KRATOS ANALYTICAL
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NEWS 02 2020

A QUICK HELLO

Welcome to the second Kratos newsletter

We were really pleased with the feedback we received after the circulation of our first newsletter in April. We've had suggestions as well as lots of ideas for items to include. We'll develop these ideas and regular features as we publish more. Our second newsletter sees us focus on Australia! This was prompted by an order for a fourth AXIS Supra on the continent and also the retirement of Dr Barry Woods after 50 years at the University of Queensland, the last 18 years of which he ran over 40,000 samples on an AXIS Ultra^{DLD}. It seems an appropriate time to spotlight our instruments and Users 'down-under'. In this Aussie special, the i-work interview is with one of our service engineers and a Q&A interview with our User at Queensland University of Technology (QUT). There's our regular 'Tips, Tricks and Thoughts from the Applications Lab' as well as a retrospective on surface analysis in Australia.

We hope you find something to interest you and we appreciate continued feedback.

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TIPS, TRICKS AND THOUGHTS FROM THE APPLICATIONS LAB.

In writing this, we hope to give some insight into things that we do in the applications lab that might help our Users in their data acquisition and processing. If you'd like to share something, why not contact us and we'll publish the best of them.

IT'S ALL ABOUT THE SAMPLES

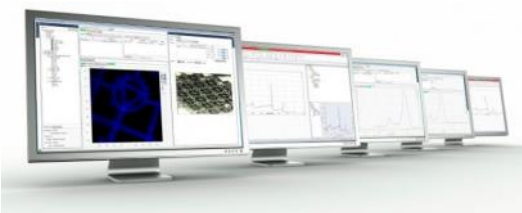
Sample mounting is often overlooked, but can be significant in defining the ease and quality of spectra and images acquired. For example, did you know that there are a series of ISO standards describing appropriate sample handling practices? I'm sure that our applications lab. is not the only one that has received samples for analysis with fingerprints, or supplied nice and clean in a plastic bag? Whilst the problems associated with measuring organics and salts from fingerprints or silicones from plastic bags are well known to the time-served X-ray photoelectron spectroscopist, they may not be to the owner of the sample. A good starting point in educating those supplying samples for analysis is the publication by Baer *et al.* 'Practical guides for x-ray photoelectron spectroscopy: First steps in planning, conducting, and reporting XPS measurements'.

So appropriate sample handling prior to analysis is extremely important, but so is the form or nature of the sample. Is it actually compatible for XPS analysis? If the sample is not UHV compatible, is cooing available to freeze it or lower the vapour pressure sufficiently? Will it physically fit into the spectrometer or does it need reducing in size without destroying the region or chemical species of interest? These are all questions that we need the owners of samples to consider before performing XPS analysis.

A further consideration prior to analysis relates to sample mounting. Typically, samples are mounted using vacuum compatible double sided tape, or where vacuum cleanliness is a serious consideration, mechanical clips or screws can be used to hold the sample in place. Either approach places the sample in electrical contact with the sample holder which ensures that the peaks appear at the correct binding energy for metallic or conducting samples. For wide bandgap, insulating samples, this method of mounting will have no influence on the peak positions as the charge neutralisation system will determine the surface potential. When the sample is not full conducting, however, mounting the sample in electrical contact with the sample holder can lead to differential charging, even when the neutraliser is used. For this type of sample, we recommend mounting the sample 'floating'. That is, not in electrical contact with the sample holder. This can be easily achieved by using non-conducting (Kapton) double-sided vacuum compatible tape. This method of sample mounting ensures that the sample as a whole behaves as an insulator and the charge neutraliser fixes the surface potential of both insulating and conducting regimes of the sample during analysis. So if in doubt, mount it floating! For the interested reader, the subject of charge neutralisation is discussed in greater detail in this recent publication.

ESCApe 1.4 RELEASE AND DISTRIBUTION

At the time of publication of this newsletter, ESCApe 1.4 is due for imminent release. This is the latest version of our acquisition and processing software for AXIS Supra, Supra^{+} and latest generation AXIS Nova instruments which are already running ESCApe software.



The software is available for ESCApe Users to download from the members' area of our website (https://www.kratos.com/members). To access this area, you must have a valid login. To help with security and GDPR compliance, we have made it necessary to reregister for a members' area password.

