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Sol-gel Spin Coating method for thin film deposition

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Abstract:

A sample may be referred to as a thin film if one of its dimensions is much smaller than the other two [1]. They consist of thin material layers that range in size from a few micrometres to fractions of a nanometre. Thin films are advantageous for optical coatings, photonic devices, electronic semiconductor devices, etc. The substrate material, the rate and angle of deposition, the ambient environment, and the deposition technique all have a significant impact on thin film attributes. High optical transmission/reflection, adhesion, hardness, porosity, chemically inert for corrosive environments, high resistivity or conductivity, and stability with regard to stoichiometry, orientation in single, and temperature and polycrystalline films are among the specific film properties required by the particular applications. Additionally, amorphous films have been proven to be beneficial in lasing materials [5] and optical sensors [2-4].

Techniques for thin film deposition

Thin film deposition is the process of applying a thin layer of any substance with appropriate adhesion to a substrate surface. Physical and chemical deposition methods are the two main categories under which deposition techniques fall.

A film's deposition procedure may be broken down into three main stages:

1. Making the particles (atoms, molecules, and clusters) that make the film
2. Movement of particles from the source to the substrate
3. Adsorption of particles onto the substrate and the creation of films

Physical Deposition Methods

For physical deposition techniques, a vacuum of 10-6 Torr or higher must be maintained. They cover a variety of techniques, including cathodic arc deposition, thermal evaporation, pulsed

laser deposition, and sputtering. In addition to these, other techniques like as liquid phase epitaxy, molecular beam epitaxy, atomic layer deposition, reactive sputtering, etc. are also employed to achieve better film quality by carefully regulating the deposition conditions. The substrate temperature, rate of deposition, residual gas pressure in the system, ambient conditions, the purity of the material to be deposited, inhomogeneity in the films, and structural or compositional variations of the films in localized or wider areas all affect the electrical, optical, magnetic, and surface properties of the deposited films [6].

Chemical Deposition Methods

Chemical vapour deposition, electroplating, plasma accelerated chemical vapour deposition, organometallic solutions, and other techniques are examples of chemical deposition techniques. The application of organometallic solutions to a substrate's surface by dipping, spinning, or spraying is a non-vacuum method for creating thin optical films. After that, the film is dried and cooked in a furnace. As the film undergoes thermal breakdown, the organic components evaporate and the film is transformed into pure oxide. By modifying the solution's temperature, viscosity, and other characteristics, thickness may be controlled. This method has been effectively applied to coating high power optical laser components with antireflection designs that have highly damaged thresholds, in addition to being continually employed in materials-related research. We shall talk about sol-gel spin coating with organometallic solutions in this chapter. The films produced via sol-gel spin coating have a thickness of a few hundred nanometres. ZnO-based films may be made using a variety of techniques, including dip coating, sol-gel, chemical vapour deposition, and spray pyrolysis. In addition to creating high-quality films, sol-gel is the most straightforward of these techniques.

Sol-gel process

Thin films, nanostructures, nanorods, and other materials are frequently deposited using the sol-gel method in chemical deposition. Any metal oxide may be prepared sol-gel by mixing reactants and then solidifying the resulting solution to create an amorphous oxide gel [7]. Densified glasses and polycrystalline solids are produced by heating the porous oxide [8]. Dopant incorporation and composite synthesis are simple processes [9]. An alternative to vacuum

deposition techniques is the sol-gel approach. Since it permits maskless processes like micro contact printing, inkjet printing, and roll printing technology. Metal oxide semiconductor films for optoelectronic and microelectronic devices may be formed using solution-based techniques, which might reduce costs and boost mass manufacturing throughput [10].

There are basically three processes in the sol-gel process [11]-

a. The creation of low viscous solutions of appropriate precursors, such as organic or inorganic metal derivatives, which may ultimately result in the oxides or metal oxides. Homogeneity is ensured via low viscosity.
b. During desiccation, a uniform sol is formed and allowed to gel, giving the ceramic product chemical homogeneity.
c. Forming into the final fibres, surface coating, etc., during or after gelation prior to annealing.

The primary elements influencing the sol-gel process are the temperature, the medium's pH, and the anions or complexing agents in the solution [12]. The sol-gel technique is utilised to create thin-film transistors [13,14], high-temperature excitonic devices [15], antireflection coatings, solar-reflecting glass, protective coatings, magnetic films [17], transparent conducting films [16], sensor films [4], and more.

Sol and precursors

The first chemicals, known as "precursors," are mixtures of pertinent substances that function as solutes in the sol-gel process [18] and ought to be capable of forming reactive inorganic monomers or oligomers. Precursors must be enough reactive to take part in the gel-forming process and soluble in the reaction medium. The chemical makeup of the precursor and the reaction circumstances that are used determine its reactivity.

