

A H_2S Intermediate-Temperature Fuel Cell with Nano-Composite Li_2SO_4 Proton-Conducting Membrane*

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Abstract: A nano-composite Li_2SO_4 proton-conducting electrolyte and a new preparation procedure of membrane-electrode assembly (MEA) were developed for the electrochemical oxidation of H_2S . Instead of the traditional screen-printing method, in the MEA, both the anode and cathode catalysts were simultaneously pressed to form the cell with nano-composite electrolyte. This allows the design to possess some advantageous configurations that can diminish the Ohmic resistance between the electrolyte and the electrodes, enhance the mechanical and electrical properties, and improve the performance of fuel cells due to the membrane thickness reduction and the good contact between the electrolyte and the electrodes. The electrolyte was then characterized by scanning electron microscope (SEM) and electrochemical impedance spectrum techniques. The results indicate that the nano-composite materials improve the electrolyte integrity, and that no cross-over of H_2S through the improved electrolyte occurs due to its high density, good compactivity and gas-impermeability. Moreover, MEA is stable in H_2S stream. For a single cell with the configuration of H_2S , ($\text{MoS}_2/\text{NiS} + \text{Ag} + \text{electrolyte} + \text{starch}$)/ $\text{Li}_2\text{SO}_4 + \text{Al}_2\text{O}_3$ /($\text{NiO} + \text{Ag} + \text{electrolyte} + \text{starch}$) and air in a MEA thickness of 0.8 mm and a Li_2SO_4 to Al_2O_3 weight ratio of 65:35, the maximum power density is about $130 \text{ mW}/\text{cm}^2$ and the corresponding current density is about $200 \text{ mA}/\text{cm}^2$ at 680°C .

Key words: solid oxide fuel cell; membrane-electrode assembly; hydrogen sulfide; proton-conducting membrane

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0 Introduction

H_2S is one of the most noxious, poisonous and abundant air pollutants, which is found in natural gas and several processing and industrial effluent gas streams. There are various prior art methods for H_2S disposal. H_2S is converted to SO_2 and elemental sulfur using the Claus process. In the Claus process, the heat generated by oxidation reaction is generally not uti-

lized, and the capital investment can not be economically justified. Other disadvantages of the Claus process are that it is of a H_2S conversion efficiency of only about 92% and other pollutants, such as CS_2 and COS , are normally produced. Solid oxide fuel cells (SOFCs) provide an economically and environmentally desirable alternative H_2S processing capability by producing high-grade electric power from large amount of chemical energy associating with the oxidation of H_2S ^[1-3]. The first direct H_2S -air solid oxide fuel cell was reported by Pujare et al^[1] in 1987. Then, efforts have been directed toward the improvement of cell performance^[2-8]. High-performance composite metal sulfide-based anodes for H_2S solid oxide fuel cell (SOFC) were developed recently in our group. A composite Ni-Mo-S catalyst derived from MoS_2 and NiS , admixed

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with YSZ as ionic conductor and Ag as electronic conductor, had good performance and durability^[7]. The nature of the electrolyte determines the manufacturing requirements and operation mode of conventional H₂ fuel cell, and this is also true for H₂S fuel cells. The ions (H⁺ or O²⁻) conducted within the electrolytes determine the electrochemical processes occurring in the fuel cell, as shown in Table 1. When proton-conducting electrolytes are used in an H₂S fuel cell, high-purity sulfur is the only product obtained in the anode chamber and water is the only product formed in the cathode chamber. With oxide ion-conducting electrolyte, element sulfur, H₂O and SO₂ are formed in the anode chamber, thus requiring further removal of SO₂.

Table 1 Electrochemical reactions of H₂S SOFC with different electrolytes

Electrolyte type	Anode reaction	Electrolytic conduction	Cathode reaction	Overall cell reaction
Proton-conducting	$\text{H}_2\text{S} - 2\text{e}^- \rightarrow 2\text{H}^+ + 1/2\text{S}_2$	proton transfer from anode side to cathode side	$\text{O}_2 + 4\text{H}^+ + 4\text{e}^- \rightarrow 2\text{H}_2\text{O}$	$\text{H}_2\text{S} + \text{O}_2 \rightarrow 2\text{H}_2\text{O} + 1/2\text{S}_2$
Oxide ion-conducting	$\text{H}_2\text{S} - 2\text{e}^- \rightarrow 2\text{H}^+$ $\text{H}_2\text{S} + 2\text{O}^{2-} \rightarrow 2\text{H}^+ + \text{SO}$	oxide ions transfer from cathode side to anode side	$\text{O}_2 + 4\text{e}^- \rightarrow 2\text{O}^{2-}$	$2\text{H}_2 + \text{O}_2 \rightarrow 2\text{H}_2\text{O}$ $2\text{H}_2\text{S} + 3\text{O}_2 \rightarrow 2\text{H}_2\text{O} + 2\text{SO}_2$

