KRATOS ANALYTICAL
A SHIMADZU GROUP COMPANY

NEWS 03 2020

Tips, Tricks & Thoughts from the Apps Lab.

Service Support : Covid update and new array group analysis in ESCApe 1.4

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Meet our Users

Dr Kaveh Adib, Corning Inc.

Looking back at development of Kratos spectrometers

Part 3: Spherical Mirror Analyser

FROM THE EDITOR

Welcome to the third Kratos newsletter

It's been a busy few months since the last Kratos surface newsletter. We continue to adapt to a new working practices with colleagues that can work from home being asked to do so for the rest of this year. This measure helps to ensure that those involved in the assembly, testing and shipping of our instruments can work in as safe an environment as possible. Their hard work has ensured that we have continued to ship instruments to new customers throughout this challenging period.

The advantages of a fully automated XPS instrument have become even more apparent, with the applications team being able to analyse samples, generate reports and conduct instrument demonstrations without being on site. We have had similar feedback from customers which is very positive.

One area that has proved challenging is service support. Whilst the support group continue remote diagnosis of faults and coordinate replacement parts, travel restrictions have prevented most 'on-site' work. This is reviewed further by our Service Manager later in this Newsletter.

Also in this newsletter we have the regular 'Tips, Tricks and Thoughts from the Applications Lab'. For those that have not visited Kratos HQ, the Apps. Lab. for surface and MALDI products is located behind the large windows most prominent in the image above. The i-work interview this quarter is with our US Service Manager, Danny Ovens-Smith. We catch up with one of our AXIS Supra Users at Corning Inc. and have look back at the development of the spherical mirror analyser which created a step-change in XPS imaging when launched as part of the AXIS Ultra in 1997.

We hope you find something to interest you and appreciate your 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.

SERVICE SUPPORT

Covid update

Defining 'normal working' over the last 3 months has been impossible and continues to be challenging going forward. The Kratos Support Group, based at our Manchester headquarters, is continuingly adapting to provide the best possible service support we can under the prevailing restrictions. Our covid -safe guidelines mean that, where possible, staff that are able to work from home are doing so for the rest or 2020. This means that fewer colleagues are working on site, ensuring their safety and allowing the normal Support Group activities to function. This seems to be mirrored by our Users, who after initially being unable to access labs to run their Kratos spectrometers are now allowed limited access to mount samples and start analysis. With this gradual return to work we are seeing an increasing demand for Support.

For Customers with a service contract we can offer remote support using TeamViewer and VoIP telephone support. Obviously, this requires the PC to be connected to a network and the internet and we would encourage all Users to ensure this is the case to help us with remote support of your instrument. ESCApe software has significant reporting and logging functionality which allows our Engineers to help tune or diagnose faults with ease. If we identify a fault and where appropriate we can dispatch replacement parts to restore functionality of your spectrometer. By holding a service contract with Kratos you enable us to employ a dedicated service team available to support your instrument. As well as remote support, a service contract also allows you to access service loan stock and provides availability for service exchange stock. All service contracts include an annual preventative maintenance (PM) visit, ensuring your instrument remains calibrated and in prime working condition!

We understand that some faults cannot be resolved through remote support. In this case we will do our best to schedule a visit of one of our engineers. Of course, visits can only be arranged where UK Government and local restrictions allow travel. We will also work with Users to follow local working practices and ensure the health and safety of our employees and Users. In supporting our Users we are prioritising emergency visits to customer site. In recognition of the challenges in scheduling site visits during this period, Kratos Analytical Ltd* will rollover any unused 2020 preventative maintenance days into the next contract period and commit to performing the PM visit at the earliest opportunity.

Currently, the best way to contact us is to e-mail support@kratos.co.uk. This e-mail can be used for specific instrument support requests or more general contract enquiries.

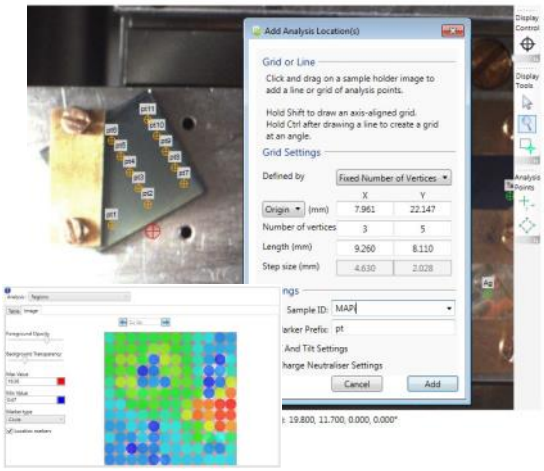
* where the service contract is with KAL, UK.

