

Review on Deposition Techniques and Protective Coatings of Metallic Interconnectors for Solid Oxide Fuel Cell

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ABSTRACT: To realize the commercialization of solid oxide fuel cells (SOFC), their cost should be competitive enough in comparison to alternative power generation systems. Hence there is a modern technology drive to manufacture the SOFC components at much lower cost. SOFC interconnect and current collector meshes are the major components which significantly contribute to the system cost. With reduced operating temperature of solid oxide fuel cells (SOFCs) to the range of 600°C-800°C, metallic interconnectors with high oxidation resistance are used to replace conventional ceramic interconnectors. Metallic interconnections are of advantage over ceramic interconnections as regards to output, expense, mechanical strength and electrical conductivity. To Date, all Cr-containing alloys which are promising candidates for metallic interconnections are prone to dynamic Cr migration causing deterioration of the cells. Protective coatings to effectively inhibit Cr migration have been developed; as well as maintaining excellent electric conductivity and a good resistance to oxidation. This article reviews the progress and technical challenges in developing metallic interconnects; different types of protective coatings and deposition techniques for metallic interconnects for intermediate temperature SOFC applications.

KEY WORDS: SOFC, interconnectors, protective coatings.

I. INTRODUCTION

Fuel cell bypasses the conversion of chemical energy of fuel into thermal and mechanical energy. Technology can be used in applications with a broad range of power needs. Their application ranges from systems of a few watts to megawatts. When compared to other electrochemical devices SOFCs exhibit excellent fuel flexibility, efficiency and power reliability as well as lower emission and operating cost. The SOFCs are typically designed for stationary high-power (100 kW up to megawatt), small-power (1-20 kW) and portable electronic devices (20-250 W) applications. Advances in thin-film processing and micro-manufacturing have prompted the innovative concept of using micro-SOFC as a possible battery replacement in portable electronic devices (estimated at 1-20 W). The SOFCs which have total thickness in micrometers and material microstructure at the nano scale level are indicated as micro-SOFCs[1].

Fig 1 illustrates the working principle of solid oxide fuel cell. Fuel (hydrogen) supplied to anode. Fuel is oxidized to release electrons. Oxygen is supplied to the cathode, here oxygen acts as oxidant and is reduced to oxygen ions(O₂⁻)[2]. These ions pass through the electronically separated electrolyte, from which they conduct ions and react to water at the anode with hydrogen. The electrons released then pass through an external circuit known as interconnections to produce electricity. The driving force of oxygen delivery by the thick electrolyte is the difference between the anode and the cathode in oxygen partial pressure[1].

