

Tips, Tricks & Thoughts from the Apps. Lab.

A little of what we know

i-work

Interview with an employee

Building spectrometers during a pandemic

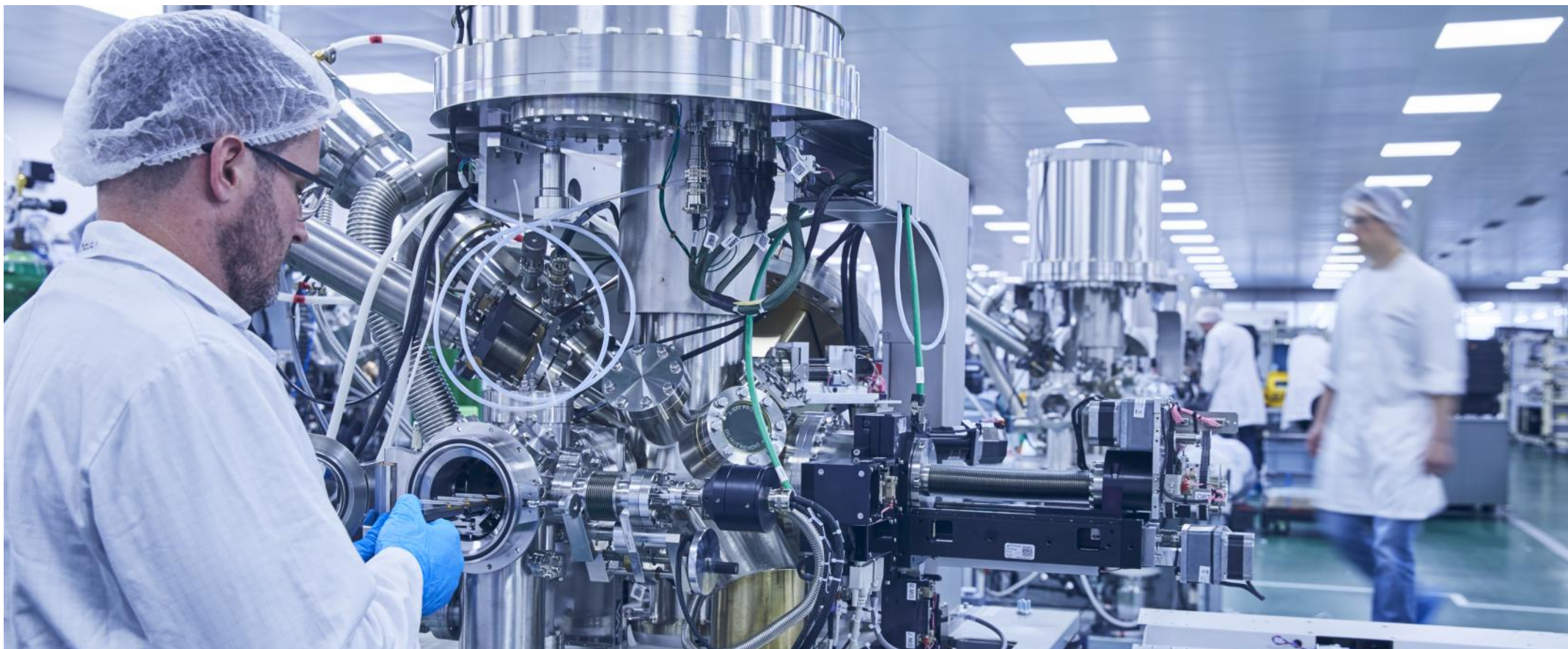
The 150th AXIS Supra is shipped to China

Meet our Users

Dr. Andreas Holländer

Looking back at development of Kratos spectrometers

Delay-line detector



WELCOME TO THE SPRING KRATOS NEWSLETTER

Adjusting to the new 'normal'

Whilst it has not quite been business as usual over the last 12 months, Kratos colleagues have adapted to constraints imposed by the global pandemic. As a manufacturing business, it's important that our site has remained open over the last 12 months. We have limited the numbers of staff on site at Kratos. They have continued to test and ship Kratos instruments. About half of our staff are working from home

with online meetings becoming the norm. Without being able to travel to customer sites we also continue to provide the best remote service support possible. All these constraints make it even more impressive that last month we shipped the **150th AXIS Supra** to its new owner.