GROUP ARRAY ANALYSIS

Enhanced functionality for ESCApe 1.4

What sampling rate provides analysis representative of the sample? Is a single, large area survey spectrum enough, or should you acquire 3 or 5 from different positions on the sample? It's never easy to decide how best to design an experiment although with the new **group array** analysis functionality in ESCApe 1.4 software, the data acquisition has become significantly easier.

The first step is to define the analysis positions on the sample using the 'add analysis locations' marker. This allows the easy definition of



either a line or 2D grid of analysis points across the sample. Further flexibility of this analysis location tool allows the grid settings to be defined by a fixed number of vertices or separation between points. Where the sample has an irregular shape the analysis positions that fall outside the area required for analysis can simply be deleted. Once added to the location table, to benefit from the group batch processing and graphical display capabilities, the data must be acquired in 'group' mode. This simple step ensures that data is acquired from each array position with identical acquisition parameters, guaranteeing that there is consistency of all data within the group node.

As with any data acquired in ESCApe, region and component models are effortlessly propagated across the group dataset. The subsequent processing step provides 'spatial analysis' of the data, allowing elemental or chemical state quantification to be displayed as a function of position across the sample. This is particularly powerful in allowing the visualisation of large amounts of spatially distributed data. It becomes easier to correlate chemical differences on the surface of the sample.

Group array acquisition and analysis is well demonstrated in recent work completed in our Applications Lab. on combinatorial exploration and mapping of a co-sputtered Ni-Ti-Co thin-film [1]. The substrate was a 3" Si wafer with 177 discrete analysis positions. Analysis took less than 3 hours. The elemental composition as well as chemical state of Ti was determined and displayed using the large sample handling capability of the AXIS Nova in combination with the group array analysis.

[1] https://dx.doi.org/10.1021/acscombsci.0c00097

i-work

Interview with an employee

Name **Danny Ovens-Smith**

Job title U.S. Service Manager

How long have you been at Kratos?

I was with Kratos UK for 8 years before moving to the U.S. in February of this year.

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

I manage our support efforts for our customers in North America and send the Service Engineers where they need go. I will also be giving them ongoing training and have many projects on the go to improve Kratos US and then allow it to continually get better. Some of these are already starting to make a difference so I am looking forward to making things even better.

Best part of your job?

There are a few things I've enjoyed through different roles at Kratos: the travelling, helping customers, overcoming challenges, and solving problems are probably the highlights for me. For my new position here in the U.S, I've enjoyed talking to and meeting a new set of customers, all with their own needs and issues to solve. It's so cheesy but I really do get a kick out of work when a customer is happy – every job I've had since I was 16 has been directly customer facing in some way so I think I'm wired that way.

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

Before moving to Kratos I used to work for a rocket company working towards Space Tourism whilst also doing educational outreach in schools teaching kids that rocket science isn't actually that difficult (despite the well known phrase!) but that the engineering of how we achieve it is the hard part. I completed my undergraduate degree in Physics from Lancaster University before that and had a few jobs through my studies and was Vice President of our Student Union for the year after I graduated before moving to Manchester.

What have you learnt working at Kratos?

I was always more of a Physics/Astro-physics nerd but here I've picked up mechanical engineering, electrical engineering, some chemistry, and even plumbing skills too in order to understand everything going on with our systems and how all these different aspects come together to function as one. It was never something I thought I would do but here I am 8 years later!

Your favourite quote / line from a film?

There are a few variations based on the translation but: 'The Earth is the cradle of humanity, but one cannot live in a cradle forever'- Konstantin Tsiolkovsky, essentially the founding father of modern astronautics who was mathematically proving rocket propulsion and space travel theories even before the turn of the 20th century.

What is your motto or personal mantra?

Growing up my family motto was always 'If at first you don't succeed, try, try again' but it was supplanted for me later in life with the seminal 'Do, or do not … there is no try' which gives me my 'if you're not going to do it right, you might as well not do it at all' attitude when it comes to my work ethic.

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

I'm actually a bit of a gamer – not sure if that counts as being 'busy' but it can serve many purposes whether that is helping me relax or keep my mind active with strategy/tactical games.

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

This is a hard one. I'm trying to think of something vaguely interesting. I used to be part of the skydiving society we had at University, The Black Knights, as we had the longest running parachute centre in the UK just 15 minutes' drive away and did several solo sky dives.

