ISSN: 2834-4928

Research Article

Journal of Electrical Electronics Engineering

Yttria Stabilized Zirconia Thin Film Electrolyte for Low-Temperature Solid Oxide Fuel Cells Using Sol-Gel Dip Coating Method

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Submitted: 2025, Apr 08; **Accepted:** 2025, May 13; **Published:** 2025, May 27

Citation: Garba, M. B., Rosnita, M., Zakaria, N. F. (2025). Yttria Stabilized Zirconia Thin Film Electrolyte for Low-Temperature Solid Oxide Fuel Cells Using Sol-Gel Dip Coating Method. *J Electrical Electron Eng, 4*(3), 01-06.

Abstract

This research investigates the development and analysis of Yttria Stabilized Zirconia (YSZ) thin film electrolytes for low-temperature Solid Oxide Fuel Cells (LT-SOFCs) using the solgel dip coating method. YSZ is valued for its high ionic conductivity and thermal stability, which are vital for SOFC operation at reduced temperatures. The sol-gel dip coating technique offers a versatile and cost-effective approach to control film thickness and microstructure. In this study, YSZ thin films are synthesized by varying precursor concentrations, coating period and annealing temperatures to optimize film properties. Structural analysis using X-ray diffraction (XRD) confirms the formation of the crystalline phase while Scanning electron microscopy (SEM) reveals a uniform and dense microstructure. Results indicate that films with higher precursor concentrations, 4 g and longer coating periods, 15 min show increased surface roughness and porosity. Atomic force microscopy (AFM) measurements reveal roughness values ranging from 57.5 nm to 172.0 nm for 4 g precursor solutions, while 2 g solutions exhibit roughness between 80.3 nm and 238.8 nm. SEM images show denser but more porous films with longer coating periods, which could affect the mechanical and electrical properties. Contact angle measurements indicate nucleation follows the Frank-van der Merwe growth mechanism, with angles ranging from 200 to 800 depending on growth conditions.

Keywords: Dip-Coating, Electrolyte, Solid Oxide Fuel Cell, Thin Film, Yttria-Stabilized Zirconia

1. Introduction

Solid Oxide Fuel Cells (SOFCs) are considered one of the most promising technologies for efficient and environmentally friendly energy conversion devices. Their ability to directly convert chemical energy from fuels into electrical energy through electrochemical reactions has garnered significant attention in recent years. SOFCs offer several advantages over conventional energy conversion systems, including high energy efficiency, fuel flexibility and low emissions of pollutants such as NOX and SOX. However, conventional SOFCs typically operate at temperatures above 800 oC, which presents several challenges, including material degradation, thermal instability and high system costs due to the need for high-temperature resistance materials [1,2].

In response to these challenges, there has been growing research interest in Low-Temperature Solid Oxide Fuel Cells (LT-SOFCs), which operate at temperatures below 650 oC. Lowering the operating temperature can significantly extend the lifespan of materials, reduce production costs and enable the use of less expensive components. However, one of the major barriers to the commercialization of LT-SOFCs is the development of electrolyte materials with high ionic conductivity at lower temperatures. This is where Yttria Stabilized Zirconia (YSZ), a widely used electrolyte material in SOFCs, becomes relevant [3].

YSZ is highly considered for its excellent oxygen ion conductivity, high mechanical strength and chemical stability at elevated temperatures. It operates by allowing oxygen ions to migrate from the cathode to the anode through oxygen vacancies in its fluorite crystal structure, thus enabling the electrochemical reaction. However, at lower temperatures, YSZ exhibits reduced ionic conductivity, which diminishes the overall performance of the fuel cell. Therefore, a critical research challenge has been improving the performance of YSZ at reduced operating temperatures while retaining its desirable properties [4].