In this newsletter, you'll find interviews with one of our German Users as well as one of our Test & Installation Engineers. There's an article on the development and introduction of the delay-line detector as well as Tips, Tricks and Thoughts from the Applications Lab.

We hope you find something to interest you and would really appreciate any feedback you would like to give.



info@kratos.co.uk



www.kratos.com

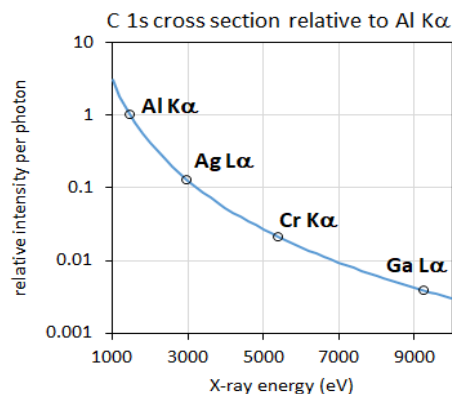
TIPS, TRICKS AND THOUGHTS FROM THE APPLICATIONS LAB.

In writing this, we hope to give some insights 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 any of your own advice with our User community, why not [contact us](#) and we'll publish the best of them.

HAXPES

In our recent poll on LinkedIn over 75% of respondents considered the photon energy required for hard X-ray photoelectron spectroscopy (HAXPES) to be greater than 5 keV. By implication this means that the Ag L α source for AXIS spectrometers is still considered to be in the soft X-ray regime, along with more conventional Al K α (1486.6 eV) and Mg K α (1253.6 eV). As we know, using the higher photon energy excitation has the same advantages of increased sampling depth, access to higher binding energy core levels and 'shifting' of Auger transition overlaps with core-level photoemission peaks.

One of the less obvious considerations in using higher photon energy sources relates to the decrease in the photoionisation cross section with increasing photon energy. This is clearly demonstrated by the plot of C 1s cross-section relative to Al K α excitation for the currently available excitation sources [1]. This is an important consideration in designing experiments using the Ag L α excitation source. It's likely that acquisition times for narrow region scans of transitions that we routinely acquire in several



minutes with Al K α X-rays will have to be significantly longer. However, the increase in collection time can be mitigated somewhat by acquiring regions with higher pass energy to increase the sensitivity. It is also noted that for elements Aluminium to Chlorine, the 1s core level and associated KLL Auger electron peak appear as strong features in the Ag L α excited spectrum.

To ensure quantitative results using the Ag L α excitation source it is important that the **instrument transmission function** has been recorded and the correct **relative sensitivity factors (RSFs)** are used. The determination of the transmission function and RSFs appropriate for AXIS instruments is presented in a paper by Shard *et al.* [1]. The supplementary information distributed with the publication allows download of the RSFs for use in generating quantitative results with alternative processing software.

For further insight on this subject, the open access article [Hard X-ray photoelectron spectroscopy: A snapshot of the state-of-the-art in 2020](#) is a recommended read [2].

[1] A. Shard *et al.*, Surf. Interface Anal., 2019, 51, 7, 763-773.

[2] Curran Kalha *et al.*, 2021 J. Phys.: Condens. Matter in press.

ION SOURCE ALIGNMENT

A significant contribution to a successful sputter depth profile is ensuring that the ion source is aligned with the analysis position.

Alignment can be checked by one of two methods. After ensuring that the sample is at the correct analysis height, it is possible to image the low KE (400 eV) secondary electrons generated by the ion beam using parallel imaging mode. Alternatively, running the ion source with no raster to etch a spot on a material that shows a colour change when sputtered, such as oxidised copper or a thin tantalum oxide film, allows quick alignment with the XPS analysis position.

