A Rotating Electrolyte Disc (RED) for Operation in Liquid Metal Electrode Solid Oxide Fuel Cells

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A novel solid oxide fuel cell with liquid metal anode (LMA SOFC) has been recently proposed as an alternative to conventional solid oxide fuel cells. LMA SOFC allows operation on a variety of carbonaceous fuels – gaseous or solid, while remaining tolerant towards fuel contaminants [1]. The liquid metal electrode resides in a layer between the fuel and solid electrolyte. During current flow oxygen ions migrate though the solid electrolyte (yttria-stabilised zirconia) to the liquid metal anode. At the interface between the electrolyte and the liquid metal the \( \text{O}^2^- \) ions react electrochemically with the metal, generating metal oxide which in turn reacts with the fuel, producing water (hydrogen as fuel) and/or carbon dioxide (carbon or hydrocarbon as fuel).

With respect to reported earlier uncertainties in performance limitation of LMA SOFC [2] greater understanding of the electrochemical reactions, redox, and transport processes within liquid metal electrodes is needed. This study discusses the development of a technique to enable the determination of the parameters controlling mass transport of dissolved oxygen (and other gases) and oxidised species including metal oxide in the liquid metal; the technique also provides the possibility of the investigation of heterogeneous and homogeneous reaction kinetics as well as the influence of electrode contaminants.

Conventional rotating disc electrode (RDE) theory is applicable to the high temperature technique discussed here. In our design a solid electrolyte disc rotates in a liquid metal electrode (e.g. tin), as opposed to a solid electrode disc rotating in a liquid electrolyte, making this concept novel and unique. The theory of the RDE is well known for aqueous solutions [3], though it has not previously been applied to the study of solid oxide fuel cells with liquid metal anodes. Operation with liquid tin instead of aqueous electrolyte makes the proposed method interesting and challenging.

A schematic of the rotating electrolyte disc (RED) setup is shown in Figure 1. The metal is contained within a closed-end zirconia tube and held at elevated temperatures by an electric furnace. The oxygen content of the liquid metal is controlled by electrochemically ‘pumping’ oxygen into or out of the liquid metal via the zirconia tube with associated external air electrode. The working electrode is liquid tin and the electrochemical reaction takes place at the base of the rotating electrolyte. A platinum counter electrode is positioned on the opposite face of the rotating electrolyte disc inside the tube to which the disc is attached.

![Figure 1. Schematic of RED setup for LME SOFC.](image1)

To demonstrate the validity of the concept and design for high temperature studies, the setup was first tested using an aqueous system at ambient temperature (equimolar solution of ferrous and ferric chloride with KCl as supporting electrolyte and a platinum disc electrode on the base of the zirconia disc), Figure 2. The obtained data show that the developed design conforms to standard RDE behavior (increasing rotation speed \( \omega \) increased the observed limiting current density). Diffusion coefficients for Fe\(^{2+}\) and Fe\(^{3+}\) were determined from Levich plots. This study also presents preliminary results in molten tin electrodes.

REFERENCES

Symmetrical cells of Pr$_2$NiO$_{4+\delta}$ on electrolytes with apatite-type structure

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Materials with the K$_2$NiO$_4$-type structure have been recently proposed as cathode materials for their use in solid oxide fuel cells. Among this family of materials Nd/Pr nickelates have extremely good performances especially at intermediate temperatures (700-800°C), where the conventional oxygen conductor electrolytes like YSZ lose their supremacy. This intermediate temperature use, together with some chemical compatibility problems found with Zr based materials, make the combination of these nickelate cathodes with apatite electrolytes very interesting. The compatibility of two cathode compositions, Pr$_2$NiO$_{4+\delta}$ and Nd$_{1.95}$NiO$_{4+\delta}$ with 3 different electrolytes, La$_{9.67}$Si$_6$O$_{26.5}$, La$_{9.67}$Si$_3$Ge$_3$O$_{26.5}$ and La$_8$Y$_2$Ge$_6$O$_{27}$ has been evaluated for different sintering temperatures. Symmetrical cells have been prepared by dip coating of dense apatite pellets on cathode pastes and have been electrically evaluated.
Low temperature SOFC operation via interstitial diffusion mechanisms
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Keywords: Solid oxide fuel cell, interstitial oxygen diffusion, proton conduction, superstructure

In the search for SOFC materials which exhibit improved oxide ion conductivity at lower temperatures, attention has recently been focussed towards materials with atypical structural chemistry and diffusion pathways. Oxygen excess materials (such as the Ruddlesden Popper, Apatite structures) have the potential for interstitial oxide ion conduction with activation barriers to oxygen transport that are typically lower compared to the more common vacancy mechanism found in traditional SOFC materials\textsuperscript{1,2}. An alternative strategy to reduce migration barriers and enhance conductivity in the intermediate regime (4-600°C) is to change the charge carrier from oxide ions to protons, and develop electrolytes that conduct interstitial protons from the anode to the cathode\textsuperscript{3}.

Fergusonite structured rare earth niobates (RENbO\textsubscript{4}) can exhibit both high protonic and oxygen diffusivity depending on the dopant strategy. For example the alkaline earth doped members (RE\textsubscript{1-x}A\textsubscript{x}NbO\textsubscript{4-δ}, RE=La, Nd, Gd, Tb, Er, A=Ba, Sr, Ca) retain protons as the dominant charge carriers up to 1000°C, with oxygen vacancy diffusion resuming after\textsuperscript{4}. The cerium analogue CeNbO\textsubscript{4+δ}, can incorporate a range of oxygen excess stoichiometries (δ=0, 0.08, 0.25, 0.33) by oxidation of Ce\textsuperscript{3+} to Ce\textsuperscript{4+} and exhibits fast oxide ion and electronic conduction\textsuperscript{5}. Unlike other rare earth niobates CeNbO\textsubscript{4+δ} conducts via an interstitial type mechanism and adopts either commensurate or incommensurate modulated superstructures showing that symmetry is not necessarily a pre-requisite for fast ion transport. Here we report the transport properties of Sr\textsuperscript{2+} doped CeNbO\textsubscript{4+δ} under oxygen and proton containing atmospheres and show that this new material exhibits activation barriers significantly lower than both the parent material and other common SOFC materials whilst highlighting the progress made in novel superstructured, interstitial oxides for SOFC applications.

