

## Innovative low temperature SOFCs and advanced materials

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### Abstract

High ionic conductivity, varying from 0.01 to 1 S cm<sup>-1</sup> between 300 and 700 °C, has been achieved for the hybrid and nano-ceria-composite electrolyte materials, demonstrating a successful application for advanced low temperature solid oxide fuel cells (LTSOFCs). The LTSOFCs were constructed based on these new materials. The performance of 0.15–0.25 W cm<sup>-2</sup> was obtained in temperature region of 320–400 °C for the ceria-carbonate composite electrolyte, and of 0.35–0.66 W cm<sup>-2</sup> in temperature region of 500–600 °C for the ceria-lanthanum oxide composites. The cell could even function at as low as 200 °C. The cell has also undergone a life test for several months. A two-cell stack was studied, showing expected performance successfully.

The excellent LTSOFC performance is resulted from both functional electrolyte and electrode materials. The electrolytes are two phase composite materials based on the oxygen ion and proton conducting phases, or two rare-earth oxides. The electrodes used were based on the same composite material system having excellent compatibility with the electrolyte. They are highly catalytic and conductive thus creating the excellent performances at low temperatures. These innovative LT materials and LTSOFC technologies would open the door for wide applications, not only for stationary but also for mobile power sources.

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**Keywords:** Low temperature solid oxide fuel cells; Ion-doped ceria; Ceria-based composite electrolyte

### 1. Introduction

In the present solid oxide fuel cell (SOFC) R&D, many efforts are contributed to reduce the operation temperature either by using thin film technology for yttrium stabilized zirconia (YSZ) electrolyte [1–4] or by using alternative electrolyte materials, such as various ionic doped ceria and lanthanum gallate electrolytes, etc. [5–12] that can make the SOFCs function at lower temperatures. All these efforts have, however, limitations due to the deficiency of technology and the instability of the material.

Our approach is to develop new hybrid and nano-composite materials based on rare-earth oxides both for electrolytes and the compatible electrodes. Low temperature, say 300–600 °C, operation for SOFCs is realized based on a research

breakthrough in innovative nano- and hybrid conducting ceria-based composite materials. These materials were prepared usually by mixing different cationic doped ceria, e.g. gadolinium doped ceria (GDC), yttrium doped ceria (YDC) and samaria doped ceria (SDC) with various salts, e.g. chlorides, fluorides, hydroxides and carbonates, or by two rare-earth oxides based on the ceria. The materials contain at least two phases, e.g. the ceria phase and the salt phase, or the ceria phase and other rare-earth oxide phase different from ceria. Previous studies were carried out on the GDC and salts, e.g. GDC–NaCl [13], GDC–LiCl–SrCl<sub>2</sub> [14], –NaOH [14], and carbonate (e.g. MCO<sub>3</sub>, M = Ca, Ba, Sr) composites, demonstrating promising results for low temperature solid oxide fuel cell (LTSOFC) applications [14,15]. This paper continues the study on the ceria-based composites. Two composite systems can be used as typical examples: (i) SDC and alkaline carbonates, i.e. M<sub>2</sub>CO<sub>3</sub>, M = Li, Na, K, composites; and (ii) ceria-lanthanum oxide composites. Their material properties and LTSOFC applications are reported in this work.

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## 2. Preparation and characterization

### 2.1. SDC– $M_2CO_3$ , $M = Li, Na, K$ composites

**Step 1.** Nano-structured ion doped ceria, e.g. samaria doped ceria (SDC) was prepared through wet-chemical route, i.e. co-precipitation approach. Raw chemicals were used as follows: cerium nitrate hexahydrate and samarium(III) nitrate hexahydrate (supplied by Sigma–Aldrich). These chemicals were prepared as 1 M solutions. Then the samarium(III) nitrate hexahydrate solution was mixed with the cerium nitrate hexahydrate solution according to the desired molar ratios, e.g. 20 mol% Sm in ceria. An appropriate amount of oxalate acid solution (2 M) was added to prepare the SDC precursor in the oxalate state. The precipitate was rinsed several times in deionized water, followed by ethanol washing for several times in order to remove water from the particle surfaces. The obtained precipitates were dried in an oven at 100 °C over night and then ground in a mortar. The resulting powder was sintered at various temperatures between 300 and 600 °C for 1 h. The sintered powders were ground completely for use.

