SGT NEWS

SEARCHING FOR COMMERCIAL SUCCESS - UK-IRELAND SOL-GEL MEETING

Sol-gel science and technology has not yet reached its full potential. The application of the process and products made in this way have not had many commercial successes. The UK-Ireland Sol-Gel Group met at Nottingham Trent University to hear from a leading figure in the search for applications, present results from recent research and discuss the problems that have arisen.

The meeting was held at the university's new Faculty of Education building and hosted by Dr Carole Perry of the Department of Chemistry. Dr Perry welcomed the participants from the UK, Ireland, Portugal and Germany to the first ever meeting at the Faculty. The UK-Ireland Sol-Gel Group is an independent body and the Society of Glass Technology has supported the meetings held since it was conceived in 1996. The Royal Society of Chemistry provided additional support for this event.

The meeting consisted of an early evening meal followed by a talk presented by Professor Helmut Schmidt of the Institut für Neue Materialen (INM), Saarbrucken. Each poster author was then given a two minute slot to present their work and introduce themselves. There was then a poster session until around 10.00pm. Delegates returned the next day after breakfast to hear presentations by representatives of research groups around their current research topics. Between sessions there were again opportunities to inspect and interrogate the poster authors.

The informality of the first evening helped break the ice and gave researchers the opportunity to put over their own experiences and ask for advice from the group on any difficulties they have come across.

SOL-GEL TECHNIQUES FOR NEW INDUSTRIAL PRODUCTS

The Institut für Neue Materialen has expanded to employ more than 250 staff concerned with finding, among other things, new uses for products made by the sol-gel process. Professor Schmidt has 320 published papers and ten patents. His talk on sol-gel techniques for new industrial products began by highlighting the need to be aware of the markets for commercial applications. Science can drive certain areas, whereas the market can sometimes 'pull' research if the demand is there. The pull is usually much more compelling unless something revolutionary is produced by science.

The sol-gel process has the major potential to produce some interesting products enabling new phenomena in materials. However development costs are high and as a result, the end product needs to sell in bulk or add more value. Good work is being done in developing applications of the sol-gel process, but these have a restricted materials market. The only successful bulk application of the sol-gel process is the 200,000 tones/year of TiO_2. Smaller companies can combine the sol-gel process and engineering together to reduce the gap between basic science, development and commercial application.

Nanotechnology is the popular term which will see further introduction of sol-gel derived products.

Clinical test are underway in Germany for a sol-gel derived, particle-containing superparamagnetic iron oxide which is preferentially ten times more likely to be taken up by tumour cells. Once absorbed, a magnetic field is applied, the cell is destroyed and the tumour disappears.

Thixotropic printing of a sol-gel substrate on foils is possible. With the correct chemistry, the printing of holographs is viable. From this work, a system is up and running which prints CDs at 60m/min or 700 times faster than by injection moulding.

A sol-gel based binder with silver (98wt% Ag) has been used to print fine patterns on many different substrates. Sintering does not change the shape of the print. This technology has been transferred to Japan for automotive applications. It has also been used to develop touch screens for computers.

A silica based sol modified with an organic resin has been used as a binder for glassfibre mats replacing the phenolic binder. The organic contents are drastically reduced and his has found a commercial application.

Some sol-gel research has been used to develop optical microprocessors. However, a spin off from this has been in diffuser lamps and embossed lenses to focus light on solar cells.

Hybrid sol-gel materials can be very good at adhering to natural fibres adding greater functionality and durability to fabrics.

A sol-gel coating has been used as a deodorising catalyst for a kitchen stove.

An anti-reflective coating for automotive glass has been produced from a combination of TiO_2 and SiO_2 and the angle dependent dip coating method. The result of this coating is that cars do not need to use light-absorbing black dashboards, white can be used instead and there will be no heat gain.

Polycarbonate coated with a sol-gel nanomer and a topcoat have performed better than glass in a standard abrader test. The mechanisms are not understood but the implications for reducing weight in critical applications are substantial.

Anti-graffiti paints have been developed using tailored coatings of fluorinated sol-gels. The application has also been developed for easy clean coatings on ceramic sanitary wear.

