Electrode design for direct-methane micro-tubular solid oxide fuel cell (MT-SOFC)

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HIGHLIGHTS
- A hierarchical structured YSZ micro-tube is fabricated.
- It is composed of self-organised micro-channels and a thin electrolyte layer.
- The self-organised micro-channels are useful for incorporation of anode components.
- Micro-channels contribute to a lower transport resistance.
- Excellent stability during long-term test with negligible coking is observed.

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ABSTRACT
Herein, a micro-structured electrode design has been developed via a modified phase-inversion method. A thin electrolyte integrated with a highly porous anode scaffold has been fabricated in a single-step process and developed into a complete fuel cell for direct methane (CH4) utilisation. A continuous and well-dispersed layer of copper-ceria (Cu-CeO2) was incorporated inside the micro-channels of the anode scaffold. A complete cell was investigated for direct CH4 utilisation. The well-organised micro-channels and nano-structured Cu-CeO2 anode contributed to an increase in electrochemical reaction sites that promoted charge-transfer as well as facilitating gaseous fuel distribution, resulting in outstanding performances. Excellent electrochemical performances have been achieved in both hydrogen (H2) and CH4 operation. The power density of 0.16 Wcm⁻² at 750 °C with dry CH4 as fuel is one of the highest ever reported values for similar anode materials.

1. Introduction
Solid oxide fuel cells (SOFCs) are types of fuel cells generally known for their high operating temperatures ranging from 600 to 1000 °C [1–3]. In addition to the typical attractive features of fuel cell technology, such as high efficiency and environmentally friendly operation, SOFCs provide some unique features, such as full solid-state operation and flexibility in fuel sources [4,5]. Hydrogen (H2) is the most commonly used fuel for SOFCs, with excellent electrochemical performances frequently reported [6,7]. However, the vast majority of H2 is produced from hydrocarbons through the steam reforming process, during which 20–30% of fuel value is lost [8]. As a result of this, the concept of direct hydrocarbon utilisation in SOFC has attracted research interest worldwide [9–12]. By using hydrocarbons, alternative cell materials need to be utilised in order to suppress carbon formation, which is an inherent problem associated with conventional nickel (Ni)-cermet anodes [13,14]. In a qualitative index of various SOFC anode materials by Ge et al., Ni-based anodes show superior electrocatalytic activity over Cu-based anodes, however the latter has the advantage of better fuel flexibility and a lower tendency towards coke formation [2]. Gorte and co-workers reported the development of planar SOFCs with Cu-based anodes operated with several types of hydrocarbons in which...
good cell performances with no or trivial coke formation have been demonstrated [8,14–16].

Fabrication is a primary challenge associated with the copper-based anodes. Copper and its oxides have relatively low melting points, viz. 1085 °C, 1326 °C and 1232 °C for Cu, CuO, and Cu2O, respectively [17,18]. This makes the preparation of a cell with Cu-based anode through standard high-temperature ceramic processing techniques (i.e. tape casting and sintering) impracticable. Instead, impregnation has been almost exclusively applied to incorporate Cu in an additional step [14,16,19]. Geometrically, a micro-tubular design offers additional benefits compared to flat and tubular designs, including improved thermal shock resistance, quick start-up and shut-down, and enhanced volumetric power density [20,21].

Anode design is a critical aspect of the direct utilisation of hydrocarbons in SOFC operation. In this study, a micro-structured yttria-stabilised zirconia (YSZ) micro-tube has been fabricated via a phase inversion-assisted process to develop micro-tubular (MT)-SOFC with a Cu-based anode which is operated using CH4 as a fuel source. One major benefit of the phase inversion-assisted process is the flexibility in control and tailoring of the micro-structure. The microstructured YSZ scaffold is composed of self-organised micro-channels and a skin layer. The thin skin layer functions as the electrolyte, whereas the micro-channels perform as a substrate for the anode materials (Cu-CeO2). These anodic materials are firmly positioned into the micro-channels, thus, deterring their thermal movement. Furthermore, this unique micro-tubular feature helps avoid delamination and defect formation, contributing towards improved structural integrity. After a complete cell was constructed, a performance test was conducted with direct CH4 utilisation.

