

Evaluation of the potential application of $\text{La}_{2-x}\text{Sr}_x\text{CuO}_{4-\delta}$ and $\text{Nd}_{2-x}\text{Sr}_x\text{NiO}_{4-\delta}$ as alternative cathode materials for solid oxide fuel cells

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Comparative analysis between two cathode materials for the preparation of solid oxide fuel cells was performed. Series of layered perovskites $\text{La}_{2-x}\text{Sr}_x\text{CuO}_{4-\delta}$ were synthesized by the nitrate citrate method with different levels of Sr - substitution. Cathode materials area specific resistance (ASR) at different temperatures and fuel cells resistances at different temperatures are the indicative criteria the present work is focused on, in order to evaluate and compare the operating characteristics of both cathodes. For the determination of these parameters electrochemical impedance spectroscopy (EIS) over “cathode/interlayer/electrolyte/interlayer/cathode” structured symmetrical cells for the ASR determination was applied. Afterwards voltammograms over “anode/electrolyte/interlayer/cathode” structured anode supported SOFC (AS-SOFC) for the determination of cells resistance were acquired. Voltammograms were recorded for different times of SOFCs exposure under real operation conditions, thus evaluating cells capacity to operate for a long period of time. AFM topography observations were performed in order to indirectly explain the differences in the behavior of the samples tested. The potential application of both cathode materials as alternative SOFC was evaluated and conclusions were drawn.

Keywords: SOFC, cathodes, $\text{La}_{2-x}\text{Sr}_x\text{CuO}_{4-\delta}$, $\text{Nd}_{2-x}\text{Sr}_x\text{NiO}_{4-\delta}$, perovskites

INTRODUCTION

During the recent decades the demand for new energetic sources has sharply increased. In this context, the solid oxide fuel cells (SOFCs) have been found to be an attractive alternative of the already utilized energetic systems. Furthermore, SOFCs could be used for co-generation of electricity and valuable products for the chemical industry as well [1 – 4]. Nevertheless, the application of SOFCs as industrial integrated products recommends standardization in order to guarantee their reliable and harmless usage for prolonged periods of time.

Each layer of the multilayered ceramic element performs its own function. The most widely spread materials for SOFC application are described in several review papers [5, 6]. Generally the SOFC is composed by cathode (air electrode), anode (fuel electrode), electrolyte, functional layers in between, interconnectors and sealings.

The requirements, related to the SOFC household and industrial exploitation, predetermine the large scientific R&D activities intended for new ceramic materials and technologies for their synthesis. Thus, studies are carried out on the

synthesis and characterization of cathode materials as: $\text{La}_{0.5}\text{Sr}_{0.5}\text{CoO}_3$, LaCoO_3 , $\text{LaNi}_{0.6}\text{Fe}_{0.4}\text{O}_3$ [7, 8], $\text{Ce}_{0.9}\text{Sm}_{0.1}\text{O}_{1.95}$, $\text{La}_{0.5}\text{Sr}_{0.5}\text{Co}_{0.2}\text{Fe}_{0.8}\text{O}_3$ [9, 10], LaMnO_3 , $\text{La}_{1-x}\text{Sr}_x\text{CoO}_{3-\delta}$, $\text{La}_{1-x}\text{Sr}_x\text{Co}_{1-y}\text{Fe}_y\text{O}_{3-\delta}$ [11, 12] and others [13]. Typical electrolyte in the SOFC technology is yttria stabilized zirconia (YSZ) owing to its ionic conductivity and electrical insulating properties at temperatures 700–1000°C. Disadvantage of this material is its relatively high chemical activity towards some elements that are often present in the cathode composition. This can be overcome by including gadolinium doped ceria (CGO) as barrier interlayers between the cathode and the electrolyte. In most cases the anode material is Ni-cermet as a porous fuel electrode.

It was established from previous works, that there is a permanently increasing interest in the development of new materials for SOFC applications [14]. Nowadays, the evolution of the SOFC technology leads to new challenges. One of the main problems to deal with appears to be the lowering of the operation temperature. New generation of fuel cells - intermediate temperature SOFCs (ITSOFCs) achieves performance at temperatures in the range of 700–850°C, which allows the use of significantly less expensive construction materials in comparison to the older generation SOFCs with an operation temperature range of 1000°C. Overcoming of the problem is

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related to structural and functional changes in the entire fuel cell device.