The focus of this research on H₂S fuel cells is therefore on the proton-conducting fuel cell, particularly on the preparation of composite Li₂SO₄ proton-conducting electrolyte as well as the development of a preparation method for membrane-electrode-assemble (MEA), considering all possible parameters such as size and formulation of electrolyte material, heat-treatment sintering temperature and duration of electrolyte. Optimal fabrication parameters can be obtained by conducting a parametric study that provides some insights for the design and preparation of MEA.

1 Experiments

1.1 Preparation of Nano-Composite Target Materials for Electrolytes

To prepare an electrolyte with a designed composition, 98% (mass fraction, below the same) aluminum nitrate [Al(NO₃)₃(9H₂O)] (produced by Aldrich Inc.) and 99.7% lithium sulfate anhydrous (Li₂SO₄) (produced by Alfa Aesar Inc.) were used as starting materials of electrolytes. A nano-composite Li₂SO₄-Al₂O₃

component was prepared using a sol-gel technique developed by the authors to prepare the target materials. The technique comprised a sequential steps of gel-solution preparation using lithium sulfate salt and an aluminum nitrate precursor, followed by sol formation, drying, calcination at 600 ~ 700 °C to remove NO_x so as to obtain Li₂SO₄-Al₂O₃ component, and grinding the solid materials so as to obtain required particle sizes in a ball-grinding machine.

1.2 Preparation of MEA

To prepare a MEA, nano-composite Li₂SO₄-Al₂O₃ powder with appropriate amount was weighted and mixed with water. Anode catalyst, namely a Mo-Ni-S-based composite anode prepared from 80% MoS₂ + NiS (1:1 in weight ratio), 5% Ag powder, 5% starch and 10% electrolyte, was first put onto a 2.54 cm cylindrical die, followed by electrolyte materials (nano-composite Li₂SO₄-Al₂O₃ powder) containing suitable moisture content. Finally, cathode catalyst, namely a NiO-based composite cathode prepared from 80% NiO, 5% Ag powder, 5% starch and 10% electrolyte, was put onto the electrolyte materials. The composite MEA sample disks were pressed at the pressures of 150 ~ 600 MPa that vary with the moisture content of electrolyte materials. The thickness of MEA is between 0.8 and 1.5 mm. The MEA so obtained was heated in nitrogen atmosphere in an oven, first at 105 °C for 60 ~ 90 min and then at 800 ~ 850 °C for 360 min. The weight of MEA was approximately 8% ~ 10% less than the initial one of the products due to the emission of water. The MEA was finally cooled to room temperature slowly under nitrogen.

1.3 Installation of MEA into Fuel Cell Test Station

A fuel cell test station similar to that shown in References [6-7,9-10] was constructed, with some minor modifications that did not alter the principles of operation. To install the MEA (2.54 cm in diameter) into a test fuel cell, the cathode side of the assembly was first attached to a supporting annular alumina disk with a diameter of 3.2 cm and a thickness of 0.3 cm using ceramic sealant (α-terpineol, from Aremco Product Inc., USA). An opening (1.1 cm in diameter) in the center of the supporting ceramic disk allowed air to access the cathode. The combination so made was

then sealed with ceramic sealant between two alumina tubular chambers with an outer dimension of 2.54 cm and a length of 40 cm, as described in Reference[9]. An additional sealant layer (3 mm in width) was applied around the sealing area to ensure a good seal. Platinum mesh was used as current collectors. The mesh surface was refreshed in the flame of a gas lamp prior to installation for each experiment^[5]. The cell was then heated in a tubular furnace (Thermolyne F7930), with nitrogen passing through the anode chamber and air through the cathode chamber. To cure the sealant, the furnace temperature was increased at 0.8 °C/min till up to 230 °C, and was held at 230 °C for 1 h. Then, the temperature was increased to a selected testing temperature, typically 600 °C, and was held at that temperature for the duration of each set of tests.