Instructions for User installation of ESCApe 1.4 are included in the download file. To help us better support you and your instrument, it is important that all ESCApe Users upgrade to the latest version as soon as possible.

USERS' MEETING 2020 ANNOUNCMENT

We've re-scheduled our biennial Users' Meeting for the **week of the 21st September**. In line with most meetings in 2020, this will be a virtual online meeting. We're still in the planning stages but we will have contributions from both Users and Kratos staff. Due to the change of format, it's likely that we will have shorter 2 or 3 hour sessions spread over the week. As there will be fewer slots available for oral contributions, we're also planning to have an online poster session.

If you have attended an online conference or meeting over the last few months and have suggestions for a format that worked well, we would love to learn from your experiences. We want to make our virtual Kratos Users' Meeting as successful as possible.

AND FINALLY...

Congratulations to Dr Jonathan Counsell, who many of our Users will know from the Applications Team. He was recently awarded the Vickerman Prize by the UK Surface Analysis Forum Committee in recognition of his work which will have a major impact in the field of surface analysis. Well done JC!

i-work

Interview with an employee

Editors note: In keeping with our Australian themed news letter, we've found a half Aussie Kratos employee to interview!

Name **Kevin Sykes**

Job title Customer Support Engineer – Surface Analysis

How long have you been at Kratos? Just over 17 years.

How would you describe your job to a 5-year-old?

I fix broken things and try to make the customers happy. A happy customer is more likely to buy from us again and will also tell others how good we are.

Customers want many things like information and advice, but they also want their Instrument to work and work reliably. Sometimes they want an upgrade or to take advantage of a new technique. Other times they want they just want their Instrument moved to a new lab, city or even country!

Best part of your job?

With all the travelling I do I have a great deal of autonomy and this gives a great sense of freedom – as long as you get the job done of course. It also means you have to think 'on your feet' and be self-sufficient.

Another important benefit of all this travel is meeting the customers, both old and new. This exposes me to many different cultures and working practices.

How did you end up at Kratos, your background and experience?

I really started out as an Electrical/Electronic Design Engineer working on BIG Flexographic Printing Presses – 50 m long, 5 m wide and 3 stories high! Next I was a Test and Installation Engineer working on metalizers. These had BIG vacuum chambers with massive pumping groups. The chambers were 3 m diameter by 5 m long and you walked inside the chamber to service them! After that I became an Implant Engineer working in silicon wafer fabrication facilities – think really BIG Ion Guns, 1.7 MeV!

The common theme here being travel and customer support, it's no wonder I became a Customer Support Engineer for Kratos!

What have you learnt working at Kratos?

Paperwork – seriously! Keeping good accurate records is really important when working autonomously as it allows other members of the team to continue helping customers whilst I am not in the country.

Oh, and quite a bit about XPS!

You travel a lot for your job, which country is your favourite?

I get asked this a lot, usually soon after meeting a new customer. This can be a genuine question or a trick question but the answer always has to be the same— "it depends upon what you mean by best? The best for scenery, holidaying, cuisine, safety, working, living, travelling, value, friendliness…" The list goes on. I never usually answer the question!

What is your motto or personal mantra?

"Best for our customers."

What keeps you busy when you're not at work?

House, family and model railways. When working away, I do quite a bit of design work on my model railway: Circuit designs, PCB layouts, code generation, research and now CAD/CAM for my CNC Router.

Tell us one thing that we don't know about you?

Whilst getting a hold of Vegemite is not a problem, I do miss "Lamingtons"!

Surface Analysis—An Australian Perspective

After a discussion with Dr Thomas Gengenbach, Dr Barry Woods and Prof Paul Pigram

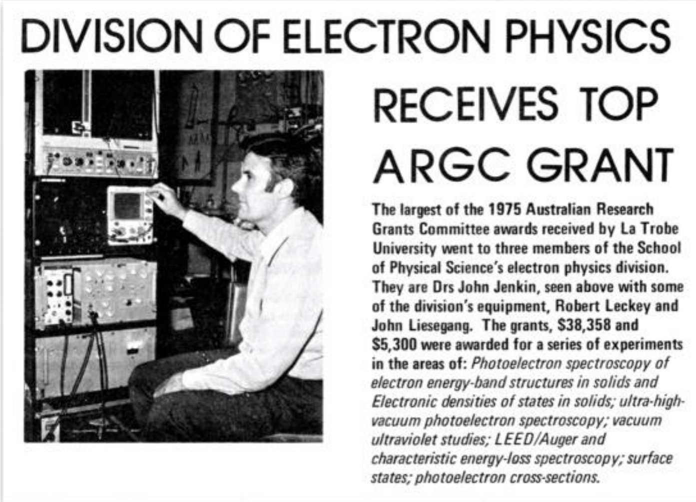
'In some respects, we're at a crossroads in Australian surface analysis' states Dr Thomas Gengenbach. When I formulated the idea to review surface analysis in Australia it was prompted by the retirement of Dr Barry Woods after a 50 year career at the University of Queensland. I wanted to look back at some of the history of XPS and surface analysis 'down under'. But as you'll read in this article, by looking at the past, our discussions quickly and easily turn to the future.

