

Insight

A regular update to the semiconductor industry



April 2008

The new face of Epichem™ Group News

SAFC® Expansion in Far East

Late 2007 saw SAFC Hitech™ take another major step toward expanding the company's manufacturing footprint in the important Asia market with the signing of a Memorandum of Understanding (MoU) with the Gyeonggi Province of South Korea. The MoU, signed by SAFC Hitech President Barry Leese and Moon-Soo Kim, Governor of Gyeonggi Province in a ceremony held in New York City, signals the company's intentions of exploring investment opportunities that may result in a facility being built in the province for the supply and manufacture of electronic materials.

The MoU is part of an ongoing program of regional investment by Sigma-Aldrich® Corporation and follows the September 2007 announcement of investment in a facility at the Wuxi-New District Park (WND) located northwest of Shanghai, China.

"South Korea is an important market for SAFC Hitech with a very strong domestic electronics business that represents an excellent opportunity for us to supply electronic chemicals for use in the semiconductor market," commented Barry Leese. "Gyeonggi Province, in particular, has been extremely proactive and supportive of our intentions. We look forward to exploring a long and mutually beneficial relationship with the region."

SAFC President Frank Wicks added, "As part of the strategic plan SAFC laid out in 2005, we expect to increase our investment in a number of regions, including the Asia-Pacific markets. By exploring investment avenues with South Korea, we have the opportunity to accelerate sales growth in the Asia-Pacific region while, at the same time, increasing our capacity and allowing us to better serve our local and global SAFC Hitech customer base."



SAFC Hitech President, Barry Leese and Moon-Soo Kim, Governor of Gyeonggi Province, South Korea at the MoU signing ceremony in New York

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The current name for Gyeonggi Province was first used in the 11th century (1026). The Gyeonggi area of the Korean peninsula has served as the capital region for more than a thousand years. It now plays a vital role in the politics, economy and culture of Korea and of course contains the key city of Seoul. Its 24 million inhabitants account for 23% of the GDRP helping Korea to its position as the 11th largest trading nation in the world.



Moon-Soo Kim
Governor of
Gyeonggi Province



Barry Leese
SAFC Hitech President

For further information about Gyeonggi Province visit <http://english.gg.go.kr/>

SAFC Hitech New Image Unveiled in 2007



The incorporation of Epichem into the SAFC Hitech business sector saw a rebranding during the year to spread the word of this exciting new combination of expertise. At a number of trade shows the stunning new booth designs were certainly noticed by customers. This distinctive marketing will be continued forward to raise awareness of our new visual identity to ensure all customers know where we are and what we can offer.

Conference Representation for 2008

SAFC Hitech personnel will again be at all the key semiconductor conferences and workshops around the world to network with customers and the scientific community. As markets move forward we ensure state of the art products and services are available to groups across a wide spectrum of application areas to offer a competitive advantage for the end user.

We are pleased to invite you to come and talk to a representative at one or more of the events listed below planned for 2008. Come for a chat about sales enquiries, technical support or the weather back home. Everyone is welcome.

| DATE | CONFERENCE | LOCATION |
|-----------------|--|-----------------------|
| 2-4 March | 2008 Key Conference on Compound Semiconductors | Key West, US |
| 18-20 March | Semicon China | Shanghai, China |
| 24-28 March | MRS Spring Meeting | San Francisco, US |
| 2-4 April | 4th Photovoltaic Science Applications & Technology (PVSAT-4) | Bath, UK |
| 6-10 April | ACS 235th National Meeting & Exposition | New Orleans, US |
| 10 April | BCGA (British Compressed Gases Association) | Leeds, UK |
| 14-17 April | CS Mantech | Chicago, US |
| 5-7 May | Semicon Singapore | Singapore |
| 5-7 May | ASMC - Advanced Equipment and Materials Processes | Boston, US |
| 7-8 May | Blue Conference - SSLS Forum | Hsinchu, Taiwan |
| 8-9 May | S2K 2008 | Cardiff, UK |
| 18-23 May | 213th ECS Meeting | Phoenix, US |
| 18-23 May | SID 2008 (Society for Information Display) | Los Angeles, US |
| 18-21 May | WOCSDICE 2008 | Leuven, Belgium |
| 25-29 May | IPRM (Indium Phosphide & Related Materials) | Versailles, France |
| 26-30 May | EMRS Spring Meeting | Strasbourg, France |
| 1-5 June | NSTI Nanotech | Boston, US |
| 1-6 June | ICMOVPE-XIV | Metz, France |
| 4-5 June | EuroLED | Birmingham, UK |
| 9-12 June | ISIF (International Symposium on Integrated Ferroelectrics) | Singapore |
| 15-19 June | 7th International Conference on Coatings on Glass and Plastics | Eindhoven, Holland |
| 25-27 June | EMC (Electronic Materials Conference) | Santa Barbara, US |
| 29 June-2 July | ALD 2008 | Bruges, Belgium |
| 15-17 July | Semicon West | San Francisco, US |
| 17-21 August | ACS 236th National Meeting & Exposition | Philadelphia, US |
| 1-4 September | European Photovoltaics | Valencia, Spain |
| 9-11 September | Semicon Taiwan | Taipei, Taiwan |
| 16-20 September | E-MRS Fall Meeting | Warsaw, Poland |
| 7-9 October | Semicon Europa | Stuttgart, Germany |
| 1-5 December | MRS Fall Meeting | Boston, US |
| 3-5 December | Semicon Japan | Tokyo, Japan |
| December | DGKK | Braunschweig, Germany |