References
Using the Rotating Ring Disk Electrode Technique to Determine the Electron Transfer Number of Metal Oxide Catalysts

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Current energy production techniques involve the burning of fossil fuels. There are a couple of main problems using this technique. The first being the dwindling supplies of fossil fuels, even with the recent emergence of using shale gas as an energy source this only postpones the problem, it doesn’t solve it. In addition the burning of these fuels produces harmful greenhouse gases such as carbon dioxide. Due to these problems different methods of clean energy production are needed with fuel cells being a potential solution.

Optimisation of fuel cell technology needs to be undertaken especially that of the non-precious metal cathodes. One of the important properties of these cathodes is to be efficient, as well as less expensive meaning that the reaction should proceed via a 4 electron process rather than 2.

The determination of the number of electrons transferred in the oxygen reduction reaction can be determined by a couple of methods. The most well-known technique analyses Koutecky Levich plots generated from Rotating Disk Electrode data. However this method is dependent on the surface area of the metal oxide catalyst which cannot be accurately determined. To this end the work presented here focuses on refining a modified RDE technique named Rotating Ring Disk Electrode.

There are numerous types of solid oxide catalysts that have been researched in the past. In this work perovskite and perovskite related materials have been investigated. There are many examples of perovskites that have been researched previously one of the most common being the (La,Sr)MO$_3$ series with M = Mn, Fe and Co. This research has focused on CaMn$_{1-x}$Ru$_x$O$_3$ as well as the perovskite related pyrochlore Ca$_2$Ru$_2$O$_7$ (Figure 1).

![Figure 1: Crystal structure of perovskite and pyrochlore materials.](image-url)
Silicon dioxide. Fluorite’s poison is perovskite’s medicine.

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Silicon oxide is widely considered a detrimental contaminant of SOFC materials, particularly electrolyte materials, as it has been reported to segregate at the grain boundaries where it forms insulating siliceous phases, lowering the conductivity, such that overall performance is degraded. For example, it has been reported that several hundreds ppm of SiO₂ can increase the electrolyte grain boundary resistance by over one order of magnitude.

Following previous observations where oxyanions (carbonate, borate, nitrate, sulfate, phosphate) were successfully incorporated into perovskite-type cuprate superconductors and related phases [1-3], we have performed the introduction of silicate groups into the structure of perovskite-type SOFC electrode materials to analyse its effect on the structural and electrical properties of these materials.

These studies on Si incorporation were carried on different systems. For instance, in cobalt-based perovskite electrode materials, the successful incorporation of Si into La₀.₆Sr₀.₄Co₀.₈Fe₀.₂O₃₋δ and Sr₁₋ₓYₓCoO₃₋δ based materials resulted in improvements in the conductivity and an enhancement in the stability towards CO₂. [4] Very recently, SrₓCa₁₋ₓMnₓO₃₋δ and SrₓCa₁₋ₓFeₓSiₓO₃₋δ cathode materials have been prepared by solid state reaction, and direct evidence for the incorporation of Si into the structure provided for the first time by ²⁹Si NMR. [5-6] In each case, Si doping is shown to enhance the conductivity, which can be attributed to electron doping (driven by the introduction of oxide ion vacancies due to the preference for Si to adopt tetrahedral coordination), as well as, for some systems, a change from a hexagonal (containing face sharing of octahedra) to a cubic perovskite (containing corner sharing of octahedra). In addition, work on SrₓCa₁₋ₓFe₁₋ₓSiₓO₃₋δ materials has shown the stabilization of the cubic form in reducing conditions, making them potentially suitable for use as cathode and anode materials in symmetrical SOFCs.

The enhancements in performance is contrary to previous assumptions of the detrimental effect of Si and can be attributed to the incorporation of the Si into the perovskite structure, whereas in prior studies of the effect of Si on fuel cell materials researchers have typically examined its addition as a secondary phase.

In this work we have developed microtubular solid oxide fuel cells (SOFCs) based on an extruded support. Firstly, Ni-YSZ (yttria stabilized-zirconia) anode is manufactured by Powder Extrusion Moulding (PEM). Feedstock composition and extruding parameters are adjusted to obtain tubular green bodies. An YSZ layer is then deposited by dip coating as the electrolyte and the sintering parameters are optimized to obtain a dense layer. An active area of ~1 cm$^2$ LSM-YSZ is deposited as the cathode.

The electrochemical performance is measured in fuel cell, electrolysis and coelectrolysis mode. At 850 °C, the power density at 0.5V was 0.7Wcm$^{-2}$ and ASR at OCV was 0.9 Ωcm$^2$. The obtained values are similar to state of the art YSZ based SOFC. In steam electrolysis mode, at 850 °C and thermoneutral voltage (1.3 V) we have measured a current density of 0.9 A cm$^{-2}$.

