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To cite this version:

Pauline Briault, Mathilde Rieu, Richard Laucournet, Bertrand Morel, Jean-Paul Viricelle. Anode supported single chamber SOFC development for energy recovery of engine exhaust gases. XIVe Congrès de la Société Française de Génie des Procédés, Oct 2013, Lyon, France. pp.No 226. hal-00930150

HAL Id: hal-00930150

https://hal.science/hal-00930150v1

Submitted on 14 Jan 2014

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Récents Progrès en Génie des Procédés, Numéro 104 - 2013
ISSN: 1775-335X ; ISBN: 978-2-910239-78-7, Ed. SFGP, Paris, France

Anode supported single chamber SOFC development for energy recovery of engine exhaust gases

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Abstract

Solid oxide fuel cells (SOFCs) operating in a mixed gas atmosphere (fuel and oxidant), the so-called single chamber SOFCs (SC-SOFCs), have been increasingly studied in the past few years. The absence of sealing between the two compartments provides an easier operation than a classical "two-chambers" SOFC. The single chamber configuration has several advantages over conventional SOFCs: new cell geometries, stack assembly and miniaturization of cells are more easily conceivable. These advantages open the way to new applications such as energy recovery in the exhaust gas by conversion of unburned hydrocarbons into electricity. For that purpose, cells would be embedded at the exit of the engine. Yano *et al.* and Nagao *et al.* demonstrated in 2008 the feasibility of such a device by performing a stack of 12 electrolyte supported SC-SOFCs incorporated at the exit of a scooter engine. However, optimization of the system including architecture, gas mixture and materials modification may lead to enhanced performances.

In the present study, a gas mixture closer to real exhaust conditions has been selected. It is composed of hydrocarbons (HC: propane and propene), oxygen, carbon monoxide, carbon dioxide, hydrogen and water. Only oxygen content has been varied leading to different gas mixtures characterized by three ratios $R=HC/O_2$. Concerning the cell components, a cermet composed of nickel and the electrolyte material, $Ce_{0.9}Gd_{0.1}O_{1.95}$ (GDC) is used as anode and two cathode materials, $Pr_2NiO_{4+\delta}$ (PNO) and $La_{0.6}Sr_{0.4}Co_{0.2}Fe_{0.8}O_{3-\delta}$ (LSCF), have been selected. Anode support is prepared by tape casting; electrolyte material is then screen-printed on top of the green tape. Half cells are co-sintered at 1400°C for 6 hours. Cathode and gold mesh screen printing and sintering conclude cell preparation.

These cells have been tested in the three gas mixtures for temperatures ranging from 400°C to 600°C. Ni-GDC/GDC/LSCF-GDC cell operation has delivered a maximum power density of 15.25 $mW.cm^{-2}$ at 500°C and $R=HC/O_2=0.21$, while lower power densities were obtained for the two other ratios, R=0.44 and R=0.67. Both cathode materials were then investigated in single chamber conditions for the R=0.21 and R=0.44 ratios and LSCF was selected as the most suitable material, delivering the highest power densities. Performances were enhanced by increasing electrolyte thickness to 30 µm, highest power value of 19.35$mW.cm^{-2}$ was obtained at 500°C for R=0.21, which is close to 20 $mW.cm^{-2}$, the power density obtained at higher temperature by Yano and Nagao during single cell tests.

Keywords : Single chamber SOFC, energy recovery, unburned hydrocarbons

1. Introduction

Solid oxide fuel cells are usually described as devices able to convert chemical energy into electrical energy. Conventional solid oxide fuel cells are separated into two compartments containing each electrode split by a gastight insulating electrolyte. Fuel (hydrogen, in most cases) and oxidant (oxygen or air) are directly supplied to each chamber and no mixing of the two gases is tolerated. At the cathode side,

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oxygen is reduced into oxygen ions (1) which travel through the electrolyte to the anode side, where they oxidise hydrogen into water (2). Electrons produced by hydrogen oxidation are then drawn to the cathode through an outer circuit. The overall reaction is the chemical oxidation of hydrogen to water (3).