It was described [15] that at low operation temperatures, the polarization losses and the importance of catalysis of the electrode reactions sharply increase. At the cathode, mixed potentials can arise when traces of combustible substances determine the electrode potential in competition with oxygen. In the present work temperatures below 700°C will not be applied during tests due to the operation characteristics of the YSZ electrolyte. It is known that its ionic conductivity cannot be activated below 700°C.

The aim of the present research work is to compare the variations of two cells resistances as a function of the operating temperature as one of the indicative criteria for the fuel cells performance. The variation of the resistance as a function of the test duration was also indicated. Symmetrical cells with cathode-electrolyte-cathode structure were used to determine the cathode resistance by electrochemical impedance spectroscopy (EIS) where the area specific resistance (ASR) was determined for both cathode materials. Later on two different anode supported fuel cells (AS-SOFC) with anode-electrolyte-interlayer-cathode structure were prepared and tested with the two different cathode materials. The variation of the fuel cells resistance as a function of the current density was determined. AFM observations were carried out in order to explain the differences between the fuel cells with the different cathodes.

EXPERIMENTAL

Cell assembling

Two kinds of test objects were used in the present work:

(i) *Symmetric cells* - They were composed by two cathode layers screen printed on both sides of the electrolyte layer. CGO containing paste was screen printed, dried and sintered as a barrier layer between the cathode and the YSZ, thus preventing the formation of insulating layers from the cathode components and the electrolyte. It was synthesized *via* classical ceramic method. Preliminarily prepared pastes containing $\text{La}_{2-x}\text{Sr}_x\text{CuO}_{4-\delta}$ and $\text{Nd}_{2-x}\text{Sr}_x\text{NiO}_{4-\delta}$, were afterwards screen printed on both sides of the sintered solid electrolyte.

(ii) *Anode supported SOFC* - For AS-SOFC preparation, anode supported half cells were used. They were supplied from SOFCPOWER S.r.l. [16], composed by YSZ- solid electrolyte and a Ni cermet anode. Functional CGO interlayer was

deposited *via* screen printing, followed by screen printing deposition of $\text{La}_{2-x}\text{Sr}_x\text{CuO}_4$, and $\text{Nd}_{2-x}\text{Sr}_x\text{NiO}_{4-\delta}$ cathode materials.

After each cycle of deposition every layer was sintered in an individual thermal cycle. The primer CGO layer was treated in the following cycle: an increase with a step of 1°C/min up to 400°C, followed by a 3°C/min step up to 1250°C. The final cooling rate was 5°C/min. The cathode layer was treated at 900°C for 2h and a cooling rate at 5°C/min was applied. Thus, the obtained bi-layer film had a total thickness of 30 µm.

Characterization procedures

The following methods were applied for characterization of the cells obtained.

(i) *Determination of the area specific resistance (ASR) of both cathode materials on symmetrical cells.* It was determined by high temperature AC impedance spectroscopy on Autolab PGStat-30, produced by EcoChemie, The Netherlands. The measurements were performed at open circuit voltage (OCV) in the frequency range of 1 MHz to 0,1 Hz in the temperature interval between 500 and 700°C. The EIS analysis yielded results on the determination of the polarization resistance of the cathode materials. The polarization resistance obtained on a Nyquist plot of the EIS curves was used in the calculation of the ASR.

(ii) *V - J characteristics of the anode supported fuel cell.* It was performed in a high temperature furnace with controlled gas flows, product of ECN - The Netherlands. Acquisition of the voltammograms: it was performed in a galvanostatic regime at 50mA/cm² for 180 sec. The voltammograms were done at the maximum threshold: 0,420A/cm² and at the minimum threshold: 500mV. The voltammetric measurements were executed at 900, 850, 800, 750 and 700°C, respectively. The test procedure was started after initial achieving of the operation temperature in nitrogen atmosphere for reduction of NiO into Ni⁰ and stabilizing the Ni:YSZ cermet anode layer and stable charging of the cell up to 0.3 A/cm² and retention of this temperature for 2 h.

RESULTS AND DISCUSSION

Determination of the Area Specific Resistance.

It was determined on symmetric cells, prepared as was described above. After the polarization resistance was obtained from the EIS measurements, the ASR was calculated according to equation 1, where S is the area of the tested

electrode. The division into two gives the value for one of the cathode layers.

$$ASR = \frac{R_p \times S}{2} \quad (1)$$

It was established that the samples of the symmetric cells with the respective cathodes show almost identical values of the ASR for the five temperatures in the range from 500 to 700°C, as is shown in Figure 1(a, b) below.