Improved Ruthenium Sources for III-V and Si Applications

In recent years Ruthenium has become of increasing interest as an element for inclusion into semiconductor devices. Whether it be for a metal electrode or as a dopant in a compound semiconductor the limited performance of standard Ru precursors has held back the integration of this versatile material. A number of linked research projects have been undertaken at SAFC to expand the approaches available for deposition of high quality Ru based films and make available a wider range of products capable of increasing device properties.

The potential use of Ruthenium to form InP based semi insulating films capable of acting as current blocking layers has been proposed to avoid intermixing of dopants and the consequent degradation of device performance. Growth of Ru doped InP has been demonstrated previously using the conventional precursor Bis(dimethylpentadienyl) Ruthenium [DMRU] [1], however for atmospheric pressure MOVPE its physical characteristics lead to limitations in its usefulness. To overcome the issues of low volatility, incorporation efficiency and stability under a hydrogen ambient a range of alternative Ru precursors were studied.

Previously reported work had indicated that substituted cyclopentadienyl compounds of the general formula $(RCp)_2Ru$ offered high potential [2], hence a more complete series of samples were prepared and characterized by a range of techniques to fully establish their physical properties. Vapour pressure measurement was employed to compare volatility whilst Thermogravimetric Analysis (TGA) and Proton Nuclear Magnetic Resonance (NMR) were used to assess thermal stability under a variety of conditions. In addition, TGA and NMR were performed under different atmospheres (N_2 and H_2) to investigate the impact on stability by changing carrier gas.

The vapour pressure results are shown in Figure 1 and confirm the suitability of $(^iPrCp)_2Ru$ and $(^iBuCp)_2Ru$. The TGA comparisons under N_2 indicate good stability whilst those taken under H_2 gave slightly different results as shown in Figure 2 and therefore NMR trials were undertaken to provide further information and prove stability of the chosen source $(^iPrCp)_2Ru$ in this environment (see Figure 3).

Having chosen a precursor physically better suited to the MOVPE process, growth trials were initiated to assess Ru incorporation compared with the conventional DMRU source. In simple films a significant improvement in dopant incorporation and control was observed. To demonstrate the effectiveness of the new sources in actual devices several test structures were fabricated and fully processed with excellent results achieved. [3] Figure 4 shows the stabilisation of dopant diffusion when Ru is employed and Figure 5 illustrates good forward and reverse bias blocking performance.

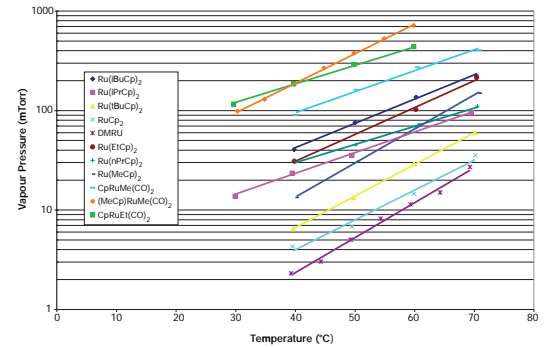


Figure 1: Vapour pressure data for $(RCp)_2Ru$ and $(RCp)Ru(CO)_2R$ compounds

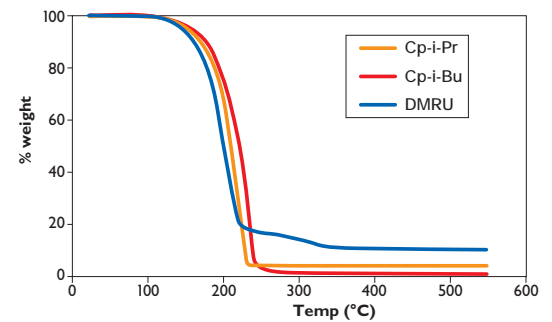


Figure 2: TGA data for $(^iPrCp)_2Ru$, $(^iBuCp)_2Ru$ and $(CH_2C(Me)CHC(Me)CH_2)_2Ru$ under hydrogen