(1) $O_2 + 4e^- \rightarrow 2O^{2-}$

(2) $H_2 + O^{2-} \rightarrow H_2O + 2e^-$

(3) $H_2 + ½ O_2 \rightarrow H_2O$

Since the early nineties (Hibino *et al.* 1995, Kuhn and Napporn 2010), SOFC have been studied in a new configuration where anode and cathode are exposed to the same gas mixture and contained in a single chamber. No sealing between compartments is thus necessary, removing one of the main challenges of conventional SOFCs. However, in SC-SOFC configuration, hydrogen cannot be used as a fuel since hydrogen-air mixtures are highly reactive, thus hydrocarbons, able to form hydrogen through partial oxidation (4), are most commonly used in the gas mixture.

(4) $C_nH_m + n/2\ O_2 \rightarrow n\ CO + m/2\ H_2$

Since there is no difference in the gas mixture at each sides of the cell, single chamber operation principle is based on the difference of electrodes catalytic activity towards hydrocarbon oxidation. When no current is drawn, the depletion of oxygen at the anode and the maximum concentration of oxygen at the cathode lead to an oxygen partial pressure gradient between both sides of the cell involving open circuit voltage (OCV) creation.

The single chamber configuration has several advantages over conventional SOFCs: new cell geometries, stack assembly and miniaturization of cells are more easily conceivable and development of new applications such as exhaust gas recycling is possible. For this process, which will be developed in the present article, cells would be embedded at the exit of an engine and would turn unburned exhaust gases into electricity, which could then be supplied to electronic devices in the vehicle. This forward-looking energy recovery device could be not only used in vehicles but also in stationary plants as well, in order to reduce hydrocarbon emissions and increase engine efficiency.

To date, only a few authors have investigated such a system. Herman *et al.* (2004) from Hewlett Packard group and Uchiyama (2007) in their patents, developed equivalent devices. Naoki Uchiyama collaborated on testing such a system in real conditions with Takashi Hibino's group; two articles dealing with the results were published in 2008 (Yano *et al.* 2008; Nagao *et al.* 2008). For an electrolyte supported Ni-$Ce_{0.8}Sm_{0.2}O_{1.9}$ (SDC)/ Yttria-stabilised zirconia, 8%Y_2O_3-ZrO_2 (YSZ)/$La_{0.8}Sr_{0.2}MnO_3$ (LSM) single cell, open circuit voltage and maximum power density reached respectively 1100 mV and 20 $mW.cm^{-2}$ at 800°C which can be considered as a too high temperature for the aimed application. A twelve-cells stack embedded at the exit of a scooter engine delivered a power output of 1.2 W, which is still not enough to provide electricity for all electronic devices of the vehicle but proves the feasibility of such a system.

Further optimization including architecture, materials and gas mixture modification might lead to better results. Through this project, some improvements towards better energy recovery are initiated. Anode supported architecture was used rather than the usual electrolyte supported since it has proven its higher ability to deliver better power densities (Shao *et al.* 2011). Indeed, the decrease of the electrolyte thickness leads to lower ionic conduction path and ohmic losses. For the cell components, most performing materials were selected: a traditional cermet composed of nickel and the electrolyte material, $Ce_{0.9}Gd_{0.1}O_{1.95}$ (GDC) as anode, and two cathode materials, $Pr_2NiO_{4+\delta}$ (PNO) (Ferchaud *et al.* 2011), only used up to now for conventional SOFCs, and $La_{0.6}Sr_{0.4}Co_{0.2}Fe_{0.8}O_{3-\delta}$ (LSCF) (Rembelski *et al.* 2012). Concerning the gas mixture used for tests, the articles and patents only exposed evasive or simplified compositions. Herman's patent described a mixture composed of hydrocarbons (e.g. methane, ethane…), hydrogen and oxygen with no exact composition. As for Uchiyama, hydrocarbons, carbon oxides and air are the components of the studied atmosphere, without any further details. Hibino's group is the only one who detailed the concentration of its studied stream for single cell test. A mixture of four hydrocarbons (methane, ethane, propane and butane), supplied at a concentration of 1000 ppm each, with oxygen ranging from 1250 to 15000 ppm is reported. Nevertheless, no exact composition is given for the stack test at the exit of the scooter. The gas mixture used for the present paper is based on a real composition corresponding to the gas mixture exiting from the catalytic converter of a petrol engine.

