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**OPTIMIZATION OF ELECTROPHORETIC DEPOSITION
PARAMETERS FOR UNIFORM ZINC OXIDE DEPOSITION ON
CONDUCTIVE GLASS SUBSTRATE AND COMPARATIVE
ANALYSIS WITH TITANIUM OXIDE**

by

MD. MUHIDUR RAHMAN

NURUL ISLAM

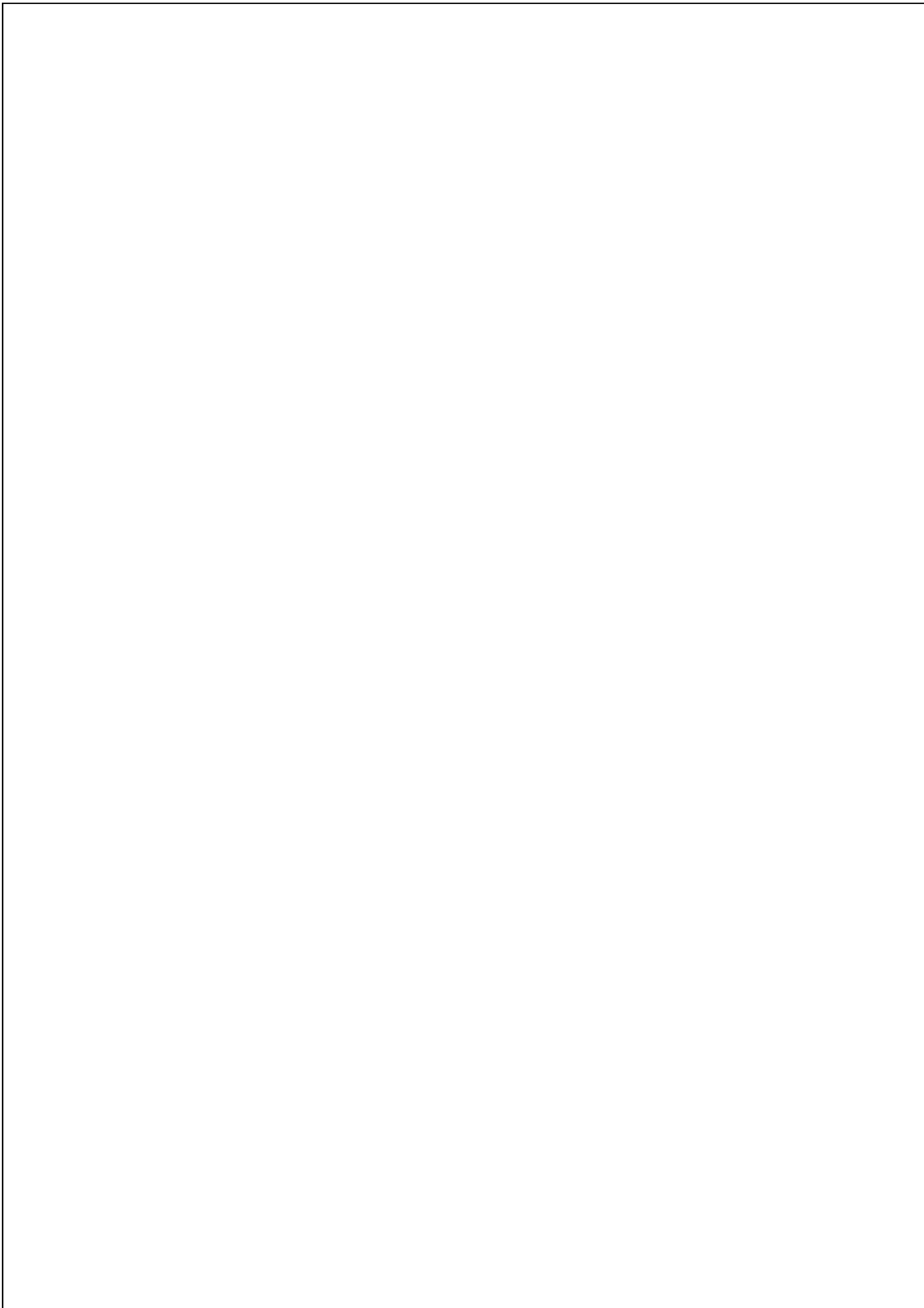
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**BACHELOR OF SCIENCE IN ELECTRICAL AND ELECTRONIC
ENGINEERING**



Department of Electrical and Electronic Engineering
INTERNATIONAL ISLAMIC UNIVERSITY CHITTAGONG

APRIL 2021



**OPTIMIZATION OF ³⁷ELECTROPHORETIC
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MD. MUHIDUR RAHMAN
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³
A thesis

submitted as partial fulfilment of the requirement for the degree of

**BACHELOR OF SCIENCE IN ELECTRICAL AND ELECTRONIC
ENGINEERING**

Department of Electrical and Electronic Engineering
INTERNATIONAL ISLAMIC UNIVERSITY CHITTAGONG

APRIL 2021

CERTIFICATE OF APPROVAL

The thesis entitled as “**Optimization of electrophoretic deposition parameters for uniform zinc oxide deposition on conductive glass substrate and comparative analysis with titanium oxide**” submitted by **Md. Muhidur Rahman**, bearing Matric ID. **ET-163001** and **Nurul Islam**, bearing Matric ID. **ET-163034** of session **Autumn 2020**, to the Department of Electrical and Electronic Engineering, International Islamic University Chittagong, has been accepted as satisfactory in partial fulfilment of the requirements for the degree of Bachelor of Science in Engineering and approved for the examination held on **30 March, 2021**.

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DECLARATION

It is hereby declared that this work has been done by us and no portion of the work contained in this thesis has been submitted elsewhere for the award of any degree or diploma.

Md. Muhidur Rahman

Nurul Islam

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Authors

ABSTRACT

This investigation is about the ⁶¹ Electrophoretic Deposition (EPD) of ZnO nanoparticles on the substrate of fluorine-doped tin oxide (FTO). It is always being a challenge to get a well-developed deposition. This study shows that smoother deposition ⁴⁸ can be achieved by controlling the deposition voltages, deposition time and deposition distance. The EPD processing is conducted via cathodic electrodeposition ¹ to get a uniform crack-free, smooth deposition of Zinc Oxide which is important to develop various electronics device applications. The work focuses on finding out the optimum condition for crack-free deposition by these three parameters mentioned above. From the observation it is seen that the amount of deposition increase when ¹ deposition time and voltage increase. On the other hand, increasing the deposition distance, amount of ¹ the deposition decreases. By observing all the data, deposition ¹ voltage, deposition time and deposition distance are 60 V, 60 s and 1.2 cm respectively produces crack-free deposition of ZnO and these are the parameters that has been proposed as an optimized deposition condition for the deposition of ZnO nanoparticles. Another comparative investigation between ZnO and TiO₂ shows, though the amount of the deposition of ZnO is low but ZnO produces smoother crack-free deposition than TiO₂.

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LIST OF ABBREVIATIONS

EPD	Electrophoretic deposition
ZnO	Zinc oxide
TiO ₂	Titanium Oxide
DSSC	Dye Sensitized Solar Cells
SEM	Scanning Electron Microscope
PVD	Physical Vapour Deposition
VET	Vacuum Evaporation Technology
EMI	Electromagnetic Interference
PPy	Polypyrrole
CVD	Chemical Vapour Deposition
TACVD	Thermally Activated Chemical Vapour Deposition
EDTA	Ethylene-Diamine-Tetraacetic Acid
PLD	Pulsed Laser Deposition
(YBCO)	Yttrium Barium Copper Oxide
PZT	Lead Zirconium Titanate
DC	Direct Current
FTO	Fluorine Doped Tin Oxide
LCD	Liquid-Crystal Display
2-D	Two-Dimensional
3-D	Three-Dimensional

CHAPTER 1

INTRODUCTION

1.1 Introduction

⁵³ In EPD, a DC electric field is applied to charged powder particles scattered or suspended in a liquid medium, and the DC field ⁷⁰ causes the charged particles to attract and deposit onto an oppositely charged conductive substrate [1,2]. The main driving forces of ² electrophoretic deposition (EPD) are the charge on the particle and the electrophoretic mobility of the particles in the solvent under the effect of an applied electric field [1]. Electrophoresis and deposition are two processes that are completed during EPD. The ⁵⁰ motion of charged particles in a suspension toward the oppositely charged electrode under the control of an electric field is known as electrophoresis. The ⁹¹ particles accumulate and form a consistent deposit on one of the electrodes is deposition [2-6]. A post-EPD processing process, which involves an appropriate heat treatment (firing or sintering) is usually necessary for further densification and removal of porosity [4,6]. By ⁶³ regulating the deposition time and applied potential in EPD, the thickness and ⁴⁷ morphology of a deposited film can be controlled [1]. Any solid which is available in the form of a fine powder (<30 μ m) or colloidal suspension can be subject to electrophoretic deposition. Examples of electrophoretic material deposition in nearly every material class include ⁹⁹ metals, polymers, carbides, oxides, nitrides and glasses [3,6]. ⁷⁷ Electrophoretic deposition (EPD) is a technique that has recently gained prominence in academia as well as the industrial field because of its ⁹ short formation time, needs simple apparatus, little restriction of the shape of substrate, ability to ²⁷ control the thickness and the morphology of the film, ² no requirement for binder burnout as the green coating ¹² contains few or no organics, its high versatility for application with different materials and combinations of materials, its cost-effectiveness, simplicity and the ¹² ability to be scaled-up to large product volumes and sizes [1,6,14,19]. The EPD methodology has ⁵ proved to be successful for thick film of silica, nano-size zeolite membrane, hydroxyapatite coating on a metal substrate for biomedical applications, luminescent materials, high-Tc superconducting films, gas diffusion electrodes and sensors, multi-layer composites, glass and ceramic matrix composites by infiltration of ceramic particles onto fiber fabrics, oxide nanorods, carbon nanotube film, functionally graded ceramics, layered ceramics, superconductors, piezoelectric materials etc [1]. By following four

mechanism charged particles can develop in suspension in EPD (1): (a) ion adsorption selectively from the liquid into the solid particle, (b) ion dissolution from solid to liquid form, (c) adsorption or orientation of dipolar molecules at the particle surface and (d) electron transfer between the solid and liquid phase due to differences in work function [3]. In EPD, there are two electrodes respectively positively charged electrode and negatively charged electrode. The positively charged particles attract by the cathode and deposit on the negatively charged electrode (cathode) and the process is called cathodic electrophoretic deposition. The negatively charged particles attract by an anode and deposit on the positively charged electrode (anode) and the process is termed as anodic electrophoretic deposition [1]. To control the EPD, the characteristics of this process need to know. Two classes of parameters govern the characteristics of this method; (i) those associated with the suspension, and (ii) those associated with the process including the physical parameters such as the electrical nature of the electrodes, the electrical conditions (voltage/intensity relationship, deposition time, etc). Particle size, the dielectric constant of liquid, conductivity of suspension, viscosity of the suspension, zeta potential, stability of suspension are parameters associated with the suspension. The Effect of deposition time, applied voltage, Concentration of solids in suspension, conductivity of substrate are parameters related to the process [1]. The coating or layer of material on the film can easily be controlled by these parameters such as deposition thickness, crack-free coating, deposition area etc. It is a challenge to know the optimal condition for uniform crack free deposition of a material on a specific film or substrate. ZnO and TiO₂ are two promising and widely used materials in the field of material science. ZnO is a well-known direct-band gap semiconductor with large exciton binding energy (60meV) at room temperature and one of the excellent semiconductors photocatalysts with a wider direct bandgap (3.37eV) [10]. Because of its distinct electrical, optical and mechanical properties, there is a great deal of interest in the fabrication of high-quality ZnO films for applications such as field emission, gas sensors, biomedical research, solar cells, superhydrophobic surfaces, optoelectronics, photo-electronic devices, diode lasers, and low-voltage, high-efficiency phosphors etc [8-15]. On the other side, TiO₂ is also a widely used material relatively with a shorter bandgap. The photocatalytic properties of titanium oxide are excellent and are thus used in antiseptic and antibacterial composites. It has the ability of organic pollutants and germs degradation and also resistant to UV. In this study by applying the EPD process, ZnO was deposited on a conductive glass substrate by varying various parameter such as

voltage, time, distance to achieve a uniform crack free ZnO layer. On the other side, a comparative study between ZnO and TiO₂ over electrophoretic deposition varying three parameters, deposition time, deposition distance and deposition voltage.

1.2 Background

Since 1808 Electrophoretic deposition (EPD) has been known. Ruess, a Russian scientist, discovered the idea of EPD in 1808 when he observed an electric field caused displacement of clay particles in water. But the methods were first applied in practice in 1933 when the deposition of thoria particles on a platinum cathode as an emitter for electron tube application was patented in the USA by Harsany [1,2,6]. EPD was primarily used until the early 1990s in the processing of conventional ceramics, including enamel and porcelain, and only little work was performed on EPD of engineering ceramics [6]. Hkamaker first studied the EPD of ceramics and the technique gained significant interest only in the 1980s in the area of advanced ceramics [1].

1.3 Objective

The main goal of this investigation is to study the principle of EPD and to optimize various deposition parameters to get a uniform crack free ZnO layer on conductive glass substrate for DSSC application and to study the characteristics of EPD on three parameters comparatively with TiO₂.

1.4 Thesis Layout

This thesis contains 5 chapters. A short description of each chapter is given as follows:

Chapter-1: Introduction: This chapter gives an overview of the whole work which consists of Introduction, Background, Objective and Thesis Layout.