Thin film electrolytes have emerged as a promising solution to the conductivity limitations of YSZ at lower temperatures. By reducing the electrolyte thickness, ionic resistance can be minimized, thereby improving overall ionic conductivity, even at reduced temperatures. Various deposition techniques, such as physical vapor deposition (PVD), sputtering and chemical vapor deposition (CVD), have been explored for fabricating YSZ thin films. However, many of these methods require high processing temperatures and expensive equipment, limiting their scalability for commercial applications [5].

In contrast, the sol-gel dip coating method offers a cost-effective and versatile alternative for depositing YSZ thin films. This wet-chemical technique enables precise control over the composition and thickness of the films at relatively low processing temperatures. The sol-gel process involves transforming a solution into a gel-like network, which can subsequently be converted into a dense ceramic film through heat treatment. In this method, a substrate is dipped into the YSZ sol, withdrawn at a controlled speed and then subjected to heat treatment to remove organic components and densify the film [6]. The sol-gel dip coating method has the potential to produce high-quality YSZ thin films that are crack-free, uniformly dense and suitable for use as electrolytes in LT-SOFCs [7].

The research focuses on the fabrication and characterization of YSZ thin film electrolytes using the sol-gel dip coating method, to enhance their ionic conductivity at low temperatures. The effects of growing parameters such as precursor concentration, heat treatment and thickness of the film on the structural, morphological and electrochemical properties of the YSZ thin films are critically investigated. Furthermore, the performance of the resulting YSZ thin film electrolyte in LT-SOFC applications is evaluated to assess their potential for commercialization in fuel cell systems operating at lower temperatures.

This study attempts, to address the challenges associated with YSZ performance at low temperatures and contributes to ongoing research efforts to advance LT-SOFC technologies by optimizing the sol-gel dip coating parameters. The successful development of YSZ thin film electrolytes could significantly reduce the operating temperature of SOFCs, opening new avenues for their application in stationary power generation, electronics and transportation.

2. Solid Oxide Fuel Cells (SOFCs)

Solid Oxide Fuel Cells (SOFCs) are fuel cells that generate electricity through the electrochemical oxidation of fuel. They operate at high temperatures (typically 600-1000°C) and are known for their high efficiency, fuel flexibility, and low environmental impact. SOFCs are composed of an anode, cathode, and electrolyte [8-11]. The electrolyte conducts oxygen ions from the cathode to the anode, facilitating the electrochemical reaction that generates electricity. The efficiency and performance of SOFCs are highly dependent on the properties of the electrolyte material [12].

The anode facilitates the oxidation of the fuel (e.g., hydrogen or hydrocarbons) and conducts electrons to the external circuit. Common materials include nickel-based cermets (ceramicmetal composites), typically nickel/yttria-stabilized zirconia (Ni/YSZ) [13]. The cathode facilitates oxygen reduction from the air and conducts electrons from the external circuit to the electrolyte [14]. Typically made from mixed ionic and electronic conductors (MIECs) like lanthanum strontium manganite (LSM) or lanthanum strontium cobalt ferrite (LSCF) [15]. The electrolyte conducts oxygen ions from the cathode to the anode and is impermeable to gases. Common electrolyte materials include yttria-stabilized zirconia (YSZ), gadolinia doped ceria (GDC), and scandia-stabilized zirconia (ScSZ) [16].

SOFCs operate on the principle of converting chemical energy directly into electrical energy through an electrochemical reaction. The overall process involves:

At the Anode: Fuel (e.g., hydrogen) reacts with oxygen ions (O^{2-}) from the electrolyte to produce water, releasing electrons, (5).

$$H + O \quad - - -> H\ O + 2e \qquad (1)$$

At the Cathode: Oxygen from the air gains electrons to form oxygen ions, which are conducted through the electrolyte to the anode, (17).

$$O + 4e \quad - - -> 2O \qquad (2)$$

Electron Flow: The electrons flow through an external circuit from the anode to the cathode, generating electrical power.

2.1 Structure of Solid Oxide Fuel Cell (SOFC)

Solid Oxide Fuel Cells (SOFCs) consist of three primary components: the anode, the cathode, and the electrolyte, Figure 1. Each of these components plays a crucial role in the efficient operation of the fuel cell, contributing to its overall performance, durability, and efficiency.