STAYING ENGAGED

USERS' SURVEY—WHAT YOU TOLD US

Just under 6 months ago we conducted our biennial Users' Survey. This is an important way for us to gather feedback from Users of our instruments to better understand aspects of the Surface Business. This feedback is an important metric within the Quality system at Kratos and provides input to the Senior Management group.

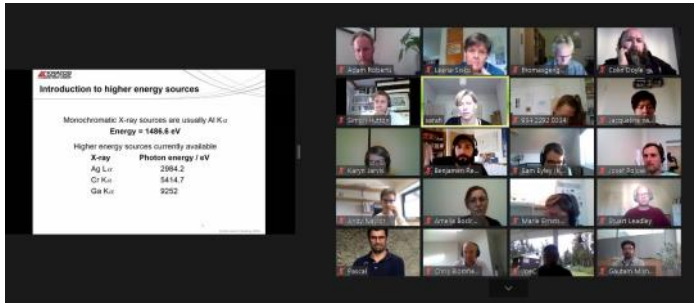
Over 150 Users completed the survey with 79% rating their 'overall experience of Kratos Analytical' either excellent or good. If we include those that rate their experience as 'average' this figure jumps to 97%. This demonstrates that positive sentiment for Kratos and our products remains at the consistently high level of previous surveys. Behind this headline figure there were regional differences. Our North American Users had lower satisfaction scores than European or Asian Users. This was pre-empted and, with the changes to structure and personnel at Kratos Analytical Inc., has been addressed. We hope that these changes will reflect in the metrics of the next Users' survey.

Engagement with our customers through service and applications support remains very good. Our survey shows 85% of global responses judged service support 'better or as good as other instrument manufacturers'. Applications support gained even higher recognition, with 92% expressing Kratos provides 'better or equivalent support'. The real value in these results is comparison to previous surveys. As with the overall experience, feedback from the 2020 survey did not show any significant changes.

It is always interesting to learn how the instruments are being used. When asked 'which features do you regularly use', not unsurprisingly the most used feature with 93% of respondents was large area spectroscopy. The next four most regularly used were ion source/depth profiling (68%), selected area/small spot spectroscopy (55%), snap-shot spectroscopy (36%) and angle resolved XPS (36%). Interestingly, the higher energy (Ag Lα) excitation source is increasing in importance relative to the 2018 survey.

Finally, we know that these headline figures are impressive but may not reflect individual experiences. If there's anything that you would like to share with us, good or bad, you're encouraged to contact us with your feedback.

USERS' MEETINGS 2020



We had a combined number of over 200 Users registered for the European & Asian and North American Users' Meetings held at the end of September. Scheduling was designed to allow maximum participation from around the globe and moving to a virtual format meant that these were our best attended meetings to date. It was with some envy that European Users watched as one of our Australian contributors enjoyed a glass of Aussie red at the end of his working day as we were on our first early morning caffeine fixes.

Feedback from both meetings was overwhelmingly positive. Both the end of meeting polls and post-meeting emails demonstrated that the content and format had been well received. Over the 2 meetings there were 30 oral contributions, with 12 from academic Users, 5 industrial, 2 national metrology labs and 11 from colleagues at Kratos. They all highlighted the use and application of our spectrometers for surface and materials characterisation.

Highlights included 'Spectroscopic studies of iron gall ink – the standard ink of the middle ages' from Karen Gaskell at the University of Maryland and the presentation of 'Argon cluster sputtering of core-shell nanoparticles' by Alex Shard from NPL.

An interesting theme during both meetings and related to an article in Newsletter#02 was that of reproducibility of XPS results in the literature and the role that Users of the technique can take in this. In his presentation 'Responding to XPS analysis challenges in publications' Don Baer concluded that this is a multi-faceted challenge in which we all need to act in addressing non-reproducibility in our own work as User, team member, mentor, manager or reviewer.

The online format also allowed us to hold a (Twitter) poster session throughout the week of the meetings. Posters were shared using **#KratosUMposter**. We acknowledge the support of AIPP in supporting this by offering a prize of 12 months subscription to their eSpectra database for the best poster. This was won by Sam Seddon of Warwick XPS Group for his poster 'Work function of GaAs (hkl) and its modification using PEI: mechanisms and substrate dependence'.

Thanks go to everyone who contributed a poster or oral contribution and to everyone who took time to attend the meeting. If you were not able to join the meeting, recordings of a number of the presentations can be viewed using links in the Kratos Members Area of our website.