Chapter-2: Deposition Techniques: In this chapter introduction to various deposition techniques are given. A literature review on Electrophoretic Deposition is also added in this chapter.

Chapter-3: Experimental: A short description of the apparatus used in this study are given and the full experimental methods are discussed in this chapter.

Chapter-4: Results and Analysis: This chapter contains the results found from the experiment and the analysis of these results.

Chapter-5: Conclusions and Future Work: Comments about the work, application and future scope of the work are discussed in this chapter.

Chapter 2

Deposition Techniques

2.1 Introduction

The processes of thin-film deposition are essentially either primarily physical, for example evaporative techniques and sputtering techniques, or purely chemical, like as gas-phase and liquid-phase chemical processes [16,17]. Physical-chemical approaches encompass a large range of processes dependent on glow discharges and reactive sputtering that incorporate physical and chemical reactions. Several methods can be adopted and the preference is mostly affected by the implementation, which primarily determines the approach applied by criteria such as deposition speed, film thickness and consistency. Some devices such as the frequency of memory changing required fast deposition and dielectric sealing of a dense and precise thin film layer of only a few nanometers [18-20]. According to the demand for the required thin film, the methods are selected. The definitions of deposition technologies are briefly described and a literature review on electrophoretic deposition are all included in this chapter.

2.2 Various Deposition Techniques

There are mainly two processes of deposition technologies of thin-film and each can be divided into two more subdivisions. These are:

- I. Physical process.
 - a) Evaporative Process.
 - b) Glow Discharge Process.
- II. Chemical Process
 - a) Gas-Phase Chemical Process.
 - b) Liquid-Phase Chemical Processes.

A classification scheme is presented in **Fig. 2.1**, a list of some typical deposition methods depending on criteria, short description about some common deposition technologies are given in the following sections.

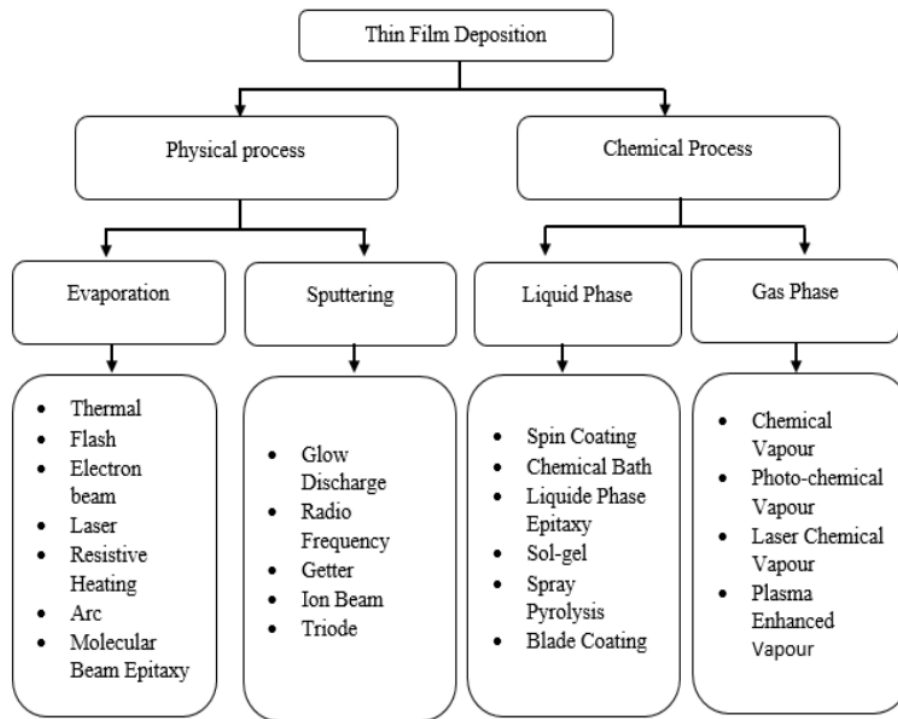


Fig 2.1 List of some typical deposition methods depending on criteria

2.2.1 Thermal Evaporation

Among the physical vapour deposition (PVD) techniques, thermal evaporation has a significant popularity to researchers. This is a method of thin film deposition, a technology of vacuum for applying pure material coatings to the surface of different artefacts. The coatings, also called films, are approximately in the range of thickness of angstroms to microns and maybe one or more materials in a structure layer.

The ingredients for thermal evaporation may be uncontaminated atomic elements, both metals and non-metals, or substances such as oxides and nitrides. It may include several items such as semiconductor wafers, solar cells, optical components or many other possibilities.

The subject is called the substratum. The heating of solid material in a high sealed container induces vapor-pressure temperature by a thermal evaporation system. The vapor cloud within a room can be increased by even lower vapor pressure from the inside

of the vacuum. It is a gas flow now passing through and sticking like a film or coating to the chamber.

Since the substance is heated up to its melting point and liquid in the majority of the thermal processes, it is generally situated in the lower part of the container, often in a kind of upright surface. The vapour rises above this base and the substrates are held inverted at the top of the chamber in suitable fixtures. Thus, the surfaces intended for coating face down to the heated raw material for coating [21].

The biggest benefit of the thermal evaporation method is that the control on the process and low temperature to deposit are fantastic. This technique also offers the advantage that elementary, ceramic and composite coatings are possible for this technique. The benefits of thermal evaporation technology are not only certain, they also have certain drawbacks, such as vacuum processes and high capital costs. Another drawback is that comparatively; low coating concentrations are possible for this method [20].

2.2.2 Vacuum Evaporation

The use of vacuum evaporation in different substrates is one of the most widely used methods for depositing operational films [22]. Vapour particles can be directly deposited on the substrate, where the vapor particles can be condensed into a solid state and thus form a practical covering. The method of vacuum evaporation includes two basic phases: the evaporation and condensation of a functional substance on the substrate. In high-vacuum evaporation, the coating materials are melted, gasified and evaporated by electrical heating or electron heating.

The vapor from the coating material then reaches the substrate's exterior and is eventually cooling; a decent quality thin film layer is successfully achieved. Vacuum is used to avoid the interaction of background gas and other harmful fragments with evaporated fragments. The top of the substrate is deposited with evaporated usable content. VET technique has been used extensively for the deposit in different substrate of practical films, such as resistance to wear, corrosion, high temperature, oxidation and radiation, as well as improved conductivity, permeability and insulation properties. VET is using a dot-source to evaporate covering materials which limits it being used in high boiling or melting points of metals and alloys. Evaporation has some inconveniences including a poor bonding power with the substratum [23].

2.2.3 Electron Beam Evaporation

Evaporation by electron ray, the material can be dissipated by charged particles in the form of an extreme laser during electron beam evaporation. The thermionic emission of electrons produces a hot filament that can provide enough energy, after acceleration, to evaporate some content. 10 kW was delivered on impact in a normal event with an emission of 1A accelerated by the 10 kV voltage drop [24]. The filament is positioned outside view of the evaporant as seen in Fig. 3.10 and the electron ray is drawn across the surface by a magnetic field, B, and the dot indicates the position in this diagram. In order to prevent the filament from melting in the incoming evaporant. The combined power, F, is known as Lorentz force for electrons in electrical (E) and magnetic fields [25]:

$$F = F_E + F_B = q_e E + q_e (v \times B) \quad (2.1)$$

Where every symbol contains usual meaning. The F_B vector is perpendicular to both v and B , as seen in Fig. 2.2 [26]. According to the Eq. (2.1), it speeds the electrons from the electrode or cathode by the first force expression. The acquired speed makes the electrons turn sideways while passing through the conjunction with the next force concept for magnetic field line of nanoparticles. The centrifugal power in radius r of the electrons balances the next factor.

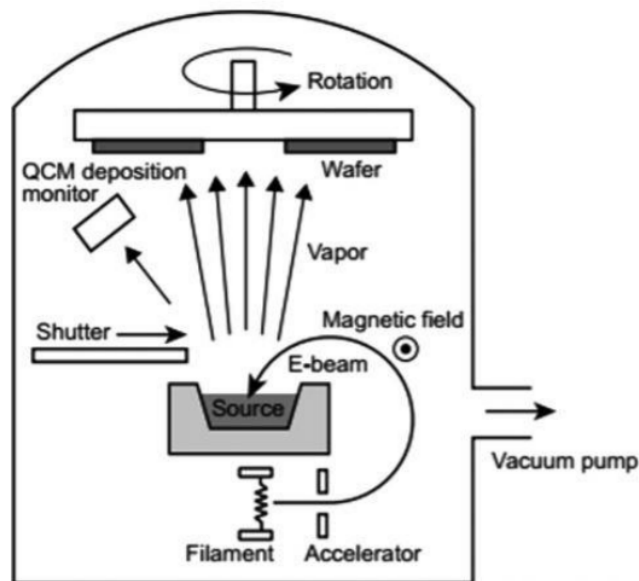


Fig. 2.2: Graphical representation of the phenomenon of the EBE system

2.2.4 Glow Discharge Sputtering Technique

The technique of glow discharge relies on plasma output in a gas tubing at low pressure, except for chemical decomposition of vapour for deposited a thin deposition on a surface, the materials emitted from the purpose are not used with plasma ions [28]. Plasma may be formed either through capacitive coupling or through inductive coupling after application of an RF field. This technique is exceptionally consistent in vast and diverse areas. The stoichiometric alloys and compounds can be easily obtained using this method. Technical glow discharge involves low wafer temperature. While this strategy has advantages, it is also disadvantageous because it needs low kinetic energy and usually near 1nm dense recesses. While this strategy has advantages, it is also disadvantageous because it needs low kinetic energy and usually near 1nm dense recesses [27].

106 **2.2.5 Radio Frequency Sputtering Technique**

Sputtering radiation frequency (RF) is also another method of sputtering, which entails alternating the electron density of the current in the void at the radio frequency to prevent the charge of accumulating certain kinds of sputtering targets. In RF sputtering, the cathode (the target) being the thin film layer and an anode are serially linked to the blocking condenser. The condenser is part of a network for balancing impedances, which transfers power to plasma emission from the RF source. The cathode is bombarded with high voltage in the sealed container, resulting in high-energy ions that sputter off ions as a coating that covers a surface at 13, 56 MHz. The nature of the capacitor is to create DC self-biases and to optimize the transmission of energy into the plasma produced for the target material. This method offers great opportunities like deposition on the on the non-conductive materials, having ability to maintain plasma at lower pressure like 0.13 to 2 Pa. It also can reduce the race path erosion at the end of the target surface and the ability to clear the target materials to reduce arcing effects [29-31].

2.2.6 Chemical bath deposition

Usually known as solution growth technique or controllable precipitation, chemical bath deposition method [32]. Bath deposition method is the earliest form in which films are placed on a surface. **Chalcogenide films as well as metal oxide films** are often prepared

by solution growth technology. Deposition at lower heat may also be achieved. The precursor solution for metal ions has to be complicated by ligands in the solution growth process. The complex model is defined nearly with a solution for ammonia, triethanol, ethylene-diamine-tetraacetic acid (EDTA), citric acid, etc. If the complexation is over, the anions should be added.

This anion comes as a source of sulfur anions or selenourea, and of sodium selenosulfate in the selenium anion for the deposition of chalcogenides from thiourea, thioacetamide, thiosulfate and sodium sulfide. Substrates are placed within the solution in longitudinal, horizontal or special position and are left to achieve the necessary film thickness. Oxide films are very distinct from chalcogenide deposition. The substance is immersed in the solution under the ideal temperature ranging from 60 to 100°C after the complexation by regulating the pH values, and is then mostly deposited under metal hydroxide films. The film will then be transmitted by the annealing to oxide [33].

2.2.7 Sol-gel Technique

For the synthesis of oxide materials, the sol-gel method has been widely used [36]. Sol-gel is among the best known wet-chemical processes. It operates at lower temperatures and improves the homogeneity of multi-component materials. A sol is the spread of a component in the fluid that suspends particles only through the Brownian movements. A gel is a phase in which liquid and solids are dispersed, and a solid network of liquid components is formed. Currently, the sol-gel covering procedure is 4 phases:

- (1) When scattered in fluid, the target colloidal particles become a sol.
- (2) The sol solvent deposition causes the spraying, sinking or flipping of the coatings on the substrate.
- (3) Solar particles are polymerized by removing the stabilizing elements, and the gel is generated in a continuous network state.
- (4) The remaining bio-or inorganic elements are pyrolyzed and amorphous or crystalline cover are formed by final thermal treatments [34, 35].

The technique Sol-gel offers the benefits of the capability to create thin bonding, providing an ideal adhesive connection in between metal layer and the top coat.

The organic-metallic precursors of the ³³ desired ceramic oxides can be combined, dissolved in a specific solvent and hydrolyzed into the sol and eventually a gel, which makes the structure highly controllable. This can be used for low-temperature sintering, typically 200-600°C, which can manufacture good quality coatings using a clear, cost-effective and efficient process. While sol-gel is advantageous, due to certain shortcomings, such as low connectivity, low wear resistance, high permeability and difficult porosity regulation, sol-gel technics cannot reach their maximum industrial potential. In fact, where the ⁴⁹ crack-free property is an important condition, the max film thickness limit is 0.5 μm.