The electrolyte in an SOFC is a solid ceramic material that conducts oxygen ions from the cathode to the anode. The most commonly used electrolyte material is yttria-stabilized zirconia (YSZ) due to its high ionic conductivity and stability at elevated temperatures [18]. YSZ is typically doped with 8-10% yttria to enhance its ionic conductivity and mechanical properties [19]. The electrolyte must be dense and impervious to gas to prevent the mixing of fuel and oxidant, which could lead to reduced efficiency and safety concerns.

The anode in an SOFC is responsible for facilitating the oxidation of the fuel. It is typically made from a composite material known as a cermet, which combines ceramic and metallic components. The most common anode material is a nickel-YSZ cermet [20]. Nickel provides excellent catalytic activity for the oxidation of hydrogen and hydrocarbon fuels, while YSZ ensures compatibility with the electrolyte and maintains the structural integrity of the anode at high temperatures [21]. The anode must be porous to allow the fuel to diffuse through it and reach the electrolyte interface where the electrochemical reaction occurs.

The cathode in an SOFC facilitates the reduction of oxygen molecules to oxygen ions, which then migrate through the electrolyte to the anode. The cathode material must exhibit both electronic and ionic conductivity to efficiently transfer electrons and oxygen ions. Lanthanum strontium manganite (LSM) is the most commonly used cathode material because it offers a good balance of electronic conductivity, ionic conductivity, and thermal expansion compatibility with YSZ [5]. The cathode must also be porous to allow the oxygen from the air to reach the electrolyte interface.

In addition to the primary components, SOFCs require interconnects and seals to function effectively in a stack configuration. Interconnects are typically made from high-temperature alloys or ceramics and serve to connect individual cells electrically while separating the fuel and oxidant gases. They must possess high electrical conductivity and corrosion resistance at operating temperatures [22]. Seals are used to prevent gas leakage between the anode and cathode compartments and must be chemically stable and mechanically robust at high temperatures.

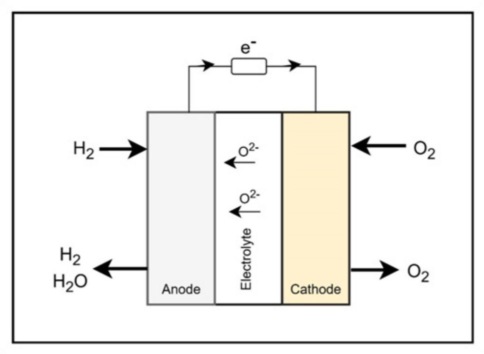

Figure 1: Schematic Illustrations of Solid Oxide Fuel Cell Structure

3. Experimental Procedure

3.1 Sample Preparation

In this research, silicon (SiO2) glass was used as a substrate material for thin-film deposition. The glass was cut to 10 mm × 10 mm and washed with acetone, ethanol, and deionized water at 200 C for 10 minutes to remove impurities and rinse away the residue.

The cleaned substrates were dried using an oven at 60°C for 15 minutes to ensure no bubbles formed on the surface of the substrate.

3.2 Preparation of Precursor Solution

The precursor solution was prepared using the sol-gel method according to the growing parameters in Table 1.0. YSZ was dissolved in 40mol ethanol, mixing with poly-viny-alcohol (PVA) and polyethylene glycol (PEG) as binder and plasticizer, respectively. The suspension was stirred on the hotplate at the temperature of 600C for 2 hours and continued ultrasonic mixed for another 1 hour in an ultrasonic bath to ensure homogeneity of the precursor solution. Table 1.0: Thin Film Growing Parameters.

