

epichem group news

Fourth Quarter 2003

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Research is Key

Collaborative research is a key element to research and development activities performed in the Epichem laboratories

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Precursors for Hafnium Silicate

Reporting on recent research, supported by Epichem Oxides & Nitrides at Liverpool University

New Oxides & Nitrides Laboratory

A new laboratory has been installed at Bromborough, UK for research and small to medium scale production of Epichem Oxides & Nitrides.

The laboratory is self-contained and includes five walk-in fume cupboards, a wash fume cupboard and local extraction points with their own air handling units and extracted air scrubbing. Flexibility has been the main requirement for the fume cupboards to ensure that future, as well as current, products are accommodated. A facility for benches



has been provided in all the cupboards that are removable and can be used at various heights. To improve access for maintenance and installation of plant items, a service corridor has been provided at the rear of the fume cupboards. This corridor is also used to locate the manifolds for the services supplying the various areas of the laboratory.

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Epichem Gases

Epichem Gases has responded to the downturn in the semiconductor business by targeting other market sectors. This strategy has proved successful and we now have new customers in the coating and chemical reagent fields.



See page 5 for the full story

Compounds

- ALUMINIUM
- ANTIMONY
- ARSENIC
- BARIUM
- BISMUTH
- BORON
- BROMINE
- CADMIUM
- CALCIUM
- CARBON
- CAESIUM
- CHLORINE
- CHROMIUM
- COBALT
- COPPER
- GALLIUM
- GERMANIUM
- HAFNIUM
- INDIUM
- IODINE
- IRON
- LANTHANUM
- LEAD
- MAGNESIUM
- MANGANESE
- NICKEL
- NIObIUM
- NITROGEN
- PHOSPHORUS
- PLATINUM
- RUTHENIUM
- SCANDIUM
- SELENIUM
- SILICON
- STRONTIUM
- SULFUR
- TANTALUM
- TELLURIUM
- TIN
- TITANIUM
- TUNGSTEN
- VANADIUM
- YTTRIUM
- ZINC
- ZIRCONIUM

Our sales and operations team work hard to ensure that our customers are happy with our products and services. The information transfer between these two departments is critical to ensure that our products reach their destination safely and as requested.

This can only be achieved with an exceptional internal support team for both sales and operations who, most importantly, have a customer focus.

Brenda Tagliavia - Operations and Sales Support Manager

Brenda joined Epichem in 1996 from Air Products and has been working within Customer Service since that time. Brenda currently manages the US Customer Service function. She assists Operations and Business Managers of both Metalorganics and Oxides & Nitride precursors. Brenda works with the Global Sales team to provide outstanding customer focused service.



Sue Edwards - Customer Service Administrator

Sue has been at Epichem since May 2000 and joined us from Rock Oil where she worked as Export Administrator. Sue has responsibility for administration, from processing customer orders to preparation of customer quotations, and specific responsibility for export license applications to ensure our products are safely and legally transported around the world. Sue is also IATA (International Air Transport Association) trained.

Lynsey Houghton - Shipping Co-ordinator

Lynsey joined Epichem in 1996 as the company Receptionist and in July 1998, she applied for a position within the Customer Care team and was successful in her application. Lynsey gained her IATA certification as well as training in hazardous awareness for ADR and IMO regulations. She now has responsibility for document preparation as well as for ensuring the safe shipment of our products and timely delivery to the customer.



Vicky Arundale - Internal Sales Co-ordinator

Vicky has been working for Epichem since January 2002, prior to which she worked for Optogas and Linde as an integral part of the sales department. Her vast knowledge of the speciality gases market means she is best placed to help the customers in this area. Vicky has responsibility for providing internal sales support and liaising between sales personnel, production and the customer with regard to product supply and availability.

Marian Traves - Internal Sales Co-ordinator

Marian initially worked for Inorgtech since joining the company in May 1996 and remained in employment when Epichem purchased the business in 2000. Since that time, Marian has had responsibility for accounts, despatch, administration and internal sales. Marian relocated to Epichem Bromborough in September this year and has responsibility for Oxide & Nitride customer service, encompassing quotations, order placement and ensuring that all orders reach our customers correctly and on time. Marian is responsible for the safe transportation of our Oxides & Nitride products and is also IATA trained.



