

STFC Early Career Researchers Conference 2016

Conference Booklet

STFCBatteries.org 

The STFC Early Career Researchers Conference 2016

Welcome

Welcome to the ECRC 2016 at Cosener's House, Abingdon. The ECRC launched successfully last year and is organised by early career researchers, for early career researchers. It provides an opportunity for those in the early stages of their research career, PhDs and Post-Docs, to present and discuss their work in the area of electrochemical energy in an environment that might be less intimidating than some of the larger conferences. Presentations will cover the topics of electrochemical energy devices including fuel cells, batteries, super capacitors and electrolyzers. In addition, four plenary talks from researchers further along in their careers will provide insight, direction, and constructive input to conference delegates.

We hope you enjoy the conference and that the experience is useful and stimulating.

- *Rhodri, Thomas and Harini*



| Day 1: April 5th | | | | | | |
|-----------------------------|-------|-------------------------------------|------------------------|--|--------------------------|--|
| 10:30 | 00:30 | Registration Open | | | | |
| | | | | | | |
| 11:00 | 00:05 | Welcome Day 1 | | | | |
| Chair: Dr. Rhodri Jervis | | | | | | |
| 11:05 | 00:30 | Session 1a | Dr. Mirella Di Lorenzo | Miniature Biological Fuel Cells for Energy Harvesting and Sensing Applications | Fuel Cells | |
| 11:35 | 00:20 | | Anna Ploszajski | Freeze-Dried Ammonia Borane-Polyethylene Oxide Composites: Phase Behaviour and Hydrogen Release | | |
| 11:55 | 00:20 | | Tanveerkhan Pathan | Electrochemical Corrosion Analysis of Functionally Coated Metallic Bipolar Plates in PEM Fuel Cells | | |
| | | | | | | |
| 12:15 | 00:15 | Coffee | | | | |
| Chair: Dr. Ishanka Dedigama | | | | | | |
| 12:30 | 00:20 | Session 1b | Dr. Veronica Celorrio | Oxygen Reduction Reaction at $\text{La}_x\text{Ca}_{1-x}\text{MnO}_3$ Nanostructures: Interplay between A-site Segregation and B-site valence | | |
| 12:50 | 00:20 | | Kaipei Qiu | Highly Efficient Oxygen Reduction Catalysts via Nanoconfinement of Maghemite into a Nitrogen-Doped Graphene Framework | | |
| 13:10 | 00:20 | | Turgut Sönmez | Spinel Manganese-Cobalt Oxides As ORR Catalyst in Alkaline Media | | |
| | | | | | | |
| 13:45 | 01:10 | Lunch | | | | |
| Chair: Dr. Denis Kramer | | | | | | |
| 15:00 | 00:20 | Session 2a | Nick Farandos | Electrochemical Performance of 3D Inkjet Printed Solid Oxide Fuel Cell Microstructures | Modelling and Tomography | |
| 15:20 | 00:20 | | Kent Griffith | Understanding the Structural Evolution of Complex Oxides upon Electrochemical Lithiation | | |
| 15:40 | 00:20 | | Dr. Antonio Bertei | Physically-based modelling of the microstructure-performance correlation in SOFC anodes | | |
| 16:00 | 00:20 | | Joshua Stratford | Investigating Sodium Storage in Tin anodes for Sodium-Ion Batteries: A Combined Pair Distribution Function Analysis and Solid-State NMR Approach | | |
| | | | | | | |
| 16:20 | 00:10 | Coffee | | | | |
| Chair: Dr. Sam Cooper | | | | | | |
| 16:30 | 00:20 | Session 2b | Donal Finegan | Understanding battery failure: a multi-scale and high speed X-ray CT approach | | |
| 16:50 | 00:20 | | Moshiel Biton | Advanced 3D Imaging and Modelling of Dendrites in the Zn-Air System | | |
| 17:10 | 00:20 | | Joshua Bailey | Monitoring the microstructural evolution of the nickel phase in SOFC anodes | | |
| 17:30 | 00:30 | | Prof. Peter Lee | Synchrotron Imaging of Energy Materials | | |
| | | | | | | |
| 18:00 | 00:10 | Close and Thanks | | | | |
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| 18:30 | 01:00 | Poster Session and Drinks reception | | | | |
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| 19:30 | 01:00 | Dinner Followed by drinks | | | | |

| Day 2: April 6th | | | | | | |
|------------------------------------|-------|---------------------------------|-----------------------------|--|-------------------------------|--|
| 09:55 | 00:05 | Welcome Day 2 and Poster Winner | | | | |
| Chair: Dr. Shane Beatie | | | | | | |
| 10:00 | 00:20 | Session 3a | Prof. Qiang Zhang | Advanced lithium-sulfur batteries: The critical role of carbon | Materials | |
| 10:20 | 00:20 | | Cheng Tang | 3D Nanocarbon/LDH Hybrids for Excellent Oxygen Evolution Catalysis | | |
| 10:40 | 00:20 | | Ian Johnson | Continuous Hydrothermal Flow Synthesis: A route to high-power and high-energy nanomaterials for Li-ion batteries | | |
| 11:00 | 00:20 | | Dina Ibrahim Abou El Amaiem | Functionalized Cellulose-based Nanocomposite 'Papers' for Electrochemical Energy Conversion and Storage | | |
| | | | | | | |
| 11:20 | 00:10 | Coffee | | | | |
| Chair: Dr. Ana Belén Jorge Sobrido | | | | | | |
| 11:30 | 00:20 | Session 3b | Ronak Janani | A New Class of Ionogels | | |
| 11:50 | 00:20 | | Weixin Song | Dandelion-shape TiO ₂ /Multi-layer Graphene Composed of TiO ₂ (B) Fibrils and Anatase TiO ₂ Pappi Utilizing Triphase Boundaries for Lithium Storage | | |
| 12:10 | 00:30 | | Prof. Magdalena Titirici | Replacing Critical Elements from Energy Materials | | |
| | | | | | | |
| 12:50 | 01:05 | Lunch | | | | |
| Chair: Donal Finegan | | | | | | |
| 14:00 | 00:20 | Session 4a | Daniela Ledwoch | The Influence of Sodium Metal in Sodium Ion Half-Cell Testing Shown on Hard Carbon Anodes | Other Electrochemical Devices | |
| 14:20 | 00:20 | | Lukas Lutz | High capacity NaO ₂ batteries - Understanding the key parameters for solution-mediated discharge | | |
| 14:40 | 00:20 | | Dr. Patrick Cullen | Liquid phase charged graphene for engineering | | |
| | | | | | | |
| 15:00 | 00:10 | Coffee | | | | |
| Chair: Harini Hewa Dewage | | | | | | |
| 15:10 | 00:20 | Session 4b | Dr. Matthew Roberts | Understanding High capacity in Li-rich 3d cathode materials | | |
| 15:30 | 00:30 | | Dr. Ian Whyte | Flow Batteries: Design and Experience | | |
| | | | | | | |
| 16:00 | 00:10 | Close, Thanks and Talk Winner | | | | |

Keynote: Miniature Biological Fuel Cells for Energy Harvesting and Sensing Applications

Mirella Di Lorenzo

Department of Chemical Engineering, University of Bath, Bath, UK

Biological fuel cells (BFCs) are electrochemical devices that take advantages of biochemical pathways to generate electrical energy. The miniaturization of this technology opens up attractive and interesting opportunities in the field of energy harvesting and sensing.

Freeze-Dried Ammonia Borane-Polyethylene Oxide Composites: Phase Behaviour and Hydrogen Release

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c) Cella Energy, Building 148, Sixth Street, Thompson Avenue, Harwell Campus, Didcot, OX11 0TR

The demand for portable energy is ever increasing, and although lithium ion batteries currently lead the field, hydrogen-based systems can offer a lighter-weight, higher energy density, faster to re-fuel alternative. Many of the current portable hydrogen fuel cell systems - including the high-profile automobiles from Toyota, Honda and Hyundai - store hydrogen in high-pressure gas cylinders. However, solid-state materials can offer superior capacity, both volumetrically and gravimetrically, compared to gaseous systems, as well as an advantageous move away from high pressure storage. Hydrogen can be stored in solid materials both physically (adsorbed onto the surface of a highly porous material) and chemically (incorporated to make compounds in metal- and complex-hydrides). Ammonia borane (AB), a complex hydride, is the hydrogen storage material of choice for the systems designed by Cella Energy (**Fig. 1**), this work's industrial partner. AB is widely recognised as a leading candidate for commercial solid-state portable systems, yet its uptake has been hindered by issues of foaming during hydrogen release, additional impurity release and difficult physical handling in a working system.