So, what of the crossroads? Thomas elaborated that 'because for most of the past, XPS and surface analysis in general, were done in groups that had established expertise and experience. However in the last 5-10 years, new groups are purchasing XPS but perhaps don't have that expertise. Groups see the need for XPS as it is a useful tool in many areas, but it is difficult to establish that expertise'. This is not only limited to Australian surface analysis but is true of the Global community and comes with the maturity of the technique. The young researchers that were at the forefront of surface analysis in the late 1960s and early 1970s are now at retirement age, and it is with the retirement of these researchers that we run the risk of losing a huge wealth of experience.

Barry remembers early in his career working in the group of Prof Lawrie Lyons. He was involved in the purchase of a chemical analysis instrument for the study of CdS/CdTe thin film solar cells. The instrument they purchased in 1982 was a Physical Electronics 560 SIMS costing Aus$ 154k. However, there was not sufficient funding to purchase the Multi Analytical Computer System (MACS). This was purchased in 1984 for $122k AUD. It seems almost inconceivable that the acquisition and processing computer could cost nearly as much as the hardware of the spectrometer! It meant that initially spectra were acquired on chart recorders. This concurs with Thomas' recollection of his early VSW instrument at LaTrobe. 'No computer at all. Just a chart recorder with spectra printed on a roll of paper'. In writing his own software in the early days, Thomas suggests that 'it really teaches you something. You have to learn about the algorithms and understand how they are used and where they might not be good'.

It's never been easier to acquire X-ray photoelectron spectra and images, but it doesn't correlate that the quality of data published has improved.

Whilst it's never been easier to acquire X-ray photoelectron spectra and images, it doesn't correlate that the quality of data published has improved. Could it be the disconnect between the data and its processing caused by modern PCs with latest generation software that contributes to presentation of erroneous data? It is not correct to lay the blame solely at the ease of data acquisition using PCs. Indeed, the *benefits* of using computers for materials surface science are easy to demonstrate. The use of computers for furthering materials characterisation has been embraced by groups such as Prof Paul Pigram's at LaTrobe. By setting up a state-of-the art visualisation and instrument remote access facility, the teaching and access to surface analysis instrumentation has been extended to a wider community both in Australia and internationally. As Paul highlights, his latest research interests involve development of artificial intelligence and machine learning which can offer great insights to large and complex datasets associated with materials characterisation which were not previously possible.


DIVISION OF ELECTRON PHYSICS

RECEIVES TOP ARGC GRANT

The largest of the 1975 Australian Research Grants Committee awards received by La Trobe University went to three members of the School of Physical Science's electron physics division. They are Drs John Jenkin, seen above with some of the division's equipment, Robert Leckey and John Liesegang. The grants, $38,358 and $5,300 were awarded for a series of experiments in the areas of: *Photoelectron spectroscopy of electron energy-band structures in solids and Electronic densities of states in solids; ultra-high-vacuum photoelectron spectroscopy; vacuum ultraviolet studies; LEED/Auger and characteristic energy-loss spectroscopy; surface states; photoelectron cross-sections.*

Looking back at early surface analysis in Australia, Thomas remembers 'when I started, I had some experience but not a lot. At the centre at CSIRO, I had good mentors and had time to read and learn about the techniques, but modern facilities seem to be under greater pressures and don't have that luxury of time'. Indeed, that's probably a widely recognised problem. As the technique of XPS has matured and the workplace has developed, it's rare to find technical experts whose time is solely dedicated to data acquisition and analysis. XPS is now a widely used technique used for surface characterisation of an ever-increasing array of simple to complex materials. As such the journals where XPS results are published are becoming more diverse. This has its own problems where peer-reviewed articles find their way to publication without the XPS results being properly scrutinised. Indeed, more than half of the scientists responding to a survey that was published in 2016 indicated that there was a "significant reproducibility crisis" in science [1], so it is recognised as a generic problem.

