British Carbon Group Newsletter

December 2011







Fditorial

The main purpose of our newsletter this month is to give advance notice of our usual Christmas and Annual General meeting. The newsletter contains a meeting notice and the provisional minutes for the previous AGM. This year the AGM will be at the Institute of Physics, 76 Portland Place, London, W1B 1NT, on 21st December, beginning with a light lunch. As has been the custom over the past few years, there is no registration fee and all are encouraged to attend. It would be helpful though for catering purposes to let our Secretary know if you would like to come. His e-mail address is peter.c.minshall@magnoxnorthsites. com The programme will include speakers on carbon for clean-up of waste water and removal volatile toxins and one on carbon nanotubes in transport. Any one who has attended past meetings will know that the presentations are always by an expert in their field, are lively and sometimes provocative and provide a clear review of the area. We also hope to make the 2011 BCG/SCI Carbon in industry award at the meeting. The AGM is normally quite short and business-like. One of the main items is the election of committee members, for which we can always do with new blood. Anyone interested in joining the committee will always be welcome but should first contact our Chairman at gneighbour@brookes.ac.uk.

We have just made the 2011 BCG/SCI award to Dr. Steve Ragan of Jacobi Carbons. I'm delighted by this news as I worked with Steve some years ago on developing carbons for storage of natural gas. He will presented with his award and give a talk at the Christmas meeting on the use of carbons in environmental clean-up.

We also have a programme of future meetings in mind that are in the process of being organised. Nicole Grobert is planning a meeting on the Public Understanding of Carbon Science. This will include a competition with a modest cash prize for school children to come up with an appropriate design.

After the great success of the meeting last year, BAT are sponsoring a meeting which we will organise on the general subject of adsorption on carbon, or at least with applications in industry. This will be after Easter 2012. NanoteC12 will return to the UK next year (see the report on NanoteC11) to its home at Sussex, under the general charge of Prof. Malcolm Heggie. Many will also be interested in ECCRIA, the meeting on basic studies of Coal: this will take place in Nottingham in 2012. Fuller details are given later in the newsletter.

Finally, as nearly all Editors do, I have often exhorted my readers to respond to the pearls of wisdom enshrined in these pages. Any (printable) reaction will be most welcome, whether it be an offer of an article on a related topic, a general account of carbon research, a picture illustrating some interesting research or even a letter complaining about the contents. In fact, the latter would be especially welcome. Controversy is always welcome to stir up debate. Your committee has listened to the heartfelt pleas of your Editor and have agreed to give a modest prize of a £10 book token to the best unsolicited contribution. So come on, I know it's only a modest reward but do respond in kind. norman.parkyns@tesco.net

21st December 2011 12:00pm

Hooke Room Great Portland Street.

Email: gneighbour@brookes.ac.ul



British Carbon Group



A half day meeting will be held on Wednesday 21st December at the Institute of Physics, London devoted to latest developments, future prospects and cross-fertilization between three principal domains of carbon research: diamond, graphite and nanocarbons.

- 12.00 Buffet Lunch
- 12.45 AGM for BCG members
- 13.30 Welcome Dr Gareth Neighbour Chairman of BCG & Oxford Brookes University
- 13.35 The SCI Carbon in Industry Award & Lecture Dr Steve Ragan (Global R&D Director, Jacobi Carbons Ltd) Aspects of Activated Carbon and Household Water Filters
- 14.25 Dr Ray Whitby (Brighton University) Carbons for Waste Water Clean-Up
- 15.05 Tea & Coffee
- 15.35 Dr Peter Branton (BAT Ltd) Activated Carbon for the Adsorption of Toxicant Vapours
- 16.15 Dr Bojan Boskovic (Director, Cambridge Nanomaterials Technology Ltd) Carbon Nanotubes in Transport
- 16.55 Meeting Close



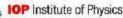




The British Carbon Group (registered charity 207890) is affiliated to The Royal Society of Chemistry, The Institute of Physics and The Society of Chemical Industry.







The British Carbon Group

Notice of 2011 Annual General Meeting

Notice is hereby given that the 2011 Annual General Meeting of the British Carbon Group will be held at the start of the Christmas half day meeting on Wednesday, December 21st 2011 at 12.45 pm.

The venue is the Institute of Physics, 76 Portland Place, London W1B 1NT.

The business of the Meeting is as follows: -

- 1. Apologies for Absence
- 2. Minutes of the previous AGM (Held at The University of Manchester, Manchester.

15th December 2010).

- 3. Matters Arising
- 4. Chairman's Report
- 5. Treasurer's Report
- 6. To Receive Notice of the Representatives of the Sponsoring Bodies
- 7. Election of Officers and committee members.
- 8. Any Other Business

At the 2011 AGM the Chairman and Vice Chairman must stand down and are not eligible for re-election. Nominations for these positions are invited. In addition the position of an ordinary Committee member falls vacant this year and nominations for this position are also invited.

