CENTRE FOR PROCESS ANALYTICS AND CONTROL TECHNOLOGIES

CPACT NEWSLETTER August 2009

APACT 09



The APACT 09 conference took place in Glasgow at the Hilton Hotel on 5-7 May 2009.

The feedback received has been excellent. A few quotes from the delegates:

"Great conference, friendly atmosphere and enjoyable social programme"

"APACT09 provided a great opportunity to network with past and future colleagues and clients in a friendly and comfortable environment. Most enjoyable".

A total of 35 presentations were given over the three days of the conference. There were 3 plenary and 8 keynote talks. 97 delegates attended from countries including Denmark, France, Germany, Netherlands, Spain, Sweden, Switzerland, UK and USA.

12 vendor companies exhibited and 22 posters were presented. A CD containing most of the presentations was compiled and included in the delegate packs distributed at the conference. The presentations are now also available on the apact website (www.apact.co.uk).

Poster prizes were awarded to Peter Hamilton, CPACT Strathclyde (1st Prize) for his poster entitled "Evaluation of particle size measurement techniques in relation to powder drying" and Tapiwa Mutasa, University of Strathclyde (2nd Prize) for his poster entitled "Process Intensification using high intensity focussed ultrasound techniques".




Clairet Continues to Expand in all Dimensions!

Time Domain: In April Clairet Scientific celebrated 15 years of applying spectroscopy to give useful information. From small beginnings in Northampton in 1984, Clairet is now well established in the world of process spectroscopy and PAT.

Frequency Domain: Clairet has always prided itself in covering the whole absorption spectrum (Ultra Violet → Visible→ NIR→ MidIR) & Raman and has recently extended that range even further with a move into the rapidly developing technique of terahertz spectroscopy (ultra Far IR). Radiation from this region of the spectrum (30 cm^{-1} - 130 cm^{-1}) is able to penetrate packaging and surface coverings to both image and obtain spectra of the layers underneath (see Figure 1). In addition, data collection is rapid (kHz) and so many samples can be analysed per second.

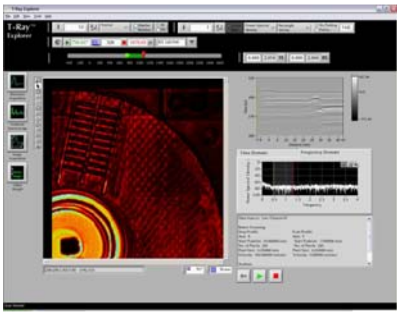


Figure 1: Terahertz image of a floppy disk showing detail through the plastic cover
(Image courtesy of Picometrix Inc.)

Figure 2: Extrel process mass spectrometer
(Image courtesy of Extrel)

Sensitivity: The range of concentrations that spectroscopic techniques can cover has also been expanded for gas analysis. Process Raman and NIR methods typically cover concentration ranges from 100% to a few 100 ppm. UV, visible and mid IR spectroscopy cover the ppm range. Clairet has recently started working with Extrel quadrupole mass spectrometers (Figure 2) giving us access (in the process & in real time) to concentrations as low as 10 ppb. Feed and off-gases from many different processes (e.g. fermentation, drying, ambient air monitoring, ammonia production, etc.) can be monitored in real time to very low levels with high accuracy.

The range of applications of process Raman Spectrometry has been extended with the Airhead Raman gas cell (Figure 3). This allows the routine analysis of process gases in hot and potentially hazardous processes. The innovative design of the probe head allows the analysis of gas phase samples from 100% to a few hundred ppm. This now permits the monitoring of homonuclear diatomic gases such as H_2, N_2, O_2, Cl_2 and F_2 as well as other inorganic and organic species (Figure 4).

Figure 3: The Airhead Raman gas cell
(Image courtesy of Kaiser Optical Systems)

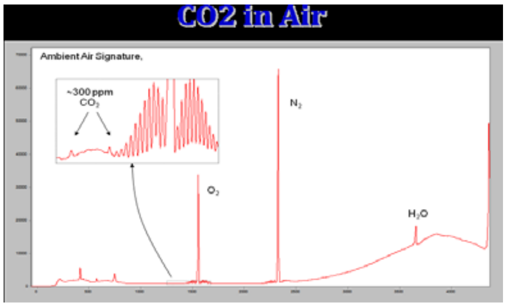


Figure 4: Raman spectrum showing 300 ppm CO_2 in ambient air.
(Image courtesy of Kaiser Optical Systems)

We would like to thank all our past colleagues and friends and are looking forward to stretching ourselves even further in the next 15 years.

Paul Dallin (paul.dallin@clairet.co.uk)

NEW APPROACHES TO INFRARED ABSORPTION SPECTROSCOPY

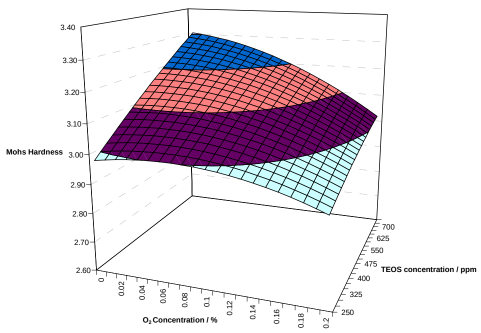
Dr Philip Martin, School of Chemical Engineering and Analytical Science, University of Manchester,
philip.martin@manchester.ac.uk

Our group in Manchester have been working for several years with tunable infrared lasers as sources for absorption spectroscopy in process analysis applications. The high intensity, coherent, infrared beams with high spectral resolution often enable changes in absorbance in the range 10^{-4} to 10^{-7} to be routinely measured with no moving parts but at the expense of narrow wavelength tuning ranges. In many ways they are complementary to traditional techniques such as Fourier transform infrared spectroscopy (FTIR) where broad spectral ranges can be covered but with lower sensitivity.