**Step 2.** SDC and  $M_2CO_3$  ( $M = Li, Na, K$ ) powders were prepared by mixing the SDC and carbonates according to the weight ratio of 80 wt.% SDC: 20 wt.% binary carbonates, e.g. Li–Na, Li–K, or Na–K carbonates. A typical composition was used like 67 mol%  $Li_2CO_3$ : 33 mol%  $Na_2CO_3$ . The mixtures of the SDC and binary carbonates were ground, then sintered at 650 °C for 0.5–1.0 h. The sintered sample was ground again for various electrical and fuel cell measurements.

### 2.2. Nano- and fine $CeO_2$ – $La_2O_3$ composites

Through wet-chemical route the  $CeO_2$ – $La_2O_3$  composites were prepared using related nitrate compounds. The 1 M solution of the mixture of  $Ce(NO_3)_3 \cdot 6H_2O$  and  $La(NO_3)_3 \cdot 6H_2O$  supplied by Sigma–Aldrich was prepared in various molar ratios for Ce:La from 5:1 to 1:4 followed by strong stirring. The precipitation was completed by adding appropriate amounts of ammonia liquid (33%). The precipitate was washed by distilled water and dried in oven at 180 °C to get homogeneous powders. The dried powders were heated at 500–700 °C for 1–2 h to get fine powders from nano to micrometer scale depending on the preparation skills and the sintering temperatures. The sintered samples were ground completely for various measurements.

### 2.3. Characterisation

The crystal structure was analyzed by X-ray diffractometer (XRD) with  $Co K\alpha$  radiation, 35 kV voltage and 30 mA current. Scanning electron microscopy (SEM), scanning probe microscopy (SPM) and energy dispersion spec-

troscopy (EDS) were used to determine the morphology and the composition of the samples.

The electrical properties were studied using a.c. impedance analysis. The measurements were conducted in the frequency range from 5 Hz to 13 MHz using a computerised HP 4192A LF Impedance Analyzer. The temperature range used for the measurements was from 300 to 700 °C. The measurements were usually performed in air. The FCs were constructed by a hot-pressing procedure. The hot-pressing technique involves loading a mold with the powders of anode followed successively by the electrolyte and finally the cathode, all being pressed in one step to form a complete FC assemble for measurements. The pressed FC cell size was 13–20 mm in diameter (two molded sizes) and 1.0–1.5 mm in thickness, which consisted of an approximate 0.3–0.5 mm thick electrolyte, 0.3–0.5 mm thick electrodes, anode and cathode, respectively. The anode was composed of NiO mixed with the electrolyte and the cathode was based on lithiated NiO with the electrolyte. Two-cell stack was constructed based on the common planar-type SOFC stack technology. Regular stainless steel was employed for the bipolar plates and stack materials. In the cathode chamber (air side) of the bipolar plate/stack was covered by a silver thin film to protect the oxidation of the stainless steel from the air and high temperature operation. The FCs were measured between 200 and 650 °C. Hydrogen was used as the fuel, air as the oxidant. The gas (fuel and air) flows were controlled between 100 and 200 ml/min under 1 atm pressure.

## 3. Results and discussion

### 3.1. Material phase and micro structures

Pure SDC has a fluorite structure, which can be crystallized from the SDC oxalate precursor above 350 °C. Temperature at 400 °C is sufficient to make the SDC good crystalline structure. Fig. 1 shows the structure of the SDC sample being heat-treated at various temperature changes from the amorphous/gel state, see Fig. 1(a), in the precursor state, to the crystalline state at 400 °C, (b) and then to remain this crystalline state for high temperatures, see (d) for 560 °C. The particle size is growing with the sintering temperature, resulting in their sharper and narrower XRD peaks. In Fig. 1, the sharpest XRD peaks are observed for 560 °C. From this narrowing effect of the XRD peaks, we can calculate the corresponding crystalline particle sizes growing from 20 (350 °C) to 50 nm (400 °C) and then to 90 nm (560 °C), respectively.