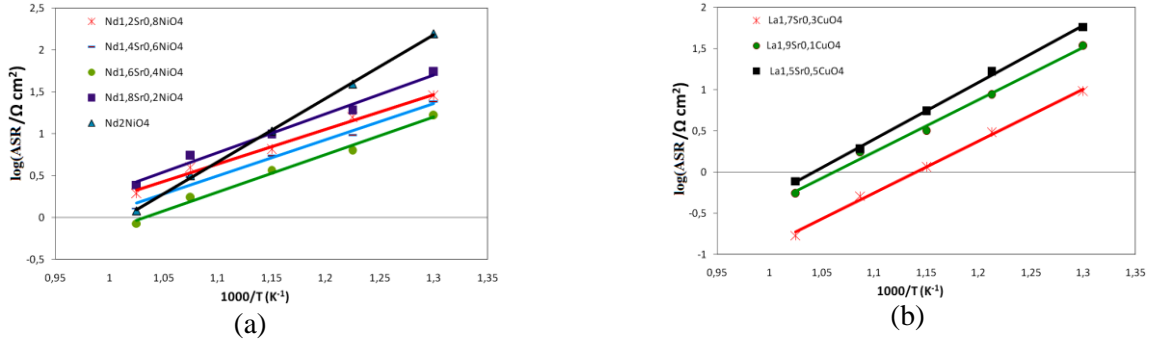


Fig. 1. Values of the area specific resistance of a) $\text{Nd}_{2-x}\text{Sr}_x\text{NiO}_{4-\delta}$ and b) $\text{La}_{2-x}\text{Sr}_x\text{CuO}_4$ for different temperatures, expressed in $1000/T$ (K^{-1})

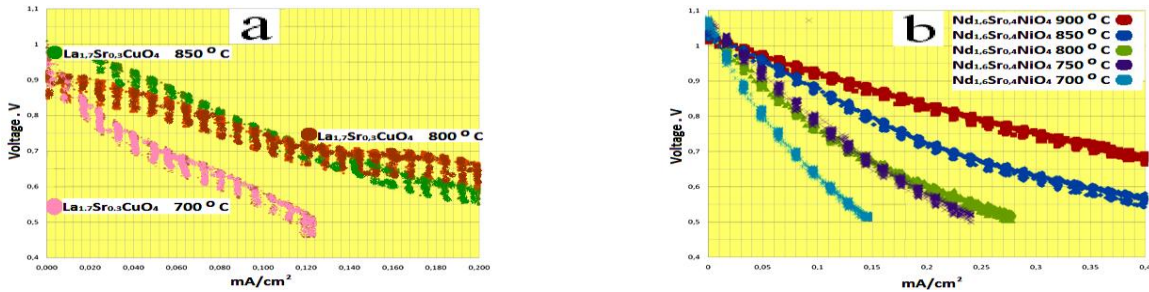


Fig. 2. SOFC Voltammograms acquired for $\text{La}_{1.7}\text{Sr}_{0.3}\text{CuO}_{4-\delta}$ at 700, 800, 850°C (b) $\text{Nd}_{1.6}\text{Sr}_{0.4}\text{NiO}_{4-\delta}$, at 700, 750, 800, 850, 900°C.

The similarity of the diagrams evinces that both electrodes possess identical area specific resistances, and even almost the same trends of dependence of ASR vs. temperature. In addition, the analysis shows a low cathode resistance that allows us to consider both materials as suitable for cathode materials application. As shown in the literature, the resistance of IT SOFC cathode should be in the order of $1 \Omega \text{ cm}^2$ at 700°C [16, 17].

The performed analysis showed different ASR values obtained for the different levels of Sr-substitution. The lowest resistance values were detected for $\text{La}_{1.7}\text{Sr}_{0.3}\text{CuO}_4$, as well as for $\text{Nd}_{1.6}\text{Sr}_{0.4}\text{NiO}_{4-\delta}$ - tested samples. The latter two cathode materials were used in the subsequent experiments of the complete AS-SOFCs tests; simplifying these materials with indexes LSC and NSN, respectively.