2. Experimental

2.1. Exhaust stream composition

The gas mixture used in this project is based on data provided by an engine company; it has been modified to fit to laboratory conditions. The given composition corresponds to the actual stream after a catalytic converter. The exhaust stream was hence composed of hydrocarbons (HC): 1/3 propane and 2/3 propene, and oxygen but also of hydrocarbons oxidation products: hydrogen, carbon monoxide, water and carbon dioxide; nitrogen was used as balance gas. Three compositions were employed for the following analyses; their main difference lies in the oxygen concentration involving three different ratios R (HC/O_2): R=0.21 corresponding to oxygen content required for total oxidation, R=0.67 corresponding to partial oxidation and R=0.44, an intermediate ratio. The compositions are detailed in table 1.

Table 1. Gas mixture representative of exhaust conditions

$R=HC/O_2$	HC (ppm)	CO (%)	H_2 (%)	O_2 (%)	CO_2 (%)	H_2O (%)	N_2 (%)
0.21	1000	0.5	0.5	0.48	5	2	91.42
0.44	-	-	-	0.23	-	-	91.67
0.67	-	-	-	0.15	-	-	91.75

2.2. Materials and cell preparation

All raw materials used for electrochemical tests and cell manufacturing are commercial powders. Suppliers and powder specifications are summarized in table 2.

Table 2. Powders specification

Name (composition) of materials	Commercial supplier
LSCF ($La_{0.6}Sr_{0.4}Co_{0.2}Fe_{0.8}O_{3-\delta}$)	Fuel Cell Materials
PNO ($Pr_2NiO_{4+\delta}$)	Marion Technologie
NiO	Sigma Aldrich
GDC ($Ce_{0.9}Gd_{0.1}O_{1.95}$) used for anode preparation	Rhodia
GDC ($Ce_{0.9}Gd_{0.1}O_{1.95}$) used for electrolyte preparation	Fuel Cell Materials

Anode-supported cells were prepared by screen printing of the electrolyte on the green anode made by tape-casting. The slurry for tape casting was prepared according to the formulation previously described by Rieu *et al.* (2012), the powder mixture used in this paper is composed of 60 wt.% of NiO and 40 wt.% of GDC.

The half cells as prepared were then co-sintered in two main steps. At first, a slow temperature increase (60°C/h) was performed up to 600°C with a 1h-dwell to remove the binders contained in the tape. Then a faster temperature increase towards 1400°C and a 6h-dwell were respected to sinter the half-cells. Reduction of the anode was carried out at 650°C for 4 hours in a 6% H_2 atmosphere. Two cathode layers were screen-printed on the reduced half-cell using a 70 wt.% cathode material and 30 wt %. GDC ink (Rembelski *et al.* 2012) and annealed at 1100°C in argon to avoid any anode reoxidation. A gold mesh was screen printed on top of the cathode to enhance the current collection during the experiments. As it is illustrated on figure 1, a typical cell used for experiment is composed of a 15-20µm cathode and a 20µm electrolyte on top of a 400µm thick anode.

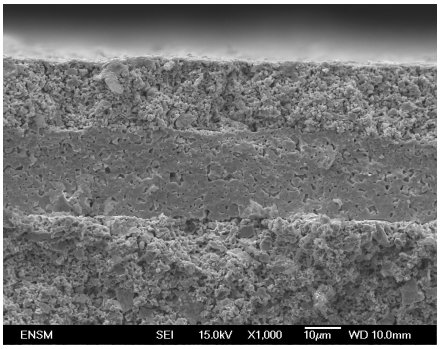

Figure1. Cell cross section observed by Scanning Electron Microscope (SEM)

2.3. Fuel cell investigation

Experiments were performed in a horizontal tubular furnace. The investigated cell was placed in an alumina sample holder. Gold meshes on each side of the cell were used to collect current; two gold wires were stuck on each gold mesh for potential measurements. Electrical measurements were performed by a VersaSTAT 3 from Princeton Applied Research.