To address these issues, our research has sought to nanostructure AB and use chemical interactions in a polymer composite; advantageous for being both low cost and lightweight. In this work, AB is combined with poly(ethylene oxide) (PEO) to make composites with great potential to impact the portable hydrogen storage arena (**Fig. 2**). These materials have been produced by freeze-drying from aqueous solutions across a full range of compositions from 0% to 100% AB by mass. The phase mixing behaviour of AB and PEO has been investigated using x-ray diffraction which shows that a new 'intermediate' crystalline phase exists, different from both AB and PEO, as observed in our previous work using co-electrospinning [1]. Hydrogen bonding interactions between the etheral oxygen atom (-O-) in the PEO backbone and the protic hydrogen atoms attached to the nitrogen atom (N-H) of AB molecules promote the formation of reaction intermediates, leading to lowered hydrogen release temperatures in the composites compared to neat AB. PEO also acts to significantly reduce the propensity for AB foaming during hydrogen release in a number of ways. A temperature-composition phase diagram has been produced for the AB-PEO system to demonstrate the relationship between the phase mixing behaviour and hydrogen release properties of these promising solid-state hydrogen storage materials.



Fig. 1: Part of Cella Energy's medium power system



Fig. 2: AB-PEO composites

[1] A. Nathanson, A. Ploszajski, M. Billing, J. Cook, D. Jenkins, T. Headen, Z. Kurban, A. Lovell, and S. Bennington, "Ammonia borane-polyethylene oxide composite materials for solid hydrogen storage," *J. Mater. Chem. A*, vol. 3, no. 7, pp. 3683–3691, 2015.

Electrochemical Corrosion Analysis of Functionally Coated Metallic Bipolar Plates in PEM Fuel Cells

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- b) Advanced Technology Institute, Faculty of Engineering and Physical Sciences, University of Surrey, Guildford, Surrey, GU2 7XH, UK
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A fuel cell is an electrochemical device that converts chemical energy of fuel directly into electrical energy via electrochemical reactions producing power with no pollution and very low environmental impact. The only by-products are heat and water. A bipolar plate (BPP) is a component in a fuel cell that separates individual cells, distributes fuel and oxidant, carries the load current to an external circuit and manages water produced in the cell. Traditionally, BPPs have been made up of graphite because of its low surface contact resistance and high corrosion resistance. However, graphite is expensive, brittle and permeable to gas and therefore ineffective for high volume manufacture. There is therefore a significant motivation for development of metallic bipolar plates. The major concern that still remains is of corrosion within the harsh fuel cell operating environment and the formation of passivation layers that reduce the electrical conductivity.

Here, we present the ex-situ interfacial contact resistance and corrosion-rate analysis of different grades of coated and uncoated steels under simulated fuel cell environment. The materials have been analysed by accelerated corrosion tests under simulated anodic and cathodic fuel cell conditions. This study has facilitated the understanding and development of corrosion resistant coatings for highly durable bipolar plates. Graphene possesses excellent chemical and mechanical strength; it is highly durable with excellent corrosion resistance and high electrical conductivity. Graphene was deposited on different grades of steel using chemical vapor deposition and electrophoretic deposition techniques. Contact resistance and corrosion tests performed on the coated samples are used to analyse the coating and help in achieving the optimum graphene coverage. We also present our results for in-situ corrosion testing in a working fuel cell for selected materials. Finally, all the materials have been analysed for their post-corrosion analysis using a suite of surface analysis techniques.

Keywords: PEM Fuel Cells, Bipolar Plates, SS 316L, Corrosion, Graphene.

Oxygen Reduction Reaction at $\text{La}_x\text{Ca}_{1-x}\text{MnO}_3$ Nanostructures: Interplay between A-site Segregation and B-site valence

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Efficiency in fuel cells is strongly linked to the performance of the electrocatalysts for the oxygen reduction reaction (ORR) at the cathode side, being Pt-based alloys the benchmark materials [1]. Transition metal oxides have been reported as active materials for ORR in alkaline media [2]. Although a number of high profile studies have proposed various descriptors for rationalizing the reactivity of TMOs in this context, a generally accepted understanding to rationally drive catalyst optimization has not yet emerged [3]. In this work, we probed that the ORR catalytic activity of transition metal oxides can be tuned by modifying the position of surface states. We shall demonstrate that by introducing Ca^{2+} into the structure of LaMnO_3 , e.g. $\text{La}_x\text{Ca}_{1-x}\text{MnO}_3$, not only the surface chemistry is changed, but also the Mn oxidation state. Our investigation reveals that by altering the redox surface states position, the ORR onset potential can be displaced.

$\text{La}_x\text{Ca}_{1-x}\text{MnO}_3$ nanoparticles were synthesized via an ionic liquid method. Surface composition studies by XPS show that A-site segregation to the surface is favoured by the presence of La^{3+} ions. Mn oxidation state was investigated by ex-situ EXAFS analysis. The electrochemistry study of these oxides on an Ar-saturated solution, provide conclusive evidence of the position of the redox states, shifting to more negative potentials with the increase in the Mn oxidation state, as observed from Figure 1. The RRDE studies in the presence of O_2 reveal that the onset potential for ORR closely matches with the position of the redox surface states. These observations provide the clearest indication that oxygen bond breaking requires changes of oxidation state within a range close to the reversible oxygen potential. Although all the $\text{La}_x\text{Ca}_{1-x}\text{MnO}_3$ oxides perform the $4e^-$ ORR pathway, the current density at 0.85 V decreases with the increase of the Mn oxidation state, identifying the reduced state of Mn as the most active specie for ORR.

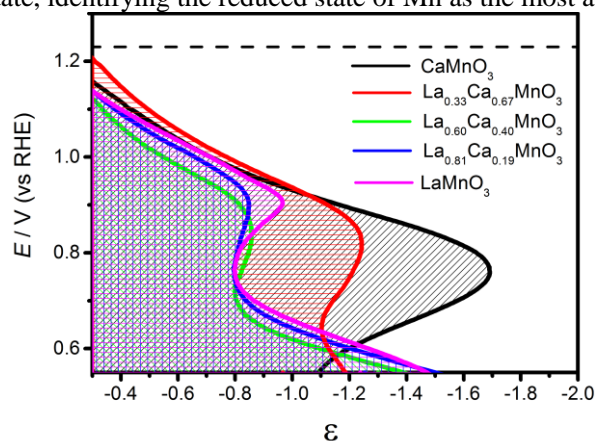


Figure 1. Derivative of change in cathodic charge normalized by the total cathodic charge as a function of potential for the $\text{La}_x\text{Ca}_{1-x}\text{MnO}_3$ electrodes.

[1] Chem. Res. 46 (2013) 1848-1857.

[2] Energ. Environ. Sci. 4 (2011) 114-130.

[3] ChemElectroChem 3 (2016) 283-291; Nat. Chem. 3 (2011) 647-651; Chem. Sci. 4 (2013) 1245-1249; Angew. Chem. Int. Ed. 47 (2008) 4683-4686; Energy Environ. Sci. 8 (2015) 1404-1427.

Highly Efficient Oxygen Reduction Catalysts via Nanoconfinement of Maghemite into a Nitrogen-Doped Graphene Framework

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Oxygen reduction reaction (ORR) is critical for electrochemical energy storage and conversion, e.g. in fuel cells and metal-air batteries [1]. A major challenge is to develop cost-effective and durable ORR catalysts, so as to replace relatively expensive platinum loaded carbon (PtC) counterpart, particularly for large-scale applications. Despite progresses over the past decades in developing efficient non-precious metal (NPM) catalysts, such as Fe/N/C based materials (the best-known alternatives), most of the reported catalytic activities are yet to match that of PtC [2].

Recognizing the challenge, herein we propose a two-step process for the production of highly efficient NPM catalysts that outperform PtC in alkaline media: 1) a hierarchical porosity of a supporting substrate is generated and optimized in advance, especially to achieve a high total pore volume for rapid mass transfer; and 2) an appropriate amount of NPM precursor is added into the optimized substrate to boost the reduction potential while maintaining the hierarchically porous structure.

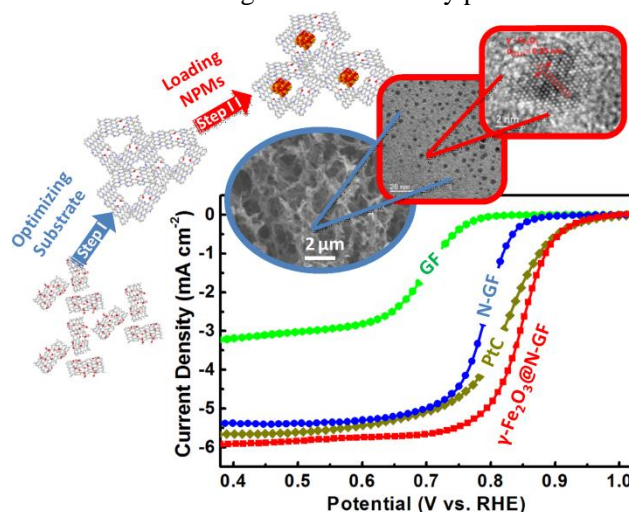


Figure 1. Proposed two-step design principle for highly efficient ORR catalysts.