*outstanding
customer focused
service*

Bubbler Specifications



Standard ref88 bubbler

Epichem products are supplied in the highest quality stainless steel bubblers with packless diaphragm valves as standard on its bubblers from 100g to 2Kg and beyond. Stringent preparation and testing ensure that the interior surfaces are contaminant-free prior to product fill and He leak testing of valves is employed to check their integrity. A standard range of bubblers exists, however, Epichem remains flexible with the number of alternative assemblies that can be fabricated upon request.

A range of specified features can be included to tailor the ampoule design to meet customer requirements without compromising quality or reliability. The available options include:

Custom body dimensions - Maximum utilisation of temperature control bath volume

Variable connection orientation - Flexibility to fit non-standard equipment

Rotated valves - Improved access to operate manual valves

Polarized fittings - Added safety to ensure correct connection direction

Thermowell - Increased temperature control capability

Bypass valve - Simplified, higher integrity purging procedures

Feet - Improved stability, increased fluid circulation in heating bath to reduce temperature variations

Hard seated valves - Higher operating temperature with metal-metal seals

Dished end ampoules - Larger fill volumes accessed with reduced weight and improved construction

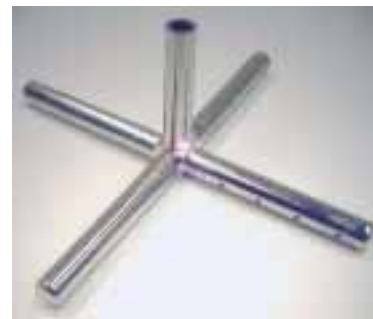
Alternative diptube geometries - Improved pick up stability and precursor usage under aggressive usage conditions.

To discuss all combinations of the above features beneficial to your requirements, contact your local representative.

The use of a dual chamber bubbler for solid TMI has been demonstrated to replicate the flux stability achievable for two conventional bubblers in series whilst providing a smaller footprint.



The cross dipleg is gaining popularity for larger ampoules to maintain flux stability at the higher carrier flows and the new perforated disc option offers even greater reliability in most user cases.



Introducing John Selsby - Global Sales Manager

Epichem Group is delighted to announce the appointment of John Selsby as Global Sales Manager. John has vast commercial knowledge and experience of major semiconductor manufacturers and is well placed to take responsibility for further developing relations with current and new customers, establishing new markets and co-ordinating our sales activities around the world.

John graduated with a BSc Chemistry from Newcastle-upon-Tyne University and most recently worked as European Sales Director for the electronics division at Praxair Inc. Prior to that, he spent 25 years with Air Products, latterly as European Sales Manager.

John has an office base in Bromborough and at his home office in Hampshire, UK. Over the forthcoming months, he will be travelling to our global locations to meet customers and colleagues alike.

I am sure you will join us in wishing John every success in his new role.



Gases are a changing scene at Epichem. For many years, this business sector relied almost solely on the semiconductor business and only two gases, silane and boron trichloride. As the downturn in the semiconductor business turned from rumour through prediction to reality, Epichem Gases expanded to target other industrial applications. While the majority of our gases still go to standard semiconductor uses, we've also found success in gaining significant customers in the coating and chemical reagent areas.

This change has required the extension and improvement of facilities. Epichem's current silane filling system is comprised of two independent manifolds, in which the "make and break" connection points are protected by a down flow of clean air, provided by air handling units and filters. An automatic purging system has been installed to ensure that all cylinders are completely emptied to a very high standard. In order to constantly have the ability to analyse both raw material and filled vessels at any time of the day or night, Epichem now has two independent analysers, capable of measuring gaseous impurities down to 0.1ppm.



Epichem analyses thousands of cylinders of silane in a year and has therefore built up an impressive database of analysis and analytical trends. Epichem now has two independent boron trichloride production streams, doubling capacity to over 150 tonnes per annum. The BCl_3 filling system has recently been upgraded to improve cylinder handling efficiency and further advance bottling capability.

Epichem signed a collaboration agreement with Degussa AG to sell and develop a number of materials. Initially, these products include chlorosilane, dichlorosilane, trichlorosilane and silicon tetrachloride. These materials have generated considerable interest and sales have resulted. The chlorosilanes are the building blocks for a range of the other materials used in the semiconductor industry which, when fully developed, will be available for supply to customers.



