

Journal of the Institute of Circuit Technology

Vol.7 no.2 Spring 2014 Issue

#### 2013 Events

26t

25th September IeMRC 2013 Conference Wednesdav at Holvwell Park. Loughborough

		iemrc@lboro.ac.uk		
h September <i>Thursday</i>	ICT	<b>Evening Seminar</b> at Newton House Hotel, Hayling Island <u>bill.wilkie@InstCT.org</u>		

- 24th October 9.30 Conformal Coating Reliability Seminar NPL & SMART Group event Thursdav at NPL, Teddington Tony Gordon info@smartgroup.org
- 5th November **ICT Evening Seminar** at Darlington Tuesday St George Hotel, Durham Tees Valley Airport, DL2 1RH 01325 332631 bill.wilkie@InstCT.org

## 2014 Events

4th June

5th June

Thursday

6th March ICT AGM and Winsford Evening Seminar Thursday Chimney House Hotel in Sandbach. bill.wilkie@InstCT.org

14th -17th April ICT Annual Foundation Course at Loughborough University Tuesday -Friday bill.wilkie@InstCT.org

7th - 9th May ECWC13 (13th Electronic Circuits World Wednesday -Convention) at Nuremberg Friday eipc@eipc.org

ICT 40th Anniversary Dinner(see backcover) at Wiltshire Golf Club Wednesday bill.wilkie@InstCT.org

> **ICT Annual Symposium** (see backcover) at Great Western Railway STEAM Museum, Swindon bill.wilkie@InstCT.org

23th September ICT Evening Seminar at Newton House Hotel, Hayling Island Tuesdav bill.wilkie@InstCT.org

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#### Editorial



We always post the amazing write-ups from Pete Starkey in our Journal, but you cannot beat the thrill of actually being there and soaking up the atmosphere. Our last Evening Seminar was held at the Chimney house hotel in Sandbach, supported by CCE Europe, with a fine friendly ambience and constant networking. It was one of those evenings when we could have run the entire event without papers and everyone would have had a good evening – but we had four excellent speakers with good technical papers.

We also had a demonstration. Andy Ballantyne, from Leicester University, was giving a talk on ionic liquids, and took two plastic jars, each with a white powder and poured the contents of the smaller jar into the larger, screwed on the cap and began to shake alongside the microphone. With the audience listening intently, we heard the course rattle of the salts change into the splash of a clear liquid – cue spontaneous applause from the delegates!

This is also a very busy year for the ICT and the Industry. Not only do we have the World Conference in Nuremberg in May, but this is also our 40th Anniversary and we will be having a 40th Anniversary Dinner before our Annual Symposium in June.

We have struck some anniversary pens, so if you can't make the Symposium, drop me a line and we will make sure you get your souvenir pen. Supplies are limited, so let me know before the end of April.

# Bill Wilkie

Council Martin Goosey (*Chairman*), Andy Cobley (*Deputy Chairman*), John Walker (*Secretary*), Chris Wall (*Treasurer*),
 William Wilkie (*Membership Secretary & Events*), Bruce Routledge (*the Journal*), Richard Wood-Roe (*Web Site*),
 Maurice Hubert, Lawson Lightfoot, Tom Parker, Steve Payne, Peter Starkey, Francesca Stern, Bob Willis.

Membership New members notified by the Membership Secretary 10305 Martin Nicholson M.Inst.C.T. 10306 Tony Sauer M.Inst.C.T. 10307 Aleksandra Pinczewski M.Inst.C.T. 10308 Jason McNally M.Inst.C.T. 10309 Lewis Skippings M.Inst.C.T. 10310 Alan Green M.Inst.C.T. 10311 Leigh Allison M.Inst.C.T. Members elected as Hon.Fellow 10056 Walt Custard Hon.F.Inst.C.T. 10206 Happy Holden Hon.F.Inst.C.T.	At the last meeting of the for council	Corrections and Clarifications			
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## Review of papers presented at the Institute of Circuit Technology Winsford Seminar on 6th March 2014 by Pete Starkey

#### Standing Room Only at Winsford.

Spring had almost arrived! The snowdrops and crocuses were in flower and buds on the daffodils, but it was a dull and drizzly day in Cheshire UK for the Institute of Circuit Technology Winsford Seminar. The event attracted a sell-out audience and it really was standing-room-only in the seminar room.

