

Thin-Film Dip-Coating Methods

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Coating is the way of incorporating a thin coating of material into a substrate by deposition in either the liquid phase (solution) or the solid phase (powder or nanoparticles), dip-Coating is one of them.

Keywords: dip-coating ; physical vapor deposition ; chemical vapor deposition

1. Introduction

Coating is the way of incorporating a thin coating of material into a substrate by deposition in either the liquid phase (solution) or the solid phase (powder or nanoparticles) ^[1]. The use of coating strategies may be tailored to meet production requirements for coated layer thickness, coated surface roughness, rate, and coating product size, which can be defined by coating velocity, coated film width, and patterning capabilities ^[2]. The coating operation may be carried out at air pressure or low vacuum conditions. It is categorized according to the solubility of the coating components used. In solution coating, a binder and target material are dissolved in a solution that may coat the substrate directly, and the solution then evaporates from the resultant wet film to produce a dry film. As opposed to this, vacuum deposition techniques including chemical vapor deposition (CVD) ^[3], physical vapor deposition (PVD) ^[4], atomic layer deposition (ALD) ^[5], plasma ^[6], flame hydrolysis deposition (FHD) ^{[7][8]}, and sputtering ^[9] are mostly used to produce thin, conductive ^[10], semi-conductive ^[11] and dielectric layers of demanded morphology that are utilized to transfer heat or electricity under low- or high-vacuum conditions. The guiding layer must have a higher refractive index than the substrate and cladding to effectively confine light ^{[12][13]}. Consequently, thin-film deposition and local bulk material manipulation are the two major methods that may be used to execute the guiding layer. The thin-layer deposition method includes procedures such as radio frequency (RF)-sputtering and magnetron sputtering, CVD, plasma-enhanced CVD, FHD, spray pyrolysis deposition, pulsed laser deposition, spin-coating, spray-coating, and sol-gel coating. The direct inscription of a channel waveguide (WG) in the substrate using an femtosecond (FS)-laser is an alternative to performing local modifications of the bulk material via ion exchange, ion implantation, or UV radiation.

Using the roll-coating process, a surface is coated with a tiny, micro nanoscale layer of liquid that has been recirculated across a sheet or web. The major goals of the thin layer coating are to increase the surface's effectiveness, service life, and quality. The coating is widely employed at the industrial level due to its useful benefits and applicability. The manufacturing of paper, paperboard, cellulose thin films, plastic coatings, fibrous fabric sheets, metallic foils, etc. are only a few of the processes in which it is primarily utilized. Most of the chemicals employed in the roll-coating procedure are non-Newtonian fluids that behave in either a viscoelastic or pseudoelastic manner ^{[14][15]}.

Due to its effective material utilization and direct and accurate patterning with a resolution of 20–30 nm, in contrast to spin-coating as well as other traditional processes, ink-jet printing techniques have drawn interest as a potentially cost-effective way for fabricating Perovskite solar cells (PSCs) ^{[16][17][18]}. Regrettably, because of its complexity and poor volume output, ink-jet printing cannot be easily adapted to mass production ^[19]. With no restrictions on substrate size and minimal polymer use, spray-coating techniques offer a promising future for large-scale manufacturing ^[20]. They are expected to replace spin-coating methods, the industry standard. The capacity to access a wide range of fluids with different rheology makes it possible to produce completely spray-coated PSC devices. Yet, the use of spray coating in the manufacture of PSC is constrained by the following major problem: a thicker and more uneven layer ^[21]. Therefore, most current research focuses on improving the morphology of an active layer employing high boiling point solvents ^[22], additives, solvent combinations, post-thermal annealing, and other spray coating techniques ^[23].

Spin-coating is a rapid and popular method for depositing thin films on substrates, and its main benefit is that it is simple to create extremely uniform films. When a solution of a particular substance is spun at a fast speed, the centripetal force and the liquid's surface tension work together to cover the substrate uniformly. Spin-coating produces a thin film with a thickness of a few nm(s) to a few microns after the surplus solvent is removed. Small substrates that range in size from a few millimeters square to a meter or more in diameter are coated using the spin-coating process. The convenience and

relative ease of setting up the process, together with the thinness and homogeneity, are the main benefits of the spin-coating approach.

