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Spin Coating: Process, Applications, Challenges, and Characterization Techniques

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Spin coating is a thin film deposition technique that is widely used due to its simplicity, low cost, and efficiency in forming homogeneous layers. It is based on the centrifugation of liquid solutions on substrates and enables the controlled deposition of polymers, semiconductors, oxides, and nanoparticles. This versatility makes it indispensable for both research and industrial applications, particularly in the fields of microelectronics, sensors, optical devices, renewable energy, and biomedical engineering. In microelectronics, it is used in the production of insulating and conductive layers for integrated circuits and OLEDs. In sensor technology and optical devices, it ensures precision in the deposition of sensitive materials. In the field of renewable energies, it contributes to the production of thin-film solar cells. In biomedical engineering, it enables the development of bioactive coatings and systems for the controlled release of drugs. Spin coating is crucial in the production of electrolytes for fuel cells, which require highly conductive, defect-free thin films to optimize ion transport and energy efficiency. Materials such as YSZ, GDC, LSGM, LSCF and SDC are widely used and each offer advantages in terms of ionic conductivity, chemical stability, and thermal compatibility. The controlled deposition of these materials allows dense and uniform layers to be achieved, reducing energy losses, and increasing device life. Continuous research into optimizing the materials and parameters of spin coating underlines its importance for technological innovation and sustainable development, as its adaptability and precision ensure that it will continue to play a key role in the development of various fields of technology in the future.

1. Introduction

The technique of layer deposition by rotation, known as spin coating, was developed in the 1920s to apply coatings to substrates. Since then, it has been widely used in the production of thin films for various industrial and scientific purposes. Its popularity stems from its simplicity and ability to produce thin, uniform layers, which has made it one of the most promising methods in materials engineering (Yada, 1995; Tyona, 2013). The method has proven to be particularly effective in the development of microelectronic devices such as sensors and optical components, as well as in biomedical applications and the production of membranes for molecular filtration. The versatility of the process enables the deposition of a wide range of materials, making it an indispensable tool for the development of new functional devices. In this process, a solution is applied to a substrate and rotated at high speed. This motion ensures uniform distribution of the liquid and removes the excess by centrifugal force, creating a homogeneous film after the material has dried or solidified (Vorotilov, 1995; Tyona, 2013; Mouhamad et al, 2014; Kalafatis et al, 2024).

The thickness of the finished film depends on several factors, such as the speed of rotation, the viscosity of the solution and the duration of centrifugation. As a rule, higher speeds lead to thinner films. Ambient conditions such as drying temperature and atmosphere also significantly affect the uniformity and quality of the film (Yada, 1995; Vorotilov, 1995; Tyona, 2013; Chou et al, 2021; Kalafatis et al, 2024). This method can be used to deposit a variety of materials in solution, suspension or gel form, including polymers, semiconductors, oxides and nanoparticles. These are commonly used in electronics, sensors, displays and optical devices. In research and

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development, spin coating plays a crucial role in the production of films for photovoltaics, biosensors and protective coatings (Vorotilov, 1995; Tyona, 2013; Chou et al., 2021; Kalafatis et al., 2024).

Spin coating has also been explored for the production of electrolytes in fuel cells, as it can produce thin and uniform layers of ion-conducting materials. Such uniformity improves ion transport and helps to reduce internal resistance, thereby improving fuel cell efficiency (Cardoso et al., 2021; Kalafatis et al., 2024). The method has been used to deposit ceramic electrolyte films based on materials such as yttrium-stabilized zirconia (YSZ), gadolinium-doped ceria (GDC), lanthanum strontium gallate magnesium (LSGM) and samarium-doped ceria (SDC), all of which are known for their high ionic conductivity and important role in solid oxide fuel cells (SOFC) (Kalafatis et al., 2024). Beta-alumina, another extensively studied material, is used in high-temperature fuel cells due to its excellent thermal and ionic properties. In addition, polymer electrolytes such as Nafion have been investigated for low-temperature applications, especially in proton exchange membrane fuel cells (PEMFC) (Vorotilov, 1995; Tyona, 2013; Mouhamad et al., 2014; Kalafatis et al., 2024).

