

Effect of Fe-Doped YSZ Electrolyte on the Performance of Co-Sintered Solid Oxide Cells

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Abstract

Solid Oxide Cell (SOC), as a class of highly efficient electrochemical energy conversion devices, has attracted considerable attention in the energy field. To address the challenges of high energy efficiency yet insufficient stability during industrial deployment, this study employs an Fe₂O₃ doping strategy to investigate the modification of yttria-stabilized zirconia (8YSZ) electrolytes. The complex interaction mechanism between Fe₂O₃ and 8YSZ is thoroughly analyzed, and SOC cells containing Fe-doped YSZ electrolytes are fabricated through a co-sintering process. In the fuel cell (FC) mode using hydrogen as fuel, the maximum power output of the cell reaches 0.86 W cm⁻² (750°C). In the electrolysis cell (EC) mode, the electrolysis current density can reach -0.85 A cm⁻² (750°C @ 1.3 V), with stable operation exceeding 500 hours in EC mode. Furthermore, by fabricating large-scale SOC cells of 12 cm × 12 cm, the effectiveness and scalability of this optimization method are further validated. The results of this study provide a novel research strategy for enhancing the initial performance and stability of SOC, advancing the industrial application process of SOC.

Full Text

Preamble

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Effect of Fe-Doped YSZ Electrolyte on the Performance of Co-Sintered Solid Oxide Cells

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Abstract

[Background] Solid oxide cells (SOCs), as highly efficient electrochemical energy conversion devices, have attracted significant attention in the energy sector. **[Purpose]** To address the challenges of high efficiency but insufficient stability during industrial deployment, this study employs an Fe₂O₃ doping strategy to modify yttria-stabilized zirconia (8YSZ) electrolytes. **[Methods]** We conducted an in-depth analysis of the complex interaction mechanisms between Fe₂O₃ and 8YSZ, and prepared SOC cells containing Fe-doped YSZ electrolytes through a co-sintering process. **[Results]** In fuel cell (FC) mode using hydrogen fuel, the maximum power output reached 0.86 W cm⁻² at 750°C. In electrolysis cell (EC) mode, the electrolysis current density achieved -0.85 A cm⁻² at 750°C and 1.3 V, with stable operation exceeding 500 hours in EC mode. Furthermore, the scalability of this optimization method was validated by successfully fabricating large-scale 12 cm × 12 cm SOC cells. **[Conclusions]** These results provide a novel research strategy for enhancing both the initial performance and long-term stability of SOCs, advancing the industrial application prospects of this technology.

Keywords: Fe dopant, Co-sintering, XANES, Electrolyte, Solid oxide cells

Introduction

Solid oxide cells (SOCs) represent highly efficient electrochemical devices that directly convert chemical energy to electrical energy, offering both high conversion efficiency and environmental compatibility [1]. However, commercialization is constrained by two major issues: suboptimal performance and inadequate long-term operational stability [2-3]. Anode-supported solid oxide cells (AS-SOCs) constitute a common design architecture, where the electrolyte and cathode layers are attached to a thick anode substrate [4]. Currently, yttria-stabilized zirconia (YSZ) is widely employed as the SOC electrolyte material due to its excellent pure ionic conductivity and chemical stability in redox atmospheres [5]. Nevertheless, YSZ presents significant processing challenges, requiring sintering temperatures as high as 1350-1400°C, which adversely affects other functional layers during cell fabrication. More critically, even at these temperatures, conventional processing struggles to achieve ideal densification of YSZ [6].

Recent studies have employed thin-film techniques such as pulsed laser deposition (PLD) and vacuum plasma spraying (VPS) to achieve near-full densification of electrolytes at lower temperatures, followed by low-temperature post-treatment [7-8]. However, these methods suffer from complex procedures and

high costs, making them unsuitable for industrial production. In contrast, multilayer co-sintering remains the most promising technical route due to its advantages for scalable manufacturing [9].