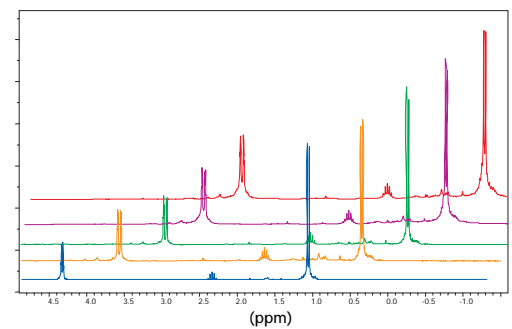


Figure 3: 1H NMR data for $(^iPrCp)_2Ru$ at $50^\circ C$ under hydrogen

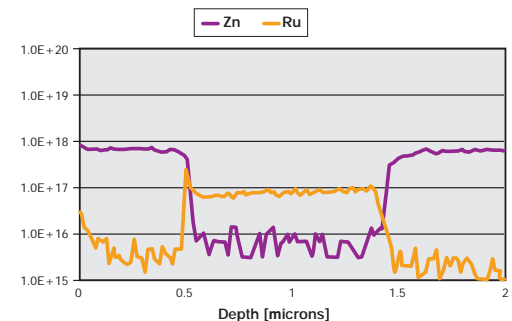


Figure 4: SIMS profile for a p-InP: Ru-InP:p-InP structure

Meanwhile in the Si industry the ongoing requirements of Moore's Law have resulted in a demand for metal gates with Ruthenium high on the list of proposed solutions. The challenges for full integration are huge and traditional sources such as $\text{Ru}_3(\text{CO})_{12}$ have been proven to be limited in performance.

Previously reported work with a number of elements (ie Hf, Zr, Ti) [4,5] has indicated that the use of a complex mixed ligand molecule can provide improved characteristics, hence a series of Ruthenium derivatives were prepared and studied. In detail the ligands employed were substituted cyclopentadienyl, carbonyl and alkyl groups (see Figure 6). [6] The range of characterisation techniques outlined above were applied to these precursors to identify the most promising candidates.

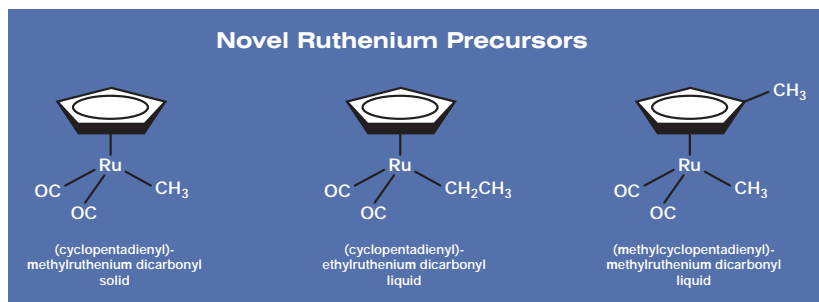


Figure 6: Schematic representations of $(\text{RCp})\text{Ru}(\text{CO})_2\text{R}$ precursors

Vapour pressure (see Figure 1) and TGA results highlight $(\text{MeCp})\text{Ru}(\text{CO})_2\text{Me}$ and $\text{CpRu}(\text{CO})_2\text{Et}$ as extremely high volatility liquid precursors and worthy of further study in an ALD process. Trials were undertaken on an SAFC tool and controlled depositions have been demonstrated to give excellent morphology and roughness results for layers on both Si and TaN surfaces (see Figures 7 and 8) along with good resistivity values (22-25 micro-ohm cm). The presence of the alkyl group bonded to the Ru centre is clearly shown to be beneficial to the ALD process.

It is clear that a detailed understanding of chemistry is key to achieving the ongoing precursor improvements necessary to support customer demands in a wide range of different fields. The Ru work presented demonstrates the complexities involved in identifying and testing sources of the same element across varied material systems using different deposition methods. The demonstration of controlled Ru doping and matrix deposition is testimony to the in depth knowledge and expertise of the SAFC research team and their ability to provide advanced source materials capable of enabling novel technologies and device structures for enhanced performance capabilities.