Preliminary results of steam and CO$_2$ coelectrolysis to produce syngas will be finally presented.
Multipurpose electrode materials for intermediate temperature solid oxide electrochemical systems

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The development of versatile electrode materials to be used in symmetrical fuel cells or reversible fuel cell-electrolysers, can enormously simplify and reduce the cost of the device processing with a huge impact on the commercialization. However, the high number of requirements that these systems must fulfil represent a great materials challenge that is preventing their implementation in the devices. Perovskite oxides are able to accommodate a wide variety of stoichiometries and crystal structures allowing the tuning of the desired properties to satisfy all the requirements for the different electrode applications by slight changes in the composition or under different environments. In this work, we have studied the composition, crystal structure and physical properties relationships of different families of perovskites to understand the complex mechanism affecting the electrochemical performances and practical limitation of these multipurpose electrodes. First, SrCoO$_3$-$\delta$ derivatives were evaluated as air mixed ionic–electronic (MIEC) electrodes for reversible fuel cell-electrolyser showing a considerable improvement of the electrode performance under anodic polarization conditions, where the material is catalysing the oxygen evolution reaction in the electrolyser, reaching overpotential values as low as 28 mV for a current density of 210 mA/cm$^2$ at 700 °C. Second, the behaviour of a number of materials under oxidising and reducing conditions, were analysed to be used as symmetrical, anode and cathode, electrodes in fuel cells. It was observed that the topotactic oxidation-reduction in perovskites facilitates their reversibility without abrupt changes in the thermal expansion. Promising behaviour was obtained for some compositions such as the La$_{0.5}$Sr$_{0.5}$Co$_{0.5}$Ti$_{0.5}$O$_{3-\delta}$ MIEC phases that may be used as a cathode or as an anode at 800 °C, yielding power densities of 220 and 170 mW/cm$^2$, respectively or simultaneously as cathode and anode a maximum power density of 110 mW/cm$^2$. 
Tailoring in situ growth of nanoparticles towards applications

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Surfaces decorated with nanoparticles play a key role in many fields including renewable energy and catalysis and are typically prepared by deposition techniques. Here we show that, alternatively, particles could be grown in situ, directly from a perovskite support though judicious choice of composition, particularly by tuning deviations from the ideal ABO$_3$ stoichiometry.$^1$ This concept seems to enable unprecedented control over particle composition, size, distribution, surface coverage and anchorage, and may serve to design sophisticated materials for several applications.

A few examples are presented to illustrate the type of particles and microstructures that can be produced but also the factors that control this phenomenon. Examples of specific applications are also addressed, including the production of alloy nanoparticles, metal-ceramic interfaces and H$_2$ production from high temperature steam electrolysis with in situ exsolution of catalytically active nanoparticles during operation.$^2$

References

Abstract for Oral Presentation

Development and Performance of (Mn,Mg)FeCrO$_4$ -based Electrodes and Coatings for Solid Oxide Fuel Cells

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Important advances have been made in SOFC development utilizing a ceramic framework based upon yttria zirconia (YSZ) electrolytes supported upon porous YSZ electrode skeletons. This ceramic framework is sintered at high temperatures with subsequent impregnation and low temperature processing of the active electrode materials. Here we seek to develop this impregnated electrode concept by investigating a novel scaffold material similar to the main corrosion product of ferritic stainless steel. The chromium rich spinel (MnFeCrO$_4$) was used as an electrode support material, either alone or impregnated with (La$_{0.75}$Sr$_{0.25}$)$_{0.97}$Cr$_{0.5}$Mn$_{0.5}$O$_{3-\delta}$, La$_{0.8}$Sr$_{0.2}$FeO$_{3-\delta}$, Ce$_{0.9}$Gd$_{0.1}$O$_2$-$\delta$, CeO$_2$ and/or Pd. In these initial studies it was found that all of the impregnated phases adhere very well to the spinel and considerably enhance performance and stability to a level sufficient for SOFC applications.$^{1,2}$

Additionally, an approach was envisaged to use these spinels as coatings to protect steel particles in e.g. steel-supported solid oxide fuel cells which are known to oxidise in high fuel utilisation conditions.$^3$ Our approach consists of scavenging the FeCr oxides formed during a controlled oxidation process into a continuous and well adhered spinel coating. In order to achieve this, MnO$_x$ and MgO were separately infiltrated into two different porous FeCr/YSZ supports as nitrates, thus capturing the oxide scales of metal supports (Fe,Cr)O$_x$ into spinels (Mg,Fe,Cr)$_3$O$_4$ and (Mn,Fe,Cr)$_3$O$_4$. Another benefit of this approach would be that the Mg/Mn-FeCr spinels are expected to have higher electronic conductivity compared to the Cr$_2$O$_3$ oxide scale.

References

In Situ Studies of Redox Exsolution in Perovskites

John Irvine, Dragos Neagu, George Carins and Cristian Savaniu.

The scope of this research is the study of SOFC anode materials in operationally representative environments. Perovskite compositions have attracted attention over the last few years as alternative anode materials. Moreover, we seek here to produce catalyst nanoparticles exsolvated from perovskites through careful control of their defect chemistry. This exsolution capability holds significant potential due to possible enhancement of catalytic activity in the oxidation of Hydrogen fuels in high, intermediate and low temperature SOFC anodes. We recently completed an initial in-situ neutron diffraction study on the nanocatalyst exsolution from strontium titanates and related compositions at ILL (France). Of interest was the nature of exsolvated nanoparticles and their anchoring to the bulk material. Figure 1 shows the diffraction patterns obtained for La_{0.8}Ce_{0.1}Ni_{0.4}Ti_{0.6}O_{3} at different stages of the in situ reduction as well as one for an ex situ reduced sample. Clear from the data were the structural changes to the parent Perovskite lattice as well as the evolution of diffraction peaks associated with nanoparticulate Ni and CeO_{2}. Other compositions studies were: La_{0.49}Sr_{0.31}Fe_{0.03}Ni_{0.03}Ti_{0.94}O_{3} and La_{0.52}Sr_{0.28}Ni_{0.06}Ti_{0.94}O_{3}. In situ neutron diffraction data analysis will be presented offering invaluable insights onto the nature and formation of the exsolutes and structural changes of the perovskite upon reduction.

![Figure 1: Neutron diffraction patterns of La_{0.8}Ce_{0.1}Ni_{0.4}Ti_{0.6}O_{3} – As prepared, reduced in situ and pre-reduced ex situ](image-url)
Can strained oxide ion conductors provide a route to low-temperature SOFCs?

