

NEWSLETTER



**Serving Electrochemical Science, Technology and Engineering within
the catchment of**

**The Royal Society of Chemistry
and
The Society of Chemical Industry**



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Editorial

Welcome to the first issue of the Electrochemistry Newsletter in 2020. Last year continued to be a busy year for the members of the electrochemical community in the area of batteries at the Faraday Institution with a number of groundbreaking research scientists continuing to work on improving battery performance. More details can be found at: <https://faraday.ac.uk/>

This issue includes a number of reports from students and postgraduates who attended or organised a conference. Students should be encouraged to apply for a contribution to the cost of presenting their work at a national or international conference or organising a postgraduate conference. The Electrochemistry Group of the RSC and the Energy Technology Group of the SCI provide the funds if the application is accepted. Candidates should apply to [Dr. Mark Symes](#).

I welcome any feedback and suggestions or contributions from readers for future issues. Many thanks to Oliver Rodriguez Martinez who helped to put together the Newsletter.

Carlos Ponce-de-León

If you wish to notify the editor with your view on the material or the content of any item in this issue, or if you wish to contribute to the newsletter, please write to the Editor-in-Chief (Carlos Ponce-de-León, Faculty of Engineering and the Environment University of Southampton) at:

capla@soton.ac.uk

Missed a copy? You can catch up on all the news *via* our web-space hosted by the Royal Society of Chemistry at the following URL.

<http://www.rsc.org/Membership/Networking/InterestGroups/Electrochemistry/news.asp>

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Conference report:

Regional Postgraduate Symposium Great Western Electrochemistry University of Bath, Monday 26th June 2019

On Monday 24th June 2019, the one-day postgraduate “GWEM 2019” symposium was held at the University of Bath. At this meeting invited guests from academia and industry presented talks together with postgraduate presenters from Bristol, Oxford, Bath, Brighton, King’s College, Swansea, and from Cardiff. Industry exhibitors (Alvatek, Blue Scientific, Whistonbrook, Metrohm) contributed with displays and prizes. There were four sessions with in total 17 talks, all chaired by postgraduate students. The major theme of the day was carbon dioxide reduction, but a much wider range of topics was covered, including photo-electrochemistry, thermo-electrochemistry, corrosion, and surface alloying.



The day started with a talk by Yuanzhu Zhao (Bath) on photo-electrochemical processes involving graphitic carbon nitride with glucose as a sacrificial quencher. Yuanzhu employed the polymer of intrinsic microporosity PIM-1 to enhance the photocatalytic process and to more effectively “harvest” hydrogen. Disah Mpadi (Bristol) then presented work on lanthanum ferrate photo-electrodes. Xin Sun

(Bristol) continued this topic with a talk on more complex photo-cathodes and their ability to generate hydrogen in the presence of light. Surface phenomena during underpotential-alloying based on Pb/Au were explained by Alicja Szczepanska (Bristol). The first session concluded with a talk on molecular hydrogen evolution catalysts based on hydrogenase molecular frameworks. Georgia Orton (KCL) presented this work as an example of interdisciplinary study of molecular structures mimicking biological reactivity.

The second session was dedicated to invited guest speakers. Dr. Charles Cummings (Qinetiq) presented work from an industry perspective focusing on the capture/conversion of carbon dioxide in submarine environments. Next, Dr. Eileen Yu (Newcastle) presented an overview of her work on carbon dioxide conversion in microbial films/cells. After a lunch break, the third session started with Dr. Enno Kätelhön (Oxford) discussing computer simulations and experimental evidence for “apparent catalysis/inhibition” and effects caused by diffusion through particulate films on electrodes. The next three contributions from postgraduate students were given by Chaolong Yang (Bristol) on “Imaging of topological insulator single crystal surfaces under electrochemical control”, by Mark Buckingham (KCL) on “An investigation into pseudo-thermocapacitance through gelled electrolyte thermo-electrochemistry”, and by Ferraraccio Lucia Simona (Swansea) on “Electroluminescence (ECL) at enzymes encapsulated in alginate hydrogels”.

After a final opportunity to discuss with exhibitors, go through poster presentations, and to refuel with coffee/tea and cookies, the final session started with Prof. Enrico Andreoli (Swansea) describing “Copper foam catalysis and electroreducing carbon dioxide to n-propanol”. This highly challenging research aim and developments towards improving yields and catalytic performance were discussed in the framework of economic opportunities for products derived from CO₂. Next, Carla Gonzalez Solinio (Bath) presented her work on “Exploring the use of printed circuit boards for energy harvesting from glucose”. Ben Howchen (Bristol) discussed “Mixed Ni/Ir lanthanide electrocatalysts for the oxygen evolution reaction. Dr. Kate Lawrence (Wiley-VCH) concluded the day with a presentation aimed at explaining “careers and trends in scientific publishing”. This presentation gave a lot of food for thought (for both postgraduates and academics) and themes for further lively discussions in the wine and nibbles reception.

All-in-all, this was a successful day of electrochemistry and a difficult final task remained for the two juries (for posters and for talks) to award prizes. The “Whistonbrook” poster prize was awarded to Muhamed Alkaliba (Bristol) for his poster on “Bifunctional oxygen electrocatalysis”. The two “ChemElectroChem” poster prizes went to Nathan Missault (Bristol) for “Thin film electrodeposition” and to Ferraraccio

Lucia Simone (Swansea) for “Electroluminescence in enzyme electroanalysis”. The Alvatek Prize for an excellent talk was awarded to Mark Buckingham (KCL) for “An investigation into pseudo-thermoelectrochemistry through gelled electrolyte thermoelectrochemistry”. The Blue-Scientific prize for an excellent talk was awarded to Yuanzhu Zhao (Bath) for “Photoelectrochemistry of immobilised Pt@g-C₃N₄ mediated hydrogen and enhanced by the polymer of intrinsic microporosity PIM-1”. Finally, the RSC prize for the best presentation was awarded to Georgia Orton (King’s College London) for her talk on the topic “Towards diiron hydrogenase mimics as electrocatalysts for H₂ oxidation”. This presentation is therefore nominated for presentation at the Electrochem 2019 conference in Glasgow.

26th June 2019, Bath
Frank Marken

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**Great Western Electrochemistry
University of Bath, Monday 24th June 2019**

Location: Lecture Theatre 3WN2.1 (behind Physics, see below)

Programme

9.15			Coffee & Posters
10.15	Yuanzhu Zhao (Bath)	Photoelectrochemistry of Immobilised Pt@g-C ₃ N ₄ Mediated by Hydroge and Enhanced by the Polymer of Intrinsic Microporosity PIM-1	
10.30	Disah Mpadi (Bristol)	Nanostructured LaFeO ₃ photoelectrodes	
10.45	Xin Sun (Bristol)	Complex metal oxide photocathodes	
11:00	Alicja Szczepanska (Bristol)	Electrochemically controlled surface alloying	
11.15	Georgia Orton (KCL)	Towards diiron hydrogenase mimics as electrocatalysts for H ₂ oxidation	
11.30			Break & Discussion & Posters
12.00	Charles Cummings (Qinetiq)	Submarine Atmosphere Control: 'Amine Degradation within CO ₂ Removal Plants'	
12.30	Eileen Yu (Newcastle)	Converting CO ₂ to fuels and chemicals with microbial electrosynthesis	
13.00			Lunch (not provided), Coffee (provided) & Posters
14.00	Enno Kätelhön (Oxford)	Nanoparticle electrocatalysis: Unscrambling illusory inhibition and catalysis	
14.30	Yuanyang Rong (Brighton)	Charge transfer nanohybrids of graphene oxide and the intrinsically microporous polymer PIM-1	
14.45	Chaolong Yang (Bristol)	Imaging of topological insulator single crystal surfaces under electrochemical control	
15.00	Mark Buckingham (KCL)	An investigation into pseudo-therm capacitance through gelled electroly thermoelectrochemistry	
15.15	Ferraraccio Lucia Simona (Swansea)	Electrochemiluminescence (ECL) at enzymes-encapsulated in alginate hydrogels	
15.30			Coffee & Posters
16.00	Enrico Andreoli (Swansea)	Copper Foam Catalysis; Electroreducing Carbon Dioxide to n-Propanol	
16.30	Isabella Poli (Bath)	Cesium Lead Bromide solar cells for water splitting	
16.45	Carla Gonzalez Solinio (Bath)	Exploring the use of printed circuit boards for energy harvesting from glucose	
17.00	Ben Howchen (Bristol)	Mixed Ni/Ir Lanthanides electrocatalysts for the oxygen evolution reaction	
17.15	Kate Lawrence (Wiley-VCH)	Careers and Trends in Publishing	
17.30			Wine and Nibbles in 1 South (Department of Chemistry)



ISE REGIONAL STUDENT MEETINGS

Graduate Students who are members of ISE and intend to organize a **Regional Student Meeting** can apply for ISE financial support. **Regional Student Meetings** are typically one-day meetings involving graduate students active in the geographic area where the meeting takes place.

The format of the meeting (oral presentations, posters, discussion sessions, other) is autonomously decided by the organizers who will be responsible for securing a venue and collecting registrations. No registration fee should be requested. No later than one month after the meeting, the organizer(s) will send to the ISE Office a report on the event, including the names and the e-mail addresses of the participants. The participants will be encouraged to apply for ISE membership. An overview of the report accompanied by suitable pictures if available will be posted on the ISE website under Student Activities.

Applications for ISE support must be sent by e-mail to the **ISE Office** (info@ise-online.org), with a copy to the Regional Representative of the country where the meeting is organized, 3-12 months before the meeting date, using the **application form**. The local ISE Regional Representative (**Dr. Tim ALBRECHT of Imperial College London, for the United Kingdom**), if requested, will assist the potential meeting organizer in the preparation of the application. Applications will be analyzed by a committee consisting of (i) ISE Secretary General, (ii) ISE Treasurer, (iii) ISE Vice President responsible for Educational Activity and (iv) ISE Vice President responsible for Regional Sections.

The response will be communicated to the applicant and to the relevant Regional Representative no later than 1 month after the application submission.

The maximum financial support will be **600 €**; the expected use of the funds must be specified in the application. Co-sponsoring by other Societies and/or institutions is possible.

Find out more:

<http://www.ise-online.org>

Student reports

Report on the BES XXV Conference

I was accepted to speak at the 25th International Symposium on Bioelectrochemistry and Bioenergetics (BES XXV), a conference organised by the Bioelectrochemical Society and held at the University of Limerick. It was an enjoyable and stimulating week of academic talks, poster receptions and networking with many top academics and fellow young researchers in the field. Attending this conference was truly a constructive experience to my development as a young researcher, and helped to gain approval and acknowledgement of my DPhil research, much needed as I approach my thesis submission date.



Rainbow over the University of Limerick campus

electron reduction of pyruvate to L-lactate. This cofactor is prohibitively expensive for industry to use at stoichiometric ratios, therefore NADH should be regenerated, allowing it to be used at sub-stoichiometric amounts. This research focuses on the use of various 'NAD⁺-reductase'-modified carbon working electrodes with direct electron transfer to specifically reduce NAD⁺ back into the biologically active 1,4-NADH, allowing for transfer of electrons between the two enzymes and reduction of the substrate C=O bond of interest with high Faradaic efficiency. I developed this electrode interface into an electrochemical flow cell, with the ambition of using this module within the fine chemical and pharmaceutical industries.

I have been working with the Vincent Group at the University of Oxford for two years, with a project to develop a biological electrocatalyst for specific C=O reductions within a flow cell. In nature, C=O bonds are often reduced by a class of enzymes called *NADH-dependent oxidoreductases*. For example, an *L-lactate dehydrogenase* (LDH) enzyme uses the labile hydride provided by the nicotinamide enzyme cofactor 1,4-NADH, for the specific two



"Guinness is good for you..."



Panorama of King John's castle and of the city of Limerick

The conference proceedings were dominated by discussions on *sensing biointerfaces* and *bioenergetics*. Other session topics included *electrophysiology*, *materials for bioelectrochemistry* and *protein electrochemistry*. Speakers of particular personal interest included Daniel Bond, who spoke about his group's study into cytochrome conduit complexes in the genome of *G. sulfurreducens*, *giving insights into the electron transfer mechanism of this microorganism's metal reduction pathway*. Another was Laura Wey's investigation into the presence of soluble endogenous electron mediator in cyanobacteria *Synechocystis* sp. PCC6803, which nicely complements studies of photosystem II photoelectrochemistry.



Bunratty castle, near Limerick

In addition to proceedings, there was a social trip to two castles and an historical Irish village, which was certainly an interesting change of pace to learn about the local culture and history. Afterwards was the conference dinner, which was great firstly for the unlimited Guinness, a very important and delicious drink in Ireland, and secondly for the disco at the end. Nothing sums up the end of a long

conference week like dancing with everyone from the students to even the most established professors!

Overall, the BES conference was a fantastic experience and was vital for the development of my research. Through presenting and answering questions about my talk, and with informal discussions with many academics in the bioelectrochemical field, I returned home inspired with fresh experimental ideas to try out in the lab. I would like to thank the RSC Electrochemistry Group for helping to fund my attendance at BES XXV – it was certainly a valuable experience for me and my research.

Justin R. Weeks

DPhil candidate

Vincent Group, University of Oxford



Everyone who attended BES XXV!

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Student reports

SSI2019: 22nd Conference of the International Society of Solid State Ionics, 16-21 June 2019 Conference Report (International): Steffen P. Emge, PhD student, University of Cambridge

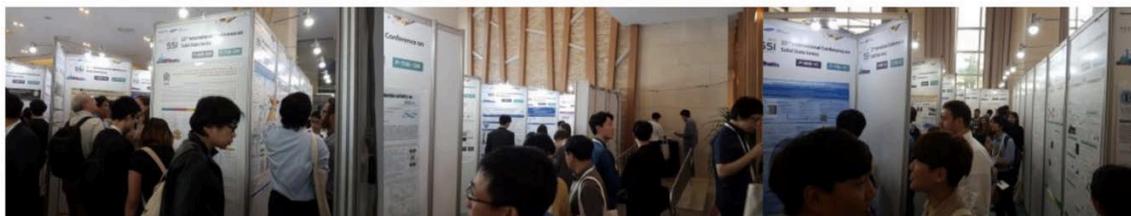


Solid State Ionics is held biennially and one of the biggest conferences dealing with this topic. The 22nd Conference was held in PyeongChang, the venue of the last Winter Olympics, and drew attendees from all over the world. It focused on various areas of research related to Solid State Ionics, with one Fundamentals session and various applied sessions (i.e. Batteries, Fuel Cells, Sensors etc.) to cover different interests.



Due to my research I attended mostly the Battery session, but also the Fundamentals and Industry sessions. Each day began with a plenary lecture by a prominent researcher of the field, e.g. Prof. Manfred Martin (“History” of Solid State Ionics), Prof. Eric Wachsman (Li Anode, Dendrites and LLZO) and Prof. Ryoji Kanno (Development of Sulfur based highly conducting solid electrolytes).

A major focus of the battery section was on layered transition metal oxides (LiCoO₂, NCA, NMC etc.). The talks about anionic redox in cathode materials, what causes it and how to observe it, were particularly enlightening. Another large topic were solid-state batteries, with a big part about different solid electrolytes and how to combine these with electrode materials to achieve a solid-state battery. For me of great interest were the talks on how to enable Li metal as anode and prevent dendrites in solid electrolytes and why dendrites grow, which was also part of one of the plenary talks (by Eric Wachsman). The industry session had an interesting talk by LG about next generation batteries, their requirements and how to achieve them. The fundamentals session had a quite enlightening group of talks regarding disorder and defects and their influence as well as experimental techniques for observing defects.



The poster sessions were held Monday and Tuesday evening and, although a bit crammed, it was a very rewarding experience with many helpful scientific discussions. On the second day I was able to present my poster on the local structure of a garnet-type Li-ion solid electrolyte (LLZO) studied by solid state NMR. I got some helpful feedback and was able to network with other people over our research. At the end, I was so fortunate to receive one of the best poster awards for my poster.



The broad range of topics made sure that basically everybody could find a session of interest. The organization of the conference was impeccable with a Welcome ceremony on Sunday evening (as well as Tutorials during the day) and the scientific program from Monday to Friday. The conference culminated in a brilliant Gala dinner, which was a great networking opportunity, on Thursday and the closing ceremony on Friday, where amongst other things Poster Awards and the next location for SSI-2021 were announced (MIT).

I highly appreciate the support by the RSC Electrochemistry Group (as well as St. Catharine's College and the Department of Chemistry, Cambridge), which made it possible for me to attend this conference and I can only recommend this conference to anyone in the field of Solid State Ionics.



As nice treat of traditional iced Korean tea and sweets due to the hot temperatures in Korea at the time.

Student Conference Bursaries

The Student Bursary Scheme provides financial support to promising postgraduate students to attend a major electrochemistry conference abroad. This includes UK based students travelling to a conference abroad and students based abroad wishing to attend a conference in the UK. The Bursary Scheme is open to all postgraduate student members of the RSC's Electrochemistry Group undertaking research in electrochemistry. Applications shall consist of:

- (i) the application form (see <http://www.rsc.org/ScienceAndTechnology/Funding/TravelGrants/InterestGroups.asp>),
- (ii) the abstract submitted to the conference organisers,
- (iii) one A4 page *curriculum vitae* stressing academic and scientific achievements (e.g., research articles, oral and poster presentations **made by the applicant**).

Applications may be made at any time of the year and shall be submitted to the Group Secretary in electronic form.

The selection committee of the Electrochemistry Group shall decide the sum awarded. Under normal circumstances this sum shall not exceed £300.

Successful applicants shall produce a conference report article for the Newsletter.

Candidates should submit their applications directly to the RSC Electrochemistry Group Secretary:

Dr. Mark Symes : (e-mail: Mark.Symes@glasgow.ac.uk)
Prof. Upul Wijayantha (email: U.Wijayantha@lboro.ac.uk).

Related: also see RSC travel bursaries

<http://www.rsc.org/Membership/Networking/InterestGroups/Electrochemistry/StudentBursaryScheme.asp>

Echem.NET

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More Meetings:



International Society of Electrochemistry

2020

29 March - 1 April, Tainan, Taiwan (*26th ISE Topical Meeting*)

Electrochemical Deposition for Semiconductor and Green Energy

18 - 21 May, Salt Lake City, Utah, USA (*27th ISE Topical Meeting*)

Electroanalytical Chemistry and Bioelectroanalysis

30 August - 4 September, Belgrade, Serbia (*71st Annual Meeting*)

Electrochemistry towards Excellence

2021

29 March - 1 April, Santiago, Chile (*28th ISE Topical Meeting*)

Challenges in Molecular Electrochemistry and Surface Reactivity

18 - 21 April, Mikulov, Czech Republic (*29th ISE Topical Meeting*)

Energy and water: electrochemistry in securing the sustainable society development

29 August - 3 September, Jeju Island, Korea (*72nd Annual Meeting*)

Electrochemistry from Fundamentals to Products

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The Electrochemical Society

Advancing solid state & electrochemical science & technology

2020

237th ECS Meeting with the 18th International Meeting on Chemical Sensors (IMCS 2020)

May 10-15, 2020 — Montréal, Canada

Palais des Congres de Montréal

PRiME 2020

October 4-9, 2020 — Honolulu, HI

Hawaii Convention Center & Hilton Hawaiian Village

2021

239th ECS Meeting

May 30-June 3, 2021 — Chicago, IL

Hilton Chicago

240th ECS Meeting

October 10-15, 2021 — Orlando, FL

Orange County Convention Center

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Where science meets business

Electrochemical Technology

The Electrochemical Technology Technical Interest Group is involved in all aspects of the application of electrochemical science and engineering. The Group's aim is to promote research and development of electrochemistry which leads to the production of appropriate technologies and industrial and consumer products. The Group provides an interface between academia and industry and is a forum for promoting research and collaboration between a range of scientific and engineering disciplines.



Industrial sectors

Electrochemical activities cut across all industrial sectors, including chemical, pharmaceutical, electrical, electronic and micro-electronic, information technology, mining and metallurgical, biotechnology, transportation, medical, water and wastewater. As such, the Group's interests include applications of electrochemistry in:

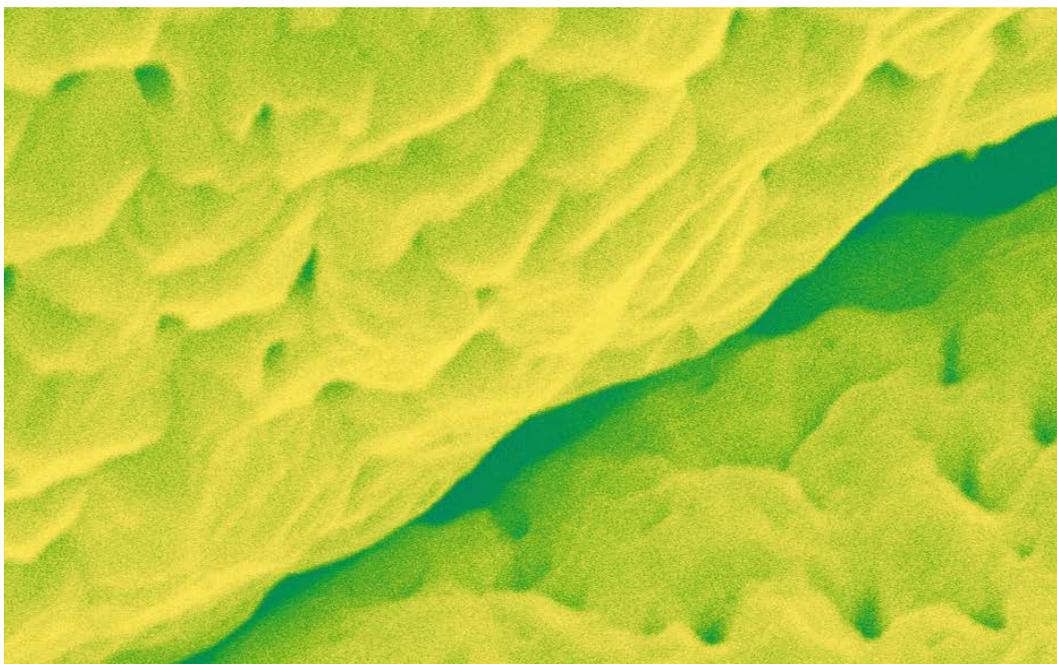
- ▶ sensors and monitors
- ▶ energy conversion and storage
- ▶ synthesis of chemicals, pharmaceuticals, biochemicals, polymers and electronic materials
- ▶ materials protection, processing and fabrication
- ▶ environmental protection and control

Join at:

<http://www.soci.org/membership-and-networks/technical-groups/electrochemical-technology-group>

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RSC Electrochemistry Group



RSC Electrochemistry Group

This RSC Group is part of the Faraday Division, involved in all aspects of electrochemical processes (fuel cells, energy sources, analytical devices and sensors, electrochemical planting and synthesis, fundamental research etc).

Activities:

- The Group organises the annual 'Electrochem' meetings (Faraday Medal) to reward outstanding international scientists. For up-to-date information, go to the RSC's web pages for the Electrochemistry Group.
- The Electrochemistry newsletter: available quarterly, in pdf, from our RSC web pages, it highlights events' reports and general sector's news and insights.
- Student bursaries: to support/encourage graduate students giving lectures on their PhD work at national and/or international conferences.
- Outreach: activities involving the public and schools to raise awareness of the fundamental importance of electrochemical processes today.

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Chemical Sciences

www.rsc.org/electrochemistry

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Join at:

<http://www.rsc.org/Membership/Networking/InterestGroups/Electrochemistry/>

The Electroanalytical Sensing Systems Group

The Electroanalytical Sensing Systems Group is one of the RSC's many Interest Groups. The Interest Groups are member driven groups which exist to benefit RSC members, and the wider chemical science community, in line with the RSC's strategy and charter.

Join at:

<http://www.rsc.org/Membership/Networking/InterestGroups/Electroanalytical/>

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Prizes

Castner Medal Winner

[Professor Keith Scott](#),
University of Newcastle,

Faraday Medal Winner

[Professor Martin Winter](#),
University of Munster

Fleischmann Lecture

[Professor Karen Faulds](#),
University of Strathclyde

Parsons Medal Winner

[Dr. Alison Parkin](#),
University of York

Evans Medal Winner

[Professor Tetsuo Shoji](#),
University of Tohoku

Paul McIntyre Award

[Dr Steve Paterson](#),
Robert Gordon University

**Electrochemical engineer awarded prestigious
Schwäbisch Gmünd Prize for Young Scientist**

[Dr Luis Fernando Arenas](#)
University of Southampton

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Welcome from the Local Organising Committee

Welcome to Electrochem2019 in Glasgow!

This year's Electrochem is a joint venture between the University of Strathclyde and the University of Glasgow and is being held in the University of Strathclyde's fantastic Technology and Innovation Centre right in the heart of Glasgow.

According to the readers of *Rough Guides* travel books, Glasgow is the world's friendliest city, and who are we to argue with that? We hope that you will have plenty of opportunity for catching up with old acquaintances and making new friends as part of our social programme, including a civic reception in the spectacular Glasgow City Chambers and a traditional Scottish ceilidh on the first evening, and the sumptuous conference banquet on Tuesday night.

There should be a bit of time left over for some electrochemistry too! This year will see the inaugural presentation of the RSC Roger Parsons medal for early career researchers, alongside the more established awards such as the RSC Faraday medal, the SCI Castner medal and the Institute of Corrosion's Evans award. And then there are over 60 other talks and another 60+ posters to enjoy.

Obviously none of this would have been possible without the help and support of a great many support staff from the universities and City Chambers. Special mention must go to Joy Leckie, Nic Toshner, Ewa Kosciuk and Karla Cunningham for their efforts.

Finally, we would like to thank you for participating and supporting Electrochem2019. We hope you enjoy the conference!

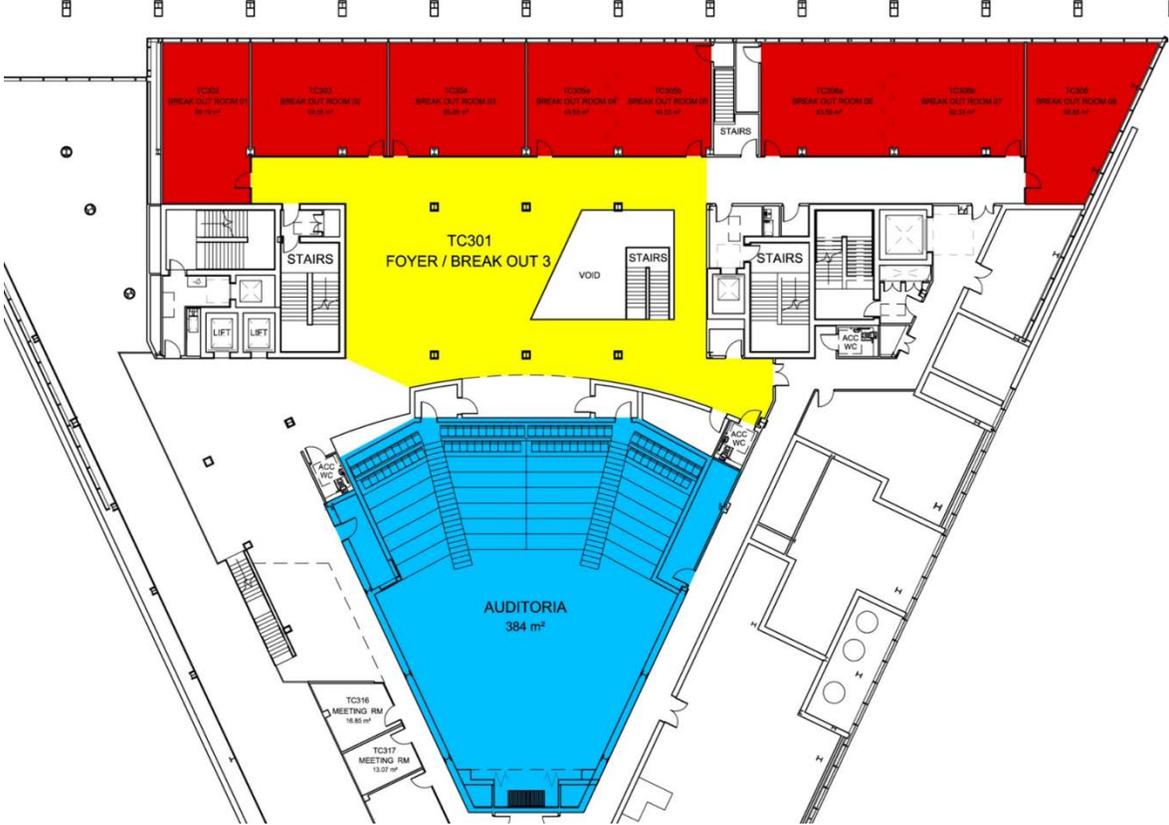
Mark Symes, Sudipta Roy, Todd Green, Damion Corrigan, Ashleigh Fletcher

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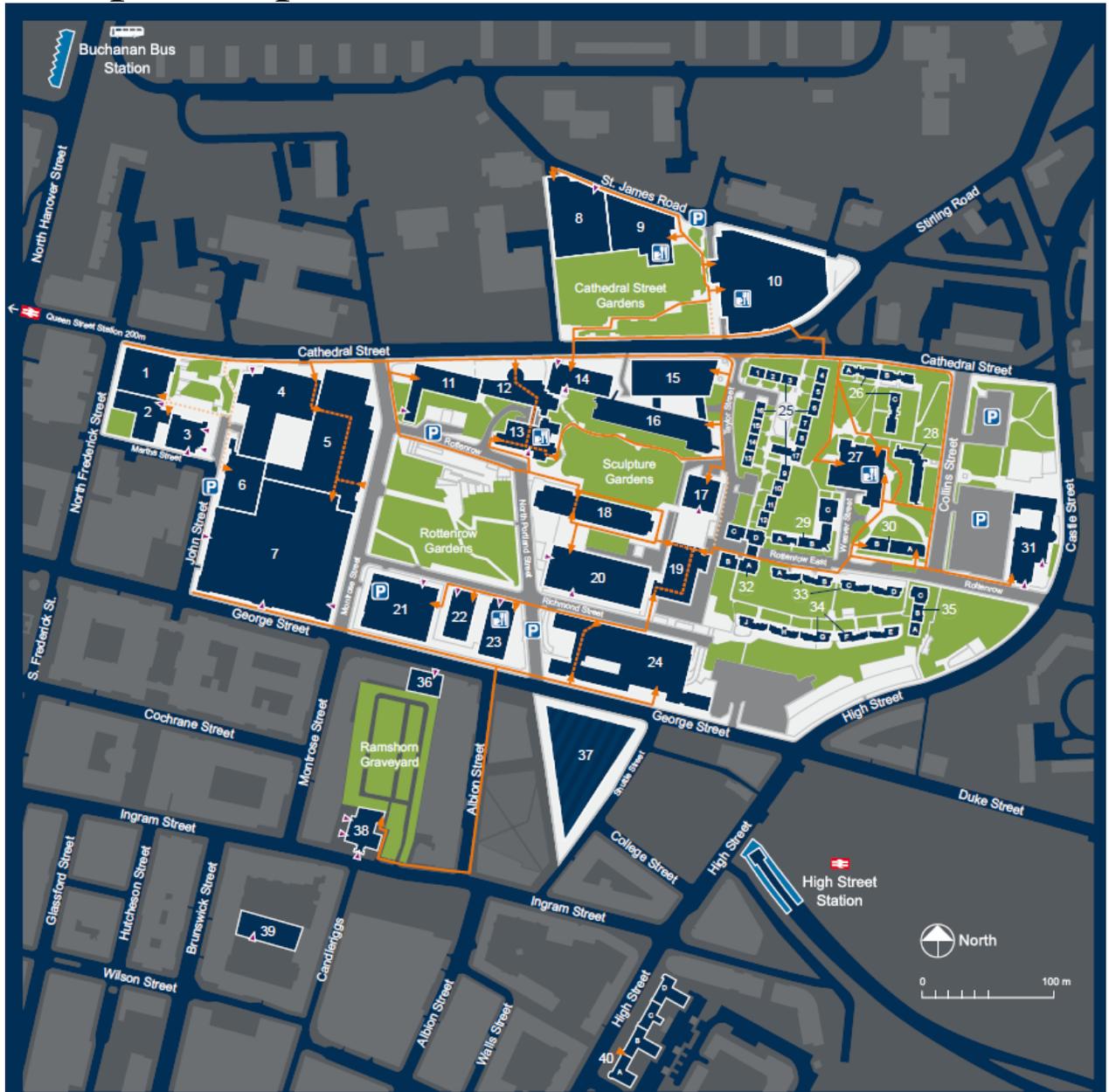
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Floor Plan (Level 3)



Campus Map



Campus Buildings

Alexander Turnbull	36	Garnett Hall	25
Andrew Ure Hall	40	Graham Hills	24
Arbuthnott (Hammett Wing)	15	Henry Dyer	11
Arbuthnott (Robertson Wing)	16	James Blyth Court	34
Architecture	18	James Goid Hall	30
Barony Hall	31	James Young Hall	35
Birkbeck Court	25	James Weir	5
Centre for Sport and Recreation	1	John Anderson	19
Chancellors' Hall	32	Livingstone Tower	23
Collins	22	(Security Control)	
Colville	20	Lord Hope	9
Curran (Andersonian Library)	10	Lord Todd (Village Office)	27
Estates Services	8	McCance	21
Forbes Hall	29	Murray Hall	28

Patrick Thomas Court	39
Ramshorn Theatre	38
Royal College	7
Sir William Duncan	13
St Paul's	3
Stenhouse	14
Strathclyde Business School	12
Students' Union	6
Technology and Innovation Centre	37
Thomas Campbell Court	
Thomas Graham	4
University Centre	2
Wolfson Centre	17

Key

- University Buildings
- Accessible Routes External
- Accessible Routes Through Buildings (Walkways 0.05m - 0.05m)
- Accessible 'Buddy' Routes (assistance required)
- Accessible Entrance
- Non-Accessible Entrances
- Public Car Parking, Pay and Display
- Catering Outlets
- Building under construction

Delegate Information

Conference Venue (Building 37 on map)

Technology and Innovation Centre
University of Strathclyde
99 George Street
Glasgow
G1 1RD

Conference Registration

Monday 26th August, 18:00-19:30 in Nourish@Aroma Café, Lord Todd Building (venue for Welcome Dinner, see below)

Tuesday 27th August and Wednesday 28th August, 08:00 – 17:00 in the reception area of the Technology and Innovation Centre

WiFi

Access via Eduroam is available to those visiting from other academic institutions. We also have a dedicated WiFi circuit available for organisers, exhibitors and delegates in all conference spaces and public areas on Levels 1, 2, 3 & 9 of the building. To connect, select TIC Conferences from the list of available networks and enter the password, which will be available at registration.

