AUTUMN 2008

# **Process**News

A Newsletter from Oxford Instruments Plasma Technology (OIPT)

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## Welcome to **Process**News

Oxford Instruments prides itself in being innovative and adapting our technologies as customer requirements change. For example, in response to the increasing international focus on energy conservation, we have consistently developed our strong leadership in High Brightness Light Emitting Diode (HBLED) processes.

Most recently our exciting acquisition of Technologies and Devices International Inc (TDI), a world leader in the development of Hydride Vapor Phase Epitaxy (HVPE) technology and processes, will enable Oxford Instruments Plasma Technology to expand the range of products it already supplies to the HBLED market.

OIPT continues to develop its offering as a leading and specialist provider of systems in areas including Failure Analysis, MEMS, Ion Beam, and Compound Semiconductors, while also investigating new technologies.

This past year was a hugely successful one for OIPT and we made a great start to the new financial year with a record order month in April. We remain firmly on track to double the size of the business from 2005 to 2010. We continue to increase our investment in R&D and additional sales and service personnel, underlining our commitment to improved customer satisfaction, through the products and processes that we develop, to the support we offer in the field.

I hope you enjoy **Process**News, and we look forward to working with you in the coming year.

**Andy Matthews**
*Managing Director*

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## HBLEDs: How Oxford Instruments is shaping the future

Lighting is one of those things we take for granted, but it forms an integral part of our daily lives, impacting enormously both on us and our environment.

Global lighting consumption is estimated to account for the output of 1000 electric power plants, and costs £100 billion a year, so the drivers for a more efficient light source are both commercial and environmental. This is where the HBLED comes in, replacing the old incandescent lightbulb technology. Theoretically, HBLEDs could use less than 20% of the power needed to produce incandescent light bulb equivalent levels of output for lifetimes up to 50,000 hours – a great leap forward.

Oxford Instruments is playing an increasingly important role in the manufacture of HBLEDs, enabling huge changes in the global lighting industry.

*Story continues on page 2*

**PlasmaLab**133-ICP380

OXFORD INSTRUMENTS
*The Business of Science*®

# HBLEDs: How Oxford Instruments is shaping the future

**Mark Dineen** PhD, Principal Applications Engineer, OIPT

*Continued from cover*

## Producing an HBLED with the help of Oxford Instruments

Starting with a flat substrate, commonly sapphire, to improve the quality of the device the surface is sometimes patterned with raised features, and this requires etching. The Oxford Instruments **Plasmalab**®System133-ICP380 is used by a number of leading HBLED manufacturers to perform this function.

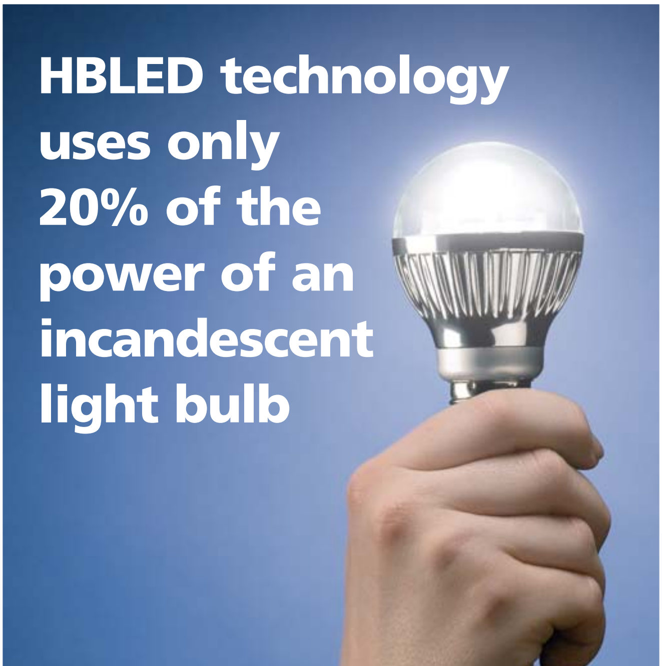
GaN material is then grown on the sapphire using MOCVD, MBE or HVPE. The final structure is a p-n junction with the p-type on the top surface and the n-type layer next to the substrate. Because of the insulating nature of the sapphire it is now necessary to etch down to the n-type layer.

The **Plasmalab**®System133-ICP380 can etch 21 x 2″ of these wafers at one time, giving world leading throughput.

Some further surface preparation can now be performed, for example photonic crystal etching, to enhance the light extraction from the devices, this is followed by the device isolation etch. Both these steps are achievable using the **Plasmalab**®System133-ICP380.

The final process is to enclose the device in a protective dielectric layer and the **Plasmalab**800Plus DP800 is the system of choice to do this for a large number of HBLED manufacturers. The advantage of the **Plasmalab**800Plus DP800 is the leading batch size, as it is capable of a processing mammoth 40 x 2″ wafers in a single run, giving superb throughput on a reliable platform.

The dielectric isolates the device and protects it from contamination. It is now ready for packaging and making into a light.



**HBLED technology uses only 20% of the power of an incandescent light bulb**

## What the future holds

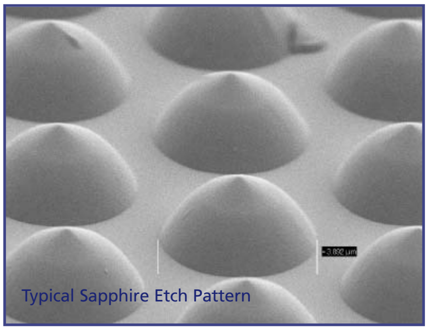
As the industry moves on to the larger 4″ substrates, it becomes a much more attractive proposition to process single wafers at a time rather than batches. Oxford Instruments already has the technology to perform high yield, high speed processes, and the company is ready to progress to the next stage.

## Single Wafer Etching using an Electrostatic Chuck

The benefit of moving to single wafers is that the wafer may now be clamped more readily. New technology allows clamping of sapphire wafers using a piece of equipment called an Electrostatic Chuck. The benefit of this is that the wafer is held without touching the sensitive topside – an important improvement over mechanical clamping, as this reduces particles and gives improved wafer cooling, allowing increased etch rates with photoresist masks.

## ICP-CVD Passivation

Traditionally PECVD has been used for the deposition of the final protective layer, however this requires high temperature to give the quality of film needed for HBLEDs. Some studies have shown that HBLEDs perform better if the temperature is kept below a certain limit during the manufacturing process. ICP-CVD allows very high quality films to be grown at low temperature and high rate.



Typical Sapphire Etch Pattern

## Looking to the future

HBLEDs will have a very positive impact on our lives but technical advances still need to be made to facilitate it. Through technology and experience, Oxford Instruments is uniquely placed to make this future possible.

**We now have electrostatic chuck capability for clamping four, six and eight inch wafers in materials such as sapphire, silicon carbide and silicon.**

**The hardware has been further developed for single wafer multi chamber use in high bias chlorinated conditions.**

# MEMS – Fabrication of silicon micro-needles using isotropic and anisotropic plasma etching techniques for biomedical applications

**Dean Stephens,** Senior Applications Engineer, OIPT

When people think of the term MEMS (Micro-electro-mechanical-systems) it generally conjures up images of micro sized turbines, motors and accelerometers.

However, when applied to the field of BioMEMS (MEMS for use in biomedical situations), most individuals would struggle to imagine many current applications.

## Micro-needles for pain-free drug use

One exciting use of this technology is the fabrication of arrays of silicon micro-needles, for the transdermal delivery of drugs or, conversely, blood sampling.