New Precursors for La-oxide and La-silicate Dielectric Films



Lanthanum oxide (La_2O_3) and lanthanum silicate (LaSi_xO_y) are currently being investigated as alternatives to SiO_2 in silicon-based field effect transistors. In recent research, supported by Epichem Oxides & Nitrides, at Liverpool University, new precursors have been developed for the deposition of La_2O_3 and LaSi_xO_y by liquid injection MOCVD.

There has been considerable recent interest in the use of lanthanum oxide (La_2O_3) and lanthanum silicate (LaSi_xO_y) as alternative high-k gate dielectric layers in silicon-based field effect transistors^[1-3]. Lanthanum oxide has a variety of other useful physical characteristics, such as high mechanical stability and good optical transparency from ultraviolet to infrared, leading to applications in protective^[4] and optical coatings^[5]. It is also a component of the ferroelectric oxides, $(\text{Bi},\text{La})_4\text{Ti}_3\text{O}_{12}$ ^[6] and $(\text{Pb},\text{La})(\text{Zr},\text{Ti})\text{O}_3$ ^[7] used in non-volatile FERAMs, and is present in the conducting oxides $\text{La}_{1-x}\text{Sr}_x\text{CoO}_3$ ^[8] and LaNiO_3 ^[9], potential electrode materials in ferroelectric devices.

MOCVD is the preferred technique for the deposition of electroceramic thin films, having advantages such as good composition control, high film densities and deposition rates, and excellent conformal step coverage. However, the growth of high quality La_2O_3 by MOCVD has not yet been demonstrated. La_2O_3 films grown from the β -diketonate precursors $[\text{La}(\text{thd})_3]$ (thd = 2,2,6,6-tetramethylheptane-3,5-dionate)^[10] and $[\text{La}(\text{acac})_3]$ (acac = acetylacetonate)^[11] showed heavy carbon contamination. There is thus an urgent requirement for new, improved precursors for the MOCVD of lanthanum-containing oxides, but there are few precursors available with the appropriate stability and volatility.

Metal alkoxides have been widely used in the MOCVD of metal oxides, but there are few reports in the literature on the use of rare-earth alkoxide precursors in MOCVD. This is because the large ionic radius of the highly positively charged lanthanide (III) ions leads to the formation of bridging intermolecular metal-oxygen bonds, so that simple $\text{La}(\text{OR})_3$ complexes are polymeric, with a corresponding low volatility. However, the sterically hindered donor functionalised alkoxide ligand 1-methoxy-2-methyl-2-propanolate, $\text{OCMe}_2\text{CH}_2\text{OMe}$ [mmp], contains two [Me] groups on the α -carbon atoms close to the metal centre, which inhibits polymerisation and increases precursor volatility, and we have recently utilised the unique properties of this ligand to synthesise $[\text{La}(\text{mmp})_3]$, a rare example of a volatile rare earth alkoxide^[12].

Thin films of LaO_x were deposited over a wide range of substrate temperatures (250 - 600°C) using a $[\text{La}(\text{mmp})_3]$ / tetraglyme mixture in toluene (see Figure 1)^[12]. The atomic composition of the LaO_x films was determined using Auger electron spectroscopy (AES), which gave O:La ratios of 1.8-2.4, consistent with the films being La_2O_3 containing excess oxygen (expected O:La ratio in $\text{La}_2\text{O}_3 = 1.5$). Very significantly, carbon was absent from all of the films at the estimated detection limit of < 0.5 at.-%, a remarkable result considering the range of growth temperatures used.

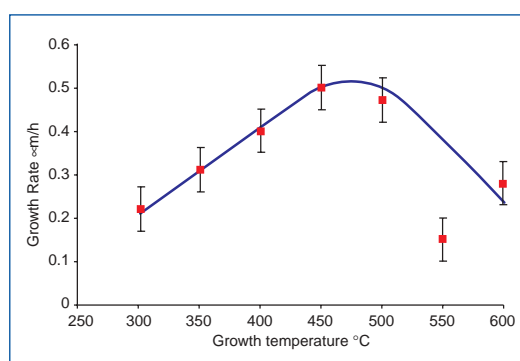


Figure 1
Variation in growth rate with substrate temperature for La-oxide films grown by liquid injection MOCVD using $[\text{La}(\text{mmp})_3]$

The X-ray diffraction pattern (see Figure 2) of a film deposited at 450°C (PAW 309) exhibits three dominant diffraction peaks attributed to the (100), (002) and (101) reflections measured at 2θ values of 25.1°, 27.9° and 29.7° respectively, which is consistent with the random powder diffraction pattern of La_2O_3 with a hexagonal structure. A scanning electron micrograph (SEM) of a fracture sample from a lanthanum oxide film deposited at 450° (Figure 3) displayed a columnar growth habit with associated 'hillock' features^[12].