3.2. V–J characteristics

There is a clear deviation between the theoretical and the real correlation values of the electric potential and the current density [18]. This difference is determined by losses caused by various sources, as for example: 1 – losses from

activation of the electrodes; 2 – losses from Ohmic polarization, (i.e: polarization resistance R_p), and 3 – losses from the gas transport, caused by the diffusion between the feeding combustible gas and the gaseous products. Here it should be mentioned that the presence of the combustion products decreases the concentration of the feeding gas and consequently – its partial pressure. It is clear that the rate of all these losses depends on the composition of the SOFC components and on their properties, (i.e. density, thickness, porosity, etc.). That was the reason we performed a comparative analysis on the electrochemical behavior of cells, either with $\text{La}_{1.7}\text{Sr}_{0.3}\text{CuO}_4$, or with $\text{Nd}_{1.6}\text{Sr}_{0.4}\text{NiO}_{4-\delta}$ at different operation temperatures. The results obtained are presented in Figure 2 (a,b) for both LSC and NSN samples.

Figure 2 shows the considerable difference in the behaviour of the respective cells. The correlation of the results of Figure 1 and Figure 2 leads to a surprising fact: although the characteristics shown in Figure 1 are almost identical, the voltammograms in Figure 2 reveal remarkable differences for the complete ITSOFC with the respective cathodes. The open circuit

voltages for the LSC sample, position (a) are in the range of 850 mV and do not reach 1000 mV, whereas they are between 1000 and 1100 mV in position (b) for the NSN sample. Additionally, at 700°C the cell with the former cathodic material achieves the 500 mV threshold at 0,120mA/cm², while that with the latter one reaches it at 0,150mA/cm². Higher differences between LSC and NSN cathode cells even arise at elevated temperatures. Thus, at 800 and 850°C the cells achieve the 500 mV threshold at about 0,240 mA/cm², whereas the cells with Ni-based cathodes reach it at 0,275 mA/cm² at 800°C, and at more than 0,400 mA/cm² at 850°C, respectively. At 900°C no voltammogram for the lanthanum cathode cells could be detected, because the material suffered partial fusion at this temperature.

Furthermore, the shapes of the curves reveal supplemental differences. Possible explanation about the phenomenon in the latter case could be the decomposition of the water molecules, rendering additional H₂ supply. In this way, at higher current densities, the additional decomposition of the water, (coming either from the fuel humidity, or as a product of the H₂ → H₂O_(g)), could deliver additional quantities of hydrogen, thus increasing the efficiency of the entire ITSOFC.

The curves for 800 and 850°C of position (a) reveal almost indistinguishable double inflections, indicating the presence of different processes in the respective ranges of the voltammograms. They could originate from structural changes in the respective cathode materials, as is explained in the next paragraph.

The polarization resistance R_p could be calculated by equation 2, taking in account that the polarization resistance expresses the relation

between the changes of current density (ΔJ) and the voltage (ΔV) in a given range.

$$R_p = \frac{\Delta V}{\Delta J}, \Omega/\text{cm}^2. \quad (2)$$

Both cells showed their lowest R_p values at 700°C. Their values were estimated to be 3.666 kΩ/cm² for LSC and 3.333 kΩ/cm² for NSN, respectively. In other words, at 700°C the cell with the NSN electrode slightly exceeds that with the LSC one. Nevertheless, at 850°C the R_p value for nickelate was estimated to be 0.75kΩ/cm², whereas for LSC it reaches 1.5 kΩ/cm². This fact is an additional evidence for the better behavior of Nd_{1,6}Sr_{0,4}NiO_{4-δ}, compared to La_{1,7}Sr_{0,3}CuO_{4-δ}, at higher temperatures. The contradiction between the identical ASR behavior in Figure 1, and the remarkable differences in Figure 2 is a direct evidence for either the incompatibility of LSC with the rest of the components of the fuel cells, or the presence of cell “destructive” processes at temperatures above 750°C. This contradiction reveals that the cells possess rather distinguishable electrical characteristics, although they are identical for both cathode materials.

3.3. Durability tests

The duration of the time elapsed until the breakdown of whatever industrial product at operation conditions, (i.e. temperature, pressure, etc.), is considered as durability. The breakdown of the respective product appears at the moment of the sharp dissipation of its parameters from their nominal or initial values. Thus, in the present case significant difference between the respective cells arises regarding their durability. Figure 3 shows voltammograms recorded for different times of SOFCs exposure to real operation conditions.