This technique is currently at the forefront of 'pain free' delivery of drugs, as it does not penetrate deeply into subcutaneous tissue that is full of nerve endings, merely piercing the epidermis, and also has the benefit of greatly reducing the possibility of infection of the injection site itself. Likewise, in blood sampling for making blood-glucose measurements, the use of an array of micro-needles increases the permeability of the skin by many orders of magnitude, enabling larger sample volumes without the pain normally associated with this procedure.

## Fabrication with **Plasmalab**®System100

The fabrication of such micro-needles, in silicon, has long been an aim of the biomedical industry due to the high strength of the material and the potential to create huge numbers of identical devices simultaneously in plasma etching equipment.

A technique for fabricating these devices repeatedly, without having to go to the lengths of using many 'dry' and 'wet' methods, has been demonstrated at OIPT, using a **Plasmalab**System100 ICP380 etch tool.

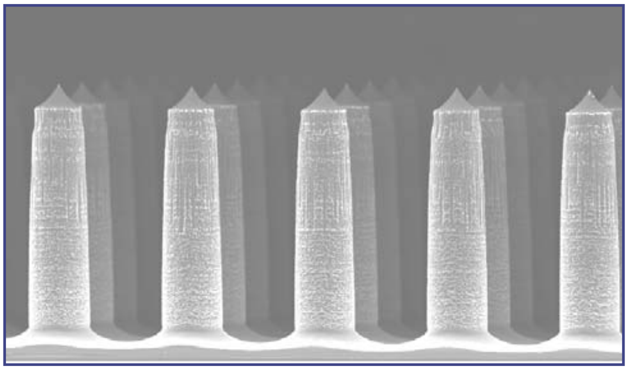
A photoresist masked silicon wafer, with features the size of the desired width of the needle shaft, is first etched isotropically to create the 'point' of the needle, beneath the mask. This feature is not initially etched to a complete needle-tip, as the mask would then detach, but stops short of this at a few microns width. Then, using a deep **Bosch** etch to the targeted depth of the micro-needle array, the needle is anisotropically etched beneath the tip.

The mask is now plasma stripped from the tops of the needles, and another isotropic etch performed to 'sharpen off' the very tips of the micro-needles.

This was all performed in the same plasma chamber, using a combination of both isotropic and anisotropic etching. The arrays can then be separated and the internal holes produced.

## Plasma Precision

Because of the flexibility of this technique the point radius can be tailored precisely depending upon the conditions of the isotropic etch. Equally the angle of the needle shaft can be stringently controlled by adjusting the parameters of the **Bosch** etch, to give the desired sidewall angles.



# New Faces

## Around the World at Oxford Instruments

**Dan Ayres,**
*Operations Director*
Bristol, UK

**Bob Gunn,**
*Applications Team Leader*
Bristol, UK

**Bernard Scanlan,**
*General Manager, Technologies and Devices International, Inc. (TDI)*
Maryland, USA

**Holger Spira,**
*Sales Manager*
Wiesbaden, Germany

**Hirokazu Suzuki,**
*Sales Manager*
Oxford Instruments Asia, Japan office

**Darren Tang,**
*Customer Support Engineer*
Oxford Instruments Asia Team, Penang, Malaysia

**Edwin Kong,**
*Applications Engineer*
Oxford Instruments Asia Team, Singapore

# Hydride Vapour Phase

## For wireless, LEDs, and laser diodes

**Larry Leung PhD,** Product Manager, TDI

On the 9th April Oxford Instruments PLC acquired Technologies and Devices Inc (TDI) who are based at Silver Spring, Maryland, USA

TDI, founded in 1997 by Dr. Vladimir Dmitriev, are a world leading company in the development of Hydride Vapour Phase Epitaxy (HVPE) processes and techniques for the production of novel compound semiconductors such as GaN, AlN, AlGaN, InN, InGaN. These materials are used in a variety of applications, the primary ones being solid state lighting, short wavelength optoelectronics and RF power electronics.

## The HVPE Process

In the HVPE process, Group III nitrides (e.g., GaN, AlN) are formed by reacting hot gaseous metal chlorides (e.g., GaCl or AlCl) with ammonia gas (NH3) (Refer to diagram below). The metal chlorides are generated by passing hot HCl gas over the hot Group III metals. All reactions are done in a temperature controlled quartz furnace.

*e.g., Hot* $HCl\ (g) + Ga\ (l)$ ------> $GaCl\ (g)$

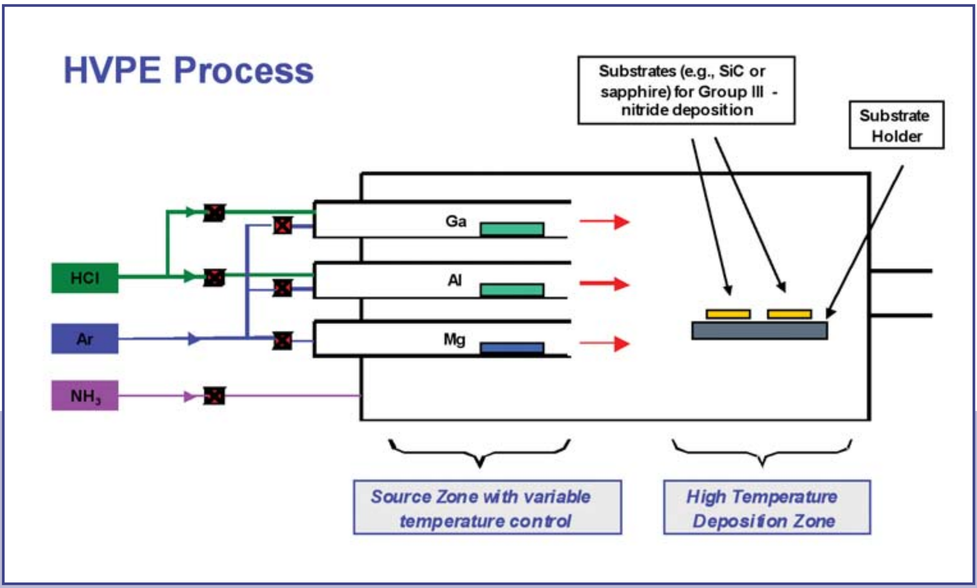
$GaCl\ (g) + NH_3\ (g)$ -------> $GaN\ (s) + HCl\ (g) + H_2\ (g)$

The GaN or AlN templates have been grown on substrates such as SiC or sapphire. p-type GaN or AlN can be achieved by using Mg during the process and n-type by silane gas with Argon as the carrier gas.

## Advantages of HVPE

Developed in the 1960s, it was the first epitaxial method used for the fabrication of single GaN crystals. One of the key features of the technique is its high growth rate (at up to 100 µm per hour) which is almost two orders of magnitude faster than typical MOCVD and MBE processes.

The technique is able to produce crack-free, high quality GaN epitaxial layers (e.g., a typical dislocation density can be as low as $10^7/cm^3$ for a 10 µm thick GaN template on sapphire.) Figure 1 shows the X-ray diffraction of a 10 µm thick GaN template on sapphire. The narrow FWHM of 250 arcsec measured at ω-scan (0002) peak demonstrates excellent material quality.