2.2.8 Blade Coating

³⁸ Blade coating is a method of manufacturing for the production of large-area films on rigid or flexible substrates such as knife coating or doctor blade. The well-defined density is influenced primarily by the distance between the blade and the surface. The blade is pushed around a ⁴⁴ flat surface for laboratory-scale testing. The blade is attached to the shifting substratum ¹¹¹ for large-scale R2R processes. The ink is held in front of the razor, which is relatively moving towards the blade. Adjustable space widths allow variable wet layer thicknesses to be deposited. Based on the cover speed and flow activity, ⁴⁴ the final wet layer thickness is approximately half the distance width^[37]. ³⁸ The atomic structure of the substrate, interfacial tension of liquid, speed of covering, viscosity and surface temperatures shape other coating parameters that affect film forming. The analytical partnership is able to measure the thickness of the dry coating, d,

$$d = \frac{1}{2} \times g \times \frac{c}{\rho} \quad (2.2)$$

⁸ Where, g is the gap width, c the concentration of the solids in the ink in g/cm³, and ρ the density of the material in the final film in g/cm³ [38].

⁸³ 2.2.9 Chemical Vapour Deposition

Chemical vapour deposition (CVD) belongs to the methods familiar by which a solid substance is deposited in or above a naturally heated substratum by a vapor by a chemical ³⁶ reaction. A thin coating, powder, or single crystal is the resulting form of solid substance. ³⁶ Materials with a differing number of diverse physical, tribological and chemical characteristics may be grown through varying control parameters, which include

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electrode materials, substrate temperature and reaction gas mixture quality, total pressure gas flows, etc. An advantage of the CVD technique is its high throwing strength, which makes it possible to produce evenly-formed substrates, coating with uniform thickness and characteristics with low porosity. The ability to locate or selectively deposit on shaped substrates is another significant attribute.

In several thin film implementations, CVDs and relevant systems are employed, which include dielectrics, leads, passivation coating, obstacles to oxidation, conductive oxides, tribological and Corrosion-resistant layering, heat-resistant coating and microelectron epitaxial layers. Other CVD uses include the processing of tungsten and ceramic materials, and the manufacture of solar cells, high-temperature fiber composites and well defined-size particles. 17
Recently, this method has also been used to manufacture high Tc superconductors and, most recently, carbon nanotubes [39]. Because during deposition accurate vapor oxygen behavior can be monitored, no oxygen reinforcement is necessary for superconductivity.

Many CVD processes are available. The deposition is started and sustained by heat in thermally activated CVD (TACVD). Even though, photons, electrons and ions will cause and sustain CVD reactions as well as the mixture (plasma-activated CVD) [40].

2.3 Principle of Electrophoresis

Electrophoresis refers to electrical field along with phoresis, which results in migration [41]. electrophoresis is a separation technology by which ions molecules move to the applied electrical champ with differential speeds. The atoms move in the direction of the opposite electrode. These molecules migrate towards the cathode with a positive ions and the negatives move towards Anode. Migration is because of the load on molecules and the applied potentials through electrodes, the speed of movement depends on the net load, weight, height, form and electrical current of the substances. As power is used, the molecules move to the corresponding electrodes. The higher molecular weight molecules move more slowly. While they run quicker with limited mass. In addition, activity also has an impact on its scale. The large molecule has greater friction than smaller molecules. Dependent on their charge, weight and shape, these molecules move at varying speeds and lengths. The electrophoresis theory is shown in Fig. 2.3

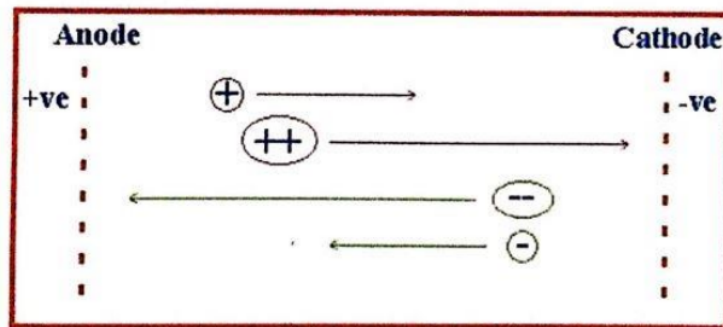


Fig. 2.3 Principle of Electrophoresis

2.4 Introduction to Electrophoretic Deposition

The concept electrophoretic deposition (EPD) involves electrical coating, cathodic electrodeposition, anodic electrodeposition and electrophoretic coated or electrophoretic coating. A distinctive characteristic of this mechanism is that, in a fluid medium suspended, colloidal particles relocate and store on an electrode underneath the effect of an electrical field (electrophoresis). Electrophorous deposition should be used with all colloidal particles and can produce better suspension and bear charges. Materials like polymers, pigments, dyes, pottery and metals include this. This technique is beneficial for application of materials on a conductive substrate. In the real manufacturing environments and facilities that can be used, the components that are placed are the main determinant.

Since electrophorus painting processes in many industries have been widely employed, aqueous EPD is the most commonly used commercially. However, applications for non-aquatic electrophoretic filing are also very popular now. Non-aquatic EPD applications for use in electronic device production and ceramic coating application are currently being examined. Non-aqueous processes have the advantage of avoiding the electrolysis of water and the oxygen evolution which accompanies electrolysis.

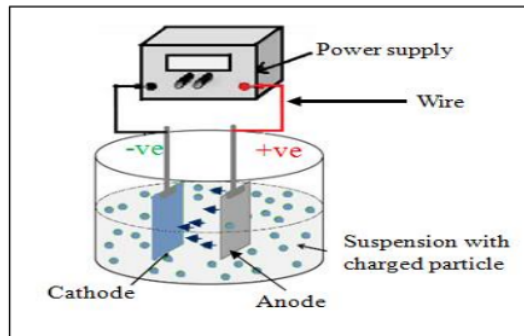


Fig.2.4 Principle of EPD

Film deposition is the method used to apply a film to the surface of a substrate or to create layers on a previously deposited cover. Film deposition EPD is one of the most prominent methods in colloidal deposition in which colloidal particles immersed in a fluid solution are moved and deposited on an electrode by means of an electrophoresis principle. EPD has benefits of shorter formation time, requires simple devices, no substrate shapes limit, no binder burnout requirement, since the green layer includes very little organics. The EPD method is very flexible compared to other specialized forming methods because it can be quickly changed for a particular use. For eg, deposition may be performed with only small changes in design and positioning on a smooth, cylindrical or some other shaped substrate. In particular, while the wet method is a simple modification of deposition time and applied potential, the EPD provides easy control of the thickness and morphology of a stored movie. Dispersed in or suspended in a liquid medium. Charged particles of powder are attracted in EPD and deposited in the presence of a DC electric field on conductive substrate of opposite charges.

2.4.1 Factors influencing EPD

The EPD system includes charged particles which are stored under the control of the applied electrical field in a suspension to an electrode. There are mainly two types parameters; i) the suspension parameters, and ii) the process parameters which can determine the characteristics of this process, including those relating to the physical parameters such as the electrical behavior and electrical properties of the electrodes.

2.4.1.1 Parameters related to the suspension

The parameters related to the suspension are:

- I. Particle size
- II. Dielectric constant of liquid
- III. Conductivity of suspension
- IV. Viscosity of suspension
- V. Zeta potential
- VI. Stability of suspension

i. Particle size

While there is no universal thumb rule to determine electrophorus particle sizes, there are strong deposits for a number of ceramic and clay systems within the range 1-20 μm [55]. This does not actually imply; however, it is not possible to deposition particles outside this size limit. For smooth and uniform deposition, the particles must stay totally dispersed and stable. Furthermore, a high surface charge must be achieved in order to achieve electrophoretic deposition of larger particles otherwise the electric double layer area must expand over time. It was also observed that particle size had an influential effect during drying on the cracking of the deposit.

ii. Dielectric constant of liquid

In several organic media, power [44] studied beta-alumina suspensions and calculated the probability of deposition in accordance with the dielectric fluid standard and fluid conductivity. The conductivity has been significantly increased with the dielectric constant, evidently referring to the fluid in its purest state. It is also worth noting that impurities, especially water, affect conductivity and that, as a result of dissociative or adsorbent modes, conductivity for milled suspension is very different from that of pure liquid. Powers were provided with only liquid deposits for which the dielectric constant ranged from 12 to 25. Deposition fails due to inadequate dissociative strength, with an excessively low dielectric constant, and the high ionic concentration in the liquid decreases the size of the double-layered area and hence the electro mobility.

iii. Conductivity of suspension

In the experiments with the EPD the conductivity of the suspension was suggested by Ferrari and Moreno[30]. It was observed that the particle movement is too poor when the suspension becomes too conductive, and if the suspension is too resistive the particles electronically charge and equilibrium is lost. They discovered the presence of a small conductivity band where the layer is formed at a differing dispersant quantity and temperature. Leads from this area do not qualify for EPD and restrict the opportunities for formation. However, for the various systems, this suitable range of conductivity should be different.

iv. Viscosity of suspension

In casting processes, the main controlling parameter is the viscosity. Rheological measurements on concentrated slips give us a good idea about the optimum dispersing state when adding dispersants. In EPD process, the solid loading is very low and the viscosity cannot be used to evaluate the dispersion state [45,46]. But the desired properties in the suspension vehicle are low viscosity, high dielectric constant and low conductivity.

v. Zeta potential

Throughout the electrophoretic deposition process, the zeta potential of molecules is significant. A strong and consistent surface load of the suspended particles must be achieved. This plays a major role in: a) stability of its suspension by defining the rate of repulsive particle interaction; b) determination of the path and momentum of EPD movement of particles; c) determination of its green repository density. The overall structure performance involves the presence of discrete suspension particles. The contact caused by electrostatic forces and Van der Waals is affected by two factors.

vi. Stability of suspension

Because of the brownian movement colloidal particles with average diameter of 1 μm or less appear to remain suspended for long periods. Particles bigger than 1 μm must be suspended with constant hydrodynamic perturbation. Settling rate and the inclination to endure or prevent flocculation are characteristic of suspension stabilization. There is no

inclination for static suspensions to flocculate, settle gradually and form thick deposits that adhere closely to the reservoir base. Suspension flakes fit quickly and form low volume deposits which adhere weakly. Is if suspension becomes too consolidated, there seems to be no electric field to counteract the electrostatic repulsion between the molecules and no deposition. The suspension should be fragile in the region of the electrodes according to a few electrophoretic deposition models [49].

2.4.1.2 Parameters related to the process

The parameters related to the process are:

- i. Effect of deposition time
- ii. Applied voltage
- iii. Concentration of solid in suspension
- iv. Effect of deposition distance

- i. Effect of deposition time

Basu et al.[47] observed that deposition rates reduced with increasing or extended time for a fixed field. Chen and Liu made similar observations[48]. During the preliminary deposition period the deposition is linear. However as time is spent decreasing and a plateau reaches very high deposition times, the deposition rate drops. At steady EPD voltage, this is supposed to occur because even though the possible variation of electrode levels continues, with the forming of an insulating outer coating of ceramic particles on the electrode surface the electrophoresis influential field declines with deposition time [50].

However, there is usually a significant correlation between deposition mass and time throughout the initial phase of the EPD. Even if the deposition period rises, the particles in the material stored decrease over time for the continuous electrophoresis. This reduces the velocity of the electron and the deposition current. Sarker and Nicholson used EPD kinetics with $m(t)$, m_0 as the original mass, t as the time of deposition, μ described the time scale features [68], Eq. (2.3) as the EPD kinetics.

$$m(t) = m_0(1 - e^{-t/\tau}) \quad (2.3)$$

ii. Applied voltage

The deposit balance normally increases and the applicable opportunity is increased. Basu et al. [47] find that more uniform films are placed in modest fields of application (25–100 V/cm) while film quality decreases if comparatively higher fields are applied (>100 V/cm). Due to the fact that particulate film forming on the electrode is a kinetic process, the particle aggregation rate determines the packaging behaviour in the coating. The coating can be disrupted by the fluxes in the ambient material and during its deposition for a higher area that can result in turbulence in the spring. Furthermore, molecules can move so quickly that they cannot have sufficient time for a tight-packed system to remain in its best places. Finally, laterally mobility if deposited on the surface of the already deposited layer in high field situations is reduced, since higher applicable potential exhibits greater velocity on particular flow and movement, the force implemented affects the deposit rate and the deposit structure.

iii. Concentration of solid in suspension

The solid particle size plays a vital role in the solution, notably for EPDs. While each particle categories have the identical substrate charge sign, they can (based on the size fraction of solids in the suspension) deposit at various rates. In several cases, the powder deposits are equal while the particle size of solids remains high. However, is if volume of solids is small, the particles may be deposited at speeds corresponding to their respective mobility [51].

iv. Effect of deposition distances

Deposition distance means the inter-electrode distance or the distance between the two substrates. Deposition amount for a fixed applied field decreases with increased deposition distance. When the deposition distance is decreased, more particles are deposited in the substrates. When the deposition distance is increased, fewer particles are deposited in the substrates. Shen-Chang Lin et al. showed when deposition distance increases, it increases possibility to collide each other which led to deceleration of deposition rate [67].

2.4.1.3 Other Influencing Parameters

It's been seen that there are some other parameters which can influence the Deposition quantity, quality and speed. Hasanpoor et al. has showed with the increment of the concentration of the nanoparticles, the number of the charged particle increases which results in the increment of the current density [66]. This influences the thickness formed on the substrate. They also showed that the type of alcohol also can influence the deposition result. B. Ferrari et al. has founded that the surface of substrate also has influence on the Electrophoretic Deposition. Polarizing electrodes or blocking elements have a significant interface barrier, which means that the voltage gradients in the suspension are lower than anticipated, resulting in an important potential decrease near electrodes (e.g. activation overvoltage, concentration overvoltage) [63-65].