To enable SOC fabrication via multilayer co-sintering, researchers have optimized the sintering process by adding sintering aids such as alumina and transition metal oxides [10]. The Gauckler group first demonstrated the promoting effect of sintering aids on YSZ electrolyte densification in AS-SOC systems [11]. However, existing literature shows contradictory conclusions regarding the impact of dopants on electrolyte performance. Herle and Vasquez observed no significant change in YSZ conductivity in air with 1 at.% Mn and 2 mol% NiO doping [12], whereas Kawada et al. reported a slight conductivity enhancement with Mn doping [13]. Most studies indicate that transition metal doping negatively affects the intrinsic properties of YSZ [14-15]. These conflicting results suggest that while doping effectively improves sintering behavior, the complex interaction mechanisms between dopants and electrolyte materials require further investigation.

Subsequent research has gradually revealed the positive effects of Fe doping on electrolyte conductivity and SOC performance. Zhang et al. demonstrated that Fe_2O_3 doping effectively suppresses the detrimental effects of SiO_2 impurities in ceria-based electrolytes, significantly improving grain boundary conductivity in silicon-containing materials [16]. Ghuman et al. employed molecular dynamics simulations to systematically investigate the segregation behavior of Al and Fe impurities at YSZ grain boundaries, finding that Fe impurities exhibit a certain segregation tendency that stabilizes the YSZ crystal structure. As the diffusion barrier for ions at grain boundaries is enhanced, the overall conductivity of YSZ is further affected [17]. However, the existing form, distribution patterns, and specific effects of Fe doping on YSZ crystal structure remain unclear. Moreover, systematic studies on Fe diffusion behavior in electrode materials and the long-term stability of doped electrolytes are lacking.

Based on these considerations, this study employs an Fe_2O_3 doping strategy to modify YSZ electrolytes and optimizes SOC cell fabrication through co-sintering processes. The research aims to achieve two key objectives: (1) enhance ionic conductivity and low-temperature sintering densification through optimized doping concentrations, enabling thinner electrolyte layers; and (2) effectively suppress the formation of resistive phases at the YSZ electrolyte-GDC interface during co-sintering. We systematically investigated the effects of Fe doping on the microstructure of YSZ powders and electrolytes, and conducted in-depth studies on the electrochemical performance and long-term stability of SOCs constructed with Fe-doped YSZ co-sintered electrolytes.

1.1 Sample Preparation

This study employed 5 cm \times 5 cm anode-supported SOCs with an effective electrode area of 16 cm². The cell architecture consisted of a NiO-3YSZ support

layer, NiO-8YSZ hydrogen electrode functional layer, Fe-doped 8YSZ electrolyte layer, GDC ($\text{Ce}_{0.9}\text{Gd}_{0.1}\text{O}_{1.95}$) barrier layer, and LSC ($\text{La}_{0.6}\text{Sr}_{0.4}\text{Co}_{0.2}$)-GDC oxygen electrode.

To prepare Fe-doped 8YSZ electrolyte powder, 95 g of 8YSZ powder and 5 g of Fe_2O_3 were weighed and subjected to high-energy ball milling, followed by pre-sintering at 800°C . The pre-sintered powder was then dispersed in a slurry prepared from terpineol, carbitol, PVB binder, DOP plasticizer, SN348 defoamer, and other additives to produce the electrolyte slurry.

The complete SOC fabrication process is illustrated in Figure 1 [Figure 1: see original paper]. Since Fe acts as an effective sintering aid that significantly reduces the sintering temperature of YSZ while suppressing excessive grain growth, it facilitates the formation of a uniform fine-grained structure and enhances the densification of the functional layer, electrolyte, and barrier layer during co-sintering [18-19]. Consequently, SOCs with Fe-8YSZ electrolytes can be prepared via co-sintering. Unlike the multi-stage heating and cooling procedures required for separate sintering of the hydrogen electrode and GDC, the co-sintering method involves only a single thermal cycle, substantially reducing energy consumption and processing time.

In the fabrication process, the GDC barrier layer was screen-printed onto the Fe-8YSZ-coated half-cell substrate. The composite structure was then heated to 1300°C and held for 4 hours to complete sintering, yielding a half-cell with the barrier layer. Subsequently, the LSCF-GDC oxygen electrode was screen-printed onto the half-cell and fired at 1000°C for 3 hours to obtain the full cell. Compared to the traditional process requiring separate screen printing of the hydrogen electrode functional layer and conventional 8YSZ electrolyte on the green substrate with two repeated thermal cycles before GDC printing and oxygen electrode fabrication, the introduction of Fe significantly reduces overall processing time.