2. Spin coating technology

The combination of these materials with the spin-coating technique enables the production of ultra-thin and uniform layers that ensure higher efficiency and durability of the energy conversion devices. Precise control of the thickness and morphology of the resulting layers is crucial for optimizing the performance of fuel cells, making this approach a promising alternative to produce advanced electrolytes (Kalafatis et al., 2024).

With advances in nanotechnology and the growing need for materials with unique optical, electronic, magnetic, and mechanical properties, research into and improvement of spin coating has become inevitable. The greatest advantage of this technique is its ability to produce thin films efficiently, cost-effectively and with high reproducibility. Among the film deposition techniques such as dip coating, sputtering and spin coating, the biggest challenge is to find a balance between quality and cost. Sophisticated equipment is often required to ensure film uniformity, which increases production costs (Yada, 1995; Kalafatis et al., 2024).

Spin coating, on the other hand, is characterized by the fact that only the rotation and viscosity of the material need to be controlled. This simplifies the process and enables the formation of coatings with good physical and chemical properties. The applicability of this technique extends to the semiconductor industry, biomedical engineering, optical coatings and even the production of active layers for chemical and biological sensors. In addition, spin coating has been explored in the renewable energy industry to produce perovskite layers in solar cells, which has contributed to significant advances in energy efficiency (Yada, 1995; Vorotilov, 1995; Tyona, 2013; Mouhamad et al., 2014; Kalafatis et al., 2024).

The production of electrolytes for fuel cells is another important application of this technique, which ensures the formation of thin and dense layers that maximize the efficiency of ion transport. These thin films act as selective barriers for the passage of ions, avoiding short circuits and optimizing the conversion of chemical energy into electrical energy, which is essential for the development of clean and sustainable energy sources.

The growing demand for thin films with improved properties has driven the development of new deposition techniques, among which spin coating stands out as one of the most studied and applied options due to its accessible and efficient approach, as its versatility and low cost make it an excellent alternative for the production of functional coatings in various fields that promote technological and scientific innovation

Continuous research to improve deposition parameters and the development of new materials compatible with this technique promise a further expansion of applications. Spin coating will therefore continue to play a key role in the production of next-generation technological devices, including the development of advanced electrolytes for fuel cells, and make an important contribution to the sustainable energy sector and scientific innovation.

The process of film formation by centrifugation is shown in Figure 1. In the first phase, a volume of the material is applied to the substrate and the system is rotated with a certain acceleration up to a set speed: during such rotation, part of the solution is expelled from the substrate. After complete evaporation of the solvent, the film finally forms on the surface of the substrate. The speed of rotation can be varied, and its ideal value depends on several factors, such as the type of material used, the volatility of the solvent and the desired thickness of the dry film (Yada, 1995; Tyona, 2013; Mouhamad et al., 2014; Chou et al., 2021; Kalafatis et al., 2024).

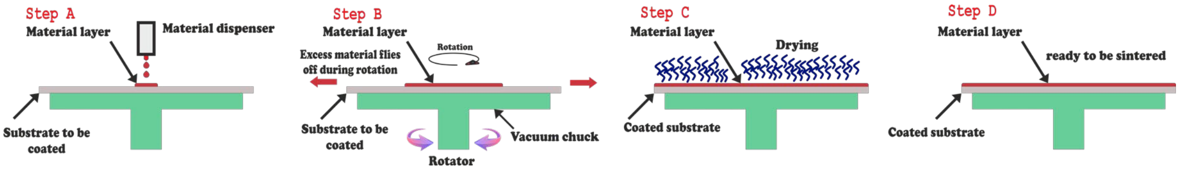

Figure 1: Procedure for preparing thin films using spin coating.