Sample Code	**YSZ Mass (g)**	**Coating Period (min)**	**Annealing Temperature (oC)**
YSZ 1	2.0	5	400
YSZ 2	2.0	10	500
YSZ 3	2.0	15	600
YSZ 4	4.0	5	400
YSZ 5	4.0	10	500
YSZ 6	4.0	15	600

The cleaned prepared samples were attached to the dip coater machine's sample holder for the thin film fabrication. The film was set to be prepared for a single layer and the withdrawal speed of 150mm/min, remained constant throughout the process, the samples then were dried for 24 hours at room temperature. All samples were treated with a pre-heat of 100 °C for 1 hour before being sintered at 400 oC, 500 oC and 600 oC at a 2°C/min rate for 2 hours respectively to densify the films.

3.3 Sample Characterization

The phase structures of the YSZ thin films were characterized by an X-ray Diffractometer (XRD). Moreover, the morphology, thickness and grain size of the thin films were characterized by employing Scanning Electron Microscope (SEM).

4. Results and Discussion

4.1 Structure Characterization

Figure 2.0 shows 3D AFM images of YSZ thin films grown by sol-gel dip-coating with different growth parameters. Figures 2.0 (a) to (c) are 2 g precursor solutions meanwhile

Figures 2.0 (d) to (f) refer to 4 g solution. (a) and (d) is for 5 mins dipping coat, (b) and (e) for 10 mins and (c) and (f) are related to 15 mins dipping coats. From the AFM result, we can measure the root mean square (RMS) roughness of the thin film surfaces. RMS reflects the average deviation of the surface height from the mean plane. Higher RMS indicates a rougher surface on the thin film. From the result obtained in AFM the surface roughness is getting thicker from 2 g to 4 g due to differences in precursor solution. The surface roughness increases as the dip coating is left for a long time. Table 2.0 listed the RMS of YSZ with different precursor solutions and growth dipping coating.

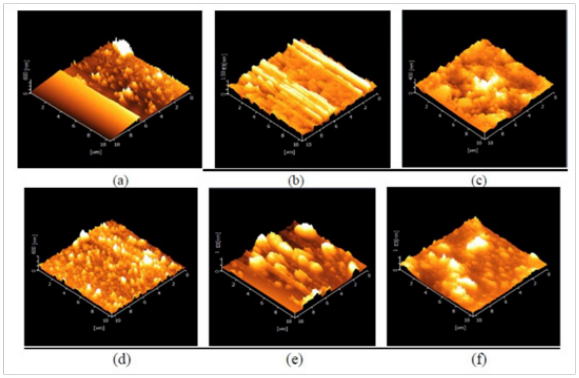

Figure 2: AFM Images of YSZ thin film (a) – (c) for 2 g and (d) – (f) for 4 g solution

Coating Periods (Min)	Root Mean Square (nm)	
	2 g	4 g
5	80.3	57.5
10	238.8	172.0
15	58.8	117.9

Table 2: RMS of YSZ Thin Films with Different Precursor Solutions and Coating Periods

Figure 3.0 shows the scanning electron microscopy (SEM) of YSZ thin films grown with different growth parameters. Figures 3.0 (a) to (c) are 2 g precursor solution with increasing time dipping from 5 to 15 mins. Figures 3.0 (c) to (f) are related to 4 g solution with the same increasing time dipping from 5 to 15 mins. It shows that the surface roughness increases when the dip coating time increases. With a longer growth time, the film may undergo further densification as particles rearrange and voids shrink. However, this densification can be incomplete, leaving behind some porosity. Additionally, longer growth times may promote grain growth within the film, which can create intergranular porosity between grains. During deposition, gas species like water vapor or residual solvent molecules can become trapped within the growing film. Longer growth times may allow for more of these gas species to become trapped, contributing to porosity. Higher porosity can lead to lower mechanical strength and increased film brittleness. Depending on the application, porosity can have a complex effect on electrical properties. Due to the ability to control film thickness through multiple layers, sol-gel allows for achieving denser YSZ films with shorter overall growth times compared to techniques requiring a single thick deposition. This can be beneficial as it minimizes the risk of excessive grain growth, or other undesirable effects associated with longer deposition times.