As well as being a useful source for the deposition of high-k gate dielectric films, where low temperature deposition is a necessary requirement, $[\text{La}(\text{mmp})_3]$ is also likely to be a good precursor for the deposition of La-containing ferroelectric films, such as $(\text{Bi},\text{La})_4\text{Ti}_3\text{O}_{12}$.

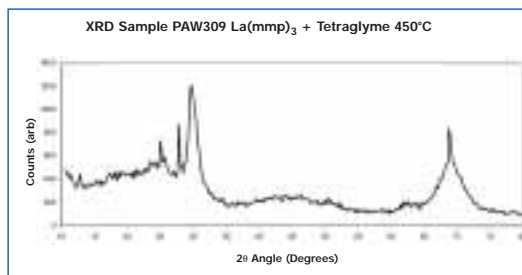


Figure 2
XRD spectrum of lanthanum oxide deposited at 450°C using $[\text{La}(\text{mmp})_3]$

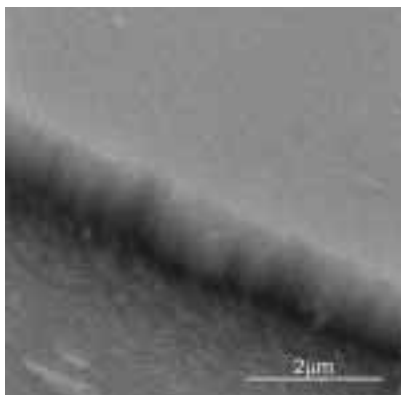


Figure 3
Scanning electron micrograph of a lanthanum oxide film deposited at 450°C using $[\text{La}(\text{mmp})_3]$

A potential problem with La_2O_3 is that it is chemically unstable in air, reacting with CO_2 to form $\text{La}_2(\text{CO}_3)_3$ ^[13], and adsorbing water to give $\text{LaO}(\text{OH})$ and $\text{La}(\text{OH})_3$ by an extensive surface hydroxylation process^[14]. This can result in oxygen vacancies and excess positive charge in the oxide film layer, leading to unwanted flat-band voltage shifts in the metal-oxide dielectric structure^[1]. In addition, La-oxide films deposited on Si substrates have shown a tendency to form a lanthanum silicate interfacial layer, and so a number of recent studies have focused on the deposition of LaSi_xO_y ^[3]. Although the permittivity of LaSi_xO_y will inevitably be lower than the pure La_2O_3 ^[11], this may be an acceptable trade-off for improved interface stability.

We have now found that thin films of LaSi_xO_y can be deposited by liquid injection CVD using $[\text{La}(\text{N}(\text{SiMe}_3)_2)_3]$ in the presence of oxygen over a wide range of substrate temperatures (Figure 4)^[15]. Auger electron spectroscopy (AES) showed that all films were lanthanum silicate, with silicon concentrations ranging from 8.5 at.% to 15.2 at.% which have no obvious dependence on substrate temperature.

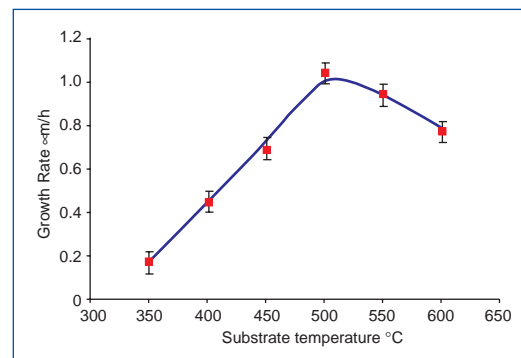


Figure 4
Variation in growth rate with substrate temperature for La-silicate grown using $[\text{La}(\text{N}(\text{SiMe}_3)_2)_3]$

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Carbon and nitrogen were not detected in the films (estd. detection limit 1 at.%). Analysis of the films by scanning electron microscopy (SEM) showed that all of the as-grown films were smooth and featureless on the micron scale, and Raman spectra of the as-deposited films suggested an amorphous network structure consisting of (La_2O_3) and (SiO_2) molecular sub-units. Upon annealing at 800°C and above, the films undergo crystallisation to form a complex range of lanthanum silicates^[15].