References

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- [4] J Niinisto et al, Chem. Mat., 19, 3319 (2007)
- [5] SAFC patent WO2006131751A1
- [6] SAFC patents pending

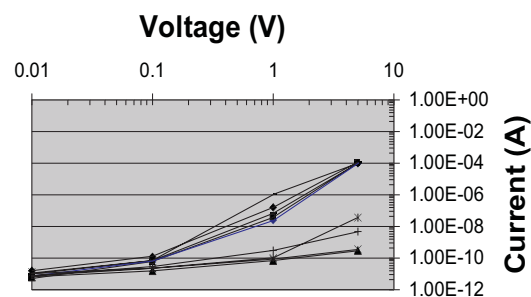


Figure 5: IV data for multiple devices in forward bias

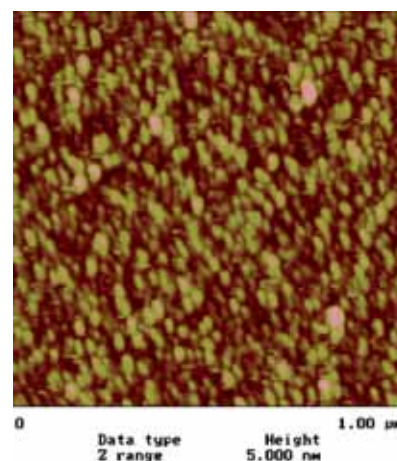


Figure 7: AFM of Ru layer on Si

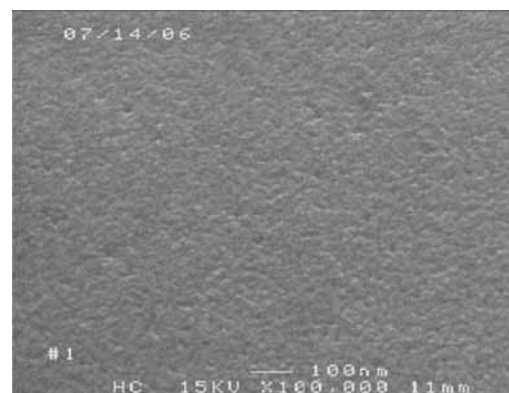


Figure 8: SEM of Ru layer on TaN

Customer Satisfaction Survey Results

SAFC Hitech strives to achieve the highest standards of customer service and to assess performance in this area conducts an annual survey to allow customer perceptions to be evaluated. Feedback is highly valued and numerous improvement projects have been implemented based on customer input. The objective is always to maintain our highly valued relationship with product users and ensure all areas of the SAFC Hitech/customer interface remain at the highest level.

SAFC Hitech would like to thank all of the respondees for their time and comments which will be used to improve our performance. A prize draw was held for all completed forms and this year's winner of a state of the art iPod is Erwin Kessels from Technical University Eindhoven in the Netherlands.