Such a scheme was successfully applied to a case of nanoconfined maghemite ($\gamma\text{-Fe}_2\text{O}_3$) in a nitrogen-doped graphene framework (Figure 1). The resulting catalyst system surpasses the performance of the equivalent commercial PtC, in terms of a higher reduction potential, a significantly lower peroxide formation ratio, more than tripled kinetic current density, smaller Tafel slope and better durability etc. The reported catalyst is also among the best of all the existing Fe based ORR catalysts, indicating the great potential of $\gamma\text{-Fe}_2\text{O}_3$ for ORR in practical applications.

[1] Ge, X.; Sumboja, A.; Wu, D.; An, T.; Li, B.; Goh, F. W. T.; Hor, T. S. A.; Zong, Y.; Liu, Z. *ACS Catal.*, **2015**, 5 (8), 4643–4667.

[2] Garsuch, A.; Bonakdarpour, A.; Liu, G.; Yang, R.; Dahn, J. R. In *Handbook of Fuel Cells*, John Wiley & Sons, Ltd: 2010.

Spinel Manganese-Cobalt Oxides ($\text{Mn}_x\text{Co}_{3-x}\text{O}_4$, $0 \leq x \leq 2.0$) As ORR Catalysts

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The oxygen reduction reaction (ORR) has a paramount importance in energy conversion and storage technologies such as fuel cells and metal-air batteries.¹ The electrochemical reduction kinetics of oxygen are sluggish in nature, which requires excessive amounts of noble-metals (Pt-based)[1] to speed up the reaction kinetics. However, the high cost, poor durability and scarcity of platinum remain as a big obstacle to these clean-energy technologies becoming available commercially[2]. Manganese doped spinel cobalt oxide ($\text{Mn}_x\text{Co}_{3-x}\text{O}_4$) has been considered as one of the most promising non-platinum catalysts for ORR in alkaline conditions, due to its high activity, corrosion stability, low cost, availability and simple preparation[2, 3].

It is important to understand the effect of dopant on the catalytic activity of spinel Co_3O_4 towards ORR behavior in alkaline electrolyte. Therefore, the aim of this study is to understand how the composition of the spinel affects the electrocatalytic behavior, especially for the ORR reaction. A series of manganese doped cobalt oxides ($\text{Mn}_x\text{Co}_{3-x}\text{O}_4$, $x = 0.0, 0.5, 1.0, 1.5$ and 2.0) were produced via thermal decomposition method and characterized using Thermogravimetric Analysis (TGA), X-Ray Diffraction (XRD), Raman Spectroscopy, Brunauer-Emmett-Teller (BET), Scanning Electron Microscope (SEM) and Energy-Dispersive X-ray Spectroscopy (EDX). The electrochemical behaviors were also tested using coated Rotating Disk Electrode (RDE) and Rotating Ring Disk Electrode (RRDE). The MnCo_2O_4 catalyst shows the greatest activity with the lowest onset potential (see Fig.1). It has been found that the atomic ratio of Mn influences the crystal phase (cubic vs. tetragonal), which exhibits different ORR activities and onset potentials. Intensive electrochemical analyses show that the dopant has an enormous effect on the catalytic behavior of spinel cobalt oxide.

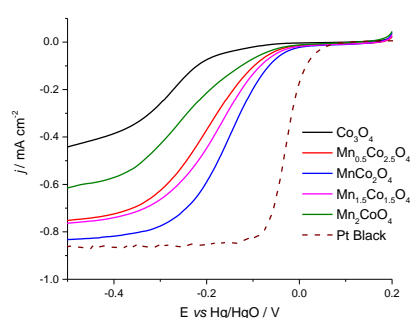


Fig.

1. ORR behavior of $\text{Mn}_x\text{Co}_{3-x}\text{O}_4$ ($x = 0, 0.5, 1.0, 1.5, 2.0$) in 1 M KOH purged with O_2 (30 mins); 298 K; 5 mV s⁻¹ scan rate; 100 RPM. Pt black is shown for comparison purpose.

- [1] F. Y. Cheng, J. A. Shen, B. Peng, Y. D. Pan, Z. L. Tao, and J. Chen, "Rapid room-temperature synthesis of nanocrystalline spinels as oxygen reduction and evolution electrocatalysts," *Nature Chemistry*, vol. 3, pp. 79-84, Jan 2011.
- [2] Y. Liu, Y. Wang, X. Xu, P. Sun, and T. Chen, "Facile one-step room-temperature synthesis of Mn-based spinel nanoparticles for electro-catalytic oxygen reduction," *Rsc Advances*, vol. 4, pp. 4727-4731, 2014 2014.
- [3] V. Nikolova, P. Iliev, K. Petrov, T. Vitanov, E. Zhecheva, R. Stoyanova, *et al.*, "Electrocatalysts for bifunctional oxygen/air electrodes," *Journal of Power Sources*, vol. 185, pp. 727-733, Dec 1 2008.

Electrochemical Performance of 3D Inkjet Printed Solid Oxide Fuel Cell Microstructures

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The electrochemical performance of solid oxide fuel cells (SOFCs) can be increased primarily by increasing the density of active triple phase boundaries (TPBs, located at the interface of electrode, electrolyte and pore phases), where reactions occur. SOFC composite electrodes with infiltrated scaffold structures have been shown to have greater TPB densities compared with structures fabricated by conventional power-mixing methods;^[1] however, methods to reproducibly fabricate scaffolds with pre-determined architectures and minimal processing steps is a challenge. Hence, we employ a Ceradrop X-Series 3D inkjet printer to fabricate yttria-stabilised zirconia composite electrode scaffolds with ca. 5 μm resolution, prior to sintering. Printed geometries will be presented,^[2] along with preliminary electrochemical results of printed planar electrolyte structures.^[3] The results of finite element models for potential and secondary current distributions within realistic 3D structures will be presented (**Figure 1**).

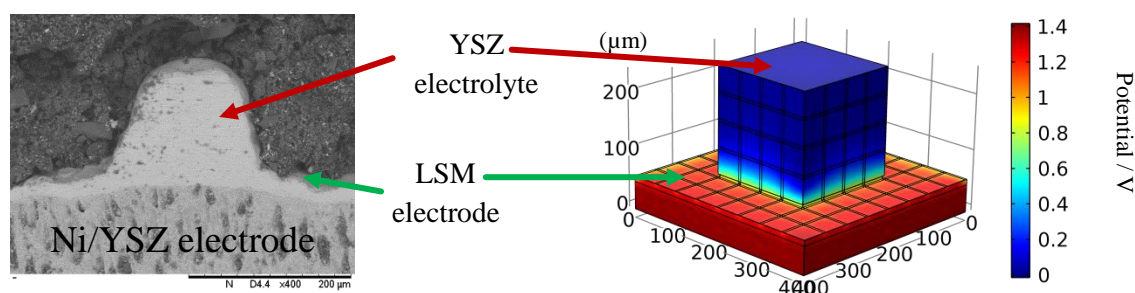


Figure 1. 3D printed yttria-stabilized zirconia (YSZ) pillar coated in lanthanum strontium manganite (LSM) and modelled potential distribution within the pillar.

[1]. Kishimoto M., Lomberg M., Ruiz-Trejo E., Brandon N. P. Enhanced triple-phase boundary density in infiltrated electrodes for solid oxide fuel cells demonstrated by high-resolution tomography. *J Power Sources*. 2014;266:291-5.

[2] Farandos N. M., Kleiminger L., Li T., Petit C., Kelsall G. H. 3D Inkjet Printed Ytria-Stabilized Zirconia Microstructures. *J. Eur. Ceram. Soc.* Submitted Feb. 2016.

[3] Kleiminger L., Farandos N. M., Li T., Hankin A., Kelsall G. H. 3-D Inkjet Printed Solid Oxide Electrochemical Reactors. I. YSZ Electrolyte. *Electrochim. Acta*. Submitted Feb. 2016.