When generating a depth profile, the widely accepted guideline is that the sputter crater dimensions are 10x greater than the analysis area. Following this advice, combined with correct alignment, will ensure that the XPS measurements are acquired from the bottom of the sputter crater. In the applications lab. we routinely use the 110 μ m aperture which gives good sensitivity whilst limiting the analysis area, allowing use of a 1.5 mm raster. Good alignment between the ion beam and analysis position becomes vital when profiling with 20 keV cluster ions as the *maximum* raster size for these very high energy ions is *ca.* 2 mm.

AND FINALLY...

On 31st March we said **farewell to Dr. Simon Page** as he

started his retirement. Some of our Users know Simon and have had correspondence



with him in his role as Engineering Manager and laterally, Manager of the Physics Group. Even if you didn't know Simon, you have direct experience of his contributions to Kratos Analytical. Simon started his career at Kratos 33 years ago and was inventor of the spherical mirror analyser introduced on the AXIS Ultra which is still used in the current generation of spectrometers. As you can read later in this Newsletter, he was also co-inventor of the delay-line detector. Simon has been at the heart of instrument development since the 1990's. During his career it is estimated that Kratos have sold in excess of 600 instruments, of which the AXIS Ultra alone has generated over 30,000 publications. Our spectrometers have contributed to the development of new materials as diverse as catalysts for green chemistry to novel bio-materials within academic and industrial settings. This is a fantastic legacy which we're sure we can continue thanks to the foundations that Simon has laid. We wish Simon the very best in his retirement.

i-work

Interview with an employee

Name Dr Alex Tonkins

Job title Test & Installation Engineer

How long have you worked for Kratos?

I have been at Kratos for nearly 7 years.

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

If you think of our product as if it were a guitar, I am the guy that lovingly adjusts the strings so that it can make beautiful music.

What makes a good test engineer?

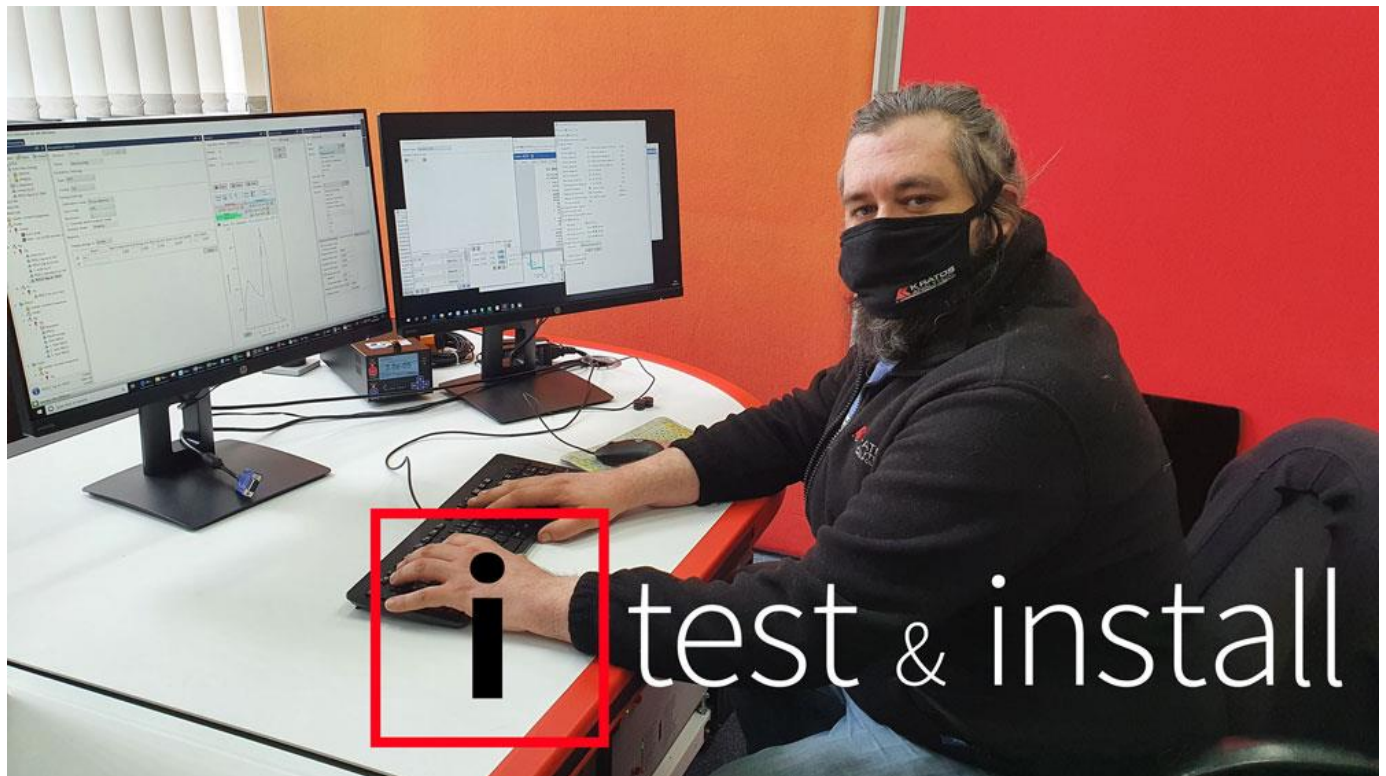
Personally I think that a conscientious attention to detail is a great asset. Getting the small details right is essential for precise scientific instrument. Being able to think out of the box is important and so is effective communication between team members. Our team has such a wealth of knowledge and if we put our heads together, there is little we cannot solve.

