






Electrophoretic deposition of YSZ layers on pyrolytic graphite and a porous anode substrate based on NiO–YSZ

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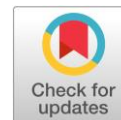
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Abstract

Solid oxide fuel cells are promising hydrogen energy devices. The goal of this research was to create ceramic layers for SOFCs based on yttria-stabilized zirconia (YSZ) and to investigate their parameters. YSZ ceramic layers with a thickness of 5.14 μm on a porous NiO–YSZ substrate and 7 μm on pyrolytic graphite were obtained by electrophoretic deposition. X-ray diffraction and electron microscopy were used to determine the composition, structure, and morphological features of ceramic layers. The effects of the substrate's nature, the degree of dispersion of the initial YSZ powder, and the heat treatment conditions on the properties of the ceramic layer YSZ were considered.

Keywords

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Key findings

- Ceramic layers based on zirconium dioxide stabilized with yttrium oxide were obtained by electrophoretic deposition.
- The thickness of the obtained ceramic layers was 5.14 μm on a porous NiO–YSZ anode substrate and 7 μm on pyrolytic graphite.
- The dependence of the EPD process efficiency on the precursor dispersion degree was established.

1. Introduction

Currently, there is an increased interest in the development of novel electrical energy storage and production facilities, in particular, power plants based on solid oxide fuel cells (SOFC) [1, 2]. The advantages of such devices are the absence of harmful emissions and thermal pollution, higher efficiency, scalability, noiselessness in operation, and low energy consumption [3–5]. However, the mass production and use of solid oxide fuel cells are constrained by their high operating temperatures (1073–1273 K), at which the conductivity of the main component of the SOFC – the oxygen-ion electrolyte – reaches a sufficient level. At high temperatures, degradation processes and chemical interactions among components in the membrane-electrode block of fuel cells are accelerated [6]. All this imposes strict requirements on the materials used, causes a high cost of

SOFCs, and significantly limits their further production [7]. Application of a thin-film electrolyte based on zirconium dioxide stabilized with 3 to 10 mol.% of yttrium oxide is an effective solution to the problem of reducing the operating temperature of the SOFC since decreasing the thickness of the electrolyte leads to a decrease in the internal resistance of the element and an increase in its power [8, 9]. One of the most rapidly expanding fields in electrochemistry right now is the development of fuel cells using a thin-film solid oxide electrolyte [10, 11]. The electrophoretic deposition (EPD) of a solid electrolyte on the surface of the anode or cathode appears to be the most appealing among the potential techniques for producing such films because it is cost-effective and does not require complicated hardware design [12–14]. The cost of obtaining ceramic layers of YSZ will be significantly reduced by using precursors from

domestic production, such as high-quality powder of the DCI-1 brand with an affordable price and large volumes of industrial production. The EPD method allows controlling the morphology and output parameters of the resulting ceramic coatings and obtaining layers with good adhesive properties and with a higher density compared to coatings obtained by other methods [15, 16]. The purpose of this research was to obtain ceramic layers of YSZ and analyze their morphology and crystal structure. Electrophoretic deposition was carried out on pyrolytic graphite and a porous NiO-YSZ anode substrate.

2. Experimental

The EPD process includes three main stages. The first stage is the preparation of a stable suspension of the applied oxide material in a suitable liquid dispersion medium. The second one is the application of an electric field to the suspension, causing the movement of the particles to the electrode and their deposition on it. The last one is the drying and sintering of the resulting coating.