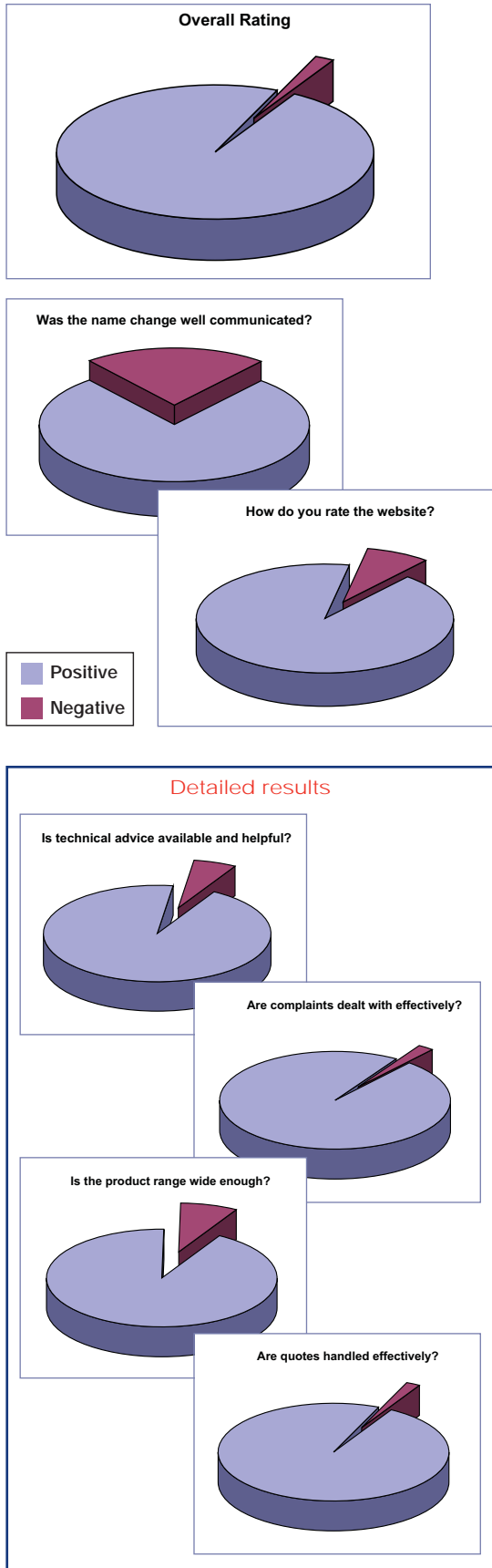


The findings of the survey allow corrective actions to be established to counter negative experiences as well as ensuring newly introduced protocols are successful. Positive feedback demonstrates that dealing with SAFC Hitech has met or surpassed customer expectations.

Overall the majority of responses were positive indicating an excellent perception of SAFC Hitech as a precursor supplier. This year the main topics of concern were the communication of our name change from Epichem and the functionality of the newly launched website. In particular the technical support through this medium was marked down. These issues are being addressed to clarify company changes and improve accessibility to detailed product data on the website.

As in previous years all customers who returned negative comments will be contacted directly by their account manager to discuss these concerns and determine a strategic plan to resolve any difficulties. A key target for 2008 will be to maintain effective communications between all our customer and SAFC Hitech contacts in Sales, Customer Services, Technical Services and beyond. Furthermore a new look catalogue is available to highlight the product offerings across the different application fields. Coupled with the planned improvements to the website we hope to demonstrate the extensive options available to all customers to ensure purchase of the precursors best suited to their processes in the most efficient manner.

Whilst customers whose complaints are satisfactorily resolved often become more loyal than customers who were never dissatisfied it is SAFC Hitech's goal to reduce the number of complaints or concerns expressed by its customers and to increase the range and quality of service offered.



Successful Upscaling of TMI Delivery Technologies

The requirement to provide increasing volumes of Me_3In to production reactors in a reliable, consistent fashion has seen numerous bubbler designs evaluated. The most successful trials on small scale have been reported previously and involved novel interior design features (See Figure 1 and ref [1,2]). This approach has now been successfully expanded to larger scale with customer results showing good control of dosimetry for 900g fill levels through to end of life. The stability of output for run to run is exceptional even under extreme conditions as shown in Figure 2 below obtained on SAFC Hitech's own transport assessment equipment.

References

- [1] Transport properties for different bubbler designs, M S Ravetz et al, Proc. IPRM 2001, p310
- [2] TMI transport studies, effect of different bubbler designs, L M Smith et al, J. Crystal Growth, 272 (2004) p37