2.4.2 Types of EPD

There are two major categories of EPD processes:

- (i) Anodic EPD.
- (ii) Cathodic EPD.

Anion materials are collected during the anodic procedure on the positive charged electrode or anode. Cation material is placed on the negative terminal or cathode during cathodic phase. Both charged organisms move by electrical field to the electrode at the opposite polarity by the mechanism of electrophoresis. Both methods are still in commercial usage, even though the anodic process is often used for a prolonged period of time industrially and is thus deemed the oldest of both methods. For all kinds of systems, there are benefits and drawbacks and different specialists have positive and negative aspects. Cathodic electrophoretic deposition is carried out in this study.

The main benefits for the anodic mechanism are normally:

1. Lower costs compared to cathodic process.
2. Simpler and less complex control requirements.
3. Fewer problems with inhibition of cure of subsequent top coating layers.
4. Less sensitivity to variations in substrate quality.

5. The substrate is not subjected to highly alkaline conditions, which may dissolve phosphate and other conversion coatings.

⁴ The major advantages that are normally touted for the cathodic processes are:

1. Higher levels of corrosion protection are possible.
2. ⁸¹ Higher throw power can be designed into the product.
3. Oxidation happens mostly in the anode and so staining and other issues created by the ⁸⁹oxidation of the electrode substrate itself would be eliminated in the cathode phase.

2.4.3 Application of EPD

Any material that is usable as a small particle (<30 μm) or colloidal suspension can be used for electrophoretic deposition. Electrophoretic material deposition illustrations of nearly any material type, such as metals, oxides, polymers, carbides, nitrides and glasses, can be found. The method could also ²¹be used to produce clothing, to shape monolithic, laminate and graded stand-alone products, and to infiltrate pore materials and actually perform woven fibers for the manufacture of composites.

2.4.3.1 Coatings

In the field of automobile, devices and general industrial (organic) composites, electric deposition of the coatings already has achieved global recognition. The benefits that have resulted in use of electrophorous deposition are high automation, low emission levels, high throws and the uniformity of the coating. Additional benefits in comparison to the submerged or sprayed coatings are an increased adherence to the electrophorus deposits.

2.4.3.2 Shaping Free-Standing Objects

2.4.3.2.1 Monoliths

The real benefit of electrically-powered deposition is the ²¹higher speed and the low wear of moulds for standard ceramics, such as bathtubs, tiles, basin relative to slip casting [52]. As complex shapes can be covered with EPD, they can also be used to form objects. Tiles, enclosed and open-end pipes, hemispheres, diameter changed tubes and triangular parts are among some of the forms made with solid compounds usually for table and sanitary ware [52,53], but more on a lab scale.

2.4.3.2.2 Laminated Materials

EPD may also be used to manufacture substrated materials. The deposit on electrode can be transferred into a second suspension for deposition of a substrate of various compositions until the desired thickness of the initial layer is achieved. A layered content is easily modified by varying back and forth. ZrO₂/Al₂O₃ substrates with alumina coating thin as 12 μm and zirconia layers 2 μm were developed in ethanol-based solution by Nicholson et al. [54]. The borders in between layers were smooth and well defined, which shows that, while EPD can be a fast operation, the growth of the layers can be monitored properly.

2.4.3.2.3 Graded Materials

Though coated materials are collected by dipping the deposit electrode in separate bathrooms, the structure of the EPD suspension under which the material is performed can still be slowly transformed. Sarkar et al. [55] has shown the potential to shape graduated content by applying an alumina powder ethanol suspension of a yttria-stabilized zirconia powder progressively to an ethanol suspension during deposition. There has been a steady rise in the deposited aluminium content.

2.4.3.2.4 Unique microstructures and selected deposition

EPD does not yet have the full ability to provide specific microstructural functionality. In the manufacturing of non-planar laminates, Nicholson et al. [56] did several exploratory work; they were able to manufacture laminates with wavelengths by inserting a grid before the deposition of electrode. Thus, deposition can also be improved locally with the use of secondary electrodes. As a source of boron diffusion, Scala & Sandor [57] deposited B₂O₃ on silicon chips. By developing a silica lm on the wafer and removing silica just where the boron has to be deposited, the accumulation on the whole silicone wave was avoided.

2.4.3.3 Infiltration

The strength that electrophoretic deposition which can be used for infiltration or the application of an intrinsic layer in objects by composite materials. Gal-Or et al. [58]

invaded silicon carbide and silicon oxide crystal porous graphite surfaces. The molecules building in an outer layer should be reduced for effective penetration in a way that pores are not covered. The penetration was increased by ⁴⁶ lower applied voltages and a high-particle suspension density.

The penetration of fiber preform with polymer matrix for composite processing is another use of electrophoretic deposition. The preform is directly used for conducting fibers as the deposition device, while the preform is located before an electrode on non-conducting filaments. The benefit of electrophoretic deposition in the production of composites is that the cost can be reduced. In addition, electrophorus deposition is much quicker and matrix structure can be easily managed.

2.5 Literature Review on EPD

⁸ Davey and General Electric were granted the first patent for the use of electrophoretic painting in 1917. The method for the accumulation of rubber latex has been used since 1920s. The first patents describing the foundation of water dispersible water solvents explicitly developed for EPD were published in the 1930's. Electrophorus coating started to take its present form in the end of the 1950s, after the creation of the automotive coating process started by ⁴ Dr. George E.F. Brewer and the Ford Motor Company staff. In 1963 started operation of the first private, anodic car system.

In 1965 BASF AG was granted the first copyright for a Cathodium EPD device. The very first commercially cathodic EPD was introduced by PPG Industries, Inc. in 1970. In the automobile industry, first ever cathodic EPD was used in 1975. Catholic EPD form today accounts for around 70% of the amount of EPD in use worldwide, primarily because of high use of automobile manufacturing. It is perhaps the best device ever built and has led to a huge increase in the life of the car industry.

More than thousands of patents for the different EPD formulations, EPD processes and EPD-coated products have been published. Patents granted by several federal Patent Authorities, but ⁴ the U.S. Patent and Trademark Office's reviews of patents will accompany nearly all major inventions.

Zinc oxide (ZnO) has been used for Electrophoretic deposition for many applications like DSSC solar cell, nano tube and many more. C S Sebastian et al. carried out the Electrophoretic Deposition on the graphite drawn paper, which is the conductive surface

for the procedure [43]. They mixed acetone with the grounded ZnO and applied 300V to 500V for the deposition for 3 min. Nayeem Ansari et al. processed ZnO suspension for EPD with ZnO nanoparticles and acetylacetone in ethanol [42]. Where the acetylacetone used so that it can break the aggregates. To charge the suspension water (H₂O) and acetone were added and dispersed with homogenizer.

Electrophoretic deposition of TiO₂ has been performed by various researchers. A. Zaban et al. used Ethanol as organic liquid, TiO₂ (P25) as Precursor and Acetone, De-ionized water and Iodine as particle charger [59]. Wirat Jarernboon et al. used Acetylacetone as organic liquid, TiO₂ (P25) as Precursor and Iodine as particle charger [60]. Hsin-Wei Chen et al. used Isopropyl Alcohol as organic liquid, TiO₂ (P25) as Precursor and Acetone and De-ionized water as particle charger [61]. Jason Bandy et al. used Isopropyl Alcohol as organic liquid, TiO₂ (P25) as Precursor and De-ionized water and Magnesium Nitrate, Hexahydrate as particle charger [62]. In this investigation, Ethanol is used as organic liquid, TiO₂ (P25) is used as Precursor and De-ionized water is used as particle charger.

CHAPTER 3

EXPERIMENTAL

3.1 Introduction

Electrophoretic deposition (EPD) method involves the motion and deposition of charged particles in the presence of electric field onto a conductive electrode to develop thin or thick films and coatings. In the cathodic EPD, positive particles are attracted by cathode and deposit on the cathode (negative electrode); in the anodic EPD, negative particles are attracted by anode and deposit on the anode (positive electrode). The driving forces of EPD are particle charges and particle mobility's in aqueous media under an electric field. A comprehensive description of the materials which are used and the procedure used to complete the electrophoretic deposition of ZnO are briefed in this chapter.

3.2 Material Used

- a. Zinc Oxide
- b. Distilled Water
- c. Ethanol
- d. FTO coated glass substrate
- e. Clipping Arrangement
- f. Plastic Jar
- g. DC power supply
- h. Multimeter
- i. 4-digit digital analytical balance
- j. Electric blender
- k. stopwatch
- l. Hair dryer

3.2.1 Zinc Oxide (ZnO)

ZnO has a relatively large direct bandgap of ~ 3.3 eV at room temperature and an exciton binding energy of 60 meV and is capable of being used extensively on nanoscale [76]. There are applications for ZnO nanostructures in environmental, technological and biomedical purposes including dye-sensitised solar cells, lithium-ion batteries, biosensors, nanolasers and supercapacitors [77]. The benefits are related to a

large bandgap ¹⁵ include higher breakdown voltages, the ability to sustain large electric fields, lower electronic noise, and high-temperature and high-power operation. The bandgap of ZnO can further be tuned to 3~4 eV by its alloying with magnesium oxide or cadmium oxide. Electrical properties can be modified by controlling the doping standard ³⁴ from insulator through an n-type semiconductor to metal while maintaining optical transparency that makes it useful for transparent electrodes in flat-panel displays and solar cells [78].



Fig. 3.1 Zinc Oxide

3.2.2 Distilled Water

Electricity is ¹¹improperly conducted by pure water. Water with certain chemical components or elements in it at varying amounts including sodium, magnesium, calcium and chloride, is a better conductor of electricity. Generally speaking, as water salinity increases, conductivity increases alongside it. Warmer water has a higher conductivity than cooler water. The toxins of distilled water have been extracted by boiling it. it is started to captured as steam, ¹¹ it is captured as steam, and minerals and other compounds are left behind. Once the steam is cooled, it reverts to distilled water [71]. Electricity does not conduct well by pure water. ²⁶ Ordinary distilled water in equilibrium with carbon dioxide of the air has a conductivity of about $10 \times 10^{-6} \text{ W}^{-1} \cdot \text{m}^{-1}$ (20 dS/m). Since the ions carry the electrical current in solution, If the ion concentration rises, so does conductivity. When water dissolves ionic species, conductivity increases [72]. In this experiment, distilled water was combined with the ZnO and ethanol solution before the electrophoretic deposition to charge the solution.

3.2.3 Ethanol

Ethanol (also called ethyl alcohol, grain alcohol, drinking alcohol, or simply alcohol) is a chemical element that is organic in nature. It is simple alcohol with the chemical formula C_2H_6O . Ethanol is naturally produced by the fermentation of sugars by yeast or via petrochemical processes naturally produces ethanol such as ethylene hydration. Ethanol is a multipurpose solvent, miscible with water and variety of organic solvents, including acetic acid, acetone, benzene, carbon tetrachloride, chloroform, diethyl ether, ethylene glycol, glycerol, nitromethane, pyridine, and toluene [73]. Ethanol is an important commercial chemical used in alcoholic beverages, which may contain up to 50% ethanol. It is also used as a solvent in cleaners and as a fuel additive. Ethanol is used in the production of other chemicals, pharmaceuticals, perfumes, and cosmetics [74]. There are two forms of ethanol binding (Polar and nonpolar). One is the nonpolar bond that exists between the molecule of carbon and hydrogen (C-H) and another is a polar bond that is visible between molecule of Carbon and molecule of Oxygen atoms (C-O) and Oxygen and Hydrogen atoms (O-H). The polarity of a bond depends on factors like dipole moment, electronegativity and symmetry. There exists covalent bonding in the molecule of ethanol. There is a strong covalent bond between the atoms. The covalent strength of the alcohol bond is sufficient to prevent ionization in the presence of water. Therefore, in aqueous solutions, alcohol doesn't ionize. The C_2H_5OH molecule's covalent bond is sufficiently strong that atoms don't ionize. Thus, ethanol has no free or delocalized electron to flow into it and doesn't conduct electricity. One of the major reasons that alcohol fail to conduct electricity is because of this. [75].