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Developments in thin film growth methods have allowed for the fabrication of model systems of SOFC materials with an advanced level of control over the materials properties. This has opened a new route for ion transport to be studied in unprecedented detail. Coupling functional oxide films with insulating substrates of varying lattice mismatch allows direct control over interfacial phenomena such as space change regions, lattice strain and dislocations. All have all been linked to extraordinary enhancements in the oxygen ion conductivity of commercially used electrolyte and cathode materials; in some cases an improvement of up to eight orders of magnitude has been reported.¹ Such an enhancement, if it could be incorporated into a SOFC, would have huge implications for lowering the operating temperature and improving efficiency and has hence shifted the paradigm from developing new material compositions to studying nanostructured systems in order to tailor such favourable interfacial effects.

As one of the most common commercially used SOFC electrolyte materials, single thin films of yttria-stabilised zirconia (YSZ) have received much attention, with reports of conductivity ranging from an enhancement of up to three orders of magnitude²³ to a reduction by a factor of around five.⁴⁵ Dislocation networks at the film/substrate interface have often been considered as the cause yet is still a subject of debate, with concrete evidence for the phenomenon still proving elusive. In addition, electrical measurements on such films only provide a measure of the total charge transport leaving an ambiguity about the nature of the charge carrier.

In this work YSZ films have been grown on a number of different substrates using pulsed laser deposition, where the interfacial strain, microstructure and thickness can all be controlled. X-ray diffraction and high-resolution transmission electron microscopy have been used to comprehensively characterise the film orientation and microstructure. Isotope tracer diffusion combined with secondary ion mass spectrometry will be presented as a method to directly and unambiguously measure the oxygen ion transport properties in these films. Finally new evidence on the change in the conductivity as a result of locally strained YSZ via a dislocation network will be shown.

In situ investigation of curvature change induced by stress in multilayer structure during co-sintering for wavy type SOFC

Abstract

Wavy type Solid Oxide Fuel Cell (SOFC) using the co-sintering process is beneficial for improved triple phase boundary (TPB) conditions compared with planar type fuel cells of the same diameter. For application of wavy type SOFC in intermediate temperature, materials used were Ni-CGO for anode; CGO for electrolyte; and LSCF for cathode. These materials were tape-casted with 20µm thickness and stacked to form a multilayer structure via hot pressing. In-situ observation using long focus microscope, Infinity K-2, was carried out to monitor shaping. Prior to co-sintering, this structure was supported by Al₂O₃ rods to be pre-shaped as wavy type so as to aid formation of the wavy shape during co-sintering. The co-sintering process induces stress derived mainly from the different sintering behaviour and the viscosity variation between individual layers. This induced stress contributes to curvature change of the structure and that can be utilised to form the wavy type shape. In order to calculate the level of stress induced during co-sintering, bilayers, consist of Ni-CGO/CGO and CGO/LSCF, were co-sintered and their curvature developments were also monitored. The analysis of monitored results enabled to predict curvature development of a triple-layer wavy SOFC. In addition, this paper investigates how different thickness ratios affect stress developments through analysis of curvature development during the co-sintering.

NOTE: The author of this abstract is Indae Choi (Loughborough)
Solid oxide fuel cells (SOFCs) are becoming increasingly popular due to their low carbon, high efficiency operation and ability to utilise a wider range of fuels than low temperature fuel cells. However, in order to reach the full potential of such devices further knowledge is required regarding the strains experienced during start-up and operation, and subsequent failure mechanisms. Here the authors present work conducted at the Joint Engineering, Environmental and Processing (I-12) beamline at Diamond Light Source. By building a novel furnace design it has been possible to couple X-ray diffraction (XRD) with high-resolution infrared thermal imaging in order to study SOFC samples at operating temperatures (>600°C). The design of the furnace was such that thermal gradients were imposed upon the samples whilst conducting XRD to enable crystallographic strain mapping to be undertaken along isotherms at 100 μm intervals across the sample. In addition, macroscopic strains are calculated using experimentally obtained thermal data to calculate the effect of the thermal gradients imposed. These measurements have been obtained at incremental temperatures through the start-up regime allowing the full effect of heating the samples to be observed.
This presentation will focus on potential fuel cell applications in the processing of lignocellulose or wastes into clean energy generation. There are three strands to the work to be presented, as follows.

Earlier work has investigated how energy from waste generation efficiency can be greatly enhanced by the use of solid oxide fuel cells (SOFC). The work delivered systematic process integration strategies and process simulation, to improve heat recovery including waste heat, energy efficiency and cleaner operation, of biomass gasification fuel cell (BGFC) systems. The performance of the integrated BGFC system was thoroughly analysed for a range of power generations, ~100-997 kW. Increasing power generation from a BGFC system decreases its power generation efficiency (69-63%), while increasing combined heat and power generation efficiency to 80-85%.

To overcome the threats posed by climate change, advanced clean energy technologies are urgently needed. Although hybrid fuel cells have emerged as attractive power generation solutions, there is limited attempt to optimise these systems using model based engineering. The presentation will give a summary of fundamental analysis and an optimisation framework, to choose design parameters and performance sensitivity of hybrid systems working with waste-derived syngas.

My recent work focuses on the low temperature proton exchange membrane fuel cell (PEMFC) performance analysis and characterisation for hydrogen production using a range of carbon based electrodes in a range of configurations. Alongside, model-based engineering approach shows opportunity for utilisation of wastes in PEMFC for hydrogen and energy production.
Solid Oxide Fuel Cells (SOFCs) present a highly desirable form of energy production due to their high potential efficiencies. However, improving the long term performance is still key to commercial implementation. Improving the understanding of degradation mechanisms therefore offers essential help on the route to commercialisation.

The ability to predict the lifetime performance of a SOFC would give guidelines for where improvements and alterations can be made in terms of production, operating parameters and cell design. Multiple attempts have been made to implement models which can respond to all the varying parameters to give their characteristic performances over longer operational lifetimes than is feasible to physically test.