Best part of your job?

I love travelling the world, meeting our customers, and leaving them happy with their purchase.

How has the pandemic affected your job.?

I think that Covid-19 has forced us to re-evaluate the way we communicate both within the company and also with our customers. By the smart utilisation of information technology, we have been able to remain cohesive and focused regardless of physical location. We are at our best when we collaborate and over the last year we have developed many new strategies to achieve that aim.



How did you end up at Kratos? Your background/experience?

I studied chemistry and physics to a PhD level at Cardiff University and afterwards worked within the synthetic chemistry sector for many years, but eventually returned to my original passion of physical chemistry thanks to the opportunity given to me by Kratos.

What have you learnt working at Kratos?

To be flexible and think on my feet, and also that it's okay to rely on the wealth of expertise around me provided by my talented colleagues.

Your favourite quote / line from a film?

I really struggled coming up with one that wasn't expletive laden or morbid but here goes: Fight Clubs' **Tyler Durden**: "I

say never be complete, I say stop being perfect, I say let... let's evolve, let the chips fall where they may."

What is your motto or personal mantra?

"You cannot achieve anything without first taking action."

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

Currently I am spending a fair amount of my free time house hunting, although I do love a bit of Minecraft when I have the chance. When things go back to normal I am looking forward to going to some live music events and watching some stand up comedy.

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

I can do the splits!

BUILDING SPECTROMETERS DURING A PANDEMIC

The 150th AXIS Supra built shipped is shipped to China

As you'll appreciate, building an AXIS Supra⁺ is complicated. Compound this complexity with a global pandemic and the added difficulties due to leaving the EU and you might expect the task to become impossible!

Thankfully, this has not proved to be the case over the last 12 months. We have been busier than ever at Kratos and recently shipped our 150th AXIS Supra to its owner.

But how does an AXIS Supra⁺ go from a bill of materials on paper to a fully tested and installed instrument on a customer's site?

The starting point of all instruments is the sales forecast. This allows colleagues in Purchasing and Materials Management to ensure that all components required to build the instrument are on hand. Our Purchasing Manager comments 'Kratos has worked closely with our suppliers to ensure that even with challenges of Covid-19 and Brexit we could keep materials moving to ensure that instruments are completed and shipped to customers on time'.

With demand at a record high for the AXIS Supra⁺, our instruments are currently built

against an order. This has the advantage for Kratos of removing the 'guess work' in defining the configuration of instruments passed to Production. It can however frustrate customers with longer delivery times for their instrument.

Processing the order means that a number of colleagues in Sales and Accounts have already been involved with production of the spectrometer at this early stage. The manufacturing order passed to the Production department initiates the demand to build the spectrometer. It's at this point that some of the magic of the process really starts.