2.1. Preparation of the suspension

A 1:1 mixture of acetylacetone $\text{CH}_3\text{COCH}_2\text{COCH}_3$ (p.a.) and isopropanol $\text{CH}_3\text{CH}(\text{OH})\text{CH}_3$ (puriss) was used as the starting reagent for the preparation of a stable suspension. As a polymer binder that prevents cracking, a BMA-5 (butyl methacrylate-methacrylic acid copolymer, SRI Polymers, Russia) copolymer was added to the prepared mixture, which was dissolved in the mixture for 48 h. Commercial zirconium dioxide powder stabilized with yttrium oxide ($\text{Zr}_{0.97}\text{Y}_{0.03}\text{O}_{2-\delta}$) grade DCI-1, (JSC ChMP, Russia), with an average particle size of $75 \mu\text{m}$, was milled in a planetary mill, FRITSCHE PULVERISETTE 7, for 4 h at 500 rpm. Grinding with zirconium dioxide balls was carried out in an acetone environment. During the grinding process, 4 samples of YSZ powder were taken at an interval of 1 h. Figure S1 shows the X-ray diffraction pattern of the commercial powder DCI-1. Then, a portion of the milled powder in the amount of $65 \text{ g}\cdot\text{l}^{-1}$ was added to the prepared dispersion medium and dispersed on a submersible ultrasonic disperser UZDN-A at an emitter frequency of 22 kHz for 15 min.

2.2. Used substrate material

Pyrolytic graphite and porous anode substrates based on NiO-YSZ with a nickel foil sublayer were used as substrate materials for electrophoretic deposition. A nickel substrate in the form of a foil with a thickness of $100 \mu\text{m}$ provided the conductive paths, since NiO-YSZ is a non-conductive substrate [17] (Figure 1a). Pyrolytic graphite substrates were obtained by chemical vapor deposition (CVD). The thickness of the substrates was 1 mm. The ohmic resistance of the material was $2 \pm 0.4 \Omega$. Graphite substrates were used to study the influence of the nature of the substrate material on the morphology of the ceramic coating. The multilayer joint rolling of films method was used to create

porous anodic NiO-YSZ substrates (mass ratio of precursors NiO:YSZ:starch = 60:40:20). The thickness of NiO-YSZ was 0.87 mm.

2.3. Electrophoretic deposition of YSZ

Electrophoretic deposition was carried out at 298 K with a voltage of 60 V applied between the electrodes in two cycles of 10 minutes. The cathode and anode were fixed in a specially designed holder at a distance of 10 mm from each other and immersed in a reactor with a prepared suspension (Figure 1b). Pyrolytic graphite was used as a counter electrode. The resulting coatings underwent heat treatments at 973 K, 1173 K, and 1573 K for 2 h.

X-ray phase analysis was performed on a Rigaku D/MAX-2200VL diffractometer. The survey was conducted in $\text{Cu K}\alpha$ radiation at $10^\circ \leq 2\theta \leq 90^\circ$. On a JEOL JSM-5900 LV microscope, morphological characteristics of the obtained YSZ layers were examined using scanning electron microscopy (SEM). The MEASURER software was used to process the obtained SEM data in order to establish the average particle size.

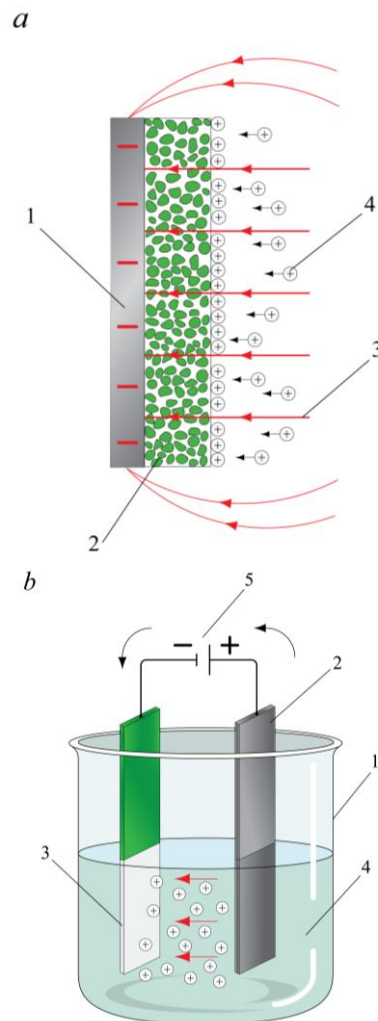


Figure 1 Electric field distribution scheme on the NiO-YSZ surface (a), where 1 – nickel foil, 2 – porous anode substrate based on NiO-YSZ, 3 – electric field lines, 4 – charged ceramic YSZ particles. Schematic diagram of an installation for electrophoretic deposition (b), where 1 – reactor, 2 – counter electrode, 3 – porous anode substrate based on NiO-YSZ, 4 – suspension, 5 – electric current source.