Understanding the Structural Evolution of Complex Oxides upon Electrochemical Lithiation

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Energy storage materials with both high capacity and high charge/discharge rate enable applications that require long life, high power, and rapid recharge. Electric double-layer capacitors offer high power and conventional battery electrodes offer relatively high capacity but the combined properties require advanced materials. While nanostructures have dominated this emerging field, there are well-known issues regarding cost, stability, scalability, and safety of nanoparticles for battery applications. Recently, complex oxide structures from facile solid state synthetic methods have shown promising rate, capacity, and stability for reversible lithium intercalation. Complex structure types with low symmetry, large unit cells, site disorder, superstructure, order–disorder transitions, local second–order Jahn–Teller distortions, and mobile lithium atoms create severe challenges and interesting opportunities for experimental and computational investigations. Herein, we discuss the structure and properties i.) crystallographic shear compounds (e.g. TiNb_2O_7) and ii.) tungsten bronze-type phases (e.g. $\text{T-Nb}_2\text{O}_5$). Electrochemical results are interpreted with insights from $^6/7\text{Li}$, ^{93}Nb , and ^{17}O solid state nuclear magnetic resonance, in situ x-ray absorption, in situ x-ray diffraction, bond valence sum mapping, and density functional theory. There is a focus on understanding these important structure families and the also applicability of advanced characterization techniques to complex functional oxides.

Physically-based Modelling of the Microstructure-Performance Correlation in SOFC Anodes

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Imperial College London, Department of Earth Science and Engineering

The performance and durability of solid oxide fuel cells (SOFCs) are strictly related to the microstructure of the electrodes. The optimization of the electrode design can be addressed with physically-based models, capable to correlate the microstructural properties to the electrochemical performance. However, before suggesting optimization strategies, models must be carefully validated by using ad hoc experimental data.

This contribution focuses on the validation of a physically-based model for SOFC composite anodes. The model is used to reproduce the experimental impedance spectra in different operating conditions, enabling the fitting of the unknown parameters of the model (Figure 1). The sensitivity of fitted parameters is discussed and their values are compared with independent sources in order to assess their soundness. The capability of the model to correlate the electrode microstructure to electrochemical performance is corroborated by analysing samples prepared with different fabrication processes or degraded in different conditions, with microstructural information obtained through 3D tomography.

The study offers an opportunity to discuss about the best strategies for model validation comprising the choice of the optimal techniques and conditions that provide the best information, the verification of model assumptions and how to deal with experimental uncertainties.

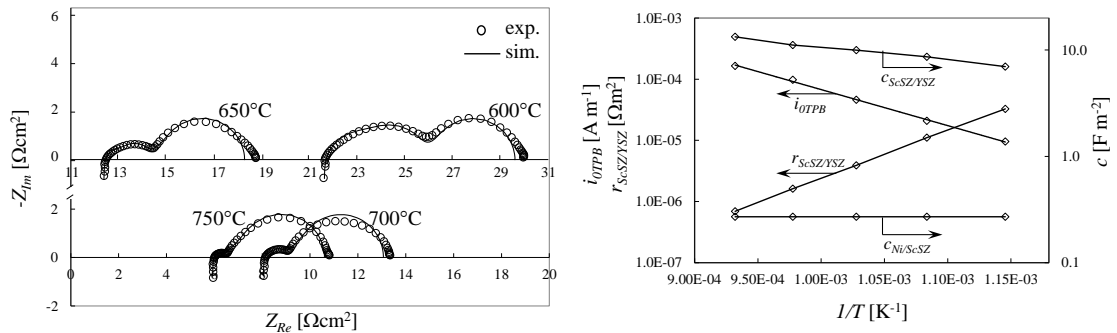


Figure 1. Fitting of impedance spectra for different temperatures (left) and Arrhenius plot of the fitted parameters (right).

Investigating Sodium Storage in Tin Anodes for Sodium-Ion Batteries: A Combined Pair Distribution Function Analysis and Solid-State NMR Approach

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O. J. Borkiewicz^d, K. M. Wiaderek^d, K. W. Chapman^d, P. J. Chupas^d,
A. J. Morris^b and C. P. Grey^a*

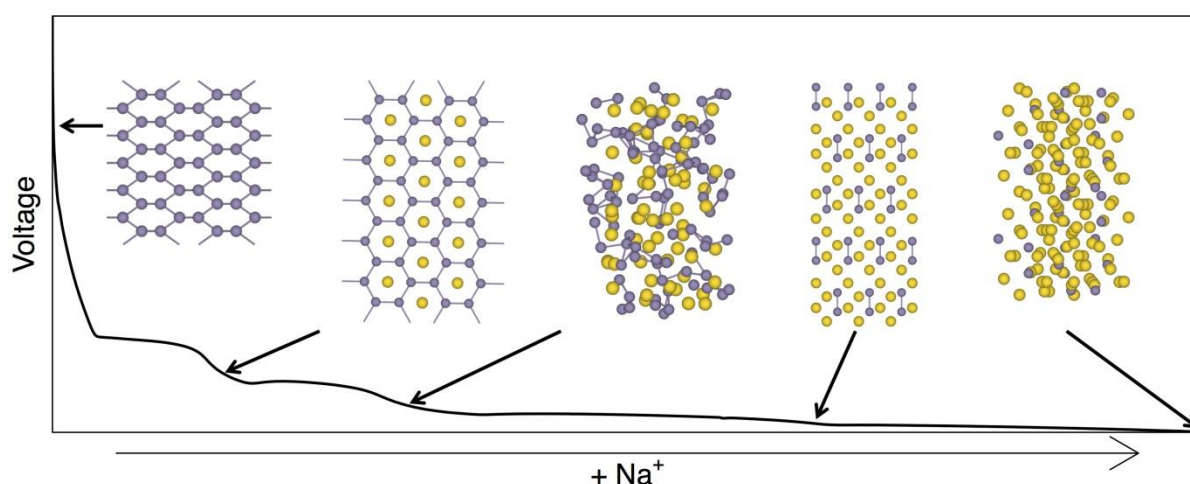
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b) Cavendish Laboratory, University of Cambridge, J. J. Thomson Avenue, Cambridge, CB3 0HE, U.K.

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d) X-ray Science Division, Advanced Photon Source, Argonne National Laboratory, Argonne, Illinois 60439, USA

Sodium-ion batteries are currently being widely investigated, particularly for grid storage applications, owing to their potential to be much cheaper than existing lithium-ion technology. However, as sodium ions are too large to intercalate into graphite, the anode of choice for commercial lithium-ion batteries, there is an ongoing challenge in finding a suitable alternative material. Here we investigate the alloying mechanism of high-capacity tin anodes for sodium-ion batteries through the use of in-situ pair distribution function analysis (PDF), ^{23}Na solid-state nuclear magnetic resonance (ssNMR) and ex-situ ^{119}Sn magic-angle spinning ssNMR. Through refinement of predicted structures using real-space and reciprocal-space diffraction data, combined with further local structural information gathered by ssNMR, we have identified two previously unknown crystalline intermediates, and one amorphous intermediate. Upon initial sodiation, crystalline tin is converted into crystalline NaSn_2 , a layered structure consisting of sodium atoms intercalated between planar layers of tin atoms. This is then broken down into a highly amorphous structure of approximate composition NaSn , which retains some of the tin-tin connectivity of the previous phases. Further sodiation results in the formation of Na_2Sn , a phase consisting of sodium atoms between tin-tin dumbbells. Finally $\text{Na}_{15}\text{Sn}_4$, consisting of isolated tin atoms surrounded by sodium atoms, is formed.