Metal hydroxides or oxides often stay in the solution during the sol-gel process, making it immediately coatable on the substrates. By altering the solution's concentration or viscosity, the film's thickness may be changed. When a soluble form of metal is unavailable, its organic form used as a precursor in an appropriate solvent. It is allowed to hydrolyse slowly and carefully until

it produces network of metal-oxygen-metal in a sol state, usually preserving the solubility and transparency of solution. The product is readily liberated [19] from the carbonaceous residue in the case of metal oxide precursors. Group I and II metal alkoxides [11] are non-volatile in nature that frequently exhibit deprived solubility in organic solvents. Metal salts having soluble nature in organic solvents and readily transformed into the oxide by thermal or oxidative breakdown, are employed as an alternative [11].

Gelation:

The rheology of fluid sols [6] determines their conversion to solidified gel [20]. Bonds are created and the gelation method decreased the distance between the colloidal molecules.

a. Water extraction or evaporation via spray drying, dispersion in an immiscible fluid, or tray drying can turn the solution [21] into wet gel.

b. by anion elimination or neutralisation, or

c. through organometallic compound polymerisation.

After homogeneous colloidal sol or solution is prepared, the network-forming mechanism, such as colloids or monomers, reacts to generate an active form [18]. Gelation is the result of reactive surface groups neutralising surface charges, aggregating, and then further condensing stable colloids.

Sol or precursor states are utilised to deposit thin films, whereas gel states are employed to fabricate fibres. High levels of control over the crystallisation and creation of various porous and dense microstructures are possible with gels [22].

Sol-gel spin coating

A straightforward, affordable, and practical method for film deposition with solution-type chemicals is sol-gel spin coating. The substrate is forced to rotate along an axis perpendicular to the coating area during this operation. There are four separate steps in the process.

i. The substrate is evenly wetted in the first stage by pouring a regulated amount of the precursor over it. Larger particles are removed from the precursor using a sub-micron filter if necessary.

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ii. To eliminate the extra fluid, the substrate is spun at the specified rotation speed in the second stage. While the substrate rotates more quickly, the fluid layer's top imposes inertia. The twisting motion caused by these two forces has the potential to create spiral vortex. Normally, however, there is no indication of a thickness difference since the precursor is sufficiently thin to continue co-rotating with the substrate. When the substrate eventually reaches its target speed, the rotational acceleration precisely balances the viscous shear drag.

iii. The fluid's thinning behaviour in the third stage is dominated by viscous forces. Films tend to develop uniformly as a result of fluid thinning. But occasionally, edge effects are also observed. Outside, the whirling fluid flows evenly. Drops must develop at the margins so they can be thrown off if there is an excess of fluid. As a result, edge effect may result from the extremities of the substrate having a little higher thickness than the middle.

iv. The fluid begins to evaporate and takes charge of its own thinning behaviour in the 4^{th} and final stage. In this process, the solvent phase is eliminated and the sol is transformed into a opaque ceramic. High-speed rotation causes the fluid's temperature to increase, which causes the fluid to evaporate. As a result, the residual solution becomes more viscous, which causes the coating to enter its "gel-state." Typically, the final film has an amorphous structure. Even though evaporation and viscous flow, the third and fourth phases, happen at the same time, evaporation takes precedence over the viscous flow impact at first.

To preserve its viscosity, the sol must be stored in an airtight flask; otherwise, it will turn into gel and be unsuitable for film deposition.

By varying the solution viscosity and other coating parameters, such as rotation rate, annealing temperature, and coating duration, among others, the film thickness may be regulated [10, 23]. Oxide film deposition has made considerable use of the sol gel spin coating technique [24, 25-28, 10, 13-16,30,31].

The sol-gel process has the following benefits:

• Thickness control;

• High degree of uniformity;

• Multilayer coating;

• Control over the thin film microstructure, including pore volume, refractive index, surface area, pore size, etc.;

• No limitations on substrate shape or size;

• Also, the substrate can be coated on both sides at the same time.