Campus Accommodation

Lord Todd (Building 27 on map)
11 Weaver Street
Glasgow
G4 0NS

Internet access available in all rooms. For check out details, please ask at reception.

Welcome Dinner

Monday 26th August, 18:00-19:30, Nourish@Aroma Café, Lord Todd Building.

Civic Reception

Monday 26th August, 19:30-20:30, Glasgow City Chambers (guides will depart from Nourish@Aroma Café at 19:20).

Ceilidh

Monday 26th August, 21:00-late, Sloan's, Argyll Street (guides will depart from City Chambers directly after the Civic Reception).

Conference Dinner (Building 31 on map)

Tuesday 27th August, 19:00-00:00, Barony Hall, Rottenrow, Glasgow (drinks from 19:00).

Sponsors

Many thanks to our generous sponsors for supporting this conference:



Wednesday				
0830-0900				
0900-1015	Refreshments, Registration in reception area of Technology and Innovation Centre			
1015-1025	Plenary - Icorr Evans Award (Shoji)			
1025-1045	Sponsors 5&6			
1045-1105	Plenary - EPSRC (Rolph)			
1105-1125	Refreshments			
1125-1145				
1145-1205	Fleischmann Lecture (Faulds)			
1205-1225	EE9 (Bentley)	MP9 (Amador)	PF9 (Hicks)	C9 (Harrison)
1225-1245	EE10 (Dickinson)	MP10 (Kim)	PF10 (Hou)	C10 (Leeds)
1245-1305	EE11 (Craig)	MP11 (Mavrikis)	PF11 (Betts)	C11 (Thomson)
1305-1400	Lunch			
1400-1420	EE12 (Kumar)	MPK1 - Keynote (Arenas) EAST	PFK1 - Keynote (Denuault)	C15 (Spajić)
1420-1440	EE13 (Wain)	Schwäbisch Gmünd Prize	PF12 (Katsounaros)	C16 (Sainis)
1440-1500	EE14 (Perry)	MP12 (Eiler)	PF13 (Huang)	C17 (Moulton)
1500-1520	EE15 (Wu)	MP13 (Daviddi)		C18 (Araujo)
1520-1550	Refreshments			
1550-1610	EE16 (Mohamed)	MP14 (Cross)	PF14 (Reeves)	C19 (Al-Nafai)
1610-1630	EE17 (Iamprasertkun, Sheelagh Campbell award)	MP15 (Aldous)	PF15 (Ivaturi)	C20 (Rzeszutek)
1630-1650	EE18 (Bhattachariya)	MP16 (Rosoiu)	PF16 (Robinson)	
1700	CLOSE			

Oral presentations at a glance

Energy and Environmental

EE1	Antonio Maia Chaves Neto	<i>Heavy metals nanofiltration using nanotube and electric field by molecular dynamics</i>
EE2	Mohamed Nasreldin	<i>Microstructured electrodes supported on serpentine interconnects for stretchable electronics</i>
EE3	Alexander Wallace	<i>The Effects of Ultrasound on the Electro-Oxidation of Sulfate Solutions at Low pH</i>
EE4	Aranzazu Carmona Orbezo	<i>Performance Optimization of Capacitive Deionization by Potentiostatic Analysis</i>
EE5	Declan Bryans	<i>Surface Treatment of Carbon Felt Electrodes and the Associated Impacts</i>
EE6	Alexander Murray	<i>Electronic tuning and applications of organic redox active charge carriers</i>
EE7	Eileen Yu	<i>Microbial electrosynthesis (MES) for Conversion of CO₂ to Fuels and Chemicals</i>

EE8	<i>Georgia Orton</i>	<i>Towards [FeFe]-Hydrogenase biomimics for H₂ oxidation</i>
EE9	<i>Cameron Bentley</i>	<i>Correlative Electrochemical Microscopy of Li-Ion (De)intercalation at a Series of Individual LiMn₂O₄ Particles</i>
EE10	<i>Edmund Dickinson</i>	<i>Theoretical insights into optimal cell configuration for reliable three-electrode impedance measurements in Li-ion cells</i>
EE11	<i>Ben Craig</i>	<i>DFT modelling of single chains of PEDOT investigating charge storage behaviour in the Al-PEDOT battery</i>
EE12	<i>Ravi Kumar</i>	<i>Protic Ionic liquid Electrolytes for Water Electrolysis</i>
EE13	<i>Andrew Wain</i>	<i>Active site manipulation in MoS₂ hydrogen evolution catalysts by transition metal ion doping</i>
EE14	<i>Samuel Perry</i>	<i>Polymers with Intrinsic Microporosity (PIMs) for Carbon Dioxide Reduction at Gas Diffusion Electrodes</i>
EE15	<i>Liang Wu</i>	<i>Electrochemical oxidation of ammonia to renewable hydrogen in an electrochemical flow reactor</i>

EE16	<i>Nourhan Mohamed</i>	<i>Anodic oxidation of nickel foam in molten KOH for supercapacitor applications: The role of overpotential on capacity and stability</i>
EE17	<i>Pawin lamprasertkun</i>	<i>Capacitance of Graphite/Electrolyte Interfaces</i>
EE18	<i>Dhrubajyoti Bhattacharjya</i>	<i>Effect of electrode processing and cell assembly on the performance of supercapacitor in prototype pouch cell format</i>

Materials and Processes

MP1	<i>Vivek Padmanabhan</i>	<i>Tailoring the Stability of Lithium-Oxygen Battery Electrolytes with the Use of Redox Mediators</i>
MP2	<i>Marie-Laure Doche</i>	<i>Recent developments in electropolishing of additively manufactured 316L stainless steel</i>
MP3	<i>Santiago Pinate</i>	<i>Electrocodeposition of nano-SiC particles under an adapted Pulse-reverse waveform</i>

MP4	<i>Eden May Dela Pena</i>	<i>Research Efforts on the Application of Ionic Liquid Technology for Surface Modification of Metals at the University of the Philippines</i>
MP5	<i>Ruoyu Xu</i>	<i>Nanoporous carbon: liquid-free synthesis and geometry dependent catalytic performance</i>
MP6	<i>Nicola Comisso</i>	<i>Preparation of electrocatalysts for OER via gas bubble templated deposition and galvanic displacement steps: New evidences on secondary oxide growth</i>
MP7	<i>Ignacio Tudela</i>	<i>Sonoelectrochemical degradation of chlorinated organic pollutants in water: 1 plus 1 equals 3?</i>
MP8	<i>Jean-Yves Hihn</i>	<i>Sonoelectrochemistry: reactor characterization tool and for electrochemical processes accelerator</i>
MP9	<i>Lucia Lain Amador</i>	<i>Copper electroformed getter-coated vacuum chambers for particle accelerators: from concept to development</i>
MP10	<i>Donghoon Kim</i>	<i>Enhancing magnetoelectric coupling for the efficient magnetic field induced electrochemical reactions</i>
MP11	<i>Sotiri Mavrikis</i>	<i>Carbon-based Materials for Anodic H₂O₂ Electrosynthesis</i>

MPK1	<i>Luis Fernando Arenas</i>	<i>Catalytic metal coatings on porous and 3D printed electrodes for electrochemical flow reactors: Prospects and the role of X-ray computed tomography</i>
MP12	<i>Konrad Eiler</i>	<i>Electrochemical synthesis of magnetic mesoporous Ni-Pt alloy thin films for hydrogen evolution reaction</i>
MP13	<i>Enrico Daviddi</i>	<i>Revealing spatially-dependent electron-transfer kinetics within a phase-separated conductive polymer blend at the nanoscale</i>
MP14	<i>Emily R. Cross</i>	<i>Electrochemical Gels for Biological Applications</i>
MP15	<i>Leigh Aldous</i>	<i>Thermogalvanic cells: Progress in both fundamental understanding and application</i>
MP16	<i>Sabrina Patricia Rosoiu</i>	<i>Electrodeposition of Ni-Sn alloys and Ni-Sn / rGO composite coatings from deep eutectic solvents and their physicochemical characterization</i>

Physical and Fundamental

PF1	Angel Cuesta	<i>The potential profile across the electrode-electrolyte interface: cation size effects and implications for CO₂ electrocatalysis</i>
PF2	Sarah Horswell	<i>The effect of hydrogen bonding capacity on lipid bilayer structure and function</i>
PF3	Yvonne Grunder	<i>Probing the charge distribution at the electrochemical interface</i>
PF4	Gilles Moehl	<i>Following the formation of Electrochemically Assisted Self Assembled silica films in real time by in situ Grazing Incidence Small Angle X-ray Scattering</i>
PF5	Reena Saxena	<i>Modified Multiwall Carbon Nanotubes (MWCNT) based Electrochemical sensor for Thallium in water</i>
PF6	Anthony Lucio	<i>Optimisation of boron-doped diamond electrodes for the quantitative detection of free chlorine species at high and low concentrations</i>

PF7	<i>Albert Schulte</i>	<i>Greening analytical voltammetry through electrochemical cell miniaturization</i>
PF8	<i>Rhushabh Maugi</i>	<i>Aptamers and Peptides – Opening new Libraries</i>
PF9	<i>Jacqueline Hicks</i>	<i>Tracking Ultrashort Carbon Nanotube Wireless Electrodes Within NG108 Cells</i>
PF10	<i>Bo Hou</i>	<i>Chemically Encoded Self-Organized Quantum Chain Supracrystals with Exceptional Charge and Ion Transport Properties</i>
PF11	<i>Anthony Betts</i>	<i>Detection of Non-Steroidal Anti-Inflammatory Drugs Utilising Cerium Dioxide Nanoparticles Deposited on Modified Screen Printed Electrodes</i>
PFK1	<i>Guy Denuault</i>	<i>Studies of the influence of molecular oxygen on the formation and reduction of oxides on Pt electrodes</i>
PF12	<i>Ioannis Katsounaros</i>	<i>Monitoring products of electrochemical reactions in real time</i>
PF13	<i>Haoliang Huang</i>	<i>The role of SnO₂ in the bifunctional mechanism of CO oxidation at Pt-SnO₂ electrocatalyst</i>

PF14	<i>Simon Reeves</i>	<i>Fundamental Electrochemistry of P-Block Halometallates in Non-Aqueous Solvents</i>
PF15	<i>Aruna Ivaturi</i>	<i>Non-invasive Non-Enzymatic Electrochemical Glucose Sensors based on Metal Oxide Nanostructures</i>
PF16	<i>Caoimhe Robinson</i>	<i>Label-free Electrochemical Immunosensor for the Detection of IgG in Calf Serum</i>

Corrosion Science

C1	<i>Koushik Bangalore Gangadharacharya</i>	<i>Thin electrolyte thickness measurements for atmospheric corrosion modelling</i>
C2	<i>Liya Guo</i>	<i>The corrosion behaviour of magnetocaloric alloys under magnetic field conditions</i>
C3	<i>Yishuang Yang</i>	<i>Study on ion penetration within polymeric binder by ion selective electrode</i>
C4	<i>Chinmay Pardeshi</i>	<i>Calibration of a Pitting Corrosion Simulation Model using Pit Population Data obtained from White Light Interferometry</i>

C5	<i>Amelia Langley</i>	<i>Chaotic Copper Corrosion: The Influence of Dissolved Gas on the Anodic Passivation of Copper in Model Seawater</i>
C6	Lukas Korcak	<i>Introducing new electrolyte for copper plating with aluminium</i>
C7	<i>Mariana Folena</i>	<i>Evaluation of the role of acetic acid in CO₂ Top of line Corrosion using real-time corrosion measurements</i>
C8	<i>Amir Shamsa</i>	<i>Influence of Ca²⁺ ions on corrosion product formation kinetics and characteristics in CO₂ environments</i>
C9	<i>Clive Harrison</i>	<i>Electrochemical Corrosion Monitoring in the Nuclear Industry</i>
C10	<i>Sarah Leeds</i>	<i>Monitoring corrosion protection of pipelines utilizing the main survey techniques</i>
C11	<i>Hunter Thomson</i>	<i>Chemical Qualification of Corrosion Inhibitors in the Oil & Gas Industry: Impact of Test Approaches on Performance during Laboratory Screening</i>
C12	<i>Paul Lambert</i>	<i>Long-Term Corrosion Monitoring of Steel and Reinforced Concrete Infrastructure</i>
C13	<i>Steve Paterson</i>	<i>Steve Paterson: Paul McIntyre award recipient talk</i>

C14	<i>David Kumar</i>	<i>The study of hot water corrosion for fusion reactor cooling circuits</i>
C15	<i>Ivan Spajić</i>	<i>Atomic layer deposition thin films from Al₂O₃ and HfO₂ as protection of commercially pure titanium for biomedical applications</i>
C16	<i>Salil Sainis</i>	<i>Influence of size and distribution of intermetallic particles in Al-Si cast alloys on the cerium conversion coating deposition</i>
C17	<i>Jessica Moulton</i>	<i>Modelling the Behaviour of Aluminium Flakes in Marine Coatings Through the Use of Agar Gels</i>
C18	<i>Alvaro Sanchez Araujo</i>	<i>Mechanisms of life enhancement by laser shock peening surface treatment of 7XXX aluminium alloys</i>
C19	<i>Isehaq Al-Nafai</i>	<i>Electrochemical Study of the Effects of Adding Pigments to Zinc-Rich Coatings</i>
C20	<i>Katarzyna Rzeszutek</i>	<i>Reducing the zinc content in protective marine coatings</i>

Posters at a glance

P1	Ehsan Rahimi	Evaluation of electronic properties and Volta potential distribution of CoCrMo alloy in presence of protein molecule by SKPFM and electrochemical measurements
P2	Bruno Ribeiro	<i>Effect of anodizing parameters on the formation of TiO₂ nanotubes produced on titanium grade 2 and titanium grade 5</i>
P3	Víctor Rojas	<i>Electrochemical Insertion of Lithium Ion in Copper Hexacyanoferrate employed as Cathode Material in Rechargeable Batteries.</i>
P4	Mirsajjad Mousavi	<i>In-situ study of corrosion of commercial SD memory card by local electrochemical techniques</i>
P5	Isobel Wilson	<i>Modification of Carbon Electrodes from Acylated Pyridine Species: A Study of the Steric and Electronic Effects of Pyridine Radicals on the Grafting Process</i>
P6	Sanchita Singhal	<i>Comparison Between Laminate and Copolymers of 1,3,5-Tris(N-carbazolyl)benzene and 3,4-ethylenedioxythiophene: A Spectroelectrochemical Study</i>
P7	Budi Putra	<i>Materials and Architectures for Ionic Rectifiers</i>
P8	Gary Harlow	<i>Studies towards applied anodic oxides</i>

P9	<i>Mohammad Danish Khan</i>	<i>Azo dye treatment with simultaneous electricity production in an air cathode microbial fuel cell coupled activated sludge process</i>
P10	<i>Bethany Lomax</i>	<i>Lunar resource extraction: the Metalysis-FFC process</i>
P11	<i>Matthew Rees</i>	<i>Development of Ionomer Functionalised Carbon Paste Electrode and Modified Glassy Carbon Electrode for Detection of Mercury in Sea Water</i>
P12	<i>Tammy Nimmo</i>	<i>Time and pH Dependence of the Open-Circuit Potential of Pt and Au in Aqueous Electrolytes in the Presence and Absence of O₂</i>
P13	<i>Johanna Ranninger</i>	<i>On-line stability investigations of platinum electrodes in non-aqueous systems using a scanning flow cell</i>
P14	<i>Anja Drame</i>	<i>Imprinted polyaniline-based screen printed electrodes for sensing of toxic organic compounds</i>
P15	<i>Yanpeng Suo</i>	<i>Influence of the acidity of ionic liquids on the double layer properties of the Pt/PIL interface</i>
P16	<i>Jack Beane</i>	<i>Operando Studies of the Potential Dependent Structure and Stability of Cu(111) Electrodes</i>
P17	<i>Nuttanun Kutrakul</i>	<i>Carbohydrate Amperometry with Pyranose-2-Oxidase Biosensors</i>

P18	<i>Mark Buckingham</i>	<i>Thermogalvanic and pseudo-thermocapacitive conversion of low-grade thermal energy to electricity through gelled-electrolyte thermoelectrochemistry</i>
P19	<i>Alexandra Borrill</i>	<i>Electrochemical Fabrication and Characterisation of Copper Oxide Structures on Boron Doped Diamond for CO₂ reduction</i>
P20	<i>Imogen Heaton</i>	<i>Peptide Nanocarriers for the Detection of Heavy Metal Ions Using Resistive Pulse Sensing</i>
P21	<i>Yuwei Pan</i>	<i>A paper-based electrochemical aptamer biosensor for monitoring antibiotic pollution</i>
P22	<i>Declan Bryans</i>	<i>Characterisation of a 200 kW/400 kWh Vanadium Redox Flow Battery</i>
P23	<i>Kasia Clarke</i>	<i>Initiation of Stress Corrosion Cracking in Zircaloy Nuclear Fuel Cladding Examined using High-Speed Atomic Force Microscopy</i>
P24	<i>Marcus Pollard</i>	<i>Additively Manufactured Flow Resistive Pulse Sensors</i>
P25	<i>Huanxin Li</i>	<i>Ultra-fast Rechargeable Next-generation Batteries</i>
P26	<i>Iryna Kozina</i>	<i>Corrosion study of magnesium alloys coated with chitosan coating in the Hank's solution for biomedical applications</i>

P27	<i>Yoshio Matsuzaki</i>	<i>Effect of Hole-Blocking Layer of Lanthanum Tungstate on Chemical Potential Profile in Proton-Conducting BaZr_{0.8}Y_{0.2}O_{3-δ} Electrolyte</i>
P28	<i>Jiayu Liu</i>	<i>Effects of oxygen on degradation of carbon supported platinum catalysts</i>
P29	<i>Maurizio Gulino</i>	<i>Tissue response to neural implants: the use of model systems towards new design solutions of implantable microelectrodes</i>
P30	<i>Neelam Mughal</i>	<i>Molecular Metal Oxides for High Energy Battery Cathodes</i>
P31	<i>Maksudul Hasan</i>	<i>Understanding the Capacity Limitation of Na-O₂ Batteries</i>
P32	<i>Steven Gibney</i>	<i>Development of an in vivo sensor for therapeutic monitoring of tricyclic antidepressants</i>
P33	<i>Christine Enowmbi Tambe</i>	<i>Effect of additives on electroforming of nickel</i>
P34	<i>Liv Mølmen</i>	<i>Pitting corrosion on coated stainless steel PEMFC flow plates</i>
P35	<i>Calum Hope</i>	<i>Capacitive Deionisation for Phosphate Recovery</i>
P36	<i>Alfie Wills</i>	<i>A selectivity switch in the alternating potential electrolysis of 4-methylanisole</i>

P37	Jack Jordan	<i>Redox-driven Nano-confinement, Wiring, and Protection of Polyoxometalates in Carbon Nanotubes</i>
P38	Shahid Rasul	<i>Electrochemical reduction of CO₂ to formate using a modified SnO₂ with Sb and Zn</i>
P39	Nicole Briones	<i>Synthesis of ZnO-NRs/CdS/AuNPs nanocomposite and its potential application in to the fabrication of an electrochemical aptasensor.</i>
P40	Dahlia Eldosoky	<i>Promoting the Remineralisation of Subsurface Caries Lesions</i>
P41	Germán Brito	<i>Study of the electrolytic influence in the electrochemical synthesis and nanostructures formation of MoS₂: effects on the optimization of the electrocatalytic performance in the hydrogen evolution reaction (HER)</i>
P42	Emre Dokuzparmak	<i>Development of electrochemiluminescence (ECL) sensor modified with nafion film for detection of methamphetamine and its main metabolites</i>
P43	Eleni Andreou	<i>Electroforming of Large-Scale Nickel Structures for Leading-Edge Energy, Aerospace and Marine Applications</i>
P44	Athanasios Stergiou	<i>Use of Mediators for the reduction of Nitroarenes</i>

P45	Robert Euan Wilson	<i>Development of an autonomous, in situ cleaning, electrochemical sensor for dissolved inorganic carbon detection at full ocean depth</i>
P46	Harry Michael	<i>3D and 4D Characterisation of Dilated Lithium Ion Battery Electrode Microstructure using in-Situ Dilatometry and X-ray Tomography</i>
P47	John Edet	<i>Investigation of Water Absorption Profile of Mineral Wool Insulation</i>
P48	Keenan Smith	<i>Fabrication of a new composite polymer electrolyte membrane by ultrasonic spray casting</i>
P49	Fiona Moore	<i>Developing a Glucose Sensing System using Nanoband Electrodes</i>
P50	Anmol Jnawali	<i>Virtual Unrolling as a predictive analysis on the mechanism and location of degradation of cylindrical batteries</i>
P51	Anam Safri	<i>Titania Anchored Graphene Oxide Nanocomposites as Bifunctional Agents for Solar Disinfection (SODIS)</i>
P52	Nikoloz Nioradze	<i>Electrochemistry of Copper/Cysteine/Graphene Composite</i>
P53	Shaon Debnath	<i>Fibre-Optic pH Sensor Based on Silver Nanoparticles for Harsh Environments</i>

P54	Alice Llewellyn	<i>In-Operando XRD for Understanding the Degradation Mechanisms of Lithium Ion Battery Materials</i>
P55	Robert Jacklin	<i>Advanced Autoclaves for High Temperature High Pressure Corrosion Assessment</i>
P56	Francisco Willian S. Lucas	<i>Electrodeposited Intermetallic Tin-Antimony as a Novel Electrocatalyst Material for Carbon Dioxide Reduction to Formate</i>
P57	Yucheng Wang	<i>Cu-based Catalysts for Electrochemical Reduction of CO₂ using Graphene aerogel Gas Diffusion Electrode Type Device</i>
P58	Akash Ratnayaka	<i>Conformal electrodeposition of layered manganese oxide intercalated with potassium as a pseudocapacitive functionalisation of carbon electrodes in Supercapacitors</i>
P59	Terence Liu	<i>Cost Reduction of Pt Based Electrochemical Oxygen Reduction Reaction Catalysts</i>
P60	Julien Biscay	<i>Disposable Electrochemiluminescence Based Sensor for Rapid Drug Detection</i>
P61	Elaheh Esmaeili	<i>Sustainable Papermaking Using Foam</i>
P62	Sarah A. Alshehri	<i>Developing polymer Battery materials</i>

P63	Caitlin McLean	<i>Characterising the electrochemical response of novel 3D printed CNT electrodes to the virulence factor pyocyanin</i>
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Invited Speakers

Faraday Medal: Martin Winter

Electrolyte –The Unrecognized Hero of the Battery World

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Abstract:

Among the four vital parts of each battery, namely anode, cathode, separator and electrolyte, the first two have been recognized for gathering all the headlines and glamour so far. However, it is the electrolyte as an unhonoured hero of the battery world, that plays a central key role in terms of design and control of battery cell processes as well as influencing material interactions, performance, long-term stability, cost and last but not least the safety of all types of current and future batteries. The right choice of electrolyte and its components hand in hand with *ad hoc* interfacial/interphasial chemistries determine the relevant properties that dictate the overall performance of rechargeable batteries.¹

Still, the overall performance of batteries is limited by the fundamental behavior of the materials used, even for the present and potential future high-performers, the lithium ion and lithium metal batteries. Market demands for higher-energy and higher-power lithium-based batteries force researchers towards identifying new liquid electrolyte formulations, polymer and ceramic solid as well as hybrid electrolytes of them.²⁻⁴ By combining different electrolyte constituents, electrolyte and battery cell chemistry can be optimized and tailored for a specific application. Profound study by means of selected electrochemical, analytical and spectral techniques supported by theoretical calculations and simulations as well as integration of gained knowledge comprising vital parameters and processes is the key towards desired advancements of lithium-based cell chemistries and their effective performance. In this presentation, emphasis is set on the physicochemical, electrochemical and safety aspects relevant for cell operation. With this in line, performance characteristics, current limitations and recent breakthroughs will be critically reviewed and discussed.

References

1. N. von Aspern, G.-V. Rösenthaller, M. Winter and I. Cekic-Laskovic, *Angewandte Chemie*, accepted, DOI: 10.1002/anie.201901381 and 10.1002/ange.201901381.
2. I. Cekic-Laskovic, N. von Aspern, L. Imholt, S. Kaymaksiz, K. Oldiges, B. R. Rad and M. Winter, *Topics in Current Chemistry*, **375**, 2017, 37-100.
3. R. Schmuch, R. Wagner, G. Hörpel, T. Placke and Martin Winter, *Nature Energy*, 2018, **3**, 267–278.
4. L. Imholt, D. Dong, D. Bedrov, I. Cekic-Laskovic, M. Winter and G. Bruncklaus, *ACS Macro Letters*, 2018, **7**, 881-885.

Castner Medal: Keith Scott

Electrochemical Engineering Science. 40+ years of process and materials developments

Keith Scott

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Abstract:

Electrochemical Engineering Science (EES) is a multidisciplinary research activity exploring applications of electrochemistry in many areas such as; energy storage, power generation, electrolysis, green energy, sensors and environmental protection and treatment. The EES field includes novel research for fuel cells, batteries, hydrogen generation, bioelectronics, cellular and microbial catalysis, etc and explores new techniques to synthesise electro-catalyst, nanomaterials and membrane materials and optimise electrode structures. EES has at its core electrochemical and spectro-chemical analysis to understand fundamental mechanisms and the exploration of electrochemical technologies at the pilot scale for electrolysis, power generations and more [1].

Over the last 5 decades major developments have occurred in the electrochemical based industries in areas such as electrosynthesis, batteries, fuel cells, hydrogen technologies, enzyme and microbial fuel cells, effluent treatment and recycling [2]. These have been partly driven by advances in better performing materials and in cell design and operation. This lecture will take a personal, historical perspective of several of the aforementioned applications including electro-organic synthesis, power sources and metal recovery and recycling. Many of these have been commercialised or developed to high TRLs, only to be shelved due to poor long term materials performance, more economic competition, or loss of market potential.

References

1. F Goodridge and K Scott. *Electrochemical Process Engineering: A Guide to the Design of Electrolytic Plant*. 1995. Springer
2. K Scott. *Sustainable and Green Electrochemical Science and Technology* 2016, J Wiley

Parsons Medal: Alison Parkin

Fourier transforming protein film electrochemistry

Alison Parkin

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Abstract:

Redox reactions underpin the mechanisms of life and understanding how bacteria use non-precious transition metal elements to activate H₂-production, CO₂-reduction and N₂-fixation is important for learning how to design sustainable energy catalysts. Notably, such multi-electron redox reactions always occur within enzymes that contain electron-transfer centres that act as a “wire”, transferring electrons between the protein surface and the catalytic centre buried in the middle of the protein. While film-electrochemistry has proven to be a powerful technique for probing the catalytic reaction mechanisms of such enzymes, it has been challenging to unpick the mechanisms of reversible electron-transfer along the protein “wire” using traditional voltammetric methods. In collaboration with Prof Alan Bond (Monash) and Prof David Gavaghan (Oxford), the Parkin group is developing protein film alternating current voltammetry as a method which can simultaneously measure the catalytic redox activity and reversible electron transfer processes of metalloenzymes. This insight has been used to guide the design of protein mutations that produce re-wired H₂-enzymes which are more active and efficient H₂-production bio-catalysts.

Evans Award: Tetsuo Shoji

Mechanics and Mechanisms of Stress Corrosion Cracking

– Role of hydrogen as all-round player –

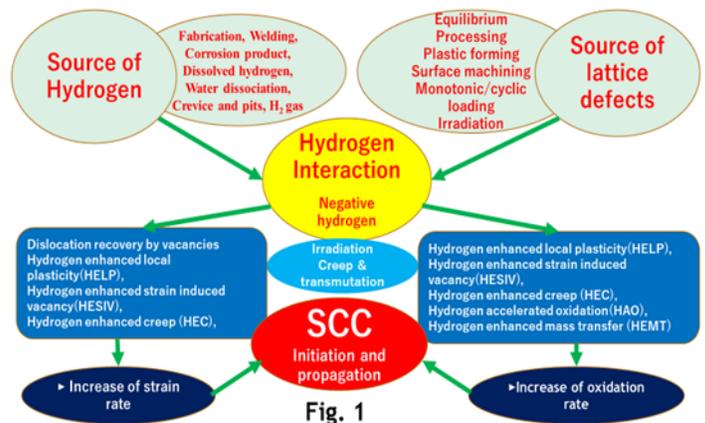
Tetsuo Shoji,

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Technology Beijing, Beijing China

Abstract: Stress corrosion cracking is a well-known degradation mode in various industries such as oil refinery plants, chemical process plants and various energy conversion plants. Stress corrosion cracking is one of the typical degradation modes caused by synergy of multiple critical factors such as mechanics, materials and environments. There have been numerous research publications on phenomenological understanding of cracking mechanisms but still fundamental mechanistic understanding needs more work to find a solution for industries. Recent atomistic modelling studies of metallic oxidation by water suggest a significance of the role of hydrogen in oxidation¹ and some experimental observations had been reported in hydrogen-vacancy clusters and promotion of diffusivity^{2,3}. Hydrogen plays a role in various aspects such as an interaction with surrounding atoms through electron transfer from metal to hydrogen to form negatively charged/positively charged hydrogen in transition metals and their alloys, with atomic defects to form hydrogen-vacancies clusters, with existing surface oxides to form degraded(non-protective) oxides, with grain boundary or interface to promote diffusivity.

Fig. 1 shows the interaction of various mechanics, materials and environmental factors in a process of SCC crack initiation and propagation including macroscopic stress field. A cooperative research work is going on to verify this scenario of critical role of hydrogen in SCC with electric power generation industry. Also, new alloys were designed and fabricated based upon this possible role of hydrogen on oxidation and SCC as well as oxygen and verification work is also going on to show an excellent SCC resistance under the environment where hydrogen is phenomenologically known to play a critical role in the system. There need much more works for better mechanistic understanding of SCC initiation and propagation with special emphasis on all-round atom, hydrogen. Some of the on-going work of PAS will be introduced in the presentation.



References

- 1 N. K. Das, K. Suzuki, Y. Takeda, K Ogawa and T. Shoji, 'Corrosion Science', Vol. 50, No. 6, June 2008, 1701.
- 2 Yuh Fukai and Hidehiko Sugimoto 2007 J. Phys.: Condens. Matter 19 436201.
- 3 E. Hayashi, Y. Kurokawa, Y. Fukai: Phys. Rev. Lett., 80(1998), 5588.

Invited Lecture by EPSRC

Electrochemical Sciences: the funding landscape

Marianne Rolph

Engineering and Physical Sciences Research Council, UK Research and Innovation, Polaris House, North Star Avenue, Swindon, SN2 1FL

Abstract: An overview of the Electrochemical Sciences research portfolio at EPSRC, and the current and future funding landscape.

Fleischmann Lecture:

Karen Faulds

Multiplexed and Sensitive Bioanalysis using SERS and SESORS

K. Faulds¹, K. Gracie, S. Mabbott¹, H. Kearns¹, L.E. Jamieson¹, F. Nicolson¹, A. Kapara¹, D. Graham¹, R. Goodacre²

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Abstract:

Surface enhanced Raman scattering (SERS) was first demonstrated by Fleischmann in 1974¹ and since then it has developed into a highly researched technique with several advantages over competitive approaches in terms of improved sensitivity and multiplexing. However, the lack of quantitative data relating to real samples has prevented more widespread adoption of the technique. Detection of specific biomolecules is central to modern biology and to medical diagnostics where identification of a particular disease is based on biomarker identification. Many methods exist and fluorescence spectroscopy dominates the optical detection technologies employed with different assay formats. We have made great progress in the development of SERS as a quantitative analytical method, in particular for the detection of biomolecules. Another advantage of SERS over existing detection techniques is that of the ability to multiplex which is limited when using techniques such as fluorescence. A focus of our research is developing multiplexed bioassays using SERS to allow the simultaneous measurement of multiple species in one measurement.

During this presentation we will demonstrate the development of new bioanalytical assays based upon SERS which have been used successfully for the detection of bacterial pathogens using modified SERS active probes.² Biomolecule functionalised nanoparticles have been designed to give a specific SERS response resulting in discernible differences in the SERS which can be correlated to the presence of specific pathogens. In this presentation the simultaneous detection and quantitation of 3 pathogens within a

multiplex sample will be demonstrated.³ Also presented will be our recently published work on the use of nanoparticles functionalised with resonant Raman reporter molecule for the visualisation of a 3D breast cancer tumour models using Spatially Offset Raman (SORS) combined with SERRS (SESORRS).⁴

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Energy and Environmental al

Oral Abstracts

***Heavy metals nanofiltration using nanotube and electric field by
molecular dynamics***

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Abstract

Nanofiltration has applications in various areas, such as desalination and separation of organic materials, among others materials floating in the water or air, depending on the structure of its holes of nanomaterial diameter. It is a very important technique because it promotes the separation of several kinds molecules from water, but it uses very high temperature and high pressure demanding very high energy and become economically highly expensive. Nanotubes demonstrates high selectivity as filter in the emerging research field. Our theoretical model has external electric field using the anode and cathode near of the nanotube openings. Also, the reference electrode will be on the nanotube. The internal van der Waals force of nanotube will select the heavy metal inside of the nanotube and only let pass the water molecules. Our results presents the thermodynamical behavior of this system under several conditions of temperature and electric field.

Microstructured electrodes supported on serpentine interconnects for stretchable electronics

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Abstract

Recently, emerging technologies like 3D-printing has doped the development of flexible electronics to design foldable displays, bioprosthesis, wearable devices, etc. Particularly, stretchable microelectronics able to adopt easily complex shapes like emulating the human body have attracted attention for smart textiles. To achieve the fabrication of such devices, innovative technologies and new designs involving the use of materials with advanced mechanical properties are required. In this work, the fabrication of lithium nickel manganese oxide (LNMO) micropillar electrodes on Al serpentine interconnects that can be stretched up to 70% without structural damaging has been achieved by laser patterning technique. Unlike compact and continuous electrode thin-films, we show that under mechanical strains, arrays of vertical micropillar supported on serpentine are carrying empty spaces that can prevent the formation of cracks and the electrode delamination.