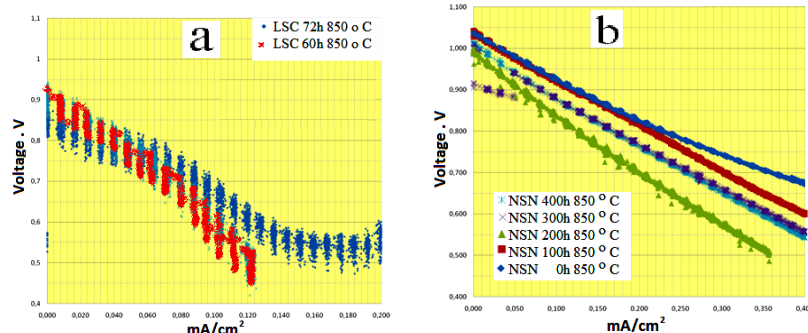


Fig. 3. Voltammograms acquired during the durability tests. (a)- $\text{La}_{1,7}\text{Sr}_{0,3}\text{CuO}_{4-\delta}$. (b)- $\text{Nd}_{1,6}\text{Sr}_{0,4}\text{NiO}_{4-\delta}$

Apparently, the durability tests prove the statement that the NSN cathode sample displays excellent durability, incomparable to that of LSC cathode cells. Indeed, the voltammograms maintain

the shape of straight lines even after 400 hours for the NSN sample, whereas the LSC sample exhibits curves with clear inflexions even after 60 h. This

means that the durability of NSN cathode exceeds that of LSC by about 6 or 7 times.

Furthermore, the $\text{Nd}_{1.6}\text{Sr}_{0.4}\text{NiO}_{4-\delta}$ reveals a surprising improvement of their voltammograms after 200 hours. The polarization resistances (R_p) for the respective curves could be calculated by the equation 1. Thus, the R_p calculations have evinced the presence of a posterior improvement after 200 hours of exposure. Immediately after the stable charging of the cell, (see experimental part), the NSN-cell sample had R_p equal to $1.000 \text{ k}\Omega/\text{cm}^2$, compared to $1.430 \text{ k}\Omega/\text{cm}^2$ after 200 hours and $1.143 \text{ k}\Omega/\text{cm}^2$ after 400 hours, respectively. The values obtained are an additional evidence of the subsequent improvement of the electric characteristics of the NSN cells. Because the structural transformations for such ceramic materials are generally inconvertible, it looks more likely that the subsequent improvement of the NSN cells after 200 h is a consequence of other

phenomena. Indeed, a hypothetical explanation can be assumed that after deterioration (or inactivation) of a part of the cathode, enhancement of another part takes place. The reason for this enhancement could be the additional portion of H_2 originating from the thermal decomposition of water. In this way, H_2 undergoes oxidation on the intact zone of the cathode, compensating the obstructed area.

The remarkable difference between the durability of LSC and NSN was observed *via* structural measurements, as well. They are shortly discussed in the next section.

3.4. Surface topology characterization

The inflections in positions (a) of Figures 2 and 3, commented above in the present text could generally be a consequence of structural deterioration of the $\text{La}_{1.7}\text{Sr}_{0.3}\text{CuO}_4$. This suggestion was confirmed by the AFM observations done and 3D surface topology shown in Figure 4.

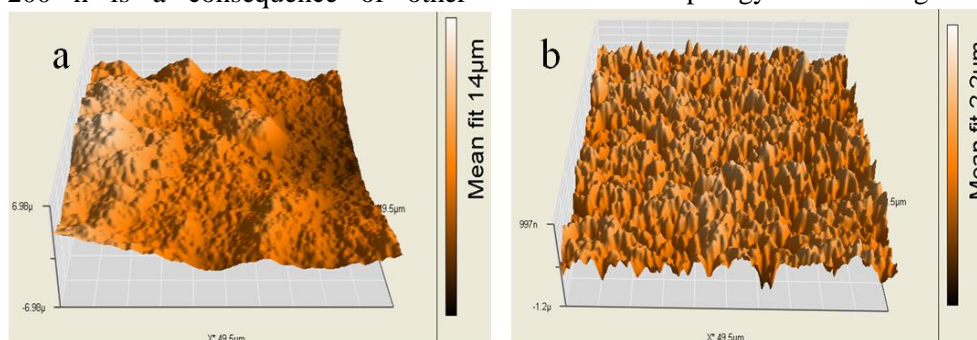


Fig. 4. AFM 3 D images of cathodes of cells after durability tests. (a)- $\text{La}_{1.7}\text{Sr}_{0.3}\text{CuO}_4$, after 72 hours. (b)- $\text{Nd}_{2-x}\text{Sr}_x\text{NiO}_{4-\delta}$, after 400 hours