High frequency capacitance-voltage (C-V) data for $[\text{Al} / \text{La}_{0.37}\text{Si}_{0.10}\text{O}_{0.53} / \text{n-Si}]$ MOS capacitor structures (see Figure 5) showed well defined accumulation, depletion and inversion regions, with a marked shift in the flatband voltage indicating the presence of a significant level of positive fixed charge ($+Q_f$) near the La-silicate-silicon interface. These C-V characteristics are similar to those of MOS capacitors fabricated using La-silicate films deposited by physical vapour deposition techniques^[16].

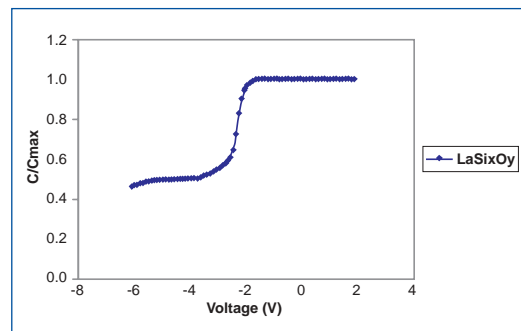


Figure 5
High frequency C-V data for an $[\text{Al} / \text{La}_{0.37}\text{Si}_{0.10}\text{O}_{0.53} / \text{n-Si}]$ capacitor structure grown using $[\text{LaN}(\text{SiMe}_2)_3]$

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Ferroelectric Meeting Highlights Nanoassembly

Delegates to the Tenth European Meeting on Ferroelectrics assembled on August 4th within the hallowed precincts of Cambridge University's Department of Earth Sciences, where the office once occupied by Charles Darwin still exists. Epichem was delighted to sponsor the opening concert in the city's Guildhall given by the Cambridge Philharmonic Society and the internationally acclaimed harpist, Alison Nicholls, playing works by Beethoven, Debussy, Elgar and Mendelssohn. The international language of music was enjoyed by 500 delegates from 41 countries.

The international language of ferroelectric science occupied the remainder of the week and a particular highlight was the subject of self-assembly of ferroelectric structures. The message from the nanotechnology sessions was that by choosing the right goal, scientists can employ natural functionality to achieve complex tasks. Such types of harnessed assembly mean that what appears to be a difficult process, turns out to be quite easy.

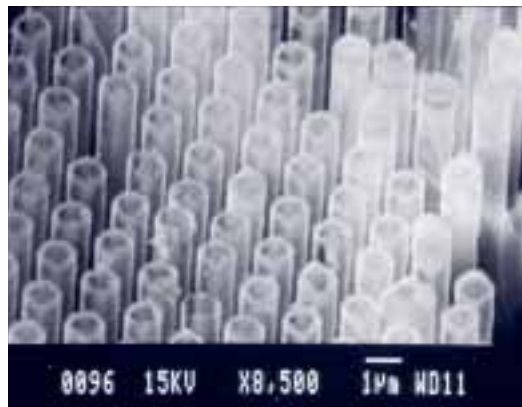
The Epichem sales booth in the poster hall was manned by Dr. Paul Williams, who has joined the European support team after several years of thin film growth experience at Liverpool University. Our sales development manager for Oxides & Nitrides, Dr. Tim Leedham, delivered an invited paper "Ferroelectric Precursors from Science to Technology" which emphasised the importance of interdisciplinary collaboration to achieve process robustness. Epichem also presented a poster on the molecular design of ferroelectric precursors.

The meeting was climatically memorable for shade temperatures approaching a hundred degrees Fahrenheit (38°C). Fortunately, Epichem was able to organise a cool supply of locally brewed Galingale beer, which was much appreciated during the poster sessions!

