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# Microtubular solid oxide fuel cells with magnetron-sputtered electrolyte

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## Abstract

A bilayer $Zr_{0.85}Y_{0.15}O_{2-\delta}/Ce_{0.9}Gd_{0.1}O_{2-\delta}$ (YSZ/GDC) solid oxide fuel cell electrolyte was first formed on a microtubular NiO/YSZ anode by the reactive magnetron sputtering method. The composite material $La_2NiO_{4+\delta}$–$Ce_{0.8}Sm_{0.2}O_{2-\delta}$ (LNO-SDC) was used as the cathode and characterised using the X-ray diffraction method. The current-voltage characteristics of a microtubular solid oxide fuel cell (MT SOFC) with anode-supporting design featuring a two-layer thin-film YSZ/GDC electrolyte and an air LNO-SDC electrode were measured. The microstructure of a single fuel cell after electrochemical measurements was investigated using scanning electron microscopy (SEM). A comparison with literature data on MT SOFCs that have similar functional layers was made.

**Keywords:** magnetron sputtering, solid oxide fuel cells

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## INTRODUCTION

Solid oxide fuel cells (SOFCs) are among the most promising devices for the transformation of chemical energy into electricity. The attractiveness of these devices is primarily due to properties such as high efficiency and environmental compatibility, as well as the possibility to make modular structures and use various kinds of fuel (natural gas, hydrogen, synthesis gas, etc.) [1–3]. Among all possible versions, microtubular solid oxide fuel cells (MT SOFCs) attract the attention of researchers due to a set of characteristics associated with the geometric parameters of these devices. The following advantages can be distinguished: high thermal cycling stability, large specific volumetric power output, rapid start-up as well as efficient shutdown capability [4]. One of the most widespread MT SOFC configurations is the structure with fuel electrode (anode) play-

ing the part of the support substrate. In comparison with electrolyte-supported SOFCs, the use of anode as the supporting layer allows a substantial reduction in electrolyte thickness. In turn, this minimises the ohmic losses, which are mainly associated with the resistance of electrolyte layer. Further decrease in the ohmic resistance, resulting from a reduction in electrolyte thickness and, consequently, improvement in productivity, can be achieved using thin-film technologies [5, 6]. Up to now, the possibility to use magnetron sputtering to form electrolyte in MT SOFCs has not been explored yet, though this method is successfully in use for a long time to make an electrolyte layer in planar SOFCs [7, 8]. The application of this method allows one to exclude the long-lasting stage of sintering the deposited electrolyte layers at a temperature of 1300 °C and higher, as it is the case with layer formation by dip-coating. Moreover, magnetron sputtering enables the possibility to deposit the coatings on large-area substrates, which is an important issue from the viewpoint of technology commercialisation.

In addition to minimisation of ohmic losses, a decrease in the losses associated with polarisation resistance also appears to be a priority task in the area of SOFC technology development and investigation aimed at the improvement of performance characteristics. To decrease the polarisation resistance of a fuel cell, cathode materials with high electrocatalytic activity are used. Among the materials used for the air electrode of SOFC, in addition to the compounds with perovskite structure, special attention is paid to the materials based on lanthanum nickelite $La_2NiO_{4+\delta}$ (LNO) [9, 10]. Due to the ability of LNO structure to embed interstitial oxygen above the stoichiometry, this oxide possesses higher ionic component of conductivity in comparison with LSCF oxide (lanthanum strontium cobalt ferrite) with perovskite structure, which is broadly used as a SOFC cathode [11, 12].

The goal of the present work was to study the possibility of using reactive magnetron sputtering to form a thin-film double-layered YSZ/GDC-electrolyte for MT SOFCs with the air LNO-SDC-electrode, to investigate the microstructure and electrochemical characteristics of the obtained elements, and to compare the achieved characteristics with the properties of a MT SOFC with similar functional layers manufactured using traditional powder methods.