This innovative approach has been used to fabricate flexible micro-battery for powering a smart contact lens. The innovative micro battery approach relies on two flexible substrates assembling consisting of polydimethylsiloxane (PDMS) supporting 1 cm² surface area disk of LNMO and LTO serpentine electrodes separated by a gel polymer electrolyte.

Interestingly, the micro battery shows in the first reversible cycle a charge and discharge areal capacities of 1.22 mAh·cm⁻² and 1.196 mAh·cm⁻², respectively. Regarding the cycling performance, the LTO/ polymer/LNMO micro battery has been assessed at fast kinetics for 30 cycles. The micro battery delivers 73.5 μAh·cm⁻² at **6C**, 47 μAh·cm⁻² at **12C** and 32 μAh·cm⁻² at **20C** with a remarkable stability.

The Effects of Ultrasound on the Electro-Oxidation of Sulfate Solutions at

Low pH

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Abstract

The electrochemical oxidation of sulfate solutions is a well-established route for the generation of powerful oxidizing agents such as persulfate. However, the effect of coupling ultrasonic irradiation with this process has attracted little attention. In this presentation, the effects of low-intensity sonication on solution-phase oxidant generation during electrochemical oxidation of sulfate solutions at low pH are discussed. Our results show that at high current densities/high sulfate concentrations, ultrasonic irradiation has little to no effect on the Faradaic and absolute yields of solution-phase oxidants. However, at lower current densities and sulfate concentrations, the amount of these oxidants in solution appears to decrease under ultrasonic irradiation. A mechanism explaining these results is proposed (and validated), whereby anodically-generated sulfate and hydroxyl radicals are more effectively transported into bulk solution (where they are quenched) during sonication, whereas in the absence of an ultrasonic field these radicals combine with one another to form more persistent species, such as persulfate.

Performance Optimization of Capacitive Deionization by Potentiostatic

Analysis

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Abstract

Capacitive deionization is a method in which water is desalinated by the application of a potential between two porous electrodes. Previous reports generally consider that the applied potential is usually divided in half for both electrodes and that the active mass of the electrodes plays an important role on the desalination performance obtained from a certain CDI system. In this work, the authors used a potentiostatic method for the determination of the electrochemical stable potential window (ESPW), and thus an optimised mass ratio for the electrode pairings, of a cell made with activated carbon electrodes and a concentrated NaCl aqueous electrolyte. The obtained results demonstrate that the ESPW in which the electrodes would not suffer from Faradaic degradation is 0.4 V for the positive electrode and -0.6 V for the negative electrode, leading to a mass balance ratio of $m^+/m^- = 2.22$. These parameters were then applied to a CDI cell, in which an improvement of 65% of the salt adsorption capacity and of 58% for charge efficiency was obtained when the system was operated with the mass balanced conditions, compared to the equivalent mass case.

Surface Treatment of Carbon Felt Electrodes and the Associated Impacts

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Abstract

Carbon felts are commonly used to increase the active surface area of the electrodes found in many redox flow battery (RFB) systems, primarily the all-vanadium RFB. However, this material requires an activation treatment to improve the performance of the electrode. This treatment, typically thermal oxidation, improves the hydrophilicity (wettability) by increasing the oxide-based functional groups on the carbon surface. This allows for an increase in the solution's permeability of the electrode as well as decreasing the pressure drop associated with the degree of hydrophobicity in the material. In addition, the fluid dispersion throughout the felt's porous structure also improves the total contact made by the electroactive species to these electrodes. This increases the electrochemical efficiencies of these redox flow systems.

The work to be presented investigated the associated impact of these surface treatments and aims to understand the effects that cause these improvements. This was achieved by:

- studying the fluid permeability of the felt materials from various treatments through contact angle measurements and visual observations;
- analysing the functionality of the surface through x-ray photoelectron spectroscopy and infra-red measurements;
- and correlating these points to the electrochemical response achieved through a series of battery cycling and electrochemical impedance measurements.

Investigating the thermal oxidation through the design of the experiment, an optimum set of conditions is reached for the activity of the electrode. Following this, a series of alternative treatment methods are to be discussed in this presentation.

Electronic tuning and applications of organic redox active charge carriers

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Abstract

Redox active organic molecules are increasingly required for a variety of uses including organic redox flow batteries (ORFBs), redox catalysts and in devices for energy conversion. Herein we present firstly a strategy for the use of phase transfer of a soluble anthraquinone sulfonate to enable the high-efficiency production of hydrogen peroxide in flow, without the use of precious metal catalysts. This enables selective H₂O₂ outputs tailored to a desired application without changing the underlying electrochemistry, and additionally circumvents the O₂ solubility limit for direct electrochemical O₂ reduction.

The investigation of quinone counterion properties is also explored, with collaborative efforts towards the understanding from ideal proton-coupled electron transfer (PCET) behaviour upon aggregation in self-assembled systems, even in buffered solutions. Understanding these processes has implications both for the fundamentals of understanding supramolecular PCET, as well as the design of supramolecular pseudo-oligomeric charge storage systems.

We finally present strategies towards all-organic redox flow batteries via the use of new, water soluble non-TEMPO based nitroxyl radicals as tunable platforms for organic energy storage. We also present work on varying alkylation states of common N-heterocycles to yield a recyclable redox mediator for all-organic RFB systems where crossover is mitigated. This 'recyclable' concept presents an alternative to 'bipolar' organic mediators.

Microbial electrosynthesis (MES) for Conversion of CO₂ to Fuels and Chemicals

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Abstract

Microbial electrosynthesis (MES) has provided the cathode-driven approach to the bio-electrochemical system (BES), by converting CO₂ to valuable chemicals of volatile fatty acids (VFAs) particularly acetate. Recently, many studies on MES focused on the economic point of view by producing longer chain VFAs than acetate or alcohols through chain elongation. However, to achieve this target, more fundamental information about the mechanism of electron transfer between the bacteria and cathode is required, particularly while the eventual target is scaling up MES for industrial applications. In this direction, we investigated the role of energy for bacteria provided by the cathodes at different applied potentials (-0.8 and -1.0 V vs. Ag/AgCl) and type of inorganic carbon sources (CO₂ and HCO₃⁻) on the bio-production. The results demonstrated the significant role of pH, biotic H₂ production and gaseous CO₂ on formation of conductive biofilm, acetate and longer chain VFAs bio-production, by suggesting the biotic H₂-mediated electron transfer. Although cyclic voltammograms confirmed the biotic hydrogen evolution in BESs which acted as a mediator between bacterial community and the cathode, it is also possible that direct electron transfer occurred due to the rich biofilm composed of acetogens formed at the cathode of BESs -1000 mV_CO₂.

Towards [FeFe]-Hydrogenase biomimics for H₂ oxidation

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Abstract

The current global energy crisis has driven research into the use of H₂ as a zero emission fuel. Nevertheless, the majority of H₂ is produced via non-renewable routes such as natural gas reforming, and energy release is achieved using the expensive, trace element Pt. Contrastingly, Hydrogenase enzymes reversibly interconvert H₂ into protons and electrons under biological conditions through complex proton-coupled redox chemistry at their active site (fig i) which features a 2Fe2S sub-cluster, a ferredoxin cluster and an azadithiolate ligand.

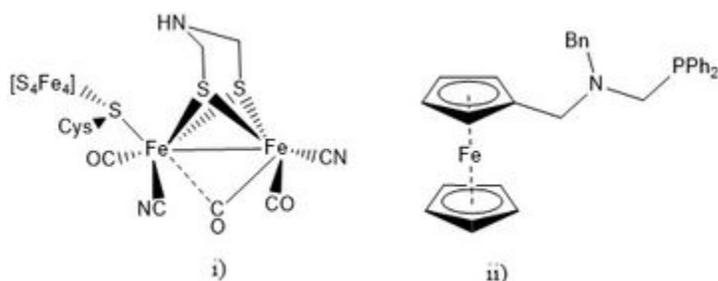


Figure: i) [FeFe]-Hydrogenase active site, ii) aminophosphino ferrocene

To facilitate similar behaviour in biomimics we have incorporated ferrocenyl ligands to mimic ferredoxin with some early successes.¹ Here we report with the incorporation of multifunctional '2-in-1' redox-active ligands (e.g. fig ii) which mimic the behaviour of both ferredoxin and the azadithiolate, and a series of ferrocene-bridged complexes. These complexes exhibit remarkably higher catalytic activities than analogues with more simple ligands and represent significant progress towards biomimics which function as noble metal free catalysts for both H₂ oxidation and H₂ production.

The redox and electrocatalytic properties of these compounds have been investigated in detail using electrochemistry and spectroelectrochemistry, and the findings corroborated by DFT. These results have driven the design of a new generation of catalysts which we expect to demonstrate superior catalytic behaviour.

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Correlative Electrochemical Microscopy of Li-Ion (De)intercalation at a Series of Individual LiMn₂O₄ Particles

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Abstract

As a promising Li-ion battery cathode material in both aqueous and organic electrolytes, spinel LiMn₂O₄ has attracted much attention in recent years due to its large theoretical capacity, high abundance and nontoxicity, although a number of problems remain to be resolved. As with much research in electrochemistry, macroscale electrochemical measurements have mainly been used to study battery materials, which for complex composite electrodes include contributions from the conductive agent, adhesive, as well as the active material. Local structure-activity relationships for battery materials are still somewhat unexplored in regards to individual active particles or particle agglomerates. To address this issue, a correlative electrochemistry-microscopy strategy has been exploited herein, which enables the structure and electrochemical activity of individual particles to be measured and compared directly.

The redox activity (Li-ion intercalation/deintercalation) of a series of individual LiMn₂O₄ particles of known geometry and (nano)structure, within an array, is determined using a correlative electrochemical microscopy strategy. Cyclic voltammetry (current–voltage, *I–E*) and galvanostatic charge/discharge (voltage–time, *E–t*) are applied at the single particle level, using scanning electrochemical cell microscopy (SECCM), together with co-location scanning electron microscopy that enables the corresponding particle size, morphology, crystallinity, and other factors to be visualized. This study identifies a wide spectrum of activity of nominally similar particles and highlights how subtle changes in particle form can greatly impact electrochemical properties. SECCM is well-suited for assessing single particles and constitutes a combinatorial method that will enable the rational design and optimization of battery electrode materials.

Theoretical insights into optimal cell configuration for reliable three-electrode impedance measurements in Li-ion cells

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Abstract

Electrochemical impedance spectroscopy (EIS) is widely used to assess material properties and electrode performance of energy storage materials. The two-electrode EIS response of an electrochemical cell includes contributions from both electrodes, and the electrolyte. A three-electrode setup with a reference electrode (RE) allows – in principle – separation of the cell impedance into two ‘single-electrode impedances’; however, such measurements are sensitive to geometric and physical asymmetries of the cell, and therefore risk yielding inaccurate results for the electrode under investigation.

We simulate a cylindrical Li-ion cell with an integrated concentric ring RE (EL-CELL, Hamburg, Germany). This work extends the theory of EIS artefacts beyond an Ohm’s law description and demonstrates the practicality of solving a Newman-type model on a realistic 2D/3D test cell geometry. We discuss and rationalise possible artefacts occurring in single-electrode impedance data for this cell, including artefacts due to geometric irregularities plausibly arising from laboratory cell assembly. The concentric ring RE configuration is insensitive to precise RE position, but even micron-scale misalignment of electrodes introduces artefacts. We recommend methods to mitigate such errors, including increased separator thickness and the use of additional spacing separators.

Our results urge caution in the interpretation of ‘single-electrode impedance’ data measured in a three-electrode cell configuration. We identify priorities for the experimental validation of our results, and for their incorporation into increasingly robust standard test protocols for the characterisation of active energy storage materials and electrodes, especially considering the dependence of electrode performance on active material properties, electrode composition, and manufacturing method.

DFT modelling of single chains of PEDOT investigating charge storage behaviour in the Al-PEDOT battery

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Abstract

Aluminium batteries are highly promising candidates for next-generation battery systems. With similar gravimetric and better volumetric energy density than lithium, aluminium has the benefits of being highly abundant and affordable, and plates reliably without dendrite formation as a metal anode. The challenge is finding cathodes to complete the battery system, as its trivalent charge causes poor reversibility when stored in cathodes. The most promising Al batteries store chloroaluminate species (e.g. AlCl_4^-) from the $\text{AlCl}_3\text{-EMImCl}$ electrolyte in the cathode when charged. Poly(3,4-ethylenedioxythiophene) (PEDOT) is one such cathode. It combines faradaic and non-faradaic charge storage, and has a high initial discharge voltage of 1.7 V (1-3).

We have investigated single chains of PEDOT using Density Functional Theory (DFT) at various oxidation levels, doped and undoped with AlCl_4^- anions. The dependence of bandgap on chain length, the minimum-energy geometries for doped PEDOT chains, and the maximum doping concentration of AlCl_4^- have been studied. These results begin to explain the behaviour of PEDOT in the cell, particularly the balance between faradaic and non-faradaic charge storage, revealing further exciting opportunities to research this promising new battery system.

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Protic Ionic liquid Electrolytes for Water Electrolysis

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Abstract

Protic ionic liquids (PILs) are promising electrolytes for electrochemical devices, owing to their wide electrochemical stability, good ionic conductivity, low vapor pressure and good thermal stability. Ammonium based protic ionic liquid such as N,N-diethylmethylammonium trifluoromethanesulfonate [Dema][TfO] has been previously demonstrated in intermediate temperature fuel cell. In this study we demonstrate that [Dema][TfO] can be used as an electrolyte for intermediate temperature water electrolysis. The cyclic voltammograms of [Dema][TfO] on platinum show a stable potential window (cathodic limit -0.5 V and anodic limit +2 V vs SHE) which is beyond the thermodynamic water decomposition potential of 1.23V. The hydrogen evolution reaction on Pt electrode in, "as prepared" [Dema][TfO] and 10% [Dema][TfO] in water exhibits two on-set potentials for hydrogen evolution. The first onset potential at -0.37 V (vs Ag/AgCl) results in a limiting current density of 0.015 mA/cm² and the second on-set at -0.68 V vs Ag/AgCl without any mass transport limitation. The in-situ mass spectrometric analysis confirmed hydrogen evolution at both potentials. The oxygen evolution reaction on IrO₂ electrocatalyst in as prepared [Dema][TfO] also shows two onset potentials and in 10% [Dema][TfO] in water the oxygen evolution occurs without any limiting current.

Active site manipulation in MoS₂ hydrogen evolution catalysts by transition metal ion doping

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Abstract

Electrochemical water splitting is becoming increasingly attractive as a means to convert electrical energy into chemical energy, allowing versatile energy storage in the form of hydrogen fuel. Despite increasing uptake of electrolyser technologies, they currently rely on rare and expensive platinum group metal catalysts, so efforts are underway to develop new catalysts based on more sustainable, earth-abundant materials. Recently, molybdenum disulfide (MoS₂) has received significant attention as an alternative catalyst for the hydrogen evolution reaction (HER) due to its high electrocatalytic activity and acid-stability. However, further performance enhancement is required in order to compete with Pt and, to that end, transition metal doping of MoS₂ has been explored as a route to enhancing its catalytic activity.

In this work, cluster beam deposition was employed to produce precisely controlled cobalt-doped MoS₂ clusters (MoS₂-Co). We demonstrate that, in contrast to previous observations of performance enhancement in MoS₂ by nickel doping (MoS₂-Ni), the introduction of Co has a detrimental effect on HER activity. The contrasting behaviours of Ni and Co doping are rationalised by density functional theory calculations, which suggest that HER-active surface vacancies are deactivated by combination with Co dopant atoms, whilst their activity is retained, or even partially enhanced, by combination with Ni dopant atoms. Furthermore, the adatom dopant-vacancy combination kinetics appear to be faster in MoS₂-Co than for MoS₂-Ni. These findings highlight a fundamental difference in the influence of transition metal doping on electrocatalytic performance of MoS₂ and stress the importance of considering surface atomic defects when predicting its behaviour.

Polymers with Intrinsic Microporosity (PIMs) for Carbon Dioxide Reduction at Gas Diffusion Electrodes

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Abstract

The substitution of fossil based production routes with new, innovative synthesis strategies for bulk chemicals and fuels will become a strategic goal within the next decade. One promising avenue is the use of CO₂ as a carbon source in the synthesis of useful materials such as methanol or ethylene.^[1] Such reactions have been successfully demonstrated at copper electrodes, which seem uniquely qualified to give a range of possible end products for CO₂ reduction.^[2] Unfortunately, this success is also the major limitation to this method, as the wide number of possible products results in selectivity issues when trying to produce one specific target molecule.

Recent works in the fuel cell community have shown that polymers with intrinsic microporosity (PIMs) enhance oxygen reduction and hydrogen oxidation activities by binding gases close to the surface of the electrode, facilitating electron transfer.^[3] This is of interest to the CO₂ reduction community, since reduction to ethylene involves twelve electrons, so increased residence time at the electrode can be expected to increase ethylene selectivity. This work shows a clear impact of PIMs on the selectivity and stability of copper gas diffusion electrodes applied to CO₂ reduction, presenting a simple means of improving existing successful catalyst materials.

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Electrochemical oxidation of ammonia to renewable hydrogen in an electrochemical flow reactor

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Abstract

Ammonia is a pollutant. One of the main sources of ammonia is from the excreta of animals and humans. Pigs in particular excrete high levels of urea which is rapidly converted to ammonia which can affect animal health. Water treatment companies spend millions of pounds each year removing ammonia from their digestate waste streams. It is possible to electrochemically oxidise ammonia to inert nitrogen and couple its removal with hydrogen evolution. Hydrogen is seen as the perfect environmentally friendly fuel of the future. It can be used in fuel cells to generate electricity and produce only water as a harmless by-product.

This paper describes the anodic oxidation of ammonia using thermally prepared IrO₂ (TDIROF) electrodes in a C-Flow laboratory electrochemical cell. The effects of electrolyte pH, mean linear flow velocity and operating temperature were investigated on the anodic reaction. Scanning electron microscope (SEM) and energy dispersive x-ray spectroscopy (EDS) were carried out to assess electrode stability. The cell voltage was monitored to detect any decrease in electrode performance. The destruction of ammonia was followed using an ion-selective electrode. Experimental tracer visualisation studies were carried out to determine the hydraulic flow patterns and evaluate the hydrodynamics of fluid passing through the C-flow electrochemical cell.

This work has demonstrated the feasibility of combining the electrochemical oxidation of a pollutant with the formation of green fuel, hydrogen. The process proposed promises environmental and cost-saving benefits for the farming and water treatment industries.

Anodic oxidation of nickel foam in molten KOH for supercapacitor applications: The role of overpotential on capacity and stability

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Abstract

Nickel oxy-hydroxide has been extensively studied as an active material for supercapacitors because of its low cost and high capacity. These properties along with wide potential range is attained through the defective structure, with high amount of interlamellar water. Direct synthesis of Ni(OH)₂ on the surface of Ni substrate is preferred as it would facilitate the charge transfer and consequently increase the capacitance of the active material. Production of the active material as a stable film by anodic oxidation in molten salt is a privileged technique because the resulting coating exhibits well adhered layer with controlled thickness and composition of the film. Moreover, alkaline molten salt medium allows oxidation of the Ni surface at lower potential compared to the aqueous solutions.

In this study, anodic dissolution and passivation of nickel foam in molten KOH is investigated by using potentiodynamic and potentiostatic techniques. Three-electrode cell consisting of Ni foam as a working electrode and as a pseudo-reference electrode and graphite crucible as a counter electrode was used. The polarization behavior was studied within the overpotential range between 0 to 1.6 V vs Ni at 200°C. The properties of the Nickel oxy-hydroxide produced at different constant overpotentials was studied. Nine electrodes were prepared by anodic polarization at constant potentials of (0.11, 0.16, 0.25 , 0.6, 1.2,1.25 and 1.3 V) each for 20 minutes. The structure and morphology of the layers were investigated by FTIR, XRD and FE-SEM. The electrochemical performance was studied by cyclic voltammetry and electrochemical impedance spectroscopy.

EE17 – Sheelagh Campbell Award

Capacitance of Graphite/Electrolyte Interfaces

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Abstract

There is enormous interest in developing new materials for energy storage, capacitive deionisation and electrowetting applications. The advent of graphene and other 2D materials has catalysed a huge activity in the application of these materials in various contexts, sometimes – however- with a questionable physical basis. To give an example, the theoretical capacitance of graphene has been widely reported to be 550 F g^{-1} on a gravimetric basis (areal capacitance of $21 \mu\text{F cm}^{-2}$, ignoring the contribution of any quantum capacitance). This value assumes the same interfacial properties of a metallic and graphene (or indeed graphite) electrode, giving the equivalent Helmholtz capacitance. We have investigated this topic by using highly ordered pyrolytic graphite (HOPG) as the bulk analogue of graphene as a model system, from which to understand the capacitance properties. Herein, we provide the first demonstration of the relationship between cation/anion identity and capacitance at the basal and edge plane of HOPG [1]. It is found that the capacitance at the basal plane depends on the ion identity, providing a range in capacitance from ca. 3 to $10 \mu\text{F cm}^{-2}$. In contrast, the edge planes display capacitance ca. 100 times higher than those of the basal plane, attributed to pseudo-capacitive processes associated with the presence of quinone groups and independent of the ion identity. This work improves understanding of capacitance of carbonaceous materials in general and so permits their continued development in many applications.

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Effect of electrode processing and cell assembly on the performance of supercapacitor in prototype pouch cell format

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Abstract

Supercapacitors are a key technology for future renewable energy storage due to their high power density, long cycle life, low maintenance cost and better safety compared to rechargeable batteries.[1] Although the commercial manufacturers mostly prefer the cylindrical cell design for fabrication of large supercapacitors, the alternative pouch cell design are extensively being adopted for a range of applications due to many advantages such as smaller dead volumes both in the cell and on the module level, lower component requirement, lightweight and higher degrees of design freedom.[2] On the other hand, the laboratory scale testing and validation of the different supercapacitor components are mostly carried out using a very small quantity of materials assembled in Swagelok cell or coin cell set up. At this level, many parameters related to electrode processing and cell-assembly such as electrode thickness, calendaring conditions, current collector surface, electrode composition and mass loading are given less emphasis. However, these parameters significantly affect the overall electrochemical performance in terms of capacity, durability, power and energy density in a commercially viable cylindrical or pouch cell. This report summarizes different problems related to electrode processing and cell assembly parameters during a prototyping effort of supercapacitor from Swagelok cell to a pouch cell using state of the art electrodes in the standard organic electrolyte and compared with an industrially manufactured electrode. Finally, multi-layer pouch cell with 25 F capacitance is demonstrated using the best-optimized parameters, which deliver higher specific energy compared to commercially available 25 F cylindrical cell.

Materials and Processes

Oral Abstracts

Tailoring the Stability of Lithium-Oxygen Battery Electrolytes with the Use of Redox Mediators

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Abstract

The rechargeable aprotic lithium-oxygen (Li-O₂) battery is a promising next-generation energy storage technology, but its practical realization is hampered by several challenges. Though a substantial progress has been made in the recent years, the challenges remain and there is still lack of adequate fundamental understanding. Here we present *in situ* and operando methodologies evaluating the feasibility of a dual redox mediator strategy to address the electrolyte stability issue in Li-O₂ batteries. With the help of *in situ* Raman spectroscopy and online mass spectrometry studies, we demonstrate how the use of redox mediators can transform the reaction mechanism and suppress the degradation of the electrolyte in Li-O₂ batteries. This study shows how an ionic liquid electrolyte composed of 1-ethyl-3-methylimidazolium bis(trifluoromethylsulfonyl)imide (EMIMTFSI), which is considered unsuitable because of its reactivity towards reduced oxygen species, can be made suitable for Li-O₂ batteries with the use of redox mediators. The mechanistic details of the role of 2,5-di-tert-butyl-1,4-benzoquinone (DBBQ) discharge mediator in suppressing the degradation of the electrolyte is evaluated using *in situ* surface enhanced Raman spectroscopy. Further, a quantitative evaluation of the oxygen consumption and evolution reactions were carried out using operando internal pressure measurements and online electrochemical mass spectrometry. A dual mediator strategy combining DBBQ and iodide redox mediators, helped to achieve a significant improvement in the oxygen evolution efficiency. Our findings advocate the selection of redox mediators based on their efficiency and catalytic selectivity rather than aiming simply for better capacity or rate capability.

MP2

Recent developments in electropolishing of additively manufactured 316L stainless steel

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Abstract

Initially, additive manufacturing (AM) was mainly used for rapid prototyping. Nowadays, all industrial sectors have adopted the technique that has increased in profitability and allows the innovative shaping of almost all alloys. However, a major impediment to the extension of this technology is due to the poor surface finish of the as built parts, characterized by a high roughness (Ra up to 40 μm) and the presence of un-melted particles and weld bead. These defects induce a weathering of the material functional properties such as an increased sensitivity to fatigue and corrosion.

Electropolishing (EP), a well-known electrochemical method for planarization of metals based on their anodic dissolution in an appropriate electrolyte, is then a technology to consider for the finalization of AM parts.

The study relates to the development of a potentiostatic electropolishing process, adapted to the high initial roughness of 316L AM components.

Experiments were first carried out in a three electrodes cell to determine the appropriate operating parameters (potential, duration, temperature...) in several electrolytes (acidic or DES). Special attention was paid to electropolishing mechanisms comprehension by in-situ AFM, and SIE and to the characterization of the post-treated samples (roughness, microstructure and corrosion resistance).

The arrangement (including a reference electrode, and a DC/pulsed potential control) was successfully upscaled in a 3 L reactor, enable handling of bigger samples ($\approx 40 \text{ cm}^2$). The right experimental sequence allowed to reach the target roughness of 1 μm while respecting the geometric integrity of the parts.

MP3

Electrocodeposition of nano-SiC particles under an adapted Pulse-reverse waveform

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Abstract

The use of nano-SiC particles in nickel electroplating has a growing interest in producing composite coatings. However, it is often reported that nano-particles codeposit in a very low fraction which can be detrimental to the final properties. To overcome this limitation, different studies explored the potential of using pulse-reverse (PR) plating for depositing rich nanocomposites. Xiong-Skiba (2005) successfully increased the nanoparticle content ($\approx 23\%w.$) by building a pulsed waveform based on the diameter of the particle i.e. the cathodic and anodic pulses were built so that the plating and stripping thicknesses are equivalent to N and N-1 particles' diameters, respectively. The main objective of this work was to test an adapted version of the method for Ni/SiC systems, electroplating the equivalent diameter of SiC 50nm particles and stripping half. Furthermore, the waveform was also modified by a pulse train, adapted from the work of Aroyo (2002) to avoid passivating the electrodes during long anodic times. To evaluate the effectiveness of this technique, Ni/SiC nanocomposites were also produced under direct current (DC) at current density corresponding to the peak and average values of the pulses. The PR waveform was also applied under high-frequency for comparison. The conventional electroplating under DC at $10A/dm^2$ achieved the highest grain refinement and doubled the SiC content ($\approx 2\%vol.$) compared to $4A/dm^2$. While the samples produced by high-frequency PR attained a similar codeposition ($\approx 2\%vol.$). The adapted long-pulse configuration successfully increased the particle incorporation ($\approx 4\%vol.$). The increase in the particle content contributed to the hardening of the deposits.

MP4

Research Efforts on the Application of Ionic Liquid Technology for Surface Modification of Metals at the University of the Philippines

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Abstract

In the Philippines, there is currently no effort towards exploiting the potentials of ionic liquid technology, especially for surface treatment of metals such as electroplating and electropolishing. A newly instituted laboratory at the Department of Mining, Metallurgical and Materials Engineering Department at the University of the Philippines is the Sustainable Electrochemical Laboratory (SETLab). This laboratory was established to initiate research on creating different surface coatings using ionic liquids. This presentation reports on some research efforts being undertaken by SETLab, including some preliminary results of different on electroplating and electropolishing experiments using choline chloride-based ionic liquids.

Nanoporous carbon: liquid-free synthesis and geometry dependent catalytic performance

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Abstract

Nanostructured carbons with different pore geometries are prepared with a liquid-free nanocasting method. The method uses gases instead of liquid to disperse carbon precursor, leach templates and remove impurities, minimizing synthetic procedures and the use of chemicals. The method is universal and demonstrated by the synthesis of 12 different porous carbons with various template sources. The effects of pore geometries in catalysis can be isolated and investigated. Two of the resulted materials with different pore geometries are studied as supports for Ru clusters in the hydrogenolysis of 5-hydroxymethylfurfural (HMF) and electrochemical hydrogen evolution (HER). The porous carbon supported Ru catalysts outperform commercial ones in both reactions. It was found that Ru on bottle-neck pore carbon shows the highest yield in hydrogenolysis of HMF to 2,5-dimethylfuran (DMF) due to a better confinement effect. A wide temperature operation window from 110 °C to 140°C, with over 75% yield and 98% selectivity of DMF has been achieved, paving the way for practical biofuel synthesis from cellulose derived 5-HMF. Tubular pores enable fast charge transfer in electrochemical HER, requiring only 16 mV overpotential to reach a current density of 10 mA× cm⁻²

MP6

Preparation of electrocatalysts for OER via gas bubble templated deposition and galvanic displacement steps: New evidences on secondary oxide growth

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Abstract

We described the preparation of active anodes for the oxygen evolution reaction (OER) through galvanic exchange reactions between electrodeposited porous PbO_2 and Co^{2+} ions dissolved in acetate media, to form secondary Co_3O_4 layers. Porous PbO_2 was prepared either by oxygen bubble templated electrodeposition [1] or by hydrogen bubble templated electrodeposition of Pb followed by electrochemical oxidation [2]. In the present communication, we report on two new aspects of our studies on related systems.

We have studied the galvanic exchange reaction between PbO_2 and solutions containing both Co^{2+} and Ni^{2+} ions, finding that the former was more reactive and its oxide was preferentially formed from solutions with identical Co^{2+} and Ni^{2+} concentration. In solutions with a 1:5 $\text{Co}^{2+}/\text{Ni}^{2+}$ ratio, a mixed oxide with a 2:1 Co/Ni ratio was deposited, with possible formation of NiCo_2O_4 , known to be active in OER. Co_3O_4 -coated and NiCo_2O_4 -coated layers had comparable activities in the OER.

Other experiments were aimed at collecting new data on the little-known mechanisms of oxide-oxide galvanic exchange reactions. These experiments included: (i) study of the dependence of the secondary oxide thickness on the reaction time, (ii) sequential deposition of secondary and tertiary layers (iii) determination of concentration profiles of the deposits by XPS. These studies showed a minor effect of the thermodynamic driving force on the oxide growth rate and a significant effect of diffusion.

[1] N. Comisso et al. *Electrochimica Acta* 253 (2017) 11–20.

[2] N. Comisso et al. *Electrochimica Acta*, 273 (2018) 454-461.

MP7

Sonoelectrochemical degradation of chlorinated organic pollutants in water: 1 plus 1 equals 3?

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Abstract

Sonoelectrochemistry deals with the combination of electrochemistry with acoustic cavitation induced by an ultrasonic field. The interaction between cavitation and the electrical field generated between electrodes can lead to interesting results for a wide variety of applications. Some of the leading electrochemistry research groups in UK (e.g. Oxford's Compton Group, Bath's Marken Group) have indeed investigated the potential of Sonoelectrochemistry for quite a long time, demonstrating the synergistic effects it may present. Despite this, Sonoelectrochemistry still is a rather unknown to the wider Electrochemistry community, who may be missing out on the opportunities that sonoelectrochemical processes represent in areas such as electrosynthesis, electroanalysis or coatings and materials' science, among others.

This presentation aims at bringing Sonoelectrochemistry closer to the Electrochemistry community by focusing on a series of studies on the sonoelectrochemical degradation of recalcitrant chlorinated compounds in water. We will not only comment on the effect of ultrasonic frequency or power, but also on the effect of concentration, electrode material, and even the presence (or absence) of a background electrolyte. Using the results obtained during our studies, we will also discuss on the synergistic combination of ultrasound and electrochemistry and indeed show that, in Sonoelectrochemistry, 1 plus 1 can equal 3.

MP8

Sonoelectrochemistry: reactor characterization tool and for electrochemical processes accelerator

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Abstract

Surface sonication has proven to offer beneficial effects not only for surface cleaning, but also for functional properties of metallic and organic coatings. Sonoelectrochemistry has attracted interests since the nineteen fifties, and several authors reported the beneficial effect of ultrasound in metal deposition and plating [1]. This is particularly competitive as it may result in a reduction of chemical additives use, or even in their complete suppression. Plating in an ultrasonic field may produce electrodeposits with an increased hardness and brightness, better adhesion to substrate, a finer grain and a reduced porosity and internal stress. This is in direct relationship with the microstructure in terms of grain size and crystalline organization, and the energy loss/beneficial effects ratio is rather positive! However, the process scale-up has failed to match laboratory observations, in particular for the design of industrial systems. To design better set-ups, it is possible to use electrochemistry as a tool to investigate phenomena at the electrode surface and at a given location in the reactor. From a process accelerated by power ultrasound, electrochemistry becomes a useful tool to quantify the ultrasonic energy scattered at the immediate electrode vicinity [2].

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[2] Hihn J.-Y., Doche M.L, Mandroyan A., Hallez L. Pollet B.G., *Handbook on Applications of Ultrasound: Sonochemistry for sustainability* by CRC Press Taylor & Francis 2011

MP9

Copper electroformed getter-coated vacuum chambers for particle accelerators: from concept to development

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Abstract

The aim of this work is to develop a novel procedure to produce small diameter pipes with a functional coating on the inner surface. The application behind this development is, for instance, a thin-film coated chamber for ultra high-vacuum applications.

Non-evaporable getter (TiZrV) thin film coatings provide ultra-high vacuum conditions in particle accelerators. Such films are deposited on the internal walls of the vacuum chambers, transforming them from a gas source into a chemical pump. The trend in electron accelerators design consists in approaching the poles of the steering magnets close to the electron beam. This implies reducing the magnet aperture and using very small diameter vacuum pipes¹. The application of the thin film by conventional physical vapor deposition (PVD) in such small diameter chambers becomes then very difficult.