3. Results and Discussion

One of the main aspects of obtaining a high-quality coating in the EPD process is the presence of a stable dispersion of YSZ. The formation of a dense, well-anchored coating is possible only if the EPD proceeds in sedimentationally and aggregatively stable suspensions [18–20]. The stability of the suspension is influenced by the choice of the dispersion medium and the ratio of the initial components. According to [21–24], it is preferable to carry out electrophoretic deposition in non-aqueous organic media, as alcohols, ketones, and their mixtures are most often used. When conducting EPD in aqueous media, electrochemical reactions occur that are accompanied by the release of gasses that significantly affect the process [25]. For this reason, an anhydrous dispersion medium consisting of acetylacetone and isopropanol has proven itself in the best way. This is due to the fact that isopropanol is less hygroscopic than ethanol, which ensures the stability of the physicochemical characteristics of the suspension. Acetylacetone contains the carbonyl group, which is capable of donor-acceptor interaction with the surface of the oxides [26].

Indeed, the stability of such suspensions, according to light transmission data at wavelengths of 540, 800, and 1000 nm, was at least 50 h. The spectral methods, in particular, spectrophotometry, quite accurately indicate the beginning of the sedimentation process in the suspension volume, which is caused by the aggregation and coagulation of particles over time. The gradual removal of small particles from the suspension leads to an increase in transmission at the beginning at small wavelengths, with the gradual settling of large particles at large wavelengths. Figure 2a shows a plot of the stability of the suspension over time.

An important condition for successful EPD is the size of the YSZ particles in the suspension and the absence of aggregation of these particles in the dispersion medium. It is worth noting that with a size of less than 1 μm , particles can remain in suspension for a long time due to Brownian motion [27]. Also, for a positive result in conducting EPD, it is necessary that the YSZ powder has a narrow particle size distribution. To achieve this, grinding in planetary mills and ultrasonic dispersion are used.

According to the results, an increase in the powder grinding time leads to a decrease in the average particle size from 4 μm to 0.9 μm with a single-modal distribution (Figure 2b). The percentage of particles with a size of from 2.5 to 7.5 μm after 1 h of grinding was 84%, but after 4 h of grinding, 90% of particles were from 0.5 to 2 μm (Figure 2c).

After dispersing the ground ceramic particles from their suspensions by the EPD method, YSZ-based layers with a thickness of up to 7 μm on pyrolytic graphite were successfully obtained. However, the high-quality coatings without cracks and with a small particle size distribution were obtained from suspensions containing powder ground

in a planetary mill for 4 h on NiO–YSZ substrates up to 5.14 μm thick.

The X-ray phase analysis showed that the formed ceramic coating consists of two phases: zirconium dioxide stabilized with yttrium oxide in an amount of 3 mol.% (95.68%) and zirconium dioxide in an amount of 4.32%, which is apparently due to the presence of residual ZrO_2 in the initial commercial powder DCI-1. Figure 3 shows the X-ray diffraction pattern of the YSZ sample deposited on a porous NiO–YSZ anode substrate. According to the XRD data, zirconium dioxide, stabilized by 3 mol.% yttrium oxide, has a tetragonal structure with crystal lattice parameters $a = 3.6249(3)$ \AA , $c = 5.1275(1)$ \AA with a predominant growth direction (101). The phase of individual zirconium dioxide has a cubic lattice with a parameter $a = 5.1522(1)$ \AA and a predominant growth direction (111).