# Epitaxy (HVPE)

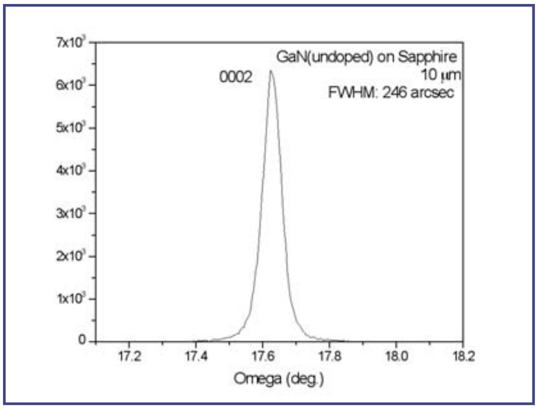


**Figure 1:** The X-ray diffraction of a 10 µm thick GaN template on sapphire

Another advantage of HVPE is its ability to grow thick, high quality of AlGaN and AlN for use in optoelectronic and RF electronic devices. The technique has been demonstrated by TDI to grow thicker high-quality AlGaN-based active regions of shorter wavelength emitters, which have a high radiative recombination efficiency – an essential feature for high-efficiency UV LEDs. Unlike MOCVD, the HVPE process does not involve metalorganics, thus providing a 'carbon-free' environment for epitaxial growth. In addition, the use of gaseous hydrogen chloride also provides an impurity 'self-cleaning' effect, which results in epitaxial layers with low background impurities and more efficient doping level.

TDI has demonstrated the industry's first HVPE-grown, multilayer, submicron AlGaN/GaN heterostructures. Figure 2 shows the multi-layer structure of AlGaN/GaN with sharp interfaces.

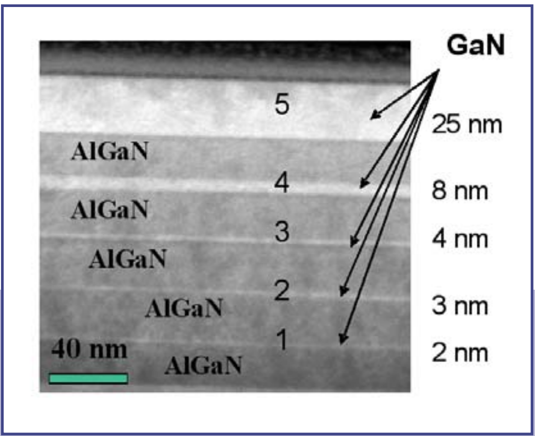


**Figure 2:** Pioneer Research on InGaN growth by HVPE

InGaN is one of the key compound semiconductor materials used for the fabrication of GaN-based blue, green and white LEDs and blue laser diodes. Most of the existing LEDs reply on MOCVD to produce the quantum well structures for the InGaN emitters. Recently, TDI has developed the HVPE technology to control the growth of InGaN to very low levels of about 0.5 to 1 µm per hour needed to make quantum wells structures. Figure 3 highlights the smooth surface morphology of InGaN grown by HVPE.

The development of InGaN materials, for the first time, will allow the fabrication of blue and green LEDs using the HVPE method. In fact, TDI has recently been awarded a significant funding by the DARPA VIGIL program to develop green laser technology based on InGaN-GaN materials

**Figure 3:** Optical image of In0.29Ga0.71N surface (image width 60 µm)

## Nitride-based Templates by TDI

The company produces a wide range of materials on different substrates, including the followings:

- GaN on Sapphire – Sizes from 2" to 4" – Ideal for Blue and White LED applications
- AlN on Silicon Carbide – Sizes from 2" to 4" – Typically used for RF electronic devices such as HEMT
- AlGaN on Sapphire – Sizes of 2" or 3" – Used in optoelectronic devices operating in UV spectral region
- InN on Sapphire – Research grade available in 2" – for work on sensors and high frequency electronic devices
- InGaN on Sapphire – Available in 2" for Green LED and green laser developments

The team at Silver Spring is proud to have joined Oxford Instruments and look forward to continuing support from their existing customers as well as discussing nitride requirements with a range of new customers.

# High deposition rate processing using ICP-CVD

**Owain Thomas PhD,** Senior Applications Engineer, OIPT

## Deposited films such as Silicon nitride and silicon oxide are used in HBLEDs to passivate the final devices.

Current methods include batch PECVD processing which has a typical load of up to 8 x 4” substrates or 40 x 2" substrates with a growth rate of 14-15 nm/min. Considerable amounts of interest have recently been directed towards single wafer LED processing which requires higher deposition rates to maintain throughput requirements. It is also known within the consortium, that the deposition temperature must also be kept low to achieve functional devices. These requirements restrict the ability of conventional PECVD which require high temperatures and low deposition rates in order to allow high quality material to be deposited, probably through allowing sufficient time for excess hydrogen to outgas from the growing film.

High density films can be deposited at low temperatures (<150°C) using the ICP-CVD technique but with typical deposition rates of 10nm/min. Recent development work at OIPT has achieved much higher deposition rates of > 140nm/min at the same low temperatures, whilst maintaining good film quality, film thickness uniformity and film stress control.

Presented here are additional ICP-CVD process repeatability tests which have been conducted by depositing high deposition rate $SiO_2$ (>140nm/min) at low temperatures (<150°C) on 75 x 100mm wafers. Results are shown in figure 1, 2, and 3 below.

Figure 1 below shows wafer to wafer deposition rate repeatability of <+/-2% with film thickness uniformity of <+/-3% over 100mm wafer.

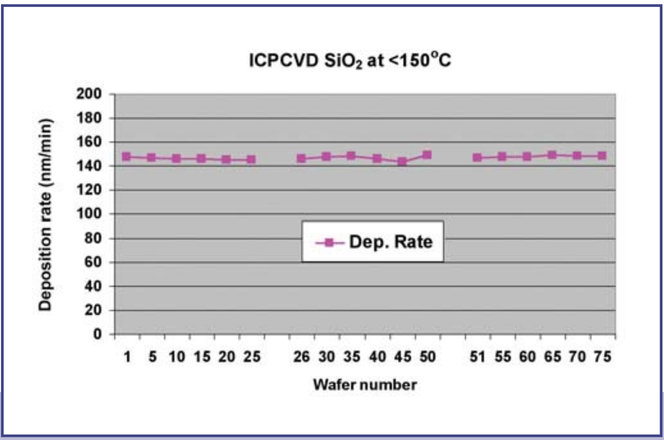


Figure 2 shows wafer to wafer refractive index repeatability of <+/-0.3%

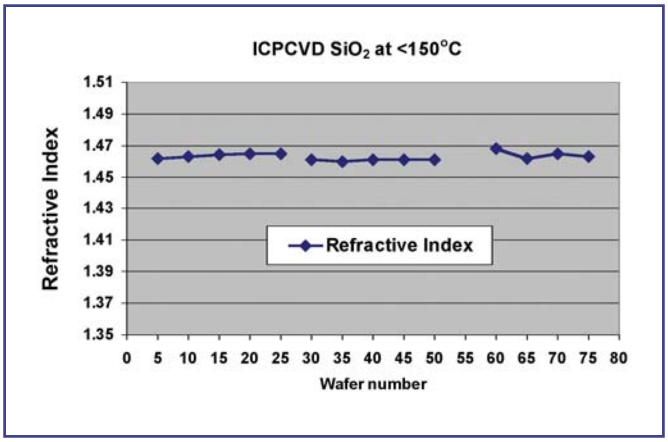
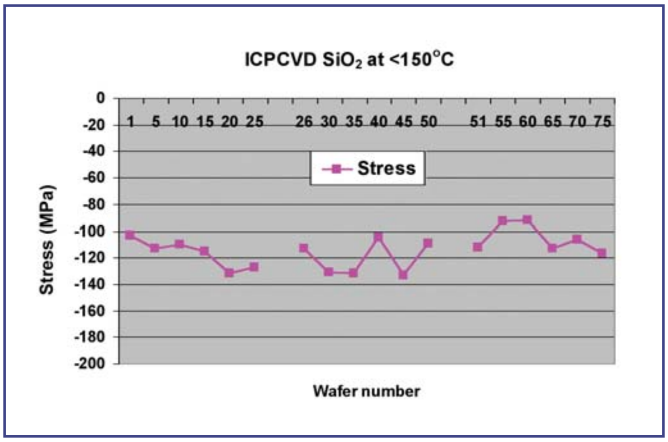


Figure 3 shows $SiO_2$ film stress of <150MPa compressive over 75 wafers.