1.4 Characteristics of MEA

The morphologies and microstructures of electrolyte materials and MEA were determined with scanning electron microscope (SEM) technique using a Hitachi S-2700 Scanning Electron Microscope. MEA was installed in the fuel cell test station, with Pt contacting lead attached to the positive and negative electrodes of MEA. The resistance of each membrane or MEA was measured at the selected temperature (500 ~ 700 °C). The temperature was adjusted (either increased or decreased) at 5 °C/min to the minimized thermal shock. After the selected temperature had been maintained for 30 min, resistance data were acquired using the conventional electrochemical impedance spectrum (EIS) methodology, and then the electrical conductivity of membrane or MEA, 0.001 ~ 0.155 S/cm at 500 ~ 700 °C, could be calculated under the operating conditions.

1.5 Measurement of Fuel Cell Performance

Cell performance was determined, using pure H₂S as the anode feed and air as the cathode feed. The cell open circuit voltage (OCV) was monitored as a function of time on stream. Data were recorded with a Gamry electrochemical measurement system (PC4-750). Initial electrical performance data were evaluated to determine cell integrity. Typically, a cell that had no leaks showed a steady OCV value after about 30 min. An OCV that fluctuated, or that had a persistently low value (0.5 ~ 0.6 V), indicated possible

leaks in the cell, and no further tests were conducted using that MEA. After a steady OCV was achieved, EIS measurements were performed to determine the cell resistance and the conductivity. The frequency region was 0.2 ~ 100 000 Hz and a stimulated alternative current (AC) signal of 5 mV was imposed onto OCV. Potentiodynamic measurements were carried out to determine the cell current-voltage and current-power performance in their compensation mode using the Gamry system at a scanning rate of 1 mV/s^[8].

2 Results and Discussion

2.1 Preparative Method of Electrolytes and MEA

Some proton-conducting electrolytes had been prepared based on Li₂SO₄. However, it was found to be difficult to obtain an integral wafer or MEA by pressing Li₂SO₄ alone since these electrolytes or MEA were quite powdery after being heated in air. Particles loosely adhered to each other and were easily abraded from the electrolyte or MEA. Preparative methods of proton-conducting electrolytes with improved mechanical and electrical properties had been developed using a mixture of Li₂SO₄ and Al₂O₃. However, it was still difficult to prepare integral and compactive electrolytes and MEA by pressing Li₂SO₄ and Al₂O₃ alone. Now it have been found by the authors that the addition of water in the mixture powder greatly enhances the adherence of electrolyte wafer or MEA when it is heated in air. A key factor seems to be the moisture content of the mixture powder that can be lost on heating the wafer or MEA. Several levels of water contents were used during the molding of powder mixtures: wet mixture (excess water wrings while pressing); semi-dry mixture, with the water content no greater than the one that the powder can absorb from humidified air; and dry mixture, using mixtures dried to constant weight at 105 °C. The suitable moisture content in this study is about 8% ~ 10% (semidry mixture), but the reason for this amount is still unknown.

Electrolytes containing different ratios of Li₂SO₄ and Al₂O₃ obtained after a single cycle of mixing, grinding, pressing and heating have similar electrolyte morphology. They are all fragile and very porous. Powder can easily be detached from each electrolyte. Opti-

cal microscopic inspection indicates loose adherence between particles, and many cavities are found on the electrolyte surface. The electrolytes are sufficiently porous to allow H_2S cross-over. The low-density electrolytes so made are not structurally sound. It is found that a procedure comprising mixing, drying and then a repeated sequence of grinding, pressing and heating (3 ~ 5 times) afford a more dense electrolyte. Similar procedures with two cycles of the sequence have been described elsewhere^[3,5].