Incoming gas mixture was fed to the reactor by monitored mass flow meters. Water was generated by nitrogen bubbling at controlled temperature. At the beginning of each test, temperature is increased to 600°C under nitrogen ($10 L.h^{-1}$) and gas mixture is supplied to the cell during the isothermal stage. Different temperature dwells were done every 50°C between 600°C and 400°C. At each stage, electrical measurements were performed.

After tests, cells microstructures were characterized by a scanning electron microscope (SEM) Jeol JSM 6500. X-ray diffraction (XRD) analyses were made on a Siemens D5000 using Cu Kα radiation at 40 kV and 40 mA.

3. Results and discussion

3.1. Cell operation with three ratios R=HC/O_2 using LSCF as cathode material

Polarization curves were measured for each ratio at different temperatures, an example is illustrated on figure 2. Area specific resistances (ASR) were calculated by determining the slope of the voltage curve at low current densities. Evolutions of the maximum power density, open circuit voltage (OCV) and area specific resistance (ASR) as a function of the temperature are illustrated on figure 3.

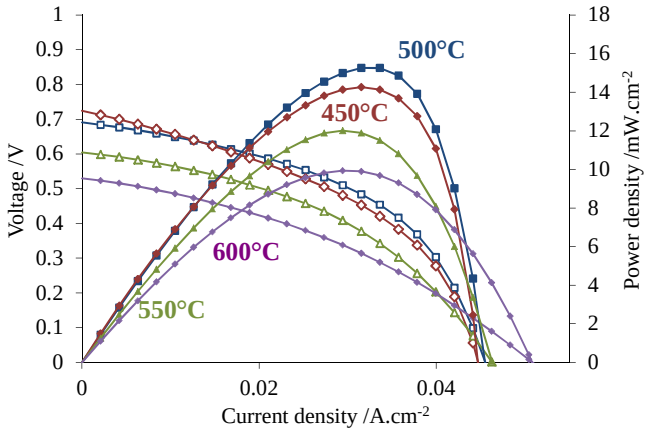

Figure 2.Polarization curve of a cell operated in a R=0.21 gas mixture

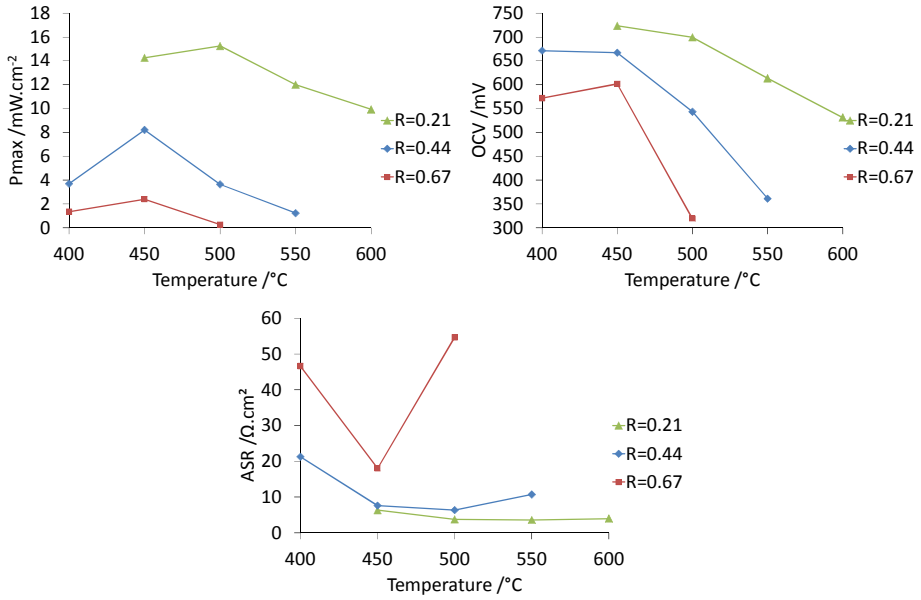

Figure 2.Evolutions of maximum power density, OCV and ASR measured for the three ratios versus temperature

For R=0.67 and R=0.44, no measurements were performed above 500°C and 550°C, respectively. Indeed gas mixtures for these ratios showed that they were not stable at these temperatures forming carbon by reactions between gas components.