Nominations duly proposed and seconded and with the consent of the nominee, should be received by the Honorary Secretary before 17th December, 2011 at the following address:

Dr. P. C. Minshall
Oldbury Technical Centre,
Oldbury Naite,
South Gloucestershire
BS35 1RQ
e-mail: peter.c.minshall@magnoxnorthsites.com

Minutes of the 2010 AGM held at the University of Manchester 15th December 2010

Present

Gareth Neighbour (Chairman), Tony Wickham (Treasurer), committee members Norman Parkyns, Mhairi Gass, Ian Kinloch, John Fisher and 20 BCG members

Apologies for Absence

Peter Minshall, Chris Ewels, Valeria Nicolosi, Nicole Grobert, Ashleigh Fletcher, Sergey Mikhalovsky, Jon Andresen.

Minutes of last AGM

The minutes of the last AGM held at the University of Oxford 18th. December 2009 were accepted as a true record with no matters arising.

Chairman's Report

Presented by Gareth Neighbour

Many thanks to all of you for coming today!

It has been a privilege to occupy the post of Chairman over the past year. I have always tried to remember that we are indeed a charity with the mission to promote carbon science through a variety of activities including education and research. I believe this year we have continued to pursue this goal. For example, in the award of bursaries to students, opening up meetings free of charge such as the one this afternoon, and of course the SCI Carbon in Industry Award recognizing leadership in industrial carbon science and technology.

Firstly, I would like to extend my sincere thanks to my fellow officers and committee members that have supported me over the last year. In terms of the committee, this year, John Andresen, Sergey Mikhalovsky and Ashleigh Fletcher must stand down as elected committee members. I would like to extend my thanks to John, Sergey and Ashleigh for their excellent service. John has kindly agreed to remain as SCI Representative replacing John Fisher and I am sure Sergey is not going far given he is organizing the "Applications of Carbon Science in Medicine" meeting next year. I would like to take this opportunity to thank in particular John Fisher who has given many, many, years of service right from the inception of the joint carbon group between the three institutions, IOP, RSC and SCI. He has been a mainstay of representing the SCI in BCG. This year, he has decided to retired as SCI Representative. We have valued enormously his contribution over the past 'thirty years' in various roles including secretary of the group. It is only befitting therefore that the SCI have chosen to recognize his contribution with a special recognition award. I'd like to call upon John to come up and collect his award.

This year we have run three meetings. The first was the 'Carbon and Industrial Applications' meeting held at the BAT Southampton organized by Peter Branton. This was an excellent meeting which attracted 49 attendees from 8 countries overseas and there's a photo in our last newsletter. I felt this meeting was very successful at bringing people together from essentially academic and industrial communities which the BCG has tried to foster in recent years. These types of meetings are very important given the need to network and to fully understand the end-user and ultimate application of the science and technology that we discuss. We also hosted again NanoteC in Oxford this year at the beginning of September organized by Dr Nicole Grobert, our Vice-Chair. The meeting was excellent for its scientific content and discussion with well over a hundred attendees taking in the delights of Corpus Christi.

We also hosted INGSM, the International Nuclear Graphite Specialists Meeting, at the Eastbourne Conference Centre. Special thanks go to Tony Wickham for organizing this meeting and as with every meeting Tony organizes, this meeting was a resounding success with well over a hundred attendees. This meeting travels around the globe every year, with previous locations involving the North America and Asia, and it was a great pleasure welcoming many international colleagues to debate the challenges of nuclear graphite.

We are a busy group so it was pleasing to note that the Royal Society of Chemistry were keen this year to show our activity as part of its Annual Congress. Perhaps it is no surprise that we have seen our membership grow over the last two or three years. Last, but not least is the meeting today which I am thankful to you all in attending and very thankful to Manchester and Tony for organizing.

I would also like to take this opportunity to single out Norman Parkyns for his excellent work in editing, once again, our newsletter, a task which is by no means easy collecting the copy!

We also continue to celebrate the life of the late Prof Brian Kelly in the award made for a paper presented at the international carbon conference and this year this went to Dr Jean-Philippe Tessonnier of the Fritz-Haber-Institute der Max Planck in Berlin. We also continue to be active in the European Carbon Association and indeed the ECA is likely to increase in size in the next year or two with probable applications to join from the Ukraine and Turkey. As you may remember, we established formal links with the Brazilian Carbon Group last year. This year, we were able to enter another bilateral agreement with the Ukrainian Carbon Group. Here, members of either group are eligible for the host nation's members' rate to register and attend the respective group's scientific meetings. Further, each body desires to promote and encourage opportunities for collaboration in carbon science between their respective memberships.

Finally, I should like to thank all the committee, especially the newer members who have given freely of themselves at a demanding time in their careers and injected so much enthusiasm. May I also take this opportunity to encourage everyone to participate in our Group at meetings, in representing the BCG through other fora and by putting forward yourselves for election to the Committee. The long term success of any group lies with welcoming new blood. To close, I would like to wish all of us, and BCG in particular, a very merry Christmas and a happy new year and let's hope 2011 is as successful as this year!

Treasurer's report

2009 Financial Report presented by Tony Wickham.

Group has a very healthy bank balance, topped up in previous years by income from International Carbon Conference, of £48,800 (including Brian Kelly award fund of approximately £15.000).

Income from interest in 2009 amounted to only £384 and in 2010 is expected to be even lower. Account is invested on our behalf by the RSC (very poor return on investment).