For the SDC composites, the introduction of the carbonates does not change the SDC phase structure. For example, in the SDC-carbonates composites, a typical composition: 80 wt.% SDC: 20 wt.% (67 mol%  $Li_2CO_3$ : 33 mol%  $Na_2CO_3$ ) written as SDC–20NLCO, only one set of SDC XRD pattern is observed, which is the same as (b). In this

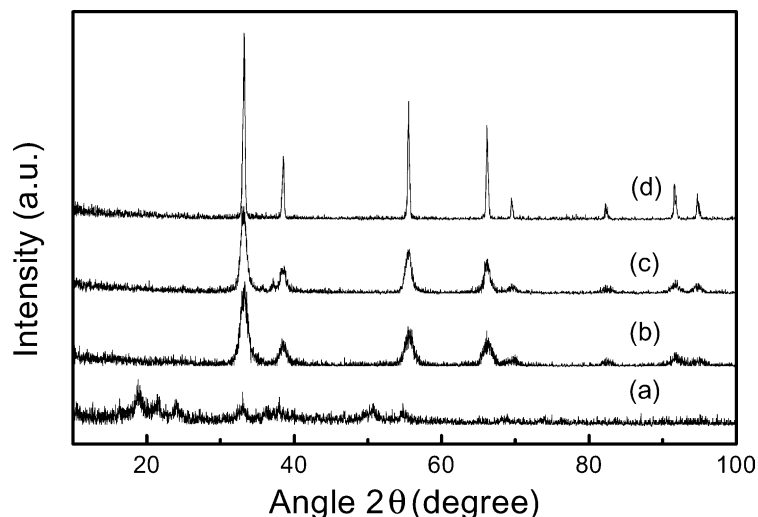


Fig. 1. XRD patterns for SDC and SDC-20NLCO at various temperatures: (a) SDC precursor state; (b) SDC at 400 °C; (c) SDC-20NLCO at 400 °C; (d) SDC at 560 °C.

case, the NLCO may be assumed to exist as an amorphous phase co-existing with the SDC, highly likely to cover on the SDC particles because during the heat-treatment the NLCO can be melted and be coated on the SDC particles. On the other hand, there are no any new XRD patterns observed for the SDC-20NLCO, which means there is neither a chemical reaction nor any intermediate compound between the SDC and the NLCO materials/phases. The microstructure of the SDC-20NLCO was further studied by SEM. Significant changes from the particle topography can be seen from the SEM analysis as shown in Fig. 2. In the SEM analysis, the nano-grain size cannot be clearly and directly observed because the particles are always formed in an aggregate group during the preparation and a heat-treatment process. For low temperatures, e.g. at 400 °C, heat-treatment, see Fig. 2(a), a clear separated interface and individual particles can be identified. However, a homogenous composite bulk was formed without clear interfaces for the sample heated at 500 °C, see Fig. 2(b). The particle (aggregate) geographies

are quite different at the different heat-treatment temperatures. For the 500 °C heat-treatment, the surface morphology of the SDC-20NLCO composite particles (aggregate) appears very ambiguous to form a homogenous bulk, which may be caused by the molten NLCO covering homogeneously on the SDC particle surfaces, thus the particles form as a nano-composite based on a nano-SDC core; while for the heated temperature below the NLCO melting point, e.g. 400 °C, the particles (aggregate) look finer with clear interfaces, see Fig. 2(a).

These studies on the phase and microstructures can help us to better understand the ion-conduction and FC processes and performances for the SDC-carbonate composite materials.