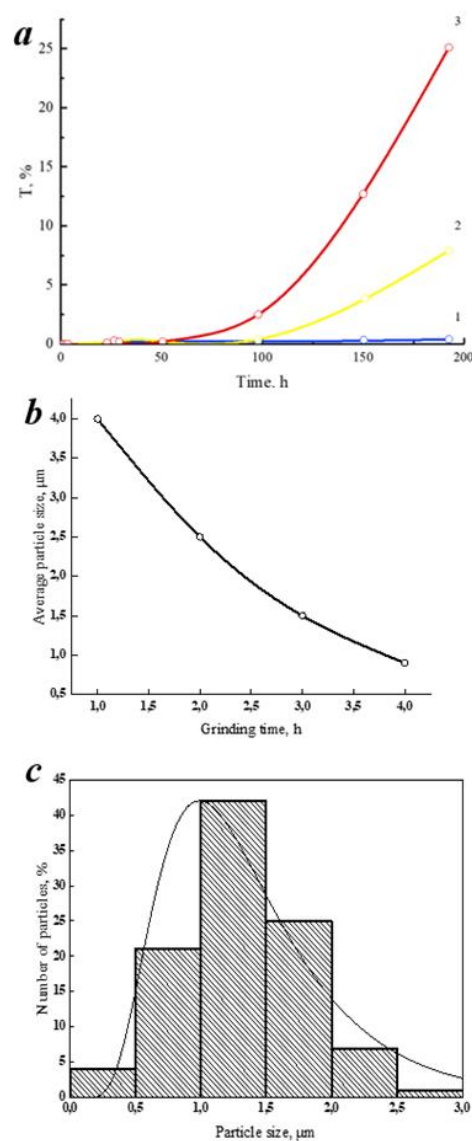


Figure 2 Dependence of the light transmission of the suspension on time, at wavelength (a), nm: 540 (1), 800 (2), 1000 (3). Average YSZ particle sizes with grinding time (b), in h: 1, 2, 3, 4. A histogram of the distribution of YSZ particles by size. The grinding time of the precursor was 4 h (c).

Pyrolytic graphite and a porous anode substrate NiO-YSZ with a nickel foil sublayer were used as the substrates for the deposition of YSZ in order to study the impact of the substrate material on the coating structure. It was possible to create a uniform coating made of zirconium dioxide with a thickness of up to 7 μm thanks to the higher conductivity of graphite substrates. After drying for 24 h at room temperature, the ceramic layer was fixed and free of visible cracks. However, following heat treatment at temperatures between 973 and 1173 K, the YSZ layer peeled off the substrate material and microcracks as large as 3 μm in width appeared. This phenomenon can be attributed to significant variations in the thermal expansion coefficients of graphite and YSZ [28]. In addition, thin layers of graphite deform at high temperatures, and the graphite substrate collapses, which leads to direct deformation and cracking of the deposited layer of zirconium dioxide stabilized with yttrium oxide on this substrate material.

Deposition of YSZ on porous anode substrates based on NiO-YSZ gave different results. The high porosity of the material allowed YSZ particles to penetrate into the near-surface layer during electrophoretic deposition and fix themselves on the substrate. The surface of the ceramic YSZ layer that was deposited on a non-conductive NiO-YSZ coating is depicted in the SEM image in Figure 4a. Since in the course of thermal studies it was found that the temperature of 1173 K is not sufficient for sintering the deposited layer consisting mainly of irregularly shaped particles, due to the higher melting point of the NiO-YSZ anode substrate [29], the YSZ functional layers were annealed at a temperature of 1573 K for 2 h. Figures 4b and 4c show SEM images of the surface of the ceramic layer and the end part of the YSZ layer on the NiO-YSZ substrate after heat treatment. The resulting layer had a thickness of 5.14 μm . The YSZ particles can be seen to have started to sinter with one another, losing the distinct grain boundaries, and there is also no evidence of the particle aggregation that was seen during the deposition on graphite.

4. Conclusions

In this work, YSZ suspensions based on an isopropanol, acetylacetone, and BMA-5 copolymer mixture were successfully produced. It was discovered that as the initial YSZ powder was ground for longer periods of time, the suspensions became more stable as the average particle size decreased from 4 to 0.9 μm and the quality of the ceramic coatings that were deposited improved. The prepared suspension's stability time exceeded 50 h. By using the EPD method, ceramic layers on NiO-YSZ and pyrolytic graphite substrates were successfully produced. The obtained YSZ layers have the crystal lattice parameters $a = 3.6249(3)$ \AA , $c = 5.1275(1)$ \AA and are composed of tetragonal zirconium dioxide stabilized by 3 mol.% yttrium oxide.