Most BCG meetings have broken even.

Annual account shows year end reduction due to timing of expenditure / income of conferences.

Overall, bank balance remains stable at around £48,000 overall.

Election of Officers and Committee Members

Under the constitution all four group officers have to stand down at the 2010 AGM and must offer themselves for re-election. All are willing to stand for another term and as there have been no other nominations for the posts the following are proposed as the group officers.

Chairman Gareth Neighbour Vice Chair Nicole Grobert Hon.Treasurer Tony Wickham Hon. Secretary Peter Minshall

John Andreson, Sergey Mikhalovsky and Ashleigh Fletcher will retire at the AGM and are not eligible to stand again for 12 months.

The following have been nominated and seconded for the vacant posts;

Chris Fwels

Peter Branton

John Fisher

There are no other nominees for the committee posts.

All the above were elected to the posts unopposed.

The following will be notified to the sponsoring bodies as representatives for 2011:

SCI - John Andresen

RSC – Norman Parkyns

IOP - Jon Goss

After the elections the AGM was closed

Graphene à la mode

It seems that graphene research continues to be the latest band wagon to scramble aboard. In the non-scientific media the name keeps on coming up. Even the Chancellor of the Exchequer has hitched a lift by announcing a large grant for research in this area. As a Group, we are rather under-represented by graphene researchers, despite this simply and solely consisting of carbon atoms. I hope that we can attract at least some of them under our umbrella. Testifying to the general interest that is being shown are the articles printed below. The first comes from an article in Chemistry World. The second was written by a graduate student at Cambridge, following NanoteC11 that he attended with our support. Finally, I repeat the announcement sent to me by Chris Ewels that appeared in the last newsletter about EU funding of research on graphene. Details are on the website www.grapheneflagship. eu/GF/index.php. There is clearly a lot of money out there waiting to be claimed. Let's hope that the technological consequences of this investment come up to the politicians' hopes.

Graphene by the kilo.

There is an article that you may not have seen in Chemistry World for September 2011 by Sarah Houlton entitled "Graphene by the kilo". It describes the activities of a company in Durham in association with the University that intends to turn out graphene by the kilogram. It uses a form of CVD where the feed is an alcohol which is passed over a suitable catalyst. The man in charge, Karl Coleman, has been making carbon nanotubes for some time but found that a relatively simple modification of feedstock and catalysts does the job. He has got together money from various sources and plans to set up the company Durham Graphene Science. He says he has considerable industrial interest for his product that he hopes to sell at around \$100/ kilo, the current price for carbon nanotubes. He sounds very enthusiastic in the article about the future of his product, so we will watch developments with interest

ndp

Towards graphene on the large scale

In spite of all the research interest that graphene has generated, useful applications for widespread utilization are yet to be realised. Several applications for graphene have been suggested and conceptually proven¹ but are limited by the lack of a cost effective, scalable process to produce high quality graphene. Chemical vapour deposition (CVD), a process in which transition metal surfaces are exposed to a gaseous carbon precursor at elevated temperatures has emerged as one of the many technologies that look quite promising this context. Graphene CVD over large areas has been demonstrated on sacrificial polycrystalline metallic films/foils of Ni, Cu etc. However, the understanding of the role of transition metal templates in CVD of graphitic carbon nanostructures like graphene and carbon nanotubes remains incomplete. This limits a widespread utilisation as control over their growth, remains empirical and rudimentary.

My research at Dr. Hofmann's group at the Engineering Department at the University of Cambridge focuses on bottom up growth of graphitic nanostructures like graphene and carbon nanotubes from novel catalyst systems. We use a combination of high-pressure X-ray photoelectron spectroscopy (XPS)² and in-situ X-ray diffraction (XRD)² experiments analysing the behaviour of thick (>300 nm) poly-crystalline and nano-particulate catalysts during hydrocarbon exposure at temperatures ranging from 500-1000°C. We focus on the comparison of transition metal catalysts like Ni and Cu and novel oxide catalyst systems like ZrO₂³ and HfO₂⁴. Our time-and depth resolved in-situ measurements allow a clear identification of the catalyst state at any point of growth. Hence we can experimentally study the process of carbon incorporation in the growing nanostructure as it happens. This is a critical step in the direction towards understanding the fundamental growth mechanisms. Further, we have also used this method to study alloy catalysts (Ni-Au)² and demonstrated a technique to rationally engineer catalysts for low temperature graphene nucleation and growth. This is another important step in improving CVD technologies for graphene synthesis, essential for realising widespread application of graphene. With a final objective towards scalable device integration of graphene from our experiments we consider HfO₂ as a model system to study graphene growth directly on a dielectric which will circumvent the existing problem of post synthesis transfer from sacrificial metal catalysts.⁴ An understanding of the growth mechanism of graphitic nanostructures from oxides remains incomplete to date and is an active area of research. It is however, widely expected to be different from that of transition metals due to the large difference in carbon solubility in these systems.

References:

- (1) Geim A K and Novoselov K S Nature materials 2007, 6, 183-91.
- (2) Weatherup et al. Nano Letters 2011.
- (3) Steiner et al. JACS 2009, 131, 12144.
- (4) Kidambi et al. Phys. Status Solidi RRL 2011, 5, No. 9, 341–343.