1.2 Testing Methods

Field emission scanning electron microscopy (FE-SEM) was employed to characterize the microstructure of Fe-8YSZ electrolytes and the fabricated full cells. Secondary electron detection mode was used with an accelerating voltage of 2-5 kV. Compositional analysis was performed using energy-dispersive X-ray spectroscopy (EDS) attached to the SEM at an accelerating voltage of 15 kV. X-ray absorption near-edge structure (XANES) spectroscopy was utilized to determine the valence state of Fe, and hard X-ray microfocus testing was conducted in fluorescence mode at beamline 15U of the SSRF to investigate Fe diffusion behavior during cell sintering. Hard XAS data normalization and processing were performed using the ATHENA software package according to established protocols.

For FC performance evaluation, open-circuit voltage and power density were used as metrics. At 750°C , samples were reduced for 2 hours using a H_2/N_2

gas mixture (1:3 volume ratio). Electrochemical performance testing was then conducted using a Maccor4000 charge-discharge device. Nickel mesh and gold mesh served as current collectors on the hydrogen and oxygen electrode sides, respectively, with gold sealing rings employed to prevent gas leakage.

For EC performance assessment, I-V curves were measured at 750°C. On the hydrogen electrode side, a mixture of O₂ and H₂ was used to generate steam in situ (70% H₂O and 30% H₂), while air was supplied to the oxygen electrode side to facilitate rapid product gas diffusion. Electrochemical impedance spectroscopy (EIS) was performed using an Autolab PGSTAT302N electrochemical workstation under open-circuit conditions, with frequency range set from 10⁻¹ to 10⁵ Hz and AC amplitude of 5 mV. The obtained impedance data were fitted and analyzed using ZView software.

2 Results and Discussion

Microstructural Characterization of Fe-8YSZ Electrolyte

To systematically investigate the crystal structure and phase composition of Fe-8YSZ composites, X-ray diffraction (XRD) analysis was performed, with results shown in Figure 2. The XRD patterns reveal that both Fe-doped and pure 8YSZ exhibit peaks corresponding to cubic YSZ (PDF 30-1468), indicating a cubic fluorite structure without detectable secondary phases. This confirms that Fe doping does not alter the crystal structure or compromise electrolyte purity. A slight shift of diffraction peaks toward higher 2θ values is observed in Fe-doped 8YSZ, indicating lattice distortion when Fe enters the 8YSZ lattice to maintain charge neutrality and structural stability. This primarily results from Fe ions substituting for Zr⁴⁺, which reduces the interplanar spacing and shifts diffraction peaks to higher angles [20]. The strong interfacial interactions, such as covalent bond formation or lattice stress transfer, significantly influence the electrochemical properties.

To determine the state of Fe in the YSZ bulk phase, X-ray photoelectron spectroscopy (XPS) was employed to analyze the chemical form of Fe in Fe-doped 8YSZ thin films, with results presented in Figure 2(b). Distinct Fe 2p core levels were observed, confirming successful incorporation of Fe into the 8YSZ structure. The XPS results verify that Fe exists in mixed Fe²⁺/Fe³⁺ valence states. Theoretically, due to the significant difference in atomic radius between Fe and Zr, Fe doping may introduce oxygen vacancies and thereby enhance ionic conductivity.

SEM imaging and EDS analysis were conducted on Fe-doped 8YSZ electrolyte powder. As shown in Figure 3 [Figure 3: see original paper], no significant coarsening is observed in Fe-doped 8YSZ electrolyte, with morphology similar to pure YSZ. EDS mapping characterization confirmed uniform distribution of Fe elements across YSZ particles without particulate formation on the surface, indicating successful incorporation of Fe into YSZ.