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Expectations also shift. Anyone running an XPS instrument has surely experienced the colleague who walks into the lab with a sample in one hand and a reference in the other, asking for similar data to be acquired from their sample. On looking at the sample, the finger-prints over it suggest that the analysis could prove inconclusive at best and more likely useless, and to compound this, on reading the literature provided, it's evident that the XPS results are not worth the paper they're printed on! This view was backed by Barry who remembered providing some good data acquired from a sample to a young PhD student. 'He went away and spent days peak fitting his data, making reference to published Ni 2p spectra. When he came back to ask me to review the peak fits, I was horrified to see he'd fitted the F Auger transitions, mistaking them for the Ni 2p doublet.! He hadn't thought to identify all of the peaks in the survey spectrum and therefore missed the significance that the sample was heavily contaminated with Fluorine'.

This is a multi-faceted problem but an easy solution is education and training. Thomas thinks that this 'comes down to people with the right attitudes looking after young scientists, guiding them and introducing them to the technique.' In parallel to this, there's also a requirement to document good working practices for XPS and surface analysis. This was the motivation for the series of 'Practical guides for XPS' written by Don Baer and co-authors and published in JVST [2]. In writing these practical guides, we're trying to condense the decades of experience of researchers and experimentalists, such as Barry and Thomas, so that the next generation of X-ray photoelectron spectroscopists and materials scientists can learn and develop applications of XPS to modern materials characterisation.

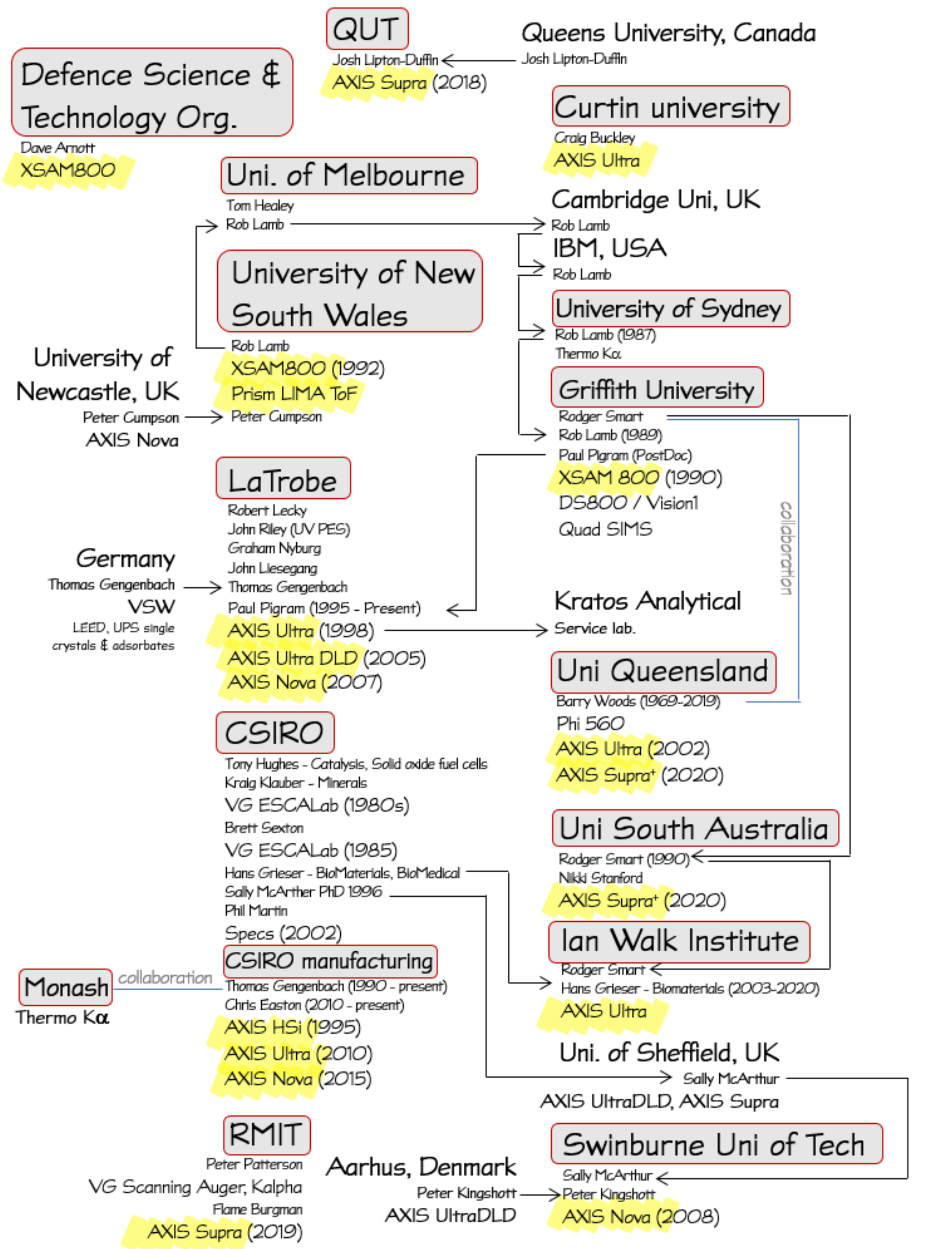
So, as we stand at this crossroad, it's hoped that we follow the path signposted by the knowledge of those that are now moving towards retirement. And perhaps pause and follow the advice stuck to both Kratos spectrometers in Thomas' lab: "This machine has no brain, use your own!".