Technical Director Bill Wilkie introduced a programme of four presentations on a diversity of subjects: bonding treatment, soldering, trace metal removal and semiconductor packaging.

Multilayer Copper Bonding



**Paul Sevriens** 

Copper surface preparation to achieve reliable multilayer bonding is a critical operation. In high frequency designs, traditional methods can result in a loss of signal integrity as a consequence of excessive roughening of the copper. Paul Sevriens travelled from MEC Europe in the Netherlands to describe an improved and simplified process of bonding treatment which gave increased peel strength and improved high temperature resistance with minimal surface roughening. A key characteristic of the process was the very low thickness of the copper-organic surface layer, typically 3 nanometres compared with 20 nanometres for competitive processes based on benzotriazole or benzimidazole. This very thin layer did not block the surface texture of the etched copper, and when peel-tested left no residue on the copper. MEC had installations in: Germany, Italy, Switzerland, England, Netherlands, Sweden and USA.

Dr Andrew Ballantyne had presented at previous ICT seminars, on

solvents v Conventional fluxes



**Dr Andrew Ballantvne** 

Soldering Fluxes : Deep eutectic applications of ionic liquids and deep eutectic solvents in metal finishing. The latest line of research at University of Leicester explored the potential benefits of deep eutectic solvents as replacements for conventional soldering fluxes, and some impressive results had been observed. Compared with conventional fluxes which relied on acidic activators, those based on deep eutectic solvents had the advantages of benign chemistry, low toxicity and environmental impact, low cost, low water sensitivity, high metal solubility and less-onerous registration requirements.

After summarising the chemistry and physical characteristics of ionic liquids in general, and entertaining the audience with a live mix-and-shake demonstration of how easily they could be prepared, Dr Ballantyne reviewed the results of solderability testing on copper, brass, nickel, stainless steel and cast iron. Although the scope of soldering technology clearly extended far beyond the field of electronics assembly, specific testing had been carried out on a range of PCB finishes including bare copper, OSP, ENIG, immersion silver, immersion tin, tin-lead HASL and lead-free HASL, on surface-mount pads, through-holes and BGA pads, all with satisfactory results and all samples passed contamination testing after normal cleaning.

In cooperation with Merlin Circuit Technology, deep eutectic solvent formulations had been evaluated as hot-air solder levelling fluxes with remarkable success. Copper was wetted more rapidly than with proprietary HASL fluxes and complete coverage was achieved in a single dip. One very interesting outcome was the ability to solder-level with lead-free solder on PCBs with electroless nickel finish, not previously achievable with conventional fluxes, and this opened up the prospect of a novel solderable finish dubbed HASLEN - Hot Air Solder Levelling on Electroless Nickel.

Copper Recovery using Crap



Professor Martin Goosey

ICT Chairman **Professor Martin Goosey** gave the third presentation, entitled Recovery of Copper from PCB Manufacturing Processes using Crab Shells, describing the TSB-funded STOWURC project whose objective was to develop sustainable materials and processes using waste products from the seafood industry to treat effluent and recover metals from PCB manufacture.

The whole supply chain, from raw material to dissemination of information, was represented in the project consortium. It had been observed that a natural component of the shells of crustaceans, chitin, was able to adsorb heavy metals from dilute solution, and that a simple chemical modification of chitin, by alkaline deacetylation to form chitosan, significantly improved the efficiency of adsorption. For example, one gram of chitosan was capable of adsorbing 250 milligrams of copper.

Professor Goosey summarised the results of initial experiments. The effects on adsorption rates of parameters such as temperature, pH, initial concentration, rate of mixing, specific metal ions had been studied, over a range of chitosan parameters including the amount of adsorbant, the degree of deacetylation and the particle size. It was possible to reduce copper concentration in effluent to the 0.1ppm level.

Having captured the metal, it was possible to desorb it with sulphuric acid and recover it by straightforward electroplating. A key issue was the residual adsorption efficiency of the chitosan after stripping of copper; there was a tendency for absorption ability to decrease with each cycle, and conditions were being optimised to minimise the effect.