## EXPERIMENTAL

### *Formation of MT SOFC anodes*

The initial raw materials for manufacturing microtubular anode substrates were the powders of nickel oxide (NiO) of specially pure grade (UZKhR, Russia) and zirconium dioxide stabilised with yttrium oxide (YSZ, $Zr_{0.9}Y_{0.1}O_{2-\delta}$) of chemically pure grade (Neokhim, Russia). The formation of MT anode substrates was performed by phase inversion [13]. In addition to a mixture of powders at the mass ratio of NiO/YSZ = 60 : 40, the paste for extrusion contained N-methylpyrrolidone (chemically pure grade) as a solvent, and polysulphone (chemically pure grade) as a plasticiser. Thus obtained anode substrates were annealed in the air at 1200–1400 °C for 1 h.

### *Formation of double-layer YSZ/GDC-electrolyte*

The deposition of double-layer YSZ/GDC-electrolyte on MT SOFC anodes was carried out by reactive anode sputtering. The scheme of experiment is shown in Fig. 1. A holder to mount the tubular node substrates in the spraying vacuum chamber was developed and made specially for this purpose. The holder is a rigid metal contour made of stainless steel. Inside the contour, there are dismountable tungsten wires 0.4 mm in diameter, on which the tubular anodes are put.

A system for substrate tube rotation in the horizontal plane during coating deposition is provided in this design to ensure homogeneity in the composition and thickness. Three tubular anodes can be accommodated at once in the substrate holder. The rate of their rotation during the deposition of the coating is about 3 r.p.m. The magnetron sputtering system and substrate heater are at the bottom and top with respect to the substrate holder in the vacuum chamber, respectively (see Fig. 1).

Before mounting anodes into the holder, they are washed in isopropyl alcohol and then in distilled water, after which they are dried in the air. After placing the samples into the vacuum chamber, it is evacuated to the residual pressure of $10^{-4}$ Torr, the samples are heated to a temperature of 300 °C, and their purification with the ion source with closed electron drift is performed at the voltage of 1500 V and discharge current 20–25 A for 10 min. The metal $Zr_{0.85}Y_{0.15}$- and $Ce_{0.9}Gd_{0.1}$-targets 76 mm in diameter (Girmet, Russia) are mounted in turn into the magnetron

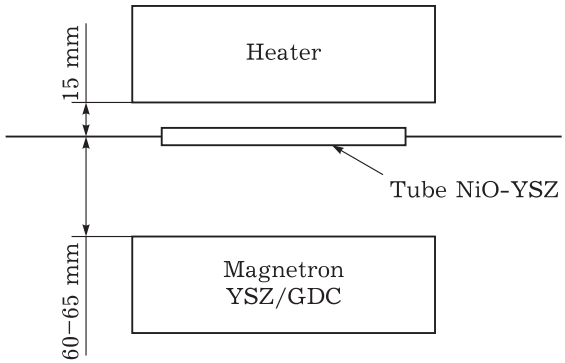


Fig. 1. Scheme of experiment on the deposition of double-layer $Zr_{0.85}Y_{0.15}O_{2-\delta}/Ce_{0.9}Gd_{0.1}O_2$ (YSZ/GDC) electrolyte.

sputtering system to deposit YSZ and GDC layers, respectively. The temperature of substrates is maintained at a level of 300 °C during coating deposition. When the film of YSZ-electrolyte is deposited, the samples are left to cool in the vacuum chamber to the room temperature, then the chamber is opened, and $Zr_{0.85}Y_{0.15}$-target is replaced by $Ce_{0.9}Gd_{0.1}$-target. The samples with YSZ-film are not taken out of the vacuum chamber during the procedure. Then heating and ionic purification of the sample surface is carried out as described above, followed by sputtering the film of GDC-electrolyte. The coatings are deposited in the argon-oxygen atmosphere at a pressure of $1.3 \cdot 10^{-3}$ Torr. In both cases, the magnetron power is 0.5 kW. The thickness of coatings prepared by magnetron sputtering can be varied by changing the deposition time. To evaluate the rate of YSZ and GDC layer growth, preliminary experiments were carried out by depositing the YSZ and GDC layers on microtubes during certain time intervals. Then the thickness of resulting coatings was measured using a scanning electron microscope (SEM) over the image of the transverse fracture of samples. The estimated rates of YSZ and GDC layer deposition were 250 and 670 nm/h. For the samples studied in the present work, the time of YSZ layer sputtering was 10 h, and GDC layer – 3 h.