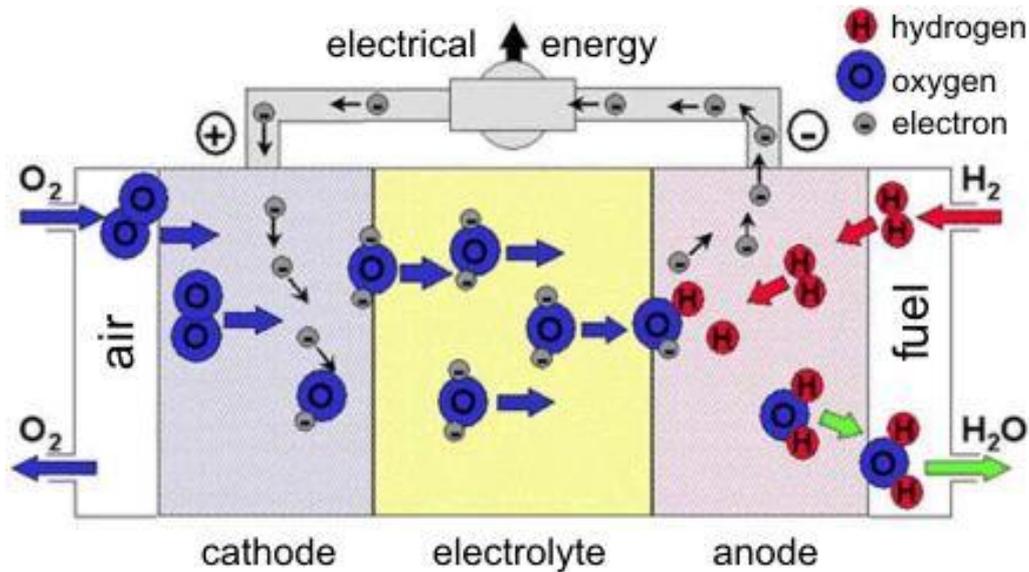


Fig 1: Working principle of solid oxide fuel cell.

In general, a single cell refers to the configuration of a thick sandwiched electrolyte between two porous electrodes. Multiple single cells are then stacked in a planar or tubular design, connected via interconnections in the electrical series to accumulate voltage output and function as an electrochemical power source[3]. Planar SOFCs, specifically anode-supported ones, possess higher power densities and lower manufacturing costs than tubular cells. However, the marketing of planar SOFCs is limited by technical obstacles in the development of highly ionic conductive electrolyte content at cell operating temperature, electrodes with sufficient catalytic activity and conductivity and cost-effective processing of metallic interconnections[4]. Interconnect operating in both oxidizing and atmospheric reduction requires the most demanding selection criteria among the four major components (anode, cathode, electrolyte and interconnect). Doped lanthanum chromites (LaCrO_3) have been favored over the decades for SOFCs at high temperatures (near 1000°C)[3]. LaCrO_3 exhibits excellent electrical conductivity, stability in reduction and oxidation environment as well as compatible coefficient of thermal expansion (TEC) matching neighboring materials[4]. Ceramic materials, however, are difficult to fabricate, thereby restricting interconnect geometric nature. In addition, LaCrO_3 , a semiconductor of type p, is deficient in oxygen with reduced partial oxygen demand, resulting in Poor Conductivity. Lanthanum is a rare earth element, which is expensive. Manganese cobalt oxide ($\text{Mn}_{1.5}\text{Co}_{1.5}\text{O}_4$) is considered to have good thermal stability, excellent electrical conductivity and a consistent coefficient of thermal expansion (TEC) with the substrate[5].

To date, protective coatings have been manufactured using a wide variety of techniques such as sol-gel, plasma spray, electrophoretic deposition etc. These techniques cannot be used for dense coatings. Additionally, sophisticated techniques such as chemical vapor deposition (CVD) RF-magnetron sputtering, EB-PVD, and pulsed laser deposition were used[6]. Techniques, coverage of wider areas and cost-efficiency are the major bottlenecks. With recent progress towards lowering the operating temperature of SOFCs within the range of 600°C - 800°C , metal alloy replacing the conventional one is used with high oxidation resistance interconnecting ceramics. Ferritic stainless steel, full of hope Candidate Metallic Interconnects, is susceptible through rapid growth of chromium at high and volatile temperatures Cr migration, causing degradation of the cells. Researchers have focused on addressing those limitations for developing Protective coatings[7].

II. METALLIC INTERCONNECTS FOR SOFCs

Interconnections provide electrical contact between individual cells and serve as a physical barrier to distinguish fuel (anode) from oxidant (cathode) gasses[8]. Strict requirements like excellent oxidation to achieve reliable performance; Air and H_2 / H_2O atmospheric resistance, excellent electrical conductivity, good thermal conductivity and compatible TEC with adjacent components for SOFC interconnecting materials[7].



Metallic interconnections show better fabrication, lower cost, improved mechanical strength and higher electrical and thermal conductivity compared to ceramic interconnections. Metallic interconnections typically possible form a continuous layer of oxide scales of alumina (Al₂O₃) or chromium (Cr₂O₃) to provide oxidation resistance under SOFC operating conditions. In this sense, because of the isolating aspect of the Al₂O₃ scales, Cr-forming alloys are favored as interconnections. Potential metallic candidates assessed, to date are all alloy-containing chromium, such as alloy based on Cr⁻, Fe⁻ - Cr⁻ and Ni-Cr⁻ [9].