This study focuses on continuous degradation mechanisms and the impact they have on the single repeating unit level, i.e. the anode, cathode, electrolyte, metal interconnect and sealants. Currently the model being developed deals specifically with anode and electrolyte degradation. Depending on operating conditions, these cells suffer from Ni particle agglomeration, coarsening, and volatisation which lead to increased overpotential in the anode. A loss in the ionic conductivity in the electrolyte is also observed (1).

Using models derived for these degradation mechanisms (1) combined with percolation models (2), Matlab® coding has been developed to outline the loss of performance in a single repeating unit for a given set of operating parameters. These equations will be tested against real life data to establish their validity and, where necessary, adjusted for accuracy. Further progress will allow for accelerated testing of components and cells to determine the long term performance of a given material alteration or design change.

Performance of micro-tubular SOFCs on simulated biogas

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To explore the ability of Nickel-Yttria stabilized Zirconia (Ni-YSZ) anode-supported micro-tubular SOFCs (mSOFCs) operating with renewable fuel, experimental works have been carried out to fuel mSOFCs with simulated biogas. Varied compositions of CO$_2$/CH$_4$ fuel mixtures (7:3, 6:4, 5:5, 4:6, 3:7 volumetric ratio) were used at operating temperatures of 800°C to 700°C. Attention was drawn to measuring power density at various current densities and observing cell degradation at constant current operation over 40 to 70 hours of operation. Beside measurements of coke formed by Temperature Programmed Oxidation (TPO) and Scanning Electron Microscope (SEM) observation of the anode surface before and after cell testing, cell impedance was also measured to diagnose causes of performance degradation. The mSOFCs utilized had a 2.3mm external diameter and 55mm length, 200µm thick Ni/YSZ anode, 15µm YSZ electrolyte and 20µm Lanthanum Strontium Manganate (LSM) cathode offering 0.8 to 1.0cm$^2$ active surface area. Results showed that mSOFCs can be operated on biogas with stable power output at 0.8 to 1.4A/cm$^2$ current density. A typical result of an mSOFC fuelled with CO$_2$/CH$_4$ at 3:7 ratio operating at 800°C and constant current of 1.25A/cm$^2$ produced 725mW/cm$^2$ power. After being tested for 70 hours, power produced from this cell was decreased to 625mW/cm$^2$ giving power degradation of 14.0%. On several cells tested, it was observed that cell performance stabilized within 15 to 20 hours of operation at constant current (with 5-7% voltage fluctuation) but significant voltage decrease occurred after 45 hours of operation. Measurements of impedance showed that cell ohmic and polarisation resistances significantly increased after the cell was fuelled with simulated biogas of low CO$_2$/CH$_4$ ratio. Visual observation of the cell after being tested and impedance results showed that cell degradation may have been caused by combined effects of coke formation, cell component cracks and anode re-oxidation. This work is still in progress. More data are being collected to further elucidate cell degradation mechanisms. However, the initial results presented in this paper show promising performance of operation of Ni-YSZ anode supported mSOFCs with biogas.

Keywords: micro-tubular SOFC, biogas, cell degradation
Lowering the operating temperature of SOFCs: Protonic conductors and the role of local structure on understanding ionic conductivity

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Inorganic materials containing mobile protons in the 300-600 °C temperature regime have found applications as protonic conductors in fuel cell devices [1]. Structural analysis of the protonic defects is not straightforward by conventional diffraction techniques, whilst ¹H solid-state NMR spectroscopy (often in combination with double resonance techniques) provides a method with which to directly probe local structure [2,3]. Some of the key questions that are important in these protonic materials are related to trapping defects where protons become less mobile and thus hinder the overall proton conduction [4]. Typical examples of such materials include cation doped perovskites such as yttrium-doped BaZrO₃ (BZY). Identification of these trapping defects, and reducing the trapping energies, are key for designing new materials having better proton conductivity.

In this study, a joint multinuclear NMR spectroscopy, and density functional theory (total energy and GIPAW NMR calculations) investigation of yttrium-doped BaZrO₃ was performed. The results are consistent with a vacancy-dopant and proton-dopant trapping model in dehydrated and hydrated BZY, respectively. The agreement between spectroscopy and first principle calculations provides a path for material design of next generation protonic conductors.

References
Characterization of a novel Ni-impregnated GDC Electrode for Solid Oxide Cells application

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One of the main challenges of Solid Oxide Cells (SOCs) is to achieve an electrode microstructure with a high number of triple phase boundaries, and good electrical and ionic conductivity, to meet both performance and lifetime targets. In this study a novel cathode structure for SOCs was made by impregnation of a porous Gd-doped Ceria scaffold with Ni nano-particles. The electrical conductivity of the cathode was assessed by the Van der Pauw method. Electrochemical performance in steam/hydrogen and carbon dioxide/carbon monoxide mixtures was evaluated using three electrode DC and AC measurements over a range of temperatures (550-750°C) and gas concentrations. The microstructure of the cathode was studied using Scanning Electron Microscopy. Encouraging results have been obtained for the performance of the electrode and results from these studies will be reported.
A Direct Methane SOFC Using Doped Ni-ScSZ Anodes For Intermediate Temperature Operation

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Abstract

Solid Oxide Fuel Cell (SOFC) systems operate at temperatures 500 – 950°C and have garnered interest in recent years due to their higher conversion efficiencies when compared to heat engines, variable fuel capability, low noise operation and cell design flexibility. While these advantages make SOFCs one of the most sought after technologies, the technical challenges associated with high temperature operation and the issues with the utilization of hydrocarbon fuels currently create economic barriers for widespread implementation. Developing SOFC systems that operate directly on hydrocarbon fuels allows immediate use of fossil fuels, eliminates the need for separate fuel reformers and purification systems and allows by-product heat to be recycled back into the cell stack or used in a cogeneration heat and power application. Direct hydrocarbon fuel utilization coupled with low temperature operation may create new operating difficulties but at the same time system stability and materials degradation may be improved so that a decrease in temperature promises major cost benefits and promotes an ever increasing interest in SOFC commercialization, solidifying their position in the new energy economy.