Li_2SO_4 content of the composite electrolyte is a critical factor to influence the properties. Li_2SO_4 is required for ion conduction. Electrolytes made of pure Li_2SO_4 are fragile and are very difficult to obtain an integral wafer by pressing. Thus, alumina is required to confer the strength to the composite. However, there is a decrease in conductivity when Li_2SO_4 content in the Li_2SO_4 - Al_2O_3 composite is less than 40%. This observation is consistent with EM theory^[10]. The structure of MEA deteriorates and its shape becomes deformed when Li_2SO_4 content is as high as 80%, for all preparative conditions. Consequently, for the present study, Li_2SO_4 content is between 55% and 75% in all formulations. The experimental data including electrolyte density and homogeneity as well as compactivity, MEA integral and fuel performance reveal that the optimum content of Li_2SO_4 is about 65% in this study.

Heat treatment parameters affect the mechanical strength of MEA, the structure formation, the morphology and phase composition of Li_2SO_4 - Al_2O_3 . Lithium sulfate and alumina do not react chemically at the sintering temperature less than 1200 °C^[11]. The quality of the interphase contact improves when the nano-composite Li_2SO_4 component is used. The phase transformation and thermal stress during the heat treatment below the melting point of Li_2SO_4 lead to the appearance of micro pores as well as poor compactivity, and these defects are mainly in the contact zone in the bulk matrix of nano-composites that has agglomerated. When the heat-treatment temperature is at least as high as the melting point of Li_2SO_4 , there are remarkable changes of the composite structure and the morphology. A melted phase causes the physical interaction between the salt and alumina particles, and the agglomerates of tightly bonded alumina grains are observed. However,

shrinkage cracks in the bulk matrix are formed in several cases, depending on the sintering conditions and the ratio of Li_2SO_4 to Al_2O_3 . SEM photographs show the results with an electrolyte composition of 65% Li_2SO_4 + 35% Al_2O_3 at various sintering temperatures. At 850 °C (above the melting point of Li_2SO_4 , 845 °C), the sample looks more dense and compact comparing with that at 700 °C, as shown in Fig. 1(a) and Fig. 1(b). Although the microstructures of nano-composite samples sintered at further elevated temperature (up to 900 °C) are more homogeneous and dense, electrolytes might be easily cracked, as shown in Fig. 1(c). The sintering duration also influences the electrolyte performance. Longer duration can possibly increase the



(a) 700 °C



(b) 850 °C



(c) 900 °C

Fig.1 SEM images of electrolyte at different sintered temperature

compactivity of membrane, but can also cause some minor cracks leading to crossover. Therefore, the suitable sintering temperature and duration in this study are 800 ~ 850 °C and 6 ~ 8 h, respectively.

2.2 Electrical Conductivity of Electrolyte or MEA

EIS technique was used to study the change of the electrical conductivity of electrolytes having different thicknesses with temperature. Fig. 2 shows the conductivity as a function of temperature for the electrolyte composition of 65% Li₂SO₄ + 35% Al₂O₃ with the thickness of 0.8 ~ 1.5 mm. A decrease in electrical conductivity occurs as a result of the increase in electrolyte thickness. The conductivity of the electrolyte with 1.5 mm in thickness is about 0.08 S/cm at the temperature above 600 °C, which is only about half of that of the one with 0.8 mm in thickness at the same temperature. An abrupt change in conductivity occurs at about 575 °C, the temperature of which is the same for each electrolyte with different thicknesses. Conductivity slightly increases with the temperature above 575 °C. Below 575 °C, it dramatically decreases. This indicates that 575 °C is the lowest operating temperature appropriate for the present Li₂SO₄-based proton-conducting electrolytes. This phenomenon is attributed to the Li₂SO₄ phase transformation known to occur at 577 °C^[3]. Li₂SO₄ is in the cubic α -phase above 577 °C, which has high proton conductivity, and in the monoclinic β -phase below 577 °C, which has low proton conductivity. It can also be seen from Fig. 2 that the conductivity keeps almost the same when the operating temperature is between 600 and 700 °C, which is the suitable operating temperature range for intermediate-temperature fuel cells.