2. Experimental

2.1. Materials

YSZ and lanthanum strontium manganite (LSM) powders were obtained from Inframat Advance Materials (USA). Polyethersulphone (PESf) (Radel A300, Mw = 42500 g mol−1, Ameco Performance, USA), N-Methyl-2-pyrrolidone (NMP) (Mw = 99.13 g mol−1 from VWR, UK), and Aralacel P135 (polyethyleneglycol 30-dipolyhydroxystearate, Uniquema) were applied as binder, solvent, and dispersant, respectively. NMP was also applied as bore fluid (internal coagulant) after mixing with ethanol (VWR, UK, NMP/Ethanol = 60/40 in weight). Denionised water was adopted as external coagulant. Anode components: 99% copper (III) nitrate trihydrate (Cu(NO3)2·3H2O) and 99.5% cerium nitrate hexahydrate (Ce(NO3)3·6H2O) were purchased from Fisher Scientific (UK). Ethylene glycol (99 + %) from Acros Organic (UK) was used as cathode ink vehicle.

2.2. Preparation of micro-structured YSZ micro-tubes

The ceramic suspension was prepared by mixing the YSZ powder with the solvent (NMP) and dispersant. The resultant mixture was milled for 60 h (MTI Corporation model SFM-1 Desktop Planetary Ball Miller). PESf was subsequently added to the suspensions and mixed for another 60 h to attain a homogeneous mixture. Before spinning, the suspension was degassed by stirring under vacuum for 2 h to eliminate air bubbles. The suspension was transferred to a stainless steel syringe and extruded through a custom-designed spinneret into an external coagulation bath of DI water. The spinneret was immersed in the coagulant bath to eliminate any air gap. The extrusion rates of the suspension and the bore fluid were precisely controlled using two Harvard PHD 22/2000 Hpsi syringe pumps. The micro-tube precursors were left in water overnight to complete the phase inversion before sintering. Table 1 presents the fabrication parameters for the preparation of YSZ micro-tube.

2.3. Preparation of a complete fuel cell

Fig. 1 shows the schematic diagram of the phase-inversion based extrusion process. The inlet (Fig. 1a) shows the picture of the dual-orifice spinneret used in this work. Fig. 1b illustrates the procedure for preparing a complete single cell. After completion of the YSZ micro-tubes (Fig. 1b(i)), a dual-layer cathode consisting of an inner LSM-YSZ layer (LSM/YSZ = 50/50 by weight) and an outer LSM layer was brush-painted onto the YSZ micro-tube and sintered at 1000 °C for 2 h (Fig. 1b (ii)). Subsequently, the anode materials (Cu-CeO2), were incorporated into micro-channels of YSZ micro-tubes via vacuum-assisted co-impregnation process. A mixed aqueous solution of copper nitrate and cerium nitrate was prepared prior to impregnation, with a concentration of 4 M and 1 M, respectively. The set-up for the impregnation process is shown in Fig S1 with additional description of the impregnation process. The co-impregnated micro-tube underwent heat treatment at 450 °C for an hour to decompose the nitrates. This impregnation was repeated until a target loading of 25 wt % was achieved.

2.4. Reactor assembly and sealing

The MT-SOFC was placed into two alumina supporting tubes (Almahn Crucibles Ltd., UK) and sealed with a ceramic sealant (Ceramabond 552-VFG, Aremco, USA). The sealant became gas-tight following successive heat treatments at 95 °C and 260 °C for 2 h at each temperature with a heating rate of 5 °C min−1. This assembly was placed inside a 300 mm long quartz tube with 20 mm outer diameter (OD) (Almahn Crucibles Ltd., UK) with stainless steel end-caps made in-house, and sealed to the quartz tube by Viton O-rings (Polymax, UK). The whole set up is shown in Fig. S2.