There are many new areas in which sol-gel derived products are appearing. Sol-gel is reaching these, not in bulk areas, but in markets with close integration between the academic input and the final product.

PRESENTATIONS

A non-hydrolytic sol-gel route has been developed in recent years as a potential alternative to the conventional hydrolytic route to inorganic oxides. However, there are very few reports of the use of this new approach as a route to organic-inorganic hybrids. Professor John Hay of Surrey University described recent examples of hybrid synthesis by each route and the advantages and disadvantages of each. Examples include hydrolytic synthesis of silica-ionene polymer hybrids and radiochemical sensors and non-hydrolytic synthesis of ormosils, organically-modified aluminosilicates and polymer-silica hybrids. The two sol-gel routes provide complementary approaches such that the selection of the appropriate route for synthesis of a particular hybrid requires consideration of a number of relevant factors.

The development of thin film structures requires detailed characterisation in order to determine their physical and chemical properties. Professor Hamid Kheyrandish, of MATS, Warrington discussed the relative merits and limitations of the various microprobe techniques before outlining the principles of both secondary ion mass spectroscopy (SIMS) and sputtered neutral mass spectrometry (SNMS). He then detailed some recent applications of SIMS and SNMS for analysis of thin oxide film structures including optical coatings and dielectric materials. SNMS has also been used to analyse of a variety of oxides and nitrides, multilayers and graded coatings.

Sol-gel produced mixed oxide materials have been extensively studied using conventional, ex situ structural techniques. Because the structure of these materials is complex and dependent on preparation conditions, there is much to be gained from in situ techniques. The high brightness of synchrotron x-ray sources makes it possible to probe atomic structure on a short timescale, hence, in situ. Professor Robert Newport, University of Kent at Canterbury reported recent results for mixed titania- (and some zirconia-) silica gels and xerogels. The results have been obtained from intrinsically rapid synchrotron x-ray experiments.

Volume manufacturing of ceramic components by a sol-gel route was described by Dr Ron Jones of

LOCAL SECTION CONTACTS
For details of forthcoming local section events in your area, contact the following.
All SGT members and non-members welcome.

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INDIA
– Dr J Mukerji,
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Horizon Technology. Gelation in certain aqueous inorganic sol systems can be brought about by the rapid removal of water during freezing. This technique has been used since the mid-60s to act as a binder system for ceramic powders in the manufacture of refractories. Freeze casting was difficult to adapt to high volume manufacturing of small parts since the casting process required the use of individual moulds for each part which would subsequently be frozen. Then the gelled part could be ejected from the mould after freezing. Development has now led to a modification of this process which allows the wet mix to be formed to the required shape then removed from the mould retaining this cast shape before entering a tunnel freezer. This has cut down mould costs and production time. A particular example is a new range of ceramic gas burners for use in domestic water heating systems.

The influence of sol-gel powders preparation on the ferroelectric properties of ceramic/polymer composites was described by Professor Jose Marat-Mendes, Universidade Nova de Lisboa, Portugal. Ferroelectric powders of lead titanate and calcium modified lead titanate were prepared by the sol-gel process. The gels were dried and heat treated near 500°C using various procedures. The final ceramic powders were mixed with a polymer and films of about 100 μm were obtained by hot pressing. The electroactive properties of the composites were studied.

Both organic and inorganic materials are widely used as coatings for the protection and decoration of steel substrates. Organic-based systems (eg epoxy matrix paints) are used for 'low' temperature applications and inorganic systems (vitreous enamels) for high temperature applications. Organic systems are mechanically flexible and tough, but have poor abrasion and thermal resistance. Conversely, inorganic systems have excellent abrasion resistance, but are brittle and require high processing temperatures. The gulf between the properties offered by these two types of coating has limited the penetration of steel into many applications. Inorganic-organic hybrid materials, which have properties intermediate between those of inorganic glasses and organic polymers, offer a possible solution to this problem. Professor Alan Atkinson, Imperial College of Science, Technology and Medicine described epoxy-based inorganic-organic hybrid coatings on steel substrates by a sol-gel process.