References

1. Joy George, 'Preparation of Thin Films' Pub. Marcel Dekker NY(**1992**).
2. S. Dixit, A. Srivastava, A. Srivastava and R. K. Shukla, J. Appl. Phys. 102. 113114 (**2007**).
3. S. K. Shukla, G. K. Parashar, A. P. Mishra, P. Misra, B. C. Yadav, R. K. Shukla, A. Srivastava, A. Srivastava, F. Deba, G. C. Dubey, Chemical Sensors, 20, Supplement B 546 (**2004**).
4. S. Dixit, A. Srivastava, A. Srivastava, R. K. Shukla, Jpn. J. of Appl. Phys. 47(7A) 5613 (**2008**).
5. H. Dong, D. Wang, K. Chen, J. Huang, H. Sun, W. Li, J. Xu, and Z. Ma, Appl. Phys. Lett. 94, 161101(**2009**).
6. Ph.D. Thesis, "Structural and Optical Properties of Zinc Oxide and Barium Titanate Based Thin Films" by Kamakhya Prakash Misra, University of Lucknow, **2010**.
7. J. D. Mackenzie and D. R. Ulrich, Proc. of SPIE Conf. on Sol-gel Optics, 1328 (**1999**) 2-13.
8. B. D. Fabes et al, Proc. of SPIE conf. on Sol-gel Optics, 1328 (**1990**) 319-328.
9. J. D. Mackenzie, Proc. of SPIE conf. on Sol-gel Optics, 878 (**1988**)128.
10. C. Y. Tsay, H. C. Cheng, C. Y. Chen, K. J. Yang and C. K. Lin, Thin Solid Films 518, (**2009**) 1603.
11. R. C. Mehrotra, History of Precursors, Proc. of the Winter School on Glasses and Ceramics from Gels; Sao Carlos (SP) Barzil 14-19 Aug **1989**, Ed. M. A. Aegerter, M. Jafelicci Jr, D. F. Souza and E. D. Zanotto, Utopia Press, Singapore pp. 1-12.
12. E. Matijevic, Accounts of Chem. Res. 14 (**1981**) pp. 22.
13. H. C. Cheng, C. F. Chen, C. Y. Tsay, Appl. Phys. Lett. 90, 012113 (**2007**).

14. W. J. Park, H. S. Shin, B. D. Ahn, G. H. Kim, S. M. Lee, K. H. Kim and H. J. Kim, Appl. Phys. Lett. 93, 083508(**2008**).
15. Y. Zhang, B. Lin, X. Sun, and Z. Fu, Appl. Phys. Lett 86, 131910(**2005**).
16. A. Jain, P. Sagar and R. M. Mehra, Materials Science-Poland, 25, 233(**2007**).
17. S. -Y. Bae, C. –S. Kim and Y. –J. Oh, J. of App. Phy. 85(8) 5226(**1999**).
18. Helmut Schmidt, 'Chemical Processing up to Gelation' Proc. of The Winter School on Glasses and Ceramics from Gels; Sao Carlos (SP) Barzil 14-19 Aug **1989**, Ed. M. A. Aegerter, M. Jafelicci Jr, D. F. Souza and E. D. Zanotto, Utopia Press, Singapore pp. 62.
19. I. Thomas, US Patent 3, 799, 754, March 26, **1974**; I. Thomas, 'Multicomponent Glasses from the Sol-gel Process' in Sol-gel Technology for Thin Films, Fibers, Preforms Electronics and Speciality Shapes, Ed. L. C. Klein, Noyes Publications, New Jersey, **1988**.
20. Sumio Sakka, Rheology of Sols in the sol-gel processing, Proc. of the Winter School on Glasses and Ceramics from Gels; Sao Carlos (SP) Barzil 14-19 Aug **1989**, Ed. M. A. Aegerter, M. Jafelicci Jr, D. F. Souza and E. D. Zanotto, Utopia Press, Singapore pp. 76.
21. Jerzy Zarzycki, New Methods in Sol-gel Synthesis, Proc. of the Winter School on Glasses and Ceramics from Gels; Sao Carlos (SP) Barzil 14-19 Aug **1989**, Ed. M. A. Aegerter, M. Jafelicci Jr, D. F. Souza and E. D. Zanotto, Utopia Press, Singapore pp. 257-269.
22. G. Westin, M. Wijk and A. Pohl, J. of Sol-gel Sci. and Tech. 31, 283 (**2004**).
23. Sumio Sakka, Rheology of Sols in the sol-gel processing, Proc. of the Winter School on Glasses and Ceramics from Gels; Sao Carlos (SP) Barzil 14-19 Aug **1989**, Ed. M. A. Aegerter, M. Jafelicci Jr., D. F. Souza and E. D. Zanotto, Utopia Press, Singapore pp. 346-374.
24. N. Kumar, A. Srivastava, Journal of Alloys and Compounds 706, (**2017**) 438-446
25. K. P. Misra, R. K. Shukla, A. Srivastava, A. Srivastava, Appl. Phys. Lett. 95 (**2009**) 031901.
26. A. Srivastava, N. Kumar, K. P. Misra, S. Khare, Mater. Sci. Semicond. Process. 26(**2014**)259–266

27. R. K. Shukla, A. Srivastava, A. Srivastava, and K. C. Dubey, J. Cryst. Growth 294 (**2006**) 427-431.
28. Nishant Kumar, A. Srivastava, Opto-Electronics Review 26 (**2018**) 1–10.
29. A. Srivastava, N. Kumar, and S. Khare, Opto−Electron. Rev. 22(**2014**) 68-76.
30. Y Natsume, H Sakata, Thin Solid Films, 372 (**2000**) 30–36.
31. Y. Cao, L. Miao, S. Tanemura, M. Tanemura, Y. Kuno, Y. Hayashi, Appl. Phys. Lett. 88, (**2006**) 251116.