These recent advances have shown the capability of ICP-CVD in achieving high quality films at low temperatures with high throughput.

Therefore due to these additional benefits of ICP-CVD several HBLED manufactures are now considering the ICP-CVD technique as an alternative to conventional PECVD.

# Low loss, high quality optical coatings using Ionfab®500Plus

**Sebastien Pochon PhD,** Applications Engineer, OIPT

## A high quality optical coating should have low optical loss. Losses inside an optical coating arise from scatter and absorption.

Ion beam sputtering produces films with total losses so low that sophisticated devices are needed to measure them. Data for the measurement of mirrors with losses less than 2ppm have been published by two independent groups. Both groups made their mirrors on Oxford Instruments Ion beam sputter deposition systems – the **IonFab**500Plus.

A major feature of an optical coating is its surface quality. The quality of the surface determines the performance of the optical device itself. High quality optical coatings start with a smooth super-polished optical substrate with roughness typically of 0.05 nm rms.

A conventionally deposited film will add roughness to the surface of the optical substrate, the degree of the roughness dependant upon the technique used. For example a film deposited by evaporative techniques produces a surface roughness of typically 1 nm rms, while ion assisted deposition techniques produce a surface roughness of typically 0.4 nm rms. Ion beam sputter deposition produces films with a surface roughness equal to that of the super-polished substrate, 0.05 nm rms.

**IonFab500Plus has been developed for customers demanding high throughput**

The figure below shows the spectrum centred at 633nm of a 36 layers mirror obtained with **Ionfab**500Plus tool. Total losses for $SiO_2/Ta_2O_5$ mirrors are less than 40ppm with repeatability of ±2%, <±0.001 R.I. and uniformity of less than ±2% across 10” planet and <±0.0005 R.I. Surface roughness increase <0.02nm RMS for initial substrates <0.07nm RMS. Loss readings are subject to suitable substrate and clean room conditions being of a suitably high quality. Oxford Instruments’ customers have reported achieving <20ppm mirrors in production.

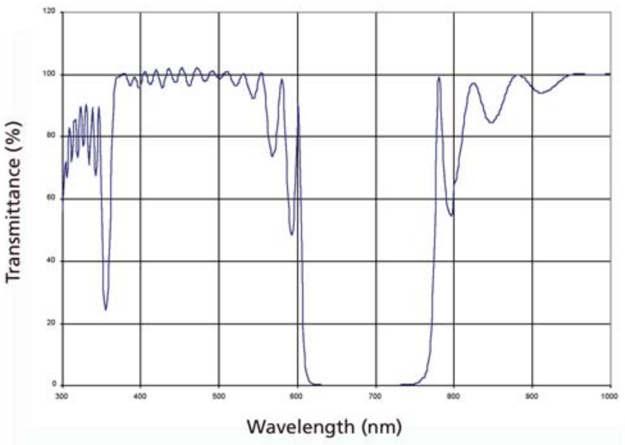


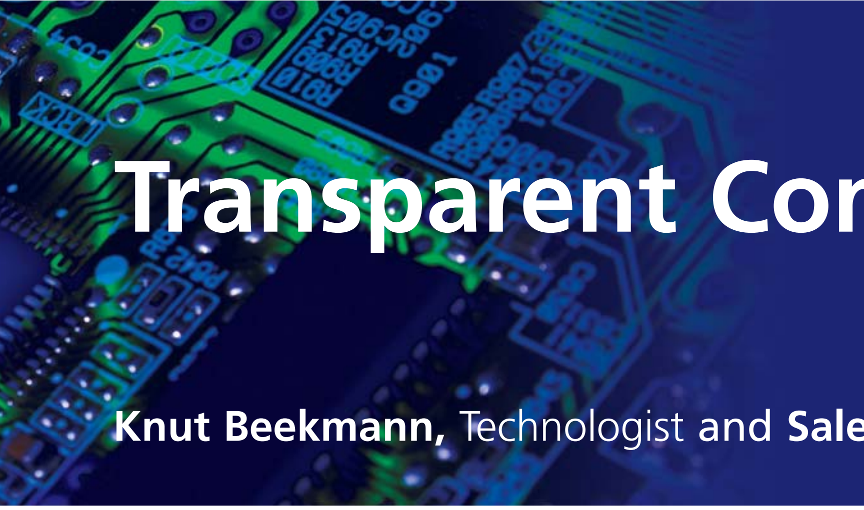
**Figure 1**: Spectrum of a 36-layer 30deg 633nm mirror

**Ionfab**500Plus enables excellent refractive index control and uniformity that provides high wafer yield.

## Significant orders received from the Russian market

We were delighted to win orders for six systems to equip a number of research & development facilities and manufacturing plants in Russia. The six systems ordered include both plasma deposition tools, and an **Ionfab**® ion beam system.

Mark Vosloo, Sales Director commented, *“We are extremely pleased to have entered the Russian market so successfully over the past few years, and these new orders only emphasise our ability to establish ourselves in an evolving marketplace. These are very significant orders for us, not only commercially, but also as an endorsement of Oxford Instruments’ position at the forefront of providing high quality, innovative process tools which are enabling the next generation of electronic and nanotechnology devices.”*



# Transparent Conductive Films

**Knut Beekmann,** Technologist and **Saleem Shabbir,** Applications Engineer, OI

## Introduction

Recent years have seen a dramatic growth in interest in transparent conductive oxides or TCOs. These materials are wide band gap semiconductors and have properties that are particularly suited for various high tech applications. They are found in solar cells, liquid crystal displays, and have found uses in gas sensors and for EMC and anti static shielding.

Indium Tin Oxide or ITO is one such TCO material with a band gap of ~3.7eV. ITO is of particular interest and is becoming a very important material in the manufacture of solar cells. Solar energy development and production is a high growth area in the technology sector. This is a direct result of the growing need to produce more energy from renewable sources in order to reduce world dependence on fossil fuels and the rising costs of extraction.

## Sputter deposition

Sputtering or physical vapour deposition (PVD) is the favoured method for depositing thin layers of ITO. Although films can be produced by reactive sputtering of an indium tin alloy target, such a process is difficult to control and suffers from poor repeatability. Oxford Instruments Plasma Technology has developed a DC magnetron PVD process to deposit ITO films using a typical commercially available ceramic oxide target. In this case, process parameters can be adjusted and therefore provide films with optimum properties such as low resistivity and high optical transmission in the visible and near infra red spectrum.

| Film Property | Value |
|---|---|
| **Film Thickness** | 100 nm |
| **Refractive Index** | 2.03 |
| **Deposition Rate** | 33 nm min-1 |
| **Transmission minimum (400-1200nm)** | > 82% (minimum @400 nm) |
| **Transmission average (400-1200nm)** | 89% |
| **Resistivity** | ~3 x 10-4 Ohm cm |
| **Roughness – Mean (Ra)** | 1.95 nm |
| **Roughness – Max height (Rmax)** | 14.65 nm |

**Table 1.** Summary film properties for PVD ITO

Important film properties such as the film crystal structure and composition determine the fundamental properties of ITO. The crystal structure is mainly dependent on the deposition temperature. Generally films deposited below 100°C are amorphous. Figure1 shows XRD data for a sample ITO film deposited at a temperature of 200°C indicating the presence of both (222) and (400) orientations.

