KRATOS ANALYTICAL
A SHIMADZU GROUP COMPANY

# NEWS 01 2020

## Tips, Tricks & Thoughts from the Apps. Lab.
A little of what we know

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## Next stop Kratos Analytical
New Metrolink line passes Kratos site

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Dr Mark Biesinger

## Looking back at development of Kratos spectrometers
Part 1: The origins of Kratos Analytical


# A QUICK HELLO

## Welcome to the first Kratos newsletter

It's been on the 'to do list' for years but never quite floated to the top. But, with unprecedented times and coronavirus changing how and what we do at work we've finally put something together. I'm sure that the content will develop with each publication. We'll be introducing the great people that work at Kratos in the 'i-work' interviews. In our first issue we meet one of our applications specialists. There'll be interviews with our Users highlighting the great results generated from Kratos spectrometers. We'll also be looking back at the technological developments of our spectrometers, highlighting the 50+ years of XPS from Kratos as well as Tips, Tricks and Thoughts from the Applications Lab.

We hope you find something to interest you and would really appreciate some feedback.

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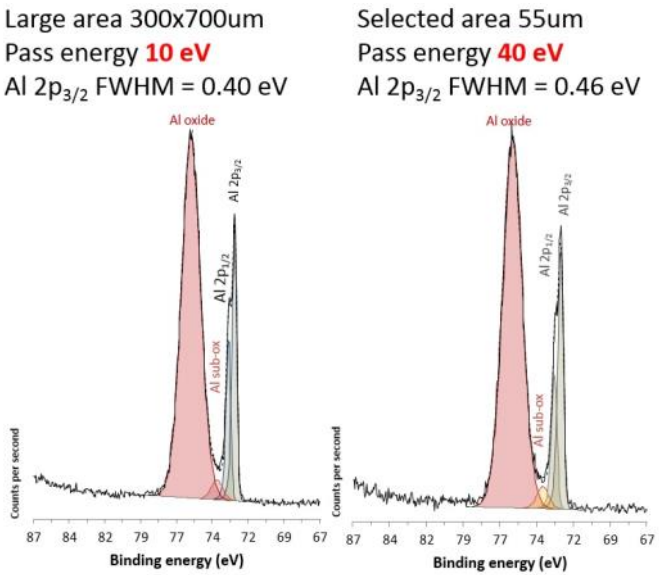
# TIPS, TRICKS AND THOUGHTS FROM THE APPLICATIONS LAB.

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. Some of these insights may be of more relevance to those using our latest ESCApe software, others will be more generic and applicable to all AXIS instruments. What would be interesting is to learn of any Tips, Tricks and Thoughts that our Users might have relating to data acquisition and processing using Kratos spectrometers and software. If you'd like to share something, why not contact us and we'll publish the best of them.

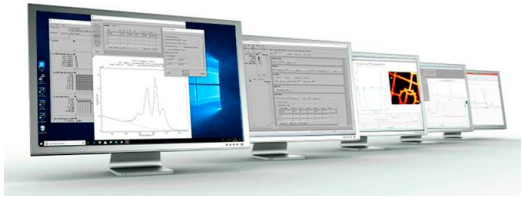
## INCREASING SENSITIVITY FOR SMALL SPOT ANALYSIS

The first of the thoughts that we'd like to share relates to selected area spectroscopy using any of our AXIS instruments. The selected area chosen for a small spot analysis is defined by inserting an aperture into the lens column. The combination of magnetic and electrostatic lenses, along with the size of the chosen aperture defines the diameter of the analysis area from which the photoelectrons are collected. A consequence of inserting the aperture into the lens column is a reduction in the number of electrons reaching the detector, or in other words a decrease in the signal. This is very apparent to anyone that uses selected area spectroscopy mode for data acquisition. A less obvious benefit of using the aperture and iris to define the small analysis area is that the spectroscopic resolution increases when compared to the large area (slot) analysis. The primary reason for this is that by inserting the aperture and closing the iris, the trajectories of the photoelectrons are narrowed and there is less electron scattering in the analyser. This effect is more apparent for the smaller 27 µm and 15 µm analysis diameters. The significance of this is that as you use smaller analysis areas, you can work at a higher pass energy and get similar energy resolution to the lower pass energies in large area mode. And, as we know, working at higher pass energy gives a higher sensitivity with the opportunity to reduce the acquisition time.