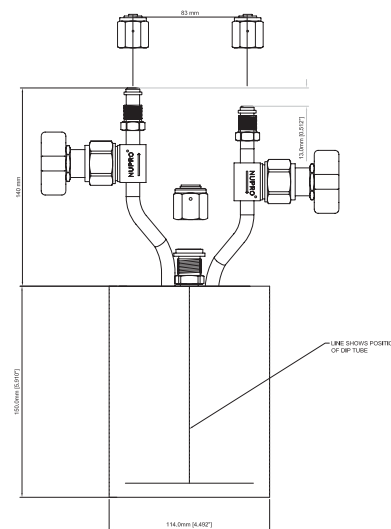


Figure 1: Bubbler schematic

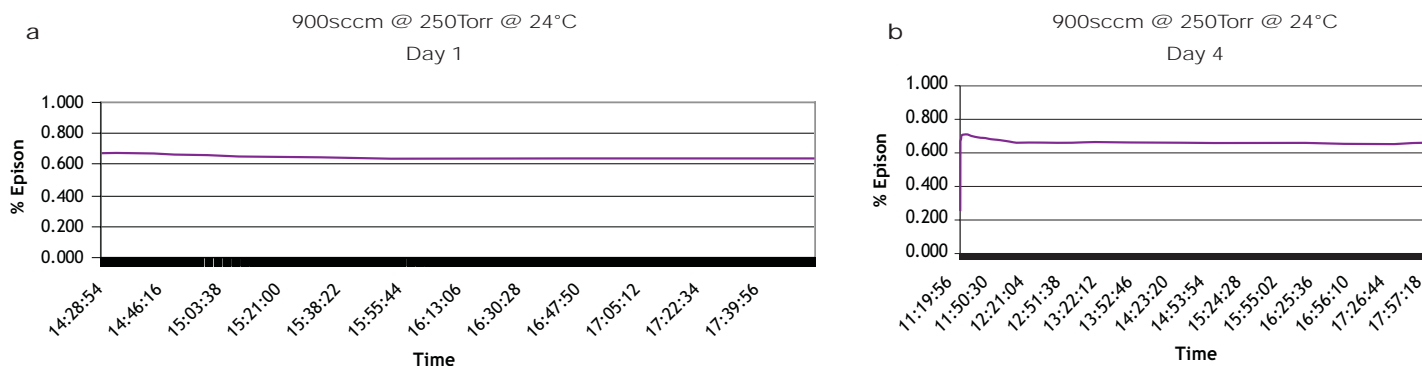


Figure 2: Output flux readings at a) beginning of life and b) end of life for 900g fill bubbler under extreme usage conditions

For more details of advanced TMI bubblers please contact your local SAFC Hitech representative.

CIA Safety Award

The SAFC Hitech site in Bromborough, UK has been recognised by the Chemical Industries Association (CIA) for its safe working achievements over recent years. In detail the threshold target for the mean lost time accident rate of 0.20 per 100,000 hours worked was surpassed and a value of 0.18 per 100,000 hours attained. This level of achievement reflects the high regard SAFC Hitech places on safety and the rigorous policies implemented throughout the organisation to maintain a healthy environment for all of our workforce when producing extremely hazardous chemicals.



Deposition of SnO₂ Thin Films by MOPACVD at Room Temperature

SnO₂ films are among the most promising materials for gas sensors, heat mirrors, photovoltaic solar energy conversion devices and transparent electrodes due to their good thermal conductivity and visible transparency [1]. For future applications the desire is to be able to deposit such thin films on to a wide variety of substrates including plastics and so reduction of the growth temperature is a key area to address. The use of plasmas to reduce process temperatures is well known with the reaction activation energy supplied by high energy electrons rather than the heat supplied in a normal CVD technique. This is clearly demonstrated by the requirement for temperatures between 320°C and 470°C to deposit SnO₂ films by CVD [2] and the ability to deposit coatings at less than 100°C using plasma assisted techniques. [3]

All of the coatings produced in this work have been deposited at room temperature using Metalorganic Plasma Assisted Chemical Vapour Deposition (MOPACVD). It had been suggested that the refractive index and coating transparency may be engineered to have specific characteristics by adjusting the oxygen partial pressure during deposition [4] and so this parameter was studied intensely.

Experimental

Tetraethyltin (TESn) was chosen as the precursor because its vapour pressure of 0.36Torr at room temperature was considered to be well suited to allow control of the vapour flow during experiments. There was a concern that the higher vapour pressure source tetramethyltin (TMSn) would lead to unwanted, excess vapour to be admitted into the reactor. The TESn vessel was maintained at a constant temperature of 30°C and vapours delivered into the chamber using a vapour draw system via a metering valve. This provided an effective, cost efficient alternative to using a vapour source mass flow controller. A standard mass flow controller was used to regulate the flow of oxygen into the chamber. Nitrogen was used to purge the system once the coating had been deposited.

Tin oxide coatings were deposited onto glass (BK7) and Ge substrates from the decomposition of TESn within a plasma having RF power of 50W.

The complete reaction leading to the formation of SnO₂ thin films can be written as :-



The water vapour and carbon dioxide are expelled through the rotary pump exhaust, leaving a coating of tin oxide on the substrate. An important factor in the completion of this reaction is the provision of sufficient oxygen to the chamber to avoid the formation of SnO_xC_y deposits with extremely high carbon content. [5]

To investigate the impact of oxygen flow on carbon incorporation the flow rate of the oxygen into the chamber was steadily increased from 0sccm up to a maximum of 20sccm. The elemental composition of the samples was determined using x-ray analysis within an environmental scanning electron microscope (ESEM). The microscope is equipped with both energy and wavelength dispersive detectors giving fine resolution where peaks occur at comparable energies. Measurements were taken at several different sites within each sample to ensure reproducibility.

Figure 1 shows a typical EDX profile for one of the SnO₂ coatings. The presence of carbon within the coating is clearly demonstrated from the peak at approximately 0.3keV. The small nitrogen peak is a consequence of a statistical averaging procedure applied by the microscope software. It also calculates the likelihood of each peak being a true representation of the composition of the sample and this strongly suggests that there is no nitrogen within these films. The small quantity of Ge is indicative of the substrate material used.