III. POTENTIAL CANDIDATES.

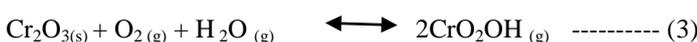
Cr-based dispersed oxide reinforced alloy, especially Ducrolloy (Cr-5Fe-1Y₂O₃, designed by Plansee Company), is considered to replace LaCrO₃ for SOFCs operating at close to 1000 C. Ducrolloy shows excellent oxidation resistance and comparable TEC fit with other components of SOFCs[8]. Excessive Cr₂O₃ concentrations, however, result in excessive high-area-specific resistance (ASR) after 75 hours of oxidation at 1000 °C. Besides that, alloy based on Cr is suitable only when a simplified manufacturing process is developed with reduced processing costs[10]. Fe-Cr-based alloys are more ductile, better working capacity and lower cost compared to Cr-based alloys. Fe-Cr-based alloy, specifically ferritic stainless steel, with an optimum Cr-content of 17-25 percent, offers excellent oxidation resistance by forming a continuous scale to Cr₂O₃. Yang et al. recorded this power output and the resistance to oxidation depends on the composition of the steel substrates; that is, the steel substrates with a relatively high Cr concentration (> 23%) and a marginal residual Si exhibit significantly reduced contact resistance and growth rate of the oxide scale[11]. The resistance to oxidation is substantially provided if the Cr-content is reduced to 5%-10%. Ferritic stainless steel's body-centric cubic construction ensures compatible TEC fit with other SOFC components. However, impurities from the steel substratum thus form continuous Al₂O₃ and SiO₂ layers and increase ASR significantly by oxidation for 4000 hours[9].

Ni-Cr-based alloys demonstrate excellent oxidation resistance and electrically conductive oxide scales; however, their inappropriate high TEC values impede the application of these alloys for interconnecting SOFCs. Ferritic stainless steel is the most promising candidate to date, since its TEC suits the surrounding cell components well and is economically processable[12].

IV. TECHNICAL CHALLENGES

There are two key drawbacks of Cr-containing alloys, namely rapid growth of Cr₂O₃ at high temperatures which increases over time electrical resistance and evaporation/ Cr (VI) species migration from interconnections into the cathode which dramatically impairs the performance of the cells. The mechanism of these phenomena and their kinetics has been extensively studied[5]. The thickness of Cr₂O₃-scales is measured in micrometers, or even tens of micrometers, after oxidation under the SOFC operating atmosphere for thousands of hours. Rapid growth of Cr₂O₃-scales, leading to increased ASR or cracking/spalling, is not acceptable[1][6]. As such, oxidation rate by adjusting the oxidation kinetics with a small amount of Ti (0.5 wt percent to 0.7 wt percent) can be effectively reduced. Nevertheless, high Ti levels (0.98 wt percent) produce excess ionic defects and reduce the resistance to oxidation[13][11].

Cr species volatilization is highly dependent on partial oxygen pressure and water content. This cycle is frequently ignored on the anode side as Cr-O-H gas species has maximum equilibrium vapor pressure is much higher on the air / cathode side (between 10⁻⁶ hp and 10⁻⁹ hp) than on the fuel / anode side (between 10⁻¹⁰ hp and 10⁻¹⁴ hp). The thermodynamic instability of the Cr₂O₃ scales formed on the Cr-containing alloys generates volatile Cr(VI) species in a cathodic environment, in particular gaseous oxides of chromium (CrO₃) and chromium oxyhydroxide (CrO₂(OH)₂), by the following reactions[14][8].



Cr poisoning mechanism and kinetics are inconsistent, particularly on the LSM cathodes. One is that the volatile Cr (VI) species is either electro-chemically reduced or chemically deposited at the triple phase boundary, the electrolyte / cathode / oxidant interface, where the deposition blocks the active cathode area, leads to cell degradation[15][8]. The second is that the nucleation theory, which suggests the chemical reduction of high-valence Cr species, is enabled by the nucleation agents on and/or at the electrode and electrolyte surfaces configuration of the electrode / electrolyte[16].