This contribution describes a method of coating/assembly, using a sacrificial aluminium mandrel as substrate of a functional thin film together with the creation of a surrounding copper pipe by electroforming².

Furthermore, the presence of hydrogen trapped in the electroformed copper was studied for different copper-sulphate baths: an additive-free solution and a solution including a sugar. D-xylose addition allowed the use of higher cathodic pulses before the starting of the copper diffusion limited range. In addition, TDS experiments showed that hydrogen is trapped in two different forms: incorporated in copper vacancies or trapped in the form of bubbles.

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[2] L. Lain Amador et al., J. Vac. Sci. Technol. A, 36 (2018)

MP10

Enhancing magnetoelectric coupling for the efficient magnetic field induced electrochemical reactions

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Abstract

Multiferroic materials have attracted great attention for the electrocatalytic and biomedical applications as they can exhibit coupling interaction between ferroic orders, such as the magnetoelectric effect. The magnetoelectric coupling between ferroelectric-ferromagnetic heteroepitaxial film is mediated by the elastic interaction at the interface; however, the mechanical constraint from the substrate hinders the electro- and magnetostriction of each layer, significantly affecting the coupling interactions. Meanwhile, it has been reported that the degree of the substrate clamping effect can be highly reduced by decreasing the lateral size of the structure. Here, we report on the enhancement of the magnetoelectric coupling in BaTiO₃/CoFe₂O₄//MgO (001) heteroepitaxial thin films by reducing the lateral size of the structures for the efficient control of the magnetic field induced electrochemical reaction. Piezoresponse Force Microscopy (PFM) analysis under the external magnetic field shows that the magnetoelectric coupling effect is enhanced as the degree of the clamping effect (the size of the lateral surface dimension) decreases.

Carbon-based Materials for Anodic H₂O₂ Electrosynthesis

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Abstract

Hydrogen peroxide (H₂O₂) is a powerful oxidizing and bleaching agent that has a variety of applications at an industrial scale including paper pulp whitening, chemical synthesis and water purification¹. The current standard for manufacturing H₂O₂, the anthraquinone process, is energy-intensive and hazardous, signifying the need for a simple and sustainable synthesis route.

Electrochemistry can offer one such means of production, utilizing just water and oxygen as raw materials. Extensive studies have been carried out on the two-electron oxygen reduction reaction (ORR) to produce H₂O₂, yet the oxidation route (H₂O → H₂O₂) has been largely overlooked, due to the reaction's thermodynamic unfavourability and the poor selectivity of available electrode materials².

Recently, it has been shown that metal oxides like bismuth vanadate (BiVO₄) and calcium stannate (CaSnO₃) can oxidise water to H₂O₂, albeit with low yields³. Interestingly, carbonaceous electrocatalysts have not been considered for water oxidation to H₂O₂, despite their high selectivity for the cathodic route. This research addresses this knowledge gap, with preliminary test results demonstrating that carbon-based materials like boron-doped diamond (BDD) are excellent candidates for the two-electron water oxidation reaction, providing an environmentally friendly and stable route to H₂O₂ production.

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2. S. Fukuzumi et al., *Chem.: Eur. J.* **2018**, 24 (20), 5016–5031(<https://doi.org/10.1002/chem.201704512>)
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MPK1 - Schwäbisch Gmünd Prize

Catalytic metal coatings on porous and 3D printed electrodes for electrochemical flow reactors: Prospects and the role of X-ray computed tomography

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Abstract

Electrochemical flow reactors have found important applications in electrosynthesis, energy storage and conversion, destruction of hazardous compounds and water treatment technology. Their performance can be improved by implementing porous electrodes, which provide them with a higher electrode surface area and an enhanced mass transport coefficient, in comparison to classical planar electrodes. This presentation describes the preparation and analysis of platinized titanium electrodes for cerium-based redox flow batteries. The platinum coatings on micromesh and felt substrates were obtained through galvanostatic and potentiostatic electrodeposition in an alkaline bath. Surface morphology and deposit distribution within the porous materials were studied using SEM and X-ray computed tomography, while their surface area was estimated from their charge transfer current ratio versus a planar electrode for a reaction of interest. Recent advances on 3D printed porous electrodes are also discussed, taking as example the combined characterization of surface area and mass transport for two different nickel-coated stainless steel structures. The coatings on these laser-sintered substrates were prepared by electrodeposition and electroless deposition. 3D printing techniques offer the possibility of creating porous electrodes by design, aiming to develop more efficient electrochemical flow reactors. Overall, the present work showcases the opportunities and challenges in the preparation of functional coatings using parallel plane electrochemical flow cells.

Electrochemical synthesis of magnetic mesoporous Ni-Pt alloy thin films for hydrogen evolution reaction

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Abstract

Hydrogen as an environmentally friendly energy vector is constantly gaining importance, and there is a high interest in finding both cost-effective and durable materials as electrocatalysts i.e. to reduce the use of Pt.

Ni-rich Ni-Pt alloy thin films are produced in a simple potentiostatic co-electrodeposition process from an aqueous solution containing Ni(II) and Pt(VI) salts. The process is micelle-assisted; i.e. an amphiphilic block copolymer is added to the bath to create porous films with a very high surface-to-volume ratio to ensure a most effective use of Ni and Pt. A variation of the deposition potential in the range from -0.6 to -1.2 V vs. Ag/AgCl results in different Ni contents ranging from 60 to 99 at%. Pores are homogeneously distributed throughout the film thickness and have a constant diameter around 10 nm.

Thorough characterisation of the thin films reveals their nanocrystallinity and single-phase structure (face-centered cubic solid solution), resulting in a good corrosion resistance and the ability to work as an electrocatalyst towards the hydrogen evolution reaction (HER) in 0.5 M H₂SO₄. Results indicate that optimal behaviour is achieved from a synergistic combination between composition and mesoporosity. In all cases, the performance is stable and reproducible, a degradation of the surface due to the exposure in acidic media is not observed.

Another interesting property of the material is the possibility to tailor its Curie temperature by tuning the Ni/Pt ratio in the films, making the system potentially interesting for magnetic devices (e.g. recording media and MEMS).

MP13

Revealing spatially-dependent electron-transfer kinetics within a phase-separated conductive polymer blend at the nanoscale

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Abstract

Conducting polymers are of great interest in the development of electrodes for innovative organic electrochemical devices, such as solar cells and supercapacitors. Electrochemical techniques are used as a fundamental tool for studying the properties of polymer film electrodes and are often the main way for benchmarking the quality of a material as an electron conductor. However, where such polymers are blended with several other components (*e.g.*, non-conductive polymers), ohmic resistance can make a significant contribution to the bulk electrochemical response, complicating the analysis of conventional cyclic voltammetric methods, particularly at the high scan rates required to probe the kinetics of rapid electron-transfer processes.¹

In this contribution, we demonstrate the use of scanning electrochemical cell microscopy (SECCM),² to map the electrochemical behaviour of a complex, phase-separated conducting/non-conducting polymer blend composed of poly(3-hexylthiophene-2,5-diyl) (P3HT) and polymethyl methacrylate (PMMA). The spatially-dependent electron-transfer kinetics associated with a rapid outer-sphere electron transfer process (ferrocenedimethanol oxidation) has been mapped voltammetrically at the nanoscale, and the resulting data have been correlated with complementary co-located high-resolution surface characterisation techniques (such as conductive AFM), to understand structure-electrochemistry in polymer blends. The nanoscale results further allow predictions of macroscopic behaviour, through finite element method simulations. A comparison of macroscopic voltammetry and simulations highlights the importance of electrode-film resistance, rather than redox reactions, in controlling measured macroscopic electrochemical properties.

Refs.

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Electrochemical Gels for Biological Applications

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Abstract

Electrochemically grown low molecular weight gels (EGLMWG) have great potential to help improve performance for a variety of existing biological technologies such as drug delivery systems, membrane technologies and cell mimicking environments. EGLMWG are grown in the presence of hydroquinone.^{1,2} The electrochemical oxidation of hydroquinone produces a pH gradient which acts as the gelation trigger on the surface of the electrode.^{1,2}

Due to hydroquinone being toxic, EGLMWG are non-cell viable, which limits the use of these materials for biological application. Currently EGLMWG are used only as moulds where the gel and hydroquinone is removed before cells are added.^{3, 4} Here, we show an alternative to hydroquinone that forms EGLMWG that are cell viable for use in biological applications as a synthetic replacement for commonly used biological gels such as Matrigel and collagen.

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MP15

Thermogalvanic cells: Progress in both fundamental understanding and application

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Abstract

Two electrodes at different temperatures separated by a common redox active electrolyte will experience a potential difference; this is known as the temperature dependence of the cell potential. While recognised as a fundamental parameter, it is increasingly being explored as a facile means to electrochemically convert a temperature gradient into electricity, with no moving mechanical parts. Electrochemical cells employing the latter process are frequently referred to as thermogalvanic cells. However, many fundamental components of such systems remain unexplored, unexplained and un-modelled.

This presentation will summarise recent investigations into the fundamentals of thermogalvanic cells, as well as their potential applications.

Our recent fundamental investigations have involved exploring the effect of ionic strength and concentration upon the temperature dependence of the cell potential and the thermogalvanic current generated. It has also investigated the previously unexplored effect of the ratio of the reduced:oxidised species in thermogalvanic cells. Notably, the potential from stoichiometric cells follow a Debye-Huckel-type relationship, whereas non-stoichiometric ratios are dominated by Nernstian-type trends. In stoichiometric cells, current was directly proportional to concentration, whereas in non-stoichiometric cells, the current was best modelled by Butler-Volmer-type kinetics.

For applications, recent attention has focussed upon the potential application of thermogalvanic cells in the production of energy-harvesting construction materials. As such, the recently patented development of 'thermogalvanic bricks' will be introduced.

MP16

Electrodeposition of Ni-Sn alloys and Ni-Sn / rGO composite coatings from deep eutectic solvents and their physicochemical characterization

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Abstract

Electrodeposited Ni-Sn alloys show enhanced characteristics compared to those of the individual elements. They represent an alternative to chromium decoration and they find applications in corrosion protection due to their good anticorrosive properties. In addition, Ni-Sn alloys are used in electronics as etch resist in printed board circuits as well as in automotive industry. Furthermore, graphene related materials are attracting the current interest owing to their unique electrical, thermal and mechanical properties.

The electrodeposition of Sn based containing alloys from aqueous electrolytes is limited by the oxidation of Sn²⁺ to Sn⁴⁺ and sludge formation which leads to the solution destabilization. Deep eutectic solvents, defined as eutectic mixtures of cholinium salts with hydrogen bond donor compounds, represent a promising green alternative to overcome this problem and due to their high viscosity, they may act as dispersants of carbon-based materials.

Here we report the synthesis and characterization of both Ni-Sn alloys and of Ni-Sn/reduced graphene oxide composite coatings from choline chloride based ionic liquids on Cu substrates. Influence of different electrodeposition parameters has been investigated. The obtained deposits are adherent and uniform onto copper substrates. SEM, Raman and XRD analysis evidenced the successful insertion of reduced graphene oxide into the metallic matrix.

Finally, the mechanical properties and conductivity have also been investigated.

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Physical and Fundamental

Oral Abstracts

The potential profile across the electrode-electrolyte interface: cation size effects and implications for CO₂ electrocatalysis

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Abstract

The effect of the nature of the electrolyte cation on the activity and product selectivity of the electrocatalytic reduction of CO₂ is well known. We present a study of the effect of the electrolyte cation on the Stark tuning rate of the C-O stretching frequency and the capacitance of a CO-covered Pt electrode. The results show a rather good correlation between the two magnitudes and offer clear evidence that the electric field felt by any molecule within the electrical double layer is different for different cations, which can lead to differences in the stability of adsorbed reaction intermediates or affect the reactivity of solvent molecules by resulting in different degrees of polarization of solvent molecules in the electrical double layer. A parallel study using Au electrodes in acetonitrile in the potential region where CO₂ reduction occurs leads to similar results. Reduction of acetonitrile yields adsorbed cyanide on the electrode surface, and we find that the rate of change of the C-N stretching frequency with potential, as well as the interfacial capacitance, are strongly dependent on the size of the electrolyte cation. In this case, the correlation between these two magnitudes is excellent, again suggesting that both magnitudes are determined by the separation between the electrode surface and the OHP.

Our results also demonstrate that the thickness of the electrical double layer is determined by the hydrodynamic radius of the cation, whereby, for example, Li⁺ is a relatively large cation in water, but a very small cation in acetonitrile.

The effect of hydrogen bonding capacity on lipid bilayer structure and function

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Abstract

Phospholipid bilayers are a popular model for a biological cell membrane because they form a natural environment for the immobilisation of membrane-associated proteins, the study of which finds application in study of protein function and in production of sensing devices. Most studies use a model lipid or lipid mixture to form the lipid matrix, although some fundamental studies of the electrochemical properties of different lipids have been made. Electrochemical measurements provide a means to determine electrical barrier properties, crucial to the function of a cell membrane, and, when combined with structural studies, can enable the effect of an electric field on lipid layer structure to be determined. To this end, more recent studies of various lipids and their mixtures have focused on ensemble structure. Some common themes have emerged but also some marked differences in behaviour. Sphingomyelin is a lipid type commonly found in lipid rafts in the mammalian plasma cell membrane. Lipid rafts host *trans*-membrane proteins and some lipids involved in cell signalling processes. Sphingomyelin has the same polar headgroup as phosphatidylcholine, one of the most common lipids in cell membranes. The key difference in structure is the backbone of the molecule, where the acyl chains are joined to the headgroup: phosphatidylcholines contains only hydrogen bond acceptors but sphingolipids contain both hydrogen bond donors and hydrogen bond acceptors. In this study, we explore how these different capacities for hydrogen bonding affect bilayer structure and function using a combination of electrochemical techniques and *in situ* infrared spectroscopy.

Probing the charge distribution at the electrochemical interface

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Abstract

In-situ surface x-ray diffraction has enabled an atomic/molecular-level understanding of the interface under reactive conditions, including its potential and time dependence, to be developed. While information about the atomic structure of the electrode surface in electrochemical in-situ cells has been widely investigated, insight into the charge distribution and the structure of the electrolyte at the interface is still lacking.

A fundamental understanding of the nature of the charge transfer, especially the influence of the applied potential and the screening by the electrolyte, is a major goal in electrochemistry to better understand electrochemical processes and charge transfer during adsorption and deposition.[1]

Thus combining x-ray spectroscopy and x-ray diffraction to gain site specific information about the charge distribution at buried interfaces is a promising tool. [2,3]

Examples of how the use of surface x-ray scattering techniques can help to characterise electrochemical interfaces in-situ in order to link, structure, reactivity and stability will be presented. [3,4] Advances in these directions offer possibilities in elucidating atomic scale models of the electrochemical interface and thus will help to establish structure-stability-reactivity relationships.

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Following the formation of Electrochemically Assisted Self Assembled silica films in real time by in situ Grazing Incidence Small Angle X-ray Scattering

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Abstract

Under the Advanced Devices by Electroplating EPSRC programme grant (EP/N035437/1) we are working on the integration of high quality chalcogenide semiconductors with microfabricated structures to be used in devices as thermoelectrics, phase change memory (PCM) and infra-red detectors. A key element of this work is the integration of nanowire semiconductor structures into electronics, for which we are looking at methods to fill larger microfabricated features (μm length scales) with aligned mesoporous templates. Mesoporous silica powders are already available in various sizes and structures for the application in catalysis, sensors, etc. Films of the same material are typically produced by evaporation-induced self assembly (EISA), but vertical alignment of the pores to the substrate is very difficult to achieve. Hexagonal arrays of vertically aligned mesopores can be achieved by electrochemically assisted surfactant assembly (EASA). The application of a negative potential to an electronically conductive substrate results in the self-assembly of a cationic surfactant (typically cetyltrimethylammonium bromide) close to the substrate surface. The resulting pore spacing and diameter of only a few nanometres make the characterisation of such structures very challenging and time consuming. Grazing incidence small angle X-ray scattering (GISAXS) allows for the non-destructive structural analysis of thin films with high statistical relevance in lateral and vertical direction. In this work, we show the results obtained from in situ GISAXS experiments done during the EASA of silica, following the evolution of the structure formation in real time with sub-second time resolution. This provides previously non-existent insight regarding the mechanism behind EASA deposition.

Modified Multiwall Carbon Nanotubes (MWCNT) based Electrochemical sensor for Thallium in water

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Abstract

The extensive use of toxic metals like Thallium (Tl), Hg, Cd and Pb in many industrial and agricultural activities poses severe global health threats. Thallium and its compounds like other toxic metals, is potentially carcinogenic and is poisonous to living organisms. The sensitive analytical techniques for rapid monitoring and quantification of Tl include AAS, ICP-MS, XRF spectrometry and GFAAS. Although these analytical techniques have high accuracy and the lowest detection limits but they are cumbersome, expensive and lack simultaneous detection with other pollutants.

The aim of this work is to fabricate a simple, rapid, low cost and sensitive electrochemical sensor as an alternative of spectrometric methods for Tl(I). The sensor is a working electrode used in differential pulse anodic stripping voltammetry (DPASV) technique which is based on Indium tin oxide (ITO) modified with functionalized multiwall carbon nanotubes (MWCNT). In this study, MWCNT was selected due to its properties such as high surface area and electrical conductivity, chemical stability and sensitivity. An ITO electrode has a promising platform due to their good electrical conductivity, transparency to visible wavelengths and high surface-to-volume ratio. The modified electrode possesses good sensitivity toward Tl(I) as compared to bare electrode using KCl as supporting electrolyte. Under the optimized experimental conditions of electrolyte, deposition time, accumulation and reduction potential the proposed sensor exhibited a linear calibration curve with a detection limit in ppb. The sensor displays good repeatability and is applied to the determination of Tl(I) in real water samples and certified reference material.

Optimisation of boron-doped diamond electrodes for the quantitative detection of free chlorine species at high and low concentrations

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Abstract

Free chlorine detection and quantification is essential to several industrial processes ranging from commercial bleaching to wastewater treatment. The two main forms of free chlorine are hypochlorous acid (HOCl) and hypochlorite (OCl⁻), which are pH dependent species. Accurate and fast detection of these analytes is vital to ensure the proper dosage is applied for a particular application. Current detection methods have known shortcomings for fast, continuous, and on-line sensing. As such, our work will evaluate the viability of using non-diamond carbon functionalised boron-doped diamond (BDD) electrochemical sensors for both high and low concentrations of free chlorine. The levels are chosen to be relevant for industrial practices that utilise chlorine as a disinfectant.

Through use of a laser micro-machining process it is possible to mechanically write robust regions of non-diamond carbon i.e. sp² carbon into an sp³ BDD electrode. These laser-machined regions activate the BDD surface towards OCl⁻ reduction and we show how the spatial pattern of sp² carbon integration affects the response of the sensor for OCl⁻ detection at high concentrations (i.e. 0.02 M to 1.50 M OCl⁻) in alkaline (i.e. pH 13), chemically oxidising solutions. Experimental electrochemical data are complimented with finite element modelling data to better understand the electrochemical system. On-going work is also examining how these hybrid sp²-sp³ BDD sensors perform for providing information on both the pH of the measurement system and the concentration of free chlorine, under pH conditions where both the HOCl and OCl⁻ forms of free chlorine exist.

Greening analytical voltammetry through electrochemical cell miniaturization

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Abstract

Analytical voltammetry in academic and industrial research or for clinical or environmental analysis is normally done in milliliter-sized beaker-type three-electrode electrochemical cells. Accordingly, the significant generation of waste from test solutions with or without toxic ingredients and the excessive reagent consumption are the objectionable side effects of an extensive application of this popular stream of electroanalysis. Ecologically aware execution of individual measurements in cell arrangements of reduced volumes is virtually the only chance to considerably decrease electrolyte waste and chemical use per analysis and let voltammetry get in line with the commonly accepted green-chemistry principles. A 30 μL three-electrode mini cell with platinum counter and miniaturized Ag|AgCl (KCl) reference electrode was shown to be an effective tool for greening voltammetry. The cell design combines matchless cheapness with unlimited reusability as well as fabrication and handling simplicity. Besides, its compatibility with all common working electrodes and its suitability for a wide range of analytes, evidenced through presentation of biomolecule and inorganic redox species voltammetry and sugar biosensing, will be discussed. Overall, the new electrochemical gadget offers a unique chance for users of analytical voltammetry to adopt an obviously desirable low-cost and eco-friendly practice on a daily basis with ease.

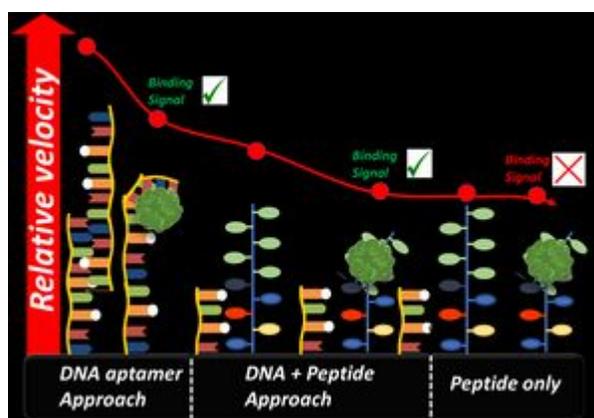
Aptamers and Peptides – Opening new Libraries

Rhushabh Maugj, Mark Platt, Bernadette Gamble
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Abstract

Resistive pulse sensing (RPS) is an electrochemical nanopore sensing platform that characterises nanoparticles that range from sub 100nm to micrometer sized particles. RPS nanopore sensors can be used as a multi-use platform to analyse ions, particles and biological targets in solution. Measuring the interaction between aptamers and small molecules, proteins, cells, virus etc. is well suited for RPS platforms. The aptamer adopts a tertiary structure in the presence of the target and results in a change in charge density around the particle which is measured as a change in translocation speed on the RPS platform.

The use of DNA modified particles has been a foundation to explore the use of aptamers in many applications, and here we present the detection of antibiotics and chemotherapeutic as the targets for the aptamers. Using indirect and direct binding techniques of aptamers to the nanoparticles surface allows for flexibility when optimising and designing an assay to detect small molecule targets. Increasing the target library allows for more targets to be detected using RPS, and a method to include peptide aptamers along with DNA aptamers can be achieved using nanoparticle surface chemistry. The ability to truncate an aptamer sequence adds to the dexterity of the assay design and helps to reduce to costs. The applications of aptamers on the particles surface has uses in disease detection, military, water sampling and therapeutics.



Tracking Ultrashort Carbon Nanotube Wireless Electrodes Within NG108

Cells

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Abstract

This work aims to combine wireless electrochemical techniques and cell culture by utilising ultrashort carbon nanotube porins (CNTPs). These CNTPs are capable of self-insertion within cellular membranes where they can function as a wireless electrodes; and through the application of a voltage, directly affect cellular functions.

CNTPs were first coated in 1,2-dioleoyl-sn-glycero-3-phosphocholine (DOPC) to facilitate their integration within the membranes of murine neuronal cells (NG108-15). 6-Amino fluorescein was covalently tagged to the CNTPs in order to track their integration and retention within the cells while FTIR was used to confirm the modification of the CNTPs. Live cell fluorescence microscopy enabled the real time tracking of the CNTPs and viability measurements.

Here we show the successful integration of CNTPs within cells and their effect on the differentiation state of NG108-15 cells. The successful incorporation of CNTPs within the cellular membrane allows the application of wireless electrochemistry and the transformation of the CNTPs into transmembrane bipolar electrodes.

Chemically Encoded Self-Organized Quantum Chain Supracrystals with Exceptional Charge and Ion Transport Properties

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Abstract

Capacity and safety are the main challenges for next-generation lithium-ion batteries. Improving the electrode design is considered the solution to increase the capability and stability in reversible lithium storage. Although several nanostructured-materials have shown promise as emerging candidates, their size-mediated lithium intercalation and diffusivity can be significantly enhanced if interparticle-coupling can be controlled while nanograin features are retained. Here, we report a nanostructure design concept consisting of weaving quantum dots (QDs) into a chained geometry to coalesce and preserve the quantum-confined boundaries for lithium transport. We exemplify this quantum chain (QC) via lead sulphide QDs, which exhibit excellent capacity and cycle performance (i.e., 685.8 mAh g⁻¹ at 400 cycles). Through systematic charge-transfer and microscopy investigations, we found that the improved capacity and stability are owing to the high boundary densities and QC-networks embodied in the diminishing lattice-stress during fast Li-diffusivity. Our findings will open new avenues in nanostructured-design for future lithium storage applications.

Detection of Non-Steroidal Anti-Inflammatory Drugs Utilising Cerium Dioxide Nanoparticles Deposited on Modified Screen Printed Electrodes

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Abstract

The detection of low levels of drugs including Non-Steroidal Anti-Inflammatory Drugs (NSAIDs) in natural waters and in wastewaters is becoming increasingly important. In this work cerium dioxide (ceria) nanoparticles were synthesised to produce mono-dispersed nanoparticles (typically 10-20 nm). These were then incorporated into the working electrodes of commercial Spin Coated Carbon Electrodes (SPCE) and used to determine paracetamol, aspirin and diclofenac (NSAIDs), as well as caffeine.

Square Wave Voltammetry (SWV) conditions were optimised in order to assist detection. Contrary to a recent report in which CeO₂ enhanced the current response obtained on a CeO₂-SPCE for the detection of paracetamol, codeine and caffeine, it was found that the currents from an SWV analysis were slightly diminished. However the use of a post-production heat treatment regime overcame this problem. Improved sensitivity was also obtained through the use of a baseline correction routine. This was especially significant at high potentials and was particularly important for the detection of caffeine. An SWV study carried out on diclofenac over a concentration range from 0.4 µM to 26 µM indicated that the response was linear with a limit of detection of 0.4 µM and a sensitivity of 0.058 µA/µM. The electrochemical response of the diclofenac was interpreted in terms of a two step EC mechanism; an initial one electron oxidation reaction and one proton transfer to form a nitrogen radical species (2), followed by a ring closure reaction, resulting in the formation of heterocyclic aldehyde and carboxylic acid species, identified from a liquid chromatograph-mass spectrometric analysis.

Studies of the influence of molecular oxygen on the formation and reduction of oxides on Pt electrodes

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Abstract

The effect of oxides on the oxygen reduction reaction (ORR) has been widely studied and a partially oxidised surface is known to be less active towards the ORR than a fully reduced one. In contrast, the effect of molecular oxygen on oxide formation and reduction has not received much attention, probably because the ORR and oxide formation/reduction occur at similar potentials. Some have studied this by forming oxides in presence of O₂ and reducing them in its absence but this requires precise degassing before the reduction takes place. Using a different approach based on the amperometric response of microdisc electrodes, our group previously reported the presence of oxygenated species that are pre-adsorbed on metallic electrodes after exposure to oxygen (1, 2) and recent results in neutral media (3) suggest that these species are related to oxide formation influenced by the presence of dissolved oxygen and by local pH changes.

In this presentation we will describe how we investigated the impact of molecular oxygen on the metal surface using a combination of transient and steady state amperometric techniques at microelectrodes and with the scanning electrochemical microscope. We will focus on the role of pH and particularly neutral conditions which offer a unique opportunity to reveal subtle effects arising from exposure of the metal surface to dissolved oxygen.

References:

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PF12

Monitoring products of electrochemical reactions in real time

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Abstract

The classical way to characterise the selectivity of electrochemical reactions involves potentiostatic or galvanostatic electrolysis and intermittent product determination, for example with chromatography methods, nuclear magnetic resonance etc. The advantage of this approach is that quantitation is possible in terms of (partial) reaction rates, yields, selectivities or faradaic efficiencies. However, the offered temporal resolution is in the order of several minutes, so transient, dynamic processes that occur within seconds cannot be captured. Differential electrochemical mass spectrometry was previously developed to address this point and was applied successfully to a wide range of reactions that form highly volatile or gaseous products.

A newly developed, internationally unique methodology will be presented that allows for the detection of electrochemical reaction products in real time, independent of their vapor pressure and the presence of salts. Some examples of applying the electrochemical real-time mass spectrometry (EC-RTMS) in multi-product reactions such as the CO₂ reduction, the oxidation of alcohols or conversions of furanic compounds will be shown.

Acknowledgments

This work was funded by the German Federal Ministry for Education and Research (BMBF) under the project grants 033RC004C (eEthylen) and 03SFK2Z0 (Power-to-X), and by the Deutsche Forschungsgemeinschaft (DFG, German Research Foundation) under Germany's Excellence Strategy - Exzellenzcluster 2186 "The Fuel Science Center".

The role of SnO₂ in the bifunctional mechanism of CO oxidation at Pt-SnO₂ electrocatalyst

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Abstract

CO oxidation is at the core of fuel cell anodic catalysis, because CO poisons Pt surface and deactivates other anodic reactions, as an impurity from reformat gas and a main intermediate from the incomplete oxidation of fuel molecules. Among CO-tolerant catalysts, Pt-Sn alloys stand out, due to a combination of the bifunctional (supply of active O species), ligand (electronic modification of the Pt sites), and ensemble (modification of adsorption sites) effects. Although the origins of the last two effects are limited to alloyed Sn for Pt-Sn catalysts, the bifunctional effect may also be attributed to unalloyed Sn such that the effects from SnO₂ may be as important as that from alloyed Sn.

To clarify the effect from SnO₂, a Pt-SnO₂ nanoparticle catalyst supported on carbon (Pt-SnO₂/C) is synthesized with careful consideration to avoid Pt-Sn bonding formation. The structure of Pt-SnO₂/C and the absence of alloyed Sn are interrogated by XRD and detailed XAS analysis. CO oxidation is strongly promoted on Pt-SnO₂/C with an onset potential of $\sim 0.3 V_{RHE}$, reproducible on mixtures of Pt/C and SnO₂ nanoparticles, and SnO₂-modified Pt electrodes. The CO oxidation on Pt-SnO₂ occurs at low overpotential with fast kinetics, such that the diffusion of dissolved CO becomes the rate-determining-step beyond $\sim 0.45 V_{RHE}$. The bifunctional mechanism of Pt-SnO₂ is proposed to originate from a co-catalysis of SnO₂, on the surface of which Sn-OH is consumed by reacting Pt-CO_{ads} at the interface and regenerated by dissociating water (Figure 1).

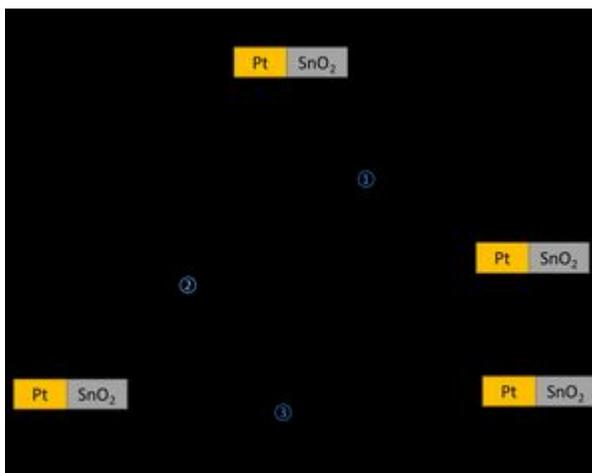


Figure 1 Catalysis cycle of CO electro-oxidation at Pt-SnO₂.

Fundamental Electrochemistry of P-Block Halometallates in Non-Aqueous

Solvents

Simon Reeves, Philip Bartlett, Wenjian Zhang, Gillian Reid
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Abstract

Electrodeposition from weakly coordinating solvents (such as dichloromethane) provides a bottom-up approach to filling high aspect ratio nanostructured templates which conventional fabrication techniques (such as vapour deposition) can struggle with. Tetrabutylammonium (TBA) halometallate salts of p-block metals and metalloids can be readily dissolved in dichloromethane. Electrodeposition of thin films on the macro- scale from this system at room temperature has been demonstrated for a variety of functional electronic materials, including InSb.¹

Electrodeposition into micro-/nanostructured templates requires different conditions than thin film electrodeposition due in part to the different electrochemical behaviours at different length scales. Therefore it is critical to understand the fundamental processes of electrodeposition of the halometallate salts in order to tailor the conditions for reproducible electrodeposition.

This presentation will cover the characterisation of TBA[InCl₄] and TBA[SbCl₄] in dichloromethane using a variety of techniques: macroelectrodes, microelectrodes, rotating disc electrodes and electrochemical quartz crystal microbalance, to further understand the electrochemical properties of these two precursors.

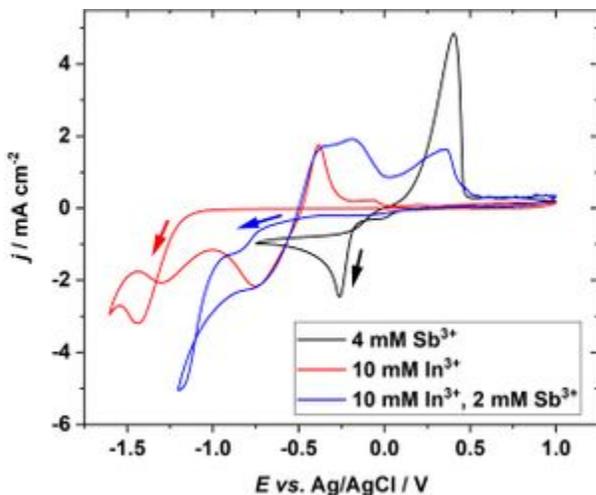


Figure 1: Cyclic voltammograms comparing TBA[SbCl₄] only, TBA[InCl₄] only and mixed TBA[SbCl₄] and TBA[InCl₄] electrolytes in dichloromethane with 0.1 M TBA[Cl]. Platinum macroelectrode $d = 0.5$ mm, $\nu = 50$ mV s⁻¹, $T = 298$ K.

This work has been supported by the EPSRC through the Programme Grant, Advanced Devices by Electroplating (ADEPT – EP/N0354371/1).

