pipes exposed to oil sand slurry, Yang et al. observed a significant increase in corrosion resistance as well as erosion resistance. Fayomi et al. reported the same outcomes on a mild steel substrate coated with Zn, Ni, and Al2O3. Furthermore, using a dihydrogen phosphate solution, Redondo et al. coated a copper substrate with a polypyrrole (PPy) coating that is resistant to corrosion.

4.2. Electrophoretic Deposition (EPD) Coating

Another type of electrodeposition called electrophoretic deposition (EPD) yields colloidal coating layers that are thicker. Colloidal particles coagulate to form thin films on substrates in a unit cell with an electric field akin to that of electroluminescence (ELD). EPD is a multi-stepprocess, in which:

1. Known as electrophoresis, an external electric field drives suspended particles in an electrolyte toward a single electrode.
2. The moving particles coagulate into a larger particle by congregating in oneelectrode.
3. The bigger particles settle on the electrode's surface, a substrate that needs to becoated.

Ultimately, a powder-shaped substrate will develop a thick coating layer on it. **Figure 6** represents a schematic of the working mechanism of the EPD process. It is advised to use densification techniques (such as sintering, furnace curing, light curing, etc.) to improve the protective layer's quality. EPD has so far been used in a wide range of applications, such as coating, graded material deposition, porous structure deposition, selective deposition, and biomedical applications. Commonly used materials in EPD include metals, oxides, phosphates, carbides, and borides. Castro et al. reported creating corrosion-resistant coatings on stainless steel AISI 304 using sol-gel and EPD, and they found that each of these processes increased corrosion resistance by two and four times, respectively. Gebhart et al. conducted a study wherein they coated an AISI 316 L stainless steel with chitosan for use in biomedical applications. They noted that this coating had a positive impact on the substrate's corrosion behavior. Additionally, they claimed that the main element regulating coating characteristics like hydrophobicity, thickness, and structure is the applied electric field in EPD. Chen et al. coated TC4 Ti-alloy orthopedic implants with graphene. According to their report, artificial joint implants coated with graphene exhibit a significant increase in longevity. They discovered that microcracks in coating surfaces were the cause of any corrosion that happened on substrates. Fei et al. successfully deposited SiC particles on paper-based friction materials and achieved an excellent wear enhancement of this material while studying the wear resistance of EPD coatings.

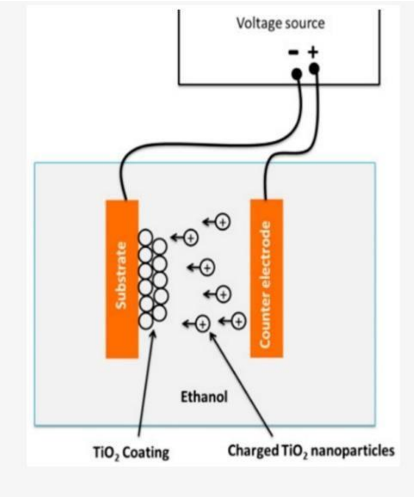


Fig.no.9 Sketch of the electrophoretic deposition process.

5. Sol–gel Coating

Coating process one of the best coating techniques for biomedical devices is sol-gel coating. Numerous studies on this process and its uses have made experiment setup and execution easier while maintaining the validity of the results. Yet, from the perspectives of ion release and corrosion, sol-gel can improve coating layers that were already in place. Sol-gel's liquid-permeating properties make it simple to seal damaged layers or porous coating structures. The solution known as Sol is made from calcium phosphorous (CaP) precursors dissolved in ethanol or distilled water. The prepared mixture is heated to various temperatures to facilitate the aqueous portion of the solution and raise the viscosity to the required level, which creates a gel phase out of the solution. The name "sol-gel" originates from this phase, which turns the mixture from a liquid solution into a gel phase. Following preparation, the components or devices are slowly submerged in the sol-gel medium. To get a thicker or multilayered coating of the same material, repeat this process. Furthermore, the coated samples can be baked to expedite the drying process or to create purposeful dehydrating cracks on the coating layer's surface in preparation for subsequent processing stages. Figure 7 represents a schematic of an example of a sol–gel (11).