Two coatings with different ratios of organic/inorganic content have been produced on glass and aluminium substrates. The coatings have been cured over a range of times and temperatures. The effects of these variables on the tribological properties of the coatings have been investigated by Dr Alan Taylor, TWI, Cambridge. Hardness was shown to vary with cure conditions and organic content. The effects of the substrate, and the benefit in measuring the hardness of the coating substrate system as opposed to the coating in isolation are demonstrated. Wear of sol-gel coatings were compared to test samples of inorganic (glass) and metallic (aluminium) substrates. Surface modification through coating is seen to change the wear mechanism of glass, and to improve the scratch resistance of aluminium.

Dr John Wright of University of Kent at Canterbury described a range of chemical sensing applications for materials prepared by sol-gel processing methods. In addition to applications of the optical properties of porous transparent glass matrices containing immobilised receptors, the talk covered the study of dimensional changes associated with varying water content, as well as applications of semiconducting nanocrystalline metal oxide materials prepared by the sol-gel process.

There is a considerable interest in the development of UV-patternable sol-gel materials for photonic applications. Upon application of a design mask during UV curing, the preparation of materials with various patterns can be achieved. While most groups have concentrated on devices for telecommunications-related applications, Dr Severine Aubonnet and colleagues at Dublin City University have been developing materials for chemical sensing, eg oxygen. During the preparation of the sol-gel solution, an oxygen sensitive dye is introduced. Variation of the pH of the sol-gel solution enables control of the oxygen sensitivity by adjusting the matrix porosity. By changing the catalyst (basic or acid) as well as its concentration, the structure of the sol-gel film is affected and hence its porosity. Oxygen measurements show that as the pH of the sol-gel solution is increased, the sensitivity is increased. The capability of the material to detect oxygen can be controlled. An oxygen sensor with tailored sensitivity can thus be prepared. Furthermore the UV-photolithographic nature of the material enables the production of sensors in a range of configuration such as arrays and waveguides.

THE ROLE OF VANADIUM IN SOL-GEL DERIVED FILMS FOR BIOMEDICAL PURPOSES.

Titanium alloys are widely used for biomedical applications, one of the most common being Ti-6Al-4V. Dr Philip Harrison and co-workers at The University of Nottingham have studied the effect of vanadia on the structure and morphology of a manufactured titania-based oxide layer. Undoped and vanadia doped titania sols have been successfully prepared using the aqueous sol-gel technique. The oxide layer has been formed by spin coating the sol onto polished commercially pure titanium and calcined at regimes between 333 K and 1273 K. The sol has also been dehydrated to a nano-crystalline powder and calcined at the same temperature regimes. Subsequent analysis has allowed parallels to be made between the behaviour of the nano-crystalline powders and the thin films.

Macroporous ceramics and glasses are used in a large number of applications, such as filters, catalyst supports, insulators, porous implants, because of the unique set of properties they offer. These properties greatly depend on pore morphology, size and distribution, as well as on the matrix characteristics. The manufacture of macro-porous ceramics with good mechanical strength is a challenge and has stimulated the development of several technologies in the last years. Foaming of ceramic fluid systems as a method to generate porosity has proved to be very successful, given that an efficient setting mechanism is also employed. Dr Pilar Sepulveda of Imperial College of Science, Technology and Medicine reviewed some of the techniques that are available today to produce cellular ceramics and glasses, with emphasis on applications in the biomedical field. Examples include porous hydroxyapatite produced by the foaming and gel-casting technique and bioactive foams produced via the sol-gel process. The sol-gel foams consist of a novel development of three-component bioactive glasses, which are known for their ability to chemically bond to bone tissue and stimulate osteogenesis. In addition to this, foaming yields large interconnected pores for tissue in-growth, thus providing the potential for applications as scaffolds for tissue engineering and as bone grafts.

POSTERS

The poster presentations made at the meeting were:

Department of Chemistry, University of Surrey

- Sol-gel matrices doped with 2,5-diphenlyoxazole - development towards a solid tritium sensor by Ian Hamerton, John N Hay, John R Jones and Shui-yu Lu.