### *Formation of cathode layer for MT SOFC*

To prepare the pastes for the functional and current-collecting layers, the powders of $La_2NiO_{4+\delta}$ (LNO), $Ce_{0.8}Sm_{0.2}O_{2-\delta}$ (SDC) and $LaNi_{0.6}Fe_{0.4}O_{3-\delta}$ (LNF) were used. The LNO and LNF powders were prepared using the citrate-nitrate method and supplied by T-SP company (Russia). The SDC powder was synthesised by co-precipitation of the corresponding hydroxides and supplied by Neokhim company (Russia). The cathode functional layer LNO-SDC (the mass ratio LNO/SDC = 65 : 35) and cathode current-collecting layer LNF were deposited by dip-coating. In addition to the solid-phase component, the pastes contained butanol, polyvinyl butyral, dibutyl phthalate and fish oil, used as a solvent, polymer, plasticiser, and dispersant, respectively. After drying the deposited layers, thus prepared units were sintered at 1000 °C for 1 h.

### *Investigation of MT SOFC characteristics*

Microstructure of the samples was studied using a scanning electron microscope S-3400N (Hitachi, Japan). The phase composition and crystal structure of the composite material LNO-SDC were studied by powder X-ray diffraction using a D8 Advance diffractometer (Bruker, Germany) with Cu$K_\alpha$-radiation within the range of 20–75° over 2θ in 0.02° increments and acquisition time 0.5 s in the air at room temperature. The phase analysis was performed using the Search-Match software. Refinement of the oxide structure by the Rietveld method was performed using Topas 4.2 software (Bruker, Germany) over the ICDD PDF-4 database (2011).

The electrochemical characteristics of a single MT SOFC cell were measured on a setup composed of a high-temperature furnace, potentiostat-galvanostat PS-50 (SmartStat, Russia) and the system of working gas supply. The rate of gas supply (at the volume ratio $H_2$/Ar = 1 : 1) during the experiment was 50 mL/min, and it was controlled by the gas flow regulator UFPGS-4 (SoLo, Russia). Measurements were performed within the range of 600–700 °C in increments of 50 °C. The current collection was performed with a Pt wire from the side of the cathode electrode and with a Ni wire from the anode side. Current-voltage curves and impedance spectra were recorded during measurements. The specific resistance of the entire fuel cell (the area specific resistance, ASR, Ohm · cm$^2$) was calculated for the linear part of the voltage-current curve. For this purpose, a narrow current density range was chosen (350–450 mA/cm$^2$ in our case), and ASR was calculated as a ratio of voltage difference ($U$) in the chosen current density range to the chosen current density range ($j$) [14–16]:

$$\mathrm{ASR} = \frac{U_{j1} - U_{j2}}{j_2 - j_1}$$

**RESULTS AND DISCUSSION**

In this work, the composite LNO-SDC was used and characterised as a material for the cathode layer of MT SOFC. The phase composition and crystal structure of composite material LNO-SDC were studied using X-ray diffraction. One can see in the data presented in Fig. 2 that the diffraction patterns of the composite after sintering at 1000 °C contain reflections characteristic of the oxides LNO and SDC. No additional phases are observed in the diffraction patterns, and this fact confirms the absence of interaction between the oxides.