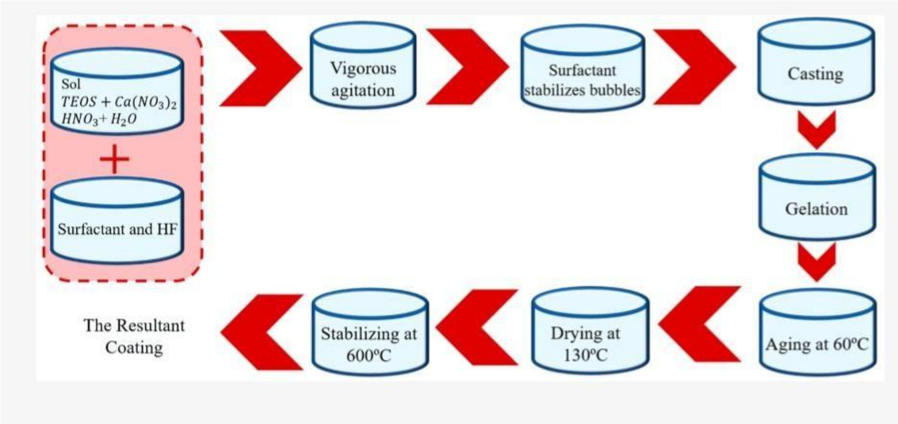


Fig.no.10 Schematic Sol-Gel coating process from solution preparation to thefinal solid structure formation.

Experimental work

1. *Reagents*

Fluka supplied the di-amino functionalized polyether (Jeffamine® D-400) and 3-isocyanate propyltriethoxysilane (ICPTES, 95%, Aldrich, Munich, Germany). Both were used as supplied, with light storage kept out of the way. Citric acid monohydrate (Merck, Munich, Germany) and absolute ethanol (EtOH, absolute 98%, Riedel-de-Haën, Seelze, Germany) were also utilized as supplied. Utilized was ultra-pure water (0.055–0.060 µS/cm) from an Elga-Enkrott, Sintra, Portugal Purelab Ultra System.

2. Sol-Gel Synthesis Procedure of Ureasilicate Coatings

The OIH matrices, which are commonly known as U(400), were synthesized in accordance with previous research [22]. The letter "U" stands for the kind of bond (urea) formed between the precursors, Jeffamine®, and the ICPTES, while the number 400 denotes the molecular weight of Jeffamine® used in the OIH coatings.

3. Coating Deposition

OIH coatings were applied to HDGS plates (5.0 × 1.0 × 0.1 cm3) that were cut from commercially available sheets and had an average Zn thickness of 16 μm on both sides. To create HDGS coated samples, metallic substrates were dipped into the prepared mixture (sol) at a withdrawal speed of 10 mm min−1 using a dip coater (Nima, model DC Small). Either nil (Rt = 0 s) or 100 s (Rt = 100 s) was the residence time. Using one, two, or three dip steps, samples were coated with or without Rt. The same curing conditions (15 days at 40 °C between each dipping step) were always used. The same conditions as previously mentioned were also used to prepare and cure samples made using three consecutive dip steps without Rt. An incubator-compressor (ICP-400, Memmert GmbH & Co. KG, Schwabach, Germany) was usedto cure the coated HDGS samples.

4. Surface Characterization

With both coated and uncoated substrates, a glow discharge optical emission spectrometer was used to perform the chemical composition depth profiling of the coatings applied on the HDGS substrates. The samples were analyzed in an argon atmosphere using an LECO glow discharge GD OES 850A (LECO, St. Joseph, MI, USA) equipped with a radiofrequency sourceand a 700 V RMS.

Using an EDS detector (Inca-xSight Oxford Instruments) and a scanning electron microscope (SEM, JEOL JSM-6400, JEOS, Peabody, MA, USA), the morphology of the OIH sol-gel coating surface applied on HDGS specimens was examined. Sputter coating was used to apply an extremely thin layer of gold to the samples' surface. To highlight the differences between the various metallic phases, back-scattered electron (BSE) detectors were used in SEM investigations of the surfaces. The substrate was used for the SEM/EDS analyses of the HDGS coated samples both before and after the samples were submerged in SCPS for eight days (12).