### 3.2. LT-SOFC performances using various ceria-based composite materials

Higher temperatures (above 400–600 °C) performances for the SDC-20 NLCO FCs were reported earlier [16]. Low

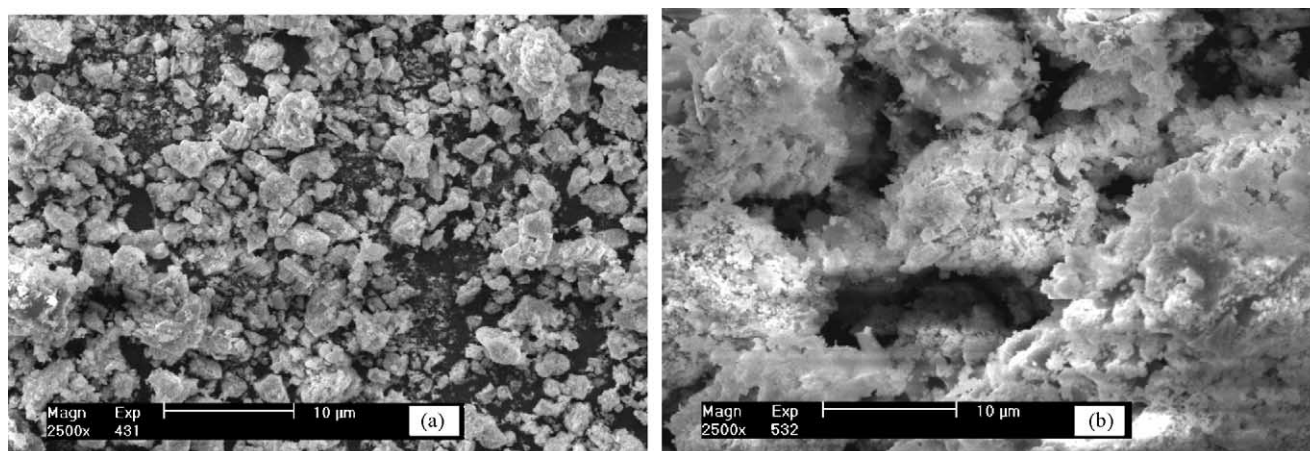


Fig. 2. SEM photos for the SDC-20NLCO: (a) 400 °C; (b) 500 °C.

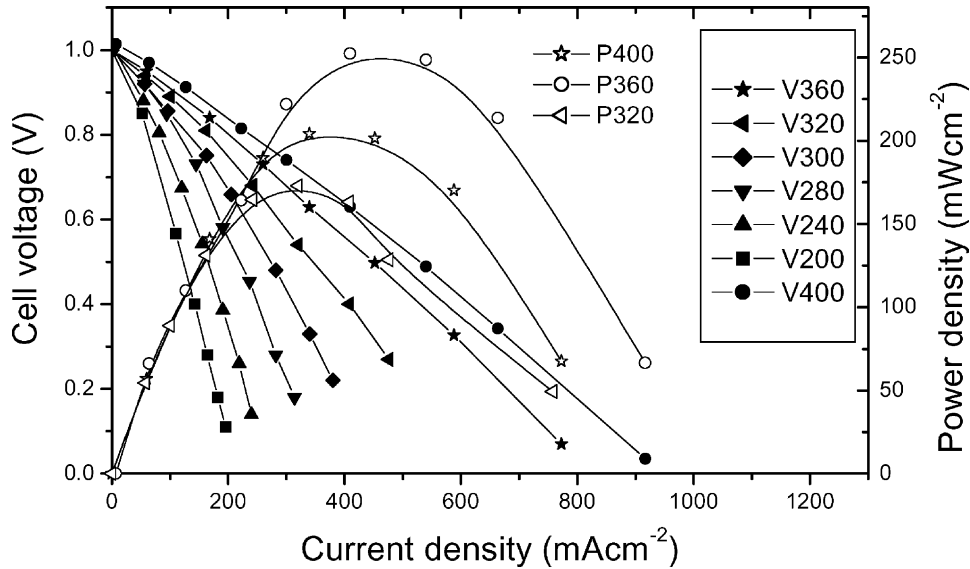


Fig. 3. The SDC-20NLCO LTSOFC performances at low temperatures below 400 °C. Fuel H<sub>2</sub>, oxidant: air, gas flow: 80–120 ml/min, gas pressure: 1 atm.