MEET OUR USERS

Dr Kaveh Adib, Corning Inc. USA

Kaveh Adib has worked at Corning Incorporated's Science and Technology Division for the last 15 years. Throughout his tenure with the company, he has co-authored 8 journal articles and has been co-inventor in 16 granted patents. Prior to that he was a Post-doc at the Brookhaven National Laboratory. He holds a PhD in Applied Physics from Columbia University in the City of New York.

What is your role at Corning?

I run the X-ray Photoelectron Spectroscopy laboratory at Corning's Sullivan Park Research Center located in western New York. I use the data from this laboratory to support a number of exploratory and problem-solving projects within Corning.

Can you describe a typical day at work?

I do not have typical days, but typical weeks: when I come in on Monday, I check that the measurements performed automatically over the weekend have run smoothly. I schedule longer term measurements over the weekend in order to utilize our laboratory facilities efficiently; one of the benefits of having your own dedicated XPS lab in an industrial setting is that you can perform extremely long measurements. I usually analyze the results during the day and prepare presentations for different projects the laboratory is supporting. On Friday afternoons I try to set aside some time to perform interesting, often exploratory, experiments.

How do you use your Kratos instrument in your role?

I use the Kratos instrument primarily to solve problems and do exploratory work. The latter gives us better insight into potential issues and affords us the ability to develop ways to solve or at least measure potential problems before they occur. Our Kratos Supra instrument is equipped with a surface science station and Low Energy Ion Scattering so that in addition to XPS data, we can obtain additional information from our surfaces that help us understand the chemistry of the surface in greater detail.

Does the industrial lab have different demands to academic lab for surface analysis?

While there are many similarities, arguably, the most important difference between academic work in general and industrial work involves time. Work in an industrial setting such as Corning can involve a very large variety of projects with different timelines. There are projects that have long time horizons in the order of several years and there are highly urgent projects with timelines in the order of weeks. For urgent projects we often have limited time to solve important problems to meet various deadlines within the company and with our various external customers who have their own rigid deadlines.
In order to meet critical deadlines, we incorporate various redundancies into our analysis work flows. For example, we ensure that the decision-making regarding performance of our surfaces is based on multiple approaches, reproducible highly credible measurements and multiple surface analysis tools.

What value does surface analysis (XPS) add to Corning products?

Many of Corning's products include a specific material or surface with different attributes. It is always our aim to measure, quantify and protect Corning's intellectual properties using XPS, which of course is an extensively used and mature analytical tool. Surface Science of glass is one of Corning's main focus areas and is a very fascinating field. Unlike crystalline materials, where the composition is essentially "locked in" by a particular crystal structure and orientation, in glass surfaces, you can readily produce surfaces with varying elemental ratios and correlate surface attributes with the changes in composition to gain great insights into fundamentals of surface chemistry. Of course, surface science in Corning is not limited only to glass surfaces.

What has surface analysis taught you?

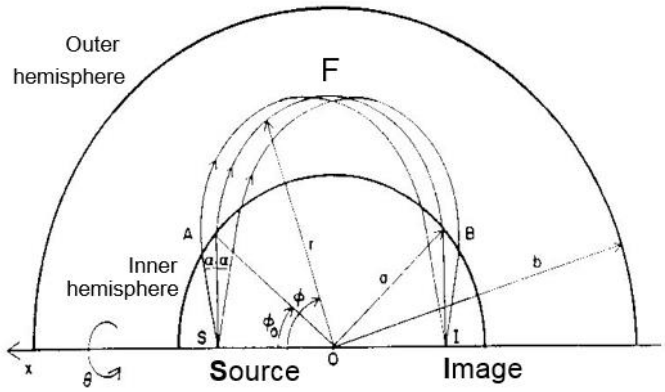
Surface analysis ultimately probes very small dimensions. Comprehending those small dimensions has taught me a sense of perspective. For example, in a typical half-liter water bottle, there are more water molecules than there are stars in our universe! If you take about 40 of those water molecules and line them up, that gives you slightly more than 10 nm, which is the typical probe depth of most commercial XPS instruments.

Any tips or tricks for surface analysts?

You should try to predict where your focus within surface science will be in the short, medium and long terms. Then, you should try to see what skills and tools you will need to make an impact within those time frames. XPS is a tool that provides meaningful insights for scientific research. It has brought great value to my research and can ultimately do the same for you and for your employer.

THE SPHERICAL MIRROR ANALYSER : parallel XPS imaging

Our review of the major developments of Kratos XPS instruments focuses on the development and integration of the spherical mirror analyser (SMA) creating one of the most successful XPS instruments ever sold, the AXIS Ultra.