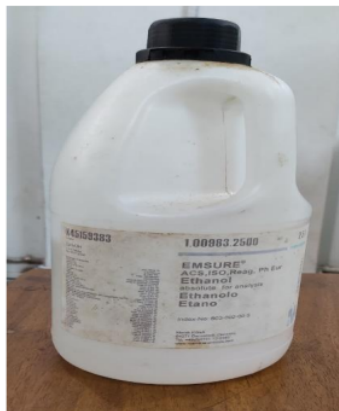


Fig. 3.2 Ethanol

3.2.4 FTO Coated Glass Substrate

Electrically conductive fluorine-doped tin oxide (FTO) coated glass is suitable for optoelectronics, touch screen displays, thin film photovoltaics, energy-saving windows, RFI/EMI shielding, and other electro-optical and insulating applications. Fluorine doped tin oxide has been considered as a potentially useful element due to its relatively stable under atmospheric conditions, chemically inert, mechanically hard, high-temperature resistant, has a high tolerance to physical abrasion and is less expensive than indium tin oxide. These FTO glass materials provide a wide range of thermal and heated glass performance properties, as well as improved light transmission and electrical conductivity. These glass substrates are obtainable in different of glass thicknesses and surface resistivity ranging from $7 \Omega/\text{sq.}$ up to $13 \Omega/\text{sq.}$ [70]

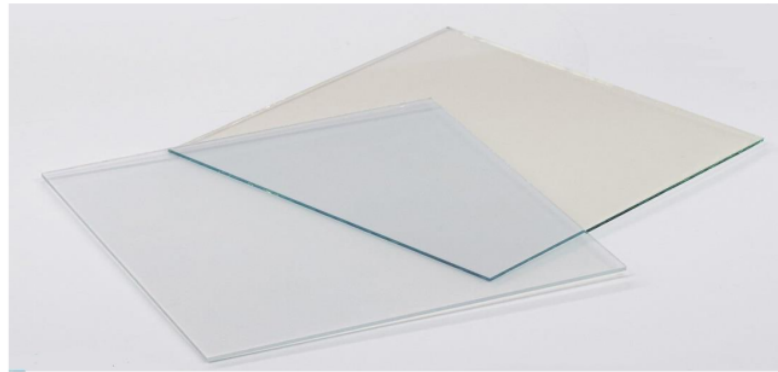


Fig.3.3 FTO Coated Glass Substrate

3.2.5 Clipping Arrangement

Clipping arrangement is used for holding the glass substrate. In the clipping arrangement, two crocodile clips and one paper clip were used. At first, both glass substrates are set face to face in the crocodile clip then the crocodile clip is set in paper clip and the distance between glass substrate is measured by scale and fixed at desire value. The distance is varied by putting nonconductive element between the crocodile clip. The crocodile clips were wrapped with rubber for avoiding short circuit between crocodile clips and paper clips .it also checked that is there any contact between two crocodile clip which is responsible for short circuit. After confirming no short circuit between two glass

substrate next step was done. The initial distance between the substrate was 0.8 cm. The distance is varied 0.8 to 1.6 during the deposition process.



Fig. 3.4 Clipping Arrangement

3.2.6 Plastic Jar

A plastic jar is needed for doing the deposition process. The size of plastic jar selected depending on the clipping arrangement and amount of solution. The size of the jar and the amount of solution such that glass substrate is immersed into the solution. The plastic jar should have a cover so that solution can be preserved for future work.



Fig. 3.5 Plastic Jar

3.2.7 DC power supply

Three DC power supply were used to apply deposition voltage. Each DC power supply can provide up to 32 V. These DC power supplies were connected in series to provide up to 90V. Before every observation deposition voltage is measured by multimeter. After confirming the deposition voltage, the deposition process was started.



Fig. 3.6 DC power supply

3.2.8 Multimeter

Multimeter was used for following tasks

- i. **To determine conductive and nonconductive surface of glass substrate:** At first multimeter was set to buzzing mode. Then positive and negative wire of multimeter was connected to same surface of the glass substrate if the surface is conductive buzzer buzzed if not buzzer didn't buzz.
- ii. **To measure deposition voltage:** Before beginning the deposition process, the deposition voltage was measured with a multimeter to determine the desired deposition voltage.
- iii. **To measure deposition current:** Deposition current is measured by multimeter when deposition process was started. For this purpose, the multimeter was connected in series between the anode and the V_{cc} of the power supplies.



Fig. 3.7 Multimeter

3.2.9 4-digit digital analytical balance

The Analytical balances are used to calculate small amounts of mass in the sub-milligram scale. It can quantify mass from 0.1 mg to 200 g. It has a ⁸⁵Stainless Steel Pan, an ⁷⁸Super bright LCD display with backlight, a high-quality glass windshield, Height adjustable feet, and a levels display in the back. It has Selectable different measurement units to choose from: 'mg', 'g', 'oz', and 'ct' [69]. Before and after the deposition, the mass ¹of the glass substrate was measured using the analytical balance. The mass of the deposition layer was calculated using the difference between these two masses.



Fig. 3.8 4-digit digital analytical balance

3.2.10 Electric blender

An electric blender was used to disperse the solution properly and equally. For this NOWAKE N-999 electric blender was used. This blender has five different speed. Homogenizers are commonly used in modern laboratories to distribute solutions. Since a homogenizer was not available in the laboratory, an electric hand mixer was used in this experiment as a replacement. Hand mixers, on the other hand, are not as effective as homogenizers. To compensate for this shortcoming, the solution was dispersed for 30 minutes when a 10-minute homogenizer dispersion would be sufficient.



Fig. 3.9 Electric Blender

3.2.11 Stopwatch

The stopwatch is needed to measure the deposition time. Stopwatch and power supply were turned on at the same time when deposition is started and also turn off at the same time when the desired deposition time is over.

3.2.12 Hairdryer

The glass substrates must be washed and dried with nitrogen gas before deposition. As Nitrogen gas wasn't available a hairdryer is used instead of Nitrogen gas.



Fig.3.10 Hair Dryer

3.3 Experimental setup

The full experimental set up is shown in Fig. 3.11. From Fig. 3.11 it is seen that, there is ZnO, ethanol, distilled water, the clipping arrangement, power supply, multimeter. Three power supplies were connected in series. The V_{cc} of the third power supply was connected to the anodic glass substrate and the ground of the first power supply was connected to the cathodic glass substrate. A multimeter was connected between the V_{cc} of the power supply and the anodic glass substrate to measure the deposition current. The analytical balance was used to measure the mass of the deposition layer. Stopwatch was used to maintain the deposition time.

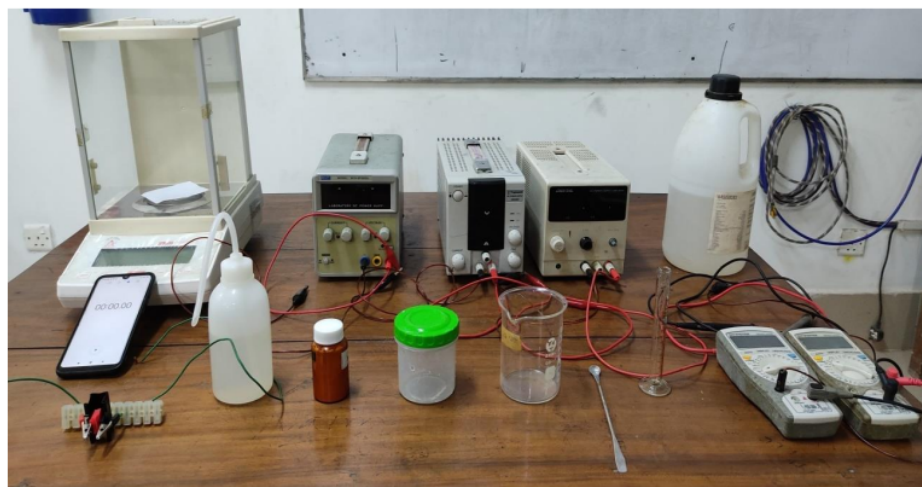


Fig. 3.11 Experimental Setup

3.4 Methodology

Total experiment was done by following steps,

Step-1: At first the glass substrates were cleaned by ethanol and distilled water. Then these cleaned and wet substrate was dried by hot air which was provided by hair dryer.

Step-2: A Multi meter was used for determining the conductive surface of the glass substrate. In case of the conductive surface the multimeter showed some resistance and the buzzer of the multi meter beeped. In case of the non-conductive side the multimeter showed infinite resistance and the buzzer didn't beep.

Step-3: The analytical balance was set in level by twirling the adjusting feet. A bubble indicator positioned at back side of the analytical balance indicates whether the balance is on level or not. Then switching ON ac power supply, the "ON" button is pressed to turn ON the digital analytical balance. After turning ON the analytical balance the LCD display showed some fractional values without any object on the balance pan. Then the value Shown in the LCD display is set to zero by pushing the 'TARE' button.

Step-4: Then the mass of the cathodic glass substrate was measured. The resultant mass was noted down and was indicated by W_1 . There is another way with which this step can be performed without noting down the Value of W_1 . For that the analytical balance is calibrated to zero by using the "Tare" button keeping the glass substrate on the pan of analytical balance. Then the glass substrate can be taken out from the pan without noting down the value. After completing the deposition process the glass substrate is put again in the pan of the analytical balance and the mass of the deposition layer or the deposition amount is found without any calculation.

Step-5: A clipping arrangement was made by a paper clip and two crocodile clips to hold the anodic and cathodic glass substrates at a fixed distance. The weighted glass substrate was set face to face at a distance of 1.2 cm by using this arrangement.

Step-6: 0.500g ZnO were measured in analytical balance. Measured ZnO and measured 80ml ethanol is taken in the jar. Then the solution was dispersed by electric blender for 20 minutes. 8 ml of purified water is then applied to the solution to charge it. It was then dispersed for another 10 minutes by a hand mixer.

Step-7: After that the DC power supply was turned ON and voltage is measured by multimeter. After setting the desired deposition voltage the power supplies were turned off for a while.

Step-8: The weighted glass substrate which was used as cathode was attached to the ground of the power supply. The other substrate which was used as anode was attached to the V_{cc} of the power supply. Another multimeter was connected in series between the V_{cc} of the dc power supply and the anodic glass substrate to determine the deposition current.

Step-9: The solution of the plastic container then had a part of negative and positive electrode. The entire substratum was ensured that the mixture did not drop and the retaining clips stayed just above solution.

Step-10: Then the power supplies and stopwatch were turned ON at the same time. Then the deposition current was measured by the multimeter which was connected in series with the power supply. The value of the deposition current was noted down. The power supply and stopwatch were turned off at the same time when desired deposition time is over. The time between turning ON and turning off the power supply is called deposition time.

Step-11: Then the substrates were picked off from the solution After turning off the power supply. A coating of ZnO nanoparticles were found on the cathodic glass substrate. This layer is called deposition layer.

Step-12: The substrate, in which deposition layer was found, was pulled out from the clipping arrangement. Then it was dried by the hairdryer.

Step-13: The dried glass substrate's mass was measured by the systematic balance. This mass was noted down and was indicated by W_2 .

Step-14: The mass of the deposition layer was found from the difference of W_2 and W_1 . If the analytical balance is calibrated to zero by using the "Tare" button keeping the glass substrate on the systematic pan before the deposition process the mass of the deposition layer can be found directly and no calculation is required.

Step- 15: Steps 1-14 were performed-

- i) By maintaining the deposition voltage and deposition time constant, varying the deposition distance.
- ii) By maintaining the deposition distance and deposition time constant, varying the deposition voltage.

- iii) By maintaining the deposition distance and deposition voltage constant, varying the deposition time

3.5 Formula for calculation of the mass of the deposition layer

For every observation separate substrate was used. The sizes of the all substrates were not same. All the substrates were partially immersed on the solution and immersed portion of all substrate was not same. Because of these reasons, the area where the deposition layer took place on each substrate was not fixed. So, deposition layers of some substrates are large and deposition layer of some are small.

The deposition level has been calculated in mass per unit area to solve this challenge. The deposition layer mass was divided by the deposition layer area. The forms of the layers of deposition is rectangular.

At first, the mass of the deposition layer is measured in grams. Then the length and width of the deposition layer is measured in centimeter.

Let,

The mass of the glass substrate before deposition = W_1

The mass of the glass substrate after deposition = W_2

So, the mass of the deposition layer is $W = W_2 - W_1$

The width of the deposition Layer = w cm.

The area of the deposition Layer, $A = l \times w$ cm²

So, Deposition Amount per area = $\frac{W}{A} = \frac{W_2 - W_1}{l \times w}$ gcm⁻² (i)

CHAPTER 4⁵⁶

RESULTS & DISCUSSION

4.1 Introduction

This chapter is divided into two segments. The first segment contains the calculation and results from the analysis of Electrophoretic Deposition of ZnO on the FTO type glass substrate based on three parameters. In the second segment, the results of the ZnO will be compared with the TiO₂. The objective of this chapter is to give an overview of the calculation and analysis of the results.⁶⁸

4.2 Calculation and result analysis of Electrophoretic Deposition of ZnO

The three parameters of deposition voltage, deposition time and distance in between substrates were varied as defined prior to the deposition process. The gap in between substrates was initially varying, maintaining the voltage and deposition time fixed. The deposition voltage was then varied to maintain a certain difference in between substrates and the time of deposition. The deposition time was then varying and the distance between the substrates and the deposition voltage was set.

4.2.1 Calculation of deposition amount

Deposition amount is calculated by using the formula of calculation of deposition amount given in section 3.5. At first, the values of the mass of the glass substrates before and after the deposition and value of the length and width of the deposition layer are measured and noted down. Finally, putting the value in equation (i) deposition amount per unit area is calculated.⁶⁶

4.2.1.1 Calculation of deposition amount for deposition distance variation¹

Here, deposition amount is calculated when the deposition distance (D) was varied; the deposition voltage (V) and deposition time (T) were kept fixed.