2.5. Characterisation

The morphology of the micro-tube was examined using scanning electron microscopy (SEM) (JEOL JSM-5610 and LEO Gemini 1525 FESEM). Samples were gold-coated under vacuum at 20 mA for 60 s (EMITECH Model K550), and the SEM images with various magnifications were acquired. Energy dispersive spectrometry (EDS, JEOL JSM-6400 electron microscope) analysis was undertaken to evaluate the elemental distribution of anodic materials. The gas-tightness of the sintered micro-tubes was assessed using N2 permeation method. The N2 permeance was calculated from the pressure drop over 8 h using the following equation:

$$P = \frac{V}{RT\times A_{m}t}\ln\left(\frac{P_{0} - P_{t}}{P_{0}}\right)$$

(1)

where

$$A_{m} = \frac{[2\pi(R_{0} - R_{m})L]}{\ln(R_{0}/R_{m})}$$

(2)

where P denotes the N2 permeance of the tested membrane (mol m⁻² s⁻¹ Pa⁻¹); V is the volume of the test vessel (m³); R is the gas
Fig. 1. Schematic diagram of the phase inversion process; (a) inset shows the dual-orifice spinneret, (b) steps involved in the overall fabrication of cell components of MT-SOFC.

Fig. 2. SEM images of the sintered YSZ micro-tube: (a) overall cross-section, (b) magnified cross-section, (c) and (d) cross-section and magnified cross-section showing the thickness of the skin-layer region, respectively, (e) and (f) inner and outer surface, respectively.
constant (8.314 J mol⁻¹ K⁻¹); T is the measured temperature (K); and \( p_0, p_a, \) and \( p_i \) indicate the initial, atmospheric and final pressure readings (Pa), respectively. \( \Delta m \) is the membrane area (m²); \( t \) is the measurement time (s); \( L \) is the length of the micro-tube; and \( R_o \) and \( R_i \) represent the outer and inner radiusses of the tube, respectively. The mechanical strength was examined through a three-point bending method using a tensile tester (Instron Model 5544) with a load cell of 1 kN. The specimen was placed onto two sample holders with a gap of 30 mm. The bending strength \( (\sigma_f) \) was calculated from the obtained fracture force using the following equation:

\[
\sigma_f = \frac{F}{BL^2} 
\]

where \( F \) represents the measured fracture force (N); \( B \), \( L \), \( D_o \) and \( D_i \) denote the mass, length, outer and inner diameters of the micro-tube (m), respectively.

The average porosity of the micro-tube was studied using pycnometer (Micromeritics Accupyc II 1340). The porosity \( (\varepsilon_v) \) was calculated using the following equations:

\[
\varepsilon_v = \frac{p_{p} - p_{microtube}}{p_{pyc}} 
\]

\[
p_{microtube} = \frac{4m_{sample}}{\pi(D_o^4 - D_i^4)_{sample}} 
\]

Where \( p_{pyc} \) represents the skeleton density (g cm⁻³) measured by pycnometer, \( m \), \( L \), \( D_o \) and \( D_i \) denote the mass, length, outer and inner diameters of the sample (cm), respectively. In addition, an assumption has been made whereby each sample has a uniform structure with identical dimension all through the micro-tube.

For a single cell, the current was collected from the cathode by wrapping silver wires on cathode surface with additional silver paste to improve the contact. For current collection at the anode, silver wires were passed through the lumen, with additional silver wool and silver paste to enhance contact between the anode surface and silver wires. Both wires from anode and cathode were connected to a potentiostat/galvanostat (Iviumstat, Netherlands) for current-voltage (I-V) measurement. The cell current-voltage testing was conducted across the cell [28, 29], indicating that our micro-tube is suitable for the fabrication of the MT-SOFC.