This is demonstrated in the figure where the Al 2p region from aluminium foil was acquired from a large area (700x300 µm) at a pass energy of 10 eV where Al $2p_{3/2}$ FWHM = 0.40 eV, and a 55 µm diameter area at a pass energy of 40 eV where Al $2p_{3/2}$ FWHM = 0.46 eV.

## UPGRADE TO WINDOWS 10



With Windows 7 now no longer supported by Microsoft, we are getting more and more enquires about upgrade paths to the latest operating system. The good news is that for nearly all Kratos instruments there is now a route allowing you to run your data acquisition and processing software on a Windows 10 PC.

For the oldest **VME based instruments** it is possible to run Vision software on the Win10 PC. The limitations in the availability of driver software for cameras however means that any upgrade to these systems might result in some loss of functionality.

**AXIS Ultra DLD** instruments can be upgraded to the last release of Vision software running on Win10. The same is true for **AMICUS / ESCA3400** instruments.

For **AXIS Nova** spectrometers running on Vision, there is a requirement to upgrade both the PC and the acquisition and processing software so that the instrument runs ESCApe on Windows 10.

And finally, early **AXIS Supra** instruments that were installed with Win7 can be upgraded to Win10, in some cases using the same PC.

As Users will appreciate, there are a significant number of permutations and combinations in hardware and software. To discuss the upgrade route available for your instrument we encourage you to contact us.

## AND FINALLY…

A common method for mounting powder samples for analysis is by pressing them into 'wells' with the back of a spatula. The significant advantage of this approach being that there is no risk of silicone contamination occurring from the adhesive tape that might otherwise be used. Similarly, it removes the question that any of the signal might be coming from the adhesive due to incomplete coverage by the powder.

One trick to getting the best energy resolution from powder samples is to ensure that the sample is as flat as possible as this eases charge neutralisation and minimises scattering of the electrons as they leave the material.

With the ability to mount so many samples on a bar, just remember not to mix powders pressed into wells with those for ARXPS! Otherwise you'll be hunting for the vacuum cleaner!

# i-work

## Interview with an employee


Name Dr Nikki Gerrard

Job title Applications Specialist

### How long have you been at Kratos?

Time has flown by since I started at Kratos, it's coming up to 16 months now.

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

I am part of the sales and applications team at Kratos. From a sales perspective, my role involves dealing with customers – from the analysis of samples for prospects thinking about buying an instrument through to after sales User training on site. I perform demonstrations of the Kratos surface instruments to prospects to give them an idea of how the instrument works and what it's really good at. From an applications perspective, I'm involved in collaborations with scientists outside of Kratos, from universities to industrial companies, to apply XPS and other surface analysis techniques to real life samples.

### Best part of your job?

I have two! Having the opportunity to travel globally to train customers on site and see what they do in their laboratories is one of my favourite parts of my job. The other is the diversity of the samples I get to analyse in the applications lab. We receive samples from so many different areas. One day I'll be analysing the organic content of soil, the next, a device from a silicon chip fabrication lab. There's never a dull day in the lab with such a diverse range of samples to analyse which one wouldn't have the opportunity to analyse in many other roles!

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

After completing my undergraduate master's degree in Chemistry, I decided to pursue a PhD in the Surface Science Research Centre at the University of Liverpool. With water/metal interfaces being of great importance in the fields of corrosion, catalysis and ice nucleation, my studentship focused on the use of surface analysis techniques to investigate surface interactions within these hydrogen-bonding systems. Once I'd finished my PhD, I started my position at Kratos to expand my knowledge of surface analysis techniques and their applications.

### What have you learnt working at Kratos?

A lot! It is a huge bonus to be a part of the applications team, to work with the different departments and have such a close insight into the development of the instruments and software. But also from a business perspective, working at Kratos has allowed me to see how a manufacturing business is run with production, R&D, software and sales all in house.

### What is your motto or personal mantra?

Team work makes the dream work! - our motto in the applications lab.