For each ratio, power density increases with decreasing temperature until a maximum, and then decreases. The cell operating in the R=0.21 gas mixture was the most performing one. Highest value of 15.25 $mW.cm^{-2}$ was obtained at 500°C for this ratio while only 8.21 $mW.cm^{-2}$ and 2.34 $mW.cm^{-2}$ were measured for 0.44 and 0.67 ratios, respectively. The same trend can be observed for OCV values and ratios can be ordered as follows: $OCV_{0.21}>OCV_{0.44}>OCV_{0.67}$. The relatively low values of open circuit voltage could be attributed to the porous electrolyte letting hydrogen going through the pores from anode to cathode where it consumes oxygen. The gradient of oxygen partial pressure between electrodes is thus decreased and so is the OCV (Rembelski 2012, Suzuki *et al.* 2005). ASR of the R=0.67 ratio is more elevated than for the two other ratios. ASR for R=0.21 and R=0.44 are close, especially when temperature is decreased, their ASR are almost equal for T=450°C.

Post-mortem SEM images were realised on the samples. Sponge-like particles located at some points of the anode were observed for the R=0.21 and R=0.44 ratios, but none was noticed for the 0.67 ratio. This kind of particles was previously seen by Wang *et al.*(2011) and is representative of nickel reoxidation, however it does not seem to affect a lot cell performances.

Moreover, carbon whiskers and nodules were observed on the tested anodes especially for the R=0.67 sample. This can explain the higher value of ASR and the lower OCV obtained during cell operation for the R=0.67 ratio, carbon particles may indeed block the catalytic sites of nickel (Koh *et al.* 2002). On the contrary, less carbon deposition on the R=0.21 cell might explain the highest performance obtained.

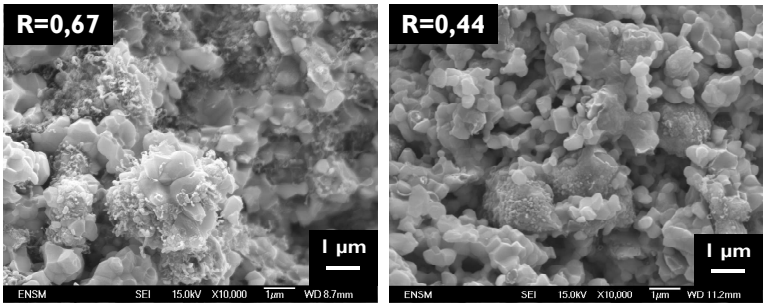

Figure 4. SEM images of carbon deposition for R=0.67 operated cell and nickel oxidation for R=0.44 operated cell

3.2. Cathode material investigation

Considering the results on LSCF for the R=0.67 ratio, this experimental condition was dismissed with PNO cathode material and only tests for the two other ratios were performed with this material. Only R=0.21 results will be exposed here. Evolutions of the maximum power density, OCV and ASR as a function of the temperature for the R=0.21 ratio are presented and compared on figure 4 for both cathodes.