temperature (below 400 °C) studies for the SDC-20NLCO FCs are thus being focused on here. The SDC-20NLCO composites have actually sufficient conductivities at low temperatures, e.g. larger than  $10^{-2}$  S cm<sup>-1</sup> for temperatures above 300 °C [14]. In order to develop high performance LTSOFCs at lower temperatures, e.g. below 400 °C, we have to use the compatible catalyst electrodes. Our targets are to develop high catalyst and functional electrode materials without noble element containments. In recent achievements, we have successfully obtained these new high performance electrodes through a novel microwave-synthesis procedure. With this achievement, the LTSOFC performance can be improved significantly by these high catalyst electrodes at low temperature operation. Fig. 3 shows some typical cell performances, *I-V* and *I-P* characteristics, for a

cell using the SDC-20NLCO as the electrolyte between 200 and 400 °C. The cell power output can reach 250 mW cm<sup>-2</sup> (the peak value) at 400 °C, and 155 mW cm<sup>-2</sup> at 320 °C. The cell can even function at as low as 200 °C.

On the other hand, without composing carbonates, pure oxide composites based on ceria can also function for high performance LTSOFCs. Earlier studies were carried out on the CeO<sub>2</sub>-Al<sub>2</sub>O<sub>3</sub>, and -SiO<sub>2</sub>, which were discovered as high conductive nano-composite materials with potential applications for SOFCs [17,18]. However, their conductivities are still not sufficiently high to meet the demands for high performance LTSOFCs. In this work, we have developed further two rare-earth oxides composite based on ceria, e.g. CeO<sub>2</sub>-La<sub>2</sub>O<sub>3</sub>. Fig. 4 displays some typical results for the LTSOFCs using the CeO<sub>2</sub>-La<sub>2</sub>O<sub>3</sub> prepared by the wet-

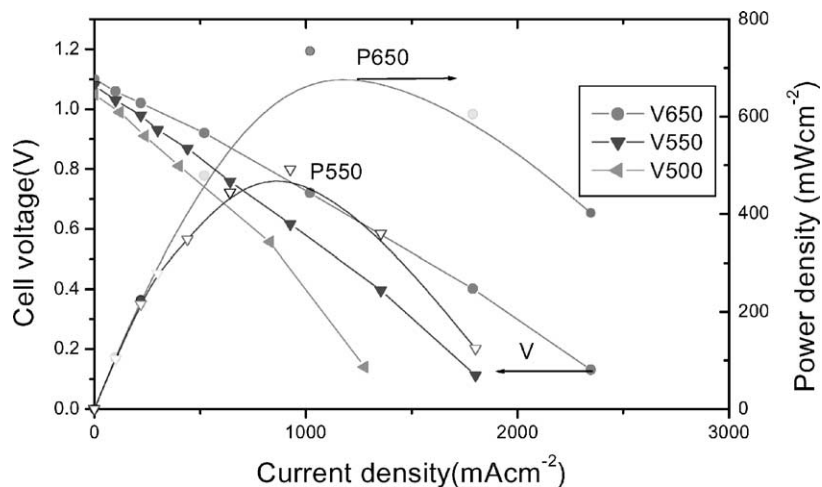


Fig. 4. *I-V/I-P* characteristics of a LTSOFC with the CeO<sub>2</sub>-La<sub>2</sub>O<sub>3</sub> (3:2 in molar ratio) as the electrolyte at various temperatures. Fuel: H<sub>2</sub>, oxidant: air, gas flow: 80–120 ml/min, gas pressure: 1 atm.