Chitosan had the ability to adsorb a wide range of metals found in PCB effluent, nickel for example, and there were many applications in the general metal finishing industry. It also provided the opportunity to recover endangered or rare metals from low concentration solutions. Professor Goosey mentioned platinum, palladium, rhodium, osmium, iridium, mercury and gold as examples, but made it clear that each metal would need an optimised combination of process parameters and conditions.

# Applications of PCBs in miniature.



**Steve Payne** 

Past Chairman of ICT **Steve Payne** gave a thought-provoking overview of laminate-based semiconductor packaging and system-in-package technology – effectively an insight into some applications of PCBs in miniature.

With some silicon dies now having as many as 5,500 input-output connections, wire bonding was a not a practicable interconnection option and a flip-chip approach was preferred, with a laminate substrate within the package. This was compatible with BGA-style area-array packaging and gave the electrical benefit of very short interconnects, but mechanically the interconnections were stiff and rigid. Therefore for high reliability applications there was a critical need to match thermal expansion properties of silicon and substrate, and to aim for a low modulus to aid mechanical compliance. Very thin substrates were preferred, to minimise z-axis stresses, and materials without woven-glass reinforcement were favoured, which gave the additional benefits of more efficient laser drilling and the absence of CAF effects.

Typical substrate design rules were 25-35 micron track and gap, to enable conductor routing from flip-chip solder bumps at pitches trending towards 100 microns, 50 micron via holes and 100 micron capture pads, with a construction based on build-up technology having dielectric thicknesses of 40-50 microns. Semiconductor packaging and system-inpackage could use the same substrate technology, and there had been heavy investment in Asia to satisfy high volume requirements for smart phones and tablets. The global market for semiconductor packaging materials was expected to grow to over \$25 billion by 2015, of which laminate substrates represented the largest market segment, with a growth forecast of 8 percent over the next five years. Although the manufacture of semiconductor substrates was technically challenging, they were in fact miniature PCBs and Payne indicated that there was a significant and growing requirement for high reliability medium volume substrates in Europe which represented a substantial market opportunity, with typical selling prices in the range £20 - £40 per square inch.

Bill Wilkie brought proceedings to a close, acknowledging the generous support of CC Electronics, and reporting a record number of advance registrations for the ICT/NUKCG Foundation Course in April and a high level of interest in the ICT Annual Symposium and 40th anniversary celebration dinner in June (full details at <u>www.instct.org</u>).

The Institute of Circuit Technology continues to gain momentum, with membership now well into the 300s, and the Winsford Seminar was another excellent opportunity for members to share knowledge and to network with their peers in the UK PCB industry.

> Pete Starkey March 2014

## Sustainable Solder Flux from Novel Ionic Liquid Solvents: Greener, Cleaner, Cheaper

by Andrew D. Ballantyne<sup>1</sup>, Christopher J. Zaleski<sup>1</sup> Robert C. H. Harris<sup>1</sup> Dennis Price,<sup>2</sup> and Karl S. Ryder <sup>1</sup>

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Andrew D. Ballantyne



Christopher J. Zaleski



Robert C. H. Harris



Dennis Price



Karl S. Ryder

#### Abstract

Soldering technology is of key importance for the PCB and electronics industries. In order to achieve a suitable solder bond a flux is required to remove surface oxide, maximising the wetting interaction between the substrate and the solder. Conventional fluxes are composed of rosin or inorganic salts, both of which have their associated hazards and limitations.

Here we report the use of deep eutectic solvents (DESs) as a novel solder flux. These liquids are composed of environmentally benign, bulk commodity chemicals yet due to their unique properties are excellent at solvating a wide variety of metal species.

These liquids show excellent substrate wetting rates for a wide variety of PCB surface finishes including bare copper and electroless nickel immersion gold (ENIG) as measured by Tri-Moore and solder wetting balance testing. Solder wetting balance testing shows efficient wetting of bare Cu is achieved using a DES flux much faster than that of the standard rosin based flux Actiec5. Cross sectional analysis shows that the resulting solder bonds are pore free. The bond to copper shows intermetallic formation in the range of 2-3 µm while ENIG has limited intermetallic formation.