Conventional nickel-yttria stabilised zirconia (Ni-YSZ) is the most developed and most commonly used anode because of its low cost and exceptional performance in H₂ rich environments but under hydrocarbon operation, Ni-YSZ can deteriorate significantly due to low sulphur tolerances and carbon poisoning. Literature states that Ni-based cermet containing metals and metal alloys demonstrate high catalytic activity for hydrocarbon oxidation and are slower for carbon catalysis than Ni alone. Power densities of .33W/cm² (800°C) have been obtained for single cells using Cu-Ni-CeO/YSZ anodes (YSZ electrolytes) and .75W/cm² (600°C) for single cells using Ru-Ni/GDC anodes (GDC electrolytes) both operating on direct methane. While these studies show proof of concept, extensive research is necessary to find cheaper, better performing catalysts for nickel-zirconia anodes that exhibit performance stability on hydrocarbon fuels over extended lifetimes and at lower temperatures.

The aim of this work is to demonstrate direct methane SOFC operation by developing new Ni based ZrO₂ anode formulations that suppress carbon formation and are stable against sulphur impurities without sacrificing cell performance. Alternative electrolyte systems will be examined to measure their impact on cell performance and intermediate temperature operation.

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Towards Apatite based SOFCs: Investigating alternative synthesis methods and dopant incorporation into apatite electrolytes.

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Keywords: Solid oxide fuel cell, apatite, electrolyte, electrode, silicon

Introduction

The performance of SOFCs depends on the efficiency of the individual materials, as well as the interfaces between them. Doped lanthanum silicate/germanate apatites (La$_9$Sr$_6$Si$_6$O$_{26.5}$, La$_{9.33+x}$Si$_6$O$_{26+x/2}$) have attracted considerable attention on their own as electrolyte materials.[1] However, there has been some discussion regarding the use of apatite silicate electrolytes due to the high temperature of synthesis and sintering conditions required to produce dense electrolyte membranes, as well as concerns over the possibility of silicon/germanium diffusion from the apatite phase poisoning the electrode materials. In this work hydrothermal and sol gel routes have been investigated for the synthesis of apatite electrolytes, and these methods have been extended to the preparation of doped systems.

Results and Discussion

Apatite silicate electrolytes have been examined with the interest to lower the synthesis temperature through hydrothermal methods. The successful synthesis was demonstrated in an alkali metal hydroxide solution using synthesis temperatures as low as 200°C. However, the results indicated incorporation of the alkali metal into the apatite phase, lowering the interstitial oxide ion content and hence also the conductivity.

A sol gel route to Ge based apatites has also been demonstrated, with formation of the apatite phase at temperatures as low as 700-800°C. This method has been extended to the preparation of V doped apatites. The partial replacement of the Ge$^{4+}$ ions with the higher charged V$^{5+}$ ions increases the oxygen stoichiometry. The limit of this replacement is seen around La$_8$Y$_2$Ge$_5$V$_{0.5}$O$_{27.25}$, and conductivity measurements are currently underway. Further work will be undertaken to involve the apatite electrolytes in fuel cell tests, with the long term stability of the conductivities of these systems being examined.

References

Electrochemical synthesis of ammonia directly from air and water using a Li⁺/H⁺/NH₄⁺ mixed conducting electrolyte

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ABSTRACT
Ammonia has been successfully synthesised directly from air and water using an electrochemical cell based on H⁺/Li⁺/NH₄⁺ mixed conducting membrane and Pt/C electrodes. It is found that the Nafion 211 membrane exhibits mixed H⁺/Li⁺ conduction after exchanging in 0.1 M Li₂SO₄ solution. The ionic conductivity of the mixed conductor is slightly lower than that of H⁺-form Nafion 211. The introduction of Li⁺ ions to the cell did not improve ammonia formation rates in our experiments. Reasonably higher temperature may favour ammonia formation and the highest ammonia formation rate (9.37×10⁻⁶ mol m⁻² s⁻¹) and Faraday efficiency (0.83%) has obtained at 80 °C when a voltage of 1.2V was applied. Ammonia formation rate decreased while 0.1 M Li₂SO₄ solution instead of water was used in the cell. In our experiments, the cell became unstable at higher applied voltages and the best applied voltage was 1.2V.

Keywords: ammonia synthesis, air, water, mixed conductor, Li₂SO₄

Figure 1 The ammonia formation rate (A) and Faraday efficiency (B) of the cell at different temperature and applied voltage.

References:
Abstract

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The experimental evaluation of a cleaning gas system at low temperature (<400 °C) for conditioning the producer gas of biomass gasification in a two air supply stages co-current gasifier, as fuel to a Solid Oxide Fuel Cell (SOFC) are presented. Analysis methodology for impurities composition (tar particles, H2S, NH3 and HCl) and permanent gases composition (H2, CO2, CO, N2, CH4) and temperature profiles inside the gasifier are also presented. The comparison of impurities removal using a primary method (two air stages reactor) and secondary method (external cleaning gas system) was made. The gas cleaning system tested is composed of a sand filter, a venturi scrubber, a spray scrubber, a fixed bed reactor and a ceramic filter.