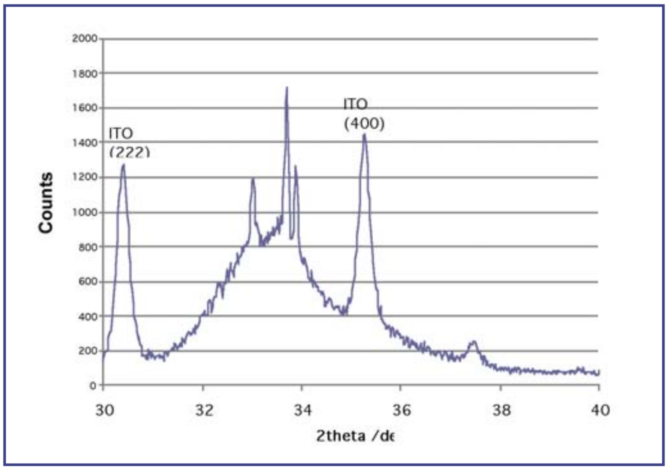
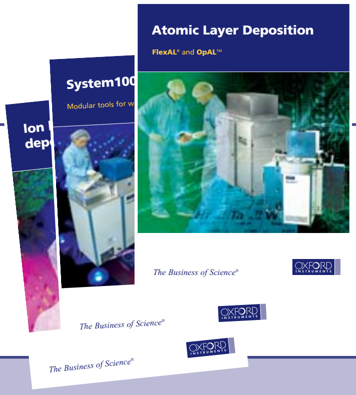


**Figure 1.** XRD analysis of ITO deposited onto thermal SiO$_2$

Optical transmission properties can be tuned by the addition of oxygen to the argon in the deposition process. The transmission has been measured in the wavelength range from 400nm to 1200nm. Processes without oxygen exhibit relatively poor transmission in this range however, as shown in Figure 2, low flows of oxygen produce films with significantly improved transmission.



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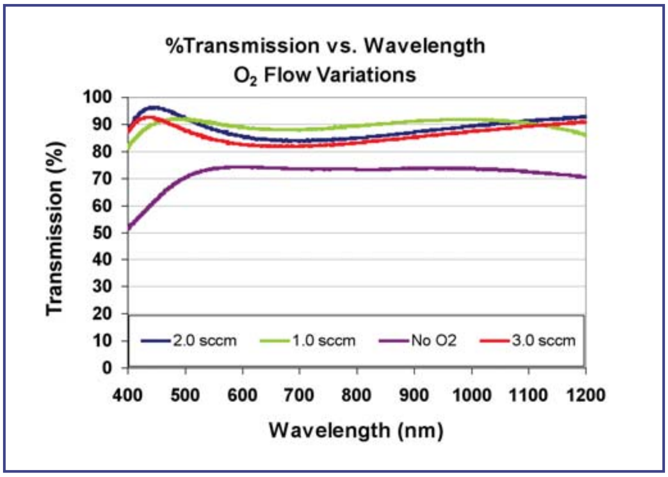


**Figure 2.** Optical transmission of ITO deposited onto glass

The oxygen flow also has an influence on the film resistivity. The general trend is for lower flow oxygen addition to produce lower film resistivity as shown in figure 3. It is therefore necessary to tune the oxygen flow taking into account both the transmission and minimum resistivity requirement.

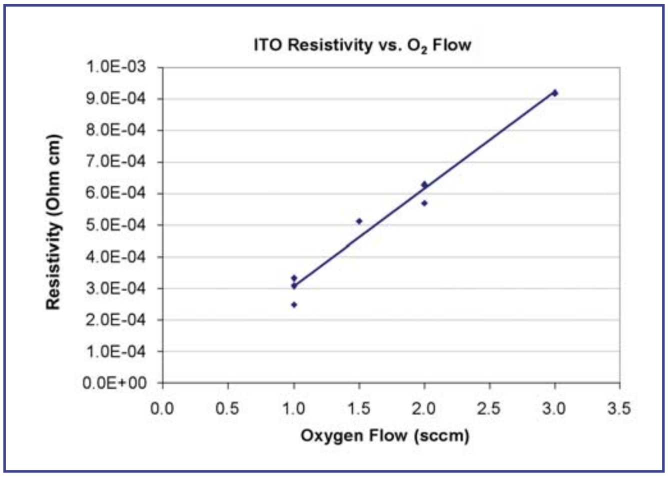


**Figure 3.** ITO resistivity of 100 nm films deposited onto thermal $SiO_2$

## Summary

Oxford Instruments Plasma Technology has developed a DC magnetron sputter deposition process that will be useful in various applications requiring good optical transmission and low resistivity. The process can be easily controlled by varying several process parameters in order to achieve optimum film properties.

**For further information:** Email: **plasma.technology@oxinst.com** Tel: +44 (0)1934 837 000 **www.oxford-instruments.com**

# Ion Beam Etching and Reactive Ion Beam Etching

**Sebastien Pochon PhD,** Applications Engineer, OIPT

Ion beam etching is a versatile etch process in which the substrate to be etched is placed in a vacuum chamber in front of the broad-beam ion source. Ions (typically argon) are generated inside the ion source and are accelerated into a broad beam, and to a defined energy, by the extraction grids on the front of the source.

As the ion beam etches the surface, the substrate is tilted to an angle in the beam and continuously rotated in order to optimize the uniformity of the etch. If a pattern is being etched by the use of a photomask, the use of tilt and rotation allows the user to adjust the wall angles in the resulting etch. If one uses an inert gas such as argon, the process is relatively slow, (typically 50 -100nm/minute,) and the heat that is generated must be removed with care, via He back cooling.

Ion beam processing has been transformed over previous years as OIPT continues to develop a range of inductively-coupled ion sources that in the case of the 35cm ion source allows the etch of substrates up to 8” diameter. The 35cm ion source uses a 2MHz RF generator and the 15cm ion source uses 13.56MHz. The ion source is virtually maintenance-free. This is in stark contrast to the earlier ‘Kaufman’ ion sources that were first used in the 1970s and had filaments that lasted as little as 10 hours.

The ion beam source is able to produce a collimated beam in combination with the tiltable platen control of its incidence at a substrate surface ranging from normal incidence to glancing one. Adding reactive gas such as $CHF_3$, $CF_4$ or Chlorine to argon in RIBE mode enables better control over selectivity between a mask and an etched material as well as an increase in the material etch rate. Increasing selectivity offers even more possibilities of etch structure with better control over sidewalls.

## The Ion Beam Range

**IonFab**®300Plus

**Etch & deposition processes in one tool**

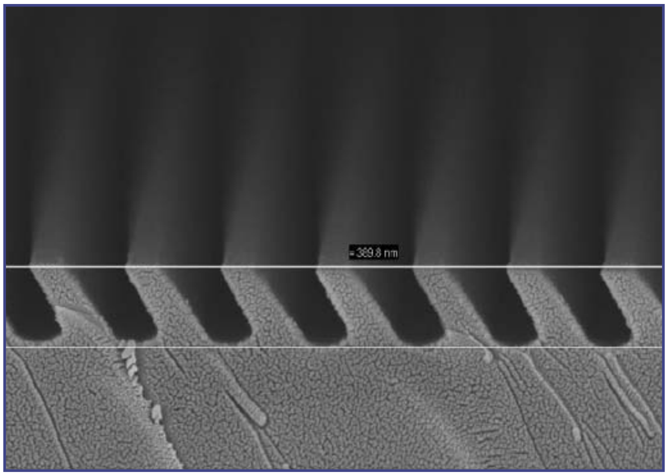
**Optofab**®3000

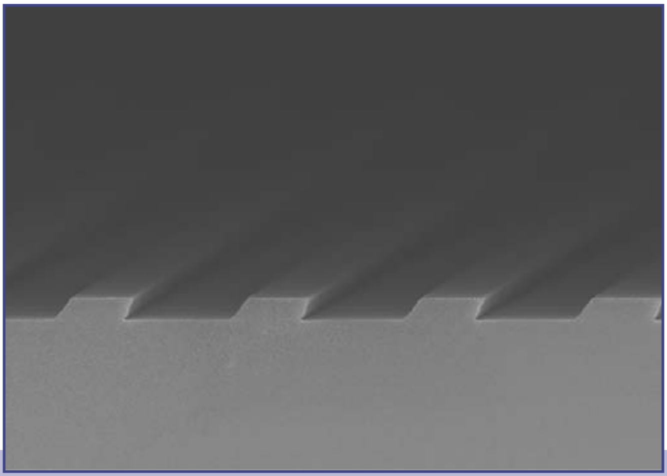
**Purpose made system for optical coatings**

**IonFab**®500Plus

**Specialist high precision ion beam deposition system**

RIBE can be used for manufacturing binary slanted gratings in large quantities. Their applications are high efficiency light in-and out-coupling with Plastic light guides. Light coupling is highly dependent on the grating period as well as the slanting angle. Sample tilting combined with RIBE offers endless possibilities in creating and controlling slanting etch angle which ultimately control grating optical properties [1]. Below are two different slanted gratings at various angles and periods of grating.