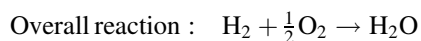
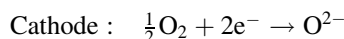
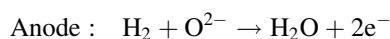
chemical approach mentioned as above. It reaches a peak power output of 0.72, 0.50, and 0.35 W cm<sup>-2</sup> at 650, 550 and 500 °C, respectively. Corresponding to the FC performances, the conductivity of the CeO<sub>2</sub>–La<sub>2</sub>O<sub>3</sub> electrolyte was measured at 4 × 10<sup>-2</sup> to 10<sup>-1</sup> S cm<sup>-1</sup> between 500 and 600 °C. This conductivity value is excellent for developing high performance LTSOFCs. It is interesting to notice that the independent CeO<sub>2</sub> and La<sub>2</sub>O<sub>3</sub> are insulators, or being very low conductive oxides depending on the impurity containments; but the composite of these two phases is highly conductive. Similar phenomena were observed before for CeO<sub>2</sub>–Al<sub>2</sub>O<sub>3</sub>, and –SiO<sub>2</sub>.

Nano-technology for preparation of the ceria-based composites is of high value, especially in a good controlling level both for the microstructure and the molecule. The host oxide structure-dependent properties of these ceria-based composites prepared by the nano-technology are significantly different from those of the conventional bulk materials in many cases [18]. High defect concentration existing in the nano-structured host oxide phase can provide a large number of active sites for ion conduction as well as gas–solid catalysis. In addition, high diffusivity through nanometer-sized interphase boundaries promotes fast kinetics of catalyst activation and ion transportation.

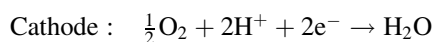
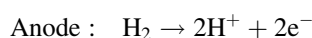
It should be pointed out that the difference between the conventional ionic doped ceria and the ceria-composites is that the former is a single phase material (so called solid solution) with strict limits for the dopant kinds and doping level/amount [19]; while the ceria-composites are two or multi phase materials, whose respective oxide/salt component remains as its own phase structure, and the mixed amount is far larger than the doping level. In the ceria-composites, significant enhancement for the ionic conductivity and diffusivity can be caused by the interfaces between two phases in contrast to the single phase. In the nanometer particle surface of CeO<sub>2</sub>, some additional oxygen ions can be adsorbed on the surface of CeO<sub>2</sub> particles with the n-type semiconduction, which essentially consists of an interfacial conducting path for oxygen ions. Consequently, some enhanced oxygen ionic conduction takes place. Thus, the nano-meter CeO<sub>2</sub>–La<sub>2</sub>O<sub>3</sub>, –Al<sub>2</sub>O<sub>3</sub>, –SiO<sub>2</sub> are highly conductive though the CeO<sub>2</sub>, La<sub>2</sub>O<sub>3</sub>, Al<sub>2</sub>O<sub>3</sub> and SiO<sub>2</sub> are insulators, respectively.

For the ceria-composites, either ceria-salt composite or two oxides based on ceria in the reduced atmosphere, e.g. hydrogen of the fuel cell, the CeO<sub>2</sub> is exposed in the hydrogen of the fuel cell atmosphere, which can cause a certain electronic conduction (n-type semiconduction) on the CeO<sub>2</sub> particle surface due to the reduction of Ce<sup>4+</sup> to Ce<sup>3+</sup>, extra electrons were produced [20–22], these ceria particles thus forming a semiconducting surface. Thus, the adsorbents, such as O<sub>2</sub><sup>-</sup>, O<sup>-</sup>, and O<sup>2-</sup>, could cover the ceria surface. These negatively charged oxygen adsorbents, especially, the additional oxygen ions on the surface of the ceria particles may essentially consist of an interfacial conducting path. Consequently, some enhanced oxygen ionic conduc-

tion occurs. This conducting mechanism is probably essential for the function and enhancement of the LTSOFC performance using these ceria-based composites as the electrolytes. In addition, the ceria-salt-composites have a hybrid conduction of oxygen ion (from ceria) and proton conduction (from the salt). By using this type of material as the electrolyte, the fuel cell processes would be performed by both proton and oxygen ion conduction. In the oxygen ion conduction case and H<sub>2</sub>-air (O<sub>2</sub>) fuel cell, the cell reactions take place as:



While in the case of proton conducting H<sub>2</sub>-air (O<sub>2</sub>) fuel cell, the reactions as:



Both oxygen ion and proton conduction have the same overall cell reaction, resulting in the same fuel cell voltage, open circuit voltage (OCV) (PS: under standard conditions this OCV is 1.23 V at 25 °C), but the cell reaction product, H<sub>2</sub>O, will be produced simultaneously at both anode and cathode sides. This is neither the case in conventional SOFC with solely oxygen ion conduction nor the case of the proton conduction fuel cell. In these cases, the water can only be formed either in the anode or in the cathode side. In our experiments, water was indeed observed in both electrode sides suggesting the existence of both proton and oxygen ion conduction. However, because of predominant oxygen ion conduction over proton conduction, most water was observed in the anode side for the fuel cells investigated. The combination of proton conduction has dual effects. On one hand, it enhances the material conductivity; and on the other hand, it may promote the electrode reaction and the kinetics between the electrolyte and electrode interfaces, consequently enhancing the current exchange rate resulting in the high current outputs. In fact the electrode–electrolyte interfaces are keys to determine the FC performance as observed for other SOFCs using the pure oxygen ion conducting electrolytes, e.g. YSZ.

Using the ceria-based composite electrolytes, a two-cell stack was constructed based on the common planar-type SOFC stack technology and tested. The tested single cell size was 20 mm in diameter, and two single cells were constructed in a series connection. Fig. 5 shows this stack performance, see curve (b), in contrast to the single cell, curve (a). As expected this two-cell stack can reach almost double power output than that of the single cell.

Finally, a typical ceria-based composite electrolyte LTSOFC was examined by experiencing a long-term life testing experiment. The cell was tested once every week for

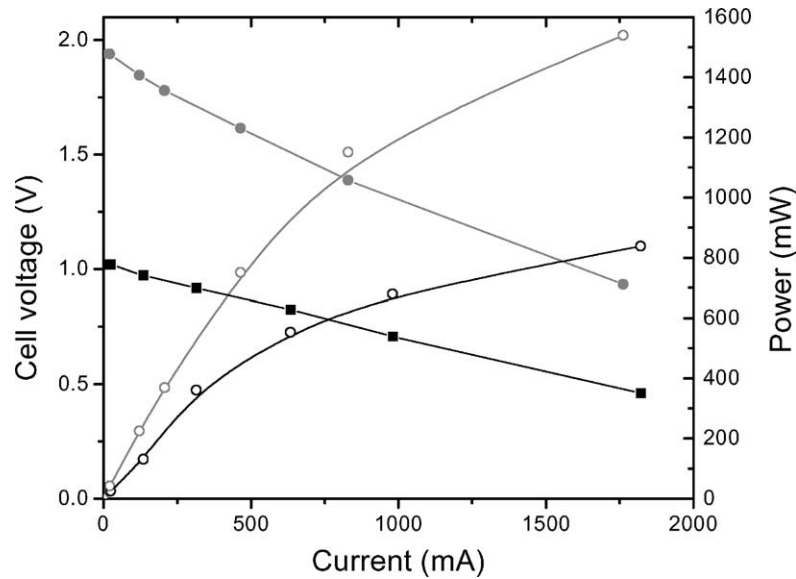


Fig. 5. Cell performance for a two-cell stack at 600 °C H<sub>2</sub>/air operation.