According to the data of X-ray diffraction analysis (Table 1), the oxide LNO has $I4/mmm$ crystal structure with unit cell parameters $a = b = 3.874(1)$, $c = 12.743(1)$ Å, and the oxide SDC has $Fm\bar{3}m$ crystal structure with unit cell parameters $a = b = c = 5.438(1)$ Å. The parameters obtained by Rietveld refinement of the diffraction patterns under consideration allow us to state a good correspondence between the experimental and calculated profiles of the samples.

The measured electrochemical characteristics of a single MT SOFC cell of anode-supporting structure with a thin-film double-layer YSZ (2.5 μm)/GDC (2 μm) electrolyte (with layer thickness indicated in parentheses) obtained by magnetron sputtering, and a LNO-SDC cathode layer are shown in Fig. 3. One can see in the voltage-current characteristics (see Fig. 3, *a*) that the open-circuit voltage values measured at a temperature of 600, 650 and 700 °C are close to the theoretical value, 1.2 V, which is evidence of the high gas impermeability of electrolyte layer and good air tightness of the cell. The voltage-current curves are nonlinear. In the region of low current density, until 200–250 mA/cm$^2$, a sharp voltage drop is observed (especially at the working temperature of 600 °C), associated with activation loss. This is due to substantial slowing of chemical electrode reactions, caused by a decrease in temperature. The maximal specific power determined for single cells was 276, 382 and 521 mW/cm$^2$ at a temperature of 600, 650 and 700 °C, respectively (see Fig. 3, *b*).

The values of area-specific resistance (ASR), which provides an integrated account of all losses arising during the single cell running, are shown in Table 2. The values of ASR were calculated from voltage-current characteristics: 0.93, 0.67 and

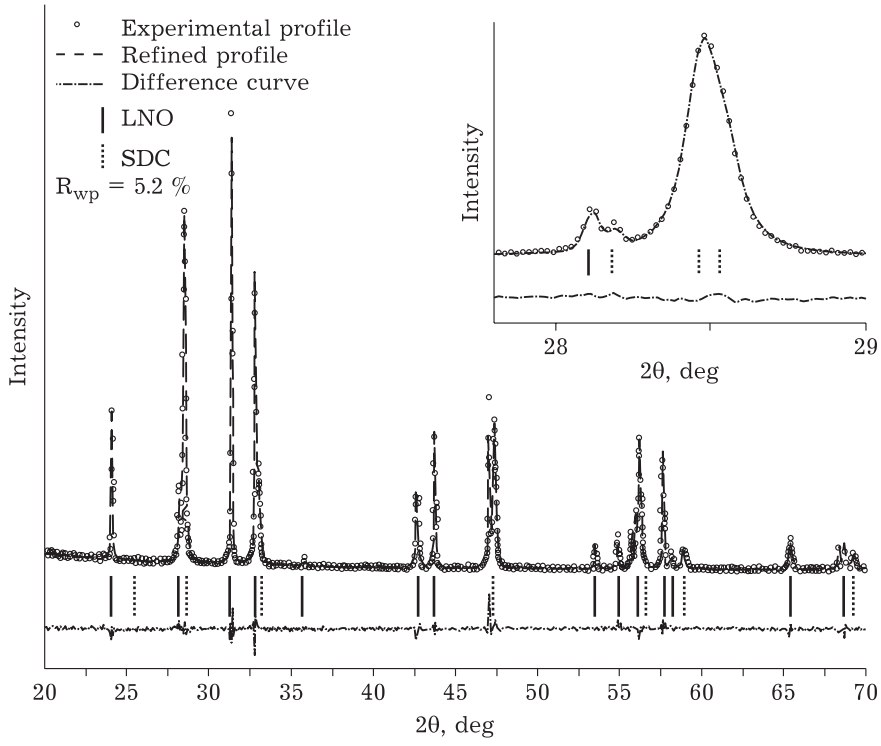


Fig. 2. Diffraction patterns of the composite material LNO-SDC ($La_2NiO_{4+\delta}$–$Ce_{0.8}Sm_{0.2}O_{2-\delta}$) after sintering at 1000 °C (enlarged fragment is shown in the insert). The pattern obtained experimentally, and the pattern calculated using the Rietveld method (refined profile) are shown along with the difference curve.