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Non-invasive Non-Enzymatic Electrochemical Glucose Sensors based on Metal Oxide Nanostructures

Aruna Ivaturi

Smart Materials Research and Device Technology (SMaRDT) Group, Department of Pure and Applied Chemistry, University of Strathclyde, Glasgow, United Kingdom

Abstract

According to World Health Organisation, 347 million people (~5% world's population) suffer from diabetes. Furthermore, there are 183 million people worldwide that are currently undiagnosed. Predicted to become the seventh leading cause of death, diabetes is dangerous because of its complications: cardiovascular diseases, blindness, risk of amputation, kidney failure etc. Glucose concentration in blood is the key parameter for diabetic patients: monitoring and hence maintaining it at an appropriate level allows these complications to be postponed. Most of the commonly used glucose sensors are based on invasive techniques requiring frequent finger pricking. Current invasive methods are thus too onerous and many patients discontinue to monitor their blood glucose leading to other complications. Also, most of the commonly used diabetes sensors are based on enzymes (e.g. glucose oxidase) and hence need special conditions for storage and don't have long shelf life. To address these issues, we have developed non-enzymatic, non-invasive electrochemical glucose sensors based on metal oxide nanostructures, which will be discussed in more detail in this talk.

Label-free Electrochemical Immunosensor for the Detection of IgG in Calf

Serum

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¹Tyndall National Institute, Cork, Ireland. ²Teagasc, Cork, Ireland

Abstract

Electrochemical transducers have been a favourable option for point-of-care immunosensors to improve diagnostic efficiency. Such devices are simple to use, have fast analysis times and can be fabricated at low cost. Electrochemical devices can be designed to allow for simultaneous measurements and be compatible with integrated circuits and microfluidics.

Currently, Ireland is the 5th largest beef exporter worldwide and the largest in Europe. Therefore, monitoring of a herd's health is important to maintain economic stability for this large industry. This monitoring can start at birth to ensure a calf will thrive, securing the herd's future. Calves are born without antibodies (IgG) and thus are vulnerable to diseases. Until they can actively produce their own, they rely on maternal antibodies transferred to them through colostrum. Low levels of IgG can put a young calf at risk of infection, often resulting in pre-mature death. Pen-side recording of IgG levels after birth would enable veterinarians to provide specific treatment strategies on-farm, thereby significantly improving survival rate of calves.

In this study, electrochemical impedance spectroscopy was employed to monitor the change of charge-transfer resistance associated with material binding at electrode surfaces. Gold microband electrodes were modified using electropolymers to immobilise anti-bovine IgG biomolecules onto the surface. Impedimetric measurements monitored the corresponding binding of bovine IgG to the immunosensor surface. The successful passive transfer of antibodies to calves was identified by comparing the IgG levels in the serum of a new born calf before and after colostrum feeding.

Corrosion Science

Oral Abstracts

C1

Thin electrolyte thickness measurements for atmospheric corrosion modelling

Koushik Bangalore Gangadharacharya, Nils Van den Steen, Daan De Wilde, Darja Pecko, Raf Claessens, Herman Terryn
Research Group Electrochemical and Surface Engineering, Department of Materials and Chemistry, Vrije Universiteit Brussel, Brussels, Belgium

Abstract

The evolution of geometric thickness of a thin electrolyte layer (TEL) on the metal surface, due to condensation/evaporation of moisture in the air, is one of the prime factors influencing the corrosion rate. The dynamic variation of thickness is mainly caused by varying surface temperature, environmental relative humidity and temperature. Additionally, the presence of hygroscopic salts also has a major impact on the condensation/evaporation dynamics. In order to quantify their influence, experiments are conducted under strict environmental control to directly measure the thickness evolution of uniform TEL under condensation and evaporation regimes. The experimental results are used to validate the Dynamic Electrolyte Film Model (DEFM), which forms the primary part of the atmospheric corrosion model developed in our group [1]. DEFM numerically simulates the thickness evolution of a uniform TEL due to condensation/evaporation of moisture, both with and without the presence of hygroscopic salts. A continuous measurement of thickness variation as function of surface temperature is attempted using interferometry. Both condensation and evaporation of TEL is promoted by varying the surface temperature below and above the dew point for a given environmental relative humidity and temperature.

Reference

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The corrosion behaviour of magnetocaloric alloys under magnetic field conditions

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Abstract

The corrosion mechanism of the magnetocaloric refrigerants $\text{La}(\text{Fe},\text{Mn},\text{Si})_{13}\text{Hx}$ in the active magnetic regenerator system has been examined in detail. Factors representative of the real complex operating conditions within a refrigeration cycle, including magnetic field direction, magnetic state of the material and magneto-volume change under an alternating changing field, were considered. The corrosion behaviour can be explained by consideration of both the Lorentz and the field gradient force acting at and near to the magnetocaloric electrode surface. Whilst the Lorentz force accelerates the mass transport of reactive solution species and removal of corrosion products, the field gradient force attracts corrosion products and shows an inhibiting effect. No dramatic effects were observed under alternating field conditions suggesting that localised cracking during volume expansion does not contribute significantly to the corrosion over the short time period spanning the study. The current work suggests that for this material the magnetic field effects are largely due to magneto-transport and are most apparent when the material is in its paramagnets with high corrosion rate (~ 20 mmpy): the worst conditions are not *a priori* obvious and so conditions for accelerated tests and for applications should be carefully chosen.

Study on ion penetration within polymeric binder by ion selective electrode

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Abstract

Water, oxygen and ion reaching the interface of organic coating and metallic substrate by penetrating polymeric binder can lead to corrosion initiation, propagation and shorten lifetime of organic coating. Chloride ion is a typical aggressive ion that encourages corrosion process. Hence, understanding of chloride ion penetration is important and essential. Ion selective electrode (ISE) is expected to use for monitoring the ions in coating and indicating the penetration process of the ions, especially for particles with small transport pathway that is difficult to be observed by electron microscopy. ISE is introduced in this project in order to monitoring chloride ions within epoxy binder and understanding the ion migration process. Besides, EIS is also applied to study the epoxy coating and is contributed to prove ion penetration through the bulk of binder. Coating surfaces are observed by SEM.

C4

Calibration of a Pitting Corrosion Simulation Model using Pit Population

Data obtained from White Light Interferometry

Chinmay Pardeshi^{1,2}, Bart Van den Bossche¹, Herman Terryn²

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Abstract

The annual cost of corrosion worldwide is about US\$ 2.5 trillion (i.e. 3 to 4% of GDP of industrialised countries). Out of all types of corrosion, pitting corrosion is most dangerous form because it is very difficult to detect. Once these pits are evolved in to sufficiently large stable pits, they act as a potential source for crack initiation. This is very dangerous for the safety of the structures. It is one of the pressing concerns of our metal based civilisation. Within the framework of mCBEEs Marie Curie ITN, the purpose of this study is to create a simulation tool to predict pitting corrosion behaviour of passivating materials (e.g. AA2024) using numerical modelling.

The objective is to develop and validate a mathematically sound deterministic pitting corrosion model. The challenge here is how to account for and predict various stages of these pits over time. What makes it peculiar is the fact that many factors are involved in this process. The goal is to have conservation equation for pit populations as well as kinetic equations for the accumulation rate of meta-stable, stable pits, and for the re-passivation of large pits. As precipitates induced local potential differences at the surface heavily influences pitting corrosion, the aim is to include those local electrochemical effects in the numerical modelling.

The presentation will cover a brief overview of the idea and methodology of using white light interferometry to model pitting corrosion. Furthermore, the challenges involved in the process will be discussed.

Chaotic Copper Corrosion: The Influence of Dissolved Gas on the Anodic Passivation of Copper in Model Seawater

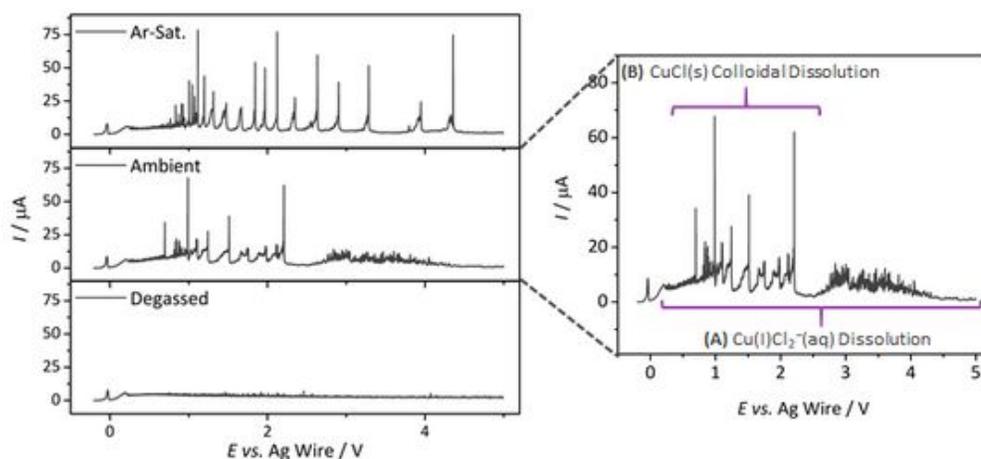
Amelia Langley, Frank Marken
University of Bath, Bath, United Kingdom

Abstract

Chaotic potentiostatic phenomena have been observed for copper during anodic polarisation when immersed in model seawater, linked to the formation of a passivating CuCl(s) film.^{1,2} Such phenomena have, until recently, been attributed only to the dissolution of CuCl₂⁻. The proposed mechanism consists now of two concomitant pathways:³

(A) the dissolution of Cu(I)Cl₂⁻ and,

(B) the formation of CuCl at the Cu | NaCl(aq) interface followed by colloidal dissolution.



Pathway B is observed as characteristic noise in voltammetry data, which changes significantly upon altering the environment. Here, the effects of dissolved gas are explored where, more specifically, the effects of industrially relevant O₂, CO₂ and H₂ are studied. Inert gas (He and Ar) and degassing (removal of gas) effects are also explored. The colloidal mechanism, (B) is demonstrated to be dependent on both the presence and type of gas, subsequently affecting anodic passivation to a lesser or greater extent. A mechanism is proposed, linked to the role of gases as surfactants.

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C6

Introducing new electrolyte for copper plating with aluminium

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¹Institute of Technology Carlow, Carlow, Ireland. ²University of Leicester, Leicester, United Kingdom. ³University of Strathclyde, Glasgow, United Kingdom

Abstract

Failure of copper-cored magnet wires used in the power industry occur due to the break-down of the insulating polymer layers causing localised copper oxidation and rapid performance deterioration of the components. One potential solution is to apply an aluminium coating which will be selectively oxidised at and near the Cu-Al interface and will provide long-term protection of the core. Aluminium plating on copper substrates is very complex, due primarily to the sensitivity of the process to water-absorption and the resultant necessity for using non-aqueous electrolytes in controlled environments. Newly discovered electrolytes based on Aluminium Chloride (AlCl₃) have been shown to facilitate Al-plating without the necessity of a glow box or a protective gaseous atmosphere, even under atmospheric conditions. Water absorption by the Aluminium Chloride solution can be restricted by applying a protective layer of liquid hydrocarbons. This insulating layer also positively impacts the plating time, which is greatly reduced by a factor of 10 or more. The deposited 5 µm+ coating is evenly distributed adherent deposition of metallic aluminium on the copper substrate. Deposition time for the aluminium layer was closely monitored using quantifiable variance (drop-off) of the plating current. Concerns exist in terms of Al-Al layer adhesion, raising concerns regarding the suitability of this process aluminium-on-aluminium plating. However, further testing of this novel technology is on-going. Deposited layers were inspected by optical topography microscope and Scanning Electron Microscopy (SEM). It was found that scaling-up of the plating process (increasing thickness) did not present any significant difficulties.

Evaluation of the role of acetic acid in CO₂ Top of line Corrosion using real-time corrosion measurements

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Abstract

Based on a review of both literature and field data, it is apparent that the role of acetic acid (HAc) in oilfield brines is extremely controversial. Although it is commonly believed that the presence of this organic compound enhances both the general and the localised corrosion rate of carbon steel, HAc has recently been reported to also act as a weak inhibitor in specific N₂-saturated environments. Such behaviour has been attributed to the fact that in these specific conditions, the corrosion current is under charge-transfer control. These observations prompted a study into whether such behaviour is apparent in a CO₂ Top of Line Corrosion (TLC) scenario. Therefore, within this study, the role of HAc on the kinetics and mechanism of carbon steel dissolution was investigated under four carefully selected TLC conditions. For each system, the corrosion response was characterised through the implementation of a miniature three-electrode setup developed for extracting real-time electrochemical measurement. Surface analysis techniques were also performed to complement the electrochemical results. Collective consideration of the corrosion response and condensate chemistry indicate that similar effects were observed compared to those reported recently in N₂-saturated systems i.e. the introduction of HAc can result in either an accentuation or diminution in corrosion rate depending upon the operating conditions. In fact, a decrease in corrosion rate was reported despite a reduction in condensate pH in specific environments.

Influence of Ca²⁺ ions on corrosion product formation kinetics and characteristics in CO₂ environments

Amir Shamsa

University of Leeds, Leeds, United Kingdom

Abstract

The role of Ca²⁺ ions on the corrosion behaviour of carbon steel in CO₂-corrosion environments was evaluated at 80°C and 150°C. In the presence of Ca²⁺ ions there was a more significant effect on the general corrosion rate at the lower temperature of 80°C where the protection afforded by the corrosion product layer was reduced. The extent of localised/pitting corrosion increased through the addition of Ca²⁺ ions indicating that Ca²⁺ plays a role in promoting localised corrosion. XRD based calculations, along with focused ion beam EDX-line scans confirmed that the mixed Ca/Fe carbonates precipitated with concentration gradients across their thicknesses.

Electrochemical Corrosion Monitoring in the Nuclear Industry

Clive Harrison, Graham Quirk, Katy Greer, Chris Greenlees
Wood, Warrington, United Kingdom

Abstract

Electrochemical corrosion monitoring can play a valuable role in understanding the nature and causes of corrosion in the nuclear power industry. However, it is not as commonly employed within the nuclear power industry, especially within the UK, as in other industries such as oil and gas, and the corrosion is usually discovered during inspections during station outages or when a component fails during operation. Nevertheless, there are numerous examples of where electrochemical corrosion monitoring has been used to facilitate the investigation of complex corrosion issues, which can be addressed subsequently by the modification of the plant environment or operational decisions once the surveillance has linked the corrosion activity to specific operational decisions and durations in the power generation cycle. Electrochemical corrosion monitoring has proven particularly useful for localised corrosion activity such as pit propagation or stress corrosion cracking (SCC, where crack propagation can be fast and brief).

This presentation will demonstrate the value of electrochemical corrosion monitoring by reference to a number of plant corrosion monitoring installations carried out by the authors over the past 30 years. These installations will include details of the corrosion data sought, the electrochemical techniques used with their benefits and disadvantages, the difficulties and issues involved in the installations and how the electrochemical data was used to mitigate the corrosion damage occurring.

C10

Monitoring corrosion protection of pipelines utilizing the main survey techniques

Sarah Leeds

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Abstract

External corrosion control of buried pipelines uses two techniques, a protective coating and because no coating is fault free, Cathodic Protection (CP) is applied to prevent corrosion of steel exposed at fault locations. Cathodic Protection does not stop corrosion instead it slows it down to a very slow rate. The rate of corrosion is dependent on the applied potential and the nature of the soil in which the pipe is buried. These two works together to convert any dissolving steel due to the corrosion process into forming a protective film covering any exposed steel.

In trying to understand and monitor corrosion protection of pipelines and hence minimize coating faults it is necessary to consider both the properties of the coating and the interaction of the coating with the local environment at the faults. The most dangerous chemical that affect coatings is water within the soil that is driven to the steel surface by the CP and the CP electrochemical reactions that consume oxygen in the water present in soil and the decomposition of water resulting in both reactions generating sodium hydroxide. Hence in trying to understand coating fault properties and what must be monitored by any detecting technique then the most important inputs must be to understand the influence of CP and the local electrolyte. All the above ground inspection techniques commonly used to monitor corrosion protection will be assessed.

Chemical Qualification of Corrosion Inhibitors in the Oil & Gas

Industry: Impact of Test Approaches on Performance during Laboratory Screening

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Scaled Solutions Ltd, Livingston, United Kingdom

Abstract

Laboratory based assessment of corrosion inhibitors is critical in selecting and qualifying effective chemicals prior to field deployment. Final chemical qualification generally relies on approaches designed to replicate as close as possible, the full corroding environment. However the time and costs associated more sophisticated techniques means that such investigations are carried out in an iterative screening program; starting with simple tests followed by product deselection and followed by further more complex of the tests. The simplified screening tests then determine the preferred products and dose rates that pass to the next stage of testing. Therefore it is vital that these screening tests are carried out in as appropriate a manner as possible.

We have conducted research into how, seemingly small, changes to test methodology or conditions in these screening tests can have a significant impact on the performance of different products. Results illustrate the controlling effect of aspects such as pre-corrosion, the impact of the brine chemistry, CI composition, as well as the effectiveness of partitioning for different chemicals, on the performance and relative performance of different products. Results are presented which demonstrate considerable differences in the observed performance, ranking and outcome of the pre-screening of corrosion inhibitor chemicals caused by relatively small changes in the methodology adopted. This can affect which candidates are taken forward for qualification, and ultimately selection for application in the field.

This work highlights that understanding and a through design of corrosion inhibitor screening programs is important to eliminating possible errors and test artefacts.

Long-Term Corrosion Monitoring of Steel and Reinforced Concrete

Infrastructure

Paul Lambert

Mott MacDonald, Altrincham, United Kingdom

Abstract

Major infrastructure is rarely intended to last less than 50 years and more commonly expected to exceed 100 years or its usefulness. Such structures rely heavily on the durability of the materials of construction, principally steel and reinforced concrete. While many components lend themselves to relatively easy maintenance or replacement, the main structural elements can be difficult to access and even harder to repair or replace. Monitoring the condition of such structures to predict if and when some form of intervention is required can therefore be a valuable activity but brings complications. Such structures are typically massive and, if designed and constructed appropriately, unlikely to suffer any significant problems for several decades. Any monitoring therefore needs to be robust, future-proofed and correctly located to be of benefit. This presentation will describe a number of applications where corrosion monitoring, both quantitative and qualitative, has been used to predict the service lives of structural components, identify when intervention was required and demonstrate the effectiveness and longevity of remediation.

C13 – CED McIntyre Award

Steve Paterson: Paul McIntyre award recipient talk

Steve Paterson

Arbeadie Consultants, steve.paterson@rbd247.com

Abstract

After receiving the award, I will describe my experiences of corrosion engineering during my long career in the oil and gas industry, and my involvement in standards development, European collaboration and training future corrosion engineers.

The study of hot water corrosion for fusion reactor cooling circuits

David Kumar¹, Angus Siberry¹, Stacy Moore¹, Robert Burrows², Lucy Platts², Reuben Holmes², Alexander Warren¹, Ross Springell¹, Chris Harrington³, Tomas Martin¹

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Abstract

The corrosion processes in light water reactors (LWR) are currently mitigated through changes in water chemistry trialled in existing water-cooled fission reactors. However, the effect of different chemistry regimes on newly commissioned nuclear reactors is not fully characterised. The next generation of Gen IV reactors and water-cooled fusion reactor designs will present new challenges to water chemistry regimes.

One cooling design in nuclear fusion reactors is the water-cooled lithium-lead (WCLL) blanket, which will be tested in ITER and may be employed in DEMO. One benefit of using a water-cooling system is that best practice corrosion mitigation techniques from current LWR fission reactors may be introduced in the WCLL designs. However, these effects must be investigated for the different structural materials designed for fusion reactor environments.

This presentation will explore the effects of simulated fusion reactor conditions on the reduced-activation ferritic-martensitic steel Eurofer-97 which is Europe's candidate for structural materials in the WCLL blankets. As well as aqueous corrosion, Eurofer-97 in a coolant loop will be subjected to high neutron flux, temperatures, pressures, and magnetic fields. The resultant microstructural changes in the material needs to be investigated to determine the suitability of Eurofer-97 in the WCLL.

To investigate the effects of parameters such as pressure, temperature, chemistry, and applied field, an autoclave flow loop facility is being developed to simulate reactor conditions. Characterisation of samples exposed to reactor conditions will be presented through techniques including electron-microscopy, electron backscatter diffraction, X-ray diffraction, focused ion beam milling and high-speed atomic force microscopy.

Atomic layer deposition thin films from Al₂O₃ and HfO₂ as protection of commercially pure titanium for biomedical applications

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Abstract

In this study we focused on the deposition of Al₂O₃ and HfO₂ films on commercially pure titanium (CP-Ti) to explore their effect on corrosion properties of CP-Ti under simulating physiological solution. Atomic layer deposition (ALD) was explored as a preparation method using a trimethylaluminium as a precursor for Al₂O₃, and tetrakis(ethylmethyloamido)hafnium(IV) as a precursor for HfO₂ films. The film thickness was around 150 nm. CP-Ti samples were prepared in two different ways: (i) grinding using 500-grit SiC emery papers and (ii) grinding followed by polishing using silica suspension. Chemical composition analysed using time-of-flight secondary ion mass spectrometry and energy-dispersive X-ray spectroscopy analysis using focused ion-beam system confirmed the presence of stoichiometric Al₂O₃ and HfO₂ films.

Electrochemical measurements were carried out in Hanks' physiological solution at 37 °C. Both samples, Al₂O₃ and HfO₂ ALD-coated CP-Ti, exhibited for ca. 5 orders of magnitude lower current density in the passive region than bare CP-Ti, as shown by potentiodynamic polarization curves. Electrochemical impedance spectroscopy measurements confirmed that ALD Al₂O₃ and HfO₂ films on CP-Ti reached large impedance values at low frequencies, ca. 10⁹ Ω·cm² after 1h immersion in electrolyte. Only in the case of HfO₂ films on ground titanium surface the values were smaller, slightly below 10⁸ Ω·cm². Additionally, at prolonged immersion up to 40 days only ALD Al₂O₃ and HfO₂ films on polished titanium surface retained good barrier properties. Accordingly, films deposited on polished surface exhibit higher protection against corrosion than the films deposited on ground surface.

Influence of size and distribution of intermetallic particles in Al-Si cast alloys on the cerium conversion coating deposition

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Abstract

Aluminium alloys are susceptible to corrosion due to microgalvanic coupling of the Al matrix and intermetallic particles (IMP). Among different protection strategies, conversion coatings can be selectively deposited on more noble sites. Cerium conversion coatings (CeCC) have been proven to inhibit the cathodic reaction by preferentially depositing on the cathodic IMP. Their spontaneous deposition achieved by immersion of aluminium substrate in Ce-ion containing baths is a consequence of the localised pH increase because of oxygen reduction reaction at local cathodic sites of the microstructure. The influence of bath process parameters (like cerium salt type, cerium ion concentration, pH etc.) have been widely investigated primarily on AA2024 and AA7075, but the more general role of microstructure remains to be clearly understood. Due to the localised nature of deposition reactions, the microstructure, in the sense of chemical composition, size and distribution of the second phase or IMP play a crucial role in the deposition behaviour. This research investigates the role of IMP size and distribution in the microstructure, i.e. how far or close they are spaced from each other, on the deposition of CeCC. Two model cast Al-7wt.% Si alloys are selected with 2wt.% Cu and 1wt.% Fe respectively. Different IM distribution in the microstructure is achieved by implementing slow and fast cooling rates for solidification of these hypoeutectic Al-Si alloys. CeCC coatings are spontaneously deposited by immersion in cerium chloride salt solutions. Bare metal and CeCC deposited microstructure is characterized with optical microscopy, scanning electron microscopy and energy dispersive x-ray spectroscopy.

Modelling the Behaviour of Aluminium Flakes in Marine Coatings Through the Use of Agar Gels

Jessica Moulton¹, Stuart Lyon¹, Douglas Beaumont², Charlotte Moon²

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Abstract

Offshore structures are commonly protected from corrosion through the combination of anticorrosive coatings and cathodic protection. Although this combination is effective at preventing corrosion, it can lead to cathodic disbonding, a coating degradation mechanism where the anticorrosive coating is displaced from the substrate. This removal of the coating is due to the presence of a high pH aqueous film between the coating and metal substrate.

Aluminium flakes can be formulated into marine coatings in order to delay the onset of cathodic disbonding. However, the mechanism behind this affect is not fully understood. One mechanism under investigation is a pH reduction. In this proposed mechanism the aluminium oxide present on the aluminium flake's surface reacts with the hydroxyl anions present. This would lead to a reduction in the local pH and thus, prevent cathodic disbonding until there was no more aluminium oxide present.

To begin to understand the role aluminium flakes play in marine coatings, a model system was developed. This system was comprised of agar gel, sodium chloride, aluminium flakes and either phenolphthalein or universal indicator. pH indicators have previously been used to study localised corrosion mechanisms, however when used previously, the indicators have only been incorporated into transparent, pigment-free coating or gel samples. In this work, these agar gel samples have been used in an electrochemical cell in order to observe the aluminium flake's pH behaviour during corrosion. These experiments were able to show the different pH regions within the agar gel.

Mechanisms of life enhancement by laser shock peening surface treatment of 7XXX aluminium alloys

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Abstract

Laser shock peening (LSP) is an up and coming surface treatment used in the aerospace industry to improve fatigue performance of aircraft components. There is a growing interest to investigate and understand the effects of LSP on corrosion performance on aerospace grade aluminium alloys. In this study, two types of LSP treatments were used on aluminium alloy 7075-T651. LSP with ablative coating (Ab-LSP) and without ablative coating (nAb-LSP) were performed to generate a compressive residual stress field at and below the alloy's surface. X-Ray diffraction and centre-hole drilling showed up to 300 MPa and 400 MPa compressive residual stresses in Ab-LSP and nAb-LSP samples, respectively; with residual stresses present up to 1 mm depth. Characterisation techniques such as contact profilometer, micro and nano-hardness, SEM-EDS and EBSD were used to understand the effects of LSP on the roughness, hardness and microstructure of the treated samples. Corrosion tests were performed on untreated, Ab-LSP and nAb-LSP AA7075-T651 with the aim to compare corrosion performance and understand the mechanisms that lead to changes in corrosion behaviour. Potentiostatic polarisation was used to generate discrete localised corrosion features for assessment of long-term corrosion performance. Variable focus microscopy and X-ray CT-scanning, together with image processing techniques, are used to analyse the corroded samples in 2D and 3D, to assess and compare pit population density, aspect ratio and morphology at the LSP surface and affected volume, as well as looking at interactions between pit features and alloy microstructure.

Electrochemical Study of the Effects of Adding Pigments to Zinc-Rich

Coatings

Isehaq Al-Nafai, Stuart Lyon

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Abstract

This study aims to investigate the possibility of adding other conductive pigments to ZRC and to test their anticorrosion efficiency with and without the additional inhibiting pigments. The question is: can pigments such as graphene, carbon black or aluminium enhance the corrosion protection by offering more electrical contact between zinc particles, or do they have the opposite effect? To test the effect of adding other pigments to ZRC, working electrodes of glassy carbon (GC), aluminium, zinc and iron were used. The primary methods in this study were the potentiodynamic polarisation and the EIS. The coupling of these electrodes with the smart potentiostat revealed the corrosion potential (E_{corr}) of each of them. The most active E_{corr} was for zinc which explains its use in ZRC while the noblest E_{corr} was for glassy carbon. On the other hand, aluminium shows passivity which can block the electrical connection between zinc particles. Moreover, the EIS data supported these conclusions by the very high impedances of GC and Al. As a result, the recent trends of adding carbon to ZRC raise a concern of whether adding carbon to ZRC will enhance corrosion protection or has adverse effects. Additionally, the influence of phosphate inhibitor has been investigated on pure WEs and standard ZRC where it was found to partly passivate and protect the metal substrates. Future work with model coatings containing Zn, carbon black, Al and graphite with different ratios will illustrate a clearer view on the effects of adding conductive pigments to the ZRC.

Reducing the zinc content in protective marine coatings

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Abstract

Every year around 4 million tonnes of zinc is used in the form of coatings to protect roughly 100 million tonnes of steel. The majority of zinc or zinc-alloy coatings are applied from a molten bath of metal or are electroplated. A rather significant amount comprises “cold galvanising” where the zinc metal is contained within an organic coating which is applied to steel like paint. It is assumed that zinc-rich coatings can only work in a similar fashion to metallic coatings, i.e. sacrificially. Nevertheless, environmental and cost pressures are strong drivers encouraging the reduction in the quantity of zinc in such materials. Interestingly, dissolution of the sacrificial zinc during service will release into the environment divalent zinc cations, which are extremely effective corrosion inhibitors. It remains unknown whether these species also contribute to the corrosion protection performance of zinc-rich paints.

The research explores the hypothesis that zinc-rich paints are effective primer coatings on steel via two mechanisms: (a) the zinc corrodes sacrificially to galvanically protect the steel; (b) a sacrificial corrosion of zinc causes zinc cations to be released which act as effective corrosion inhibitors. This presentation will discuss the quantitative characterisation of zinc rich coating’s performance with regards to the amount of zinc that corrodes during sacrificial protection in different model zinc coating systems. Potentiostatic polarisation, Electrochemical Impedance Spectroscopy (EIS), and Scanning Electron Microscopy (SEM) have been employed to characterise the zinc rich paints.

Poster abstracts

P1

Evaluation of electronic properties and Volta potential distribution of CoCrMo alloy in presence of protein molecule by SKPFM and electrochemical measurements

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Abstract

In this research, the effect of protein absorption in a phosphate buffered solution containing bovine serum Albumin (BSA) protein on the semiconductor characters of passive film formed on CoCrMo alloy was studied by means of SEM/EDS, atomic force microscopy (AFM), scanning Kelvin probe force microscopy (SKPFM), and electrochemical measurements. Different concentrations of BSA have been employed and for the entire concentration range of BSA from 0 to 2 g.L⁻¹, a lower corrosion potential and differences on the passivity region were detected. The potentiodynamic polarization and electrochemical impedance spectroscopy analyses indicate that BSA concentrations, lower than 0.5 g.L⁻¹, provide higher passive current density and lower the charge transfer resistance. Likewise, the higher concentrations of BSA, till 2 g.L⁻¹ have considerably increased the corrosion resistance of the formed passive film. This is due to the fact that the BSA molecules strongly adsorb on the passive film by electrostatic or hydrophobic interactions and thus inhibit the electrochemical reactions. Mott-Schottky analysis demonstrated that the BSA protein in the high concentrations has a positive effect on reducing the density of defects including n-type or p-type semiconductor characters and space charge regions in the passive film. AFM and SKPFM mappings visualized the presence of BSA protein adsorption on passive film with different topography and Volta potential distribution with respect to the matrix. SEM images showed that BSA protein/passive film interface and surface of protein is a susceptible sites for corrosion initiation due to different Volta potential and suitable place for adsorption of counterions e.g. Cl⁻.

P2

Effect of anodizing parameters on the formation of TiO₂ nanotubes produced on titanium grade 2 and titanium grade 5

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Abstract

Ti-based implants are the most widely used solution for bone repair and joint replacement, due to the favorable properties of titanium and its alloys when compared to other commonly used metals, namely, stainless steel and cobalt-chromium alloys. In short, titanium grade 2 and titanium grade 5 present good mechanical properties and biocompatibility without showing toxicity or immune rejection. However, although the inertness of titanium has been considered a favorable characteristic for years, its lack of bioactivity also presents some challenges, regarding inability to actively regulate osteoblast behavior or battle colonization by pathogenic microorganisms.

Titanium anodization is a simple and effective surface treatment for the improvement of osteointegration and antimicrobial activity. Extensive work has been performed on the study of the production titania nanotubes on the surface of commercially pure titanium (grade 2) but little work exists on the production of Ti nanotubes on the surface of Ti6Al4V (grade 5) alloy the most widely used alloy in orthopedic implants. The aim of this work is to produce and characterize titania nanotubes on the surface of Ti gr.2 and gr. 5 using different anodizing parameters. An ethylene glycol electrolyte containing 0.5wt.% NH₄F and 2.5% V H₂O was used for the anodization at room temperature. The applied voltage varied from 20 to 120V and the anodization time from 30 to 180 min. The obtained films have been characterized by SEM and XRD regarding the microstructure with particular attention to the structure of TiO₂ nanotubes on the α and β phase grains of Ti6Al4V.

Electrochemical Insertion of Lithium Ion in Copper Hexacyanoferrate employed as Cathode Material in Rechargeable Batteries.

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Abstract

The Prussian blue analogues (PBAs), are a promising candidate to use in large-scale energy storage application because of their long cycle life, high power density and high energy efficiency. Along with the change of transition metal, some structural parameters and some key properties of PBAs may be varied. Therefore, achieved voltage, specific energy and rate capability of the battery may be affected.

In this work, the process of electrochemical insertion of lithium ions in PBAs, Copper Hexacyanoferrate (CuHCF_e), was studied. This was synthesized by a hydrothermal process and was used as an active material in cathodes of Lithium-ion batteries.

In a first stage, the synthesis of CuHCF_e was carried out employing a factorial design as an exploratory study of the variables significance such as precursor concentrations, temperature and reaction time.

The samples obtained by the design were characterized by Field Emission Scanning Electron Microscopy (FESEM) and by X-ray diffraction (XRD). In the CuHCF_e samples, cubic structures with a high degree of crystallinity are obtained, in addition a dependence of the particle size with the variation in the synthesis conditions is evident.

Finally, Lithium-ion batteries were built using the CuHCF_e as the active material. Cyclic voltammetries and charge/discharge curves were performed to obtain the capacity, cyclability and efficiency values. The values of specific capacity for CuHCF_e ($C_T = 85,1 \text{ mAhg}^{-1}$) were close to 60 mAhg^{-1} for smaller samples size and close to 30 mAhg^{-1} for large samples size showing the influence of particle size on the performance of the battery.

In-situ study of corrosion of commercial SD memory card by local electrochemical techniques

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Abstract

Nowadays, due to living in an era of highly sophisticated electronic and communication technology, micro- and nano-electronic devices have extended to all aspects of our daily life. Corrosion deterioration in the micro- and nano-scale of electronic devices, in which a number of metallic components exist in different forms, leads to a decrease in the electrical performance, efficiency and lifetime. Due to a colossal surface-to-volume ratio of small-scale electronic devices, corrosion deterioration including galvanic coupling, crevice corrosion, and even uniform corrosion significantly influences the performance and lifetime of the devices. Fundamental understanding of corrosion behavior of miniature electronic devices in their service environments could lead to superior long-term stability of the system through a knowledge-based design of materials and protective coatings. The objective of this work is to obtain a comprehensive understanding of the localized corrosion behavior of the final metallic finish of the commercial SD memory cards, electroless nickel immersion gold (ENIG), by applying surface analysis and scanning electrochemical techniques with high-lateral resolution. In order to achieve this, Atomic Force Microscopy (AFM), Scanning Kelvin Probe Force Microscopy (SKPFM), Scanning Electrochemical Microscope (SECM), Scanning Vibrating Electrode Technique (SVET), and Scanning Electron Microscopy (SEM) are used to investigate the in-situ corrosion phenomena exposed to different corrosive environments. Combining the morphological and electrochemical techniques will provide complementary information to elucidate the in-situ corrosion mechanisms possibly leading to electronic device failures, i.e. predestined sites for initiation, and kinetics and preferable direction of corrosion propagation.