Be nice to everyone! You never know what kind of day someone is having.

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

I have a young family which is a fulltime job in itself. So my time outside work is spent with them.

# NEW METROLINK LINE RUNNING PAST OUR FRONT DOOR

## And how we moved it

Manchester's newest Metolink line opened in March, serving Trafford Park, Europe's largest industrial estate with leading brands such as Kelloggs, Unilever, Adidas and Kratos Analytical! The nearest stop to our Headquarters is Wharfside, a short 50 m walk to the main entrance. The line continues down Trafford Wharf Road, past our main entrance, but not as close as Transport for Greater Manchester (TfGM) had originally outlined!

As soon as the plans were opened for public consultation it was apparent that the proposed line ran directly adjacent to our boundary. This set alarm bells ringing regarding electromagnetic and vibration interference with our surface analysis instruments. Led by Dr Simon Page, Kratos Analytical started dialogue with TfGM around this issue. It seemed unlikely that Kratos could really have very much influence on this £350 M project. However, through a series of meetings between Kratos and TfGM we convinced the project leaders that we were committed to staying at our Wharfside location and that our business contributed not only to the local Trafford Park economy but also the North West regional and national economies with our network of suppliers.

One of the biggest concerns of the tram running so close to the factory was the electromagnetic fields that would be generated as the tram passes the building. This could potentially play havoc with the low kinetic energy electrons that we're measuring in XPS, Auger and SEM. The problem was compounded by the fact that the fields generated by the moving trams are dynamic and will be different depending whether the trams are accelerating or decelerating and also the number of people aboard. Modelling by Dr Page demonstrated the adverse effect of the original proposed line position and showed that by moving the tram track towards the ship canal and designing mitigation magnetic field cancellation into the power infrastructure along the Trafford Wharf section, the interference could be reduced to below our site specification requirements. These proposals stood-up to the scrutiny of the TfGM engineers who extended the simulations to include expected tram movements and potential fault conditions. A revision to the track position was approved and recent testing after construction has shown the success of the magnetic field cancellation.


*'It seemed unlikely that Kratos could really have very much influence on this £350 M project.'*

It's testament to Dr Page's efforts that TfGM accommodated our proposal. The revised line now runs along a section of the Central Bay Wharf – originally constructed in the Victorian era. Significant ground works were required and additional costs incurred due to rerouting the line. There were two unforeseen benefits of the project to accommodate Kratos' requirements. The first involved TfGM's subcontactors being awarded a prize for the design and implementation of the mitigating infrastructure and operating procedures. The second, more significantly for Kratos, was the demolition of the vacant single-story units opposite our site, so we now have an uninterrupted view to the Manchester Ship Canal and waterside location.

# MEET OUR USERS

## Dr Mark Biesinger : Surface Science Western, Canada

Dr Mark Biesinger is the Director of Surface Science Western (SSW), Canada's leading surface analysis and materials characterization facility located at Western University in London, Ontario, Canada. Mark has several highly-cited publications* in the field of X-ray Photoelectron Spectroscopy (XPS) which focus on improvements in both sample analysis and data interpretation techniques, particularly in the analysis of transition metals. Additionally, Mark also authors and maintains the X-ray Photoelectron Spectroscopy (XPS) Reference Pages, www.xpsfitting.com, a repository of techniques, tips and reference materials designed to help XPS users worldwide.

### What is your role at SSW?

I became SSW's Director in June of 2019. Prior to that I held Research and Business Development Manager and Research Scientist roles. I joined SSW as a scientist in 1995.

### How do you use your Kratos instrument in your role?

Time constraints with my new role means I've had to give up some of my other analysis duties, but I continue to run our two Kratos XPS instruments for our industrial and academic clients. London has strong ties to energy, automotive and other manufacturing sectors in addition to being a major medical research hub. Canada also has a very strong mineral resources sector. SSW's work tends to focus on research and problems in these industries. We use XPS to analyze items such as adhesion failures, surface contamination problems, corrosion and material degradation issues, along with the chemistry of mineral surfaces. We are also heavily involved in research on the copper coated steel containers that will be used to store nuclear fuel waste in the deep geologic repository for the next million years or so. A lot of the work I've done on characterizing copper and other transition metal species by XPS has been driven by this research and other research in the nuclear energy field.