Piran R. Kidambi

British Carbon Group & SCI

"Carbon in Industry award 2012"

(As I report in the editorial, Dr. Steve Ragan is this year's winner of the this award. He will receive it at the Christmas meeting The citation is printed below.)

Nomination for the SCI "Carbon in Industry" Award

Nominee: Dr Steven Ragan Affiliation: Jacobi Carbons Ltd

Citation

Dr Ragan has been with Jacobi since 2003 and is based in the UK. As Director of R&D, Steve is responsible for all Jacobi's laboratories in Europe as well as all R&D activities within the Group. He is responsible for all new technologies and new applications. His current duties are split between UK, Vierzon and Premnitz to coordinate laboratory and R&D activities. He is a widely known and respected figure within the international activated carbon and adsorption community.

Dr Ragan's achievements include:

- · Board member of Sutcliffe Speakman Carbons Ltd and Lakeland Carbons.
- · Lead role in the installation and commissioning of new manufacturing processes and linking QA, R&D and production.
- Introduction of new water filters and helping companies to achieve ISO 9001 and 9002 accreditations.
- · Winner of the 2005 Plastics Industry Award Best sales & marketing initiative for the Aqua Optima water filter cartridge.
- · At Brita, Steve was offered the Chairmanship of the Technical Advisory Committee of the US Water Quality Association.
- · Steve has 21 published articles and 26 full conference and short papers. He is a member of the British Carbon group and has been nominated to join its executive from Jan 2012.He is also a member of the Technical Advisory Committee of WQA.

Future meetings

9th EUROPEAN CONFERENCE ON COAL RESEARCH AND ITS APPLICATIONS: ECCRIA 9

University of Nottingham

The British Carbon Group will be assisting with the administration of the 9th European Conference on Coal Research and its Applications, which will be held at the Jubilee Campus of The University of Nottingham from September 9th – 12th 2012.

More information will be available nearer the time. Any enquiries relating to this event should, in the first instance, be directed to Professor John Patrick: john.patrick@nottingham.ac.uk

Monday 10th to Wednesday 12th September 2012 9.eccria.org Organised by the Coal Research Forum http://www.coalresearchforum.org

Building on the success of the previous ECCRIA conferences, this ninth conference is to be held at the University of Nottingham. Situated in the East Midlands region of England on the edge of Sherwood Forest, the legendary home of Robin Hood, Nottingham has had a colourful past of legend and lace and caves and castles. Beneath the bustling city streets is a unique labyrinth of man-made caves, and above the ground, Nottingham Castle stands high on the city skyline. Nottingham also has an abundance of restaurants, cafes, pubs and bistros. This friendly and vibrant city provides an excellent base for the conference and a showcase for the English East Midlands, where it is hoped that delegates from outside the UK will continue to support this event and will thoroughly enjoy their stay in Nottingham.

SCOPE AND PURPOSE

The purpose of this conference is to bring together researchers in universities with participants from industry, who are also carrying out research or are interested in the application of the research in industry. Papers are invited which describe applications in coal utilisation and preparation with particular reference to the following areas: improving efficiency and reducing emissions for clean coal technologies in conventional combustion power generation, including co-firing with biomass and wastes, ash and slag issues; advanced power generation, including enabling technologies; industrial combustion; gasification; CO2 removal, transport and storage technologies, including oxyfuel combustion and chemical looping; coal characterisation and plant diagnostics; all emissions issues, including alternative NOx reduction techniques to meet 2016 NOx limits; carbonisation and other metallurgical uses; coal-derived products; coal preparation and handling; underground coal gasification and coal bed methane, and all other coal conversion processes, including coal to liquids. Research students are also strongly encouraged to submit papers, attend and give presentations and

there will be a reduced fee for students.

CALL FOR PAPERS AND PRELIMINARY REGISTRATION

Prospective authors are asked to make a preliminary registration, then submit one or more abstracts at the conference website 9.eccria.org by 2nd March 2012. Abstracts MUST be submitted via the website. For papers accepted, one author must present the paper at the conference either as an oral presentation or as a poster.

PROCEEDINGS

All authors of accepted papers are invited to submit, by 30th September 2012, a full paper for possible inclusion in a Special Issue of FUEL. The papers submitted should be prepared strictly in accordance with the Guide for Authors and MUST be submitted electronically through http://ees.elsevier.com/jfue/. Authors should clearly indicate that the paper is for the 9th ECCRIA Special Issue. All full papers will be subject to the normal refereeing requirements of FUEL.