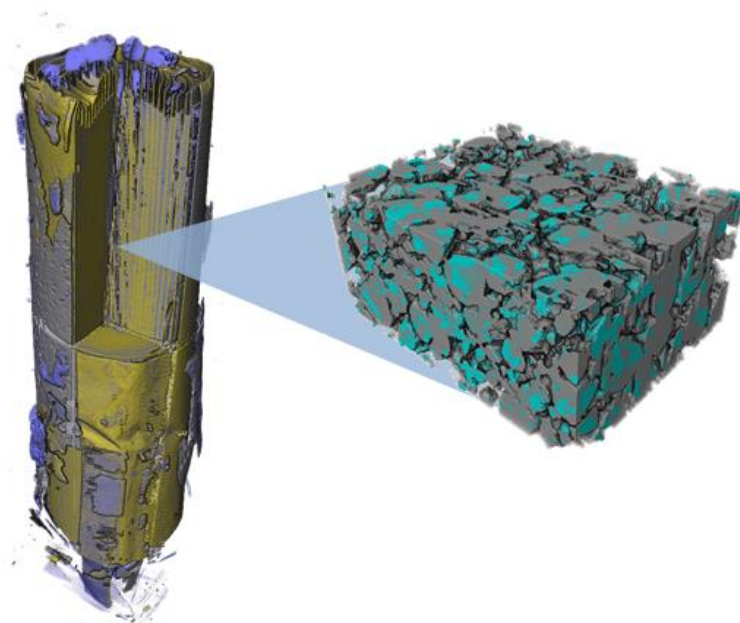


Understanding battery failure: a multi-scale and high speed X-ray CT approach

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Li-ion batteries are an integral technology in the process of achieving a clean and sustainable energy future. The safety of Li-ion batteries is of utmost importance in particular for demanding applications such as electric vehicles and other mission critical systems. The thermal response of a cell is one of the most important characteristics to understand when assessing the safety of a cell design. However, there is limited understanding of the dynamic mechanisms associated with thermally induced failures. X-ray tomography has become a widely used technique for 3D structural analyses of electrochemical materials. Two of the major advances in tomography techniques in recent years are the reduction in tomogram acquisition time and the increased spatial resolution. In this study, high speed synchrotron X-ray CT of commercial Li-ion batteries during operation and failure was performed. Tomograms were captured at a rate of up to 2.5 Hz allowing us to study some of the most rapid failure mechanisms including those associated with thermal runaway in 3D. From millimetres to nanometres, multi-scale post-mortem tomography analyses of failed battery materials reveal a large degree of structural degradation from the cell scale down to the particle scale. Features which may be indicative of temperatures reached and reaction pathways during failure are identified via inspection of electrode microstructures. For example, surface layers of transition metals on electrode particles are seen; suggesting numerous exothermic reduction steps in the presence of electrolyte. Significant changes in particle morphology are also observed which can affect the rate of heat generation and onset temperatures of thermal runaway. This combined high-speed and multi-scale tomography approach uses cutting-edge imaging techniques to put a spotlight on failure mechanisms which were previously unexplored. New insights into the structural and thermal dynamics leading up to and during thermal runaway and failure are achieved. The thermal response and catastrophic outcomes observed on the macro scale are linked to the structural properties of active materials on the micro-scale, revealing scope for further investigations on the impact of particle morphology on failure mechanisms of Li-ion cells.



Advanced 3D Imaging, Analysis and Characterisation of battery materials

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The performance of the battery is dependent on the nano and micro-structure achieved during manufacture. Furthermore microstructural evolution during operation may degrade electrochemical performance. The growth of dendrites represents a limiting failure mechanism in some battery systems; in particular this can be a challenge in zinc-air batteries. Furthermore volume expansion during lithiation is another major failure mechanism. Tomographic techniques allow the direct 3D imaging and characterisation of complex microstructures, including the observation and quantification of dendrite growth and volume expansion.

Here we present results from 3D x-ray and FIB-SEM tomography of Zn dendrite formation in a zinc-air battery, down to resolutions of tens of nanometers, enabling analysis of complex micro-structures.

This approach is shown to be effective in understanding how dendrites grow and demonstrates that tomography coupled with modeling can provide new insights into degradation mechanisms associated with dendrite growth.

Moreover a new methodology of contrast enhancement for multi modal 3D imaging, including novel advanced quantification, on a commercial Lithium Iron Phosphate (LFP) LiFePO_4 cathode. This enabling higher FIB-SEM resolution (3D imaging), which is amongst the highest ever reported for carbon containing electrode materials (e.g. composite LFP cathodes) using FIB-SEM. In turn it means that the particles are well defined and the size distribution of each phase can be analysed accurately from the complex 3D electrode microstructure using advanced quantification algorithms.

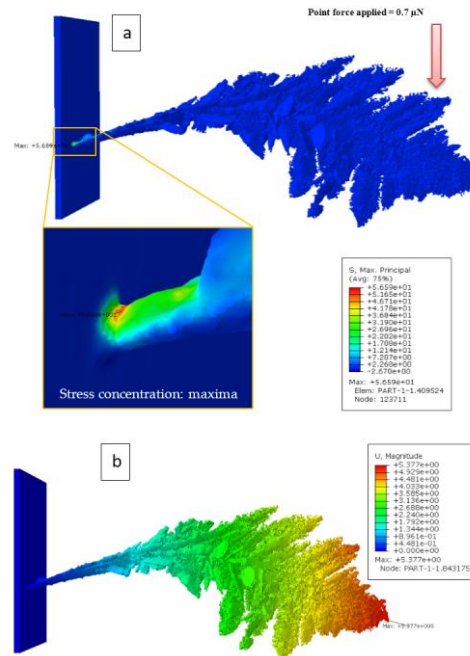


Figure 1. (a) Maximum principal stress (unit=100MPa), the maximum stress at the neck = 5.659×10^2 MPa, which is just above the fracture strength (500 MPa). (b) Displacement (unit=0.1 μm), the maximum displace is 0.538 μm at the top of the dendrite.

Characterisation of the Microstructural Evolution in SOFC Anodes

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Solid oxide fuel cells (SOFCs) used in stationary applications require an operational lifetime of 40,000 hours to be commercially viable. Aside from various degradation pathways in other components, the fuel electrode is known to undergo many different changes over a wide range of time and length scales. The fuel electrode is traditionally a porous composite of nickel and yttria-stabilised zirconia, providing electron and ion-conducting phases, respectively. The one-dimensional interface between these phases, and the porosity that allows for gas flow in and out of the electrode, is known as the triple-phase boundary (TPB), which is surmised to be where electrochemical fuel oxidation occurs. However, due to high temperature operation ($>800^\circ\text{C}$), over time a phenomenon known as nickel agglomeration takes place. This degradation pathway is a thermally-induced capillarity effect wherein nickel grains have a tendency to coarsen, leading to a loss both in electronic percolation [1] and in TPB density [2, 3], limiting the current feasibility of SOFCs for long-term use. The phenomenon has not yet been completely understood, neither in terms of the degree of TPB loss associated solely with nickel agglomeration, nor the relationship between operating time and the electrochemical

performance loss.

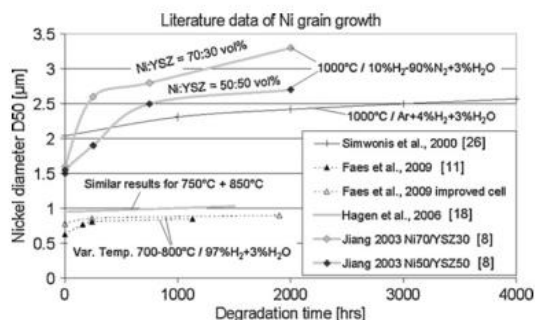


Figure 1. Literature degradation rates

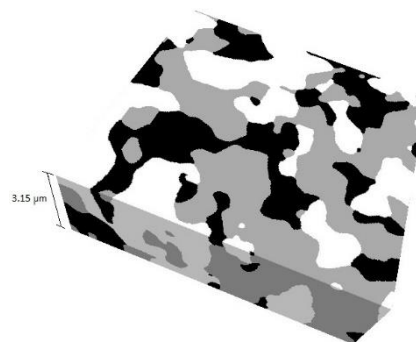


Figure 2. FIB-SEM tomographic reconstruction (Ni = white, YSZ = grey, Pore = black)

This drop in performance appears to vary widely and is susceptible to a whole host of parameters, including the original composition and microstructure, humidity of fuel gas, temperature and the level of other impurities present. The variability in growth rates to date can be seen in Figure 1 [4]. Moreover, a detailed understanding of the mechanism of this grain growth at the atomic level is also lacking.

Through a combination of lab-based X-ray computed tomography, focussed-ion beam scanning electron microscope (FIB-SEM)-based tomography (Figure 2), and mechanistic annealing studies, this work aims to uncover some of the missing links between the aforementioned parameters and nickel agglomeration in long-term operation of SOFC anodes. It focusses on accelerated testing, monitoring morphological changes by imaging techniques and combining these with real-time electrochemical measurements. Furthermore, there is a range of models in the literature that attempt to simulate the microstructural impact of the nickel's tendency to reduce its surface area as well as attempting to provide a link between this microstructural evolution and the concomitant loss in electrochemical performance. The aim is to compare experiment with these theoretical accounts to understand and therefore predict the lifetime of the anode in terms of this type of degradation, at the same time as identifying potential mitigation strategies.

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Keynote: Synchrotron Imaging of Energy Materials

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A summary of the work carried out at the Manchester X-ray Imaging Facility and the Research Complex at Harwell.

Advanced lithium-sulfur batteries: The critical role of carbon

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With the commercial application of graphite anode, lithium-ion batteries (LIBs) are extensively applied in numerous portable devices such as smartphones and laptops. However, current LIBs based on conventional intercalation mechanism cannot meet the requirements for the electronic industry and electric vehicles yet. Therefore, it is extremely urgent to seek for the systems with high energy density. Among various promising candidates with high energy densities, lithium-sulfur (Li-S) batteries with a high theoretical capacity and energy density are highly attractive;¹⁻² while the commercial application of Li-S batteries still faces some persistent obstacles, such as the low electrical conductivity of sulfur and lithium sulfide and the dissolution of polysulfides. The introduction of nanocarbon materials into the field of Li-S batteries sheds a light on the efficient utilization of sulfur by improving the conductivity of the composites and restraining the shuttle of polysulfides. In this presentation, the recent progress of the carbon/sulfur composite materials, especially carbon nanotube, graphene, porous carbon and hybrid materials in my research group will be reviewed.³⁻¹⁰ New insights on the relationship between the structure and the electrochemical performance, and proposed some prospects on the future development of Li-S batteries.