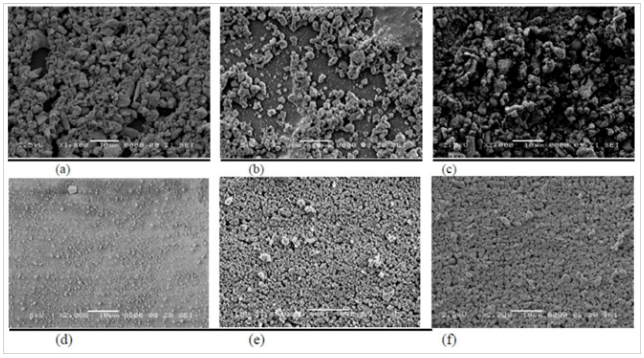

Figure 3: SEM images of YSZ thin film (a) – (c) for 2 g and (d) – (f) for 4 g solutions

Figure 4 shows the measurement of the contact angle between the substrate surface and nucleation of YSZ particle for 4 g precursor solution with 5 mins dip coating. The measured contact angle is 45 degrees, and the nucleus seems to be stable on the surface. Nucleation plays a prominent role in the condensation and evaporation of crystal growth and deposition of thin films. They need to nucleate before a new phase can be formed. From the Young equations, a stable nucleus is governed by the equilibrium of wetting conditions between the nucleus and substrate and thus will form a dome-like shape of nanofilm. This growth parameter (Figure 4.0) follows the Frank-van der Merwe growth mechanism with theta < 90 degrees and surface energy between the nucleus and substrate is less than one. The film atoms are more strongly bound to each other than to the substrate. Table 3.0 lists the contact angle measurement of the samples in this study.

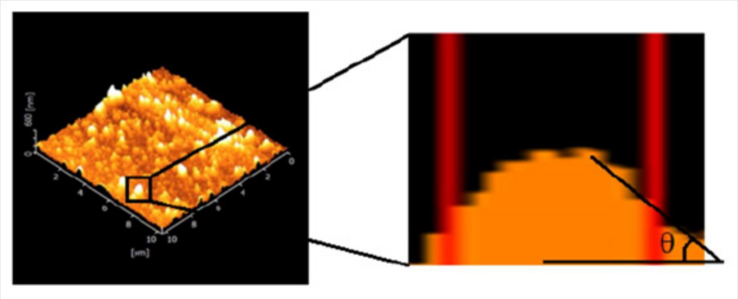

Figure 4: AFM of YSZ particle (4 g, 5 min) (a) YSZ particle and (b) contact angle Table 3.0 The contact angle of YSZ particles with different precursor solutions and time dipping.

Coating Periods (Min)	Contact Angle of YSZ Particle (Degree)	
	2 g	4 g
5	20	45
10	5	80
15	10	60

5. Conclusions

The study successfully demonstrated that the sol-gel dip coating method can produce YSZ thin film electrolytes with desirable structural and electrochemical properties for LT-SOFCs. The key experimental parameters, including precursor concentration and dip coating period, significantly impacted the film's microstructure, surface roughness and porosity. A shorter coating period and lower precursor concentrations yielded smoother, more uniform films. In contrast, longer coating periods increased porosity and brittleness, potentially affecting the films' mechanical and electrical properties. The analysis of nucleation behavior further confirmed the stability of YSZ particle deposition, following a Frank van der Merwe growth mechanism. Overall, the results affirm that optimizing the sol-gel dip coating parameters can enhance the ionic conductivity of YSZ at lower temperatures, advancing LT-SOFC technology. Future work should focus on refining the microstructure to achieve higher conductivity and explore scaling up the process for commercial applications, particularly in stationary power generation and transportation.

Acknowledgement

The authors would like to express appreciation for the support of the sponsors [GUP IE: Q. J130000.3854.19J18 and UPMU analysis grant], University of Technology Malaysia analysis grant & Tertiary Education Trust Fund (Tet Fund) Nigeria, staff training development.

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