Other meetings

(This is a commercial meeting, nothing to do with BCG but the contents are of interest)

Graphite Conference

06 December 2011 - 07 December 2011, Jumeira Carlton Hotel, London

Key Speakers

- · Gary Economo, Chief Executive Officer, Focus Metals Inc, Canada
- · Klaus Rathberger, Managing Director, Georg H. Luh GmbH, Germany
- · Gerry Hand, Vice President of Marketing, Superior Graphite, USA
- · Matimba Khoza, Managing Director, Jonkel Carbons and Graphites (Pty) Ltd , South Africa
- · Jack Lifton, Founding Principal, Technology Metals Research LLC , USA
- · Juan Carlos Zuleta, Lithium Economics Analyst, Seeking Alpha, Bolivia
- · Jonathan Lee, Battery Materials and Technologies Analyst, Byron Capital Markets, Canada
- · George Hawley, President, George C. Hawley and Associates Minerals Consultants, USA
- · Sandio Pereira, Superintendente de Novos Projetos Minerais, Magnesita, Brazil
- · Dr Julian Norley, Sr Corp. Fellow, Advanced Energy, GrafTech International Holdings Inc, USA
- · Mark Muzzin, Managing Director, Strategic Energy Resources, Australia

Industrial Mineral Events would like to announce the launch of The Graphite Conference 6-7th December 2011, the world's first dedicated natural and synthetic graphite industry event.

Touted by industry analysts as the next strategic mineral, a diamond in the rough for investors, the cornerstone of green industries and a 'wonder material' with applications from high technology to water purification; natural graphite, and synthetic graphene are set to be big industries of the future.

Our brand new event will shed light on the increasing industry excitement around existing applications and the potential of graphites as a replacement for ageing processes and materials.

Reasons to attend:

- ~Gain extensive knowledge of the carbon industries and the surrounding macroeconomic environment
- ~Hear from a roster of the best speakers and experts on current and future graphite and graphene applications.
- ~Network with delegates from mining and refining to end product manufacture and investment, to make contacts and win business.

Topics include

- ~Graphite is this the next strategic mineral?
- ~Price rises and availability
- ~Revival of traditional markets and the growth of new applications
- ~Natural Graphite exploration and mining projects
- ~The increasing demand for synthetic Graphite
- ~A diamond in the rough for investors?
- ~Graphite in the Green Industry Electric Vehicles, Solar and Nuclear
- ~Graphene a revolutionary 'wonder material'?

Reports of meetings

3rd EdF-Energy Conference on Nuclear Graphite: "Modelling and Measuring Reactor-Core Graphite Properties and Performance University of Aston, Birmingham, 1st – 3rd November 2011

This was the third (and probably the last) in a series of meetings on nuclear-graphite issues relating to the continued operation and life extension of the UK's Advanced Gas-Cooled Reactors (AGRs); the first two meetings were held in Cardiff and Nottingham and the proceedings published in book form by The Royal Society of Chemistry.

The meetings were proposed by EdF Energy (Generation) UK Ltd (formerly British Energy) as a forum to explain and discuss the very large body of work which has been put into understanding the irradiation behaviour of the Gilsocarbon graphite used both as a moderator and as the structural material of the core. Since irradiation damage by fast neutrons leads to dimensional changes, bowing of bricks and channels, and changes in critical properties like strength, Young's modulus, thermal conductivity and the coefficient of thermal expansion, with the effects modified by irradiation-induced creep, and all of this is further complicated by the simultaneous graphite oxidation in the coolant brought about by ionising radiation, important matters like predicting the onset of component cracking (a degree of which can be tolerated by the structure) and ensuring continued access by control rods and fuel stringers into the channels which lie within the graphite have been the focus of attention for the Company for a long time.

Drawing on expertise from a pool of experienced industry organisations and individuals, together with a number of Universities with particular specialities, EdF Energy has invested well in excess of £ 30 million on graphite-related research, measurements and modelling, this apparently vast sum of money being readily justified in terms of the value of the electricity generated through life extension of the 14 AGRs. EdF Energy works closely with the regulators (now the Office for Nuclear Regulation, formerly the NII) and informs them fully at all stages of the process.

This present meeting focussed on four themes: Mechanistic Understanding, in which Professor Brian Rand (formerly of the Universities of Leeds and Pretoria [RSA]) gave a keynote lecture which was followed by eleven contributed papers; Empirical Modelling (keynote by Professor James Marrow, University of Oxford and fifteen other contributed papers); Statistical Methods for Data Analysis (keynote by Dr. Philip Maul of Quintessa Ltd and three contributed papers); and Plant Issues (keynote by Mr. Bryan Banahan of Magnox Ltd) and ten contributed papers). Each of the four sessions was followed by a themed discussion, the outcome of which was recorded and will be published alongside the technical papers by the RSC, probably early in 2013. The book editor is our Chairman, Gareth Neighbour.

84 delegates enjoyed this residential meeting in the excellent facilities at 'Conference Aston', with an excellent conference banquet held at 'Bank' in the centre of the city adjacent to one of the many canal junctions.

-Tony Wickham

NanoteC11

Institut des Materiaux Jean Rouxel (IMN), University of Nantes, 31 August 2011

This year's NanoteC conference was held at the Institut des Materiaux Jean Rouxel (IMN) at the University of Nantes on the west coast of France. Starting on Wednesday 31st August over 100 researchers from around the world (our furthest coming from Australia) met to present and discuss new investigations and ideas in carbon research and nanotechnology. Each year the range of talks is slightly different and reflects the trends in carbon nanoscience – this year the presence of the chemists was very notable, with interesting discussion of both new "bottom up" and refined "top down" approaches to creating new custom designed nanomaterials, as well as advances in nanocomposite design and control.