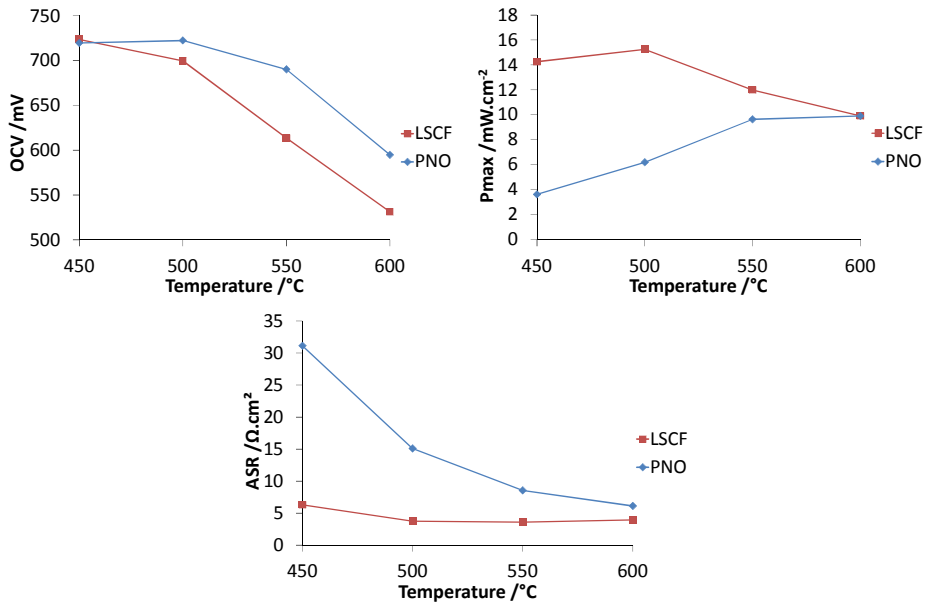

Figure 4. Maximum power density, OCV and ASR versus temperature for the two investigated cathode materials

PNO cathode involves a better OCV than LSCF material for both ratios. However, no greater power density was delivered. At 600°C, maximum power densities of both materials are equal, then, values with PNO cathode decrease with lowering temperature while they increase with LSCF cathode. ASR evaluation showed that PNO owns a higher resistance than LSCF, especially for lower temperature than 600°C.

Indeed, it was observed that, at the end of each test, the gold mesh was completely delaminated from the PNO cathode which can explain the higher resistance and thus the low performances of the cell.

In literature, it is well-known that PNO is a non-stable structure, able to decompose in oxidizing atmospheres (Kovalevsky et al. 2007). XRD analyses were carried out after cell test on this cathode and other phases than the initial PNO were observed, mainly praseodymium oxide Pr_6O_{11}. The newly formed phases might be the origin of the ASR rise by limiting the grip of the current collector on the cathode.

This cathode material was thus considered as non-suitable for cell operation in the present conditions and further experiments were made using only LSCF as cathode.

3.3. Electrolyte thickness

As the 20 µm electrolyte was not fully dense, increasing its thickness might decrease its open porosity going from one electrode to the other and block gas diffusion through these pores (Rembelski 2012, Suzuki et al. 2005). Open circuit voltage would thus be enhanced thanks to an increased oxygen partial pressure gradient between electrodes. Experiments were thus performed for cell with thicker electrolyte (30 µm) and compared to the previous ones made with a thinner electrolyte (20µm). Results for the R=0.21 ratio are presented on figure 6.

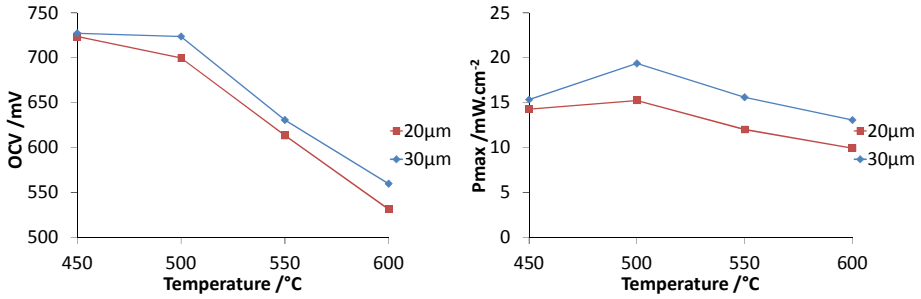

Figure 6. OCV and maximum power density versus temperature for the two investigated electrolyte thickness

An effective improvement of open circuit voltage for the 30 µm electrolyte cell was noticed especially at highest temperatures. A greater maximum power density was thus involved. The highest value obtained at 500°C increases from 15.25 mW.cm^{-2} for a 20 µm electrolyte to 19.35 mW.cm^{-2} for a 30 µm electrolyte, reaching a very close result to that of the previous work by Yano *et al.* and Nagao *et al.* but for a lower temperature.

4. Conclusions

This paper deals with an innovative concept of energy recovery from exhaust gases produced by a vehicle exhaust. For this project, most performing architecture and materials were used in exhaust gas conditions and a more realistic temperature range was studied. Three different gas mixtures were investigated for an anode supported Ni-GDC/GDC/LSCF-GDC cell. The lowest oxygen containing ratio (R=0.67) was ruled out because of huge carbon formation on the anode side. However, the two other ratios (R=0.44 and R=0.21) have delivered highest power densities, reaching 15.25 mW.cm^{-2} at 500°C for the oxygen richer ratio (R=0.21). PNO was also investigated as cathode material. Although a greater open circuit voltage was created, the maximum power densities were lower than those obtained with LSCF especially when the temperature is decreased. Indeed, new phases formed during the cell operation have probably caused the increase of the ASR.