Spin coating is a widely used technique for producing very thin films, typically in the order of micrometres, and with uniform opacity. The desired film can be achieved by optimizing some parameters such as the concentration of the solution, the proportion of other additives, the viscosity of the solution and the rotation speed. This makes it possible to control the dynamics of film formation, which are inextricably linked to the final structure of the film. Spin coating technology was originally developed for the deposition of light-sensitive layers in the field of microelectronics and later for the deposition of various materials such as light-emitting diodes, photovoltaic cells, transistors, screens, sensors and biosensors, anti-fog coatings, Antireflective coatings and many other applications such as ceramic materials for the production of electrolytes for fuel cells, since the spin coating film formation technique has the advantage of being a very simple method that can be carried out with low-cost equipment both in terms of acquisition and assembly and maintenance (Kalafatis et al., 2024).The optimization of the various factors to obtain high quality films is relatively simple and the process of film formation is fast and reproducible (Vorotilov, 1995; Tyona, 2013; Mouhamad et al., 2014; Kalafatis et al., 2024).

As it is a batch process, it cannot be adapted for a continuous deposition process, which limits its use on very large deposition areas. The use of flexible or very thin substrates requires the use of additional supports to hold the substrate and prevent deformation due to suction and rotation. The main disadvantage of this technique is the large loss of material, especially when the material is deposited statically, as up to 90% of the material is usually ejected from the substrate and, in most cases, cannot be reused (Tyona, 2013; Kalafatis et al., 2024).

3. Types of deposition: static or dynamic

The formation of a film by centrifugation begins with the deposition phase, which can take place in two different ways. In static deposition, the material is deposited in the centre of the substrate while it is stationary, and after a few moments, rotation is activated (Tyona, 2013; Kalafatis et al., 2024).

In dynamic coating, the material is applied while the substrate rotates at a low speed, which reduces the influence of initial acceleration. After application, the rotation speed can be increased, but care must be taken to prevent the solution from being thrown off by centrifugal force. Regardless of the technique, it is essential that both the rotor and the base are completely flat, as any tilt may compromise the film's uniformity (Yada, 1995; Kalafatis et al., 2024).

The choice of substrate must be made carefully and adapted to the desired application. For the solution to spread well, the surface energy of the substrate must exceed the surface tension of the liquid. In addition, surfaces with rough edges can impede the flow of the solution and promote the accumulation of material in these areas (Yada, 1995; Vorotilov, 1995; Tyona, 2013; Mouhamad et al., 2014; Chou et al., 2021)

The choice between static or dynamic deposition depends on the properties of the system under investigation. Dynamic deposition is generally preferred for films that are produced at a speed of more than 1,000 rpm. In this context, a pipette can be used to apply the material quickly and precisely. The amount of solution required depends on the size of the substrate, the concentration, and the viscosity of the material (Chou et al., 2021).

Some precautions should be taken during dynamic deposition. The material should preferably be applied in the middle of the substrate to minimize irregularities in the film. In addition, the application should be done in a single motion, quickly and evenly, to avoid the formation of multiple layers. It is also important that the dispenser does not directly touch the surface of the substrate, to avoid irregularities (Chou et al., 2021; Kalafatis et al., 2024).

For efficient dynamic application, the substrate rotation must reach high values, or the amount of material must be sufficient to ensure complete coverage. In some cases, the viscosity and concentration of the solution must be adjusted to obtain a homogeneous film at high speeds (Mouhamad et al., 2014, Kalafatis et al., 2024).

For static coating, to ensure uniformity, it is recommended to cover the entire substrate with the material before rotation, although this is not an absolute requirement If the solution does not spread naturally, more material can be added or manually redistributed with a blade before continuing the process (Vorotilov, 1995; Chou et al., 2021; Mouhamad et al., 2014, Kalafatis et al., 2024).

Both static and dynamic techniques can be performed automatically with a programmable dispenser. Automation is particularly useful when the coating needs to be applied at a specific time or at a specific application rate, as it ensures greater accuracy and reproducibility compared to manual application.

During the acceleration phase, the substrate becomes fully covered with the applied material, while the spinning motion drives excess liquid toward the edges. This promotes uniform film formation. An ideal balance between rotation speed and material concentration ensures that viscosity does not hinder proper flow over the substrate. However, this balance cannot be defined by a fixed rule, as film formation depends on multiple factors, including material–substrate interaction, solvent volatility, acceleration, rotation, airflow, and ambient conditions like humidity. Thus, experimental adjustments are usually required to determine optimal parameters.