Materials and Fabrication Process

During the coating process, a thin layer of materials in either the liquid or solid phase (solution) are deposited onto a substrate. Coating strategies may be used based on the requirements for coated layer thickness, coated surface roughness, rate, and coating product size (which can be derived from coated film width, coated velocity, and designing capabilities). Due to the material's potential optical properties and applications, silica titania (SiO_2: TiO_2) has been extensively studied. It is a material that, when combined with the aforementioned dip-coating technique and sol-gel method, forms the new low-cost technological platform for PICs. Given that its spectral range spans from visible to near infrared, and its R.I. can vary from 1.6 to 2.2, SiO2:TiO2 is an extremely appealing material in the field of integrated photonics. . Furthermore, the material and this fabrication method allow for the creation of optical interconnects with low transmission losses. We will first talk about the process of fabricating a single SiO2:TiO2 waveguide film. This is a fairly straightforward process, as Fig. 1 shows.

The following elements are necessary in order to complete the fabrication procedure: 1. It will completely cover the substrate, or the material on which SiO2:TiO2 will be deposited, forming a complete SiO2:TiO2 waveguide film, such as soda-lime glass or BK7 glass. Because of its lower surface roughness and lower thermal expansion coefficient, BK7 glass is recommended.
2. Sol precursor: a colloidal solution that has been previously made and contains the covering material, such as SiO2:TiO2. Tetraethyl ortosilicate (TEOS) and tetraethyl ortotitanate (TET), the precursors for SiO2 and TiO2, respectively, are added to a homogenizing factor—in this case, ethyl alcohol (C2H5OH)—to prepare the sol precursor for SiO2:TiO2. Hydrochloric acid (HCl) must be used in order to complete the hydrolysis and condensation reactions.

The primary advantages of this technique over other traditional thin-film deposition techniques like physical vapor deposition (PVD), low-pressure chemical vapor deposition (LPCVD), and plasma-enhanced chemical vapor deposition (PECVD) are that it is much simpler to carry out, substantially less expensive, and does not require foundries or sophisticated equipment or labs. The creation of waveguide structures, including interconnects, comes next after the platform is ready. These structures can be fabricated using a variety of techniques.Within our consortium, we are currently investigating all three of the following approaches (13).

Efficiency of Dip-Coating

Dip coating is a widely used technique in various industries for the deposition of thin films on substrates. The efficiency and scale-up potential of dip coating depend on several factors, including the specific application, the characteristics of the coating material, and the productionrequirements.

Uniformity of Coating

One of the critical factors influencing efficiency is the ability to achieve a uniform coating thickness. The control of parameters such as withdrawal speed, viscosity of the coating solution, and drying conditions plays a crucial role in ensuring uniformity

Reproducibility

Efficient dip coating should be reproducible, allowing for consistent results in terms of coating thickness and quality across multiple batches (14).

Scale Up potential of Dip-Coatings

Optimization of Parameters: Successful scale-up involves optimizing the process parameters for larger batches. This includes addressing challenges related to the drying time, temperature control, and substrate size.

Automation: Implementing automation in the dip coating process can enhance scale-up potential by ensuring consistent and controlled processing on a larger scale (15).

Applications and Case Studies:

Investigate specific applications where dip coating has been successfully employed. For example, in the field of photovoltaics, dip coating is used for depositing thin films on solar cellsubstrates (16).

Challenges and Limitations

Advanced Materials and Emerging Technologies:

Explore the use of novel materials and emerging technologies in dip coating. This could include advancements in nanomaterials or innovative modifications to the dip coating process (17).

Film Thickness Control:

Challenge: Achieving precise control over the thickness of the coated film can be challenging, especially when dealing with highly viscous or thin solutions (18).

Uniformity Issues:

Challenge: Ensuring uniformity across large or complex substrates may pose difficulties, and variations in coating thickness can occur (19).

Drying and Solvent Evaporation:

Challenge: Controlling the drying process is crucial for preventing defects such as cracks,pinholes, and uneven coatings. Rapid solvent evaporation can lead to non-uniform films (20).

Limited to Small-scale Production:

Limitation: The dip coating process may face limitations when it comes to scaling up for large-scale production due to challenges in maintaining uniformity and reproducibility (21).

Substrate Compatibility:

Challenge: Compatibility issues may arise with certain substrates, as not all materials aresuitable for dip coating. Adhesion problems or substrate damage can occur (22).

Limited to Liquid Solutions:

Limitation: Dip coating is primarily applicable to liquid solutions, restricting its use formaterials that are not soluble or form suitable solutions (23).

Environmental and Safety Concerns:

Challenge: The use of certain solvents and materials in dip coating may raise environmentaland safety concerns, requiring careful consideration and compliance with regulations (24).

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