Assembly of an AXIS Supra⁺ happens at a remarkable pace. This is largely due to the fact that many of the instrument's sub-assemblies, such as the autostage, lens and analyser, have already been built by our team of highly skilled Fitters.

The instrument is assigned a testing bay on the shop-floor, where it will remain throughout build until it is switched off at the end of Test. As soon as the frame and UHV chambers are issued, a flurry of activity sees the instrument rapidly assembled and wiremen busy adding electronic units and cabling the spectrometer.

Although the pandemic has meant that we have had fewer staff on site, production on the shop-floor has been 'business as usual'. Albeit, as socially distanced as possible.

After a few weeks, the fully built instrument is

passed to one of our Test & Installation Engineers. Over the next several weeks the instrument is transformed, butterfly-like, from a bag-of-bits to a fully functioning, state-of-the-art spectrometer. This is credit to the hard work and dedication of the Test Engineers. At the end of the test period, they have confirmed that the spectrometer and all its accessories meet our internal specifications, which are typically more rigorous than the actual sales specification. Next, the instrument is inspected and passed by engineers from the Quality Department.

Even at this stage, there's still plenty to do to get the instrument ready for its journey to the customer site. It's prepared for shipping by the Fitters and passed to colleagues in Shipping. Attention to detail sees the spectrometer shipped in a hermetically sealed bag inside a bespoke shipping crate. Each spectrometer is shipped with a number of sensors measuring ambient conditions and shocks or knocks during its journey from Kratos to the customer site. With so much effort and attention to detail we need to ensure that the instrument is in prime condition when it arrives on site.

Whilst not an exhaustive overview of the production of an AXIS Supra⁺, it's hoped that it gives you some insight into the process. From order to delivery, building the AXIS Supra⁺ has taken the input of over 150 people at Kratos.



MEET OUR USERS

Dr. Andreas Holländer: Fraunhofer Institute for Applied Polymer Research

What is your role at the Fraunhofer-Institute for Applied Polymer Research?

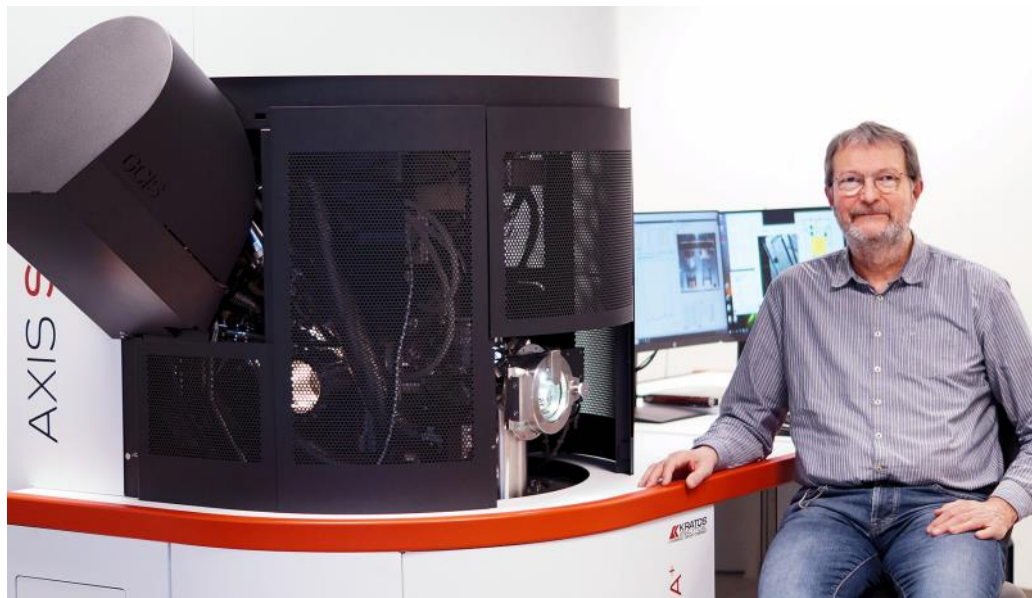
As a Senior Scientist I am involved in the preparation of projects, organizing the work in these projects and reporting the results. The projects range from analytical services to larger cooperative projects with industry. The vast majority of these projects are related to surface science, surface technology and surface analysis of polymers.