The final rotation speed of the substrate usually varies between 1,000 and 6,000 revolutions per minute, depending on the properties of the material and the substrate used. The time required for this step can vary from seconds to several minutes and is mainly influenced by the volatility of the solvent. This time should be long enough to ensure pre-drying of the film and homogeneous distribution of the material during rotation.

In many cases, the solvent evaporates completely during the deposition of the thin film. However, if low volatility solvents are used, it may be necessary to add a final step to remove residues. This can be done by heating the film in an oven, exposing it to a reduced pressure chamber or applying a gas flow to assist in the removal of the solvent residue (Tyona, 2013; Mouhamad et al., 2014; Chou et al., 2021; Kalafatis et al., 2024).

4. Mathematical basis

The mathematical modelling of the spin coating process requires the consideration of several parameters that influence the final thickness of the deposited film. These include the viscosity and concentration of the material, the speed and acceleration of the spin system and the changes in these properties during deposition. Due to the interdependence of these factors, accurately describing the process is a major challenge (Tyona, 2013). Viscous flow analyses show that the coating remains uniform during spin coating if the fluid behaves like a Newtonian fluid whose viscosity depends linearly on the shear rate, and if the initial liquid layer is uniformly distributed over the substrate. The result is a homogeneous film with consistent thickness (Tyona, 2013; Mouhamad et al., 2014; Chou et al., 2021). For practical purposes, the simplified models discussed here assume a static system. In this framework, the final thickness of the film depends directly on the viscosity and concentration of the solution and inversely on the angular velocity of the system. Therefore, increasing the rotational speed while decreasing the viscosity and concentration of the solution generally results in thinner films (Tyona, 2013; Chou et al., 2021).

Regardless of the initial material distribution, it is possible to achieve uniform films that become thinner over time. The time required to reach a certain thickness can be estimated based on the rotational speed of the system. This relationship is described by Equation 1, where h is the final thickness, h_0 is the initial thickness, ω is the angular velocity, v is the kinematic viscosity (equal to the dynamic viscosity divided by the density) and t is the time. This formulation is known as the EBP model after the initials of its authors and can be used to determine either the final thickness or the time required to obtain it. Equation 2 shows the rearranged form for calculating the time, where h and h_0 retain their meaning and ω corresponds to the rotational speed. Another model extends this analysis to non-Newtonian fluids. In such cases, if the solution does not reach a high enough concentration to mimic Newtonian behaviour, the final coating becomes non-uniform. Equation 3 expresses this by relating the final thickness h to a constant k, the initial concentration c and the rotation speed ω.

$$h = h_0 \times \left[1 + 4 \times \left(\frac{\omega^2}{3\nu}\right) \times h_0^2 \times t\right]^{-\frac{1}{2}} \quad (1) \quad | \quad t = \frac{3}{4\omega^2} \times \left(\frac{1}{h^2} - \frac{1}{h_0^2}\right) \quad (2) \quad | \quad h = k \times \frac{c^2}{\omega} \quad (3)$$

The mathematical framework also considers the evaporation of the solvent, which is important during and after the initial flow phase. Initially, a uniform layer of liquid covers the substrate, without edge barriers that would prevent redistribution of the liquid. This structure ensures an even distribution of the material (Tyona, 2013). At the beginning, the dynamics are determined exclusively by the viscous flow. During this phase, the centrifugal force and the viscosity of the liquid control the spreading of the layer and ensure a uniform distribution until the thickness decreases to about one third of the original value. As the layer becomes thinner, evaporation begins to play a greater role (Tyona, 2013).

The loss of solvents increases the concentration of solutes and promotes rapid solidification. As the thickness decreases, the increasing viscosity restricts the mobility of the liquid. Eventually, the viscosity becomes high enough to stop the flow completely and the film stabilizes. From this point on, only evaporation is effective, bringing the system to its final state. Assuming that evaporation is negligible at the beginning, the solute concentration c corresponds to the initial concentration c_0 and the flow velocity v to its initial value v_0. When the thickness decreases to about half of the initial value, the evaporation rate e is equal to the flow velocity and marks the transition to a state dominated by evaporation (Tyona, 2013).