Abstracts of the meeting are available at <http://www.the-conference/JConfAbs/8/>. Proceedings will be published in the journals Ferroelectrics and Integrated Ferroelectrics, whose offices are located in Philadelphia. (Phone 1-215 625 8900)



Prior to the Epichem-sponsored concert, members of the Cambridge Philharmonic Society rehearse Mendelssohn's Symphony No. 4 "The Italian"



Micrograph of SBT nanotubes self-assembled from an ultrasonically dispersed precursor solution provided by Epichem Oxides & Nitrides. Micrograph courtesy of Prof. J.F. Scott, University of Cambridge



Recent research, supported by Epichem Oxides & Nitrides at Liverpool University, has shown that Hf-silicate films can be deposited using either a single source precursor molecule $[\text{Hf}(\text{OSiBu}^t\text{Me}_2)_4(\text{Et}_2\text{NH})]$, or two separate sources, $[\text{Hf}(\text{NMe}_2)_4]$ and $[\text{Bu}^t\text{Me}_2\text{SiOH}]$.

ZrO_2 and HfO_2 have been investigated in depth as alternatives to SiO_2 as the gate dielectric material for sub- $0.1 \mu\text{m}$ complementary metal-oxide-semiconductor (CMOS) technology. However, neither ZrO_2 and HfO_2 are completely chemically or thermally stable on silicon, and both form interfacial layers of silicon dioxide or metal silicates, which leads to high leakage currents and a decrease in the performance of transistors incorporating these materials as gate dielectrics. A possible solution to this problem is to deposit a metal silicate, such as ZrSi_xO_y or HfSi_xO_y directly on to the silicon substrate^[1]. These form amorphous films, which are thermodynamically stable on silicon. The permittivities of these pseudo-binary alloys are inevitably lower than the pure metal oxides, but this is considered to be an acceptable trade-off for the greatly improved interface stability. For instance, ZrSi_xO_y has an intermediate dielectric constant (κ) of approx. 12 - 25, but is still significantly higher than that of SiO_2 ($\kappa = 3.9$)^[1].

ZrSi_xO_y and HfSi_xO_y thin films can be deposited by physical vapour deposition techniques, such as MBE and reactive sputtering^[1], but there have been intense recent efforts to develop MOCVD or ALD processes, which have the major advantages of high film uniformity, and superior conformal step coverage. A variety of precursor combinations have been investigated; for instance ZrSi_xO_y and HfSi_xO_y have been deposited by liquid injection MOCVD using $[\text{Zr}(\text{NEt}_2)_4]$ ^[2] or $[\text{Hf}(\text{NEt}_2)_4]$ ^[3] in combination with $[\text{Si}(\text{NMe}_2)_4]$, but the Si incorporation can show a sharp dependence on substrate temperature^[2], and differences in the thermal stability of the Group IVB and Si precursors may reduce layer uniformity. This problem can be alleviated by the use of single source precursors, such as $[\text{Zr}(\text{acac})_2(\text{OSiMe}_2\text{Bu}^t)_2]$ ^[4], and we have now shown^[5] that the novel "single source" precursor $[\text{Hf}(\text{OSiBu}^t\text{Me}_2)_4(\text{Et}_2\text{NH})]$ (Figure 1), allows the reproducible deposition of a level of HfSi_xO_y by liquid injection MOCVD, containing ~ 10 at.% Si, which showed little variation over a range of growth temperatures and oxygen partial pressures.

A problem of the "single-source" approach is that it lacks flexibility, as the fixed Hf-Si ratio in the precursor makes it difficult to alter the metal-silicon concentration ratio in the film, which limits the ability to fine-tune the film properties. However, it has now been shown^[5] that the simultaneous injection of solutions of $[\text{Hf}(\text{NMe}_2)_4]_2$ and $\text{Bu}^t\text{Me}_2\text{SiOH}$ allows the growth of HfSi_xO_y in which the silicon level in the films can be well controlled by altering the gas-phase concentration of $\text{Bu}^t\text{Me}_2\text{SiOH}$ (Figure 2). It was proposed that a single source precursor species, such as $[\text{Hf}(\text{OSiBu}^t\text{Me}_2)_4]$ (see Figure 1) is formed *in situ* in the gas phase prior to HfSi_xO_y film growth, resulting in a process with the combined advantages of the single source and dual source approaches.