References

[1] Baker M, Nature, 533, 452–454 (2016)

[2] D.R. Baer *et al.*, J Vac Sci Technol A., 37, 031401 (2019) doi: 10.1116/1.5065501

Australian surface analysis family tree*



* apologies for any omissions & inaccuracies

MEET OUR USERS

Dr Josh Lipton-Duffin, QUT, Brisbane, Australia

Dr Josh Lipton-Duffin works at Queensland University of Technology's (QUT) Institute for Future Environments (IFE) in the Central Analytical Research Facility (CARF). He's cross-appointed in the Science and Engineering Faculty's School of Chemistry and Physics.

What is your role at QUT?

My current role at QUT is titled as Senior Research Officer for Surface Science; this is an academic role with a heavy technical element. My main duty is to push innovative use of the surface science technology at QUT, not only in terms of my own research, but also to connect potential users who may not have had the background or experience to understand what surface analysis could do for their research. I find it both challenging and rewarding as I often end up far out of my depth in discussions on topics where I have no scientific background, so I get to learn from the users just as much as they learn from me. In practical terms, I am responsible for a pretty diverse portfolio of instruments, from ambient scanning probes, to the Kratos AXIS Supra, to an elaborate UHV SPM/PES playground system.

Can you describe a typical day at work?

There's not really any such thing as a 'typical' day for me – I might find myself training users, performing analyses for clients, writing papers or grants, teaching courses, or crawling underneath microscopes to troubleshoot a problem. It's never boring, although sometimes a little bit hard to plan my time. I try to keep to what the calendaring app says, but that can often get thrown out the window when unexpected things happen in the lab.