The concentrations of carbon monoxide, hydrogen and methane in the produced gas were of 19.01, 16.21 and 1.50%v, respectively, with a lower heating value around of 4.61 MJ/Nm3. Variations on the air ratio (Ra) and the total airflow in the primary method have a significant effect on temperature, allowing a reduction of 87%, 70%, 89%, 43% and 54% at tar, particles, H2S, NH3 and HCl respectively. Secondary method, external cleaning gas system reduced the tar, particles, H2S, NH3 and HCl concentrations up to 96%, 97%, 92%, 60% and 95% respectively. The minimum values for tar, particles, H2S, NH3 and HCl were 12.51 mg/Nm3, 6.02 mg/Nm3, 2.85 mg/Nm3, 121.72 mg/Nm3 and 9.18 mg/Nm3 respectively. These results are low enough, and the gas will be used as fuel in a SOFC.

Keywords:
Biomass, Double stage gasification, downdraft reactor, Low temperature gas cleaning, SOFC.
A challenge to form pinned Cu-based alloy nanoparticles on the surface of oxides for SOFC applications

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Solid oxide fuel cells (SOFCs) are one of the important energy generation technologies which are environment friendly due to low emission of noxious gases. These have capabilities to convert chemical energy of fuels into electrical energy with high efficiency. The state of art cells use Ni/YSZ cermet as fuel electrode since Ni possesses excellent catalytic activity towards the oxidation of various fuels including hydrocarbons at higher temperatures. However, it has serious issues of long term stability due to coke and/or sulphur poisoning under hydrocarbon fuels, and poor redox tolerance. It has been reported in the literature that the carbon deposition can be suppressed if Ni is used as Ni-Cu alloy because Cu has very low activity towards the formation of C-C bond when exposed to hydrocarbon fuels. However, fabrication of electrodes based on Cu alloys like Cu-Ni, Cu-Fe and Cu-Co is problematic due to low melting point of Cu. Furthermore, the high mobility of Cu on the surface of oxides results in performance degradation issues of these electrodes. Such electrodes containing uniformly dispersed pinned Cu-based alloy nanoparticles throughout could be fabricated by directly growing these nanoparticles in-situ through exsolution process. In this method, a metal of interest is initially substituted into the perovskite lattice of a perovskite, typically in oxidising conditions. On reduction, the doped metal exsolves from the host lattice and forms the nanoparticles on the parent perovskite surface. Compared to initial approaches of this technique, our approach employs advanced control over perovskite nonstoichiometry and subsequent defect chemistry to tailor the exsolution process. The nanoparticles produced by this method have good anchorage onto the host lattice which enhances not only the stability of the catalyst but also reduces the aging effect of the catalyst. Therefore, in-situ exsolution can be considered a way to produce robust catalyst and thus it can play a vital role in tackling improved performance and long term stability issues of SOFCs. In this study we developed new compositions with copper doping in titanate perovskites. We adopted various strategies to tune defect chemistry for optimal copper solubility in the perovskite lattice and the exsolution itself from the resulting perovskites. Our study revealed that the La contents higher than 32 mole percent tend to form La-Cu based oxygen excess type compound as a secondary phase. New copper doped LST perovskites seemed to be stable under 5%H2/N2. We also found that the exsolution of copper from the LST perovskites lattice strongly influenced by the extent of both A-site and oxygen deficiencies, and surface ordering due to the enrichment of A-O on the surface. We treated the surface of these newly developed Cu based perovskites by quenching and polishing techniques separately and studied their effect on exsolution. Quenching treatment, particularly in Ce containing composition, found to be very effective to drive the exsolution. Surface polishing proved very effective in increasing the nucleation point for the formation of nanoparticles. We further replaced some of the copper with very catalytic active metals like Ni, Co and Fe to produce Cu-M (M= Ni, Co and Fe) alloy nanoparticles. We will demonstrate the potentials of Cu-M alloy nanoparticles produced by in-situ exsolution for SOFC anode applications.
Manufacturing of DC-SOFC components by direct ceramic inkjet printing

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Electromagnetic drop on demand direct ceramic inkjet printing (DCIJP) was employed to fabricate components of electrolyte supported direct carbon solid oxide fuel cells (DC-SOFC). Anodes (NiO-8YSZ; Gd:CeO\textsubscript{2}) and cathodes (LSM, LSCF) were printed using suspension inks with different mass loads and compositional ratios. The technology allows for easy modification of the coatings, including thickness and compositional control, porosity graduation and precise infiltration with catalytically active materials. Printing parameters including pressure, nozzle opening time and lateral drops overlapping were studied in order to optimize the surface quality of the coatings. A number of button cells as well as large area (50x50 mm area) square cells were successfully prepared. Inks rheology and printing patterns optimizations were required to eliminate any detrimental “coffee staining” effects. Scanning electron microscopy (SEM) revealed highly coherent electrolyte/electrode interfaces and uniform porosity distribution successfully and repeatedly created in coatings thinner than 10 μm. The cells were tested with non-purified carbon by-product of direct RF plasma reforming of natural gas. The comparative tests showed similar performances for screen printed and inkjet printed cells. DCIJP was used also for impregnation of the anode’s porous scaffoldings with Cu based sol inks leading to an overall improvement in the DC-SOFCs performance. The work also demonstrated that DCIJP can be potentially cost effective route for SOFC components processing.
Pro II is a software package designed to allow a user to build a process model and then simulate the model without tedious calculations such as mass and energy balance. This software will be used to model an integrated gasification/Solid Oxide Fuel Cell (SOFC) system utilising experimental data from the hydrothermal conversion of biomass. Ultimately, the gasification of an unconventional biomass to power a SOFC from bio-gas will be modeled using Pro II.

There are several challenges to overcome with the development of such a model. Due to the complexity of natural matrices such as biomass, all examples in the literature that consider the modelling of a gasification process use model compounds e.g. glucose and glycerol, and consequently there have been extensive reports on the gasification reactions that occur. Glycerol was therefore selected as an initial basis for the model. In addition to the literature data, results generated from small-scale gasification of glycerol will be used to develop a comprehensive integrated model.