The mix of physicists and chemists led to some stimulating debate, and as Malcolm Heggie informed everyone, "A physicist is a Fourier-transformed version of a Chemist".

The four day event included a poster session with two poster prizes and four honorary mentions, won this year by Songül Güryel for her poster "Theoretical Analysis of the Intrinsic Mechanical Properties of Graphene", and J. David Nuñez for "Hydroxyapatite-CNT Nanostructured biocomposite for Tissue Engineering Scaffolds".

Conference Dinner was in a restaurant in a former biscuit factory (the famous 'LU' biscuits come from Nantes), now one of Nantes' most modern and hip places down near the banks of the River Loire. A wine tasting with wines from the Loire Valley region opened the conference dinner, thanks to the support of one this year's sponsors, Solvay. We were also very grateful for financial support from Linde, the University of Nantes, the COST Project NanoTP, the Pays de la Loire region, and the C'NANO nanoscience network in the north west of France. In the spirit of entente cordiale between carbon groups, we are also pleased to say it was also supported by the GFEC (Groupe Français d'Etude des Carbones).

Overall, NanoteC11 was a big success for sharing the views of the international science community and the state of the art in carbon related research. Hopefully there was a friendly and open atmosphere, that ended well as the end of the conference overlapped with a large open air free jazz festival in Nantes, so dazed carbon scientists were able to stagger mentally overloaded out of the lecture theatre and amble down beside the River Erdre and chill out all weekend to the mellow tones of jazz!

Next year NanoteC12 will return to its roots, back at Sussex University next to Brighton,

starting on the Wednesday August 29th until Saturday 1st September. Further details will soon be available on the BCG website at http://www.britishcarbon.org/nanotec

-Philipp Wagner and Chris Ewels

Brian Kelly award 2011

The recipient of our premier award, the Brian Kelly award, for 2011 was Monica Haus of the University of Sydney, New South Wales for her paper, an abstract of which is given below.



The picture shows Monica receiving her award from Gareth Neighbour at Carbon 2012 in Beijing

FIXED-AND FLUIDISED-BED SYNTHESIS OF COILED CARBON FIBRES ON IN SITU GENERATED H,S-MODIFIED Ni/Al,O, CATALYSTS

Monica J. Hanus and Andrew T. Harris

Laboratory for Sustainable Technology, School of Chemical and Biomolecular Engineering, Chemical Engineering Building (J01), University of Sydney, NSW, 2006, Australia

INTRODUCTION

The remarkable properties and the demonstrated potential applications of coiled carbon fibres (CCFs) $^{[1]}$ make large-scale synthesis highly desirable but despite this CCFs are not yet commercially available. The lack of scalable synthesis method and the universally poorly understood synthesis processes are the main barriers to the large-scale CCF synthesis. Using a NiSO₄/Al₂O₃ catalyst precursor we developed an in situ generated H₂S-modified Ni/Al₂O₃ catalyst suitable for scalable fluidised-bed CCF synthesis. The influence of reduction and synthesis temperatures, the duration of the catalyst reduction period, the influence of H₂ addition during synthesis, synthesis duration, C₂H₂ concentration in the synthesis gas and catalyst loading on CCF formation were explored.

EXPERIMENTAL

NiSO₄/Al₂O₃ catalyst precursors were produced by a wet impregnation method whereby NiSO₄·6H₂O was dissolved in deionised water, mixed with calcined Al₂O₃ (1:20 wt.Ni:Al₂O₃) and dried in a 60 °C oven to form a free-flowing powder that was subsequently calcined in static air at 500 °C for 5 hrs^[2].

For CCF synthesis the NiSO $_4$ /Al $_2$ O $_3$ catalyst precursor was heated to the reduction set point temperature(650 °C) under N $_2$, subjected to reduction conditions (H $_2$, N $_2$), subjected to synthesis condition (C $_2$ H $_2$, H $_2$, N $_2$) and then cooled down under N $_2$. The fixed bed-reactor consists of an electrically heated, 0.7 m long, 0.034 m I.D horizontallyorientatedAl $_2$ O $_3$ tube (MTI Corporation) and the fluidised-bed reactor consists of 1 m long, 0.052 m I.D. verticallyorientated Inconel 601 tube located within an electrically heated furnace.

Details of reduction and synthesis conditions used in the fixed and fluidised-bed reactors are provided elsewhere^[2]. The influence of parameters on CCF synthesis was explored using a high-throughput reactor by changing one parameter of a standard experiment at a time. The high-throughput reactor consisted of a bespoke thermogravimetric analyser (TGA)connected to a system of external mass flow controllers and software to allow the addition of the required gases and realtime weight monitoring. The standard experiment and a summary of all conditions that were substituted into the standard experiment are provided elsewhere^[3].

The catalyst precursor and fixed and fluidised-bed synthesized catalysts and products were characterised using x-ray diffraction (XRD), x-ray fluorescence (XRF) and x-ray photoelectron spectroscopy (XPS) analysis. All synthesis products were characterised by scanning electron microscopy(SEM). Instrument settings and sample preparation details are provided elsewhere^[2].