Equation 4 expresses the evaporation rate, where is the evaporation, c is the solute concentration, ω is the angular velocity, h is the current thickness and v is the kinematic viscosity. Equation 5 provides a more comprehensive prediction of the final film thickness h in the presence of evaporation, based on the constants c_0, ω, v_0 and e, which describe how these parameters interact in the final drying phase (Tyona, 2013; Mouhamad et al, 2014; Chou et al, 2021; Kalafatis et al, 2024).

$$e = \frac{(1-\mathrm{c}) \times 2\omega^2 h^3}{3\nu} \quad (4) \quad | \quad h = \frac{3^{\frac{1}{3}}}{2} \times c_0 \times (1 - c_0)^{-\frac{1}{3}} \times \omega^{-\frac{2}{3}} \times \nu_0^{\frac{1}{3}} \times e^{\frac{1}{3}} \quad (5)$$

5. Defects in films produced by spin coating.

The most important defects in films produced by spin coating are shown in Figure 2. To ensure the production of consistent and defect-free coatings, it is important to understand the causes of each type of defect and apply appropriate control strategies. High quality films are expected to have a smooth and homogeneous appearance, a result that is often achieved through iterative process optimization. Many defects can be minimized by adjusting process parameters, especially in terms of solution formulation and rotational dynamics.

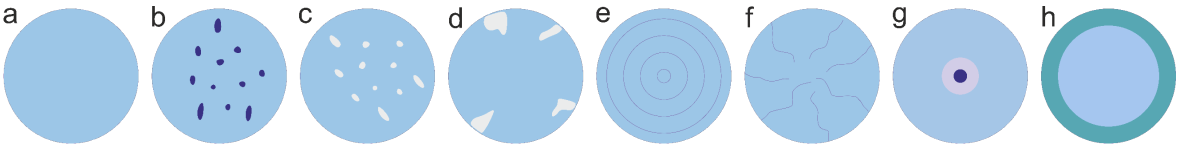

Figure 2 – Representations of spin coated films: (a) no visible defects; (b) grains; (c) pinholes; (d) incomplete coverage; (e) uneven thickness; (f) streaks; (g) rotor mark and (h) thick edges.

A film without visible defects (Figure 2a) represents the desired result, but several common defects can occur. The formation of grains (Figure 2b) can result from agglomeration of particles in the solution or uneven drying and can affect the smoothness of the surface and optical clarity.

Pinholes (Figure 2c), often caused by trapped air bubbles during evaporation of the solvent, affect the barrier properties of the film. They can be prevented by degassing the solution and controlling both the evaporation rate and the spin speed. Incomplete coverage (Figure 2d) can be caused by low rotation speed, insufficient solution volume or poor wettability of the substrate, resulting in exposed areas on the surface.

Uneven film thickness (Figure 2e) is usually due to off-center deposition, high viscosity or rapid solvent loss. Such irregularities affect the performance of the device and must be corrected by fine-tuning the coating conditions. Streaks (Figure 2f), which appear as radial patterns, are related to gradients in surface tension and can be reduced by optimizing the solvent or adding surfactants.

Rotor marks (Figure 2g) occur when the substrate is not properly aligned or secured during the spin cycle, resulting in mechanical deformation of the film. Finally, thick rims (Figure 2h) are caused by liquid accumulation at the periphery during deceleration and are influenced by the rheology of the solution and the final drying phase.

The influence of the viscosity of the solvent and the evaporation on the uniformity of the film are decisive factors for the quality of the finished film. Solutions with higher viscosity tend to produce thicker films and can lead to edge formation due to slower radial spread.

Conversely, low viscosity formulations result in films that are more prone to instability during drying. In addition, a fast evaporation rate can lead to defects such as pinholes and surface roughness, while slower evaporation favors better molecular arrangement and uniformity. These results highlight the need to optimize both solution properties and processing conditions to obtain high quality spin-coating films.

Understanding the origin and impact of each defect is key to improving film quality and ensuring reliable performance in applications ranging from optics to electronics (Tyona, 2013; Chou et al., 2021; Mouhamad et al., 2014; Kalafatis et al., 2024).