1) When, D = 0.8 cm; V = 70V; T= 60 s:

$$W_1 = 1.7801$$

$$W_2 = 1.7832$$

$$W = W_2 - W_1 = 1.7832 - 1.7801 = 0.0031\text{g}$$

$$l = 1.5\text{ cm}$$

$$w = 1.9 \text{ cm}$$

$$A = l \times w = 2.85 \text{ cm}^2$$

$$\text{Deposition Amount per area} = \frac{W}{A} = \frac{0.0031}{2.85} = 0.001088 \text{ g/cm}^2$$

2) When, $D = 1 \text{ cm}$, $V = 70 \text{ V}$, $T = 60 \text{ s}$:

$$W_1 = 1.6637$$

$$W_2 = 1.6670$$

$$W = W_2 - W_1 = 1.6670 - 1.6637 = 0.0033 \text{ g}$$

$$l = 1.5 \text{ cm}$$

$$w = 1.9 \text{ cm}$$

$$A = l \times w = 2.85 \text{ cm}^2$$

$$\text{Deposition Amount per area} = \frac{W}{A} = \frac{0.0033}{2.85} = 0.001158 \text{ g/cm}^2$$

3) When, $D = 1.2 \text{ cm}$, $V = 70 \text{ V}$, $T = 60 \text{ s}$:

$$W_1 = 1.9439$$

$$W_2 = 1.9474$$

$$W = W_2 - W_1 = 1.9474 - 1.9439 = 0.0035 \text{ g}$$

$$l = 1.6 \text{ cm}$$

$$w = 1.7 \text{ cm}$$

$$A = l \times w = 2.74 \text{ cm}^2$$

$$\text{Deposition Amount per area} = \frac{W}{A} = \frac{0.0035}{2.74} = 0.001277 \text{ g/cm}^2$$

4) When, $D = 1.4 \text{ cm}$, $V = 70 \text{ V}$, $T = 60 \text{ s}$:

$$W_1 = 1.5141$$

$$W_2 = 1.5163$$

$$W = W_2 - W_1 = 1.5163 - 1.5141 = 0.0022 \text{ g}$$

$$l = 1.6 \text{ cm}$$

$$w = 1.6 \text{ cm}$$

$$A = l \times w = 2.56 \text{ cm}^2$$

$$\text{Deposition Amount per area} = \frac{W}{A} = \frac{0.0022}{2.56} = 0.000859 \text{ g/cm}^2$$

5) When, $D = 1.6 \text{ cm}$, $V = 70 \text{ V}$, $T = 60 \text{ s}$:

$$W_1 = 1.7840$$

$$W_2 = 1.7844$$

$$W = W_2 - W_1 = 1.7844 - 1.7840 = 0.0004\text{g}$$

$$l = 1.5\text{ cm}$$

$$w = 1.9\text{ cm}$$

$$A = l \times w = 2.85\text{ cm}^2$$

$$\text{Deposition Amount per area} = \frac{W}{A} = \frac{0.0004}{2.85} = 0.000140\text{ g/cm}^2.$$

4.2.1.2 Calculation of deposition amount for deposition voltage variation

Here, deposition amount is calculated when the deposition voltage (V) was varied; the distance between the substrates (D) and deposition time (T) were kept fixed.

1) When, V = 50 V; D = 1.2 cm; T = 60 s:

$$W_1 = 1.8798$$

$$W_2 = 1.8805$$

$$W = W_2 - W_1 = 1.8805 - 1.8798 = 0.0007\text{ g}$$

$$l = 1.4\text{ cm}$$

$$w = 1.9\text{ cm}$$

$$A = l \times w = 2.66\text{ cm}^2$$

$$\text{Deposition Amount per area} = \frac{W}{A} = \frac{0.0007}{2.66} = 0.000263\text{ g/cm}^2$$

2) When, V = 60 V, D = 1.2 cm, , T = 60 s:

$$W_1 = 1.8251$$

$$W_2 = 1.8271$$

$$W = W_2 - W_1 = 1.8271 - 1.8251 = 0.002\text{ g}$$

$$l = 1.5\text{ cm}$$

$$w = 1.9\text{ cm}$$

$$A = l \times w = 2.85\text{ cm}^2$$

$$\text{Deposition Amount per area} = \frac{W}{A} = \frac{0.002}{2.85} = 0.000702\text{ g/cm}^2$$

3) When, V = 70 V; D = 1.2 cm; T = 60 s:

$$W_1 = 1.9435$$

$$W_2 = 1.9455$$

$$W = W_2 - W_1 = 0.002\text{ g}$$

$$l = 1.4 \text{ cm}$$

$$w = 1.9 \text{ cm}$$

$$A = l \times w = 2.66 \text{ cm}^2$$

$$\text{Deposition Amount per area} = \frac{W}{A} = \frac{0.002}{2.66} = 0.000752 \text{ g/cm}^2$$

4) When, V = 80 V; D = 1.2 cm; T = 60 s:

$$W_1 = 1.7805$$

$$W_2 = 1.7834$$

$$W = W_2 - W_1 = 1.7834 - 1.7805 = 0.0029 \text{ g}$$

$$l = 1.5 \text{ cm}$$

$$w = 1.9 \text{ cm}$$

$$A = l \times w = 2.85 \text{ cm}^2$$

$$\text{Deposition Amount per area} = \frac{W}{A} = \frac{0.0029}{2.85} = 0.001018 \text{ g/cm}^2$$

5) When, V = 90 V; D = 1.2 cm; T = 60 s:

$$W_1 = 1.7888$$

$$W_2 = 1.7905$$

$$W = W_2 - W_1 = 0.0017 \text{ g}$$

$$l = 1.4 \text{ cm}$$

$$w = 1.9 \text{ cm}$$

$$A = l \times w = 2.66 \text{ cm}^2$$

$$\text{Deposition Amount per area} = \frac{W}{A} = \frac{0.0017}{2.66} = 0.000639 \text{ g/cm}^2$$

4.2.1.3 Calculation of deposition amount for deposition time variation

Here, deposition amount is calculated when the deposition time (T) was varied; the distance between the substrates (D) and deposition voltage (V) were kept fixed

1) When, T = 30 s; D = 1.2 cm; V = 70 V:

$$W_1 = 2.2166$$

$$W_2 = 2.2183$$

$$W = W_2 - W_1 = 2.2183 - 2.2166 = 0.0017 \text{ g}$$

$$l = 1.5 \text{ cm}$$

$$w = 1.9 \text{ cm}$$

$$A = l \times w = 2.85 \text{ cm}^2$$

$$\text{Deposition Amount per area} = \frac{W}{A} = \frac{0.0017}{2.85} = 0.000596 \text{ g/cm}^2$$

2) When, T= 40 s, D = 1.2 cm, V = 70 V:

$$W_1 = 1.8117$$

$$W_2 = 1.8143$$

$$W = W_2 - W_1 = 1.8143 - 1.8117 = 0.0026 \text{ g}$$

$$l = 1.5 \text{ cm}$$

$$w = 1.9 \text{ cm}$$

$$A = l \times w = 2.85 \text{ cm}^2$$

$$\text{Deposition Amount per area} = \frac{W}{A} = \frac{0.0026}{2.85} = 0.000912 \text{ g/cm}^2$$

3) When, T= 50 s; D = 1.2 cm; V = 70 V:

$$W_1 = 1.8101$$

$$W_2 = 1.8128$$

$$W = W_2 - W_1 = 1.8128 - 1.8101 = 0.0027 \text{ g}$$

$$l = 1.5 \text{ cm}$$

$$w = 1.9 \text{ cm}$$

$$A = l \times w = 2.85 \text{ cm}^2$$

$$\text{Deposition Amount per area} = \frac{W}{A} = \frac{0.0027}{2.85} = 0.000947 \text{ g/cm}^2$$

4) When, T= 60 s; D = 1.2 cm; V = 70 V:

$$W_1 = 1.7878$$

$$W_2 = 1.7908$$

$$W = W_2 - W_1 = 1.7908 - 1.7878 = 0.003 \text{ g}$$

$$l = 1.5 \text{ cm}$$

$$w = 1.9 \text{ cm}$$

$$A = l \times w = 2.85 \text{ cm}^2$$

$$\text{Deposition Amount per area} = \frac{W}{A} = \frac{0.003}{2.85} = 0.001053 \text{ g/cm}^2$$

5) When, T= 70 s; D = 1.2 cm; V = 70 V:

$$W_1 = 1.6670$$

$$W_2 = 1.6682$$

$$W = W_2 - W_1 = 1.6682 - 1.6670 = 0.0012 \text{ g}$$

$$l = 1.5 \text{ cm}$$

$$w = 1.9 \text{ cm}$$

$$A = l \times w = 2.85 \text{ cm}^2$$

$$\text{Deposition Amount per area} = \frac{W}{A} = \frac{0.0012}{2.85} = 0.000421 \text{ g/cm}^2$$

4.3 Results and analysis of variation of the three parameters

The results of the variation of the three deposition parameters and the analysis of those results are given in the following sections.

4.3.1 Variation of deposition distance

The deposition distance was varied between 0.8 and 1.6 cm. The deposition voltage and deposition time was fixed for 70 V and 60 s respectively. The results are shown in Table 4.1.

Table 4.1 Variation of deposition distance (D)

Deposition distance (cm)	0.8	1	1.2	1.4	1.6
Deposition amount (gcm ⁻²)	0.0031	0.0033	0.0035	0.0022	0.0004
Deposition Current (mA)	0.7	0.8	0.9	0.8	0.7

From Table 4.1, it can be observed that with the increment of the distance, current also increased proportionally but when the distance between the substrates greater than 1.2 cm the current started to drop proportionally.

The deposition current depends on various factor, one of those is the concentration of the solution. If the concentration of the solution increases deposition current increases and if the concentration of the solution decreases deposition current decreases. As in every process, ZnO particles were deposited in the glass substrates, the concentration of the solution decreases. As well when the distance between two substrates increases, the possibility of collision of the ZnO particles increases which could lead to the decrement of the deposition current. That is why deposition current was decreased with time and distance.

The distance vs. deposition amount curve is shown in Fig. 4.1 and the distance vs. deposition current curve is shown in Fig. 4.2. The value of deposition current at the starting of the deposition process is taken to obtain the distance vs. deposition current curve. The deposition layer on each cathodic glass substrates in terms of variation of deposition distance are shown in Fig. 4.3.

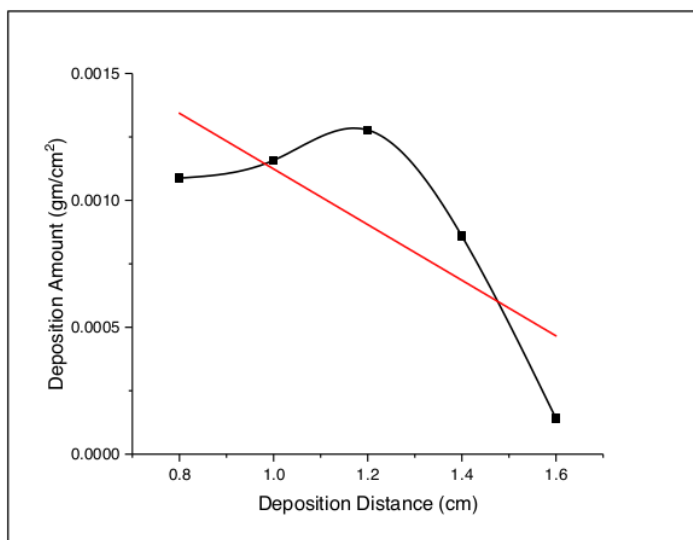


Fig. 4.1 Deposition distance vs. deposition amount curve

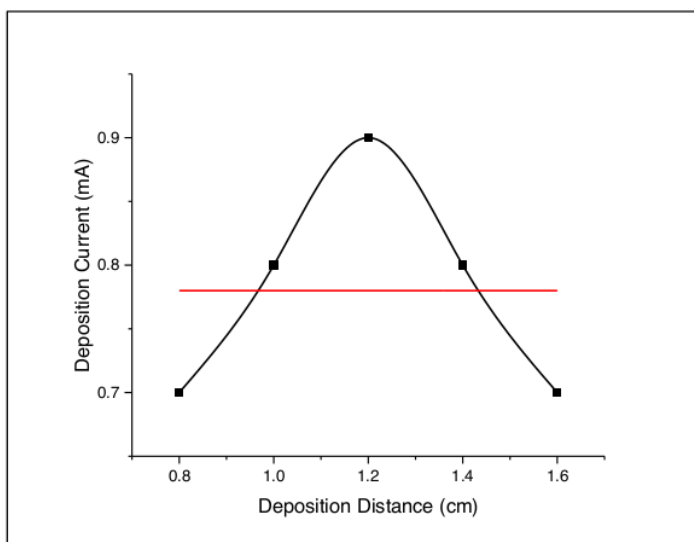
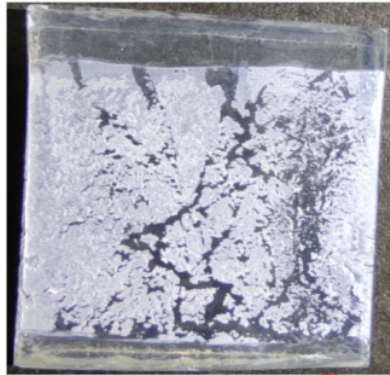


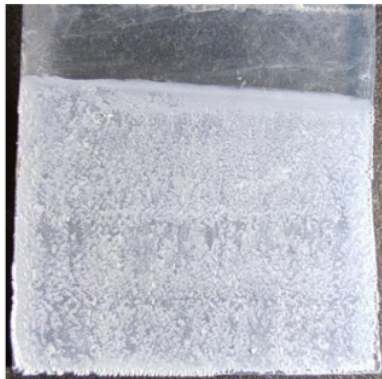
Fig. 4.2 Deposition distance vs. deposition current curve



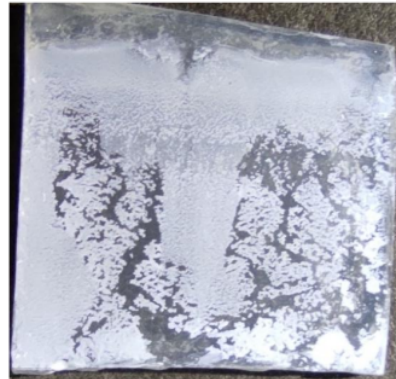
When, $D = 0.8$ cm, $V = 70V$, $T = 60$ s



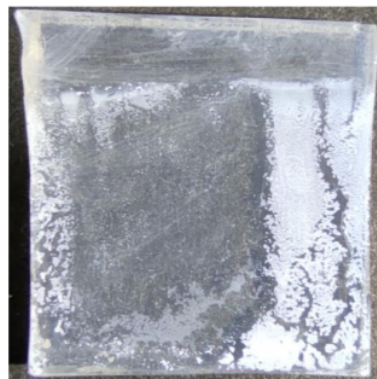
When, $D = 1$ cm, $V = 70V$, $T = 60$ s



When, $D = 1.2$ cm, $V = 70V$, $T = 60$ s



When, $D = 1.4$ cm, $V = 70V$, $T = 60$ s



When, $D = 1.6$ cm, $V = 70V$, $T = 60$ s

Fig. 4.3 Deposition layer on the glass substrates for variation of deposition distances.