The data obtained will be used to generate the glycerol gasification model, which will be integrated with the SOFC model. Ultimately, results from the small scale gasification experiments, will be used to accurately model the gasification of biomass and fuelling of a SOFC with the product bio-gas.
The aim of this research is to develop a real-time three-dimensional temperature map of a SOFC stack while delivering minimum disturbance to the normal operation. A specially designed multi-junction thermocouple array was sputtered on a SOFC substrate and tested in furnace to assess the potential of cell integrated thermocouples for in-situ monitoring of SOFC temperature. The multi-junction design enabled to reduce the number of external wiring required. Alumel (Ni:Mn:Al:Si – 95:2:2:1 wt%) and Chromel (Ni:Cr – 90:10 wt%) of 99.99% purity were chosen as the thermocouple materials. The external lead wires were chosen from the same material as the sensor, in order to guarantee elimination of noise introduction at the junction between thin films and lead wires. A simulation code was developed using MATLAB to assess the theoretical performance of sensors under varying design conditions in order to facilitate the sensor design. Seebeck coefficients of the thermocouple materials at 20°C and 1000°C were taken from published data as reference values. The values corresponding to other temperatures, in the range from 20°C to 1000°C, were linearly interpolated to compute sensor outputs at different temperatures. A virtual instrumentation panel was developed using LabVIEW for data acquisitioning, monitoring and analysing. The functions of the simulation code were validated by comparing the performance curves generated by the code for commercial thermocouples with the corresponding standard performance curves; they matched satisfactorily. The functions of the virtual instrumentation panel were validated via temperature measurement tests using commercial thermocouples; they also produced reliable outputs. A commercial sputter coater, Quorum Q150T, was used to deposit the thermocouple array and sputtering was identified to be an effective technique for such fabrication. The analysis of the experimental results proved that multi-junction thermocouple concept works satisfactorily and revealed the potential of this concept for in-situ monitoring of SOFC temperature. Overall, this research is moving successfully towards building in-situ 3D temperature map of a SOFC stack in order to improve cell and stack design, as well as being able to calibrate cell and stack models, thus improving system operation.
An Innovative Solid Oxide Fuel Cell Tri-Generation System for Low Carbon Domestic Buildings

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ABSTRACT

As a result of growing concerns of climate change and ever increasing cost and scarcity of fuel resources, fuel cells with their high electrical efficiency, low emissions and useful heat output have been identified as a key technological option for improving both building energy efficiency and reducing emissions. A 1.5kW, low temperature solid oxide fuel cell (SOFC) liquid desiccant tri-generation system has been proposed as an environmentally friendly way of providing heating, cooling and electricity to low carbon domestic buildings. The described system will be constructed and trialled at The University of Nottingham as part of a European Union (EU) funded project named Durable Solid Oxide Fuel Cell Tri-generation System for Low Carbon Buildings (TriSOFC).

The aim of this paper is to provide an introduction to the EU TriSOFC project along with some experimental results from the SOFC and desiccant air conditioning unit. The paper is split into three parts. Section one provides details of the TriSOFC system along with project aims and objectives. Section two presents a review of the state-of-the-art surrounding fuel cell and desiccant based tri-generation systems. Conclusions of the review are discussed in terms of their impact on the TriSOFC system - most significantly, (1) a considerable gap in the literature currently exists surrounding tri-generation energy systems based on SOFC combined with liquid desiccant air conditioning technology and fuel cell based tri-generation systems in residential applications, (2) the operating temperatures and heat output of a SOFC are in good thermal compatibility with desiccant regeneration temperatures and heat requirements, making them appropriate for integration in a tri-generation system, and (3) maximising thermal energy utilisation in combined heat and power (CHP) and tri-generation systems is challenging, but can have a large impact on system efficiency. Section three presents experimental results from the SOFC and desiccant air conditioning unit. These components have not yet been combined to form the complete TriSOFC system; however results from these studies are significant, as they will, (a) serve to validate and prove the viability of the complete TriSOFC system, and (b) aim to generate a greater understanding of the technologies, particularly in a domestic setting. An experimental rig was designed and built to test the SOFC tri-generation system. A heat load was used to simulate thermal output from a SOFC at an operating temperature of 50°C, and the results showed that for a heat input of 1kW a cooling output of 1.5kW could be achieved, corresponding to a coefficient of performance of approximately 1.5. Experimental SOFC results will be used to develop the system for installation in the field trials planned for 2014/2015 using a commercially available 1.5kWe BlueGEN unit installed at The David Wilson House, The University of Nottingham. The liquid desiccant air conditioning unit is studied in a laboratory setting and results presented look to the influence environmental conditions (air temperature and relative humidity) and operating settings have on the performance of the air conditioning cycle. Outcomes of the SOFC and liquid desiccant air conditioning studies are discussed and their influence on the TriSOFC system commented upon.

Keywords: Tri-generation, solid oxide fuel cell, liquid desiccant air conditioning, low carbon buildings
Composite Cathode by Electrospinning for Intermediate Temperature Solid Oxide Fuel Cells

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Cathode material $\text{La}_{0.6}\text{Sr}_{0.4}\text{Co}_{0.2}\text{Fe}_{0.8}\text{O}_3$ (LSCF6428) and electrolyte material $\text{Ce}_{0.9}\text{Gd}_{0.1}\text{O}_2$ (CGO91) were synthesized as nanofibers by electrospinning method followed by a simple thermal treatment. The morphology and structure of as obtained nanofibers were characterized by field-emission scanning electron microscopy (FE-SEM) and X-ray diffraction (XRD). The typical ceramic fiber diameter is 100-200 nm. The results showed LSCF6428 had a single perovskite phase. Then the two types of nanofiber and co-sintered to fabricate composite cathode. The electrochemical properties of the composite cathodes were determined using electrochemical impedance spectroscopy (EIS) analysis. The area specific resistance (ASR), and activation energy of the two composite cathodes were compared at temperatures from 500 to 650°C, which suggested the interconnected nanofibers of cathode and electrolye materials has better performance for IT-SOFC than does the nanoparticles.