Early studies of the SMA were published by an Israeli scientist Sar-El in 1966 [1] and later by Tremblay & Roy [2] and Diamon [3] in the 1980's. Sar-El considered an electron energy analyser with a pair of concentric hemispheres where the source, S, and image, I, were within the inner hemisphere. He noted that the SMA showed point-to-point focus within the inner hemisphere. This was a rare case of perfect focussing where all electrons at the pass energy converge at I regardless of their starting direction at S. The electron transport properties of the SMA also imply unit magnification and zero distortion, properties that are important requirements of an imaging analyser!

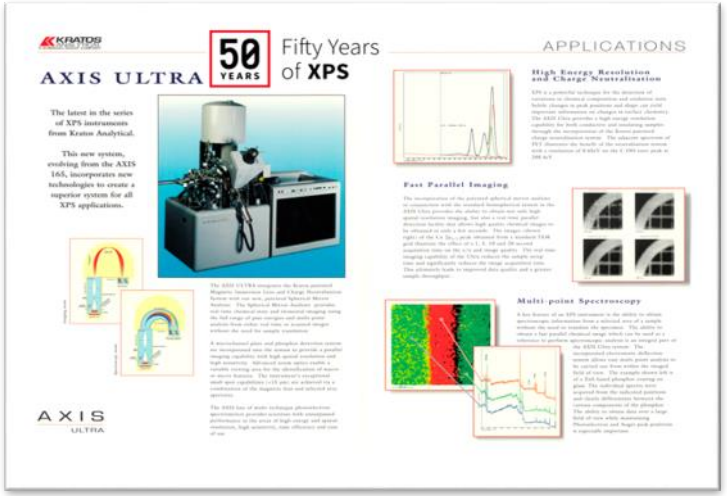
The novel step patented by Dr Simon Page during his early career at Kratos was to integrate the SMA with the conventional hemispherical sector analyser (HSA) already widely used for electron energy analysis in XPS instruments. The European and US patents for this work were filed in 1991 and lead to the development and launch of the AXIS Ultra in 1997. As stated in the patents 'the present invention provides an analyser having the possibility of providing spatial information relating to chemical composition over the surface of a sample'.

By combining the hemispherical sector and spherical mirror analysers, Dr Page and Kratos had developed an XPS instrument that allowed the highest energy resolution spectroscopy to be combined with fast, chemical state parallel imaging.

At this point it is worth highlighting the fundamental differences with between parallel imaging and mapping modes. In parallel imaging mode, the photoelectrons are energy selected by the retardation lens and focussed at the entrance slit to the analyser. Their 2-dimentional distribution is retained as they move through the HSA and into SMA where they are focussed at the 2-dimentional detector. By integrating the photoelectrons arriving at the detector an image is acquired in a few tens of seconds. This contrasts with the conventional mapping mode where a focused probe is rastered across the surface and the electron intensity integrated as a function of the position of the probe on the surface to map of the surface. Mapping acquisition mode was used by the AXIS instruments using the ACIL lens discussed previously in newsletter#2.

As well as greatly improved speed of image acquisition, parallel imaging also has a significant advantage of greater spatial resolution. When launched in 1997, the imaging resolution of the AXIS Ultra was 3 um which was a dramatic improvement over the 27 um spatial resolution offered by the AXIS 165 in mapping mode. Current ultimate spatial resolution guaranteed for the AXIS Supra$^+$ is 1 um. In the last decade the excellent energy and spatial resolution of the SMA, coupled with the fact that the analyser works in FAT mode (meaning that they energy resolution remains constant as a function of kinetic energy of the analysed photoelectrons) has allowed the SMA to be used for spectromicroscopy applications. The development of multivariate analysis of multi-spectral images by Walton and Fairley [4] allows acquisition of over 65,000 spectra from the imaging field of view to be realised in realistic acquisition times, providing spectra from much smaller areas than possible with conventional selected area XPS.

There are over 500 Kratos AXIS instruments with the spherical mirror analysers around the world. This demonstrates that a step-change development in the late 1990's continues to be an important component for X-ray photoelectron imaging nearly 3 decades later.



[1] Sar-El, Nuclear Instruments & Methods **42** (1966) 71-76

[2] Tremblay & Roy, Nuclear Instruments & Methods in Physics Research **220** (1984) 270-287

[3] Daimon, Rev. Sci. Instrum. **59**, 545 (1988)

[4] Walton & Fairley, Surf. Interface Anal. 2008; 40: 478–481