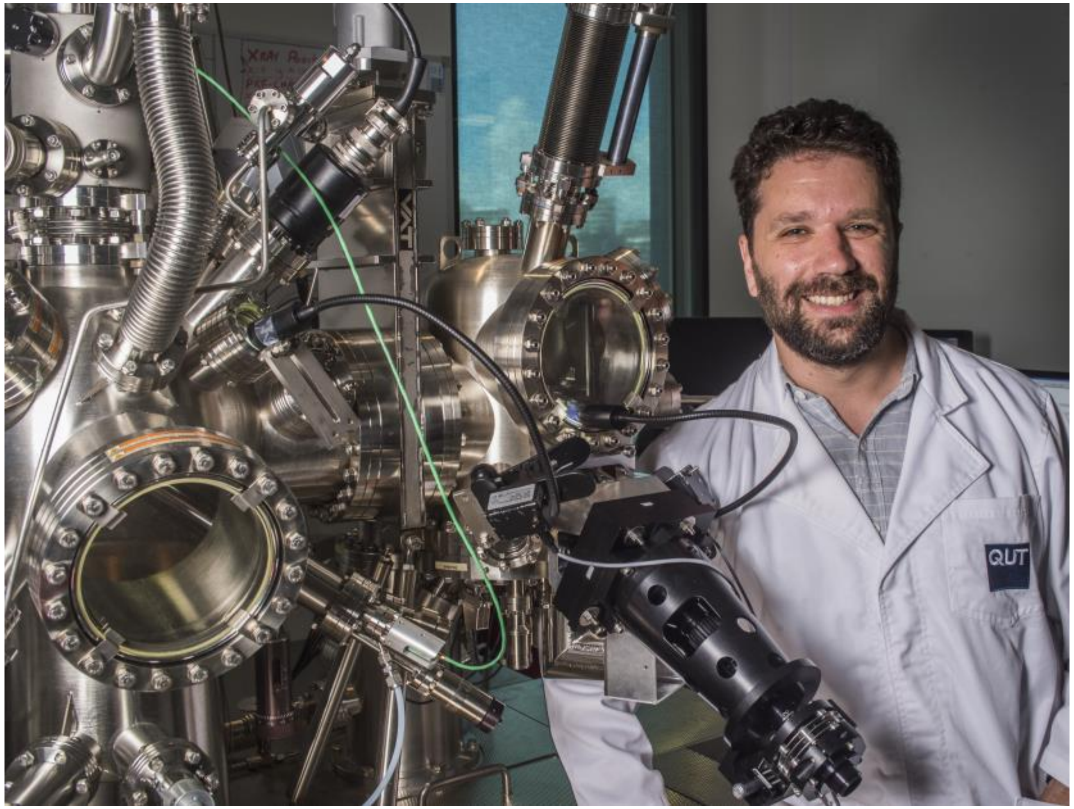
How do you use your Kratos instrument in your role?

The AXIS Supra is QUT's first real analytical XPS machine, and I have to say it's a great instrument for a first foray into the technique, and definitely a trainer's dream. On the Supra I can get most users to basic competence within a couple of hours as there's really very little damage to be done. Most issues that arise with the machine can be solved remotely, so I rarely have to leave the office to bail anyone out. By contrast I came up in a lab where everything was a homemade rig, designed to do one measurement exceptionally well, but with virtually no automation or labour-saving devices (I suppose that we, the graduate students, were the automation layer). We were really fortunate that the University recognised the value of XPS, both in terms of increased publication output and increased engagement with industry, and thus financed the instrument directly!

Our Supra gets used for a whole range of different samples – QUT has real research strengths in soft matter, biofabrication, and device technology, so we tend to see a lot of bespoke polymers, 3D printing materials and a lot of thin films and layered compounds. Our instrument runs pretty much 24 hours a day, with most users having mastered the ESCApe queuing system well enough to know how to load and submit their samples without disturbing the previous user's runs.

What do you see as the value of surface analysis?

From a mechanistic view surfaces are where the action happens. The way in which materials



"Surfaces can present a whole slew of odd and surprising behaviour"

interact is via their surfaces, so all fields of chemistry, catalysis, epitaxy, and even biology to an extent are inherently surface-driven. It's important to have tools to study surfaces exclusively because the physics are simply different on a surface than in the bulk of a material. Everyone understands surface to volume ratio goes up as dimensions shrink, and by extension the trend towards miniaturization means that surfaces and interfaces are more important than ever before.

What has surface analysis taught you?

I guess surface analysis has taught me patience and scepticism! Surfaces can present a whole slew of odd and surprising behaviour that can also double as instrumental artefacts, and the data does not come quickly. Nevertheless, it's highly rewarding to do surface analysis and it can really connect you to many different fields of science that you might not have otherwise had the opportunity to cross.

Any tips or tricks for surface analysts?

My supervisor used to tell me that three hours in the lab can save you one hour in the library! A corollary to that is that the *analysis* time is generally much longer than the *acquisition* time. So make sure the measurements you are doing are really worth both your time and the instruments'. And don't skip the survey spectrum!