The dip-coating technique is a quick, easy, affordable, and high-quality coating method that is utilized in both industrial and lab applications [23]. The dip-coating method is frequently used for optical coatings, including large-area antireflective coatings for sun control glasses and the manufacture of vehicle rear mirrors. In the dip-coating procedure, a substrate is submerged in a solution of coating components before the solution is drained away. The procedure may be described as the solution-based deposition of an aqueous-based liquid phase onto a substrate's surface. The requisite material is typically dissolved in solutions and directly applied to the substrate surface. There are several complicated chemical and physical variables used in the dip-coating process. The duration of immersion, speed of withdrawal, dip-coating cycles, density and viscosity, surface tension, substrate surface, and coating solution evaporation factors all affect the thickness and shape of the film.

2. Dip-Coating

One of the easiest and most common methods for producing thin films from a wide range of inorganic, hybrid, and nanocomposite materials is the sol-gel method [24][25][26]. Considering that it provides for coating a broad range of substrates and complicated geometries, including substrates with holes or intricate patterns, it provides for a high degree of control over the crucial parameters and offers flexibility that cannot be achieved with other traditional processes. There are several ways for wet thin-film coating, namely, dip-coating [27][28], spin-coating [29], spray-coating [30], and flow-coating [31], among others. Fundamentally, the substrate to be coated is submerged in the initial solution and then pulled out at a consistent withdrawal speed during the process, which is carried out under well-measured temperature and air conditions. A fine-tuning of the film properties, including thickness, optical constants, and interior structure, is made feasible by precisely controlling the withdrawal rate and evaporation circumstances. The solution uniformly spreads out along the surface of the substrate because of the collective effects of viscous drag and capillary action. Evaporation takes place at the process' last stage, resulting in the gelation of the film. The coated substrates often receive a post-heat treatment, which affects the properties of the films [32]. The dip-coating process is shown in **Figure 1**.

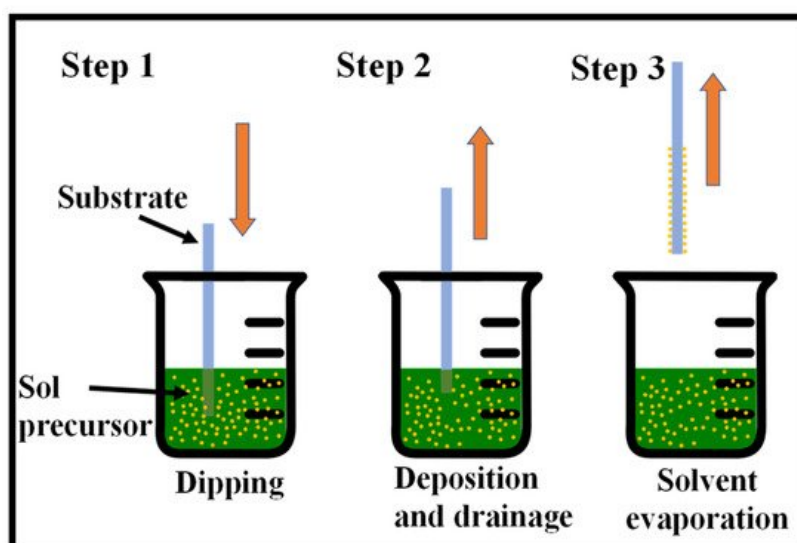


Figure 1. Sequential stages of the sol-gel dip-coating method for thin film deposition: Stage 1—the substrate is dipped and immersed in the sol precursor, Stage 2—the substrate is withdrawn at a steady rate, Stage 3—solvent evaporation produces the gelation of the layer.

The effectiveness of the dip-coating process can be directly impacted by several factors, such as pH and solution concentration, by changing, for example, its viscosity. However, as contemporary studies employ coating procedures that have already been documented in earlier works, most authors don't talk much about pH and solution viscosity. Fewer repetitions of the dip-coating process are required to produce a certain thickness or amount of deposited mass when using more viscous solutions since they lead to greater agglomeration and thicker layers of coating [33]. However, the resultant coating could potentially break and clump particles in undesirable places. To obtain past these issues, the dip-coating technique can provide a more homogenous coating by using a less viscous solution in combination with multiple repeats. The structure's rate of immersion in the solution is yet another factor that might affect the coating's features [34]. A high rate of immersion, for example, may produce thicker layers because the solution's viscous forces are increased, causing greater accumulation on the structure's surface [35].