Figure 4 obviously confirms that the surface of the NSN cathode material is much rougher and its structural conformation is preserved even after 400 hours. This roughness could be an indirect evidence for better porosity and higher specific area. On the contrary, on the image of the LSC cathode (Figure 4-a) different morphology is observed. The reason for this lower porosity is that the cathode material has suffered partial fusion in the high temperature operation conditions. Afterwards, the fused liquid material has filled the pores. Subsequently, the presence of liquid (fused) phase in the electrode obviously leads to posterior agglomeration processes in the structure of the LSC electrode. That is the reason for the uniform distribution of the pores in the latter case (see Fig.4 position b), compared to the irregular distribution of the roughness in the former case (see Fig.4 position a). In other words, the 6 to 7 times lower durability of the former material is caused by partial diffusion of the material. Consequently, the lower durability of LSC originates from its lower thermal resistance.

4. CONCLUSIONS.

After the analysis of the results obtained, the following conclusions could be drawn:

1) Both tested LSC and NSN cathode samples reveal similar trends of evolution of the ASR with the temperature. However, the operation characteristics for all cell samples are rather distinguishable. The remarkable differences between the respective LSC and NSN fuel cells are a direct evidence either for the incompatibility of LSC with the rest cells components or for the incapability of this cathode to operate in the temperature range chosen for the present work. This contradiction reveals that the SOFC possesses rather distinguishable electrical characteristics, although the ASR values of both cathodic materials are identical.

2) After the acquisition of the operation characteristics for the entire cells, it was proved that

LSC samples suffer structural deterioration at temperatures higher than 700°C. The respective voltammograms are curved, due to presence of inflexions for the SOFC containing $\text{La}_{1.7}\text{Sr}_{0.3}\text{CuO}_{4-\delta}$ cathode. The voltammograms of the NSN cell are straight lines, showing the thermal stability of the NSN cathode material. Furthermore, at 850°C the polarization resistance of LSC (1.5 kΩ/cm²) is twice higher than that of NSN (0.75 kΩ/cm²), despite their equal values at 700°C. This is a clear indication that both cathodes are more suitable for work in the intermediate rather than in the high temperature range.

3) Besides the confirmation of the results from the previous measurements, the durability tests performed show that NSN is 6-7 times more durable than the LSC cathode.

4) The AFM surface topology study undoubtedly shows that the LSC material undergoes partial fusion accompanied by agglomeration processes.

A general conclusion can be made based on the analysis performed. The NSN cathode is a promising material to be included in the given configuration of anode supported zirconia based ITSOFC. As for the LSC material, its potential should be tested in combination with different electrolytes that can achieve ionic conductivity at less aggressive temperatures that should not lead to phase transformation in the cathode structure.

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ИЗСЛЕДВАНЕ И ОЦЕНКА НА ПОТЕНЦИАЛНОТО ПРИЛОЖЕНИЕ КАТО
АЛТЕРНАТИВНИ КАТОДНИ МАТЕРИАЛИ ЗА ТВЪРДООКИСНИ ГОРИВНИ КЛЕТКИ
НА $\text{La}_{2-x}\text{Sr}_x\text{CuO}_{4-\delta}$ И $\text{Nd}_{2-x}\text{Sr}_x\text{NiO}_{4-\delta}$,

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(Резюме)

Проведен беше сравнителен анализ между двата катодни материала за прилагането им в твърдоокисни горивни клетки. Серия от т. нар. слоеви перовскити тип $\text{La}_{2-x}\text{Sr}_x\text{CuO}_{4-\delta}$ бяха синтезирани по нитрат-цитратен метод с различно степен на заместване със Sr. Специфичното повърхностно съпротивление (ASR) при различни температури, както и съпротивленията на самите горивни клетки при различни температури са показателния критерии върху които се фокусира настоящата работа с цел да се сравнят операционните характеристики на двата катода. За определянето на тези параметри бе проведен анализ със импедансна спектроскопия (EIS) върху симетрично структурирани клетки “катод/междинен функционален слой/електролит/междинен функционален слой/катод” за определянето на ASR. След което бяха получени волтамограми от анодно базирани горивни клетки “анод/електролит/междинен функционален слой/катод” за определянето на съпротивлението на така окомплектованите горивни клетки. Бяха снети волтамограми при различно време на подлагане под реални операционни условия, за оценка на капацитета на работа за дълги периоди от време. Атомно силова микроскопия AFM беше приложена за наблюдения на топографията и косвеното обяснение на разликите в поведението на тестваните образци. Потенциалното прилагане и на двата катодни материала като алтернативи на съществуващите беше оценено със съответните изводи и заключения.