University of Kent at Canterbury

- The atomic structure of titania- and zirconia-silica xerogels by Gavin Mountjoy .
- Spectroscopic characterisation of mixed Titania-Silica oxide xerogel catalysts by Mark Holland.
- Sol-gel films for optical detection of copper in water by S Savin and J D Wright.

Imperial College of Science, Technology and Medicine, London

- Processing and characterisation of bioactive gel glasses for tissue engineering by E Fielder and Larry L Hench.
- Preparation of CaO-SiO_2 glasses by the sol-gel method by Priya Saravanapavan and Larry L Hench.

AWE plc, Aldermaston

- Applications of Sol-Gel Technology at AWE by N J Bazin, H A McInnes and J E Andrew.

Nottingham Trent University

- The aqueous chemistry of zinc acetate by Martin Gardener and Carole C Perry.
- Speciation studies of aluminium in the preparation of oxide phases by Kirill Shafran and Carole C Perry.

National Centre for Sensor Research, Dublin City University

- ORMOSIL-based optical sensor for carbon dioxide employing fluorescence resonance energy transfer by C von Bültzingslöwen, A K McEvoy, B D MacCraith and C McDonagh.
- Erbium-doped hybrid planar waveguides: preparation and characterisation by J Xu, S Aubonnet, B D MacCraith and H F Barry.
- Characterisation of sol-gel derived porous films for optical oxygen sensing by P Bowe, B D MacCraith & C McDonagh.

Institute of Polymer Technology and Materials Engineering, Loughborough University

- Improved photoanodes for The dye sensitised solar cell via sol-gel processing routes by L. Weng, S N B Hodgson.
- Sol-gel processing of ceramic and metal foams by A P Baker and S N B Hodgson.

University of Dundee

- Investigation into the physical and catalytic properties of silica zirconia mixed oxide catalysts by D Rosenberg and J A Anderson.

The £100 poster prize was won by Martin Gardener.

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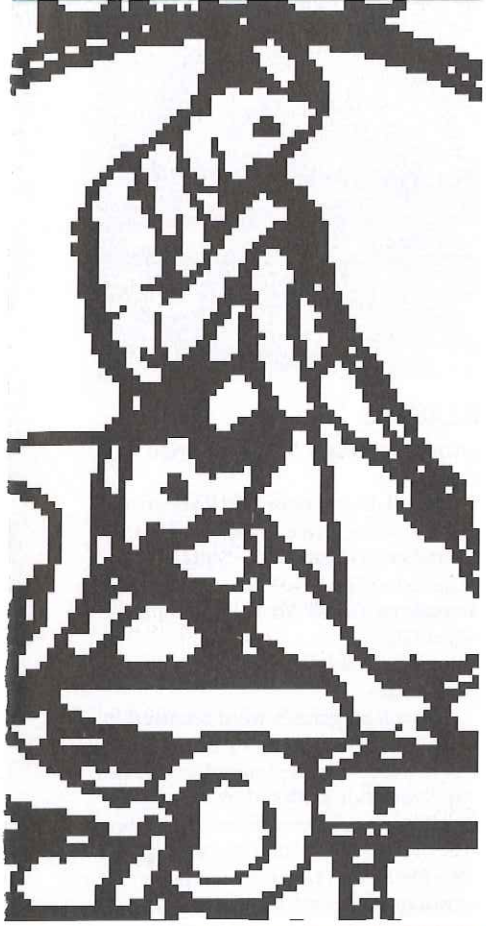

DECEMBER 2000

SGT NEWS

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INSTITUTE OF MATERIALS REGIONAL PROGRAMME

Below is a selection of meetings taken from the Institute of Materials' regional programme for the UK. Like the Society, the Institute has regional organisations that hold their own evening meetings, dinners and works visits. The full listing of events and contact details is available at the Institute's web site: http://www.instmat.co.uk/reg-div/regions/regstaff.htm