## Introduction

Soldering is a key technology in the electronics industry used in both PCB manufacture and surface mounting of components onto PCBs.<sup>1</sup> Hot air solder levelling (HASL) is a widely used PCB surface protection method, protecting the underlying Cu substrate from oxidation by covering it in a thin film of solder. In addition the majority of components in an electronic assembly are fixed to a PCB by mounting to the surface through solder bonds as they are highly conducting while also having high tensile strength. When soldering it is essential to use a solder flux which serves the purpose of cleaning and removal of oxide from the substrate and solder surface. Typically these are either rosin or inorganic salt based fluxes, each of which has their own respective drawbacks. Rosin is an irritant and skin sensitizer which is expensive and has problems with batch consistency due to its supply from natural products.<sup>3</sup> In addition, strong acids and activators are often added to rosin to increase the activity of a flux, introducing further associated hazards. Inorganic salts are often irritants as well as often possessing more dangerous hazards such as high toxicity<sup>4</sup>

To that end we have looked at the use of deep eutectic solvents (DESs) as a novel solder flux. DESs are low melting liquids that are formed by the interaction of an organic salt with a donor molecule.The donor molecule interacts with one or both of the ions, perturbing the intermolecular structure causing a dramatic lowering of the melting point.<sup>5</sup> They are commonly composed of bulk, commodity chemicals with little or no hazards and low cost. Importantly, for the application of a solder flux, they have good solubility of metal salts<sup>6</sup> and as such have been used in a variety of metal processing applications such as electropolishing<sup>7</sup>, electroplating<sup>8 - 10</sup>, immersion coatings<sup>1</sup> - 12, and metal recycling<sup>13</sup>. Here we detail some of our efforts studying the use of these novel liquids as a solder flux for applications in the electronics industry.

### Experimental

Samples for Tri-Moore wetting trials were prepared by cutting small sections (approximately 20x60 mm) from the Atotech SFTB1 Rev1 test PCB board which were treated with a variety of surface finishes including bare copper, electroless nickel immersion gold (ENIG), immersion silver, immersion tin, lead free HASL and lead HASL coatings. These boards were produced by Merlin PCB. Cross section microscope images were captured on a Zeta Instruments Zeta 2000 optical profiler using the inbuilt Zeta3D software version 1.8.5

SEM and EDX line analysis were recorded on a Phillips XL-30 Field Emission Gun scanning electron microscope (FEG SEM) equipped with a Bruker AXS XFlash 4010 EDS detector operating at 25 kV. Secondary electron imaging (SEI) was performed with a working distance of ca. 5 mm and accelerating voltage of ca. 20 kV.

Solder wetting balance measurements were recorded on a Gen3 Systems Must System 2 solder wetting balance machine using an untreated bare Cu wire suspended over a bath of SAC 305 lead free solder at a temperature of 260 °C. The wire was immersed to a depth of 5 mm in the solder bath and the resulting force exerted on the Cu wire measured for a period of 20 s.

## **Results and Discussion**

Initial investigations of the efficacy of these novel liquids as a solder flux were carried out via Tri-Moore testing. This technique involves passing a pre-fluxed PCB coupon through a bath of molten solder with a qualitative understanding of the wetting ability determined by visual and microscopic inspection of the solder pads as well as cross sectioning analysis. This method enables the ability to test a large number of samples, enabling the rapid testing of the solderability of a wide variety of PCB surface finishes. In this case we have studied bare copper, electroless nickel immersion gold (ENIG), immersion tin, immersion silver, lead free hot air solder levelled (HASL) PCBs and leaded HASL.

**Figure 1a** shows the top of a standard PCB coupon which did not have a protective finish and coated in a DES flux which has been passed through the Tri-Moore tester. All exposed pads show uniform surface coverage with solder suggesting that good wetting occurs across the entirety of the solder pads.