SOC Cell Structure and Electrochemical Performance

Comparative analysis of cross-sections from SOC's prepared by conventional versus optimized processes reveals clear structural differences, as shown in Figure 4 Figure 4: see original paper and (b). The Fe-YSZ electrolyte exhibits a thinner morphology with significantly higher densification. This characteristic stems from Fe's optimization of 8YSZ sintering behavior, where Fe ion introduction reduces sintering activation energy and promotes atomic diffusion, enabling closer particle packing during sintering and effectively reducing porosity to form thin, dense electrolyte layers [22]. Additionally, the GDC and YSZ layers prepared by one-step co-sintering demonstrate better interfacial bonding compared to step-sintered cells, likely due to atomic diffusion of Fe during sintering that promotes interfacial sintering and bonding.

To investigate the influence of Fe doping on elemental diffusion and sintering behavior during SOC fabrication, microfocus scanning was employed to obtain Fe XANES spectra across cell cross-sections. The distribution, electronic structure, and local geometric configuration of Fe in the functional layer, electrolyte, and barrier layer were systematically studied through layer-by-layer scanning from bottom to top. As shown in Figure 5 [Figure 5: see original paper], Fe diffusion occurs during co-sintering of Fe-doped 8YSZ electrolyte-based SOC's. Fe is distributed in both the upper barrier layer and lower functional layer near the electrolyte, with strong Fe signals observed near the GDC barrier layer interface. This indicates significant upward diffusion of Fe during sintering, with Fe readily reacting with GDC and entering the Ce lattice to dope it. This doping modifies GDC sintering activity, resulting in improved densification of the GDC barrier layer [23]. At the 8YSZ electrolyte layer interface, Fe primarily exists in Fe-O bonding configurations. The relatively uniform Fe distribution in 8YSZ, combined with the absence of impurity peaks in XRD, suggests that Fe mainly enters the YSZ lattice by occupying Zr sites and binding with O. The atomic radius mismatch between Fe and Zr exposes more oxygen vacancies, thereby enhancing ionic conductivity. At the electrolyte/functional layer interface, Fe signals are weaker but still present as Fe-O, indicating preferential Fe penetration within YSZ [24]. Moreover, with hydrogen introduction maintaining a reducing atmosphere in the functional layer, Fe remains stably as Fe-O rather than forming Fe-Ni alloys. This unsaturated Fe-O state enhances Ni stability and suppresses Ni agglomeration and coarsening that cause cell deactivation [25]. The overall performance improvement results from Fe occupying Zr sites in YSZ and Ce sites in GDC, providing additional oxygen vacancies and higher ionic conductivity that accelerate OER/ORR reaction rates, which also plays an essential role in long-term electrode stability.

To compare the electrochemical performance of both cell types, FC testing was conducted at 750°C. The conventional 8YSZ electrolyte cell exhibited an open-circuit voltage (OCV) of 1.06 V, while the Fe-8YSZ electrolyte cell showed an OCV of 1.15 V, close to the theoretical value and indicating good gas tightness. The OCV difference is attributed to different electrolyte compositions, as dense

electrolytes effectively block fuel and oxidant gas crossover. Gas permeation causes direct chemical reaction rather than electrochemical energy release, leading to chemical energy loss and “fuel crossover” phenomena that reduce OCV [26]. Dense electrolytes minimize this gas permeation, reducing energy loss and maintaining high OCV.

As shown in Figure 6 Figure 6: see original paper, the Fe-doped YSZ electrolyte SOC prepared by the optimized process achieved a maximum power density of 0.86 W cm^{-2} , representing a 51% improvement over the conventional 8YSZ electrolyte SOC (0.57 W cm^{-2}). EC mode steam electrolysis performance is presented in Figure 6(b). At 750°C and 1.3 V, the Fe-doped 8YSZ electrolyte SOC reached a current density of -0.85 A cm^{-2} , a 23% improvement compared to the conventional cell (-0.69 A cm^{-2}).