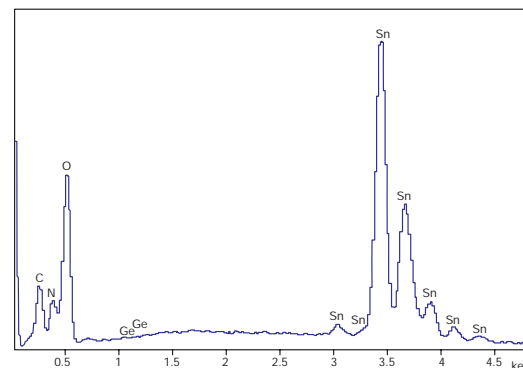


Figure 1: Results of EDX analysis performed on SnO₂ coating deposited by MOPACVD

Figure 2 shows a clear correlation between the amount of carbon incorporated within the film and the flow rate of oxygen. A flow rate of 10sccm of oxygen appeared to be optimum for the reduction of carbon.

In obtaining the results shown in Figure 2, all the other experimental parameters remained constant. It was noted that the substrate temperature increased steadily from room temperature up to approximately 35°C during the course of the deposition due to heating from ion bombardment.

Optical Properties of SnO₂ Thin Films

The visible and NIR transmission of a SnO₂ coating deposited using a flow rate of 10sccm compared to a coating produced by thermal evaporation is shown in Figure 3. Both films were deposited onto BK7 glass substrates. It is clear that the electronic absorption edge is shifted to shorter wavelengths when the coating is deposited by CVD compared to thermal evaporation. This is consistent with the CVD coating possessing a wider band gap of 2.7eV compared to 1.3eV for thermally evaporated thin films.

Refractive index (n) for the MOPACVD films agrees well with the values published by S Hamzaoui et al [6] but is slightly lower than most other values reported in the literature. [7] The infrared transparency suggests the inclusion of water vapour and other species such as CO₂, CO and CH₄ that may have been incorporated during film growth. This would imply that the packing density of the coating is somewhat reduced which could explain the slightly lower refractive index obtained. Densification would normally be optimised by increasing the energy of the reaction process. An elevated substrate temperature or increase of plasma voltage might address this issue and is an area of ongoing experimentation. The susceptibility of SnO₂ coatings to the inclusion of water vapour has also been reported by Li-Jian Meng et al [8] who noted pores within the columnar structure of their sputtered films.

Conclusions

Various SnO₂ films have been deposited onto infrared and visibly transparent substrates at low process temperatures. The oxygen flow rate is seen to be an important parameter in the reduction of carbon from SnO_xC_y thin films. For a flow rate of 10sccm of oxygen, the content of carbon in the coatings was reduced from 40 at.% (for 0sccm) to 14 at.%. It appears as though the energy band gap (2.7eV) of SnO_xC_y does not vary significantly with the ratio of oxygen to carbon but further investigation is required to assess the effect of varying the substrate temperature and RF power on the film composition and optical properties.

Acknowledgement

Research performed at Reading University in collaboration with S J Wakeham.

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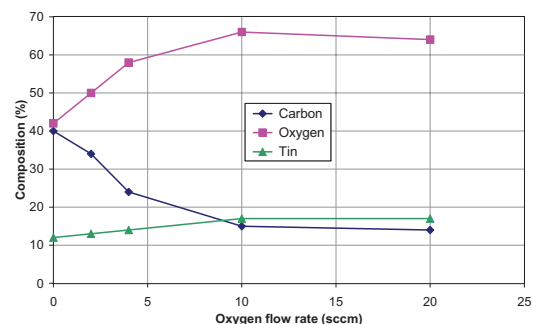


Figure 2: Change in elemental composition of SnO_xC_y coatings with variation of O₂ flow rate

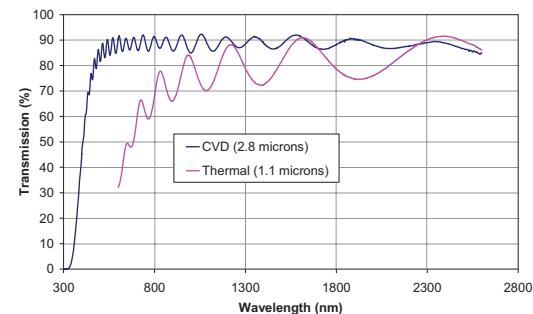


Figure 3: Spectral comparison of CVD and thermally evaporated SnO₂ coatings in the visible and NIR wavelength region

SAFC Hitech Sales Force Reorganisation



John Selby
Global Sales Manager

The merger of Epichem and Sigma-Aldrich sales networks has been ongoing since 2007 with careful attention paid to all existing customers. A number of personnel changes have led to the formation of a revitalised team focussed on taking forward the improved product offerings of the expanded SAFC Hitech business sector to a global market whilst maintaining the years of excellent customer focussed service and individual relationships forged with clients. To ensure full information is available on the sales team contacts in all areas, an up-to-date list of the SAFC Hitech representatives is presented below and features very familiar faces to guarantee customer satisfaction.