TABLE 1

Data of X-ray diffraction analysis
of the composite LNO-SDC ($La_2NiO_{4+\delta}$–$Ce_{0.8}Sm_{0.2}O_{2-\delta}$)

| Space group of symmetry | Unit cell parameters, Å | Rietveld refining parameters | |
|---|---|---|---|
| *I*4/*mmm* (LNO) | $a = b = 3.874(1)$ | $R_{exp}$, % | 4.7 |
| | $c = 12.743(1)$ | $R_{wp}$, % | 5.2 |
| $Fm\bar{3}m$ (SDC) | $a = b = c = 5.438(1)$ | $\chi^2$ | 1.48 |

*Note.* $R_{exp}$ and $R_{wp}$ – expected *R*-factor and weighted profile factor, respectively.

0.49 Ohm · cm$^2$ at 600, 650 and 700 °C, respectively. The ohmic resistance ($R_{ohm}$) values were determined using impedance spectroscopy: 0.18, 0.14 and 0.12 Ohm · cm$^2$ at 600, 650 and 700 °C, respectively. The single cell of MT SOFC with a similar composition (NiO-YSZ-anode | ScYSZ (7.2 μm)/SDC (2.2 μm)-electrolyte | LNO-SDC-cathode) in which the electrolyte was formed by dip-coating has been studied by us previously [17]; it demonstrated the maximal specific power 533 mW/cm$^2$ at a temperature of 750 °C, while ASR = 0.77 Ohm · cm$^2$, and $R_{ohm}$ = = 0.38 Ohm · cm$^2$ at this temperature. Therefore, for MT SOFC elements with similar functional layers, electrolyte preparation by magnetron sputtering provides lower ohmic and overall specific resistance in comparison with electrolyte preparation by dip-coating.

For comparison, the characteristics of single cells of MT SOFCs obtained by other researchers are listed in Table 3. The cell of MT SOFC with a YSZ/GDC-electrolyte deposited by magnetron sputtering has been shown to be characterised by a rather high level of specific power. Higher values of this parameter have been obtained only in [21], where the cell with YSZ-electrolyte 18 μm thick, formed by spray coating, exhibited specific power about 700 mW/cm$^2$ at 700 °C. The authors explain so high characteristics by the use of efficient cathode material based on neodymium nickelite ($Nd_2NiO_{4+\delta}$, NNO). Therefore, the presented data demonstrate the potential of using magnetron sputtering to form a thin-film electrolyte layer for MT SOFC in order to improve its power output characteristics.

After electrochemical tests, the microstructure of the cross section of the single cell was studied using SEM. In the images (Fig. 4), one can clearly see the porous layers of cathode and anode with a thin double-layered electrolyte between them. The electrolyte is represented by a dense layer, with uniform structure and thickness. The absence of cracks and any indications of layer delamination should be particularly emphasised,