### 3.3. Vacuum-assisted co-impregnation process

The incorporation of the anode material was conducted through the vacuum-assisted co-impregnation process. A number of impregnation cycles are required to ensure sufficient anode materials to form a continuous phase. Fig. S 4 shows the relationship between the anode loading and the impregnation cycles. The anode loading is 28 wt% of the full cell after 15 cycles of impregnation (65:35 wt% for CuO:CeO₂). The loading of anode materials increased almost linearly with the number of cycles, suggesting appropriate controllability of this impregnation process.

The porosity of the YSZ scaffold was measured to be 43%, and the porosity of the micro-tube after 15 cycles of the co-impregnation process was 36%. The porosity decreases after the impregnation as the anode materials occupied the void of the YSZ scaffold. Both values for the porosity are within the required optimal anode porosity range, between 30% and 40% [30].

It has been previously reported that CuO could agglomerate and thus form isolated, spherical clusters [31]. This phenomenon can be linked to the low thermal stability of Cu as Cu and its oxides have considerably low melting temperatures. Thus, the co-impregnation approach has been adopted and no metallic phase agglomeration or blockage of the channel entrance was observed, as depicted in Fig. 3b. In comparison to Cu and its oxides, CeO₂ has better thermal stability due to its higher melting temperature of 2602 °C [2]. Therefore, the presence of CeO₂ may assist in scattering Cu throughout the entire area, as well as improving the thermal stability of the anode composite.

It is noteworthy that one of the main advantages of using a composite Cu-CeO₂ anode is that both materials do not form a solid solution, with each species potentially retaining their specific functionality. In this study, Cu is required for electronic conductivity whereas CeO₂...
functions primarily as a catalyst for fuel oxidation. One critical property of an anode is a continuous electronic conducting phase to ensure smooth electron transport, which lowers ohmic losses. SEM image (Fig. 3b) shows the homogeneous distribution of both particles onto the inner surface of the micro-tube, which could also be shown by elemental mapping. Both materials are uniformly dispersed over the entire region of the inner surface and into the micro-channels (Fig. 3a and b). The vacuum-assisted co-impregnation process applied in this work has demonstrated good Cu continuity, which is crucial for fuel cell performance.

Fig. 4 shows the chemical mapping of the co-impregnated micro-tube. As can be seen, both materials are uniformly dispersed over the entire region of the inner surface and into the micro-channels. In addition, the higher resolution of the SEM images (Fig. 3d) of the impregnated micro-tubes depict the flake structure of the Cu-CeO₂. The thickness of the anode catalyst layer is approximately 5 μm. This particular structure could lead to an increase in surface area, which contributes to a larger TPB at which the electrochemical reaction takes place.

3.4. Electrochemical performance test

Electrochemical performance tests were conducted at 650, 700, and 750 °C using a ramping of 5 °C min⁻¹ with 30 ml min⁻¹ of dry H₂ or CH₄ as fuel, and 50 ml min⁻¹ of air as the oxidant. The co-impregnated cell (15 cycles) was used to conduct this study. Three cells were tested with a standard deviation less than 10%. The estimated active area was calculated to be approximately 0.45 cm². The open circuit voltage (OCV) can reach up to 1.19 V for H₂ at 650 and 700 °C and 0.95 V for CH₄, respectively, indicating a proper gas-tightness of the YSZ electrolyte. Note that the OCV for a cell operating under CH₄ is slightly lower than the predicted value determined from Nernst potential equation (~1.04 V) [32]. This discrepancy for the CH₄ system could be due to the presence non-electrochemical surface reactions [33]. Nevertheless, both OCV values for systems operated with either H₂ or CH₄ as reported here agreed well with other studies [8,18,34–36].