[1] Tapani Levola and Pasi Laakoonen, “Replicated slanted gratings with a high refractive index material for in and outcoupling of light”, Optics Express 2074, Vol 15, N°5 (**2007**).

# Ultra thin silicon nitride films by plasma ALD using Plasma FlexAL® and OpAL®

**Qi Fang PhD,** Senior Applications Engineer, OIPT

## Qi Fang discusses his successful development of an industry leading silicon nitride ALD process, and describes how to master this notoriously difficult process.

Silicon nitride is a well known material that played an important role in the microelectronics industry for many years; it is CMOS compatible, a good passivation layer, good diffusion barrier and exhibits low leakage. A wide variety of techniques such as PECVD, LPCVD and ICP-CVD have been employed to deposit silicon nitride, but with the current relentless drive towards nano-scale technology a new demand has arisen for ultra-thin and highly conformal layers of $Si_3N_4$. Atomic Layer Deposition is the ideal technique to meet these demands.

Silicon nitride has long been a challenge by ALD. By their nature ALD precursors tend to be highly moisture sensitive and the preferential reaction is always with oxygen containing species such as background moisture in the chamber. Oxford Instruments' applications engineers have invested a great deal of time and taken certain steps with the hardware to get from the starting point of $SiON_x$ with only 5% nitrogen to $SiN_x$ and only 3% oxygen; better than any published literature at this deposition temperature using a metal organic precursor.

The developed $SiN_x$ process uses an amino-silane precursor and nitrogen/hydrogen plasma as the two halves of the surface reaction. Comparatively few literature reports exist on $SiN_x$ by ALD, but those that do mostly use plasma ALD – from the literature and the author's own experience it is doubtful that satisfactory film quality can be achieved by pure thermal ALD methods and especially using non-chlorine based precursors at less than 400°C deposition temperature.

The refractive index measurement is very sensitive to oxygen contamination; a lower refractive index means more oxygen contamination in the film. Figure 1 shows the refractive index measured using in situ spectroscopic ellipsometry as high as 1.95.

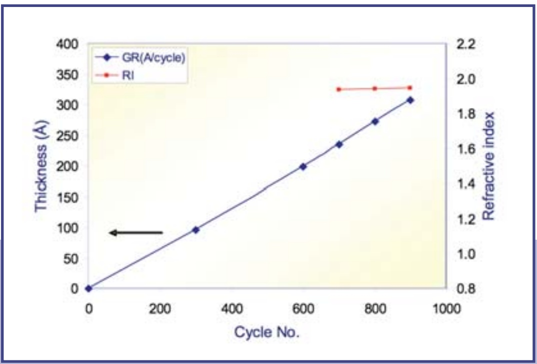


**Figure 1**: Refractive index measured by in situ spectroscopic ellipsometry at 1.95 for thicker films where accurate modelling is possible.

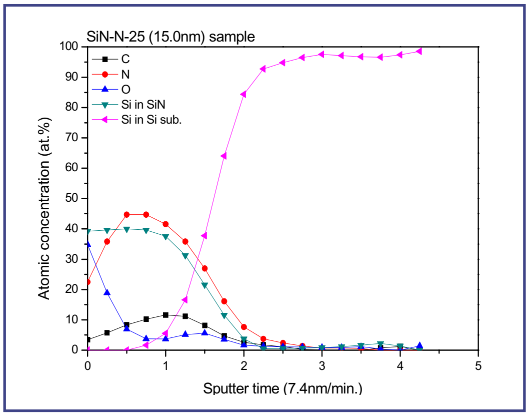


**Figure 2**: Showing $Si_3N_x$ with as low as 3% oxygen in the bulk.

The Auger Electron Spectroscopy trace (figure 2) of ALD $SiN_x$ deposited using 3DMAS at 350°C shows the ratio of N/Si in the $SiN_x$ film is around 1.1 and the carbon in the film is in a range of 5-10 atomic %. The oxygen (< 3% in the bulk) is observed mainly at the surface and interface of SiN/Si substrate (the wafers were not HF dipped). The wet etch rate of the $SiN_x$ films is comparable to those of PECVD deposited films at similar temperatures, see figure 3.

| Film | Wet etch rate |
|---|---|
| **$SiN_x$ (ALD)** | 7.7 nm/min |
| **Thermal $SiO_2$** | 60 nm/min |

**Figure 3**: Wet etch rates of $SiN_x$ are comparable to PECVD films deposited at a similar temperature. A thermal $SiO_2$ wafer was dipped for comparison in the 10:1 BHF solution.

Oxford Instruments is continuing to develop the $SiN_x$ process to lower the carbon and oxygen levels even further using alternative precursors which cannot be disclosed for commercial reasons.

**The $SiN_x$ precursor has also successfully been used to deposit $SiO_2$ by ALD with a stoichiometric ratio of Si:O at 1:2 and less than 3% carbon impurity.**

# Notch-Free Silicon-on-Insulator Etching in **Plasmalab**System100 **ICP Tools**

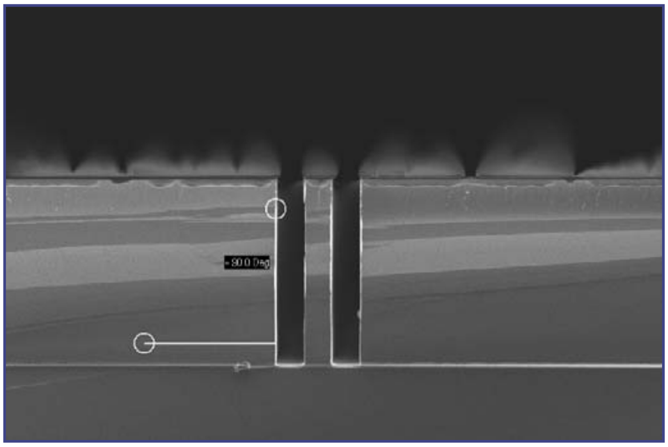
**Colin Welch,** Principal Applications Engineer, OIPT

The Bosch process is routinely used for the deep etching of silicon typically for MEMS applications (Micro-electro-mechanical systems).

One important aspect of MEMS technology is Surface Micro-machining involving a relatively thin (<100μm) device silicon layer above a thin buried insulator (usually $SiO_2$) on a thicker substrate wafer. The thinner SOI layer is structured to form high performance devices such as accelerometers. However etching the SOI down to the buried insulator presents an additional challenge: 'notching'.