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3D Nanocarbon/LDH Hybrids for Excellent Oxygen Evolution Catalysis

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Oxygen evolution reaction (OER) has attracted tremendous explorations in both fundamental and application fields recently, due to its core status in next-generation energy conversion and storage technologies, such as water splitting and metal-air batteries. Transition metal-based compounds, especially the NiFe layered double hydroxides (NiFe LDHs) have been well-established as the most high-effective and cost-efficient electrocatalysts to boost the sluggish water oxidation and improve the energy efficiency. Nevertheless, a favourable substrate is highly required to expose the poorly conductive active phases and enhance reactivities of OER. Nanocarbon (NC) materials are promising candidates for a strongly coupled hybridization with LDHs towards superior OER performances. Herein, we proposed a series of 3D NC/LDH hybrids for OER, with a systematic investigation of the relationship between structure and properties and the underlying mechanism (**Figure 1**).

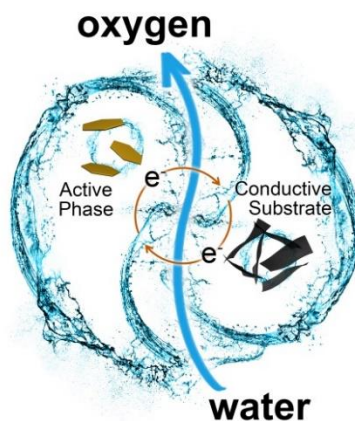


Figure 1. The wise integration of highly active phases (LDH) and multi-functional conductive substrates (NC) for superior OER catalysts.

On one hand, the highly conductive NC materials can help to connect active phases effectively. we found that a thin layer of graphene deposited on the nickel foam (NF) in advance can provide a more hydrophobic surface and modulate the growth of NiFe LDHs with better dispersion. The graphene connected all LDH flakes with a strong coupled interface (**Figure 2a**).^[1] Therefore, a high utilization efficiency of active phases was achieved, resulting in enhanced activity with a low overpotential of 325 mV for 10.0 mA cm⁻² in 0.10 M KOH. Furthermore, the carbon nanotubes (CNTs) grown out from the nanoparticles on LDH flakes were demonstrated to connect all the active sites into the conductive network *via* “point-line-point” contacts, thereby giving rise to a more effective catalyst, as illustrated in **Figure 2b**.^[2]

On the other hand, the NC scaffold can also regulate the structure of LDHs and construct a 3D interconnected active framework. A nitrogen-doped mesoporous graphene framework grown on mesoporous MgO templates was scrupulously designed to hybridize nanosized NiFe LDHs with an *in-situ* defect-anchored nucleation and spatially confined growth, leading to a uniformly decorated nano-sized active particles and strong coupled interface between active phase and conductive substrate (**Figure 2c and d**). The nNiFe LDH/NGF composite was demonstrated to overperform commercial Ir/C catalysts and compete favorably against reported alternatives for high-performance OER catalysis (**Figure 2e**).^[3] This spatially confined strategy can also be generalized to other NC materials, such as G/CNT hybrids.^[4] Additionally, based on this novel material platform, the different roles of Ni/Fe were probed. With the Fe concentration increasing, the as-obtained electrocatalysts evolved from well-crystallized hydroxides (α -Ni(OH)₂ and NiFe LDHs) to amorphous oxyhydroxides (Ni substituted FeO(OH) or FeO(OH)

polymorph). The moderate guest metal substitution into the host oxyhydroxide framework (Fe into Ni or Ni into Fe) substantially enhanced the OER activity with a decrease of both the Tafel slope and overpotential (**Figure 2f**).[5]

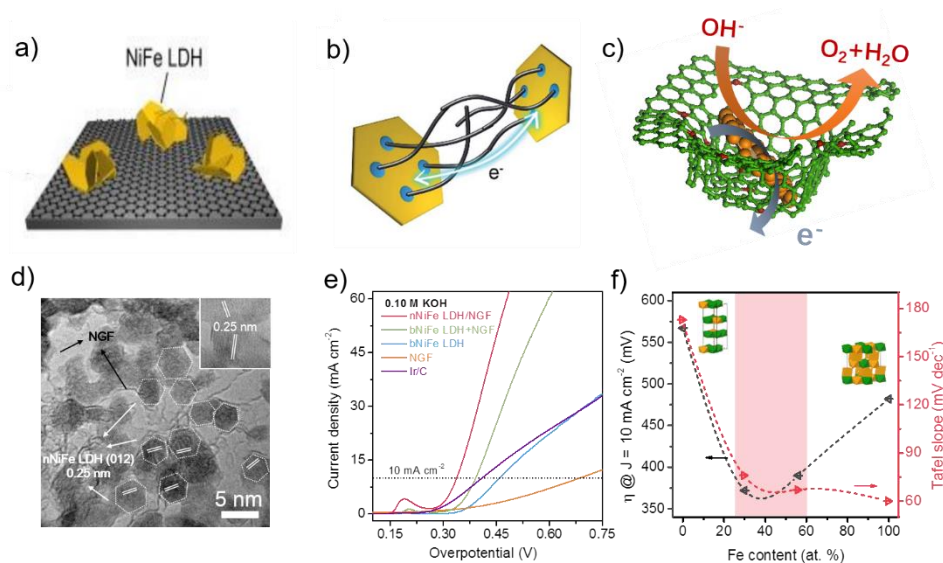


Figure 2. a) Scheme of LDH/G/NF hybrid catalyst. b) Scheme of the “point-line-point” contacted LDH/CNT hybrids. c) Schematic of the spatially confined hybrids. d) TEM image and high-resolution TEM image (inset) of nNiFe LDH/NGF electrocatalysts. e) LSV curves in 0.10 M KOH electrolyte. f) OER performance as a function of Fe content in consideration of both the activity (overpotential required to achieve 10.0 mA cm⁻²) and the kinetics (Tafel slope).

These advances are expected to provide stimulatory knowledge and shed fresh light into the development of advanced functional materials with a wise hybridization of active phases and conductive substrates.

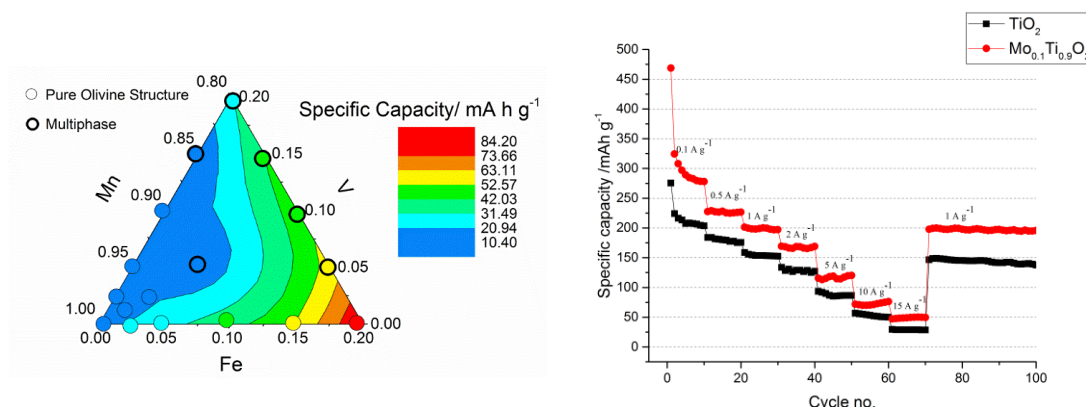
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Continuous Hydrothermal Flow Synthesis: A route to high-power and high-energy nanomaterials for Li-ion batteries

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Continuous Hydrothermal Flow Synthesis (CHFS) is a scalable, low temperature technique for producing high surface area and surface-functionalised nanomaterials. Several materials, such as doped TiO_2 , Nb_2O_5 , VO_2 , LiFePO_4 and LiMnPO_4 have been generated using this technique, and have all shown high power and/or high energy capability within Li-ion batteries [1-5]. Of great interest is the pseudocapacitive energy storage mechanism in the very high surface area (up to $300 \text{ m}^2 \text{ g}^{-1}$) anode materials (TiO_2 , Nb_2O_5 and VO_2) where the high rate capability is a result of fast Faradaic reactions responsible for charge storage near the surface [1-4]. On the cathode side, dopants within LiFePO_4 and LiMnPO_4 have been assessed combinatorically to assess their effect, with clear trends observed throughout, leading to improved rate capability for both materials [5].