To elucidate the impact of Fe-doped YSZ electrolyte on SOC electrochemical performance, impedance spectroscopy was performed on both cell types, with results shown in Figure 6(c). An equivalent circuit model was used for fitting analysis, where R1 corresponds to the high-frequency intercept reflecting ohmic resistance, while R2, R3, and R4 represent charge transfer and mass transfer resistances at electrode surfaces and interfaces, collectively constituting polarization resistance [27]. The Fe-doped YSZ electrolyte SOC exhibits significantly lower ohmic and polarization resistances than the conventional cell. Since YSZ primarily conducts oxygen ions, higher ionic conductivity reduces ohmic resistance. Fe doping introduces additional oxygen vacancies or modifies vacancy distribution, promoting oxygen ion migration and reducing ohmic resistance. The thinner, denser Fe-doped YSZ electrolyte structure shortens ion transport paths while preventing gas permeation, further reducing ohmic resistance. Fe doping also promotes elemental diffusion between the electrolyte and GDC layer/electrodes, optimizing interfacial structure stability and reducing interfacial contact resistance and polarization effects. The DRT spectrum in Figure 6(d) shows peaks corresponding to different electrode reaction processes, with peak areas reflecting impedance values. The low-frequency region (10^1 - 10^2 Hz) relates to electrode gas diffusion, while high-frequency peaks (10^3 - 10^4 Hz) correspond to electrode electrochemical reactions [28]. The Fe-doped YSZ electrolyte SOC shows substantial improvement in both electrochemical reaction impedance and gas diffusion impedance, benefiting HER/ORR reactions.

To ensure long-term operation, durability testing was conducted on the optimized Fe-doped YSZ electrolyte SOC. As shown in Figure 7 [Figure 7: see original paper], the cell operated stably for over 500 hours at a constant current of 0.5 A cm^{-2} in EC mode, with a degradation rate of only 2.3%. In contrast, the conventional YSZ electrolyte SOC exhibited obvious degradation after approximately 40 hours with a 5% degradation rate. This consistency with XANES analysis demonstrates that Fe diffusion enhances GDC barrier layer densification, effectively suppressing interfacial side reactions between the LSC oxygen electrode and YSZ electrolyte (such as formation of resistive LaZr_2O_7 phases), thereby improving chemical stability at the electrolyte-electrode interface [29].

The unsaturated Fe-O compounds formed after Fe diffusion to the electrode also interact with active Ni centers, suppressing Ni particle agglomeration and migration issues and significantly enhancing long-term durability [30].

To validate the industrial scalability of Fe-doped YSZ electrolyte SOCs, a 12 cm \times 12 cm cell was fabricated using the same method, as shown in Figure 8 Figure 8: see original paper. The photograph demonstrates tight bonding between the large-area Fe-doped YSZ electrolyte and electrode layers with a flat, defect-free surface. Mechanical cutting into four 5 cm \times 5 cm cells for cross-sectional SEM analysis (Figure 8(b)) revealed uniform electrolyte thickness and consistent microstructure. Electrochemical performance testing of three cells (Figures 8(c) and 8(d)) showed highly consistent FC and EC performance. This excellent process stability confirms that Fe-doped YSZ electrolyte SOCs prepared by co-sintering meet industrial production requirements. This study not only provides an important reference for SOC technology commercialization but also expands the application of doped electrolytes in large-scale cell fabrication through process innovation.

This work systematically investigated Fe₂O₃ doping modification of SOC electrolytes, revealing the complex interaction mechanisms between Fe and 8YSZ. Through co-sintering optimization, large-scale SOCs with Fe-doped 8YSZ electrolytes were successfully fabricated. Experimental results demonstrate that Fe diffusion during cell preparation significantly improved electrolyte and barrier layer densification while suppressing formation of resistive interfacial phases. At 750°C, the cell achieved 0.86 W cm⁻² maximum power in FC mode and -0.85 A cm⁻² electrolysis current density at 1.3 V in EC mode, with stable operation exceeding 500 hours in EC mode. The successful fabrication of 12 cm \times 12 cm cells further validated process scalability. These findings provide new strategies for improving both initial performance and long-term stability of SOCs, paving a viable path toward industrial application through optimized material design and fabrication processes.

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Author Contributions

YANG Chunhui: Experimental design and execution, initial manuscript writing
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LI Rengang: Cell structural characterization
LI Aiguo, HE Yan, ZHANG Jichao: XANES testing
ZHANG Linjuan, LIN Xiao, WANG Jianqiang: Project conceptualization and supervision, experimental design guidance, data analysis, and manuscript revision

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