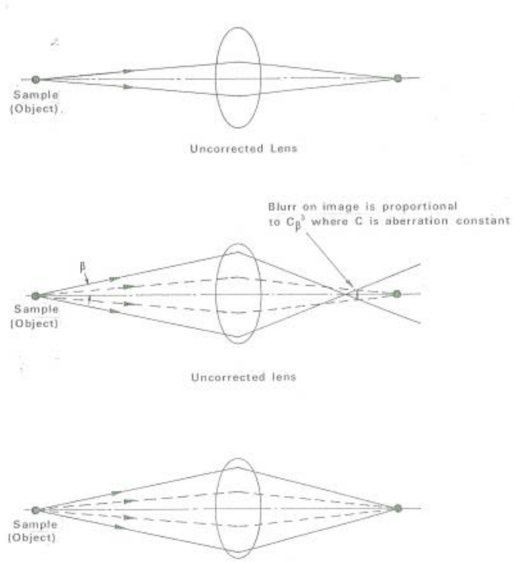
THE ABERRATION COMPENSATED INPUT LENS (ACIL®)

Following on from our look at the origins of Kratos Analytical in our last newsletter, this short article highlights one developments which has proved to be significant in the design of Kratos photoelectron spectrometers; the Aberration Corrected Input Lens, or ACIL for short. Indeed, the principle of keeping the aberrations of the input lens low remains at the heart of our current AXIS spectrometers .

In 1984, Kratos Analytical launched the XSAM series of X-ray photoelectron spectrometers. Fifteen years after the first commercial X-ray photoelectron spectrometer, the XSAM would go on to define Kratos' role as a leading supplier of surface analysis instrumentation. Analysis requirements for chemical information from a localised sample area led to the development of the unique microfocus X-ray source for small area 'focused' XPS capabilities (SAXPS). Further innovation was provided by the advanced Aberration Compensated Input Lens (ACIL) giving the ability to 'select' a small analysis area. The XSAM brochure states that a combination of the monochromatic X-ray source, ACIL, 180 degree hemispherical analyser and multidetector system ensured that the XSAM provided good signal to background ratios, high sensitivity and high throughput with excellent resolution. Claims that are as relevant today as they were in 1984!

The importance of the aberration correction is shown in light optical terms, which are analogous to electron optics, in the figure below. In brief, the aberration correction ensures that the photoelectrons which enter the electron transfer lens are all focussed at the same plane. For the XSAM, this would be at the entrance slit to the hemispherical analyser. The aberration correction is fundamentally important for ensuring good spectral energy resolution for electrons collected from a large solid angle. The ability to collect photoelectrons from a large solid angle meant a significant increase in the overall sensitivity of the spectrometer.

Effect of Lens Aberration in Optical Terms



Aberration Compensated Input Lens

The ACIL lens also meant that data could be collected from large or micro-selected areas down to 100 µm diameter. Consequently, the XSAM was one of the first X-ray photoelectron spectrometers with small area analysis capabilities. As the analysis area was defined by the ACIL system, selected area spectroscopy was possible with either monochromated or brighter, non-monochromated X-ray sources. For the Mg Kα source, running at 450 W large area performance gave a specification of 340 kcps at 0.82 eV FWHM on the Ag $3d_{5/2}$ peak, increasing to 6.3 Mcps at 1.3 eV FWHM.

It is however the performance of the Al monochromatic source that catches the eye. Data from our archive shows that typical performance of the XSAM using Al Kα monochromatic excitation was approximately 400 cps. This suggests that acquisition times for 'real' samples would have been significant and demonstrates the remarkable improvement in performance of modern AXIS instruments.

The final iteration of the successful XSAM series was the XSAMi, with its pioneering "imaging XPS" capability combined with multi-technique feature. The capability of the ACIL led naturally to the introduction of the scanning system as foreseen in the original ACIL patent. XPS maps were acquired by rastering the collection area of the photoelectrons across the surface of the sample. Importantly, the capabilities of the ACIL provided the analyst with an easy to use microprobe-like feature, enabling exact correlation of classic spectroscopic analysis with XPS and physical images. This was the advent of spatially keyed spectroscopy, where an area of interest is identified from a map of the lateral distribution of an element.

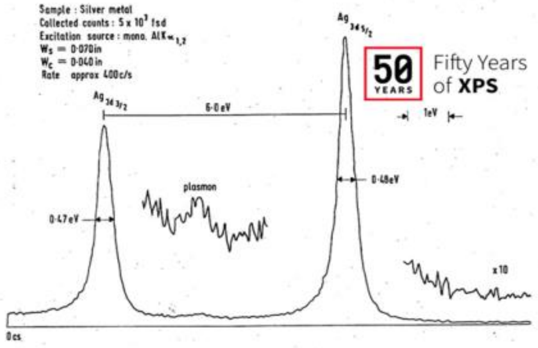


Fig. 1 High resolution XPS spectrum of Silver using the monochromator