Greatest performances were obtained with a Ni-GDC/GDC/LSCF-GDC cell by increasing the electrolyte thickness up to 30 µm, 19.35 mW.cm^{-2} were delivered at 500°C. Similar results to literature ones were thus reached but for lower temperatures and gas mixture closer to real conditions.

Acknowledgements

Funding for this project was provided by a grant from la Région Rhône-Alpes, France.

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Récents Progrès en Génie des Procédés, Numéro 104 - 2013
ISSN: 1775-335X ; ISBN: 978-2-910239-78-7, Ed. SFGP, Paris, France

Développement de piles à combustible SOFC monochambres pour la récupération d'énergie des gaz imbrûlés d'un moteur thermique

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Résumé

Les piles à combustible SOFC (Solid Oxide Fuel Cell) en configuration monochambre ont fait l'objet de nombreuses études ces dernières années. L'absence de scellement entre les compartiments anodique et cathodique permet de simplifier leur utilisation par rapport aux piles à combustible conventionnelles. Dans cette étude, une pile SOFC monochambre est développée en vue d'être utilisée comme outil de production d'électricité à partir d'hydrocarbures imbrûlés par la combustion d'un moteur thermique, améliorant ainsi son efficacité énergétique. Les cellules seraient incorporées dans la ligne d'échappement d'un véhicule en sortie de moteur. Yano *et al.* et Nagao *et al.*, en 2008, ont démontré la faisabilité d'un tel système en étudiant un stack de douze cellules en sortie de moteur d'un scooter. Une optimisation du procédé incluant des modifications de l'architecture de la pile, des matériaux de cellules et du mélange de gaz pourrait mener à de meilleures performances.

Notre projet est axé sur l'étude de piles SOFC monochambres en architecture anode support fonctionnant dans un mélange d'hydrocarbures HC (propane et propène), d'oxygène, de monoxyde de carbone, d'hydrogène, de dioxyde de carbone et d'eau. Seule la quantité d'oxygène a été modifiée et trois compositions de gaz différentes désignées chacune par un rapport $R=HC/O_2$, ont été étudiées.

Un cermet composé de nickel et du matériau d'électrolyte, $Ce_{0.9}Gd_{0.1}O_{1.95}$ (GDC) a été choisi comme matériau d'anode et mis en forme par coulage en bande. L'électrolyte GDC a été sérigraphié sur l'anode crue puis la demi-cellule a été co-frittée à 1400°C pendant 6 heures. La cathode et la grille d'or utilisée comme collecteur de courant sont ensuite déposées par sérigraphie et recuites consécutivement.

Ces cellules ont été testées pour les trois rapports R pour des températures allant de 400 à 600°C. Une cellule Ni-GDC/GDC/LSCF-GDC a délivré une densité de puissance maximale de 15.25 $mW.cm^{-2}$ à 500°C et $R=HC/O_2=0.21$, tandis que de plus faibles densités de puissance ont été obtenues pour les deux autres rapports, R=0.44 and R=0.67. Deux matériaux de cathode ont été étudiés dans les mêmes conditions et comparés : $La_{0.6}Sr_{0.4}Co_{0.2}Fe_{0.8}O_{3-\delta}$ (LSCF) et $Pr_2NiO_{4+\delta}$ (PNO). LSCF a été considéré comme étant le matériau le plus adapté au fonctionnement dans les conditions choisies, les cellules Ni-GDC/GDC/LSCF-GDC ayant fourni les meilleures puissances. Les performances de ces cellules ont pu être améliorées en augmentant l'épaisseur de l'électrolyte et ont atteint une valeur de 19.35$mW.cm^{-2}$ à 500°C pour R=0.21, résultat relativement proche de 20 $mW.cm^{-2}$, la densité de puissance obtenue à plus haute température (800°C) par Yano et Nagao durant leurs tests en monocellules.

Mots-clés : SOFC monochambre, récupération d'énergie, hydrocarbures imbrûlés

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