16 JANUARY

Nano materials - from sunscreen to solar cells.
Professor Peter Dobson, Engineering Dept, Oxford University
Dept Nuclear Physics, Banbury Road, Oxford. 6.00pm for 6.30pm

7 FEBRUARY

Electrodeposition: Multi-layers and more.
Dr W Schwarzacher, University of Bristol
Room 1.15, 7.30pm.
Contact: Geoff Coates
Tel: 01935 704160

8 FEBRUARY

The BA London Eye.
Dr John Roberts, Institute of Structural Engineers, Joint Meeting with Welding & Joining Society

22 FEBRUARY

Lasers - material analysis.
Dr S Lawson, Applied Photonics
Lever Hall, Corus Ebbw Vale Works 7.15pm.
Contact: Brian Baker
Tel: 01495 334230

27 FEBRUARY

Is fibre optics a new technology?
Prof Animesh Jha, University of Leeds.
Refreshments: Common Room at 5.30pm. Meeting: Lecture Theatre C, Houldsworth Building, Clarendon Road, University of Leeds at 6.15pm

8 MARCH

Optic fibres - The glass ceiling.
Professor Angela Seddon, University of Nottingham
Dept of Materials Engineering, University of Nottingham
6.30pm for 7.00pm

27 MARCH

GUEST NIGHT - With what degree of doctor? - Problems in forensic pathology.
Mike Green, Emeritus Professor of Forensic Pathology, The University of Sheffield. Holiday Inn, Sheffield
5.30pm. for 6.00pm

27 MARCH

Glass cords - not giving an inch!
Dr Chris Stevens, NGF Europe Ltd
Room D14, Manchester Materials Science Centre. 6.45pm

17 MAY

Conversazione
World of Glass, St Helens
Contact: Dr Mark Jackson
Tel: 0151 794 4205

25 JUNE

Materials Recycling.
Napier University (Craiglochart Campus)

NEW SOCIETY PRESIDENT

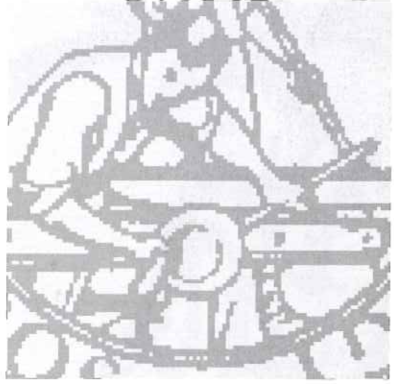
John Henderson is the President of the Society of Glass Technology. As the senior Vice-President with the longest membership he has replaced Richard Bell who has had to stand down.

Restructuring at Brunner Mond led to the departure of Richard Bell. Part of the severance terms set by the company was a break of all contacts with the glass industry including the Society of Glass Technology.

PROFESSOR RONALD WALTER DOUGLAS, HON FSGT

Professor Ron Douglas, Society of Glass Technology President 1963-65 and Honorary Secretary 1948-63, died peacefully at the age of 90 after a short illness in Ottery St Mary Hospital on 14 November 2000. He was Professor of Glass Technology at the University of Sheffield and previously a leading scientist at GEC Wembley Research Laboratories. An active figure in glass science around the world, he was President of the International Commission on Glass 1972-75.

The Society has been fortunate to have the continuity of long serving honorary officers and Professor Douglas was part of this tradition. Following the tradition of Professor W E S Turner, he was Honorary Secretary and editor of the *Journal of the Society of Glass Technology*. As editor he saw through the transition to *Glass Technology* and *Physics and Chemistry of Glasses* in 1960 and groomed his eventual successors, Professor Michael Cable and Professor Alan Owen, respectively.