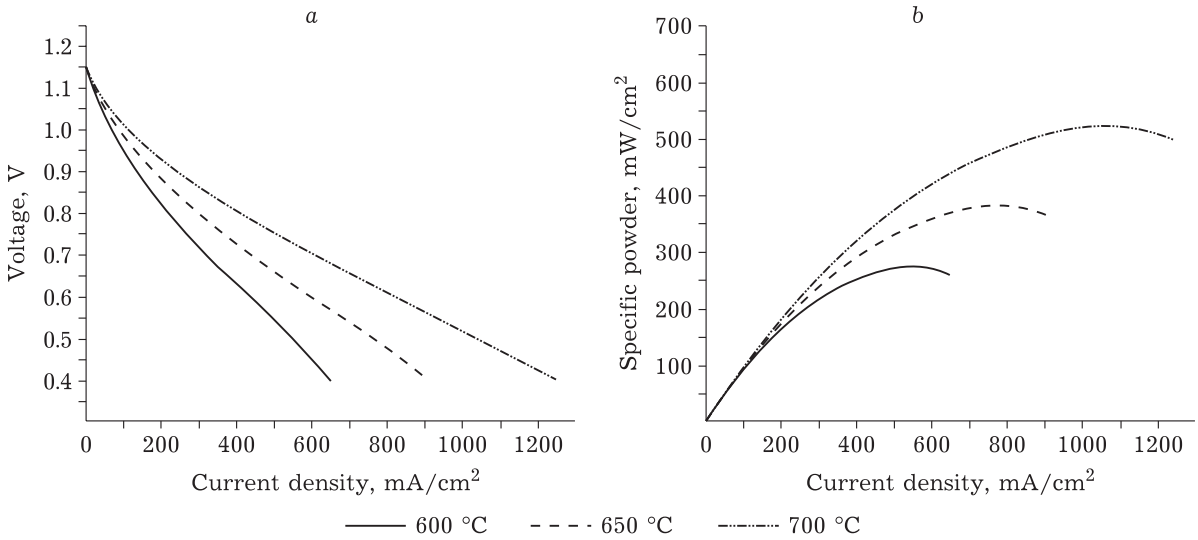


Fig. 3. Dependence of electrochemical characteristics of investigated MT SOFC: voltage (*a*) and specific power (*b*) on current density at different temperatures.

as this is the evidence of rather high mechanical stability of investigated MT single cells. The thickness of YSZ and GDC layers according to SEM data was about 2.5 and 2 μm, respectively. A good adhesion of the electrolyte to the fuel electrode is also observed. As far as the electrolyte/cathode interface is concerned, one can see numerous voids, where no contact between the air electrode and the electrolyte is observed. Insufficient contact between the electrolyte and cathode can have e negative effect on fuel cell characteristics, especially for long-term performance. For this reason, in our opinion, further works aimed at an increase in MT SOFC characteristics can be directed to improvement of the contact between these two layers. This can be implemented, for example, by forming a thin fine-pored functional cathode layer on the electrolyte surface, by changing the composition of the cathode layer or its sintering temperature.

The results presented in the work show that magnetron sputtering provides the formation of a high-quality thin-film defect-free gas-tight electrolyte layer. A decrease in electrolyte thickness led to a decrease in ohmic losses and, as a consequence, improvement of the power output characteristics of the fuel element. Summarising the data obtained in the work, we may conclude that magnetron sputtering is a promising method for forming a thin-film electrolyte of MT SOFC.

## CONCLUSION

Reactive magnetron sputtering has been used for the first time to deposit the layer of electrolyte for MT SOFC with an anode-supporting design. Cathode material LNO-SDC has been characterised using X-ray diffraction, which demonstrated the absence of any interaction between the components of the composite at a temperature of 1000 °C. It has been determined from the measurements of electrochemical characteristics that the maximal specific power values were 276, 382 and 521 mW/cm$^2$ at a temperature of 600, 650 and 700 °C, respectively. It has been determined by SEM that after electrochemical tests, MT SOFC exhibits no delamination or other kinds of destruction in functional layers, indicating that its high mechanical stability is maintained.

Thus, the deposition of a dense thin-film electrolyte by magnetron sputtering allows a decrease in the ohmic resistance of the cell, which enables an increase in power characteristics. In spite of the necessity of further studies aimed at optimi-

TABLE 2

Ohmic resistance ($R_{ohm}$) and area specific resistance (ASR) of investigated MT SOFC at different temperatures

| $T$, °C | $R_{ohm}$, Ohm · cm$^2$ | ASR, Ohm · cm$^2$ |
|---|---|---|
| 600 | 0.18 | 0.93 |
| 650 | 0.14 | 0.67 |
| 700 | 0.12 | 0.49 |