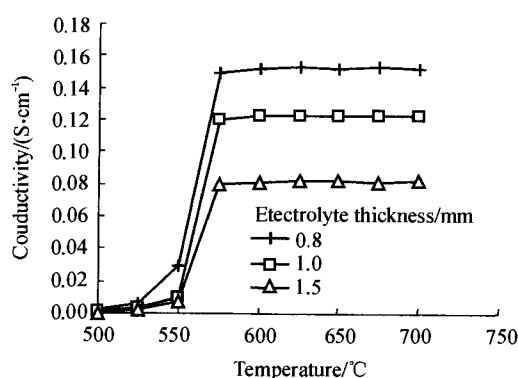


Fig. 2 Change of conductivity with temperature
The weight ratio of Li₂SO₄ to Al₂O₃ is 65:35

2.3 Electrochemical Behavior of Fuel Cell

2.3.1 Comparison of Anode Catalysts

Both Pt and the composite material derived from NiS/MoS₂ + Ag had been used as the starting materials for anodes in oxide ion-conducting (YSZ) H₂S fuel cells^[7-8]. To compare the performances of H₂S fuel cells using proton-conducting electrolytes (1.0 mm in thickness) with the composition of 75% Li₂SO₄ + 25% Al₂O₃, membranes were prepared from the wafers prepared by doubly grinding, pressing and heating the material, and then MEA was pressed with electrodes once. Fig. 3 compares the performances of the fuel cells with Pt and metal sulfide-based composite anodes, and composite NiO cathodes at 650 °C. Both cells exhibit similar performance with the OCV of about 1.1 V and the maximum current density of about 160 mA/cm². In the low current region, the metal sulfide-based composite anodes show a higher current output, as compared with that of Pt anode at the same voltage, which indicates that the metal sulfide-based composite anodes have a great catalytic activity. In addition, the metal sulfide-based composite anode is more stable than Pt over time on H₂S stream. Pt reversibly forms PtS during the cell operation due to the oxidation reaction of Pt and H₂S on anode catalyst, leading to its delamination from electrolyte. The metal sulfide-based composite anodes do not detach and they indeed show the potential of MoS₂/NiS as active anode catalysts. Another advantage of metal sulfide-based composite anode is that it is possible, by adjusting the chemical composition, to tailor its activity and stability to the conditions of the system and its thermal compatibility with the electrolyte^[6-8].

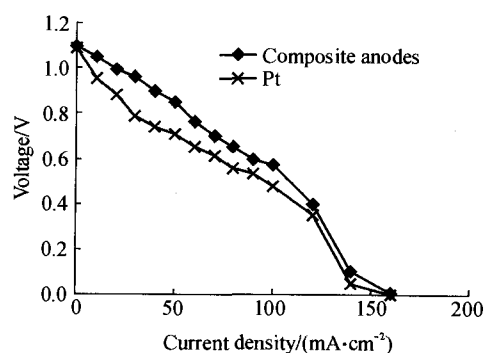


Fig. 3 Comparison of current-voltage relationships obtained for composite anodes and Pt at 650 °C

The weight ratio of Li₂SO₄ to Al₂O₃ is 75:25; electrolyte thickness 1.0 mm

2.3.2 Fuel Cell Performance

Figure 4 demonstrates the current-voltage characteristics for a cell configuration of H_2S , ($\text{MoS}_2/\text{NiS} + \text{Ag} + \text{electrolyte} + \text{starch}$)/ $\text{Li}_2\text{SO}_4 + \text{Al}_2\text{O}_3$ /($\text{NiO} + \text{Ag} + \text{electrolyte} + \text{starch}$), air with a Li_2SO_4 to Al_2O_3 ratio of 65:35 and MEA with a thickness of 0.8 mm at 600, 650 and 680 $^\circ\text{C}$, respectively. It is found that the OCVs are respectively 1, 1.13 and 1.19 V at 600, 650 and 680 $^\circ\text{C}$, and that the maximum current densities are 130, 170 and 320 mA/cm^2 at 600, 650 and 680 $^\circ\text{C}$, respectively. It indicates that the increase of temperature can significantly improve the cell performance. This is due to the characteristics of mass-transport limitation in electrochemical reaction^[8]. The effect is greatly reduced when the temperature increases to 680 $^\circ\text{C}$. At 680 $^\circ\text{C}$, the rate of H_2S desorption and transport of sulfur from the vicinity of the active sites increases, which thereby enhances the electrochemical reaction rate. Fig. 5 shows the current and power density curves having the same cell configuration as that in Fig. 4 at different temperatures. It is observed from the data in Fig. 5 that the maximum power densities are 35, 46 and 130 mW/cm^2 at 600, 650 and 680 $^\circ\text{C}$, respectively, and that the corresponding current densities are respectively 60, 76 and 200 mA/cm^2 . The MEA at 680 $^\circ\text{C}$ has superior performance considering the open circuit voltage, the current density and the power density characteristics. The results achieved so far have suggested that the MEA fabricated from Mo-Ni-S-based composite anode, nano-composite Li_2SO_4 proton-conducting electrolyte as well as NiO-based composite cathode is successful for developing H_2S solid oxide fuel cells to function at intermediate temperatures below 700 $^\circ\text{C}$.