Modification of Carbon Electrodes from Acylated Pyridine Species: A Study of the Steric and Electronic Effects of Pyridine Radicals on the Grafting Process

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Abstract

The modification of carbon electrodes via the attachment of pyridine radicals is well documented. [1] - [3] It remains an attractive area of electrochemistry due to the opportunity to further react the pyridine molecules after they have bonded to the electrode surface. [4] In the past, modification was carried out via the reduction of diazopyridinium cations which, after the loss of dinitrogen, produced a pyridine radical that bonded to the carbon electrode surface. [5] In this study we synthesised acylated pyridine cations that, upon reduction, formed an organic layer on the surface of the carbon electrode. This was verified via a range of techniques, including but not limited to: Cyclic Voltammetry (CV), Scanning Electron Microscopy (SEM) and X-ray Photoelectron Spectroscopy (XPS). Further investigations identified that the presence of different substituents on the pyridine ring either hindered or encouraged the formation of the organic layer. Density Functional Theory (DFT) calculations were then carried out to investigate the correlation between the substituents and possible binding sites on the pyridine ring.

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P6

Comparison Between Laminate and Copolymers of 1,3,5-Tris(N-carbazolyl)benzene and 3,4-ethylenedioxythiophene: A Spectroelectrochemical Study

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Abstract

A strategic approach to evident the copolymer and laminate formation was performed by chronoamperometric technique. Poly(1,3,5-Tris(N-carbazolyl)benzene) and its laminate and copolymer with 3,4-ethylenedioxythiophene were electrochemically synthesized. All the spectroelectrochemical measurements were performed in TBAPF₆/ACN. The studies revealed that the spectroelectrochemistry is a powerful tool to distinguish between the two. Also, laminates and copolymers show different optoelectronic properties and found promising applications in electrochromic devices. The effect of monomer concentrations on the band gap of copolymers was also studied.

Materials and Architectures for Ionic Rectifiers

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Abstract

Ionic rectifiers or diodes could be useful in water purification and in sensing process. Ionic diode phenomena occurs for example at asymmetric ionomer | aqueous electrolyte microhole interfaces. Depending on the applied potential, either an open or a closed diode state is observed switching between a high ion flow rate and a low ion flow rate. The ionic diode concept is introduced and explained based on the example of titanate nanosheet ion conductor. The strength of the 2D-titanate is controlling selectivity for ion transport through lamella-type spaces between the nanosheets. However, there is exciting materials such as graphene oxide behave as a cationic diode are highly interesting for water purification. In addition to the materials and ion transport effects in various types of pore spaces, the architecture of ionic diode devices is of interest. The first example of polymer intrinsic of microporosity (PIM) | Nafion interfaces and their properties provide improved rectification effects. The further work of this heterojunction has been exploited in diode mechanism in a potassium sensor context. The mechanism of potassium diode sensing based on precipitation reaction of potassium cations with perchlorate anions to give insoluble KClO_4 at interface PIM | Nafion interface and shown to be fast and reversible.

Studies towards applied anodic oxides

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Abstract

Conventional top-down fabrication processes such as nano-lithography have been hugely successful for device fabrication, especially in the semi-conductor industry. However, further opportunities to produce new geometries and structures are offered by the tantalizing possibility of bottom-up, atom-by-atom, fabrication of real-world devices and catalysts. Mastering electrochemical and electroless deposition processes, particularly in the confined spaces seen in cheap nanoscale templates such as porous anodic alumina (PAA) is an important step towards such technology.

In this contribution a summary of our recent work using various synchrotron-based X-ray techniques such as Grazing-incidence Transmission Small Angle Scattering, X-ray microscopy, X-ray adsorption and X-ray fluorescence as well further characterizations is presented. We are trying to understand both the formation of such templates, their customization, optimization, and how electrodeposition processes within-in nanopores take place and the impact of confined spaces upon the crystallinity of deposits formed. The existence of the barrier layer at the base of PAA creates challenges for homogenous electrodeposition directly into anodized alumina, we have investigated how to some extent this can overcome by chemical etching and reducing the anodizing potential towards the end of anodization. Furthermore, we have produced samples containing various Pd deposits and have investigated the impact of the confined porous geometry of anodic alumina towards the oxidation of CO in the gas phase using planar induced laser fluorescence.

Azo dye treatment with simultaneous electricity production in an air cathode microbial fuel cell coupled activated sludge process

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Abstract

A combined anaerobic–aerobic process involving an air-cathode microbial fuel cell (MFC) followed by an aerobic downstream treatment process was used for the complete removal of reactive orange 16 using acetate as co-substrate. The discharge of dye wastewater is undesirable, not only because of color but also because many azo dyes and their breakdown products are toxic and/or mutagenic and obstruct the light penetration and oxygen transfer into water, hence affecting aquatic life. The concept of MFC has been already well established for the production of electricity; however, not much work has been published regarding dye decolorization with simultaneous electricity generation. Biodecolorization via anaerobic reductive process is usually very slow and requires an electron donor (organic co-substrate) to create necessary reductive conditions. The degradation of azo-dye in MFC results in the formation of aromatic amines with simultaneous electricity production. The degradation products were further treated in an activated sludge downstream process. More than 90% of the chemical oxygen demand was removed in the combined process. The maximum output cell potential and the coulombic efficiency were 423 mV and 3.4% respectively. Surface characterization was studied by SEM. Cyclic voltammetry revealed that a redox process took place. The results of the GC/MS technique showed that RO 16 was first converted into aromatic amines in MFC which were further transformed into phthalic acid and finally into benzoic acid. The results demonstrate that complex and toxic dye wastewater can be successfully treated in MFC followed by aerobic post-treatment along with energy recovery.

Lunar resource extraction: the Metalysis-FFC process

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Abstract

The production of oxygen in-situ on the lunar surface would help to enable in-space refuelling and sustainable space exploration. Moreover, the simultaneous production of oxygen and metals/alloys would be highly beneficial. The most plentiful and accessible feedstock source on the lunar surface is regolith, a thick layer of unconsolidated fine-grained material covering the entire lunar surface. The Fray-Farthing-Chen (FFC)-Cambridge Process, invented in the late 1990s as a direct electrochemical method for producing metals, has been suggested as an efficient method for the production of both oxygen and useful metals from lunar regolith. In the terrestrial context, a carbon-based anode facilitates the removal of oxygen from the system in the form of carbon dioxide and carbon monoxide. In the lunar context an inert anode can be employed to directly produce oxygen. In the almost two decades since this novel process was first reported, significant progress has been made in developing this technology. Metalysis (UK) have successfully scaled-up and commercialised the production of a number of metals and alloys. The current research investigates the reduction of lunar regolith simulant with the Metalysis-FFC process, applying the processing innovations of the past decade. Maximising efficiency, in terms of quantity of oxygen extracted and the functionality of materials produced versus energy consumption and process complexity are key goals. The processing knowledge gained in the years of terrestrial technology development will inform the design of a process that has the ability to be scaled up and operate sustainably and autonomously on the lunar surface.

Development of Ionomer Functionalised Carbon Paste Electrode and Modified Glassy Carbon Electrode for Detection of Mercury in Sea Water

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Abstract

Anion-conducting polymers are increasingly being used in the development of alkaline anion-exchange membranes (AEM) / membrane fuel cells (AEMFC), holding promise for of non-Pt group of electrocatalysts. The polymers used in this work are ETFE-g-poly(vinylbenzyltrimethylammonium chloride) – (BTMA)¹ and 2,2'',4,4'',6,6''-hexamethyl-p-terphenylene poly[2,2'-(m-mesitylene)-5,5'bis(N,N'-methylbenzimidazolium)] (HMT-PMBI)².

Carbon paste electrodes (CPEs) were fabricated using graphene nanoplatelets, BTMA and Nujol. The paste is used in an electrode and polished for electrochemical investigation using cyclic voltammetry (CV).

GCE were coated with HMT-PMBI dropcasting 5µl polymer.

The modified CPE and GCE have been used to incorporate negatively charged redox mediators, potassium hexacyanoferrate(II) and Potassium Hexachloroirridate(IV) in background electrolyte 0.1M NaCl, investigating the compliance with Randles-Sevick equation where the peak current is proportional to the square root of scan rate, all measurements taken using 1% polymer.

The modified GCE and CPE electrodes were utilised for the detection of mercury in the presence of excess copper as interference using differential pulse voltammetry (DPV). The limit of detection of mercury is 100nM using DPV in 0.5M NaCl and 300nM in Seawater sample (Swansea Bay) adjusted to pH4, after 30 minutes preconcentration in stirred solution, open circuit, at 0.03V (vs. KCl saturated Ag/AgCl).

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Time and pH Dependence of the Open-Circuit Potential of Pt and Au in Aqueous Electrolytes in the Presence and Absence of O₂

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Abstract

Platinum remains the benchmark catalyst for oxygen reduction in electrochemical technologies such as fuel cells and sensors.¹ During operation a bias potential/current is applied to control the electrode reactions, however imbalanced conditions are experienced at start-up and shutdown where the device is near its rest potential.^{1,2} Local O₂ starvation or surplus at the catalyst may alter its polarisation behaviour and contribute to degradation.² The time and pH dependence of the open-circuit potential (OCP) of a Pt rotating disc electrode is analysed in oxygen-saturated and oxygen-depleted aqueous media. The OCP is sensitive to the presence of O₂ and shifts positively into the Pt oxide region. This behaviour is interpreted as a mixed potential between hydroxyl adsorption and O₂ reduction at different sites of the equipotential surface,^{1,3} balancing to zero net current (Figure 1). The dynamics of Pt oxide growth and stabilization of the OCP depend on the flux of O₂ to the electrode. The OCP at Pt and Au under inert conditions shows a complex dependence on time, the state of the electrode, and pH. In the absence of a driving force to pin the OCP, background processes including surface mixed potentials³ and interfacial charge

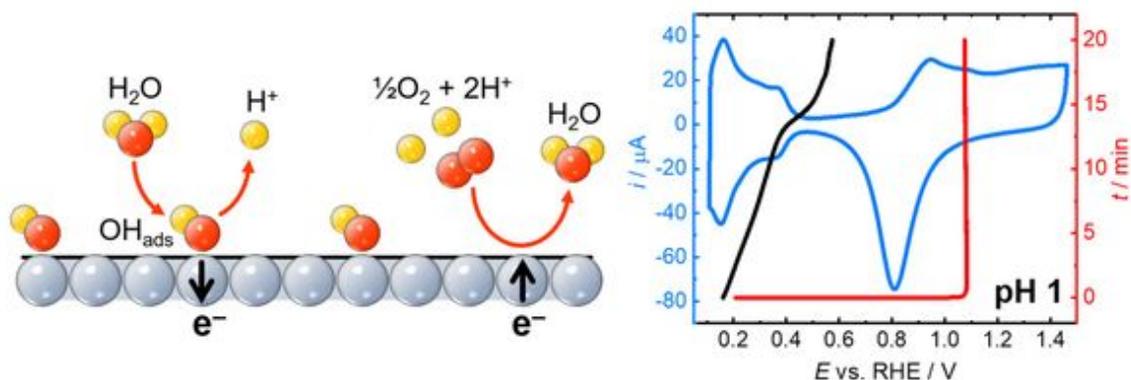


Figure 1: Schematic illustration of the mixed potential at Pt exposed to oxygen, and a plot of the voltammetry at Pt overlaid with the OCP transients in oxygenated (red) and deoxygenated (black) 0.1 M HClO₄.

contribute to establishing the potential with time. This presentation will discuss the processes that control the Pt-aqueous interface at rest under these conditions, giving insight into the effects on the catalyst surface.

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On-line stability investigations of platinum electrodes in non-aqueous systems using a scanning flow cell

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Abstract

The scanning flow cell (SFC) combined with inductively coupled plasma mass spectrometer (ICP-MS) has been proven to be a powerful technique for high-throughput investigations of stability of electrocatalysts. This measurement set-up gave valuable insights into fundamental dissolution mechanisms, effecting electrocatalytic processes in aqueous media.

However, electrochemistry in aqueous media has its limitations which include, the narrow electrochemical window, limited stability of metals and solubility of substances with potentially interesting electrochemistry. Therefore, many applications like batteries, capacitors or sensors require organic electrolytes. In order to develop economically feasible processes and devices, long-term durability of electrode materials is essential.

In this work, we present applications of the SFC ICP-MS setup for non-aqueous media. The adaptation for non-aqueous systems might sound straightforward, but its implementation is far from trivial and requires major changes to the presently successfully used system. Starting with selecting stable materials in aggressive organic solvents or, finding a suitable electrode configuration that is compatible with the low conductivity of non-aqueous electrolytes. Furthermore, the analysis of organic samples in the ICP-MS requires significant modifications and adjustments of the device. For instance, addition of oxygen is essential to prevent soot formation that leads to orifices clogging of the cones or, using platinum instead of nickel cones, due to better stability in aggressive plasma conditions.

As a proof of concept, we present platinum dissolution in methanol based electrolytes and show the completely different characteristics of these processes to aqueous electrolytes. The influence of cations, anions and water contamination on platinum dissolution is discussed.

Imprinted polyaniline-based screen printed electrodes for sensing of toxic organic compounds

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Abstract

Polyaniline (PANI) as a basic receptor material was electropolymerized on the commercial gold screen-printed electrode by cyclic voltammetry in acid solution. According to the FEG-SEM analysis, the gold electrode is firstly covered with a nanolayer of deposited polymer and with time polyaniline starts to form nanofiber structure on the surface. An emeraldine salt, the most conductive form of polyaniline, was confirmed by its characteristic peaks obtained by FTIR and by the observed distinctive green colour of the electrode. Imprinted PANI was prepared by adding propanamide into starting polymerisation suspension. FTIR results showed two new peaks in the range of 2890 and 3320 cm^{-1} , characteristic for alkanes and amides, which confirms successful imprinting of propanamide. Upon observing the redox-reversible polyaniline transition between emeraldine and leucoemeraldine by cyclic voltammetry, it was possible to observe the difference in current responses in the case of imprinted and non-imprinted polymer. This was attributed to the PANI conductivity as a consequence of delocalized electrons of conjugated π -bonds. With the addition of a non-conductive organic molecule (template molecule), the electron transfer along the polymeric chain is assumed to be suppressed, as observed in our experimental study. Using imprinted PANI enables a semi-direct electrochemical sensing of structural analogues of propane amide, like acrylamide, without using redox probe molecules.

Influence of the acidity of ionic liquids on the double layer properties of the Pt/PIL interface

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Abstract

Nowadays, an important application of fuel cells is in the automobile industry. Intermediate temperature polymer electrolyte fuel cells (IT-PEFCs) have much easier water and heat management, and a higher CO tolerance in the feed gas, which can be operated in a temperature range of 120-140 °C.^[1] Proton conducting ionic liquids (PILs) exhibit a high ionic conductivity, a wide electrochemical window and a high chemical and thermal stability.^[2] Therefore, they can be used as electrolytes in IT-PEFCs.

In this contribution, a study on the double layer properties of the interface between platinum and the PILs is investigated by electrochemical impedance spectroscopy (EIS). Three ionic liquids ([2-SEMA][TfO], [EIm][TfO] and [DEMA][TfO]) with different acidities will be compared.

Plots of the potential-dependent data from EIS measurements in the complex capacitance plane (CCP) show, that at least two differential double layer capacitances are obtained, depending on the potential, water concentration and temperature. The differential capacitance of the fast process can mainly be attributed to charge redistributions at the interface and the ion transport in the double layer. The slow capacitive process is caused probably due to charge redistributions in the innermost ion layer. This process requires a higher amount of activation energy for reorientation of the ions, since they are strongly bond to the electrode in this layer.

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Operando Studies of the Potential Dependent Structure and Stability of Cu(111) Electrodes

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Abstract

Copper and copper oxide electrode surfaces are particularly suitable for CO₂ electro-reduction, an important process for the futures of renewable energy and the environment, with the product selectivity being strongly influenced by the surface structure of the copper electrode. Surface x-ray diffraction can be used to study the Cu surface structure in the electrochemical environment thus allowing the electrochemical reactions to be correlated with the changes in the atomic structure at the surface. The Cu(111) electrode surface was investigated in a phosphate buffered electrolyte solution, with pH = 8. A roughening of the copper surface due to the formation of a mixed copper-oxygen layer is observed as the phosphate adsorption and deprotonation of (di)-hydrogen phosphate processes occur. The results show the importance of the preparation of the Cu electrode, as the morphology of the Cu surface is strongly dependent on the method of preparation. The presence of copper islands on the surface of the Cu(111) electrode leads to irreversible changes in surface morphology via a 3D Cu growth mechanism. Measurements of the Cu(111) electrode in acetonitrile electrolytes give additional insight into the restructuring of the Cu surface. In this case Cu islands are formed during potential cycling due to Cu dissociation and re-deposition. The mechanisms for formation of the twinned Cu(111) surface in both phosphate and acetonitrile electrolytes can be compared. Knowledge of the electrode surface structure is essential as it correlates strongly with electrochemical activity.

Carbohydrate Amperometry with Pyranose-2-Oxidase Biosensors

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Abstract

Pyranose-2-oxidases (P2Os) are biocatalysts that facilitate the electron transfer from aldopyranoses to molecular oxygen, generating 2-keto-pyranoses and hydrogen peroxide as products. The P2O from the white rot fungus *Trametes multicolour* was covalently bound to precoated protein thin films on platinum disk electrodes. This enzyme immobilization produced biosensors for sensitive amperometric carbohydrate detection with a simple anodic H₂O₂ readout. The performance of the obtained sugar detectors was tested in calibration trials against the most prominent P2O substrate glucose and for the less common P2O sugar targets galactose, xylose, arabinose, cellobiose, maltose, mannose, fructose, sucrose, lactose and rhamnose. Obtained raw data of performed P2O sugar amperometry and related representative calibration curves will be shown as experimental evidence for the noticeable influence of the sugar identity and hence varied strength of the affinity of enzyme/substrate interaction on the biosensors current signalling and their detection capability. Feasible for glucose as best target of P2O biosensing were a clear sub- μ M practical detection limit and a prompt sensor response. And the assessment of model samples with known since adjusted glucose content revealed adequate analyte recovery rates.

P18

Thermogalvanic and pseudo-thermocapacitive conversion of low-grade thermal energy to electricity through gelled-electrolyte thermoelectrochemistry

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Abstract

Thermoelectrics are solid-state semi-conductor devices that can convert a temperature gradient into electricity (without moving parts). However, most thermoelectrics are based upon rare earth metals, limiting their application. Thermoelectrochemistry can be used to develop thermogalvanic devices; either gelled or liquid-state systems that also convert a temperature difference between two electrodes directly into electricity without moving parts, *via* entropy-driven redox processes.

Iron is abundant, cheap, and both the $[\text{Fe}(\text{CN})_6]^{4-/3-}$ and $\text{Fe}^{3+/2+}$ redox couple is well established. Both of these redox couples have a high Seebeck coefficient of either -1.4 mV K^{-1} or $+1.4 \text{ mV K}^{-1}$ [1]; the negative or positive sign indicates that the hotter electrode acts as the anode or cathode respectively. [1] Thermal energy can either be thermogalvanically converted directly to electricity, or harvested and stored by a pseudo-thermocapacitive mechanism.

In this study, we have investigated gelled electrolytes containing the $[\text{Fe}(\text{CN})_6]^{4-/3-}$ and $\text{Fe}^{3+/2+}$ redox couples. The gels were optimised for thermogalvanic thermal energy conversion, where the gels were found to only display thermogalvanic conversion under short timescales. Under longer timescales significant pseudo-thermocapacitance was observed, altering the electrode surface from graphite to high electroactive surface area electrodes, such as carbon nanotube-based modified carbon cloth electrodes were found to further increase the pseudo-thermocapacitance. With the highest absolute capacitance reaching *ca.* 200 F per gel, and a capacitance density of *ca.* 150 F cm^{-2} .

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Electrochemical Fabrication and Characterisation of Copper Oxide

Structures on Boron Doped Diamond for CO₂ reduction

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Abstract

Copper oxide (Cu₂O) has gained ground as one of the most promising electrocatalysts for electrochemical reduction of CO₂. However, chemical synthesis of these structures often involves several complex steps.^{1,2} We present a simpler electrochemical method of producing cubic Cu₂O nanostructures on boron doped diamond (BDD) electrodes. BDD makes an excellent substrate for electrocatalysts due to its inert nature, reducing side reactions that could interfere with products. Recent advances in BDD processing enable its use as a combined transmission electron microscopy (TEM) – electrochemical substrate,³ allowing direct electrochemical deposition of Cu₂O cubes on the BDD surface for high resolution analysis. Direct electrodeposition on the BDD-TEM substrate removes several delicate steps usually required for the transfer of particles to a TEM suitable substrate.⁴ Experiments are presented, which couple the Cu₂O – BDD electrodes with on-line electrochemical - mass spectrometry analysis to identify product formation during the CO₂ reduction process.

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Peptide Nanocarriers for the Detection of Heavy Metal Ions Using Resistive Pulse Sensing

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Abstract

The use of nanocarriers within resistive pulse sensing facilitates the detection and quantification of analytes. To date the field has been dominated by polyionic carriers or nanomaterials. These combine the recognition elements of a ligand and a stable support to facilitate the sample handling, decrease analysis times and enhanced signals. Here we develop the use of peptide aptamers on superparamagnetic nanocarriers to extract and quantify metal ions in solution. The selective binding of the Nickel ions to the peptide ligand is measured as a change in translocation velocity of the carrier. The velocity of the nanocarrier is proportional to the concentration of the metal ions in solution. Unlike DNA aptamers that have a polyanionic back bone, the peptide aptamers were neutral under the assay conditions. To attempt an enhanced signal, we engineered charged groups within the peptide to explore the effects on the signal. In all cases the metal ion binding dominated the velocity of the carrier. The assay was shown to work across three orders of magnitude and can detect Ni²⁺ in the presence of other heavy metal ions. We demonstrate this by quantifying Ni²⁺ in both tap and pond water. The work allows for future multiplexed sensing strategies using both peptides and DNA aptamers in resistive pulse sensors.

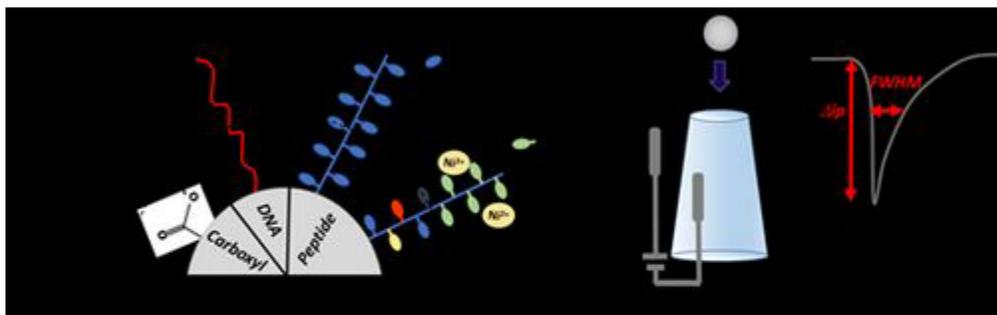


Figure 1: a) Schematic of the nanocarrier surface and the ligands used. The sections inner and outer refer to the part of the DNA or peptide closest and furthest from the particle surface. b) Schematic of a nanocarrier traversing the RPS device, and the signal. Pulse magnitude Δi_p and Full width half maximum are shown.

A paper-based electrochemical aptamer biosensor for monitoring antibiotic pollution

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Abstract

Antibiotic pollution has become a major global public problem. The widespread use of antibiotics often results in significant negative impacts on water and soil resources. This may accelerate the emergence of antibiotic resistance in natural bacterial flora in the future. Antibiotic resistance can cause diseases that used to be easily treated with antibiotics to become dangerous infections. In this work, a novel paper-based electrochemical aptamer biosensor was developed for monitoring oxytetracycline (OTC) in water. Amino functional graphene/thionine/gold nanoparticles (NH₂-G/Thi/AuNPs) nanocomposites were coated on screen-printed working electrodes (SPWEs) to improve the detection sensitivity, and a probe DNA aptamer was immobilized to capture OTC. Cyclic voltammetry (CV), differential pulse voltammetry (DPV) and electrochemical impedance spectroscopy (EIS) were used to detect OTC. The biosensor shows a linear response ranging from 100 pg μL^{-1} to 50 ng μL^{-1} , with a detection limit of 100 pg μL^{-1} . As a proof-of-principle, our work demonstrates sensitive and selective detection of antibiotics, with potential application for the rapid and low-cost monitoring of antibiotic pollution.

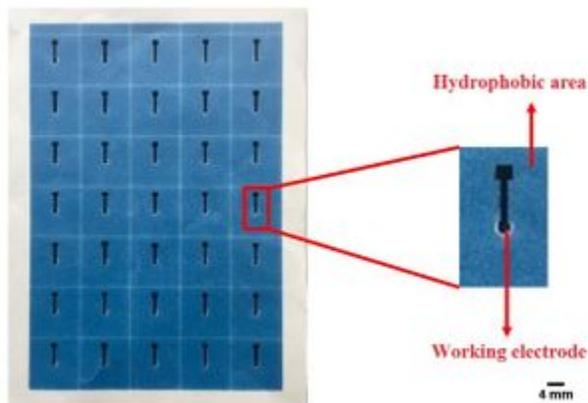


Figure 1. Screen-printed working electrodes (SPWEs).

Characterisation of a 200 kW/400 kWh Vanadium Redox Flow Battery

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Abstract

A full system characterisation of a 200 kW/400 kWh all-vanadium redox flow battery has been carried out. The battery is in Switzerland as part of the Grid2Mobility Demonstrator at the EPFL Energy Centre. The data revealed an overall system AC efficiency varying between 48% and 60% over the AC power range of 50–200 kW. However, analysis of the data indicated that lower system efficiencies largely originated from the regulatory procedures (including the centrifugal pumps and cooling system) as well as the AC/DC and DC/DC converter losses which combined to contribute up to 24% of the system energy consumption. From the electrochemical characteristics of the battery, the voltage and coulombic efficiencies were evaluated and were found to be in the range of 80–93% and 73–97%, respectively. The main source of electrochemical energy loss for this battery originated from the energy consumed during the charge part of the cycle with the temperature of the electrolyte playing a large role here. This energy loss could be minimised by monitoring the electrolyte temperature and operating the charge cycle when the temperature was below 40 °C.

A primary goal of this work was to analyse how this energy storage system could support the energy security of the local community, using the intermittent energy from the renewable generation for storage or as a cheaper, back-up power source for the 50 kW alkaline electrolyser or the 50 kW electric vehicle recharging application. The investigation carried out of this battery will be reported.

P23

***Initiation of Stress Corrosion Cracking in Zircaloy Nuclear Fuel Cladding
Examined using High-Speed Atomic Force Microscopy***

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Abstract

The integrity of Zircaloy nuclear fuel cladding is critical to nuclear reactor safety as cladding provides the first barrier against the release of fission products from fuel into the reactor primary circuit. Iodine, a UO₂ fission product, accelerates SCC and Iodine-SCC is generally accepted as the cause of Pellet-Cladding Interaction (PCI) failures(1).

An advanced form of High-Speed Atomic force Microscopy (HS-AFM) developed at the University of Bristol operates at speeds orders of magnitudes faster than a conventional AFM, imaging multiple frames per second in air and liquid environments, allowing for the observation of dynamic events at the nanoscale. By using the HS-AFM to map surface morphology, the technique provides a useful tool to image corrosion initiation events and crack formation under stress as proven by similar experiments on carbide corrosion in steels using a custom-built liquid cell to image in-situ under corrosive liquids (2).

This project will demonstrate the extension of the in-situ HS-AFM technique for Zircaloy corrosion and Iodine-SCC. Zr-alloys have been imaged both in an unstressed state and under tensile stress in an iodine solution to induce cracking with in-situ HS-AFM observation of crack initiation. Real time imaging of corrosion and cracking in the HS-AFM allows a mechanistic understanding of Iodine-SCC initiation at the nanoscale to better understand the corrosion behaviour in Zr-alloys which can be used to help further the development of corrosion resistant fuel cladding.

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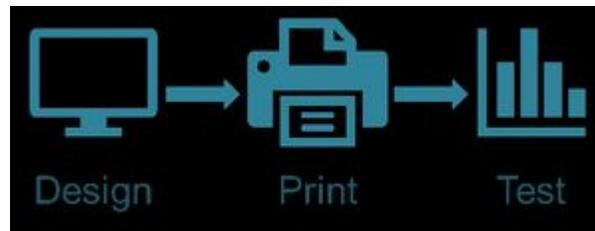
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Additively Manufactured Flow Resistive Pulse Sensors

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Abstract

Resistive pulse sensing (RPS) can provide a comprehensive characterisation of a range of analytes from nanoparticles to large cells, RPS gives information on the analytes size, shape and concentration based on the resistive pulse produced when the analyte translocates through a microscale pore. Here we use a type of additive manufacturing (AM) called Stereolithography (SLA) as a tool which can be used to rapidly fabricate a complete sensor without the need for traditional hard or soft lithographic processes, thus reducing the time and costs of producing these sensors. Herein we report an AM RPS sensor capable of detecting various sizes of microparticles at a range of concentrations across different flow rates in a variety of ionic buffers, lowest detected concentration was 10 particles per ml. The sensors we have produced can be disassembled, cleaned and reassembled several times without impacting performance. We believe this sensor has applications as an online or in-line sensor for droplet manufacturing, cell-line monitoring and environmental applications.



Ultra-fast Rechargeable Next-generation Batteries

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Abstract

Carbon-based materials play indispensable roles in energy-related systems, and constructing ultra-fast rechargeable carbon anodes is still one of the most interesting and meaningful topics in energy storage and conversion fields. Selection of an appropriate structure and composition of carbon materials can significantly accelerate the charging and discharging rates. Our group report several strategies to modify carbon materials to improve the charging rate. Specifically, we focus on introducing appropriate quantum dots and bridging suitable molecules into the carbon layer of graphite to expand layer spacing, and thus increased the migration rate for electrons and ions, shortening the charging time of batteries. We successfully built up Lithium-ion batteries which could be fully charged in 28.3 s with a capacity of 392.8 mA h g⁻¹ (the best ever reported, much faster charging and higher capacity than commercial Lithium-ion batteries), as well as Potassium-ion battery and Aluminium-ion battery with charging time of only 28.1 and 43.7 seconds, respectively.

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Corrosion study of magnesium alloys coated with chitosan coating in the Hank's solution for biomedical applications

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Abstract

In recent years magnesium and its alloys became one of most promising materials to be used in biomedical applications. Because of its strength, toughness, elasticity modulus is very similar to the human bones. Moreover, it takes part in different important living processes in nature at all, and in humans body in particular. Magnesium shows very good biocompatible and biodegradable properties but it the other hand its nonstable in the solutions containing the chloride ions. To improve corrosion resistance of magnesium it was applied natural biopolymeric chitosan coating.

In this work, the corrosion rate of Mg₁₉Zn₁Ca alloy in the Hank's solution was investigated. In order to study the corrosion degradation of Mg alloys the following electrochemical techniques were used: Linear Sweep Voltamperometry (LSV), Electrochemical Impedance Spectroscopy (EIS), chronoamperometry. The corrosion tests were performed in the Hank's solution at 37°C and pH = 7.2. The microstructure of alloys has been investigated by means of XRD and FE-SEM/EDS measurements. Surface analysis techniques like XPS and FT-IR were used to study the chemical composition corrosion products.

Magnesium alloys undergo the active corrosion in the Hank's solution. The chitosan based coatings deposited on the surface of Mg alloys significantly reduce their corrosion rate. Chitosan layer on the surface of Mg alloys produce corrosion protective film and also to shows antibacterial properties.

Acknowledgements

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P27

Effect of Hole-Blocking Layer of Lanthanum Tungstate on Chemical Potential Profile in Proton-Conducting BaZr_{0.8}Y_{0.2}O_{3-δ} Electrolyte

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Abstract

Proton-conducting solid oxide fuel cells (p-SOFCs) will be promising technology for critically-high electric efficiencies. However, the existence of the partial conductivities of hole and/or electron will cause the leakage current to deteriorate the electrical efficiency. Therefore, it is important to control the oxygen potential profile in the electrolyte because the profile should determine the degree of the leakage current. In this study, hole-blocking layers formed on the cathode -side of a BaZr_{0.8}Y_{0.2}O_{3-δ} (BZY82) electrolyte have been investigated for modifying the chemical potential profile leading to suppressing the leakage current. Proton conducting material of lanthanum tungsten oxide (La_{28-x}W_{4+x}O_{54+δ}; LWO) was used as the hole-blocking layer. The chemical potential profiles were estimated by using physicochemical parameters of the BZY82 and LWO and were found to have a large dependence on the existence of the blocking layer.

Effects of oxygen on degradation of carbon supported platinum catalysts

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Abstract

Improving the catalysts durability of PEM fuel cells is of significant importance to lower costs and achieve wide commercialization. Carbon supported Platinum (Pt/C) catalysts are widely used at both the anode and cathode. The cathode, which is at higher potential and in a highly oxidising environment, suffers more severe degradation than the anode. Therefore, it is significant to study the degradation process for cathode Pt/C catalysts, and many protocols exist^[1]. Here we extend these to include the effects of oxygen.

There are mainly three mechanisms for Pt/C degradation^[2]: 1) Pt dissolution and redeposition; 2) Pt loss caused by Pt dissolution; 3) carbon substrate corrosion, which can lead to Pt agglomeration. To study how oxygen affect these three degradation mechanisms, four different accelerated stress tests (ASTs) are applied: potential cycling between 0.6 to 1.0 V and potential cycling between 0.6 to 1.2 V in N₂ purged and O₂ purged HClO₄ electrolytes.