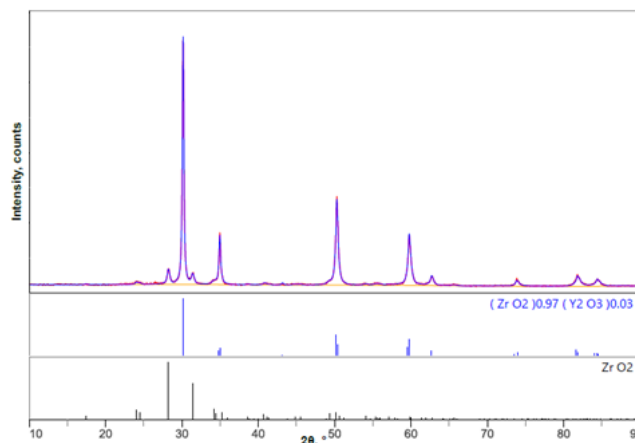


Figure 3 X-ray diffraction pattern of the YSZ layer deposited on a porous NiO-YSZ anode substrate.

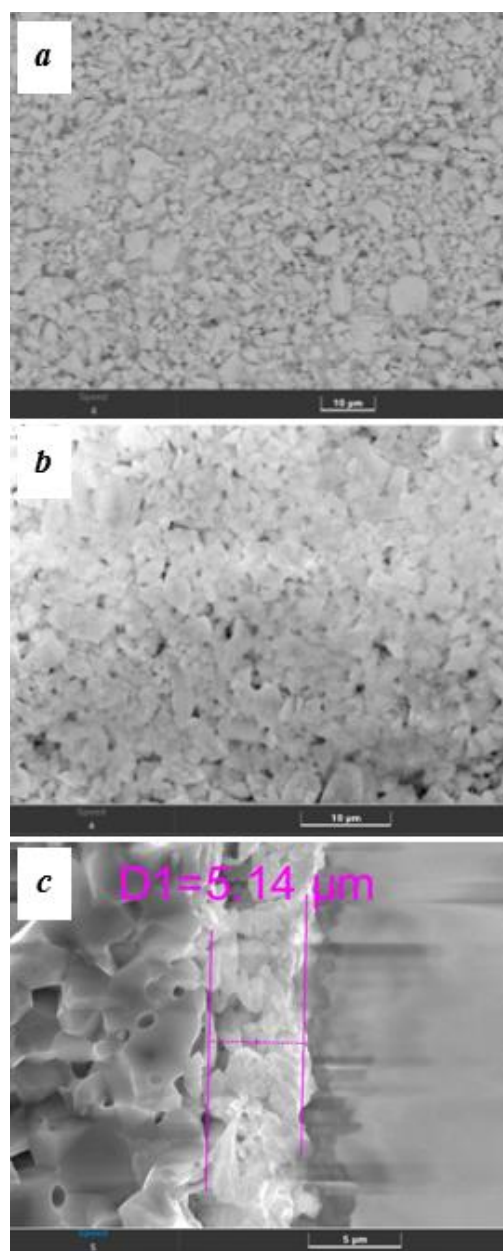


Figure 4 SEM images of the obtained YSZ layer on a Ni-YSZ substrate before (a) and after heat treatment at 1573 K (b). SEM image of the end of the YSZ layer; the heat treatment temperature was 1573 K (c).

It was determined that the EPD of YSZ undergoes successfully on a porous NiO–YSZ anode substrate with a nickel foil sublayer, ensuring that the deposited layers of YSZ are up to 5.14 μm thick. The thickness of the YSZ layers obtained on graphite was 7 μm . The EPD method can be used to apply ceramic layers on non-conductive substrates by using a nickel plate as a sublayer of NiO–YSZ. The use of EPD and inexpensive commercial powders greatly simplifies and reduces the cost of the process of obtaining ceramic coatings, which allows better integration into the green energy agenda.

Supplementary materials

This manuscript contains supplementary materials, which are available on a corresponding online page.

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Conflict of interest

The authors declare no conflict of interest.

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