**Figure 1b** shows a cross section of one of the soldered pads shown in **Figure 1a** where the copper can be seen in the bottom of the image and the solder at the top. At the interface two distinct layers can be seen which relate to the formation of intermetallics of Cu and Sn. Copper/tin solder joints are known to

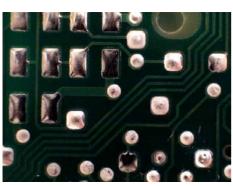


Figure 1a



Figure 1b

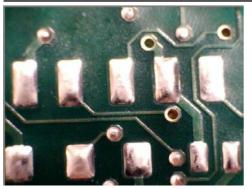


Figure 1c

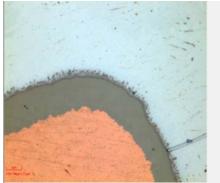


Figure 1d

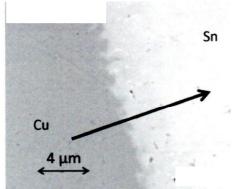
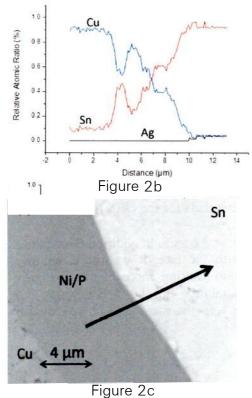


Figure 2a



form two intermetallic phases corresponding to two different atomic ratios; Cu<sub>3</sub> and Cu<sub>65</sub>.<sup>14</sup> it is likely that the intermetallic layer close to the Cu is Cu<sub>3</sub> and the darker layer is Cu<sub>65</sub>.

**Figure 1c** shows a PCB prepared in an identical manner to that of **Figure 1a** except that in this case the PCB coupon had been protected by the ENIG surface finish. Excellent wetting of the solder is observed with all of the pads showing total surface coverage of the ENIG coated pads. **Figure 1d** shows the resulting cross section of one of these pads with the solder bonded to the surface of the Ni/P which acts as a barrier between the copper and the solder. A thin intermetallic layer is visible which may be either(Ni,Cu}<sub>3</sub> Sn<sub>4</sub> or (Ni,Cu)<sub>6</sub> Sn<sub>5</sub>

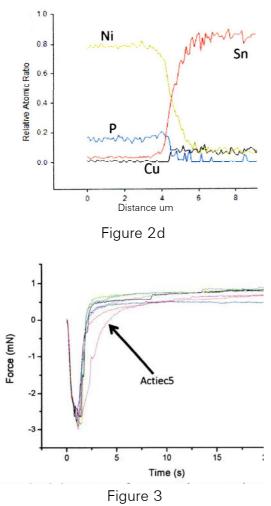
**Figure 1**: Microscope images showing surface of the pads and cross section for (a & b) copper finished PCB coupons and (c & d) ENIG finished PCB coupons which were fluxed with a DES solder flux and passed over a SN100C solder bath at 260 °C for a residence time of 6 s.

In order to obtain further understanding of the interfacial structure of these solder bonds SEM images and EDX line scan analysis were recorded for the Cu and ENIG coated samples. **Figure 2a** shows an SEM of the cross section of a copper soldered PCB coupon prepared in Tri-Moore testing using the DES flux. An intermetallic layer can be observed at the interface between the Cu substrate and Sn solder as consistent with the results shown in **Figure 1b**. EDX line analysis, shown in **Figure 2b**, measures the elemental composition of the substrate across a line. In this case a line was chosen across the interface between Cu and Sn highlighting differing intermetallic regions. This can be identified through changes in the relative atomic ratio of the Cu and Sn as is consistent with the growth of Cu/Sn intermetallics seen in conventional systems.<sup>14</sup>

Figure 2c shows an SEM of the cross section of an ENIG coated, soldered, PCB coupon prepared in Tri-Moore testing using the DES flux. In this case two distinct interfacial layers can be identified. A Cu layer can be seen in the bottom left corner of the image, then an electroless Ni/P barrier layer, then the layer of solder. Again this shows a uniform, pore free bonding to the nickel. In this case no intermetallic growth can be seen and this is consistent with the EDX line analysis shown in Figure 2d. Ni/Sn intermetallics are known to grow more slowly than their Cu/Sn counterparts and thus could be less apparent via this technique. The Tri-Moore testing provides qualitative evidence regarding the surface wetting properties of the DES solder flux showing that excellent solder uptake is seen for all surface finishes of the PCBs coupons tested. Solder wetting balance measurements provide a more quantitative analysis method, enabling direct comparison of wetting rates with common commercial fluxes.

**Figure 2**: SEM and EDX line scan compositional analysis for cross sections of (a & b) copper finished PCB coupons and (c & d) ENIG finished PCB coupons which were fluxed with a DES solder flux and passed over a SN100C solder bath at 260 °C for a residence time of 6 s.