2–3 h operation. Then the cell was interrupted from the testing and stored in the atmosphere. The tests had been lasted for 4.5 months, and further measurements are still under investigation. Fig. 6 shows the performances achieved so far from this testing cell. The cell shows better and better performance after operation during the first 3 months. This may be due to the fact that the cell performance can be improved by discharging corresponding to an improvement of the cell compatibilities and interfaces between the electrolyte and electrodes. The measurement performed 4.5 months later shows a significant degradation in the cell performance, however, it still being better than the operation of the initially fresh cell.

Based on these ceria-composite electrolytes, their LTSOFC applications have demonstrated wide fuel flexibility for direct operation with many types of fuels, such as nature gas, biomass gas, coal gas, alcohol, ammonia, etc. [23]. In addition, these new materials are extremely cost-effective and the relevant LTSOFC technology developed based on these new advanced materials is strongly market-competitive. All these exhibit an enormous potential on developing commercial-type LTSOFC technology. It is expected that the continuous R&D on these new materials may spur an acceleration of the SOFC commercialization and the change of the fuel cell R&D strategy on an advanced and marketable new generation.

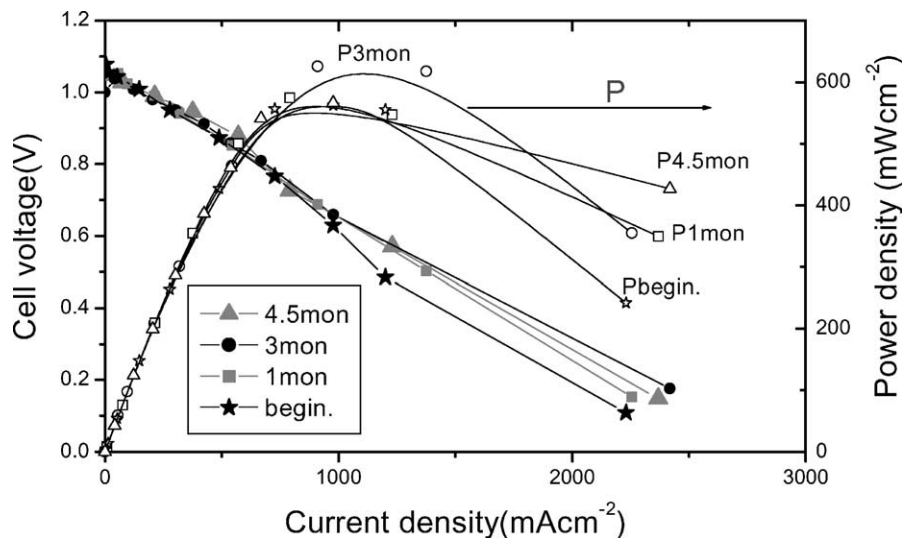


Fig. 6. Cell performances at 550 °C for lift test, H<sub>2</sub>/air operation.

#### 4. Conclusions

Material R&D focusing on LTSOFCs has been concentrated on various ceria-based composite materials. These new advanced materials have been developed based on the nano-technology, ceramic composite and hybrid conducting material technologies. They have the conductivity in the region of  $0.01\text{--}1.0\text{ S cm}^{-1}$  in the LT region, and their LTSOFC applications are successful.

Being different from the conventional YSZ and pure ion doped ceria materials used in the conventional SOFCs, the new ceria-based composite materials can be the two phase composite ceramics with both oxygen ion and proton conduction, or with two non-conducting rare-earth oxides based on the ceria. In the ceria-salt composites, one phase, e.g. ion doped ceria, has high oxygen ion conductivity; while the other phase, the salt, has significant proton conductivity. Certain proton conduction may promote the electrode reaction and the kinetics between the electrolyte and electrode interfaces, consequently enhancing the current exchange rate resulting in high current outputs.

Advanced ceria-composite electrolytes combined with the high performance and compatible electrodes have made excellent LTSOFC performances, the cell functioning at as low as  $200\text{ }^{\circ}\text{C}$ . It can be believed that these innovative advanced materials and their continuous developments will promote a new marketing SOFC technology with great marketing potential and bright future.

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