V. PROTECTIVE COATINGS

Active and low-cost protective coatings have been developed to minimize oxide growth kinetics, increase oxide scale conductivity, enhance oxide scale adhesion to metal and inhibit Cr migration from the Cr_2O_3 rich subscales to the oxide surface. This section reviews the potential of SOFC interconnect applications rare earth perovskites and composite spinel oxides.

RARE EARTH PEROVSKITES : In an oxidizing environment, rare earth perovskites exhibit p-type electronic conduction and are stable under low partial oxygen pressure. Fig 2(a) shows the perovskite unit cell defined by the general formula of ABO_3 , where A is a trivalent rare earth cation (e.g., La, Y or Sr) and B is a transition metal cation (e.g. Cr, Ni, Fe, Co, Cu or Mn)[8][15]. Doping sufficient quantities of Cu, Ni or Fe will improve the electrical conductivity and TEC efficiency of rare earth perovskites. Effectiveness of perovskite coatings such as lanthanum chromite (LaCrO_3), lanthanum strontium chromite, lanthanum Strontium manganite, lanthanum strontium cobaltite and lanthanum strontium ferrite were developed on ferritic stainless steel by sputtering, solgel and screen printing. The ionically conductive perovskites remain an ineffective barrier for volatile Cr species or oxygen diffusion despite their improvements[9][8].

COMPOSITE SPINEL OXIDES : Composite spinel oxides are described by the general formula of AB_2O_4 , with A and B as divalent, trivalent or quadrivalent cations in octahedral and tetrahedral sites, and oxygen anions in the face-centered cubic lattice sites, as shown in Fig 2 (b). Due to their capacity to suppress volatile Cr species from the Cr_2O_3 -rich scale to the scale surface, composite spinel coatings have drawn significant research attention[9][17]. The properties of a spinel can be modified by adjusting the selection of A and B cations and the ratio of those cations. Previous research investigates the thermal and electrical properties of binary spinels containing Al, Mg, Cr, Mn, Fe, Co, Ni, Cu and Zn. Numerous experiments were carried out to elucidate the behavior of oxidation and the long-term stability of spinel coatings[10]. Doping Cu into the Mn-Co device will improve sintering, electrical conductivity and TEC compatibility with other components. Solgel, plasma spraying, magnetron sputtering, electrophoretic deposition (EPD) and electroplating methods are used for spinel coating[11].