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Functionalized Cellulose-based Nanocomposite ‘Papers’ for Electrochemical Energy Conversion and Storage

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With the rapid development of the global economy, increasing environmental pollution and the depletion of fossil fuels, there is a vital need for clean, sustainable and efficient sources of energy as well as new technologies allied with energy conversion and storage [1]. Among many application fields, some of the most practical and efficient technologies for electrochemical energy conversion and storage are fuel cells, batteries and electrochemical supercapacitors. In recent years, these devices have attracted significant attention, each with recognized advantages. Driven by this need and the promise of the technology, significant progress in practical and theoretical research and development of these devices has taken place.

One of the challenges of developing these electrochemical energy conversion and storage technologies is the use of low cost and readily available materials that possess complex requirements of different applications. To overcome obstacles of high costs of raw materials and avoid usage of depleting sources, bio-based carbon materials are believed to lead the next generation of many industries including aerospace, sports equipment and electrochemical devices due to their abundance, easy processing, high chemical stability, electrical conductivity, low cost, non-toxicity, high specific surface area and wide operating temperature range [2]. Cellulosic fibers in nano and micro scale, the green and most abundant material, have eco-friendly attributes that are economically and technically feasible to replace man-made fibers. Cellulose-based nanocomposites are being reinforced recently to make environmental friendly green products including adhesives, lithium-ion batteries, electrode materials for supercapacitors and catalyst support in different electrochemical energy devices [3]. Carbonization of cellulose yields carbons, including charcoal, activated carbon and graphite fibers. The process comprises of introducing the fibers in an inert atmosphere, preheating and drying the fibers, treating the dried fibers up to a certain temperature at which they carbonize by evolution of a purging gas and finally cooling the carbonized residues [4].

In this project, bio-based cellulosic starting materials have been investigated as a candidate for supercapacitor electrode materials. The produced activated carbon materials upon carbonization of the cellulose fibers have been functionalized and characterized by different techniques to study the effect of the morphology and surface area of the carbonaceous residues on their performance in the electrochemical device. This work has been coupled with a range of electrochemical tests in two and three-electrode systems including cyclic voltammetry, electrochemical impedance spectroscopy and charge-discharge loop tests. The work highlights the importance of relating the different characterization techniques of the raw and produced materials and the effect of each on the performance of the activated carbons as electrode materials in supercapacitors.

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A new class of ionogels

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Liquid electrolytes (aqueous, organic and ionic liquids) have been widely utilized in supercapacitors and batteries due to their high ionic conductivity and transference number. However, safety concerns resulted mainly by their leakage and (in some cases) explosive nature, caused an extensive call for research on the development of solid electrolytes.

Ionogels are ionic liquids encapsulated inside a quasi-solid/solid matrix. The in-situ sol-gel technique is one of the most commonly used routes for synthesis of ionogels. Although the ability of facile sol-gel process in formation of quasi-solid to solid structures has been studied deeply in the past, the potential of inorganic silica-based ionogels as a high performance solid electrolyte requires more investigation [1, 2]. Furthermore, the possibility of mechanical and electrochemical enhancements of ionogels through application of both organic and inorganic compounds needs to be explored. The present work describes development of hybrid ionogel electrolytes based on confinement of 1-ethyl-3-methylimidazolium trifluoromethanesulfonate ionic liquid inside an organic-inorganic matrix. This new type of ionogel was created using a straightforward sol-gel process with a novel formulation of reactants, resulting in a high-performance solid electrolyte. Leakage-free electrochemical double layer capacitors were assembled by sandwiching the solid ionogel between two activated carbon electrodes. Figure 1 is the SEM image of hybrid ionogel-casted carbon electrode (cross section). As it is evident, ionogel has deeply penetrated inside the activated carbon while no sign of ionic liquid leakage is observed. The mechanically compliant ionogels described herein show better confinement of ionic liquid together with stronger mechanical structure. The electrochemical properties of the hybrid ionogels (including charge-discharge cyclic behaviour and frequency response) were characterized. The dynamic mechanical analysis results confirm an improvement in mechanical structure of ionogels while the electrochemical characterization results show comparable properties to those of conventional TEOS-based ionogels.

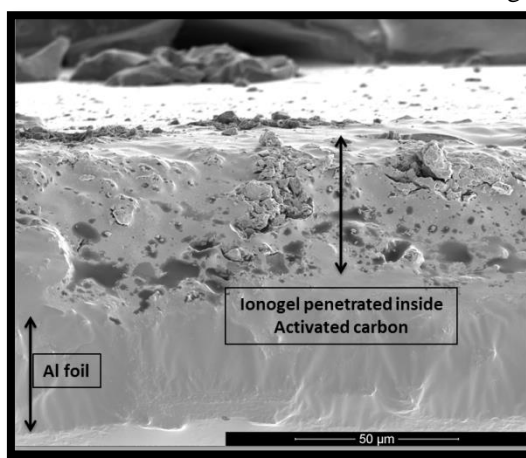


Figure1. SEM image of all solid-state hybrid ionogel coated on an activated carbon electrode. Evidently, ionogel has penetrated deeply into the coated activated carbon.

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Dandelion-shape TiO₂/Graphene Stores Lithium via Triphase Boundaries

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Titanium dioxide (TiO₂), one of the most promising anode materials for lithium-ion batteries, has been extensively studied, not only due to its low cost, abundance and non-toxicity but also on account of the negligible volume deformation during the Li-ion intercalation/de-intercalation processes and excellent gravimetric capacity ^{1, 2}. Charge stored in a Li⁺/heterophase anode system may be enhanced ³⁻⁵ through interfacial charge storage at both the solid-liquid interface and internal solid-solid interfaces. Jamnik and Maier have demonstrated increased charge storage at TiO₂-metal interfaces as a result of the metal acting as an electron sink and the TiO₂ storing excess Li⁺. In TiO₂ photochemical studies, it has been demonstrated that owing to the offset in the conduction band levels, photogenerated charge carriers can be stored at the junction between anatase and TiO₂(B)^{6, 7}. This band offset has been employed in TiO₂ engineering to yield a material in which Li⁺ and electrons are separated across TiO₂(B)/anatase TiO₂ interfaces within the anode leading to increased charge storage.

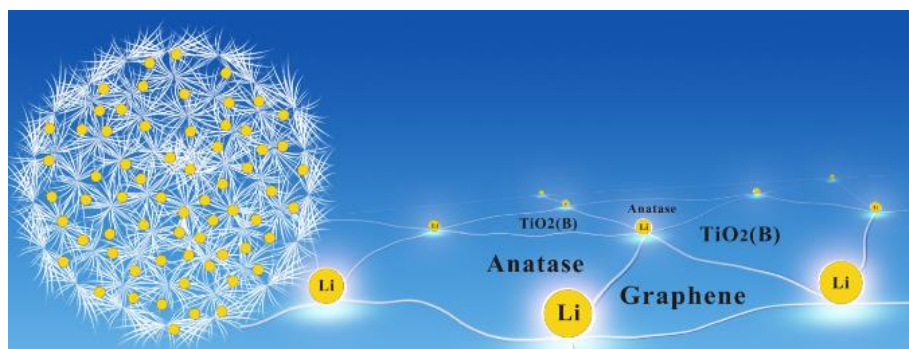


Figure 1. Schematic representation of interfacial storage for Li⁺ ions of TiO₂/MLG

In this work we have fabricated a new TiO₂-graphene composite designed to enhance the prospects of this low cost, abundant, non-toxic material as an anode in a Li-battery. The TiO₂ is dandelion shaped with nanosized TiO₂(B) fibrils capped with anatase TiO₂ pappi. Electron microscopy indicates that the composite contains triphase boundaries between anatase, TiO₂(B) and graphene, which are responsible for the enhancement of energy storage and the decrease of electrode polarization. Cyclic voltammetric investigations point to both Li⁺ insertion and pseudocapacitive contributions to charge storage. Ultrahigh specific capacities of 243 and 182 mAh g⁻¹ have been obtained at 0.1 and 1 A g⁻¹, respectively. Moreover, the excellent capacity retention can reach 99.6% after 100 cycles with almost 100% coulombic efficiency at 0.1 A g⁻¹.