The degradation of the Pt/C catalyst is characterized by the loss of electrochemical surface area (ECSA) determined from hydrogen adsorption/desorption region and CO stripping. The loss of ORR activity is measured by using the thin-film rotating disk electrode (RDE) method. The particle size distribution before and after ASTs are determined by the transmission electron microscopy (TEM).

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Tissue response to neural implants: the use of model systems towards new design solutions of implantable microelectrodes

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Abstract

The development of implantable neuroelectrodes is advancing rapidly as these tools are becoming increasingly ubiquitous in clinical practice, especially for the treatment of traumatic and neurodegenerative disorders. Electrodes have been exploited in a wide number of neural interface devices, such as deep brain stimulation, which is one of the most successful therapies with proven efficacy in the treatment of diseases like Parkinson or epilepsy. However, one of the main caveats related to the clinical application of electrodes is the nervous tissue response at the injury site, characterized by a cascade of inflammatory events, which culminate in chronic inflammation, and, in turn, result in the failure of the implant over extended periods of time. To overcome current limitations of the most widespread macroelectrode based systems, new design strategies and the development of innovative materials with superior biocompatibility characteristics are currently being investigated.

This review describes the current state of the art of *in vitro*, *ex vivo* and *in vivo* models available for the study of neural tissue response to implantable microelectrodes. We particularly highlight new models with increased complexity that closely mimic *in vivo* scenarios and that can serve as promising alternatives to animal studies for investigation of microelectrodes in neural tissues. Additionally, we also express our view on the impact of the progress in the field of neural tissue engineering on neural implant research.

Molecular Metal Oxides for High Energy Battery Cathodes

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Abstract

Sustainable energy conversion is a global issue that can be addressed using electrochemical energy storage. Next generation energy technologies incorporating materials with the energy density of batteries and power density of supercapacitors is an exciting prospect waiting to be realised. The starting point is to research new electroactive materials for enhanced electrode fabrication. Attention is turned to polyoxometalates (POMs), which are discrete molecular metal oxide clusters possessing desirable properties as an energy material. These include their rich and reversible electrochemistry, high thermal and chemical stability, as well as their tuneable synthesis. Recent research has shown that POMs can be employed to fabricate hybrid electrode materials capable of multi-electron redox processes that may enable rapid Li⁺-ion transportation whilst maintaining stable cycling.¹ However, POMs are highly soluble in a variety of solvents and their electronic conductivity is negligible. Therefore, they must be integrated into a conductive host matrix to prevent dissolution of the redox active species and augment the electronic conductivity of the hybrid material.

Here we present a class of positive electrode materials, derived from the Wells-Dawson polyoxotungstate [P₂W₁₈O₆₂]⁶⁻ motifs, for use in electrochemical energy storage devices such as Li-ion batteries. Highly redox active plenary heteropolyanions were adsorbed onto a surface-modified, conductive carbon black. The electrode performance of the resultant composite materials was investigated and the capacitance and cycling stability were established.

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Understanding the Capacity Limitation of Na-O₂ Batteries

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Abstract

Sodium (Na) or lithium (Li) dendrite formation at the anode is considered as an inherent problem of safety in metal-air batteries. The side reactions of the metallic anode with the liquid electrolyte or dendrite formation take place at the anode surface. Oxygen (O₂) may also diffuse to the anode and partly passivate the metallic surface that increases the cell impedance. The idea of using metal anode was to maximise the cell voltage and energy density compared to the counterpart metal-ion technology but currently seemed to be challenging to achieve it. It is debateable now whether the alkali metal can be used as anode or not? Therefore, most research focuses on designing a robust solid-electrolyte interphase (SEI) to protect the metallic anode against parasitic reactions with the electrolyte and dendritic deposition during cell recharge. Engineering an appropriate SEI allows a smooth deposition by lowering the activation energy barrier of the metal ion diffusion at the interphase. Here, we report how Na anode, cathode and electrolyte limit the performance of Na-O₂ batteries? The capacity limitation of the Na-O₂ batteries not only relates to the air blockage of the cathode by the discharge products but also to the metallic anode that loses its ion releasing capacity during the discharge. This study will also examine the air-cathode performance of non-precious metal oxide catalysts based on spinel-type nickel-cobalt oxide nanofibers fabricated by electrochemical method.

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Development of an in vivo sensor for therapeutic monitoring of tricyclic antidepressants

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Abstract

The ability to measure the concentration of therapeutic compounds *in vivo* can provide in-depth pharmacokinetic information while avoiding the need for recurring and difficult sampling procedures. Through a combination of electrochemistry and polymer chemistry some of the challenges which have previously limited *in vivo* sensing will be overcome to develop a sensor capable of detecting and monitoring the systemic concentration of antipsychotics.

Firstly, new polymer blends will be identified using a novel high-throughput screening approach, in particular electrochemical-atomic force microscopy (EC-AFM) will be used to assess polymers based on their conductive properties. Thus far the ability to produce thick and thin films from the conductive polymer poly(3,4-ethylenedioxythiophene) (PEDOT), through the addition of cross-linking agents and conductivity enhancers, has been investigated. These films have been interrogated using cyclic voltammetry and impedance spectroscopy to determine if they remain sufficiently electroactive to be used as a potential sensing platform.

Secondly, therapeutic analytes have been studied to build an understanding of their redox behaviour, specifically the kinetics of the tricyclic antidepressants (TCA) has been studied. Early results indicate the ability to measure at least two of the TCA family in simple solutions, however resolution in complex media requires further optimisation. If successful this project would act as a proof-of-concept for a novel sensor platform which would provide a step forward for translational research and contribute towards the growing fields of both bioelectronics and personalised medicine.

Effect of additives on electroforming of nickel

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Abstract

A range of studies have been performed to understand the effect of additives in electrodeposition. Indeed, these additives have been proven to affect deposit properties such as hardness and internal stress. Despite advances in electroplating research, very few studies have investigated the effect of addition agents in electroforming. Thus, these agents are still used in an empirical way.

Knowledge of the strengths and weaknesses of additives under different electrodeposition conditions will allow the bath chemistry of electroforming solutions to be better understood in terms of their suitability for different applications. It is therefore aimed to establish an optimum temperature and chemistry for using additives in nickel electroforming.

In preliminary studies, the effect of nickel chloride and boric acid in a nickel sulfamate bath on the electrochemical reduction of nickel has been systematically investigated with the use of cyclic voltammetry obtained on a gold electrode at room temperature. The voltammograms were recorded after obtaining reproducible traces on repeated scanning for 5 cycles.

Preliminary results convey that the addition of H_3BO_3 may have a catalytic effect, indicated by a shift in the onset potential for Ni^{2+} and H^+ reduction to more positive potentials. Conversely, NiCl_2 appeared to have a negligible effect on the onset potential for Ni^{2+} reduction. A follow-up combinatorial study of these additives performed at higher temperatures using linear sweep voltammetric, potentiostatic deposition and gravimetric techniques will be presented to reveal more information about the true effect of additives on nickel deposition in a sulfamate bath under different conditions.

Pitting corrosion on coated stainless steel PEMFC flow plates

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Abstract

The bipolar plate(BPP) constitutes up to 28% of the PEMFC stack cost[1]. Cheaper and more lightweight materials are needed, while there are strict requirements on both the mechanical and chemical stability within the acidic environment of the fuel cell. The targets set by the US DOE are a corrosion current $<1 \mu\text{A}/\text{cm}^2$ and interfacial contact resistance $<0.01 \text{ ohm cm}^2$ [2].

Stainless steel is affordable and has the mechanical stability required for the BPPs. However, SS is subject to corrosion in the PEMFC environment. To be able to reach the DOE goals, either noble metal or conductive ceramic coatings must be utilised[3]. In this work, commercially available coatings on hydroformed SS 316L flow plates are studied. A single cell fuel cell tester is used to age the samples, and the in-situ degradation is measured by impedance measurements and polarisation curves. The electrochemical micro-cell technique is utilised to study the corrosion on both the pristine and aged flow plates by polarisation. SEM is used to analyse the surface. The aim is to better understand the pitting corrosion on PEMFC flow plates.

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Capacitive Deionisation for Phosphate Recovery

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Abstract

Phosphate run-off originating from agricultural sources has negative effects on crop yield and is often a contributor to eutrophication. Moreover, natural supplies of phosphate rich rocks-phosphorites- will eventually run out. Current methods for the recovery and recycling of phosphates rely heavily on chemical precipitation e.g. struvite formation ($\text{MgNH}_4\text{PO}_4 \cdot 6\text{H}_2\text{O}$) and biological remediation e.g. EBPR (Enhanced Biological Phosphorus Removal). Electrochemical methods such as reverse osmosis and electrodialysis are primarily used for the desalination of brackish water. These techniques are highly energy intensive. Capacitive Deionisation (CDI) is one such electrochemical technique, that has a lower energy requirement, but its use on the industrial scale has yet to be realised. The research herein aims to successfully combine the recovery of phosphate from brackish water using a CDI cell. The 2,6-bis[(bis(2-pyridylmethyl)amino)-methyl]-4-methylphenol (H-bpmp) ligand has been shown to be phosphate specific, even in the presence of other anions. Following the successful synthesis and coordination of the ligand to Zn^{2+} centres, electrografting of a nitrobenzene spacer molecule, which itself is further electrochemically reduced, the functionalised ligand can be attached. The CDI system can then be tested for its suitability as a novel method for phosphate recovery.

A selectivity switch in the alternating potential electrolysis of 4-methylanisole

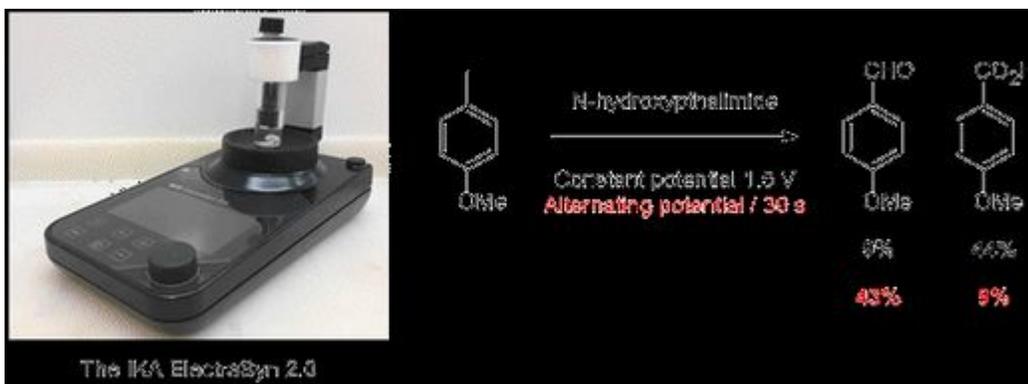
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Abstract

Synthetic organic electrochemistry has experienced a resurgence in recent years. It enables access to unique reactivity using methodology that is more environmentally benign than traditional chemical synthesis. However, the widespread adoption of electrochemistry has been slowed by the requirement for specialist equipment, as well as concerns over reproducibility. The IKA ElectraSyn 2.0 is a standardised electrochemistry set-up that has been developed to combat these issues by providing a more accessible alternative to a potentiostat and external cell for organic electrosynthesis.

The IKA ElectraSyn 2.0 has a function that periodically alternates the potential. This is generally used to prevent fouling and passivation of the electrodes during electrolysis. Whilst investigating the electrochemical oxidation of benzylic methyl groups, the use of this function inverted the selectivity for aldehyde and carboxylic acid products. Further study revealed an unexpected mode of action that leads to an uncontrolled increase in anodic potential. The mode of action for the alternating potential function is described and the mechanism by which it causes the observed change in selectivity is proposed. Because one of the principle advantages of electrochemistry is the ability to precisely control the oxidative or reductive potential of a transformation, this study holds important information for all users of the IKA ElectraSyn 2.0.



***Redox-driven Nano-confinement, Wiring, and Protection of
Polyoxometalates in Carbon Nanotubes***

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Abstract

Polyoxometalates (POMs) have received considerable attention in recent years due to their rich redox properties and potential applications in energy storage. The discrete nature of POMs means that their use as components in energy storage devices relies on their stable combination with conductive supports. Carbon nanotubes (CNTs) are stable, hollow cylinders made entirely of carbon. These nanostructured carbons are highly conductive, mechanically strong and can be functionalised. By encapsulating material within CNTs, their electronic properties and stability can be enhanced.

This work describes the first report of the encapsulation of the Keggin $[PW_{12}O_{40}]^{3-}$ and Wells-Dawson $[P_2W_{18}O_{62}]^{6-}$ heteropoly anions within carbon nanotubes along with detailed structural, chemical and electronic characterisation. Access to POM redox states are observed due to the fast and efficient electron transfer *via* the CNTs. The resultant materials are highly stable and are able to withstand multiple charge-discharge cycles, as well as withstanding conditions normally too extreme for POMs.

Electrochemical reduction of CO₂ to formate using a modified SnO₂ with Sb and Zn

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Abstract

The design of efficient and selective electrocatalyst remains one of the biggest challenges in eCO₂R process[1]. In this study for the first time, we report the eCO₂R to formate (HCOO⁻) using an Antimony (Sb) and Zinc (Zn) modified Tin Oxide (SnO₂) electrocatalysts. Modification of the electrodes was carried out in concentrations of 1 wt%, 5 wt% and 10 wt% for both Sb and Zn to compare with the eCO₂R performance of a pure SnO₂ electrocatalyst. Cyclic voltammetry (CV) analysis shows a larger eCO₂R current under a CO₂ atmosphere for both Sb-SnO₂ and Zn-SnO₂ when compared to pure SnO₂. The improved eCO₂R current is attributed to the enhanced conductivity of the modified electrocatalysts which was confirmed using electrochemical impedance spectroscopy (EIS). EIS demonstrated that Sb-SnO₂ and Zn-SnO₂ electrocatalysts improve the electronic conductivity when compared to pure SnO₂. The SnO₂ catalyst doped with 5% Sb (5-Sb-SnO₂) exhibited the highest Faradaic Efficiency (FE_{HCOO^-}) of 70% and partial current density (j_{HCOO^-}) of 14.3 mA cm⁻² at -1.3 V vs. RHE, which was 9% and 4.5 mA cm⁻² higher than the pure SnO₂ respectively. Modifying SnO₂ with small quantities (< 5%) of Sb at high overpotentials was found to be beneficial for eCO₂R activity, with increased HCOO⁻ production observed compared to unmodified SnO₂. Interestingly, Modifying SnO₂ with Zn on the other hand significantly improves the eCO₂R performance at low overpotentials, with FE found to be 2.5 times greater than pure SnO₂.

Synthesis of ZnO-NRs/CdS/AuNPs nanocomposite and its potential application in to the fabrication of an electrochemical aptasensor.

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Abstract

The authors have synthesized ZnO nanorods (NRs) closely anchored to CdS sensitized with Au nanoparticles (NPs). It is expected that the ZnO-NRs/CdS/AuNPs nanocomposite can serve as a photoactive material for use in electrochemical and photoelectrochemical detection by absorbing light efficiently and then promoting electron transfer.

ZnO-NRs were electrodeposited, varying the conditions of the synthesis process; potential, temperature, time and concentration of precursors. The above, with the objective of obtaining highly ordered nanostructures. These nanostructures were characterized by cyclic voltammetry (CV), UV-visible spectroscopy (UV-Vis), scanning electron microscopy (SEM) and X-ray diffraction (XRD), to be later sensitized with CdS formed in situ chemically on ZnO, the above, with the aim of forming a stable nanocomposite. To obtain ordered ZnO-NRs/CdS nanocomposite, the experimental conditions were varied: time, precursor concentration and temperature. This nanocomposite was characterized using CV, UV-Vis, SEM and XRD.

Finally, the ZnO-NRs/CdS nanocomposite was modified with AuNPs by drop-casting. AuNPs were obtained by chemical reduction of Tetrachloroauric Acid (HAuCl₄) with Sodium Citrate (Na₃C₆H₅O₇), obtaining a suspension that was stored at 4 ° C. This nanocomposite was characterized using CV and UV-Vis.

Promoting the Remineralisation of Subsurface Caries Lesions

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Abstract

The World Health Organisation have reported that dental caries – i.e. cavities – are a major health problem amongst the majority of adults and school children. ¹ Later stage dental caries tend to be treated through restorative means - e.g. composite resins – but are usually invasive and the effectiveness of these fillings are temporary. ^{2,3}

A new technology under development by Reminova Ltd sets out to electrochemically promote remineralisation of subsurface caries lesions. As this technique is still in its early stages of development, the aims of this research are to characterise how the nucleation and growth of calcium phosphate crystals is promoted. To achieve this, we are studying calcium phosphate crystallisation inside track-etch membranes placed inside a double-diffusion set-up. We are studying how the applied potential promotes the migration of calcium and phosphate ions and creates a local supersaturation to enable crystallisation.

This poster will present preliminary results, from *in situ* monitoring via EIS, to characterise the crystallisation process resulting from the effects of a potential. The increased rate of crystallisation hints to faster migration of ions through the membrane pores, which is consistent with subsequent SEM analysis showing the membrane pores and surfaces being filled with calcium phosphate crystals.

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Study of the electrolytic influence in the electrochemical synthesis and nanostructures formation of MoS₂: effects on the optimization of the electrocatalytic performance in the hydrogen evolution reaction (HER)

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Abstract

MoS₂ is currently known to possess promising catalytic properties for the evolution of the hydrogen reaction (HER). Although the last time its synthesis has been widely studied by chemical methods, such as hydrothermal, exfoliation by intercalation of ions, or chemical vapor deposition (CVD), the electrochemical synthesis has been less studied to date, and there is still much to be improved in terms of obtaining nanostructures and the performance for the hydrogen evolution reaction [2-3]. In the present work a systematic study of the electrodeposition of MoS₂ on FTO is presented from a precursor solution of (NH₄)₂MoS₄. The study variables for the electrodeposition process were precursor concentration (5, 2, 1, 0,5 mM), electrolyte type (KCl, K₂SO₄), electrolyte concentration (0.5, 0.1, 0.05 M), and electrochemical technique (cyclic voltammetry, and chronoamperometry). It has been shown that both the electrolyte type and the concentration of species are relevant parameters for the improvement of the catalytic performance. On the other hand, there is evidence that these changes in the electrodeposition method - with respect to the published to the date- improve the size of structures formed, allowing to reach easily a nanoscale deposit.

The electrocatalytic properties toward the hydrogen evolution were studied by linear sweep voltammetry (LSV) in the medium of 0.5 M H₂SO₄. The obtained samples were morphologically characterized by scanning electron microscopy (SEM), and compositionally by X-ray diffraction (XRD). In addition, an electrochemical characterization was performed through electrochemical impedance spectroscopy (EIS), indicating the presence of Volmer-Heyrovsky mechanism.

Development of electrochemiluminescence (ECL) sensor modified with nafion film for detection of methamphetamine and its main metabolites

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Abstract

In forensic drug analysis, the need for pretreatment in detection of amphetamine type stimulants (ATS) such as methamphetamine (MA) and amphetamine is one of the main drawbacks within the detection protocol. Electrochemical techniques such as electrochemiluminescence (ECL) enable to directly detect the drugs in street samples or biological matrices. In this context, $[\text{Ru}(\text{bpy})_3]^{2+}$ has very frequent use for detection of individual ATSs. Also, nafion composite film are generally combined with $[\text{Ru}(\text{bpy})_3]^{2+}$ to have more effective, economically and cheaper production costs. There is no need for surface modification or chain linkers in composite films and this is an advantage. Using nafion film is based on direct interaction with electrode surface. Under optimised conditions, the response of ECL linearly increased with the concentration of MA with a correlation coefficient of 0.9918. LoD for MA was observed as 74 ng/mL. Also, dynamic range was obtained for amphetamine with a correlation coefficient of 0.9934. This technique is simple, rapid, selective and sensitive and can be used for detection of MA and its metabolites in body fluids like saliva or urine.

P43

Electroforming of Large-Scale Nickel Structures for Leading-Edge Energy, Aerospace and Marine Applications

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Abstract

Electroforming is an **electrochemical additive manufacturing process**. Some of the strengths of this process include the high energy and resource efficiency coupled with low power consumption. Specifically, nickel electroforming is widely applied in aviation and space industries for the manufacturing of precision, lightweight parts, such as erosion shields. However, as current techniques rely on traditional chemistry and processing methods, utilisation of nickel electroforming remains a specialist activity.

Electroforming's boundaries can be pushed further through leading-edge electrochemical engineering. For example, an in-depth investigation of electrolyte chemistry can help to achieve desirable deposit properties, whilst raising metal deposition rates. Deeper investigation on electrolyte agitation and reactor analysis can provide information on process parameters that affect the properties of final electroformed products.

Our team aspires to bring the electroforming process to the forefront of additive manufacturing by opening new horizons for precision manufacturing and overcoming limitations which have rendered specific products "impossible to manufacture" up to this day. The first step to do so is the systematic electrochemical analysis and characterisation of the system. Electrochemical analysis techniques include linear sweep voltammetry (LSV), cyclic voltammetry (CV) and electrochemical impedance spectroscopy (EIS). Galvanostatically controlled deposition experiments are carried out and both the total process and working electrode current and potential are measured. The data is used for LSV data correction. Mass balance calculations determine the experiments' efficiency. Experimental data is gathered at 50 °C, under agitation. All lab results are tested for their reproducibility and compared to the industrial data.

Keywords: Electroforming; Manufacturing; Nickel

Use of Mediators for the reduction of Nitroarenes

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Abstract

Direct reduction of nitrobenzene to aniline leads to a mixture of products which has been previously proved.¹ The utilization of mediators for the reduction of nitroarenes is not a new research area. MacDonald et al. used polyoxometalate (POM) mediators to effectively reduce a variety of nitroarenes in stoichiometric amounts where the POM proved to be recyclable.² Herein, applying the same principals and using organic mediators and/or another POMs we aim to effectively reduce nitrobenzene for the first time using the mediator in only catalytic amounts. We screen potential mediators using cyclic voltammetry and assess the performance of promising candidates using bulk electrolysis.

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P45

Development of an autonomous, in situ cleaning, electrochemical sensor for dissolved inorganic carbon detection at full ocean depth

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Abstract

Dissolved inorganic carbon (DIC) is a key ocean chemistry parameter in climate change, and for climate change mitigation strategies. As part of a proposed complete seawater carbonate chemistry monitoring system, DIC sensors will examine ocean acidification in fishing and in ecologically sensitive areas. They will also be used to monitor sub-sea carbon storage for potential leaks of CO₂ during and after gas injection. Bespoke electrochemical cells and miniaturised potentiostats will be integrated into the Lab-on-Chip microfluidics platform, developed at the National Oceanography Centre, to create a sensor that is autonomous, and operational at ocean depths of 6 km.

We have designed and tested microelectrode sensors to measure changes in conductivity, a highly efficient gas exchange membrane, developed *in situ* cleaning methods, and custom electronics to run a suite of electrochemical techniques. Testing for several of these components has taken both in the lab, on autonomous underwater vehicles, and on the floor of the North Sea, monitoring leaks from CO₂ injections for sub-sea storage. Initial results from prototype sensors are very encouraging, with further field tests taking place in October.

3D and 4D Characterisation of Dilated Lithium Ion Battery Electrode

Microstructure using *in-Situ* Dilatometry and X-ray Tomography

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Abstract

Until now, there have been no reviews of *in-situ* dilatometry investigations combined with X-ray computed tomography (CT) to evaluate the evolution of electrode degradation in lithium ion batteries (LiBs). Progress in dilatometric investigations of electrode expansion can aid in understanding structural changes as they undergo expansion and shrinking. A greater understanding of these electrode dynamics are required to overcome limitations in current density, battery lifetime and capacity fading.

Electrochemical *in-situ* dilatometry enables analysis of intercalation induced macroscopic expansion of electrodes for LiBs using controlled pressure, conventional electrodes, electrolytes and separators. The technique is capable of showing alterations in irreversible dilation depending on electrode design and thus is a useful technique for assessing battery stability and lifetime.

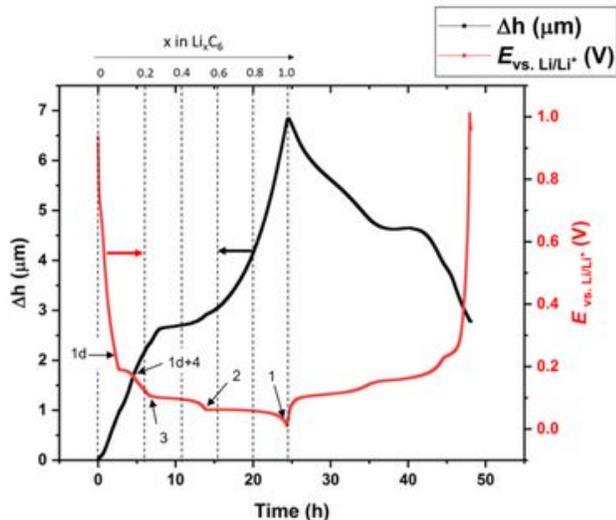


Figure 1: Potential profile and simultaneous dilatation record of graphite (NEI Corporation) during first charge/discharge.

A collaboration of *in-situ* dilatometric investigations with X-ray CT would provide insight into the field of battery degradation because a measure of the extent of dilation during battery operation can also be visually monitored using X-ray CT. This work focuses on conventional electrode materials such as silicon, graphite and NMC-811. Comparisons between calendered and uncalendered electrodes will be examined and the extent of their dilation will be imaged using X-ray CT.

P47

Investigation of Water Absorption Profile of Mineral Wool Insulation

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Abstract

This study is targeted at investigating the water absorption capacity of mineral wool insulation in order to understand its behaviour and contribution towards the occurrence of corrosion under insulation (CUI). Corrosion of insulated process facilities is either triggered by the penetration of water from the immediate external environment or condensation of water within the internal domains of insulated assets. The degree of penetration of water from the external environment to the surface of insulated process facilities reflects the performance of the insulation material.

Therefore, it is essential to investigate the water absorption profile of mineral wool insulation under different conditions such as change in pH and temperature in order to ascertain its performance when insulated on a substrate. In this study, this was conducted according to the ASTM C1511 standard.

Preliminary results revealed a rapid increase in water absorption up to 40 % within the first fifteen minutes indicating susceptibility to the absorption of water, which increases with pH. However, this absorption reduced significantly when the material was subjected to varying temperatures from 50 - 250°C reflecting its behaviour when insulated on a hot substrate.

Also, the presence of aluminium foil as a barrier in mineral wool was observed to significantly reduce the overall water absorption capacity indicating additional protection. However, should water penetrate the insulation, it will take a longer time for it to dry out when the foil is in place compared to the absence of foil.

Fabrication of a new composite polymer electrolyte membrane by ultrasonic spray casting

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University College London, London, United Kingdom

Abstract

The polymer electrolyte membrane (PEM) is an integral component of a fuel cell, selectively conducting protons from anode to cathode, driving electrochemical reactions that convert chemical to electrical energy. Efficient fuel cell operation requires the PEM to have high proton conductivity, electronic resistance, and durability, but minimal fuel crossover.

The incorporation of graphene oxide into polymer electrolyte membranes, has been shown to improve water retention and proton conduction, whilst blocking fuel crossover, due to its large surface area and hydrophilic functional groups. Additionally, selective permeation of protons through the electron clouds of pristine 2D crystals, such as graphene, has been shown to drastically mitigate fuel crossover in sandwich membranes.

Polytriazine imide is a 2D material constructed from triazine rings, forming graphitic sheets containing regular $C_{12}N_{12}$ structural voids. When incorporated into a PEM these voids can allow selective proton permeation, limiting fuel crossover, whilst the NH groups facilitate Grotthuss proton hopping. However, composite PEMs often have the drawback of agglomeration at high loading, due to the standard solution casting procedure. The use of ultrasonic spraying of a hybrid polymer mix can avert this issue due to the evaporation of solvent as it is cast, allowing the beneficial properties of the additive to be fully exploited. In addition, spray casting aids the fabrication of sandwich membranes.

Here we demonstrate the process of producing two multicomponent PEMs, and present initial results of their performance using a variety of techniques including; proton conductivity measurements, atomic force microscopy and single cell tests.

P49

Developing a Glucose Sensing System using Nanoband Electrodes

Fiona Moore¹, Camelia Dunare¹, Ilka Schmueser², Neville Freeman³, Andrew R. Mount², Jonathan G. Terry¹

¹School of Engineering, The University of Edinburgh, Edinburgh, United Kingdom. ²School of Chemistry, The University of Edinburgh, Edinburgh, United Kingdom. ³NanoFlex Limited, Daresbury, United Kingdom

Abstract

Treating diabetes costs the UK approximately £14 billion per year and a major proportion of this is from complications, including cardiovascular disease and renal damage. The health outcomes and severity of complications are considerably worse if the disease is poorly managed. Monitoring is primarily performed by measuring glucose concentration in blood via finger-prick tests which prevents continuous monitoring and can have low patient compliance. Monitoring glucose levels non-invasively and continuously would be hugely beneficial to patients' health outcomes.

Sweat is a potential alternative biological fluid to blood, which is easily accessible and has already shown its potential for detecting a range of analytes. Glucose concentrations in sweat are lower than in blood and sweat volume is limited, therefore sensitive measuring techniques are important to provide meaningful results. Electrochemical measurements using on-chip nanoelectrode arrays provide many applicable benefits including higher sensitivities, lower limits of detection and less analyte depletion.

One challenge in developing on-chip electrochemical systems is the need for a miniaturised solid-state reference electrode. These are limited by fabrication issues, which include limited layer thickness and lack of a defined filling solution. As a result, the stability and lifetime of such electrodes can be significantly affected, which represents a major obstacle in device development.

This poster will detail the development of on-chip platinum nanoelectrodes and miniaturised silver/silver chloride reference electrodes, which will enable measurement of low levels of glucose in sweat. The potential of these electrochemical systems in smart monitoring systems, including wearable continuous monitoring devices, will be discussed.

P50

Virtual Unrolling as a predictive analysis on the mechanism and location of degradation of cylindrical batteries

Anmol Jnawali, Matt Kok, Thomas Heenan, Julia Weaving, Francesco Iacoviello, Dan Brett, Paul Shearing
University College London, London, United Kingdom

Abstract

Virtual unrolling is a correlative image analysis technique that identifies the electrode rolls in the X-ray computed tomography (CT) images of batteries and translates them into a “2-D” orientation (Fig. 1) for easier comparisons of the changes in the physical conditions of cells. With enough data, a predictive element is added to the analysis – which would be useful in industry for quality assurance purposes. Preliminary results have demonstrated this predictive aspect of the analysis, whereby an imperfection in the jelly rolls of pristine cells were identified and development of deformations along those imperfections with increasing number of electrochemical cycles were observed. Preliminary hypotheses for the location of deformation have been attributed to the formation of imperfections during rolling of the jelly roll, the location of current collecting tabs, and the design of the cell – e.g. tapering of electrodes, or lack thereof. More data on cylindrical cells will be obtained to test the reliability of the predictive facet of the analysis, such as repeats, investigation of the effect of cycling at elevated temperatures, and effect of number of internal current collecting tabs. Furthermore, the application of correlative virtual unrolling with X-ray CT for pin-pointing a specific location on the physical jelly roll for easier region-of-interest (ROI) data acquisition will be investigated.

Titania Anchored Graphene Oxide Nanocomposites as Bifunctional Agents for Solar Disinfection (SODIS)

Anam Safri^{1,2}, Saima Shabbir², Ashleigh Fletcher¹

¹university of Strathclyde, glasgow, United Kingdom. ²institute of Space Technology, islamabad, Pakistan

Abstract

Synergistic water remediation effect of Titania (TiO₂) with Graphene oxide (GO) nanoparticles was investigated by synthesizing nanocomposites with different weight percentages of TiO₂ and GO in the triblock copolymer matrix through sol-gel process for their photocatalytic bactericidal degradation of *E. coli* and total fecal coliform. FESEM images demonstrated an exclusive approach of chemically exfoliating and functionalizing GO sheets before integrating with TiO₂ and polymer matrix along with observation of consequent change in the morphologies. Moreover, FTIR confirmed the chemical linkages between TiO₂, polymer matrix and GO in the obtained polymer nanocomposites films. Light absorbance of TiO₂ and GO polymer nanocomposites showed enhanced photochemical activity due to reduced band gap as and the corresponding photocatalytic inactivation of *E. coli* and total fecal coliform has been confirmed through antimicrobial activity. Results showed an improved photocatalytic bacterial inactivation with an increase in weight percent of TiO₂ but, to a certain extent. Thus, we propose an optimal weight percentage of TiO₂ and GO for complete photocatalytic inactivation of bacteria. Overall, energy efficient antimicrobial water treatment was validated without the production of disinfection byproducts along with improved optical and thermal profiles of the polymer nanocomposites.

Electrochemistry of Copper/Cysteine/Graphene Composite

Nikoloz Nioradze¹, Tinatin Dolidze², Mikheil Shushanian², Dimitri Khoshtariya^{2,3}

¹Ivane Javakhishvili Tbilisi State University, R. Agladze Institute of Inorganic Chemistry and Electrochemistry, Tbilisi, Georgia. ²Iv. Beritashvili Center of Experimental Biomedicine, Tbilisi, Georgia. ³Ivane Javakhishvili Tbilisi State University, Department of Life Science, Tbilisi, Georgia

Abstract

Since the structural and catalytic activity of copper ions bound to copper enzymes or proteins play important role in normal physiological processes and malfunctioning of the copper containing biological systems can induce the abnormal effects and pathologies, study of artificial biological nanocomposites with redox-active copper proteins mimicking the real biological systems represents the fundamental interest.

Self-assembled monolayers (SAMs) of various cysteines (L-cysteine, D-cysteine, DL-homocysteine) were used to bridge copper ions to graphene electrode via incubating of SAM/graphene composite in various concentrations of copper chloride solutions. Electron transfer reactions involving immobilized copper on SAM/graphene composites were studied systematically by electrochemical methods such as cyclic voltammetry. Cu (II) /Cu (I) ions as redox-active species presumably entrapped directly in COOH-terminated self-assembled monolayer of different cysteines showed clear electrochemical signal in the phosphate buffer solution at pH 6.2. The electrochemical signal was obtainable from copper/SAM/graphene samples prepared by deposition of copper on SAM/graphene composite from the solution of copper chloride with concentration as low as 10 nM. No significant change in electrochemistry was observed after change of the SAM chain length by changing L-cysteine by DL-homocysteine.