For thin-film coating techniques to be employed successfully in integrated optics, the film thickness must be completely under control. Because of this, thickness control is important for all thin-film development methods, including sol–gel. It has been reported that the coating speed, angle of inclination, and sol concentration have a major role in determining the ultimate thickness [36][37][38]. In addition, the ultimate heat-treated thickness can also be influenced by the sol viscosity, density, and liquid-vapor surface tension [39]. In [40], it is stated that a cleanroom atmosphere is required for the coating procedure to produce sol–gel thin films with great optical quality. To produce organic dye-doped thin films with tailored porosity for use in chemical sensing and optoelectronics, a three-step sol–gel procedure was developed [41]. Additionally, ceramic films generated from sol–gel are also presented in [24]. Here are some further important papers on the sol–gel technique [42][43][44][45][46][47][48][49][50][51].

Silica, titania, and silica-titania materials produced by the sol–gel technique have all undergone extensive research due to their potential optical uses [52][53][54][55][56][57]. The early part of the 1980s saw the beginning of their use in the production of silica-titania WG films. The first individuals to do so were Herrmann and Wildman. However, they used MERCK's [53] commercially available liquid-coat solutions rather than synthetic sols. The research team led by Lukosz produced planar evanescent WG chemical/biochemical sensors utilizing these WG films, which were coated on glass substrates using the dip-coating technique and had refractive $n = 1.8$ at $\lambda = 612.5 \mu\text{m}$ [54][55][56][58]. These films have optical losses of 2.5 dB/cm for $\lambda = 632.8 \mu\text{m}$ [58]. Spin-coating was used by Jiwei et al. to fabricate $\text{SiO}_2\text{-TiO}_2$ WG films that were then coated on SiO_2/Si (111) substrates [57]. The greatest refractive index of the films was $n = 1.87$ at $\lambda = 632.8 \mu\text{m}$, yet they had very significant optical losses of 7.4 dB/cm. It should be noted that 750°C annealing temperatures were used to generate such a high refractive index value. The phase change from anatase to rutile is expected to occur at such high temperatures, making WG films much more lossy. There are also other studies [59][60][61][62] reporting the creation and characterization of composite $\text{SiO}_2\text{-TiO}_2$ films, although their waveguiding characteristics were not studied.

If the titania concentration is greater than 20 wt.%, the sol–gel-based manufacturing process of $\text{SiO}_2\text{-TiO}_2$ is quite challenging. This is due to titania's great propensity to crystallize and produce distinct phases. As a result, manufactured films exhibit large optical losses and are not amorphous [63]. Another issue that causes optical losses to increase over time is long-term stability [64]. These challenges were overcome by Karasinski et al. by creating $\text{SiO}_2\text{-TiO}_2$ WG films with a 50% weight TiO_2 content that are low-loss and long-term stable [23]. Using the dip-coating process on BK7 glass substrates, silica-titanium WG layers with a $\text{SiO}_2\text{-TiO}_2 = 1:1$ molar ratio were created, which were subsequently heated to 500°C . The primary chemical precursors for silica SiO_2 and titania TiO_2 are tetraethyl orthosilicate $\text{Si}(\text{OC}_2\text{H}_5)_4$ (TEOS) and tetraethyl orthotitanate $\text{Ti}(\text{OC}_2\text{H}_5)_4$ (TET), respectively. Water, ethanol, and hydrochloric acid (HCl), which catalyzes the processes of condensation and hydrolysis, are the additional substances used in the procedure.

Moreover, Karasinski and co-workers produced a variety of films on glass substrates at various speeds to identify the key technical characteristic that demonstrates the link between the thickness and refractive index of waveguide films about the speed of substrate removal from the sol. After deposition, the films underwent a 60-min annealing process at 500°C . Finally, using the monochromatic, multiangle ellipsometer Sentech SE 400 adv with a wavelength of 632.8 nm, the thickness and refractive index of those films were determined. **Figure 2** displays the outcomes' features [36]. From the parameters shown in **Figure 2**, a single coating procedure can only provide a maximum thickness of roughly 230 nm in the range of withdrawal speed shown. With the repeated coating method, thicker films may be created, which is important because the sample needs to be annealed after each coating. In order to determine the properties shown in **Figure 2** of the set of ten $\text{SiO}_2\text{-TiO}_2$ waveguides that were created for this research in a single coating procedure, as well as a few more films in a double coating process.