TABLE 3

Characteristics of MT SOFC with anode-supporting design

| Structure of MT SOFC unit | Method of electrolyte formation | Maximal specific power (temperature) | Reference |
|---|---|---|---|
| anode: NiO-YSZ<br>electrolyte: YSZ (2.5 μm)/GDC (2 μm)<br>cathode: LNO-SDC | Magnetron sputtering | 521 mW/cm$^2$ (700 °C) | This work |
| anode: NiO-YSZ<br>electrolyte: ScYSZ (7.2 μm)/SDC (2.2 μm)<br>cathode: LNO-SDC | Dip-coating | 533 mW/cm$^2$ (750 °C) | [17] |
| anode: NiO-YSZ<br>electrolyte: YSZ (18 μm)<br>cathode: GDC /LSCF-GDC/LSCF | Dip-coating | ~400 mW/cm$^2$ (700 °C) | [18] |
| anode: NiO-YSZ/NiO-ScSZ<br>electrolyte: ScSZ (10 μm)<br>cathode: SDC/LSCF | Dip-coating | 418 mW/cm$^2$ (700 °C) | [19] |
| anode: NiO-YSZ/NiO-ScSZ<br>electrolyte: ScSZ (15 μm)<br>cathode: LSM-ScSZ/LSM | Dip-coating | 365 mw/cm$^2$ (700 °C) | [20] |
| anode: NiO-YSZ<br>electrolyte: YSZ (18 μm)<br>cathode: NNO-YSZ | Spray coating | about 700 mW/cm$^2$ (700 °C) | [21] |

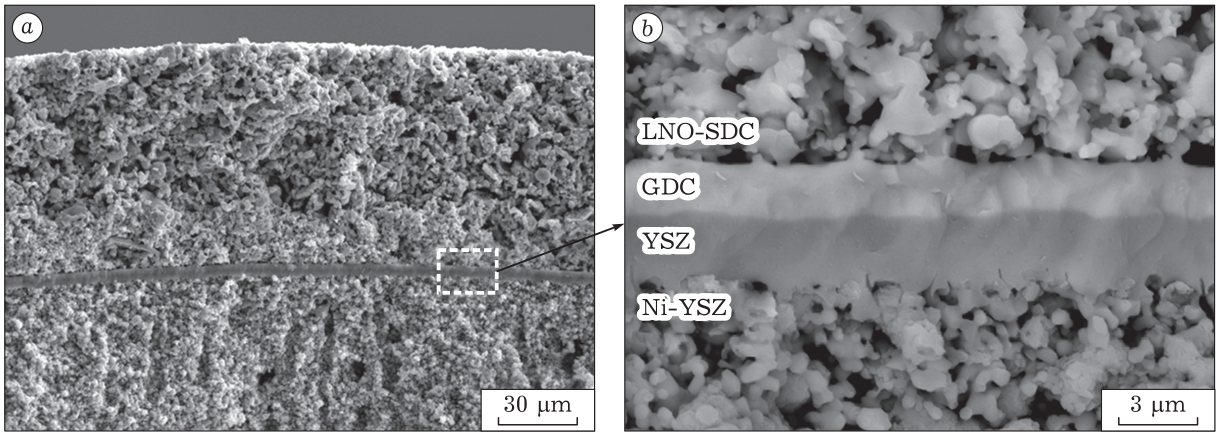


Fig. 4. SEM images of the cross section of MT SOFC after electrochemical experiment at different magnifications. Designations: LNO-SDC = $La_2NiO_{4+\delta}$–$Ce_{0.8}Sm_{0.2}O_{2-\delta}$, GDC = $Ce_{0.9}Gd_{0.1}O_2$, YSZ = $Zr_{0.85}Y_{0.15}O_{2-\delta}$, Ni-YSZ = $NiO/Zr_{0.85}Y_{0.15}O_{2-\delta}$.

sation of the formed layers, results obtained in this work allow us to conclude that the application of magnetron sputtering is highly promising to form the functional layers of MT SOFC.

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