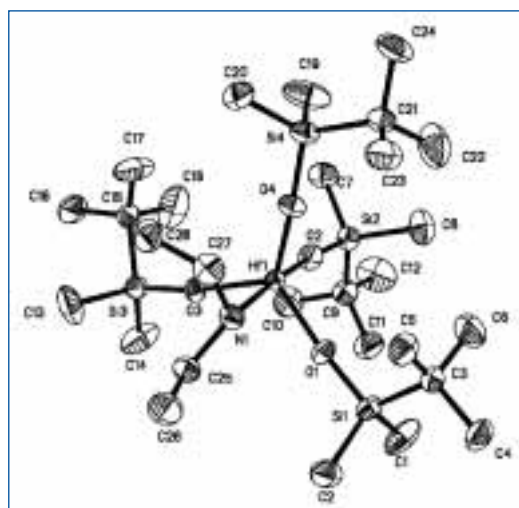


Figure 1
Crystal structure of $[\text{Hf}(\text{OSiBu}^t\text{Me}_2)_4(\text{Et}_2\text{NH})]$

The dielectric properties of a HfSi_xO_y film (Si concn. = 13.4 - 18.3 at.-%) deposited at 500°C from $[\text{Hf}(\text{NMe}_2)_4]$ and $\text{Bu}^t\text{Me}_2\text{SiOH}$ were found to be very good (Figure 3), with minimal hysteresis and relatively small flatband voltage shifts.

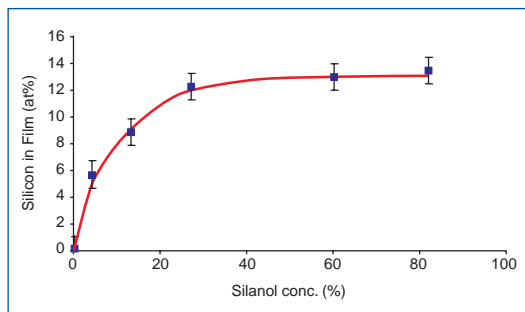


Figure 2
Variation of silicon concentration in the HfSi_xO_y film with $\text{Bu}^i\text{Me}_2\text{SiOH}$ gas phase % mole fraction $[(\text{moles } \text{Bu}^i\text{Me}_2\text{SiOH}) / (\text{moles } [\text{Hf}(\text{NMe}_2)_4] + \text{moles } \text{Bu}^i\text{Me}_2\text{SiOH}) \times 100]$

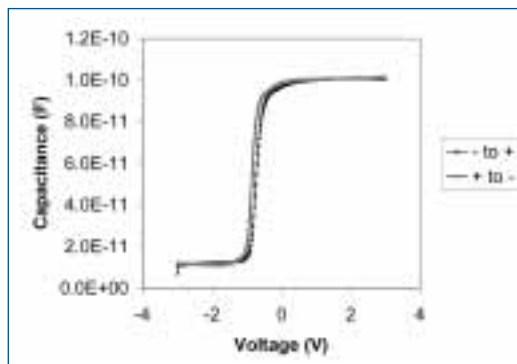


Figure 3
High frequency C-V data for a $[\text{Al}/\text{HfSi}_x\text{O}_y / \text{n-Si}]$ ($x = 13.4 - 18.3\%$) capacitor structure grown using $[\text{Hf}(\text{NMe}_2)_4]$ and $\text{Bu}^i\text{Me}_2\text{SiOH}$ precursors

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New Oxides & Nitrides Laboratory

Continued from front page

This was a fast track project, which was initiated at the beginning of May 2003 and commissioned mid-October 2003, and involved the conversion of an open plan office to a laboratory. This goal was achieved through Epichem's ability to react quickly to changes in priorities, flexibility to work on diverse projects and capability to build a relationship and work closely with the contracting company. The fume cupboards and ancillary equipment required considerable input from Epichem personnel, whilst the structural and building aspect was concentrated on by the contracting company. The two areas were then pulled together to produce a successful project.

Epichem is now looking forward to the production of Epichem Oxides & Nitrides at Bromborough in the new facility. Further expansion projects are ongoing such as a new road to improve access to the site and extra storage areas for Epichem's expanding gases products.



Epichem round the globe



*Epichem is a truly
global company*

m metalorganics

g gases

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