Apart from the fundamental interest, copper ion containing artificial biodevices could be interesting to study as effective copper ion sensors/catalysts. One more aspect of this work could be exploitation of carbon nanomaterials as a supporting substrates for self-assembled monolayers.

Acknowledgement:

Support from Shota Rustaveli National Science Foundation of Georgia, Grant YS17_32 is gratefully appreciated.

Fibre-Optic pH Sensor Based on Silver Nanoparticles for Harsh Environments

Shaon Debnath, Sudipta Roy, Todd Green
University of Strathclyde, Glasgow, United Kingdom

Abstract

The aim of the fabrication and characterization of a surface plasmon resonance (SPR) pH sensor using coatings of silver nanoparticles on optical fibre which can function in harsh environments. The objective is to determine the optimum conditions for the synthesis of silver nanoparticles which are stable and decorate them on an optical fibre to form a pH sensor. This will unveil the sensitivity of the pH sensor with regards to the minimum particle size of silver nanoparticle.

Silver nanostructured films fabricated by sol-gel techniques has been observed to exhibit a strong localized surface plasmon resonance (LSPR) at a wavelength around 400 nm [1-3]. The spectral position of LSPR is sensitive to factors such as pH and the size of the nanoparticles.

Although the synthesis of silver nanoparticles and characterization using various analytical techniques have been widely studied, the sensitivity of coating with respect to the change in pH of the solution is still required to advance the current state of knowledge. In this study, the coating of the silver nanoparticles will be prepared by chemical reduction involving a different concentrations of silver nitrate, reaction time and forming multiple layers by dip coating method. The absorbance and light scattering behaviour of silver nanoparticles will be characterized using UV-visible spectrophotometer and the particle size will be characterised by using SEM . The expected result will be used to determine the size range of the nanoparticles and the stirring time that can provide stable particles which exhibit SPS.

***In-Operando XRD for Understanding the Degradation Mechanisms of
Lithium Ion Battery Materials***

Alice Llewellyn, Paul Shearing
UCL, London, United Kingdom

Abstract

Electrochemical energy storage devices with high capacities, long cycle life and excellent safety characteristics are being intensely researched. It is therefore imperative to examine and understand both the physical and chemical changes that battery materials undergo upon cycling. The electrode materials used for an electrochemical cell greatly determine the overall performance of the battery.

During lithiation/delithiation the electrode material undergoes changes such as phase transitions and structural degradation. XRD is a crucial analytical tool for identifying changes to the crystal structure by monitoring the lattice parameters. These changes are closely related to the electrochemical performance of a cell and using *in-operando* powder diffraction to observe them could lead to explanations for electrochemical phenomena such as capacity fading.

In this project, the electrochemical, morphological and crystallographic transformations of electrode materials (mostly layered transition metal oxides, whose structural changes can be monitored by observing the changes in the (003) and (110) peaks) will be studied using *in-operando* XRD and various complementary techniques in order to connect degradation processes to their electrochemical signatures.

Advanced Autoclaves for High Temperature High Pressure Corrosion Assessment

Robert Jacklin¹, Richard Barker¹, Anne Neville¹, Yong Hua¹, Danny Burkle²

¹University of Leeds, Leeds, United Kingdom. ²LBBC Baskerville, Leeds, United Kingdom

Abstract

In the energy industry, corrosion in extreme conditions is increasingly prevalent. Access to deeper wells for hydrocarbon recovery and geothermal energy means that down-hole conditions are becoming progressively more hostile. The increasing use of high-pressure injection techniques in mature wells for Enhanced Oil Recovery (EOR) and CO₂ sequestration is also exerting greater demand on existing infrastructure such as production tubing.

Single autoclave systems present a cost-effective means to simulate high pressure and temperature environments. However, two notable disadvantages exist which cast ambiguity over the translation of laboratory results to the field. The first is the variation of solution chemistry which occurs within a closed autoclave system over time. The second is the exposure of test specimens to non-representative conditions during the heat-up period at the start, and cool down at the end of experiments. This study aims to determine the significance of these experimental limitations on the corrosion products that form when simulating CO₂ corrosion of carbon steel exposed to geothermal brines.

An initial investigation on sample-area-to-solution-volume (a/v) ratio is presented here. X65 carbon steel samples are exposed to a CO₂-H₂O-NaCl solution containing 0-5000 ppm of Ca²⁺. Tests are performed at 80°C in a standard autoclave for up to 96 hours. The corrosion rate is quantified using mass-loss measurements and corrosion products are analysed using spectroscopic and microscopic techniques. As future work, the experiments will be repeated in an autoclave modified for rapid sample insertion and retraction to evaluate the effects of heat-up and cool down times.

***Electrodeposited Intermetallic Tin-Antimony as a Novel Electrocatalyst
Material for Carbon Dioxide Reduction to Formate***

Francisco Willian S. Lucas, Fabio H. B. Lima

Institute of Chemistry of São Carlos, University of de São Paulo, São Carlos, Brazil

Abstract

It is well known that the increase in atmospheric CO₂ level is a phenomenon associated with the Greenhouse effect and climate changes. This fact has driven a large number of investments in research and development of systems for CO₂ harnessing. In this scenario, the electrochemical reduction reaction of CO₂ (ECO₂RR) to value-added chemicals has been regarded as a promising strategy. The ECO₂RR can generate different products, and formic acid is a key product amongst these; it is an important chemical reactant for the chemical industries, it is an excellent hydrogen carrier (capacity of 53 g H₂/L), and it can also be used as anode fuel in the direct formic acid fuel cell. The most widely studied electrocatalysts for selective formic acid production are based on “post-transition metals” and alloy; thereby, there is still a large gap in the literature on studies of low-cost and environmental friendly Sn-alloys. In this study, for the first time, it is presented the activity of the intermetallic SnSb as a new Sn-alloy for CO₂ electrochemical reduction to formate. This new catalyst was grown by electrodeposition and has crystalline structure and morphology completely different than its precursors (metallic Sn and Sb); however, it showed improved stability and a compatible faradaic efficiency for formate production compared to the pure Sn. The effects of the composition of the film, the potential of electrolysis, and the nature and concentration of the supporting electrolyte on the activity and long-term stability of the electrocatalyst were also evaluated.

Work supported by FAPESP(#2018/03156-5).

Cu-based Catalysts for Electrochemical Reduction of CO₂ using Graphene aerogel Gas Diffusion Electrode Type Device

Yucheng Wang¹, Hang Xiang², Eileen Yu², Yongqing Fu¹, Terence Xiaoteng Liu¹

¹Faculty of Engineering and Environment, Northumbria Univeristy, Newcastle upon Tyne, United Kingdom. ²School of Engineering, Newcastle University, Newcastle upon Tyne, United Kingdom

Abstract

Electrochemical reduction of CO₂ to low carbon organic compounds has been considered as a promising method to mitigate the greenhouse effect and produce useful energy carrying chemicals. However, the development of catalyst with high activity, selectivity, and good stability is still the bottleneck to accomplish this goal. Cu based catalysts have been reported to meet such requirement. In this work, Cu particles with nano-flower shaped oxide derived Cu/Graphene has been prepared, in order to increase the properties, and then the synthesized catalysts were applied in PEM cell with gas-diffusion-electrode (GDE) for electrochemical test. Graphene-aerogel based gas diffusion layer for electrode assembly were also involved into this project to improve the mass transfer properties. Results indicate that Cu derived catalysts exhibits a catalytic activity for CO₂ conversion to CO and formic acid with a Faradic efficiency of 80% under -1.0V vs. SHE. Online electrochemical monitoring is one of the interesting areas in this filed. In this case, we developed the optical fibre sensor for Gas/Liquid products detection.



Fig. 1 a) CuO nano needle b) CuO/graphene nano-flower catalysts c) Image of Graphene aerogel d) SEM of Graphene aerogel e) Graphene aerogel GDE

Conformal electrodeposition of layered manganese oxide intercalated with potassium as a pseudocapacitive functionalisation of carbon electrodes in Supercapacitors

Akash Ratnayaka, Upul Wijayantha, David Dibley
Loughborough University, Loughborough, United Kingdom

Abstract

Pseudocapacitance is the result of very fast redox reactions occurring on the surface of a 'pseudoactive' electrode material that result in an energy storage device showing the characteristics of a supercapacitor. Such materials usually are oxide metals that possess several valence states across the operating voltage of a device, such as ruthenium, manganese or cobalt oxides. Manganese oxides (MnO_2 , Mn_2O_3 , Mn_3O_4) have been of particular interest as a material due to their relative abundance and low toxicity compared to alternatives. We have developed a new technique for the electrodeposition of a layered Mn_3O_4 structure with intercalated potassium ions. This method creates a layer of manganese oxide which conforms to the surface shape of the deposition target with a controllable thickness. When used as anodes for asymmetric pseudocapacitors, carbon electrodes deposited with this manganese oxide layer allowed for increasing the voltage window from 1 to 2 volts with an aqueous electrode, resulting in a marked increase in energy densities of 26 Wh/kg at 1 A/g, compared to pristine activated carbon with only 4 Wh/kg at the same current density. Our asymmetric cell also demonstrated exceptional cell cyclability with coulombic efficiency losses of less than 5% after 10,000 cycles. We also demonstrate the excellent tuneability and conformity of manganese deposited with our technique (figure 1).

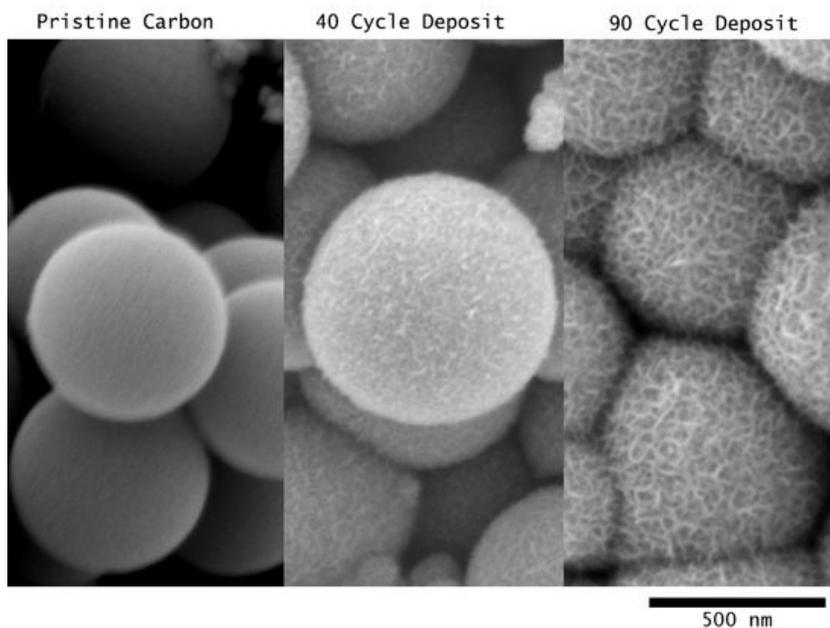


Figure 1: Activated Carbon nanospheres (left) with manganese oxide deposited at: 40 cycles (middle) and 90 cycles (right). Scale applies to all 3

P59

Cost Reduction of Pt Based Electrochemical Oxygen Reduction Reaction

Catalysts

Libo Zhang¹, Peng Gao¹, Hong Zhu¹, Terence Liu²

¹Beijing University of Chemical Technology, Beijing, China. ²Northumbria University, Newcastle upon Tyne, United Kingdom

Abstract

Active carbon supported Pt nano-particle catalyst is recognised as the best candidate for electrochemical oxygen reduction reactions (ORRs). However, its high cost and low durability hinders the mass production and wider applications, e.g. for fuel cells. Alternative catalysts to overcome such issues have been extensively studied in the past decades. This talk will presents several advanced approaches for bi-metallic and ternary metallic, as well core-shell as core-shell-shell structured Pt-based nano catalysts for ORRs. Performance including catalytic activity, durability are analysed and showed they are compatible to those from commercial Pt/C catalysts. The main objective of this work is to reduce the cost and to scale up the mass production.

Disposable Electrochemiluminescence Based Sensor for Rapid Drug

Detection

Kelly Brown, Charlotte Jacquet, Julien Biscay, Pamela Allan, Lynn Dennany
WestCHEM Department of Pure and Applied Chemistry, University of Strathclyde, Technology
and Innovation Centre, 99 George Street, Glasgow, G1 1RD, UK, Glasgow, United Kingdom

Abstract

The recreational abuse of non-traditional drugs has substantially increased over recent years.¹ Scopolamine a tropane alkaloid, falling within the same structural class as cocaine, has medical benefits as a an antispasmodic and antimuscarinic. However, its effect as an anticholinergic hallucinogen have seen its abuse both for recreational and criminal use.² The intrinsic advantages of electrochemiluminescence (ECL) make it an ideal alternative analytical technique for the rapid identification of electroactive compounds, with employment across a range of fields including medical diagnostics and pharmaceutical, food and environmental analysis.³

Here we report on a simple and rapid sensor for the detection of scopolamine within a variety of matrices, using a commercial carbon screen printed electrode modified with a Nafion® and ruthenium film. Following optimization, a calibration curve has been obtained across a linear range of 0.625 to 100mM with satisfactory reproducibility. Through our methodology we hope to drive forward ECL based sensors as viable alternatives for rapid crude drug detection systems which are not only portable and cost effective but ideal for in-field analysis with minimal sample preparation or expertise in ECL or electrochemical techniques required.

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https://www.unodc.org/wdr2018/prelaunch/WDR18_Booklet_1_EXSUM.pdf, (accessed 9th
October 2018)

2. J. Sáiz, T. Duc, M. López, C. Bartolomé, P. C. Hauser, and C. García-ruiz, "Science and Justice Rapid determination of scopolamine in evidence of recreational and predatory use," *Sci. Justice*, vol. 53, no. 4, pp. 409–414, 2013.

3. M. M. Richter, "Electrochemiluminescence (ECL)," *Chem. Rev.*, vol. 104, pp. 3003–3036, 2004.

P61

Sustainable Papermaking Using Foam

Elaheh Esmaeili

University of Strathclyde, Glasgow, United Kingdom

Abstract

It is clear that in spite the advent of the advanced technology, paper is still an important commodity, which involves not only the use of wood but also large volumes of water in the papermaking process. Increasingly however all around the world we are facing a water crisis. Therefore, measures must be taken to tackle water use, particularly for large production volume commodities such as paper. Relative to conventional water-based papermaking which involves using large flows of water, the process of making paper with foam forming utilises foam instead of water as a carrier for the fibres. Therefore, it leads to significant sustainability gains by reducing the water footprint of the process. Uptake of papermaking using foam is increasing considerably, owing to reduction of water and energy usage and better qualitative properties of the produced papers. This research aims at understanding the mechanism of how foam-fibre suspensions behave in the foam-formed papermaking process. Achieving a set of detailed guidelines on how to design and operate foam-based papermaking equipment which will be the outcome of this project can be beneficial for the papermaking industry.

P62

Developing polymer Battery materials

Sarah A. Alshehri^{1,2}, John Fielden¹, Gregory G. Wildgoose¹

¹University of East Anglia, Norwich, United Kingdom. ²princess Nourah bint Abdulrahman University, Riyadh, Saudi Arabia

Abstract

To meet our growing energy requirements in a sustainable manner, new energy storage materials are urgently required. We are developing new organic/inorganic materials for use in “hybrid–diffusional” battery devices, that combine desirable properties of traditional batteries with supercapacitors. This requires new types of cathode material to be developed that can store charge upon reduction.

P63

Characterising the electrochemical response of novel 3D printed CNT electrodes to the virulence factor pyocyanin

Caitlin McLean, Benjamin Tiller, Rolan Mansour, James Windmill, Lynn Dennany
University of Strathclyde, Glasgow, United Kingdom

Abstract

Pyocyanin (PYO), the virulence factor produced by *Pseudomonas aeruginosa*, can cause significant morbidity and mortality. Rapid detection *in-situ* is imperative for early diagnosis and treatment. [1] Electrochemical techniques allow for rapid and sensitive detection of toxins such as PYO [2], however, electrodes utilised within these techniques are often costly. 3D printing was utilised as a cheap, rapid method of electrode manufacture, using carbon nanotubes (CNTs) for incorporating conductive electrode layers, forming working and counter electrodes for electrochemical analysis.

Differential Pulse Voltmmetry revealed satisfactory detection of PYO by CNT electrodes. Linear quantification of PYO yielded an R² value of 0.968 for CNT electrodes, compared to 0.996 for the GC electrode. Promisingly, both electrodes detected PYO over the same concentration range. However, further work to improve limit of detection is required. CNT inter-electrode variability must also be progressed, through improving connections between electrodes and wires, as well as developing methods for printing a uniform and repeatable CNT layer. A long-term repeatability study gave an RSD value of 3.27% for a typical CNT electrode and 0.97% for a GC electrode.

Promisingly, the response of the CNT electrodes was reliable over multiple days, with the added advantage of their cheap manufacture. However, major steps must be taken towards developing a more robust and repeatable product regarding manufacturing methods, repeatable CNT density and building reliable connections. Future work will involve improving repeatability and striving for a cheap and disposable 3D printed electrode for infection detection within a clinical setting.

[1] <https://doi.org/10.3390/s16030408>

[2] <https://doi.org/10.1038/srep30001>

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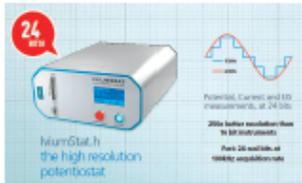
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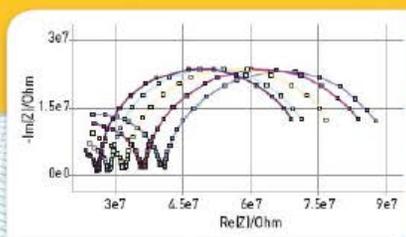
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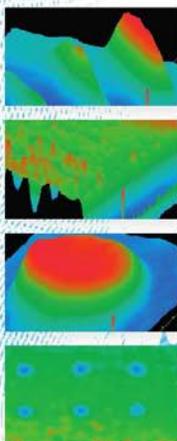
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M470

Introducing the 4th generation of scanning probe electrochemical workstations

- **9 available techniques:**
SECM, LEIS, SVP, SDS, SKP, OSP, ic-SECM, ac-SECM, ac-SDS
- High performance scanning stage:
0.09 nm ultimate z-resolution,
20 nm resolution on all axes,
100 mm scan range on all axes,
10 mm/s max scan speed
- New innovative techniques:
ic-SECM offering true simultaneous imaging of topography and reactivity,
ac-SECM offering measurement of surface conductivity without a mediator.
- Fully integrated potentiostat/galvanostat/FRA:
±10 V potential range, current ranges from **1 A to 1 nA**,
1 MHz to 1 μHz EIS capability



Product designed and manufactured by Uniscan Instruments Ltd,
a Bio-Logic SAS company

www.bio-logic.info



ec-lab

EC-Lab Ltd.
www.ec-lab.co.uk

Tel: 01753 822522
E-mail: sales@ec-lab.co.uk

New Product Information

VMP-300



POTENTIOSTAT/GALVANOSTAT



The ultimate multichannel electrochemical workstation



Modularity

- Multi-users
- Up to 16 independent channels
- EIS capability (10 μ Hz to 7 MHz)
- Ultra Low Current (100 nA to 1 pA)
- Current boosters: 1A/48V, 2A/30V, 4A/14V, 10A/5V
- Current boosters in parallel
- Linear Scan Generator (1 MV/s)

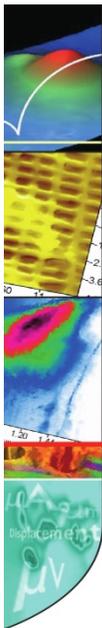


Unique features

- Up to 48 V control
- Up to 150 A (amplifiers in parallel)
- 1 pA min. current range
- 1 μ s min. acquisition time

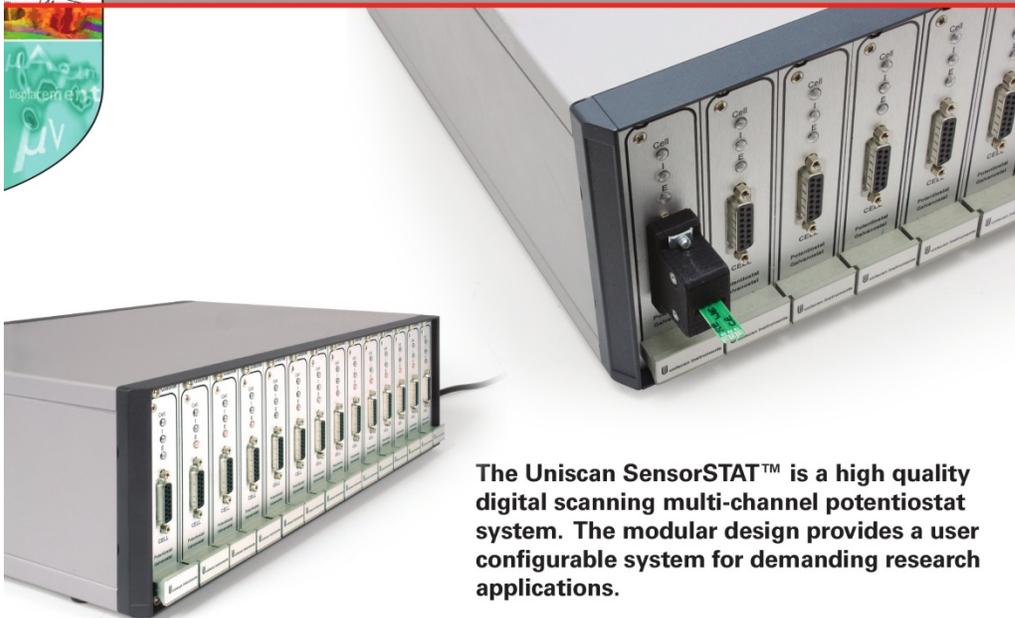


New Product Information



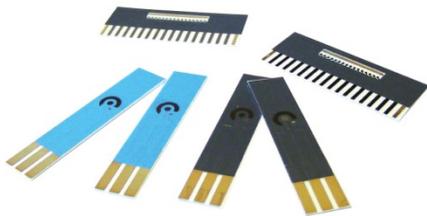
SensorSTAT

uniscan instruments



The Uniscan SensorSTAT™ is a high quality digital scanning multi-channel potentiostat system. The modular design provides a user configurable system for demanding research applications.

- Configurable for 8 to 14 channels
- Single USB connection controls all channels
- Ultra low noise current performance
- UiEChem™ software supplied with system
- Analogue triggering
- 5-WE multiplexing on each channel
- Interfaces to commercial electrochemical sensors
- User programmable techniques via macro programming
- ActiveX software for LabView™ applications



 uniscan instruments
A  BioLogic company
Science Instruments

Represented by:



ec-lab

Web: www.ec-lab.co.uk
e-mail: sales@ec-lab.co.uk

Tel: +44(0)1753 822522
Fax: +44(0)1753 822002

Metrohm Autolab



Metrohm Autolab has been a member of the Metrohm Group since 1999. Metrohm Autolab customers can look expect excellent sales and service support from a dedicated team of Electrochemists based at Metrohm's prestigious laboratories at Daresbury near Runcorn.

Metrohm Autolab produces four different potentiostat/galvanostat lines for a wide range of electrochemical applications, as well as modular extensions, software and accessories.



Metrohm
Autolab U.K.

www.metrohm-autolab.co.uk
Tel: 01928 579 600
Email: autolab@metrohm.co.uk

New Product Information

DROPSSENS

Metrohm
U.K. Ltd.

μ Stat 8000P Multi Potentiostat

Ref. STAT8000P



DropSens is proud to announce the launch of the NEW portable Multi Potentiostat μ Stat 8000P.

Our brand new instrument, of only 22x20x7 cm, includes 8 channels that can act at the same time as 8 independent potentiostats; it also includes one multichannel that can act as a potentiostat where up to 8 working electrodes share an auxiliary and a reference electrode.

With μ Stat 8000P users can perform up to 8 different electrochemical techniques at the same time; or carry out the study of one technique's parameter in just one step by applying the same electrochemical technique in several channels but selecting different values for the parameter under study. These are just examples of the enormous capabilities that our new instrument offers.

μ Stat 8000P can be applied for Voltammetric or Amperometric measurements, including 11 electroanalytical techniques. In addition, μ Stat 8000P owners can later upgrade their instrument to a μ Stat 8000 by just purchasing an extension. This self-upgrade does not require any hardware modification, but it is implemented by means of a Galvanostat software update kit.

The NEW portable Multi Potentiostat is Li-ion Battery powered (DC charger adaptor also compatible), and can be easily connected to a PC via USB or Bluetooth®.

μ Stat 8000P is controlled by the powerful software "DropView 8400" which allows plotting of the measurements and performing the analysis of results. DropView software provides powerful functions such as experimental control, graphs or file handling, among others.

Available techniques:

POTENTIOSTAT

Voltammetry

LSV	Linear Sweep Voltammetry
CV	Cyclic Voltammetry
SWV	Square Wave Voltammetry
DPV	Differential Pulse Voltammetry
NPV	Normal Pulse Voltammetry
NDP	Differential Normal Pulse Voltammetry
ACV	AC Voltammetry

Amperometry

AD	Amperometric Detection
FA	Fast Amperometry ($t_{int} < 0.1$ s)
PAD	Pulsed Amperometric Detection
ZRA	Zero Resistance Amperometry

Contact us:

email: dropsens@metrohm.co.uk | website: dropsens.co.uk | Tel: 01928 579 600

New Product Information

DROPSSENS

Metrohm
U.K. Ltd.

µStat 8000P Multi Potentiostat

Ref. STAT8000P

Instrument Specifications	
● Power	Li-ion Battery (3500 mAh) USB DC charger adaptor compatible (5 V, 15 W)
● PC interface	Bluetooth® USB
● Operating modes	8x 1 Channel Potentiostat 1x 8 Channel Potentiostat
● DC-Potential range	±4.096 V
● Current ranges (potentiostat)	±1 nA to ±100 mA (9 ranges)
● Maximum measurable current	±80 mA
● Rise time	20 µs
● Applied Potential Resolution:	1 mV
● Measured Current Resolution	0.025 % of current range (1 pA on lowest current range)
● Potential Accuracy	±0.2 %
● Current Accuracy	≤0.5 % (current range dependent)
● External inputs/outputs	- 5 Digital Input/Output pins (PIO 1, PIO 2, PIO 3, PIO 4, PIO 5) - 3 Analog Inputs multiplexing PIO 1, PIO 2, PIO 3 - 2 Analog Outputs (configurable I-out or E-out)
● Indicators	LCD display in front panel
● Dimensions	22.2 cm x 20.5 cm x 7.5 cm (L x W x H)
● Weight	1.6 kg

Control Specifications			
General Pretreatment	Conditioning stage duration:	0 – 1300 s	
	Deposition stage duration:	0 – 1300 s	
	Equilibration stage duration:	0 – 1300 s	
General Parameters	Begin, End, Base, Vertex potentials:	-4.096 V to +4.096 V	
	Step potential:	1 mV to 500 mV	
	Pulse potential:	1 mV to 250 mV	
	Scan rate:	1 ms up to 1.3 s per step	
Specific Parameters	SWV	Frequency:	1 Hz to 400 Hz
		Amplitude:	1 mV to 250 mV
	DPV, NPV, NDP	Modulation time:	1 ms to 1300 ms
		Pulse time:	1 ms to 1300 ms
	ACV	Frequency:	2 Hz to 250 Hz
		Amplitude:	5 mV to 250 mV (RMS)
	Chrono. Methods (AD, ZRA)	Interval time:	0.1 s to 1300 s
		Run time:	Hours (65000 points)
	Fast Chrono. Methods (FA)	Interval time:	1 ms to 1300 ms
		Run time:	Hours (65000 points)
PAD	Pulse time:	1 ms to 1300 ms	
	Interval time:	10 ms to 1300 ms	
	Run time:	Hours (65000 points)	

Specifications are subject to change without previous notice

Related products



CABSTAT1



CABSTATMULTI



CAST



CAST8X



8X110

Contact us:

email: dropsens@metrohm.co.uk | website: dropsens.co.uk | Tel: 01928 579 600

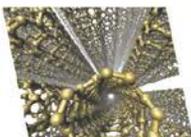
PARSTAT™ MC

multichannel potentiostat/galvanostat

Protect Your Experiment From
the Unexpected



Designed to protect your experiment from the unexpected, the PARSTAT™ MC is the most modular and robust multi-channel electrochemical testing platform on the market. It builds on our industry-leading 50+ years of experience in potentiostat development and software user-interface design.



- The *ultimate* in modular design
- Widest dynamic current range of 2 Amps to 4 nA (120 fA resolution) as standard - No need for expensive hardware options
- Hot-swappable channels allow potentiostats to be added or removed without interruption of experiments on other channels
- Fast data acquisition at 500 kS/sec allows for a wide range of high speed applications
- Features the most popular electrochemical acquisition and analysis software solution, VersaStudio
- Floating ground allows testing of multiple samples in the same cell



www.princetonappliedresearch.com
pari.info@ametek.com
P: 865.425.1289 F: 865.481.2410



New Product Information



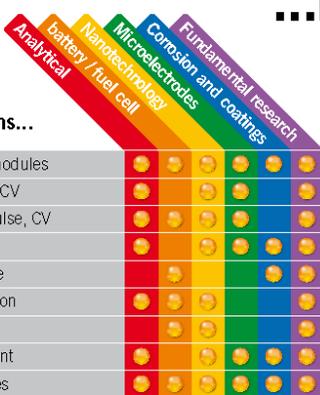
Not so much an instrument...



...more an orchestra.

The definitive modular system for electrochemical research, ModuLab delivers sublime performance for a vast repertoire of applications...

High performance 'Plug & Play' modules	●	●	●	●	●
64 MS/s smooth scan - LSV, LSP, CV	●	●	●	●	●
Up to 1 MS/s data acquisition - pulse, CV	●	●	●	●	●
100 nA current resolution	●	●	●	●	●
Up to ±25 A current - scan / pulse	●	●	●	●	●
±100 V compliance and polarization	●	●	●	●	●
10 μΩ impedance measurement	●	●	●	●	●
>100 TΩ impedance measurement	●	●	●	●	●
Multiple high-speed EIS techniques	●	●	●	●	●



US: Tel: 1-865-425-1360
 Fax: 1-865-481-2410
 UK: Tel: +44 (0)1252 556800
 Fax: +44 (0)1252 556899
 Email: solartron.info@ametek.com
 www.solartronanalytical.com



ModuLab the new gold standard for electrochemical instrumentation
 To compose an electrochemical test system that's totally in tune with your research requirements, contact Solartron today.

New Product Information

New Solartron EnergyLab XM for Energy Research

The Solartron EnergyLab XM is an electrochemical impedance workstation designed specifically for energy storage research:

- Batteries
- Supercapacitors
- Fuel cells

New Application-Focussed Product Line

The EnergyLab XM is the first of a new application-specific range of potentiostats from Solartron Analytical (Ametek), with small footprints and affordable pricing. There will be four systems in the range, which will be launched over the coming months.

Follow Blue Scientific on Linked In to receive details of the new instruments as they are announced.

EnergyLab XM

The first product in the series to launch is EnergyLab XM, for impedance testing of a variety of energy storage devices, including the testing of batteries, supercapacitors and fuel cells. The system includes all components required for this area of research, eliminating the need for costly hardware add-ons. The system includes:

- A reference grade potentiostat
- Frequency response analyser (FRA)
- 2A booster

The unit may be operated in boosted or unboosted mode (with automatic switching), providing optimum test conditions and accuracy for a wide range of devices.

EnergyLab XM's extreme sensitivity is ideal for complete characterisation of prototype low current or low impedance new generation cells. If high current is needed, external boosters can be connected and automatically controlled, allowing fully integrated high current tests at up to 100A.

For more information and quotes, please contact Blue Scientific, exclusive distributor for Solartron Analytical in the UK and Ireland, on 01223 422 269 or info@blue-scientific.com

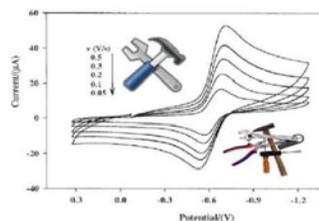
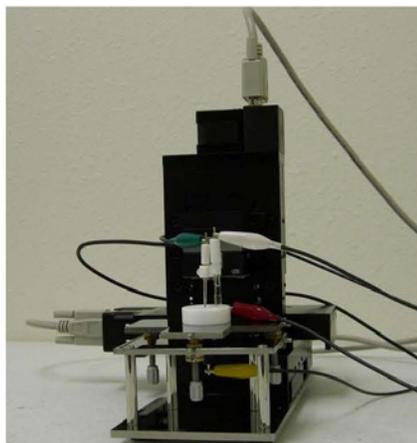


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Tools for Electrochemists!!!

CH Instruments at IJ Cambria Scientific



CHI920D SECM

The latest closed loop scanning electrochemical microscope

Products and accessories

- Wide range of electrochemical instrumentation; as well as potentiostats (and bipotentiostat) we have multiplexers, multichannel potentiostats, EQCM, and electrochemical detectors (ECDs) for LC and sensor use.
- Modules for very low current (pA range), compliance boost and rotating ring disk electrodes (RRDE)
- All instruments are very well developed and available at a very cost effective price; software included!
- In addition, we distribute the excellent ALS Ltd range of electrochemical accessories. We always keep a large stock of reference electrodes, working electrodes (including microelectrodes), and counter electrodes.
- We will almost always have the accessory parts that you require in stock for rapid delivery

Contact:

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Llanelli ♦ Carmarthen ♦ SA14 9DZ, UK
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(Mobile: 07957 287343)
IJ Cambria Scientific: www.ijcambria.com
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New Product Information



Think & Innovate



Thin-film microelectrodes

» POTENTIAL IN ELECTROCHEMISTRY

Thin-film technologies enable the manufacture of standard and customized (micro)electrodes with a low-cost, high precision and resolution. Micrux can adapt the electrochemical system to the requirements of the customers applications.

Thin-film accessories: flow cell and universal connector have been developed to use in combination with these electrodes.



Flow cell

» PROFICIENCY IN MICROFLUIDICS

Micrux has experience in developing capillary Electrophoresis microchips with electrochemical detection and the small and totally portable instrumentation to use them: Holder, iHVStat, miniPump, etc.



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Innovative Solutions for Multiple Applications



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