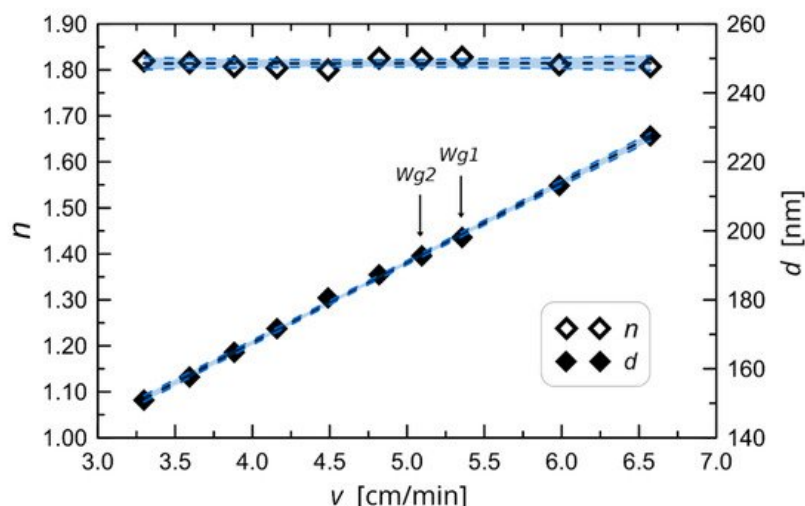


Figure 2. The experimentally determined characteristic of waveguide film thickness and refractive index against change in substrate withdrawal speed from the sol [36].

Nanoimprint lithography (NIL) is used to construct a Bragg grating (BG) device in a sol–gel silica WG for bio-phonic applications [65]. A reasonably wide area in the range of several micrometers with a resolution in the order of several nanometers is achieved by the procedure, which also achieves non-standardized lithography in sol–gel silica at a high resolution. In a sol–gel silica optical WG, structures with between 250 and 90 nm resolutions were shown for a sizable area that has not yet been tuned. For a 1 mm long region, a 250 nm periodic structure BG is produced. Two sol–gel silica BG structures, one measuring 250 nm broad and the other 90 nm wide, were then photographed using SEM as shown in **Figure 3a,b**. The transmission and reflection spectra of the BG structure are presented in **Figure 3c,d**, respectively. Based on the transmission and reflection spectra of laser light coupled into the WG at a wavelength of 1.55 μm , the efficiency of the grating structure in the WG was evaluated. The transmission and reflection spectra demonstrate that the WG grating was successfully constructed using the NIL procedure.

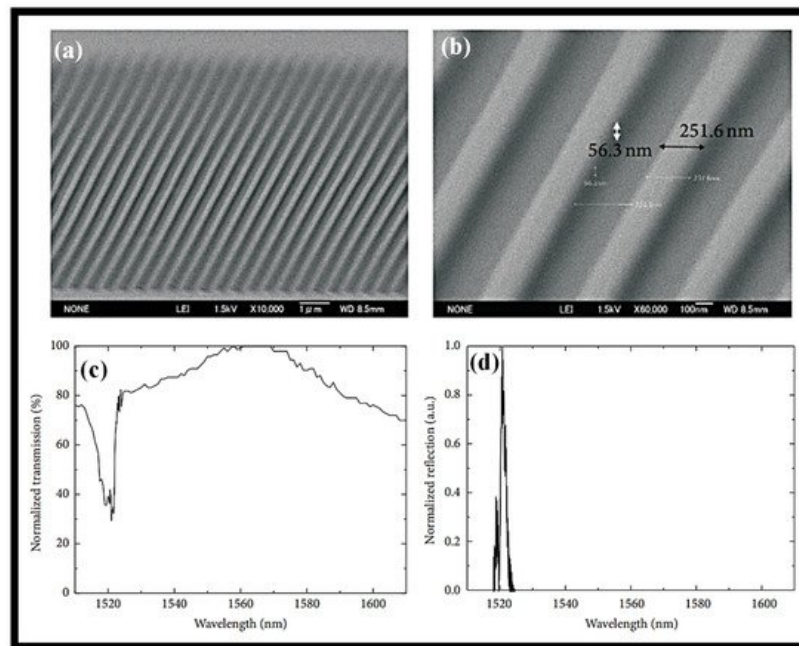


Figure 3. Sol–gel silica BG, (a) SEM image of the large area of periodic structure [65], (b) zoomed area of the periodic structure [65], (c) transmission spectrum of the BG structure [65], (d) reflection spectrum of the BG structure [65].

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