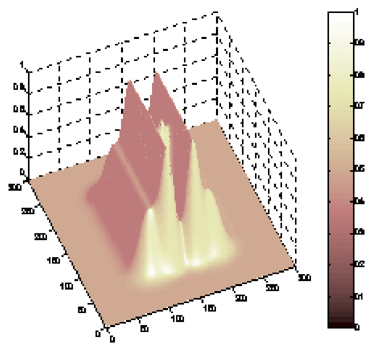
Many of the advances in the process analysis area have come from the application of near-infrared ‘telecom’ lasers. Each laser is, effectively, capable of monitoring a single gas species such as ammonia, methane, hydrogen chloride and oxygen but the combination of low noise sources and detectors leads to high sensitivity measurements despite the weaker overtone and combination band absorption lines available. Additionally, they are easily coupled to optical fibres to split the beam into multiple measurement points and also to measure at long distances. Our group have also enhanced their capability by multiplexing several lasers together to measure multiple species simultaneously. These are becoming generally accepted now in industrial environments and are supplied commercially by several companies.

In the mid-infrared range, different tunable laser options are available: quantum cascade lasers (QCL), lead-salt diode lasers, difference frequency generated lasers (DFG) and optical parametric oscillators. Again many of these have narrow wavelength tuning ranges but can access to the fingerprint region and also stronger absorptions are possible. The technology is developing rapidly and external cavity QCLs can scan over 150 cm^{-1}, for example, enabling condensed phase measurements.

An example application where we have used different mid-infrared (QCL, DFG and Pb-salt diode) and near-infrared tunable lasers in addition to FTIR and optical emission spectroscopy is plasma-enhanced chemical vapour deposition (PECVD). Atmospheric pressure PECVD can be used to manufacture functional coatings and films on heat sensitive substrates such as plastics. In this example silica films as barrier coatings have been deposition on glass substrates. The combination of different spectroscopic techniques enables the concentrations of several species to be determined both *in situ* in the reactor but also in line, both at the inlet and exhaust of the reactor. These can be combined with other process parameters and then correlated with the properties of the resulting thin film such as growth rate, surface roughness, hardness etc. A response surface methodology has been developed for optimisation purposes.



The figure shows the response surface for film hardness as a function of O_2 concentration and precursor concentration.



A second example, again from the chemical vapour deposition area but this time thermally activated, demonstrates the use of near-infrared laser absorption spectroscopy to determine spatial concentrations of key species in a reactor. In the atmospheric pressure open reactor, several integrated line-of-sight absorption measurements of HCl (a product species) were made. By making the measurements at multiple angles, the optical density at each point can be determined by tomographic reconstruction techniques. To determine the actual point concentrations, the temperature field must be known. We are developing methods to do this non-invasively from the temperature dependence of the water vapour absorption spectrum but we have also used computational fluid dynamics simulations. In principle, full 3D determination of chemical species can be extracted by these techniques for monitoring and control purposes.

In conclusion, we have shown that new developments in infrared laser technology can be used to enhance process analysis both by complementing traditional techniques such as FTIR but also to develop new capabilties such as spatial measurements and the detection of short-lived intermediates.

Dr Nordon gets Fellowship Extension

Congratulations to Dr Alison Nordon (Department of Pure and Applied Chemistry, University of Strathclyde) who has been awarded a three year extension to her Royal Society University Research Fellowship. Alison was originally awarded a Royal Society University Research Fellowship for a period of 5 years in October 2004 to work on 'new acoustic paradigms for non-invasive chemical process characterisation'. The aim of this research is to advance the use of acoustic techniques for process monitoring and control. As the information obtained using acoustic techniques is complementary to that obtained via optical techniques, the research is also focusing on the development of methodologies for the combination of acoustic and optical data.

University Research Fellowships provide an opportunity for scientists to dedicate their time solely to research and thus allow individuals to build an independent research career. Further information on the Royal Society University Research Fellowship scheme can be found at www.royalsoc.ac.uk

FUTURE EVENTS

CPACT Research Day at Newcastle University on Wednesday 23rd September 2009.

Following the success of the last Research Day at the University of Strathclyde in Glasgow, it was decided that we hold another event prior to the next CPACT Steering Committee meeting in September.

Further details will be sent out shortly, in the meantime, please put this date firmly in your diaries.

CPACT TEAM

Julian Morris
Technical Director
CPACT Newcastle
E: julian.morris@ncl.ac.uk
T: 0191 222 7342

Angela Bott
Administrator
CPACT Newcastle
E: a.m.bott@ncl.ac.uk
T: 0191 222 5785

Natalie Driscoll
Team Co-ordinator
CPACT Strathclyde
E: natalie@cpact.com
T: 0141 548 4836