Fig. 5a shows that a reasonably high power density of up to 0.55 W cm⁻² was obtained from H₂ fuel at 750 °C. This cell shows a substantial improvement over other SOFCs with similar Cu-CeO₂-YSZ composite anodes [18,37], which may be attributed to the unique hierarchical structure of the micro-tubes. The presence of micro-
channels with open entrances should help facilitate gas transport to the reaction sites. The measured peak power density is comparable to previously reported cells with Ni-based anodes [27], suggesting that the Cu-CeO$_2$ composite is a promising replacement of Ni-cermet [3,38,39].

In comparison, the CH$_4$-fuelled system demonstrated lower power density than that of H$_2$ (Fig. 5b). This finding could be related to the catalytic limitation, where it is believed that electrochemical oxidation of CH$_4$ is slower than H$_2$. Earlier work has shown that CH$_4$ is less reactive than H$_2$ in heterogeneous oxidation [40] and has lower reactivity at the anode [16]. Another reason for lower performance in the CH$_4$ system could be linked to the rate-limiting electrochemical steps at the anode. Direct oxidation of CH$_4$ in one step seems to be difficult [41], and it has been proposed that several parallel reactions occur when using hydrocarbons as the fuel [42]. This means that CH$_4$ can either be partially oxidized by the oxide ions to carbon monoxide (CO) and hydrogen (H$_2$) (Equation (6)) or fully oxidised to carbon dioxide (CO$_2$) and water (Equation (7)). In addition, there is a possibility of gradual internal steam and dry reforming (Equations (8) and (9), respectively) taking place within the system.

$$\text{CH}_4 + \text{O}^{2-} \rightarrow \text{CO} + 2\text{H}_2 + 2\text{e}^- \quad (6)$$

$$\text{CH}_4 + 4\text{O}^{2-} \rightarrow \text{CO}_2 + 2\text{H}_2 + 8\text{e}^- \quad (7)$$

$$\text{CH}_4 + \text{H}_2 \rightarrow 3\text{H}_2 + \text{CO} \quad (8)$$

$$\text{CH}_4 + \text{CO}_2 \rightarrow 2\text{H}_2 + 2\text{CO} \quad (9)$$

$$2\text{CO} \rightleftharpoons \text{CO}_2 + \text{C} \quad (10)$$

$$\text{CH}_4 \rightarrow \text{C} + 2\text{H}_2 \quad (11)$$

However, a system fuelled by methane is also susceptible to carbon formation, according to Equations (10) and (11).

Equations (6)–(9) show that there are several reacting species at the anode (CH$_4$, H$_2$ and CO), contributing to the more complex anodic reactions as compared to the H$_2$-fuelled system. Furthermore, issues surrounding the transport limitations could also be related to the larger molecular mass of CH$_4$, which causes slower diffusion and may result in greater concentration polarisation. This could be minimise by having internal reforming. The addition of reforming agents would reform the CH$_4$ into H$_2$ and CO which are relatively easy to undergo electrochemical reaction. However, in this study, we aim to operate the system directly with dry CH$_4$, thus no additional reforming agents were added.

The difference in the electrochemical performance between these fuels is reduced when the cell is operated at higher temperatures. The MT-SOFCs fuelled by CH$_4$ exhibited peak power densities of approximately 0.05, 0.09 and 0.16 W cm$^{-2}$ at 650, 700, and 750 °C, respectively.
respectively. Operation using dry CH$_4$ at 750 °C demonstrated a power density of 0.16 W cm$^{-2}$ which, to the best of our knowledge, is the highest compared to previously reported values for similar anode materials (Cu-CeO$_2$), as shown in Table 2.