**Sub-100nm sidewall roughness for Bosch DSE**

Notching means the profile of the SOI becomes strongly re-entrant and even undercut at the interface which is unacceptable to most applications. The notching effect is believed to be an effect of charging at the buried layer, although lateral surface diffusion of adsorbed chemicals may also play a role. Some over-etch is essential to be sure of completing the isolation etch of all features, but the excellent reproducibility and uniformity of the OIPT process helps to minimise that time. OIPT offer a process solution that maintain notches below 5% of the total etch depth, with a 0.5μm minimum notch undercut each side.



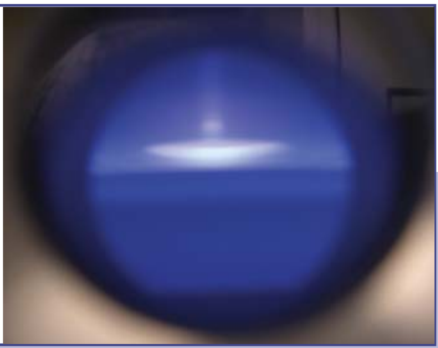
**Vertical Notch free trenches in SOI**

## LATEST UPGRADES

### Plasma Accelerator for Failure Analysis

The latest upgrade for dry etch deprocessing in semiconductor failure analysis (FA), the Plasma Accelerator for advanced die processing, has recently been released.

The Plasma Accelerator delivers increased etching speeds, simple operation and low damage. It supports a full range of dry-etch FA processes, including passivation removal, IMD (inter-metallic dielectric) and ILD (inter-layer dielectric) etch, ensuring a clean, smooth etched surface is produced with no metal de-lamination or erosion.



# Growth of Carbon Nanotubes with **Nanofab**800Agile™

**Cigang Xu PhD,** Development Scientist, OIPT

The Plasma Enhanced Chemical Vapour Deposition (PECVD) process has been employed for the growth of carbon nanotubes, in addition to techniques such as laser ablation, arc discharge and Chemical Vapour Deposition (CVD). PECVD tools provide more flexibility and control compared to a general CVD setup.

Oxford Instruments has launched the **Nanofab**700™ system for the growth of carbon nanotubes. In order to increase the temperature range and control on the ramping rate of temperature, a new system, **Nanofab**800Agile, used for the growth of nanostructured materials has been developed. In this system, the ramp rate can be higher than 40°C /min, and therefore decreases the overall process time. The system also has a loadlock that can provide a separate space for the sample to cool down without affecting the oncoming process runs in the process chamber.

Similar to Oxford Instruments **Nanofab**700 system, the Oxford Instruments **Nanofab**800Agile system has been designed specifically to provide control on the process conditions, such as the alignment of carbon nanotubes. Both the catalyst treatment and process can be performed in one process chamber, but the process and cleaning conditions vary between the systems, **Nanofab**700 and **Nanofab**800.

With the catalyst of 5nm Co on Si substrate, the aligned carbon nanotubes can be grown. Figure 1 shows the top view of carbon nanotubes, in which the nanotubes do not entangle together, as occurs in the samples grown by the thermal CVD process; Figure 2 shows the tilted view of carbon nanotubes, in which tips of carbon nanotubes are visible and carbon nanotubes are aligned!

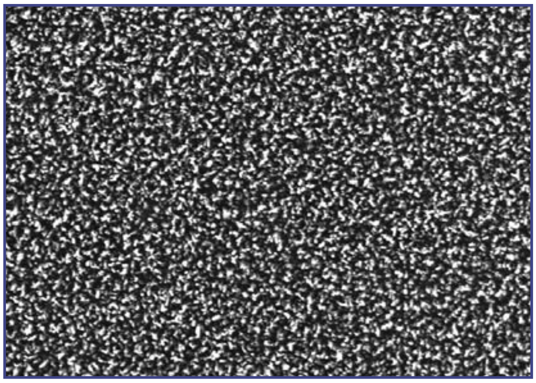


**Figure 1:** Top view of Carbon Nanotubes

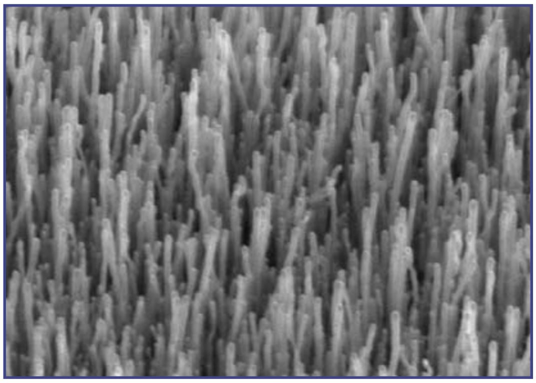


**Figure 2:** Tilted view of Carbon Nanotubes

## Customer Service and Support

Oxford Instruments Plasma Technology offers comprehensive training courses covering areas such as system operation, maintenance and fault finding, as well as giving you process tips. Why not book yourself and your colleagues on one now?

We also offer system Preventative Maintenance (PM) visits, where an OIPT engineer will visit your site and perform system maintenance on your behalf.

See **www.oxford-instruments.com/ptsupport** for further details and the full list of scheduled courses for 2008. If you wish to arrange a site visit - please contact your local support office.

# What's the wafer temperature?

**Mike Cooke** PhD, New Product Introduction Manager, OIPT

## Ask any chemical engineer what's the most important process variable, and they are likely to reply, 'temperature'.

When we plasma etch a wafer, or deposit a layer by PECVD, is the temperature just as critical? In plasma processes, most of the chemistry is driven not by the surface temperature, but by the electron temperature in the plasma. The electrons behave like a separate gas, co-existing with the neutral gases flowing into the chamber, but only weakly coupled to that gas. Because the electrons are charged particles, they pick up energy from the applied RF voltage, which they shed in collisions with neutral particles.

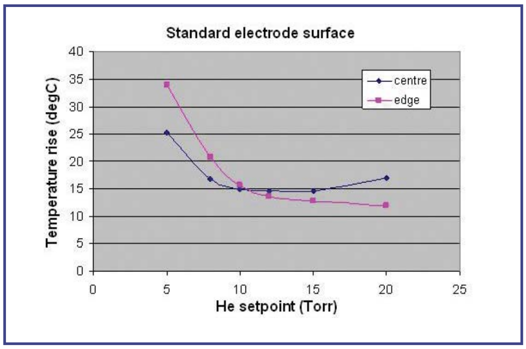
These collisions drive chemical reactions by splitting up molecules into highly reactive radicals. The electron temperature is in the range 10,000 – 40,000K, which is so far above the wafer temperature that the wafer temperature is less important. Energy is also supplied to the surface by ion bombardment, where the impact energy lies between 20eV – 1000eV, far above the thermal energy of atoms, even if the wafer is at 1000K.

But that's not the whole story. Surface processes include:

- Adsorption of gases and radicals
- Surface diffusion
- Reaction
- Desorption of reaction products

The reaction process is dominated by the plasma energies, and desorption can be stimulated by ion bombardment. But the surface temperature strongly drives adsorption, diffusion and desorption, especially on sidewall surfaces which are not strongly bombarded by ions.

While measuring and even controlling the wafer temperature may be the holy grail, the industry currently works only on the table temperature under the wafer. The wafer temperature then depends on the heat flux (either heating or cooling the wafer) and the degree of thermal coupling between the wafer and the table. Radiation coupling is weaker at lower temperatures (especially with silicon, which is fairly transparent to infra-red below 500K). At process pressures below 1 Torr, there is little conduction through the gas between the table and the wafer, so OIPT offers 'helium backside cooling'. In this technique, the wafer is clamped to the table (either by electrostatic clamping or by a mechanical clamp), and 5 -20 Torr of helium gas is maintained behind the wafer, with pressure control and flow monitoring. This pins the wafer temperature close to the table surface, even in the presence of high heat fluxes from the plasma.