STANDARD SAMPLES

SGT10 Amber soda-lime-silica container glass (mass%)										
SiO_2	Al_2O_3	Fe_2	CaO	Mg	BaO	Na_2O	K_2O	TiO_2	SO_3	Cr_2O
	3	O_3		O						3
72.8	1.62	0.32	10.6-	1.82	0.02	12.2	0.35	0.09	0.05	0.02
		5								0

SGT11 Green soda-lime-silica container glass (mass%)										
SiO_2	Al_2O	Fe_2	CaO	Mg	BaO	Na_2	K_2O	TiO_2	SO_3	Cr_2
	3	O_3		O		O				O_3
70.7	1.83	0.34	10.3	2.14	0.03	13.6	0.69	0.06	0.06	0.20
		2			1			8		5

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The Analysis & Properties Committee of the Society of Glass Technology has completed its analysis of two new certified reference materials (CRM) for amber and green coloured glasses. The standard samples will become available following approval by Council of the Society of Glass Technology in November 2000. The laboratories involved in the process have all followed ISO guidelines for the production and traceability of analytical data.

PARTICIPATING GROUPS

- Jaspar Analytical & Testing Services.
- London & Scandinavian Metals in association with Appleby Calumite Ltd.
- Sheffield Hallam University, Materials Research Institute.
- Sibelco Minerals and Chemicals (formally Hepworth Minerals & Chemicals Ltd).
- Ceram Research.
- Glass Technology Services.
- Dr Paul Watkins - independent consultant.

INTENDED USE AND STABILITY

The samples are available in the form of glass pieces and also as 40mm diameter discs. They are intended for the verification of analytical methods, such as those used by the participating laboratories, for the calibration of analytical instruments in cases where the calibration of primary substances (pure stoichiometric compounds) is not possible and for establishing secondary reference materials. The solid disc is intended for establishing and checking the calibration of x-ray spectrometers for the analysis of similar materials. The 'as received' surface should be ground and polished.

The traceability of this CRM is ensured by the use of either stoichiometric analytical techniques or methods that are calibrated against pure compounds.

There are also values for ZrO_2 and Mn_3O_4. These add to the existing range of standard sand and glass samples for analysis and calibration purposes.

GLASSES

Standard Glass No. 4. Fluoride Opal Glass
Also available as 6mm thick sheets to special order, price on application.
Standard Glass No. 5. Soda-Lime-Magnesia-Silica Glass
Standard Glass No. 6. Soda-Lime-Silica Glass
Standard Glass No. 7. Soda-Lime-Silica Glass

Two lead glasses were received by the Analysis & Properties Committee but because of time limitations Glass No. 9 was not analysed by all of the collaborating laboratories. This glass has a lower lead oxide content, about 28% PbO, than Glass No. 8 and although it cannot be offered as a certified material, it could be useful as a subsidiary calibration check.
Standard Glass No. 8. Lead oxide-potassium oxide-silica glass (30·59 wt% PbO)
Standard Glass No. 9. Probable composition available.

SANDS

Standard Sand No. 1. 200g packs at £20.00 plus postage (Al_2O_3 0·061, Fe_2O_3 0·014, TiO_2 0·026).
Standard Sand No. 6. (Al_2O_3 0·06, Fe_2O_3 0·032, TiO_2 0·024)
Standard Sand No. 8. (Al_2O_3 2·07, Fe_2O_3 0·26, TiO_2 0·073, K_2O 1·06)
Standard Sand No. 9. (Al_2O_3 1·35, Fe_2O_3 0·103, TiO_2 0·044, K_2O 0·82).

ICG2001

The programme for the International Congress on Glass is available on the Society of Glass Technology's website. As the programme takes shape, the relevant pages will be updated on a regular basis to ensure the most up to date information is available. www.sgt.org/meetings/icg2001.html

IN PRINT

In the December issue of *Glass Technology*, there are 11 peer reviewed papers and posters taken from the 1999 Borates conference. There are also papers from the Glass Opportunities – Heritage, history and conservation meeting held on 7 June at the World of Glass St. Helens.

The December issue of *Physics and Chemistry of Glasses* has the 25 poster papers presented at the Borates conference.

The Proceedings of the Third International Conference on Borate Glasses, Crystals and Melts: Structure and Applications will be available as a separate volume.

Both issues contain abstracts from the latest scientific, technological and business literature and as they are the last issues in the 2000 volume, the annual contents, annual author and subject indexes.

CHANGES TO SGT LOCAL SECTION CONTACTS

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