4.3.2¹ Variation of deposition voltage

The deposition voltage was varied between 50 V and 90 V. The deposition time and distance between the plates were fixed for 60 s and 1.2 cm respectively. The results are shown in Table 4.2

Table 4.2¹ Variation of Deposition Voltage

Deposition Voltage (v)	50	60	70	80	90
Deposition amount (gcm ⁻²)	0.000263	0.000702	0.000752	0.000102	0.000639
Deposition Current (mA)	0.9	1.1	1.5	2	2.2

From Table 4.2 it is seen that with the increasing the voltage, the deposition amount and deposition current increases.

As like distance variation, here the deposition current contains two values. The first one is the value of deposition current at the start of the deposition process and the last one is the value of deposition current at the end of the deposition process. As an example, when the deposition voltage is 40 V, the deposition current is 1.43mA at the starting of the deposition process and 1.7 mA at the end of the deposition process.

The deposition current depends on various factor, one of those is the concentration of the solution. If the concentration of the solution increases deposition current increases and if the concentration of the solution decreases deposition current decreases. As in every process TiO₂ particles were deposited in the glass substrates, the concentration of the solution decreases. That is why deposition current was decreased gradually from the beginning to the ending of each deposition process.

The voltage vs. deposition amount curve is shown in Fig. 4.4 and the deposition voltage vs. deposition current curve is shown in Fig. 4.5. The value of deposition current at the starting of the deposition process is taken to obtain the distance vs. deposition current curve. The deposition layer on each cathodic glass substrates in terms of variation of deposition voltage are shown in Fig. 4.6.

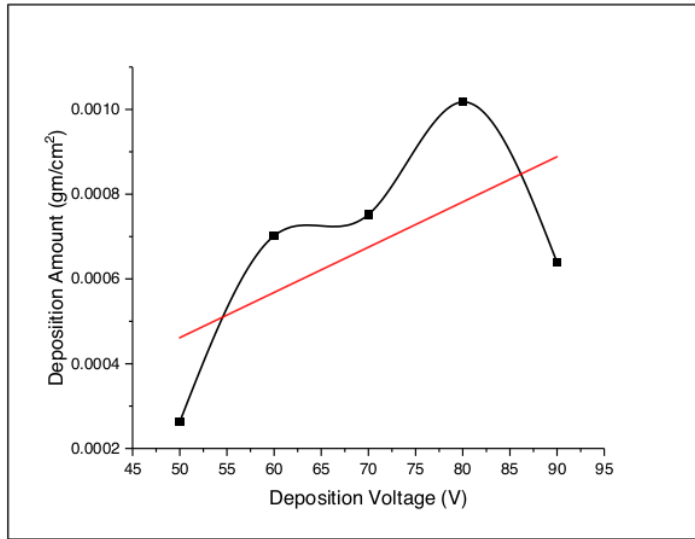


Fig. 4.4 Deposition Voltage vs. deposition amount curve

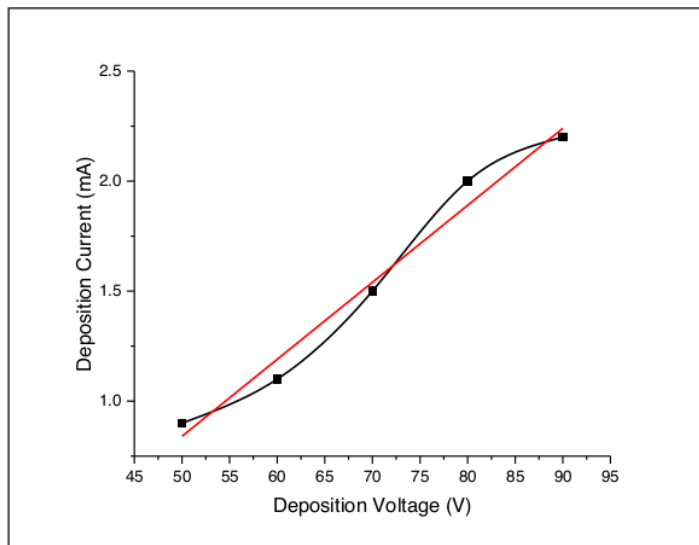
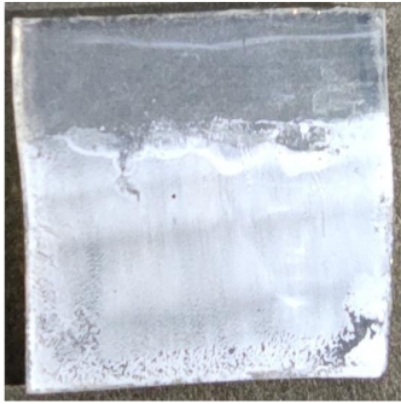


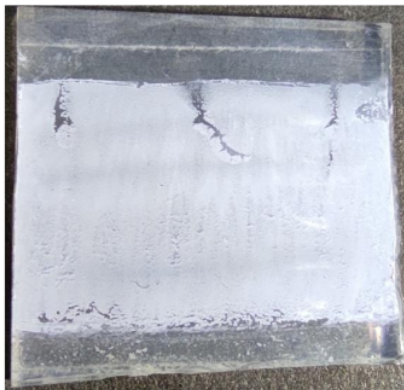
Fig. 4.5 Deposition voltage vs. deposition current curve



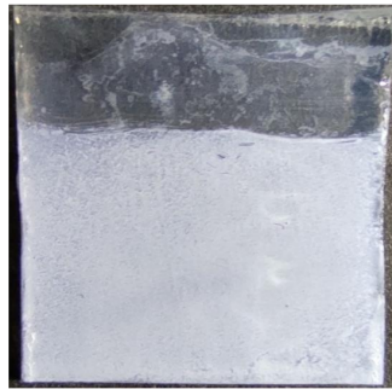
When, $V = 50 \text{ V}$, $D = 1.2 \text{ cm}$, $T = 60 \text{ s}$



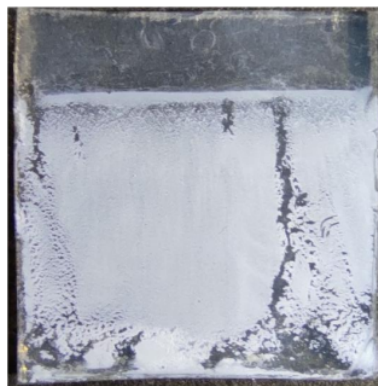
When, $V = 60 \text{ V}$, $D = 1.2 \text{ cm}$, $T = 60 \text{ s}$



When, $V = 70 \text{ V}$, $D = 1.2 \text{ cm}$, $T = 60 \text{ s}$



When, $V = 80 \text{ V}$, $D = 1.2 \text{ cm}$, $T = 60 \text{ s}$



When, $V = 90 \text{ V}$, $D = 1.2 \text{ cm}$, $T = 60 \text{ s}$

Fig. 4.6 Deposition layer on the glass substrates for variation of deposition voltage

4.3.3 Variation of Time

The deposition time was varied between 30 s and 70 s. The deposition voltage and distance between the plates were fixed for 40 and 1.5 cm respectively. The results are shown in Table 4.3:

Table 4.3: Variation of Deposition Time.

Deposition Time (S)	30	40	50	60	70
Deposition amount (gcm^{-2})	0.000596	0.000912	0.000947	0.001053	0.000421
Deposition Current (mA)	1	1.4	1.4	1.2	1.5

From Table 4.3 it is seen that increasing the deposition time the deposition amount increases and deposition current decreases.

The deposition current depends on various factor, one of those is the concentration of the solution. If the concentration of the solution increases deposition current increases and if the concentration of the solution decreases deposition current decreases. The deposition time vs. deposition amount curve is shown in Fig. 4.7 where the amount of the deposition increases with the increment of the time except the data for 70 second. As in every process ZnO particles were deposited in the glass substrates, the concentration of the solution decreases. That is why deposition amount can fall in that particular step.

In Fig. 4.8 the deposition time vs. deposition current curve is shown. As the data of the current has taken at the beginning of the process of electrophoretic deposition, it shows an uprising curve. The deposition layer on each cathodic glass substrates in terms of variation of deposition time are shown in Fig. 4.9.

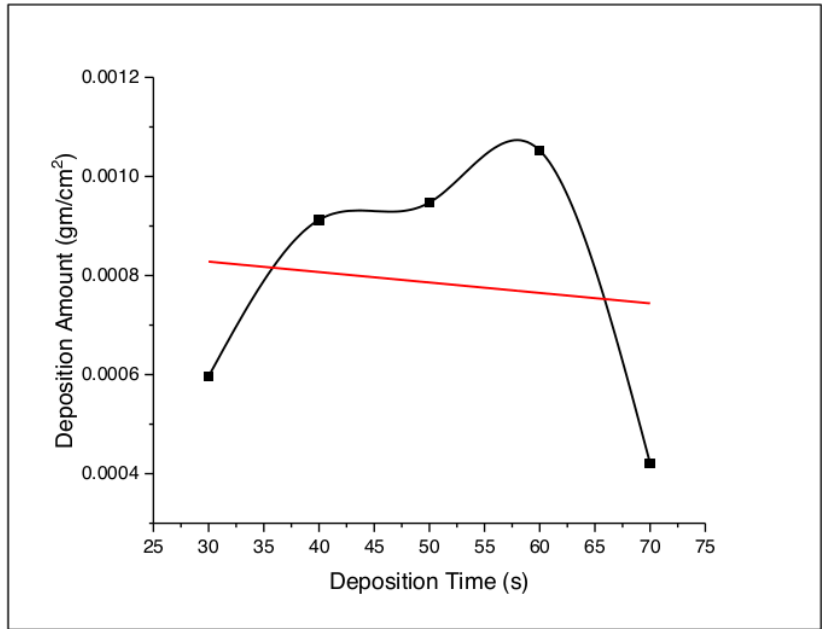


Fig. 4.7 Deposition time vs. deposition amount curve

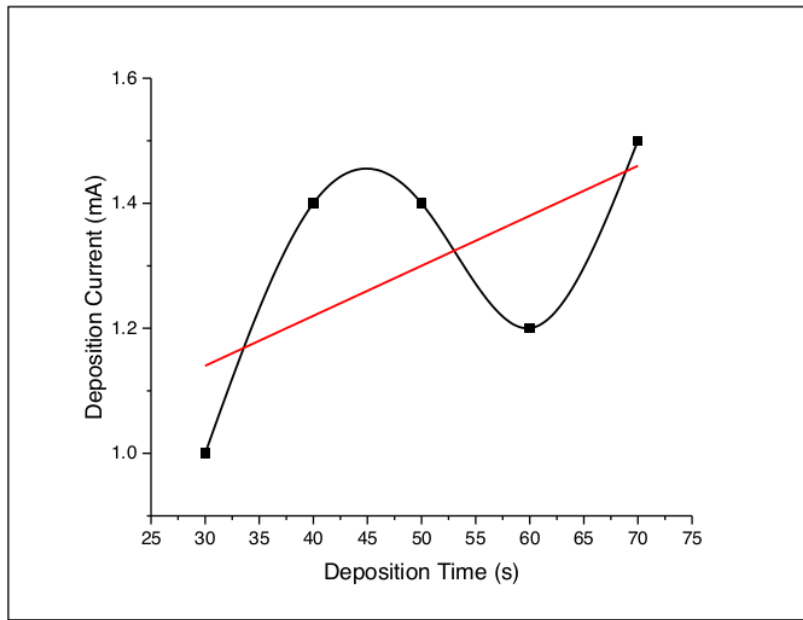
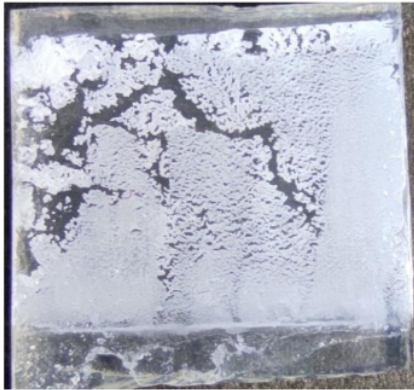
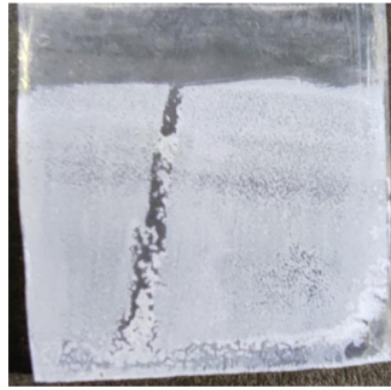


Fig. 4.8 Deposition time vs. deposition current curve



(a) When, $T=30\text{ s}$, $D=1.2\text{ cm}$, $V=70\text{ V}$



(b) When, $T=40\text{ s}$, $D=1.2\text{ cm}$, $V=70\text{ V}$



(c) When, $T=50\text{ s}$, $D=1.2\text{ cm}$, $V=70\text{ V}$



(d) When, $T=60\text{ s}$, $D=1.2\text{ cm}$, $V=70\text{ V}$



(e) When, $T=70\text{ s}$, $D=1.2\text{ cm}$, $V=70\text{ V}$

Fig. 4.9 Deposition layer on the glass substrates for variation of deposition time

4.4 Comparative study between ZnO and TiO₂ (P25)

M. S. H. Choudhury et al. has studied same **Electrophoretic Deposition of Titanium Oxide (P25)** on FTO type glass substrate as the same procedure described in 3.4 section [79]. The study was executed on the basis of three parameters as like this study which are **Deposition Distance, Deposition Voltage and Deposition Time.** On this segment both of these materials will be compared with each other.