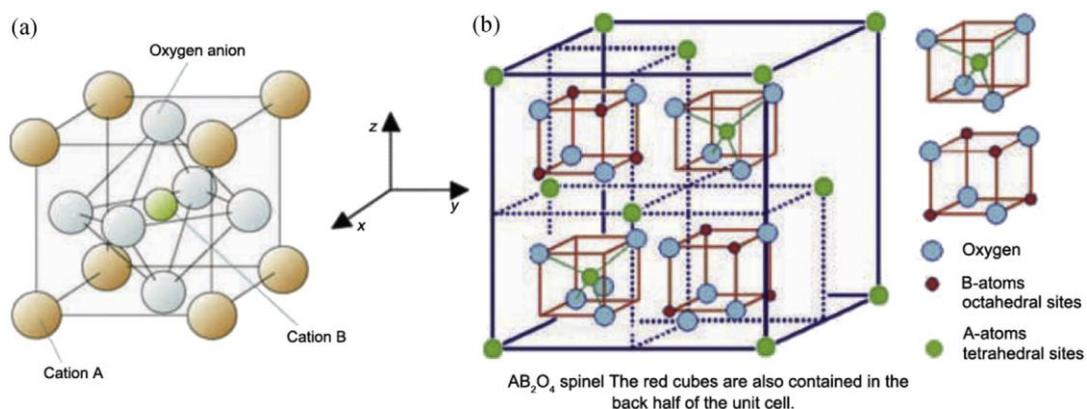


Fig 2: Unit cells of (a) Perovskites and (b) spinel

VI. STRENGTHS AND DRAWBACKS

Rare earth perovskite and composite spinel oxides are two commonly studied coating materials for SOFC interconnects[8]. These coating materials demonstrate good electrical conductivity and excellent CTE fit with adjacent components, but good oxidation resistance remains challenging in SOFC operating conditions. Recent work by Gavrilov et al[18]. further enhanced the oxidation resistance in composite spinel coating by adding REOs, especially Yttrium (Y) additives, to the Mn-Co spinel. The Y-doped specimens, however, do not show a promising ASR value as compared to those without Y-doping[19].

Table 1: Comparison of the various coating materials in terms of electronic conductivity, Cr migration inhibition and oxidation rate reduction as well as simplicity of the coating process.

Coating material	Electronic conductivity	Cr migration Inhibition	Oxidation rate reduction	Simplicity of deposition
Perovskite	Good	Fair	Poor	Fair
Composite spinel oxide	Good	Good	Fair	Good

VII. PROTECTIVE LAYER DEPOSITION TECHNIQUES

Deposition technique choice depends on the types of coating materials, the desired surface morphology and the budget available[3]. Specific methods are used for depositing various spinel forms, with various compositions[9]. This section discusses many deposition techniques including sputtering of magnetrons, sol-gel dip-coating, plasma spraying and electro deposition techniques for applying protective coating to interconnections of ferritic stainless steel.

MAGNETRON SPUTTERING: Magnetron sputtering is a technique of physical vapor deposition commonly used for developing SOFCs. Atoms of the target materials are moved to the gas phase, without chemical reaction, by physical processes[18]. The goal source is usually in solid or molten shape. Various process parameters, such as temperature can be used to obtain both porous and dense strata. Alternatively, the deposited layer's surface morphology can be easily adapted to varying deposition conditions. This method however needs high costs apparatus and low deposition levels of a few nano meter per minute[8][18].

SOL- GEL DIP-COATING: Sol-gel method is a cheap and low-temperature coating technique that allows for fine control of the coating properties, particularly of the coatings' chemical composition. Precursors, normally a colloidal solution (sol), are progressively evolving towards a gel-like system of discrete particles and continuous polymer networks[20]. Dip-coating or spin-coating typically deposits protective coating, followed by subsequent heat treatment to dry out the residual solvent. The subsequent sintering process can further manipulate the micro-structural and mechanical properties[21].

Protective coatings with excellent electrical conductivity, high thermal and structural stability, as well as good thermal expansion match to ferritic stainless steel interconnections were developed in a slurry-based coating followed by thermally grown (Mn, Co)₃O₄ layers, in particular with a nominal composition of Mn_{1.5}Co_{1.5}O₄. Doping a transition metal like Cu, Ni and Fe into the spinel Mn_{1.5}Co_{1.5}O₄ can tailor the coating material. Sol-gel dip-coated spinel layer Cu-doped Mn-Co exhibits excellent packing density, good oxidation resistance and effectively suppresses the migration of Cr-containing species from the interconnection to the cathode[22][21][6].

ELECTRODEPOSITION: Metallic alloy electrodeposition followed by subsequent heat treatment is a cost-effective method for preventing Cr poisoning of the cathode and maintaining high conductivity during SOFC activity[23]. Generally, electrodeposition can be classified as Electrolytic Deposition (ELD) and Electrophoretic Deposition (EPD).