### What do you see as the value of surface analysis?

From a business standpoint the monetary value alone can be tremendous. Every day SSW uses a wide variety of surface analysis instruments to solve short and long-term production issues, assist in quality assurance and drive research and development for a large range of industry sectors. I can think of several cases where a relatively small amount of surface analysis has led to cost savings of hundreds of thousands to over a million dollars annually. A great recent example is where we used a variety of surface analysis techniques (Tof-SIMS, SIMS, XPS, SEM/EDX) to help a gold mine increase gold recovery by over 25%, adding over $20M/year to their profits!


'It amazes me how surface analysis can be applied to so many different scientific and engineering fields.'

### What has surface analysis taught you?

How interconnected science is. It amazes me how surface analysis can be applied to so many different scientific and engineering fields.

### Any tips or tricks for surface analysts?

In a recent discussion with some other XPS experts, one of the things that came up was the value of electrically isolating or "floating" samples for XPS analysis. This technique seems to go a long way in mitigating the dreaded differential sample charging problem.

*Note from the editor: Mark's papers on the surface chemical states in XPS analysis of first row transition metals have over 3000 citations each and present data acquired using an AXIS Ultra at the University of Western Ontario. They are easily found using Google Scholar.

# THE ORIGINS OF KRATOS ANALYTICAL LTD

Interestingly, the origins of Kratos Analytical lie not with the development of photoelectron spectrometers, but with magnetic sector mass spectrometers.

In this article, we discover how In 1940 with the entry of the USA in the second World War, Nier and his co-workers at the University of Minnesota began the design and manufacture of a few sector field instruments for the analysis of isotopically enriched uranium in connection with the Manhattan Project. Approximately 40 of these instruments were subsequently produced by the General Electric Corporation (GEC) in the USA, also for use in the atomic bomb project.

The Metropolitan Vickers (MV) Research Department was approached by the Directorate of Tube Alloys of the Department of Scientific and Industrial Research in connection with the manufacture of mass spectrometers for the British part of the same project. The manufacture of the MS1 allowed MV to extend the skills in vacuum engineering and the production of precise electrostatic and magnetic field which had been developed earlier in connection with the high voltage oscilloscope and the electron microscope. Anyone wishing to learn more about the development of the magnetic sector mass spectrometers can follow this link to a short overview of these instruments from the early MS1 to the final instruments MS30, MS50, MS25 and MS80.

Kratos Analytical has its origins in Metropolitan Vickers Ltd – one of the major electrical companies dominating the British industrial scene during the early part and middle of the 20th century. Around the 1950's, Metropolitan Vickers merged with British Thompson Huston (BTH) to form Associated Electrical Industries or A.E.I. The two companies continued to trade under their own names for about 10 years before they finally changed to A.E.I. During the 1950's and 1960's A.E.I. invested heavily in pure research and they set up a small division to build and sell a range of scientific instruments coming out of this research. This was A.E.I. Scientific Apparatus Ltd. As our incorporation certificate with Companies House lists, on the 31st December 1977 the company name was changed from A.E.I. to Kratos Limited, and subsequently Spectros Limited between 1984 and 1987 with the current name, Kratos Analytical Ltd being registered on 5th August 1987. Soon after this, in 1989, Shimadzu Corporation acquired the company.

However, it was as A.E.I. that the first photoelectron spectrometers were developed. As Dr David Clark recalls 'as a consultant to AEI Scientific Apparatus (later Kratos), I was involved at an early stage in the design of the first commercial X-ray photoelectron (XPS) spectrometer, the ES100, capable of variable take-off-angle studies. With funding provided by SRC, my research group at the University of Durham took delivery in 1969 of the first commercial ES-100 to be made'. And so history was written. Kratos Analytical had sold its first photoelectron spectrometer and embarked on the development of these instruments which is continuing over fifty years later.

In future Newsletters we'll highlight some of those developments and the people behind them.


Kratos Analytical site at Barton Dock Road *circa* 1970s