4.4.1 I-V Characteristics

The current-voltage characteristics of ZnO and TiO₂ for the deposition voltage varied from 40v to 90v are shown in Fig. 4.10. Here for both materials, the I-V Characteristics shows a linear property. The Ohmic behaviour of the contact implies these characteristics and the charge transport efficiency and it also implies constant electrophoretic mobility. But the ZnO shows more vertical in Fig. 4.10 which implies better mobility and charge transport efficiency for ZnO.

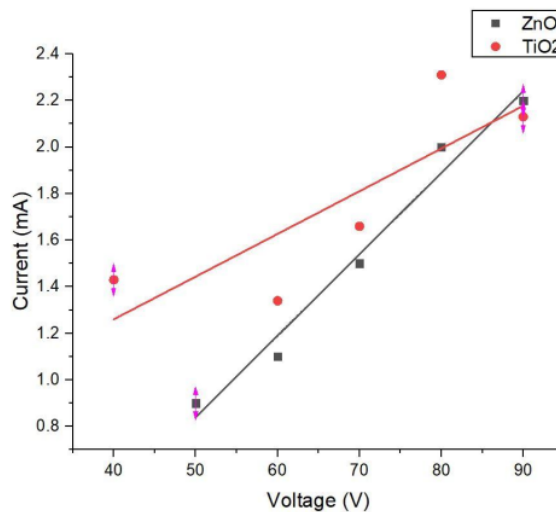
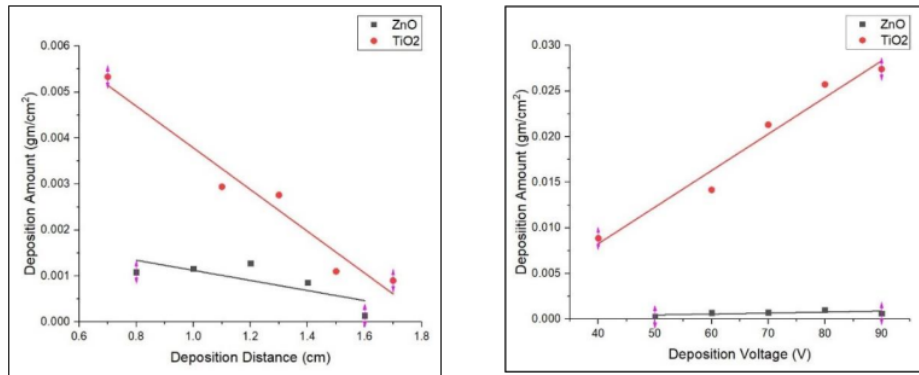


Fig. 4.10 I-V characteristics of ZnO and TiO₂

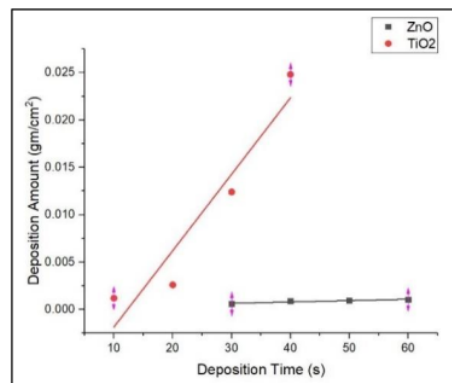
4.4.2 Deposition amount over three parameters

From the Fig. 4.11, it is been observed that for both the materials, the deposition amount follows the same trend. The amount of deposition over varying deposition distance in fig. a show the same trends of deposition. Both materials result down wards with the increase of the distance. In fig. b also follows the same trend of rising up ward for both

materials with the change of the voltage. Fig. c also shows the same results of following same trends for both materials of rising upward with the increment of deposition time. Though for the fig. b and fig. c, the deposition amount is low for ZnO compare to the TiO₂.



a) Deposition amount for variable distance b) Deposition amount for variable voltage



c) Deposition amount for variable time

Fig. 4.11 Deposition amount of ZnO and TiO₂ for different parameters

4.4.3 Introduction to tensile stress (cracking)

Whenever a layer of coating is bonded on a surface or substrate or film, there often forms a tensile stress which can cause to cracking in the deposition. This is a usual case for the metal or polymer type of coating. This tensile stress which causes the cracking problems can results in various problems like colloidal assembly and obstruct the standard photolithography. Nakamura and Kamath (1992) found that when the length of the crack is slightly larger than the thickness of the coating, can cause to the low energy release

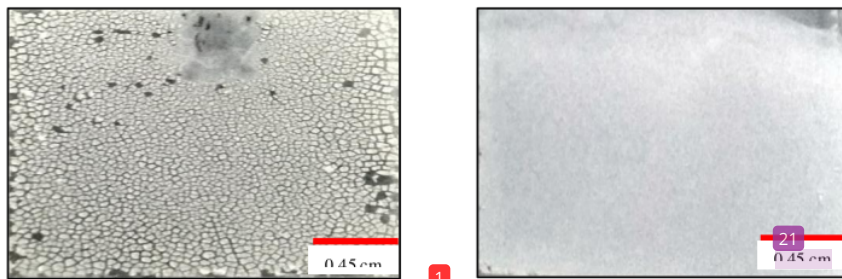
rate than steady state rate [80,81]. Beuth (1992) showed this condition of Nakamura and Kamath mathematically in the equation 4.1.

$$G = \frac{\pi (1 - \nu^2) h \sigma^2}{2 E} g(\alpha, \beta) \quad (4.1)$$

Where h refers to film thickness, E refers to the Young's modulus, ν is Poisson's ratio of the film and α, β is the elastic mismatch which is known as the Dundur's parameters. Here defines the energy release rate in steady state condition.

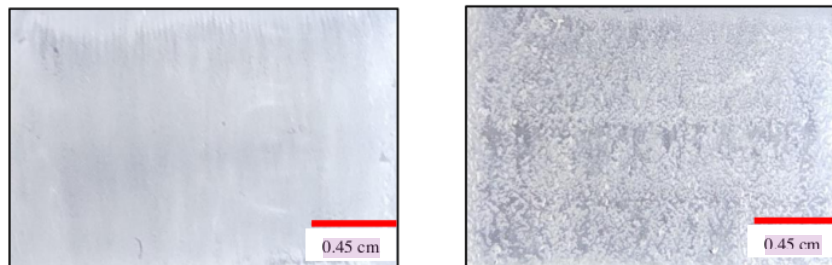
4.4.3.1 Comparative study between TiO₂ and ZnO of cracking in deposition

M. S. H. Choudhury et al. proposed in the study that with the deposition voltage of 40 v, deposition time of 60 s and deposition distance of 1.5 cm will be better option for the Titanium oxide electrophoretic deposition. The formation of crack for the electrophoretic deposition of the P25 is shown in fig. 4.10 where two figures are from two different parameters, which are variation of deposition time and variation of deposition voltage. If fig. 4.10 is compared with the fig. 4.11, it is clearly visible that the ZnO films are more crack free and with more smooth coating. That refers to more energy release rate of ZnO from the P25 which will give advantage to the ZnO.



When, V = 40 V, D = 1.5 cm, T = 60 s

Fig. 4.12 Formation of cracks on the deposition layer of TiO₂



When, V = 60 V, D = 1.2 cm, T = 60 s

Fig. 4.13 Formation of cracks on the deposition layer of ZnO

4.5 Discussion

From the above, one of the limitations has been observed that the amount of the deposition is low for the ZnO from Electrophoretic deposition comparatively. The deposition layer's thickness depends on the deposition voltage and the deposition period. However, a smooth crack free deposition is available with the correct option of the deposition voltage, deposition period and deposit distance. It is also important to refine deposition parameters so that cracks in the deposition layers are reduced in size. Electrophoretic deposition of ZnO studied on the basis of three parameters. From the fig. 4.3, 4.6 and 4.9, it is clearly visible that when the deposition voltage was set to 60 V, deposition time was set to 60 s and deposition distance is set for 1.2 cm, uniform deposition took place on the glass substrate. If the comparison between ZnO and TiO₂ (P25) is considered, Fig. 4.10 and fig 4.11 shows that ZnO deposits are smoother and crack free coating than P25.

One of the other concerns about this study is the concentration of the solution. After every deposition the concentration of the solution decreased since some ZnO particles were deposited on the glass substrate. To overcome this limitation, some ZnO has to be added so that the concentration remains comparatively unchanged. So, after every three depositions some ZnO is added in the solution and then the solution is dispersed for some time. The amount of ZnO need to be added is determined by the amount deposition took place on the substrates in previous three electrophoretic deposition process. Thus, the concentration of the solution is tried to keep constant. But as the amount is too small to calculate, there could be slight error in concentration of the solution. The exact form of the parameter deposition vs. deposition curve could not reliably be obtained, as it is known that the deposition curve has a relation to the solution concentration.

In the laboratory there was lacking of some instruments such as homogenizer, SEM etc. Some instruments were used as an alternative of these unavailable instruments. But the alternative instruments were not as useful as the unavailable instruments. As an example, electric mixer was used as an alternative of homogenizer but it was not as effective as homogenizer. So, the solution couldn't be dispersed as per expectation and the proper concentration of the solution couldn't be maintained. If it could be maintained properly the quality of the deposition layer would be better. Thus lacking of these instruments became an obstacle to get a better result of this experiment.

CONCLUSIONS AND FUTURE WORK

5.1 Conclusion

The study of Electrophoretic deposition on FTO coated glass substrates was concerned into two-part. The first portion consisted of a research on electric deposition of zinc oxide, which explored the effect of the three main process parameters. The parameters included deposition distance, voltage deposition, and deposition time. Any of the parameters has been changed several times by maintaining a constant two other parameters. At first, the distance to deposition was changed by maintaining the deposition and deposition voltage constant. The deposition voltage was then modified by maintaining a set deposition distance and deposition time. Finally, the time of deposition differed by maintaining the voltage of displacement and the deposition distance. For the variation of the three parameters, there are two types of the graph were plotted. One is the Deposition amount vs three different parameters individually and deposition current vs three different parameters individually. From the graphs, it is clear that with the increment of the distance between two substrates, the deposition amount slowly falls. When the concern is the deposition voltage, with the increment of voltage the amount of the deposition also increases. On the other hand, the amount of deposition increases with the increment of the last parameter of deposition time. If it is observed the surface morphology after every deposition parameter, a crack-free smooth coating can be found for a certain value of the parameters. So to get a crack-free smooth coating, it is been observed that the optimum condition for the deposition is when, Deposition Voltage (V) is 60 V, Deposition Time (T) is 60 s and Deposition Distance (D) is 1.2 cm.

In the second part of the analysis, a comparative study was performed between Zinc Oxide and Titanium Oxide to comprehend the characteristics of them. Both of the studies were performed based on three parameters that were described before. The study shows the amount of deposition of the ZnO is less than the deposition amount of the TiO₂. But the study clearly shows that the coating on the FTO coated glass substrate of ZnO is smoother and more crack free than the TiO₂. The I-V characteristics of both materials were linear which implies the Ohmic behaviour of the contact. This behaviour results in constant electrophoretic mobility. But the ZnO shows a more vertical graph, which implies better mobility and charge transport efficiency for ZnO.

5.2 Future Scope of EPD

In recent years, the electrophoretic process for the deposition of advanced materials has gained substantial interest. The process is simple, quick and economical and finds numerous applications, including thin and thick layer formation, layered ceramics, hybrid materials, fibre-reinforced composites and nano-composite composites, nanoscales of arranged 2-D and 3-D architectures and micro striped thin films. The procedure requires a careful selection of solvent media to stabilize the suspension as well as promote high electrophoretic mobility by developing significant surface charge magnitude in the powder surface of the suspension.

While mechanisms are not yet entirely clear, a whole new range of applications such as gas separation sensors, thermal barrier coating, etc. is opened to the option that non-leading substrate is deposited. Further research into fundamental mechanisms through adequate model development, the development of charges on non-aqueous suspension to properly monitor suspension stability and deposition is required. However, further research is needed. The non-aquatic suspensions are popular electrically deposited media. However, developing water-based electrophoretic deposition would significantly reduce deposition cost and also minimize environmental issues related to the use of non-aquatic suspensions. The procedure can be analyzed with the correlation entre the substrate and the material deposited on several substrates.

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