- a) Electrolytic Deposition (ELD): Electrolyte deposition (ELD) is an electrochemical process that produces on conductive surfaces a thick and uniform metallic coating. Both cathode into an electrolyte cell (Substratum to be plated) and anode (target metal to be plated) are immersed in an aqueous solution containing the metal ions targeted[24]. Mn-Co spinel layer can be produced by Co electrodeposition or sequential electrodeposition, followed by treatment with heat. A deep bi-layered microstructure coating by Co depositing of Mn-Co alloy, an external MnCo₂O₄ layer and an internal Cr-rich oxide layer were successfully prepared[25].
- b) Electrophoretic Deposition (EPD): Electrophoretic deposition (EPD) is a technique of colloidal processing, requiring simple apparatus and short forming time. Despite being a wet process, EPD offers easy control of thickness and morphology through simple adjustment of deposition time and applied voltage[8]. Thin films are produced by using an externally applied electric field to coagulate a dense layer of colloidal particles on the surface of the substrates[25]. It is possible to produce a relatively dense, continuous, and crack-free Mn-Co spinel coating with good packaging density, good oxidation resistance and high electrical conductivity via EPD, with reduced sintering atmosphere[23].

PLASMA SPRAY PROCESS: Plasma spraying is a highly flexible process because it is possible to deposit coatings of several material types including metals, ceramics, and composites[26]. The basic procedure is to inject powders of feedstock into a plasma flux. The molten or semi-molten powder forms would subsequently be projected toward a substratum, followed by flattening, rapid solidification, and cooling[27]. As a result, these molten particles create overlapping splats, so that the substrate is coated. Air, low pressure or vacuum spraying can be performed. Plasma-sprayed coatings may be perceived as special porous structures, due to the presence of multi-type pores (or cracks)[28].

Plasma spray coating technique was used to create a protective spinel layer on flat substrates and meshes of the SS-430. The study of oxidation of coated coupons confirmed good oxidation resistance compared with uncoated coupons which indicate the coating 's gas barrier properties[29]. The conductivity studies showed that coated mesh and plates had comparatively low ASR. However, even after 72 h, the ASR was stable suggesting the long term plasma spray coating stability[17][28].

VIII. STRENGTHS AND DRAWBACKS

Due to its ability to cover large surfaces with high purity and surface smoothness, magnetron sputtering is commonly used[30]. However, the difficulty of controlling the stoichiometric of the film and the growth of complex compounds constrains this process[31]. Sol-gel dip-coating is economically viable and, despite difficulties, involves simple equipment set-up in controlling uniformity of coating quality and thickness[21]. The deposition techniques discussed in this paper is compared and tabulated in Table 2 in terms of their process simplicity, set-up cost, coating quality.

Table 2: Comparison of various coating techniques; in terms of process simplicity, set-up cost, coating quality.

Coating techniques	Process simplicity	Set up costs	Coating Quality
Magnetron sputtering	Fair	Very Expensive	Good
Sole-gel dip-coating	Good	Inexpensive	Poor
Electrolytic deposition	Good	Inexpensive	Good
Electrophoretic deposition	Good	Inexpensive	Good
Plasma spraying	Good	Expensive	Good

IX. CONCLUSION

The main technological constraint in marketing the planar SOFCs is the production of low-cost oxidation resistance interconnecting materials with strong Cr retention capability[32]. Due to its advantage in terms of TEC match with other SOFC parts, oxidation resistance, electrical conductivity, manufacturability, and cost, Ferritic stainless steel is the preferred metallic candidates. In comparison with reactive element oxides and rare earth perovskites, composite spinel coating is more effective in retarding both Cr outward and O₂ inward diffusion to overcome rapid Cr₂O₃ scale growth and Cr evaporation in a SOFC intermediate temperature environment. Mn-Co spinel is, in particular, the most promising coating material for SOFC interconnections, despite the spreading of coating / metal interfaces and with the time oxidation rate increases. Therefore it is necessary to improve the long-term stability of interconnecting materials. It is also important to select the appropriate deposition technique, because the microstructure and surface quality of the coating is often correlated with cell performance. When making interconnections with SOFC, the deposition techniques shall focus not only on costs but also on their ability to operate on interconnections with complicated gas flow channels. Technique for plasma spraying is effective in meeting both criteria for the manufacture of spinel-coated stainless steel ferritics for intermediate temperature SOFC interconnecting applications[27].

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