Thermodynamic equilibrium models were constructed using Outokumpu's HSC Chemistry 4.1 and use a Gibbs free energy minimization routine to explore the species that predominate at particular conditions imposed in the experimental program. Details of the models are provided elsewhere^[2].

RESULTS AND DISCUSSION

NiSO₄/Al₂O₃ was found to be an effective and fluidisable catalyst precursor for CCF synthesis. Thermodynamic equilibrium modeling predicted that in both the fixed- and fluidised-bed reactors the NiSO₄/Al₂O₃ catalyst precursor reduced to form Ni/Al₂O₃ and H₂S under the reduction conditions imposed in the experimental program. Evolution of H2S during reduction was confirmed by the white ZnS precipitate formed as the reactor exhaust gases were fed through a Zn(CH₃COO)² solution. XRF and XPS confirmed S concentration post reduction to be <0.9 wt.%, while XRD confirmed that the bulk of the NiSO₄ catalyst precursor had been reduced to Ni in both the fixed- and fluidised-bed reactors and there were no significant (>0.5 wt.%) quantities of crystalline S compounds present. The catalyst

produced in situ was mainly Ni/Al₂O₂ with trace quantities of S adsorbed onto the Ni surface. CCFs were produced in both fixed- and fluidised-bed reactors on the S-modified Ni/Al₂O₂ catalyst (Fig. 1). The presence of H₂S was proven to be vital for CCF synthesis; only straight carbon fibres formed with a NiO/Al₂O₂ catalyst precursor produced by calcining the NiSO₄/Al₂O₂ catalyst precursor to a higher temperature (800 °C). In low concentrations the presence of S decreases the structural perfection of carbon fibres^[4]. It is postulated that the S adsorbs preferentially to some Ni catalyst faces resulting in anisotropic carbon extrusion from the various catalyst faces and, ultimately, CCF formation^[5]. To the best of our knowledge, this study was the first report of the synthesis of coiled carbon fibres in a scalable fluidised bed reactor. This synthesis method avoids the need to store and add a toxic S compound (e.g., H.S, or C.H.,); a requirement of conventional CCF synthesis on Ni catalysts, and presents opportunities for large-scale synthesis of these materials. Reduction and synthesis temperatures heavily affected CCF synthesis with noteworthy quantities of CCFs produced at reduction and synthesis temperatures of 650±50 °C. The NiSO, catalyst precursor decomposed to NiO during heat up to the reduction temperature and the degree of decomposition was commensurate with temperature. The quantity of H,S released during reduction was dependant on the quantity of NiSO, remaining after heat up and was a vital parameter for CCF synthesis: too high H₂S concentrations caused catalyst poisoning and at too low concentrations CCF catalysis did not occur. 650 °C was also the preferred synthesis temperature for CCF formation.

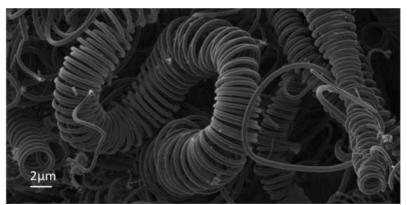


Fig. 1 SEM image of coiled carbon fibres synthesised with a NiSO $_4$ /Al $_2$ O $_3$ catalyst precursor in a fluidised-bed reactor

Reduction occurred quickly and, on average, was complete within 0.7 min of the introduction of H₂. H₂S, which forms during reduction, desorbs from Ni with time and consequently, catalyst composition varies with the duration between the conclusion of reduction and commencement of synthesis. CCF synthesis occurred when reduction duration was between 1 and 20 min with greatest coil yields observed when reduction duration was 10 min. Only straight carbon fibres formed at reduction durations of 30 min or more and this is attributed to insufficient H₃S concentration for CCF catalysis. Reduction occurred even

without the addition of H2due to C_2H_2 cracking, however this caused reduction and synthesis to occur in quick succession and when synthesis occurred <1 min after the commencement of reduction, CCF yield was poor and this was attributed to excessive catalystH $_2$ S concentrations. The presence of H_2 during synthesis was found to have a positive effect on CCF formation.

Carbon fibres began to form within 1 min of the introduction of C_2H_2 . Both thick and thin carbon fibres(~500 nm and ~100 nm in diameter respectively) formed and within 2 min most of the thick carbon fibres had begun to coil however most of the thin carbon fibres were straight at all synthesis durations examined. The catalyst began to deactivate and carbon fibre synthesis slowed after about 5 min of synthesis when many catalyst particles became encapsulated in carbon. Though carbon yield increased with increasing synthesis gas C_2H_2 concentration, the morphology of the product was largely unaffected and coils formed at all C_2H_2 -concentrations tested.

The ratio of thick to thin carbon fibres increased with catalyst Ni loading, indicating an increase in catalyst particle size, and carbon yield also increased with Ni loading. Of the catalysts tested, the greatest ratio of CCFs in the product was produced with the 1:20 wt. Ni:Al₂O₂ catalyst.