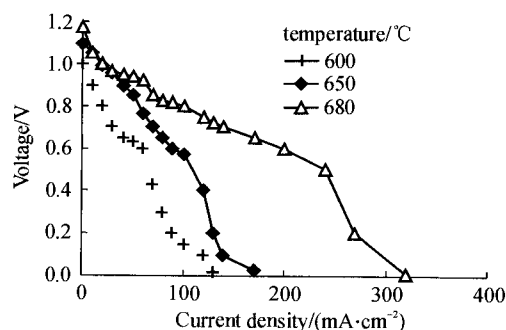


Fig. 4 Fuel cell performances at different temperatures
The weight ratio of Li_2SO_4 to Al_2O_3 is 65:35, electrolyte thickness 0.8 mm

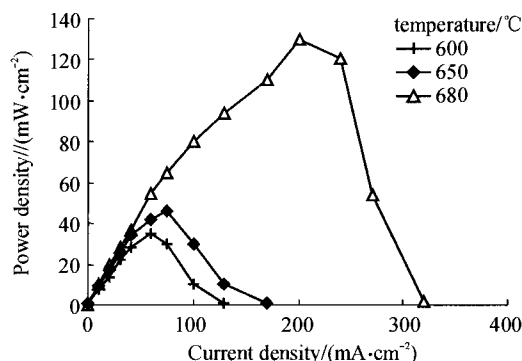


Fig. 5 Fuel cell performance at different temperatures
The weight ratio of Li_2SO_4 to Al_2O_3 is 65:35, electrolyte thickness 0.8 mm

3 Conclusions

MEA comprising a Mo-Ni-S composite anode, nano-composite Li_2SO_4 proton-conducting electrolytes and a NiO-based composite cathode has high integrity and strength as well as superior performance in H_2S stream. The effects of preparative parameters such as the moisture content, the molding pressure, the compositions of formulation and the heat treatment on membranes were studied in this paper, thus determining the suitable fabricated conditions. Electrochemical current-voltage and current-power density measurements were carried out to exhibit the performance of cell. Experimental results show that MEA is stable during the operation of H_2S fuel cells, that the metal sulfide-based composite anodes have similar activity to Pt anode, the former is more stable than the latter during the cell operation, and that the nano-composite Li_2SO_4 proton-conducting electrolyte is a potential candidate of membrane for H_2S fuel cells.

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纳米复合Li₂SO₄质子传导膜 H₂S 中温燃料电池*

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摘要: 开发了一种制备纳米复合 Li₂SO₄ 质子传导电解质和膜电极组装 (MEA) 的工艺. 与传统的丝网涂布工艺不同, 新的制备工艺是将阳极、阴极催化剂与纳米复合电解质同时一次压制成 MEA. 这就使得 MEA 的设计具有某些结构上的特点, 由于膜厚减少和电极与电解质之间的接触良好, 可以降低电解质与电极之间的欧姆电阻, 提高其机械和导电性能, 增加膜的质子传导性以及改善电池的性能. 用电子扫描电镜 (SEM) 和电化学阻抗分析技术对电解质薄膜进行了表征, 结果表明, 纳米复合材料改善了 MEA 的总体性能. 由于膜的致密性和不透性, 不会发生气体穿透过膜的现象. MEA 在 H₂S 环境中很稳定. 电池结构为 H₂S, (MoS₂/NiS + Ag + 电解质量 + 淀粉)/Li₂SO₄ + Al₂O₃/(NiO + Ag + 电解质量 + 淀粉), 空气、MEA 厚为 0.8 mm、电解质组成为 65% Li₂SO₄ + 35% Al₂O₃ 的单电池在 680 °C 时产生最大功率密度为 130 mW/cm², 相应的电流密度为 200 mW/cm².

关键词: 固体氧化物燃料电池; 膜电极组装 (MEA); 硫化氢; 质子传导膜

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