Can you describe a typical day at work?

I assume that the question does not target things like going from home to the institute in the morning and going back in the evening. Beyond that, there hardly is a typical day. In a way, this is a good thing, I don't get bored. On the other hand, there is always a pile of work in front of me. This pile contains writing proposals and quotations, planning lab work, coordinating with other parts of the teams, processing and consolidating experimental and analytical data and writing reports and publications. Beside this rather constant and recurring work, there are various activities in different networks and communities. These various parts of my work are in permanent development. For example, these days I have much less traveling than I used to have before 2020.

You recently replaced your 24-year-old AXIS 165 with an AXIS Supra⁺. What can you identify as the biggest improvements with the new instrument?

We have used the instruments – the old one as well as the new one – mostly for routine analysis of samples from the project work. This



'I am still fascinated how very small things like groups of atoms in a surface can have tremendous effects on a large scale'

means that we have a rather large number of samples every day. The high level of automation coming with the AXIS Supra is a great improvement supporting this kind of work. Despite the great improvements, we have a number of ideas and suggestions to proceed with further developments on this track.

Beside this routine work we have samples with very specific questions. The cluster ion source and the chemical state imaging give us great new options we did not have before.

How do you use your AXIS Supra⁺ in your role?

We have a team of 5 people taking care of the

instrument and the measurements, two engineers and three scientists. I am in the lucky situation that I can work hands-on the instrument, at least from time to time. Since I have been working with XPS machines for almost 30 years by now, I am the person in the team with the most experience. Therefore, I get involved if things get more complicated and when the instrument is not running the way it is supposed to.

What do you see as the value of surface analysis?

Some important properties of materials such as the wetting behaviour, the adhesion of glues, inks, paints, and also of bacteria and viruses

are determined by the outermost atomic layers of a surface. These kinds of properties are important for many products of our everyday life, be it in the final product or during the production process. Chemical surface analysis is an extremely valuable tool supporting technology development for these products and, of course, for trouble shooting in the case that something does not work like it was supposed to.

What has surface analysis taught you?

I contributed to a number of publications describing these lessons.

On a more abstract level, I am still fascinated how very small things like groups of atoms in a surface can have tremendous effects on a large scale.

Any tips or tricks for surface analysts?

For me as for the other people in our XPS team, it is a part of our work in surface technology of polymeric materials. We do not have a dedicated surface analysis group doing only analysis. I believe that the deep understanding of the materials we analyse enables us to exploit the full potential surface analysis offers.

Having this experience, I would encourage people working in surface science and technology to get involved in surface analysis. Surface analysts, on the other hand, can provide much more useful results to their peers and clients if they have a solid understanding of the chemistry of the samples they analyse.

Looking back at development of Kratos spectrometers

THE DELAY-LINE DETECTOR

Our review of the significant advances for Kratos XPS instruments focuses on the development and use of the delay-line detector (DLD) for counting photoelectrons in both spectroscopy and imaging modes.

In Newsletter03, we outlined the invention of the spherical mirror analyser (SMA) for parallel imaging. One of the unique properties of the SMA is that it produces an energy filtered 2-dimensional image of photoelectrons from the sample surface at the detector plane of the analyser. When first introduced, the imaging detector comprised a micro channel plate (MCP) to produce further secondary electrons which were projected onto a phosphor screen. The scintillations on the phosphor screen were viewed by a CCD camera from which the photoelectron distribution from the surface was integrated by the Vision 2 software.