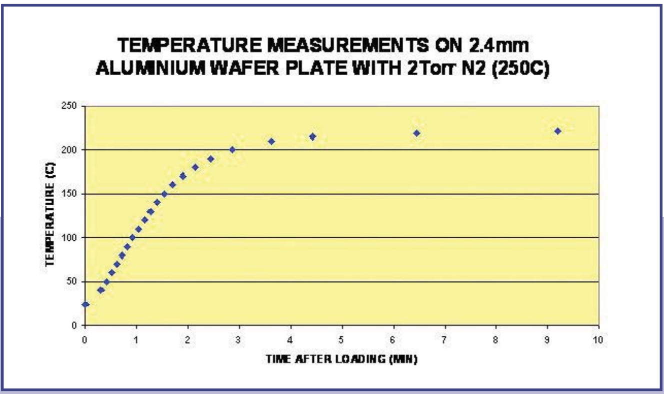


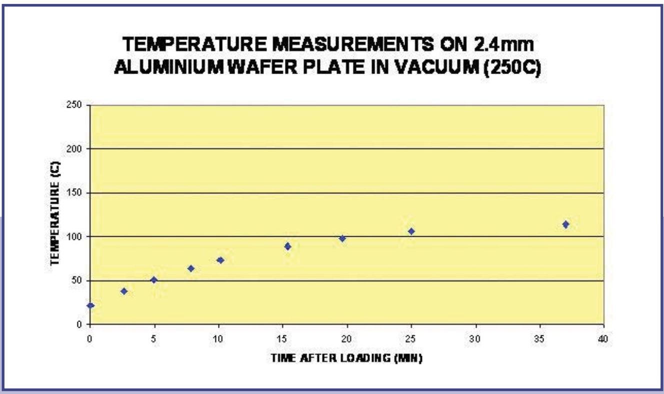
**Temperature rise of the wafer surface on a 6" wafer using mechanical clamp and standard (flat, smooth) electrode surface. Ar 100 sccm, 12mT, 2000W ICP, 0W RIE, 20°C**

In PECVD applications, the process pressure is high enough to deliver similar temperature differences between table and temperature, without needing the extra heat transfer gas feed. We have shown that it is very beneficial to raise the pressure after loading a wafer, to improve heat transfer and decrease stabilisation time. This is especially true if a carrier plate is used, because of its higher thermal capacity. Without this step, we have shown that the plate temperature deviates from the table temperature by more than 10°C. With a 2 Torr stabilization step, the plate temperature settles within 30°C of the table temperature.

## Summary

Wafer temperature does matter, even in a plasma process. A reproducible thermal stabilization history is necessary for a reproducible substrate temperature. A well-characterised heat transfer environment is essential.





# Oxford Instruments Group

## NanoAnalysis

Oxford Instruments NanoAnalysis provides an industry-leading range of accurate, fast and easy to use tools for materials analysis on an electron microscope.

## Industrial Analysis

Oxford Instruments Industrial Analysis provides materials identification and/or thickness gauging analysis instrumentation to industrial customers with diverse needs.

## Plasma Technology

Oxford Instruments Plasma Technology provides a range of high performance, flexible tools to semiconductor processing customers involved in research and development, and batch production.

## NanoScience

Oxford Instruments NanoScience provides high performance environments for low temperature and high magnetic field applications in physical science research and process development down to the atomic scale.

## Molecular Biotools

Oxford Instruments Molecular Biotools provides tools and systems based on magnetic resonance technology to advance the understanding of bio-molecular structure and function.

## Superconducting Technology

Oxford Superconducting Technology supplies world-leading superconducting wire for use in medical and scientific applications. It provides service and support for MRI customers in USA and Japan.

## Austin Scientific

Oxford Instruments Austin Scientific provides robust, high quality cryogenic vacuum pumps, helium compressors and cold heads for customers in the semiconductor, medical and research sectors. It provides high quality support, refurbishment and maintenance services for all cryogenic system users.

**For further information:** Email: **plasma.technology@oxinst.com** Tel: +44 (0)1934 837 000 **www.oxford-instruments.com**

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# Events roundup

Oxford Instruments Plasma Technology will be exhibiting at the following:

**34th International conference on MICRO & NANO engineering**
15 - 18 September 2008
Athens, Greece

**E-MRS Fall Meeting**
15 - 19 September 2008
Warsaw, Poland

**ESSDERC**
15 - 19 September 2008
Edinburgh, UK

**19th MicroMechanics Europe Workshop**
28 - 30 September 2008
Aachen, Germany

**Semicon Europa**
7 - 9 October 2008
Stuttgart, Germany

**International Workshop on Nitride semiconductors**
6 - 10 October 2008
Montreux, Switzerland

**AVS 08**
19 - 20 October 2008
Boston, MA, USA

**34th ISTFA**
4 - 5 November 2008
Portland, Oregon, USA

**MRS Fall 2008**
1 - 4 December 2008
Boston, MA, USA

**Semicon Japan**
3 - 5 December 2008
Japan

**Semicon Korea**
20 - 22 January 2009
Seoul, Korea

**Japan Nano**
18 - 20 February 2009
Japan

**Semicon China**
17 - 19 March 2009
China

**OIPT/Southampton University Seminar**
2 April 2009
Southampton, UK

# Successful 'Plasma Etch Tech 2008' seminar and workshop at Caltech

The Kavli Nanoscience Institute, California Institute of Technology held a highly successful seminar and workshop in July 2008 at the Caltech, California facility with Oxford Instruments.The wide and varied programme proved to be a huge success, and participant numbers exceeded all expectations.

The two day event started with Presentations, Panel Discussions and a Networking Lunch on day one. Key guest speakers included **Dr. Ivo Rangelow**, *Technical University Ilmenau, Plasma Simulation Program* who spoke on 'Plasma Processing Simulation Platform', **Dr. Axel Scherer**, *Neches Professor of Electrical Engineering, Applied Physics and Physics, Co-Director, Kavli Nanoscience Institute, Caltech* whose topic was 'Dry Etching of Highly Anisotropic Optical Structures', **Dr. Deirdre Olynick**, *Staff Scientist, Nanofabrication Facility, Lawrence Berkeley National Lab*, whose talk covered 'Nanoscale Pattern Transfer for Nanoscience', and **Dr. Michael Roukes**, *Professor of Physics, Applied Physics, and Bioengineering; Co-Director, Kavli Nanoscience Institute, Caltech,* discussed the Advances in Nanoelectromechanical Systems.

Speakers **Dr. Oskar Painter,** *Assistant Professor of Applied Physics, Caltech,* **Robert Gunn**, *Application Team Leader, Oxford Instruments Plasma Technology,* **Scott Sitzman,** *Application Scientist, Oxford Instruments NanoAnalysis,* and **Dr Alex Buxbaum** of *FEI* gave an insight into other key aspects of Plasma Etch.

The workshops in Caltech's laboratories on Day Two were split into four groups:

1. DRIE etching of silicon microstructures for MEMS applications – **Plasmalab**100 ICP 380 for MEMS
2. Cryo-etch of silicon for optical waveguides and mirrors - **Plasmalab**100 ICP 380 – with wide range temp electrode (-150C to 450C)
3. Low-T CVD nitride and oxide growth – **Plasmalab**100 PECVD
4. Anisotropic GaAs etching with halogen (Chlorine) gas chemistries – **Plasmalab**100 ICP380

With well over 60 people attending this was a very high level event, which could not have been so successful without the hosts and invited speakers.

Oxford Instruments will be planning another such event in California in July 2009, and also is running a similar seminar at Southampton University, UK in April 2009. For information on these and other future workshops please email: **susie.williams@oxinst.com**

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- **HVPE Tools & Substrates**

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