Table 2
SOFCs with Cu-cermet anodes fed with methane as fuel.

| Cell configuration                        | Fuel     | Temp. (°C) | P$_{\text{max}}$ (W cm$^{-2}$) | Ref. |
|------------------------------------------|----------|------------|---------------------------------|------|
| Cu-CeO$_2$-LDC/LSGM/SCF                 | Dry CH$_4$ | 800        | 0.07                            | [19] |
| Cu-CeO$_2$-LDC/LSGM/SCF                 | Dry CH$_4$ | 800        | 0.11                            | [35] |
| Cu-CeO$_2$-YSZ/YSZ/LSM-YSZ              | Dry CH$_4$ | 800        | 0.07                            | [36] |
| Cu-CoFe$_2$YSZ/YSZ/YSZ/LSM-YSZ          | Dry CH$_4$ | 800        | 0.09                            | [36] |
| 1-wt% Pd-Cu-Fe-ceria-YSZ/YSZ/LSM-YSZ    | Dry CH$_4$ | 800        | 0.12                            | [36] |
| Cu-Co-CeO$_2$/YSZ (0.23 μm)/LSM          | CH$_4$:H$_2$ | 750        | 0.09                            | [43] |
| Cu-CeO$_2$/YSZ(180 μm)/LSM-YSZ          | Dry CH$_4$ | 800        | 0.11                            | [44] |
| Cu-CeO$_2$/YSZ(10 μm)/LSM-YSZ           | Dry CH$_4$ | 750        | 0.16 This work                 |      |

Fig. 6 shows the impedance analysis for the cell with a signal amplitude of 10 mV at the frequency range from $10^2$ to 0.01 Hz. The high-frequency intercept on the x-axis indicates the ohmic resistance of the cell ($R_{\Omega}$), comprising the ionic resistance in the electrolyte, both ionic and electronic resistance in the electrodes, and the contact resistance at the interface and current collectors. The first semi-circle (high-frequency arc) corresponds to the activation polarisation ($\eta_a$), which is related to TPB area and the number of reactive sites, whereas the second semi-circle (low-frequency arc) is associated with the concentration polarisation ($\eta_c$), comprising the mass transport resistance through electrodes and interfaces. When combined, these three parts give the overall cell resistance ($\eta$), as indicated by the intercept at low-frequency.

It can be seen that total cell resistance ($\eta$) decreased when the operating temperature increases for both fuels, with this decrease being more apparent for the CH$_4$-fuelled system. This is consistent with the observation that incremental changes in power density can be caused by temperature. There is only a small difference in ohmic polarisation for cells operated at different temperatures, measured at 0.3–0.35 and 0.3–0.5 Ω cm$^{-2}$ under H$_2$ and CH$_4$ operation, respectively. In comparison, it can be observed that for CH$_4$, concentration polarisation dominates total cell resistance, with this value being much higher than H$_2$. This is in agreement with the trend of the power density, where the lower performance of CH$_4$-fuelled systems could be due to more complex reactions taking place.

Fig. 7. Current density for operation at 750 °C with dry CH$_4$ under constant 0.7 V.
4. Conclusions

In summary, a unique YSZ scaffold has been developed via a phase-inversion and sintering process, which is composed of dense outer skin layer acting as the electrolyte and a series of micro-channels for the infiltration of anode materials. This distinctive structure allows the use of Cu, which has a lower melting point than the required temperature for the YSZ micro-tube sintering process, to be incorporated as the anode, whereby this process is extremely difficult via conventional fabrication routes. Such micro-structural design also provides well-distributed gas flow passages and helps to reduce transport resistance. Outstanding electrochemical performances have been obtained from both H2 and CH4 operation. Electrochemical testing showed that the system fuelled by dry CH4 yields a power density of 0.16 W cm−2, which is one of the highest ever reported for Cu-CeO2 anode (nickel free). It has been found that only minor carbon formation could be detected and negligible degradation has been observed during long-term test. This indicates that our cell developed from the micro-structured micro-tube not only contributes to a better cell structure but is also suitable for direct CH4 utilisation.

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Appendix A. Supplementary data

Supplementary data related to this article can be found at http://dx.doi.org/10.1016/j.jpowsour.2018.03.002.

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