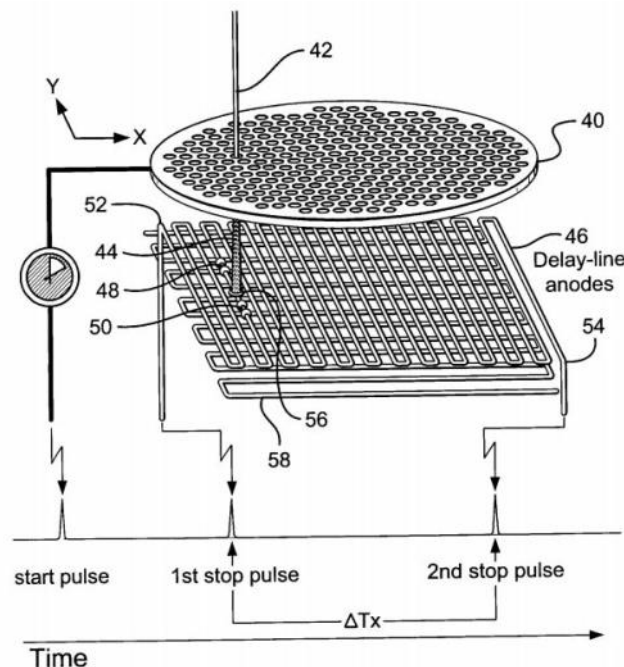
In spectroscopy mode, the photoelectrons were collected with a total of 8 channeltrons, arranged 4 either side of the MCP. This is the detector arrangement used for the AXIS Ultra, with $\frac{1}{3}$ of the area used for imaging and $\frac{2}{3}$ used for spectroscopy.

The delay-line detector replaced the discrete detectors for spectroscopy and imaging modes with a simplified, single detector. This had the immediate effect of increasing the area over which the photoelectrons could be collected in spectroscopy mode and therefore the sensitivity specification for spectroscopy mode of the AXIS Ultra^{DLD}.

A further advantage of the DLD extended to pulse-counting of the photoelectron in imaging mode. The DLD replaced potentially non-linear CCD + phosphor screen imaging detector so that *quantitative* images were derived for the first time.

A worldwide patent published in 2004 by Kratos employees, Simon Page (Physics/Engineering), Colin Park (Firmware) and

Chris Hopper (Electronics), outlines their invention relating to 'a charged particle spectrometer, method of operation and in particular, to a detector for such a spectrometer' [1].



The detector hardware, shown schematically in this figure from the patent, is deceptively simple and comprises a micro channel plate (40) above a pair of orthogonal delay line anodes (46,58). In operation the primary photoelectron (42) emerges from the energy analyser and strikes the MCP, causing a 'shower' of secondary electrons which impinge on the delay line. A 'start' pulse is generated as the photoelectron strikes the MCP. The shower of secondary electrons from the MCP creates a pair of 'stop' pulses (48,50) which propagate towards the end of the delay line (52,54). The ends of the delay line are connected to signal processing which calculates the time difference between

their times of receipt, allowing the point or origin (56) of the electrons on the delay line to be calculated. In spectroscopy mode only one of the delay line anodes is used, corresponding to the energy dispersive direction of the hemispherical analyser. The detector may be operated in conventional scanned mode where the energy of input lenses and the analyser are swept through the required energy range and the photoelectron intensities are summed across the detector. Alternatively, the spectrometer can be operated in unscanned or snapshot mode, where the input lenses and analyser are kept at a constant energy and the energy dispersion of the analyser is used to integrate a spectrum from the data channels of the delay-line detector. This mode of spectral acquisition has the specific advantage that it can be extremely fast with application to time-resolved XPS studies.

In imaging mode, the orthogonal X and Y axis delay lines are used to give a coordinate for the single event, photoelectron position on the detector. An image is generated by counting the number of photoelectrons arriving over an integration period of several seconds.

This is a simplified description of the hardware and the counting process for acquiring photoelectron spectra or images. The complex parts of the DLD are the fast electronics, with the ability to count the start/stop pulses with resolution of pico-seconds, and the software which is able to process these signals and compute the information into the spectra and images that we recognise.

The DLD and associated electronics remain in use, largely unchanged, in the latest generation of AXIS instruments demonstrating the longevity of this invention.

[1] WO2004042775 (A2) — 2004-05-21.