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In this case bare Cu wire was used as the substrate. The wire was cleaned with IPA prior to experiment but no further degreasing or etching of the wire was carried out. As such it is likely that there was still an appreciable amount of surface oxide present. The wire was dipped in the DES flux and then suspended on the wetting balance followed by immersion to a depth of 5 mm in a SAC 305 solder bath and the resulting force exerted on the balance measured.

**Figure 3** shows the solder wetting traces for 5 repeated DES flux and compared with that of the standard flux Actiec5. This is a mildly activated rosin based flux, commonly used in solder wetting balance testing<sup>15</sup> For all traces an initial rapid decrease in force measurement to a value close to -3 mN is seen due to the buoyancy effect exerted on the sample caused by the high density of the solder which the Cu wire has been immersed in. Once solder wetting starts to be observed on the substrate the force starts to increase due to the formation of a solder bond. The rate at which this increase occurs provides direct quantitative measurement of the rate of substrate wetting by the solder.

**Figure 3**: Solder wetting balance traces for untreated copper wire coated with DES solder flux and immersed in SAC 305 at 250 °C. The black line labelled is for a standard rosin based flux, Actiec5, and the rest of the traces are repeated measurements for the DES flux.

The DES solder flux visibly wets the Cu substrate faster than Actiec5. In addition the wetting profiles of the DES flux are also very reproducible with the rate of wetting remaining consistent. Due to the high buoyancy the time until zero wetting force ( $T_{zwf}$ ) has been calculated as a qualitative measurement for comparing wettability. For the DES flux the  $T_{zwf}$  is 2.1 s compared to 4.2 s for Actiec5. Thus, the DES flux wets the surface of a bare Cu wire twice as fast as that of the activated flux Actiec5.

#### Conclusions

Soldering technology is very important to the electronics and PCB industries. However, solder fluxes can be very expensive and the chemicals contained in the flux can have significant hazards associated with them. DESs are an exciting alternative flux system to the currently used commercial products due to their composition of cheap commodity chemicals, ease of production and environmentally benign nature.

Tri-Moore testing, sectioning and analysis has shown that solder bond are rapidly form onto a wide variety of PCB surface finishes including Cu and ENIG while solder wetting balance studies have shown that the DES flux enables substrate wetting at twice the rate of that of the standard test flux Actiec5.

#### Acknowledgements

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## **Corporate Members of The Institute of Circuit Technology**

April 2014

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**2014 SMART Group and NPL** three-day event **Hands-on Conformal Coating & Cleaning Experience** from 8-10 April at NEC, Birmingham.

The Conformal Coating & Cleaning Experience will provide an opportunity to spend up to three days looking at all things coating- and cleaning-related. Plan to attend the free seminars, which will take place during the NEW conference and exhibition. For information, visit three-day **Hands-on Conformal Coating & Cleaning Experience** from 8-10 April at NEC, Birmingham.

www.smartgroup.org

#### The Membership Secretary's notes - April 2014

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## Institute of Circuit Technology

#### **40th Anniversary Dinner**

at the Wiltshire Golf and Country Club (the Hotel have given us a rate of £95 for Dinner Bed and Breakfast) on

Wednesday 4th June 2014

(the night before the Annual Symposium, which is 8 miles away)

We will have a Memory Board/ Table for mementoes of those early days, including Vol.1 Issue 1 of Circuit World, kindly lent by Steve Jones and if there is anything you would like to be included, please get in touch.

We have had a good response from those wishing to take part in the anniversary dinner, but please contact Bill Willkie if you want to register for either the Symposium and/or the dinner.

bill.wilkie@InstCT.org

# Institute of Circuit Technology 40th Annual Symposium

The 40th Annual Symposium will be held at the Great Western Railway STEAM Museum at Swindon

Event Info Thursday June 5th 2014 Registration at 09:30 Symposium begins at 10:00 Great Western Railway STEAM Museum Swindon



**40th Annual Symposium** 

IT IS PLANNED TO MAKE THIS A VERY SPECIAL OCCASION

Full Agenda to Follow on our Web Site Members and non-members - £85 Tabletops - £55