Lynne Gibson
US East Coast

A key sales team policy that runs through all product lines is to coordinate accounts on a global basis to maintain a uniform supply chain to all customer production locations. Each of the team has a number of such accounts to act as primary responsible person and this has proved exceptionally valuable to larger companies with geographically separated sites. With the ever increasing demand for new materials the traditional boundaries between markets are being broken down and the SAFC sales team can provide a comprehensive portfolio to meet all customer requirements as a "one stop shop".



Ed Donohoe
Electronic & Performance Materials



Aimee Wright
US West Coast

We wish everyone success in 2008.



Ann Hughes
Europe



Chris Richards
Performance Materials



Colin Overton
Speciality Gases



Shufan Cheng
China



Gary Kameda
Japan and OEMs

Novel Deposition Technique for Complex Oxide Films

Complex oxide films for electro-optic and piezoelectric applications are currently produced using pulsed laser deposition (PLD), however this technique is limited to relatively small sample sizes. The desire to achieve good film uniformities on 100 to 150mm wafers has led to study of a new method developed at EPFL and exploited by a new company ABCD based on Chemical Beam Epitaxy (CBE) within a revolutionary novel tool.

A range of partners have been assembled to fully explore the capabilities of the system and take advantage of its systematic stoichiometric variations in combinatorial research and *in situ* structured growth control by laser assistance. The project 3D-DEMO (FR6-2004-NMP-TI4-STRP 033297) is supported by the EC. (<http://www.3d-demo.org/>)



Details of the equipment are shown in Figure 1 and it is clear that compatible precursors were necessary to ensure optimum performance. SAFC has provided a range of Li and Nb sources to allow the successful deposition of LiNbO_3 layers.

Initial work has focused on $\text{Li}(\text{O}^i\text{Bu})$ and $\text{Nb}(\text{OEt})_5$ (see Figure 2) but more advanced precursors are currently undergoing trials to improve performance and further trials are planned to further explore this material system and also other complex oxides using the most suitable sources.

| Participant Name | | Scientific role |
|---|----|--|
| Ecole Polytechnique Fédérale de Lausanne (EPFL) | CH | CBE, PLD Structural, dielectric, piezoelectric characterization |
| ABCD Technologies Sàrl | CH | CBE equipment manufacturer |
| SCIPROM Technology Sàrl | CH | Management support |
| SAFC HITECH Ltd | UK | Precursors |
| SAES Getters S.p.A. | I | LN, LT substrates, test device fabrication & evaluation |
| National Inst. Lasers, plasma & radiation physics (NIL) | RO | PLD Structural characterization |
| CNRS UMR 6174 (FEMTO) | F | SNOM |
| University of Southampton | UK | Optical measurements, device design and evaluation |

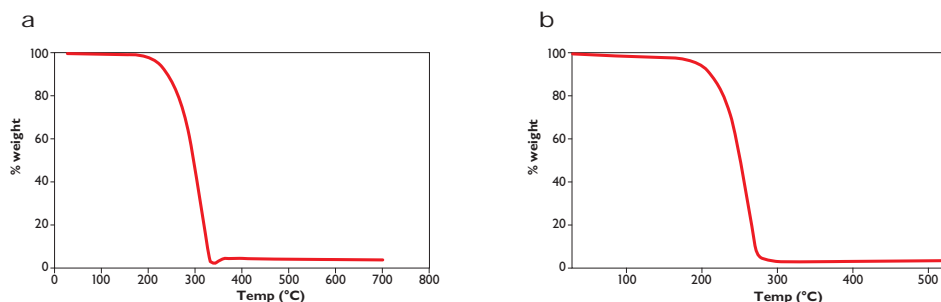


Figure 2: Thermogravimetric analysis data for a) $\text{Li}(\text{O}^i\text{Bu})$ b) $\text{Nb}(\text{OEt})_5$



Figure 1: Schematic cross section through CBE process chamber (EPFL, top), CAD drawing of new reactor (ABCD, middle), photograph of new reactor (ABCD, bottom)

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