6. Analysis of films obtained by spin coating.

Film uniformity, one of the most critical aspects, can be visually assessed, allowing for immediate adjustments to experimental parameters to improve coating quality. However, a more detailed surface analysis requires advanced microscopy techniques such as optical reflectance microscopy (OM), scanning electron microscopy (SEM), and atomic force microscopy (AFM), which provide precise information on surface morphology and identify structural defects that may affect film performance (Kalafatis et al., 2024). In addition to surface characterization, film thickness is a crucial parameter for evaluating coating quality. Common measurement techniques include optical and direct methods, with white light interferometry (WLI), ellipsometry, and scanning electron microscopy (SEM) being among the most used. SEM is particularly favoured for its high-resolution imaging, which enables precise analysis of both morphology and layer thickness.

To ensure accurate thickness analysis, the sample must be properly prepared. The process begins by embedding the coated substrate in epoxy resin to provide stability and prevent mechanical damage. After curing, a cross-section is prepared to expose the film and substrate. For precise results, the cut must use an ultra-thin wheel operated at low speed to avoid deformations in the film structure (Tyona, 2013; Mouhamad et al., 2014; Venezia, 2019; Chou et al., 2021; Kalafatis et al., 2024; Basbus, 2024). When the sample is properly prepared, SEM analysis allows clear observation of the interface between the film and the substrate and thus a direct measurement of the film thickness. The high magnification and depth of field of electron microscopy enables the identification of thickness variations, structural defects, and irregularities in the coating, contributing to the quality control of the spin coating process (Tyona, 2013; Mouhamad et al., 2014).

Figure 3 presents a scanning electron microscopy (SEM) image of a film produced using the spin coating technique. Substrate of Gadolinium-Doped Ceria (GDC) and film of Lanthanum Strontium Cobalt Ferrite (LSCF).

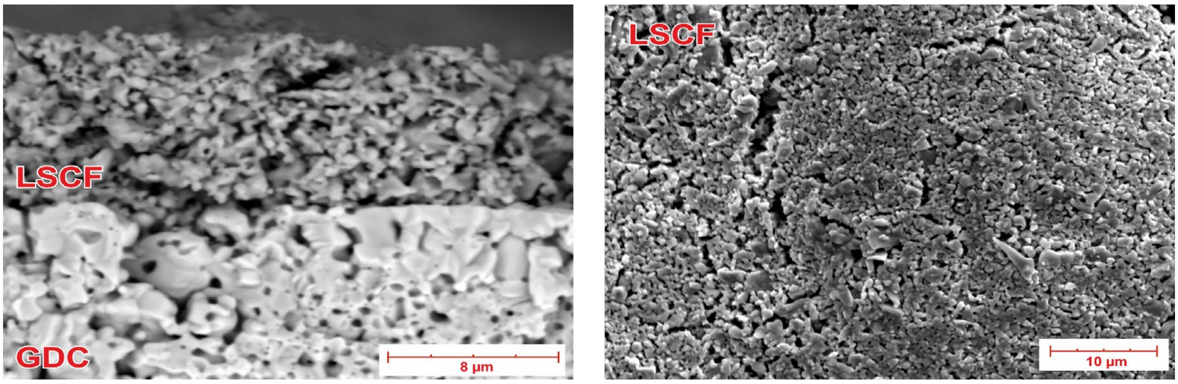

Figure 3 – On the left, a cross-sectional view, and on the right, a top-view image of a film produced using the spin coating technique.

7. Final considerations

The technique of centrifugation coating, commonly referred to as spin coating, is widely used to produce thin films on flat substrates of various shapes and dimensions. Due to its simplicity, the required equipment is easily accessible and inexpensive, making it particularly attractive for laboratory-scale research applications. However, to obtain high-quality films, numerous parameters must be precisely controlled, including the concentration of the solution, the choice of solvent, the rotation speed, the duration of spinning and drying, and the deposition method, whether static or dynamic. This technique enables the production of films with variable thickness and multilayer structures and is therefore suitable for applications in various scientific and technological fields. In addition to its extensive use in academic and experimental fields, spin coating is also becoming increasingly important in industry for the production of optoelectronic devices, biosensors and coatings for advanced functional surfaces.

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