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Keynote: Replacing Critical Elements from Energy Materials

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The creation of new and very importantly greener industries and new sustainable pathways are crucial to create a world in which energy use needs not be limited and where usable energy can be produced and stored wherever it is needed. New materials based on carbon, ideally produced via inexpensive, low energy consumption methods, using renewable resources as precursors, with flexible morphologies, pore structures and functionalities, are increasingly viewed as ideal candidates to fulfil these goals. The resulting materials should be a feasible solution for the efficient storage of energy and gases. Hydrothermal carbonization [1] is an ideal technology for the production of such low-cost but highly performing materials out of the most abundant renewable resource on the planet, i.e. lignocellulosic biomass. The practical approach is very simple and consists in placing a biomass precursor inside an autoclave, in water, followed by hydrothermal treatment overnight at 160-200°C. Since the production of carbon materials in general implies harsher and multi-step methodologies along with fossil –based precursors, this process has clear advantages in terms of sustainability and cost. Here, I wish to present some of our latest results on the production and characterization of nanostructured hydrothermal carbons (HTC) and their use in renewable energy related applications, in particular energy storage. [2-4]

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The Influence of Sodium Metal in Sodium Ion Half-Cell Testing Shown in Hard Carbon Anodes

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Development and research on room temperature sodium ion batteries started in the early 1970's alongside to lithium ion batteries. With the commercialisation of the first lithium ion cell by Sony in 1990 industry focused on lithium ion technology, which was more promising at that time. Within the last 10 year demands for energy storage technology grew rapidly due to fluctuating renewable energy production. Besides this consumer behaviour aims for more independent devices, such as cordless power tools and rechargeable electronic devices. The increase of lithium need pushes the costs further, so alternative sodium ion technologies may offer a cost and safety advantage over the typical lithium ion cells.[1],[2] Sodium is a much more abundant metal and is extracted from seawater whereas the largest lithium sources are based in countries like Bolivia and China. Although the mass of sodium is higher than lithium, the observed specific capacity of a sodium ion battery compared to lithium is not hugely compromised; this is because the proportion of the sodium and lithium content is small enough that the difference is minimal.

Sharp Laboratories of Europe Ltd have been developing a new sodium ion cell chemistry based upon a layered oxide cathode and a hard carbon anode [3]. The hard carbon anode is extremely sensitive to processing conditions and testing methods. In general all new materials are tested in half cells to investigate and understand material characteristics before full cell development is started. In our screening work we observed high hysteresis in the charge and discharge profiles and have experienced difficulties in reaching the low voltages required for complete sodiation of the hard carbon. In addition we have also observed high Coulombic inefficiencies during the testing in a sodium metal anode cell, and the instability of the sodium in certain solvents. By using 3-electrode cells we have been able to investigate the specific properties of the hard carbon in considerably more detail, and note that much of the hysteresis observed in two electrode measurements originates from the sodium metal anode. Furthermore 3-electrode testing enabled us to show that much of the hysteresis with regards to the performance of the carbon material in a 2-electrode cell is related to the sodium metal deposition and stripping.

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[2] J. Barker, M.Y. Saidi and J. Swoyer, Electrochem. Solid-State Chem. 2003, 6, A1

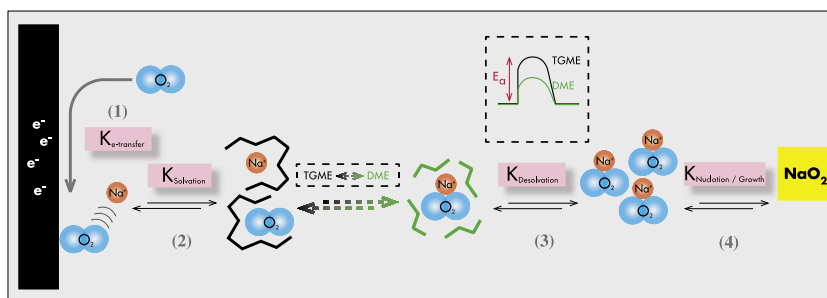
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High capacity NaO₂ batteries - Understanding the key parameters for solution-mediated discharge

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Increasing energy storage demands, driven by the needs of electric vehicles and the field of renewable energy sources, motivate the search for lower cost, higher capacity, and more sustainable rechargeable batteries.[1] Metal-air batteries are of great interest due to their large theoretical capacity, which relies on the redox reaction of gas species rather than solid-state intercalation reactions, such as those that occur in Li-ion and Na-ion batteries.[2-4] The Na-O₂ battery offers an interesting alternative to the Li-O₂ battery, which is, despite numerous works, still the source of a number of unsolved scientific questions. In spite of both being alkali-O₂ batteries, they display significant differences. For instance, Li-O₂ batteries form Li₂O₂ as the discharge product at the cathode, whereas Na-O₂ batteries form NaO₂. A very important question that affects the performance of the Na-O₂ cell concerns the key parameters governing the growth mechanism of the large NaO₂ cubes formed upon reduction. Using glyme-ethers of various chain lengths we show that, in contrast to the Li-O₂ system, high solubility's of the NaO₂ discharge product do not necessary lead to increased capacities. Herein we report the profound effect of solvation by chelation of Na⁺ on the NaO₂ growth mechanism and battery performances. This work provides a new way to look at the key role that solvents play in metal-air systems.



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Liquid Phase Charged Graphene for Engineering

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Liquid phase exfoliation of graphite has been well documented as a cheap, scalable route to the commercial application of graphene. With a new method, graphite is first intercalated with an alkali metal to create an alkali metal salt, or graphite intercalation compound, before the negatively charged graphene and positively charged metal cations are both solvated. The charge in this system can be readily used to design functional nanomaterials or electrochemically deposited graphene coatings for engineering purposes.

Understanding High capacity in Li-rich 3d cathode materials

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Lithium rich layered cathodes, which offer increased capacity, have, for several years, been regarded as one of the next major advances, able to store charge by invoking transition metal and oxygen redox processes in the same material. However, they are still challenged by problems such irreversible first cycle capacity, voltage fade on cycling related to changes in the structure. Recently important work has been carried out to better understand the origin of the high capacity and the voltage fade.

In this contribution we shall present data probing the nature of the O redox states in Li rich 3d transition metal oxides, on the nature of O loss probed by isotopic substitution and on the degree of oxygen loss vs oxygen redox. We shall also report on a new sol-gel synthesis for $\text{Li}_{1.2}\text{Mn}_{0.54}\text{Ni}_{0.13}\text{Co}_{0.13}\text{O}_2$. The electrochemical performance is shown in Figure 1. The cycling stability of the material is good with a capacity of $\sim 250 \text{ mAh g}^{-1}$ maintained after 100 cycles.

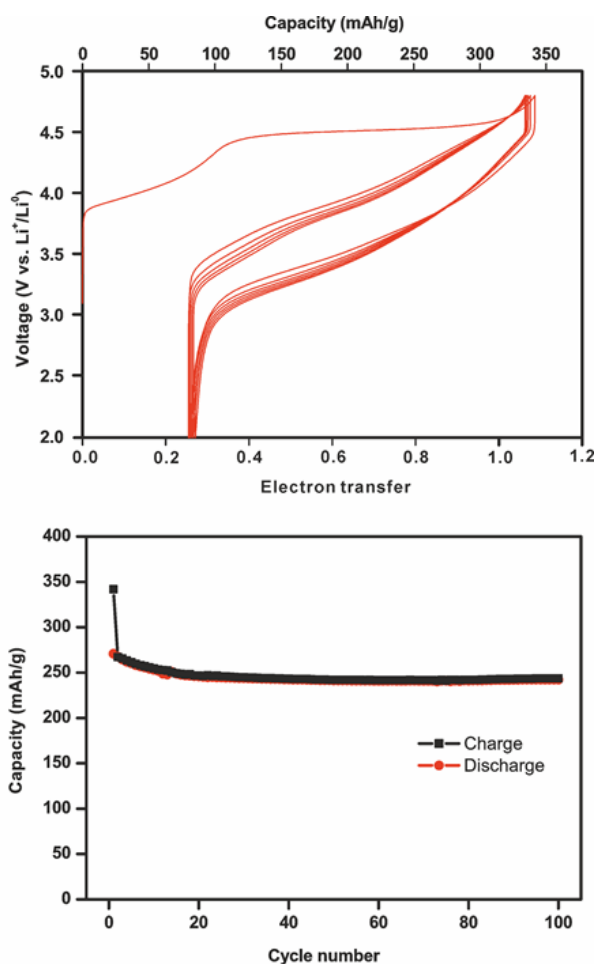


Figure 1. Load curve and cycleability data of sol-gel prepared $\text{Li}_{1.2}\text{Mn}_{0.54}\text{Ni}_{0.13}\text{Co}_{0.13}\text{O}_2$ at a rate of 50 mA h g^{-1} .

Keynote: Flow Batteries: Design and Experience

Ian Whyte

Potential Reactions Ltd, Bedford, UK

Design and operational aspects of flow batteries.