CONCLUSIONS

The use of a ${\rm NiSO_4/Al_2O_3}$ catalyst precursor allows for CCF synthesis in a scalable fluidised-bed reactor, avoids the need to store and add toxic ${\rm H_2S}$ and thus shows great potential for large-scale CCF production. CCF synthesis with a ${\rm NiSO_4/Al_2O_3}$ catalyst precursor was investigated using a high-throughput reaction system. Reduction temperature and reduction duration effect ${\rm H_2S}$ concentration and CCFs formed in only a narrow reduction and synthesis temperature range of 650±50 °C. ${\rm H_2S}$ desorbs from Ni with time and thus reduction duration effects catalyst composition and CCF synthesis occurred after reduction durations between 1 and 20 min. The presence of ${\rm H_2}$ during synthesis was found to have a positive effect on CCF formation. The ratio of thick to thin carbon fibres increased with catalyst nickel loading and the greatest ratio of CCFs was produced with a 1:20 wt. ${\rm Ni:Al_2O_3}$ catalyst

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REFERENCES

[1] Motojima S, Chen X, Yang S, Hasegawa M. Properties and potential applications of

carbon microcoils/nanocoils. Diamond Relat. Mater. 2004:13:1989-92.

- [2] Hanus MJ, Linkson PB, Harris AT. Fixed- and fluidised bed synthesis of coiled carbon fibres on an in situ generated $\rm H_2Smodified\ Ni/Al_2O_3$ catalyst from a $\rm NiSO_4/Al_2O_3$ precursor. Carbon.2010;48:3931-8.
- [3] Hanus MJ, MacKenzie KJ, King AAK, Dunens OM, Harris AT. Parametric study of coiled carbon fiber synthesis on an in situ generated $\rm H_2S$ -modified Ni/Al $_2\rm O_3$ catalyst. Carbon. 2011;Submitted.
- [4] Kim MS, Rodriguez NM, Baker RTK. The interplay between sulfur adsorption and carbon deposition on cobalt catalysts. J. Catal. 1993;149:449-63.[5] Chen X, Motojima S. The growth patterns and morphologies of carbon micro-coils produced by chemical vapour deposition. Carbon. 1999;37:1817-23

And finally...

This has nothing whatsoever to do with carbon but as I have the space and in order to cheer you up on long winter evenings, here is...

HOW TO GIVE A CAT A PILL

- 1. Pick up cat and cradle it in the crook of your left arm as if holding a baby. Position right forefinger and thumb either side of cat's mouth and gently apply pressure to cheeks while holding pill in right hand. As cat opens mouth, pop pill into mouth. Allow cat to close mouth and swallow.
- 2. Retrieve pill from floor and cat from behind the sofa. Cradle cat in left arm and repeat process.
- 3. Retrieve cat from bedroom, and throw soggy pill away.
- 4. Take new pill from foil wrap, cradle cat in left arm, holding rear paws tightly with left hand. Force jaws open and push pill to back of mouth with right forefinger. Hold mouth shut for a count of ten.
- 5. Retrieve pill from goldfish bowl and cat from top of wardrobe.
- 6 Call spouse from garden. Kneel on floor with cat wedged firmly between knees, hold front and rear paws. Ignore low growls emitted by cat. Get spouse to hold head firmly with one hand while forcing wooden ruler into mouth. Drop pill down ruler and rub cat's throat vigorously.
- 7. Retrieve cat from curtain rail, get another pill from foil wrap. Make note to buy new ruler and repair curtains. Carefully sweep shattered figurines and vases from hearth and set to one side for glueing later.
- 8. Wrap cat in large towel and get spouse to lie on cat with head just visible from below armpit. Put pill in end of drinking straw, force mouth open with pencil and blow down drinking straw.
- 9. Check label to make sure pill not harmful to humans, drink a beer to take taste away.

Apply Band-Aid to spouse's forearm and remove blood from carpet with cold water and soap.

- 10. Retrieve cat from neighbour's shed. Get another pill. Open another beer. Place cat in cupboard, and close door onto neck, to leave head showing. Force mouth open with dessert spoon. Flick pill down throat with elastic band.
- 11. Fetch screwdriver from garage and put cupboard door back on hinges. Drink beer. Fetch bottle of Scotch. Pour shot, drink. Apply cold compress to cheek and check records for date of last tetanus shot. Apply whisky compress to cheek to disinfect. Toss back another shot. Throw T-shirt away and fetch new one from bedroom.
- 12. Call fire brigade to retrieve the damn cat from across the road. Apologise to neighbour who crashed into fence while swerving to avoid cat. Take last pill from foil wrap.
- 13. Tie the little bastard's front paws to rear paws with garden twine and bind tightly to leg of dining table, find heavy-duty pruning gloves from shed. Push pill into mouth followed by large piece of fillet steak. Be rough about it. Hold head vertically and pour 2 pints of water down throat to wash pill down.
- 14. Consume remainder of scotch. Get spouse to drive you to the emergency room, sit quietly while doctor stitches fingers and fore arm and removes pill remnants from right eye. Call furniture shop on way home to order new table.
- 15. Arrange for RSPCA to collect mutant cat from hell and call local pet shop to see if they have any hamsters.

HOW TO GIVE A PILL TO A DOG

- 1. Wrap it in bacon (the pill, not the dog).
- 2. Toss it in the air