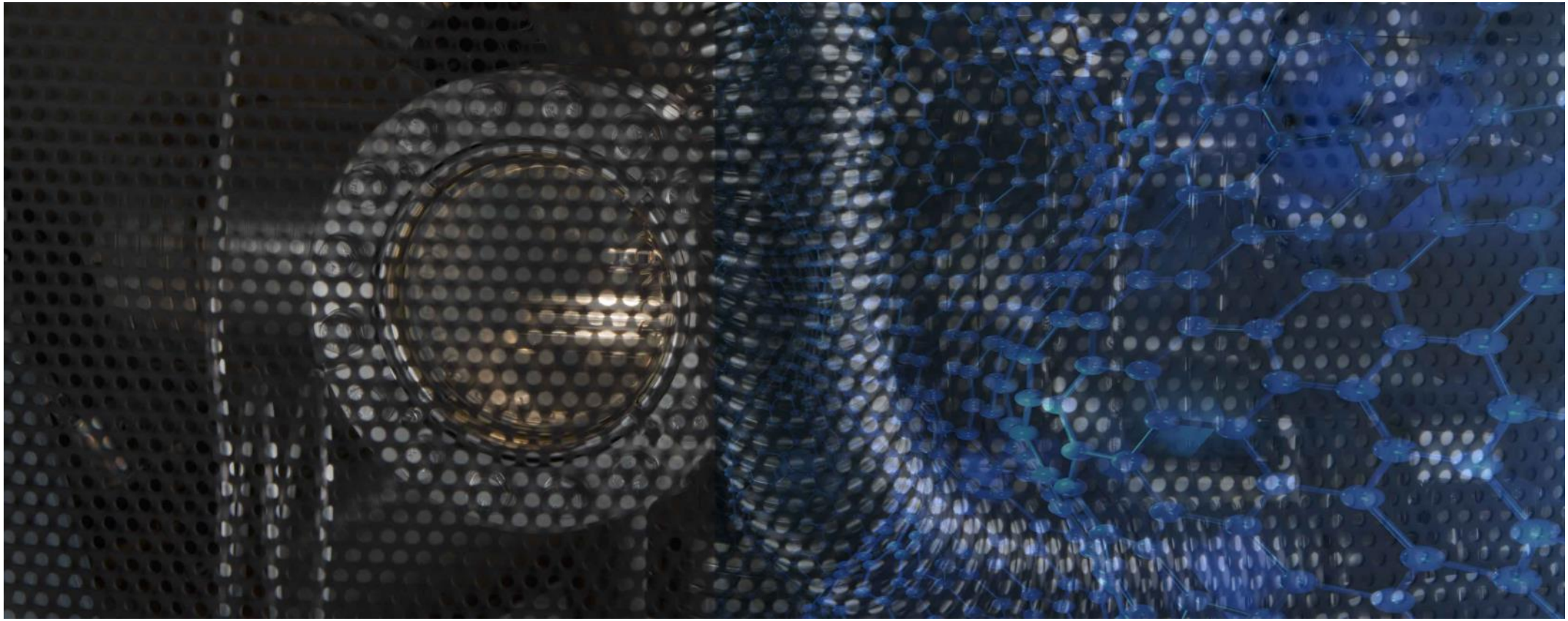
KRATOS ANALYTICAL
A SHIMADZU GROUP COMPANY

NEWS 07 2022



WELCOME TO THE WINTER KRATOS NEWSLETTER

Made to Measure

We've been designing, building and supporting our instruments from our headquarters in Trafford Park, Manchester, for over 50 years. As the world's first industrial estate, Trafford Park has a long and interesting history which we outline in this quarters newsletter.

We have two interviews. The first is with Dr. Stuart Leadley from Dow Silicones based in Belgium who recently replaced his AXIS Ultra with a new AXIS Supra^{+}. The employee interview is with our Purchasing Manager who has the responsibility, along with his team, of purchasing all the parts the company needs to manufacture our instruments.

Continuing the theme of measurements, we have a review article outlining one of the first and the latest peer-reviewed articles from our Applications lab, separated by over fifty years!

It's rewarding to think that our instruments and colleagues have been contributing to research using XPS over many decades.

In our tips from the Apps. Lab. we extend an invitation to collaborate. Perhaps your research would benefit from depth profiling with a gas cluster ion source, or using the higher photon energy Ag Lα excitation source. Read on to see how you can make this happen.

Enjoy the read.

info@kratos.co.uk www.kratos.com

TIPS, TRICKS AND THOUGHTS FROM THE APPLICATIONS LAB.

Any questions?

Have you ever wondered how something on your Kratos instrument works? Want to know the best way to process your data? Chances are that you're not alone. We'd be keen to provide answers to your questions in a new 'ask the experts' feature that we'll publish in the next Newsletter.

Submit your question and we'll forward them on to the appropriate expert within Kratos and provide answers in the Spring Newsletter.

Highlighting your research!

Our Applications Specialists have always understood the importance of our instruments in contributing new insights to materials characterisation. We strive to publish our research, including instrument developments, in peer-reviewed journals. This is highlighted elsewhere in this newsletter as we review publications from Kratos Applications specialists spanning over fifty years.

But this is only our side of the story. One of the most rewarding parts the role of Applications Specialist is travelling to customer sites to provide training and learn how the instrument will be used. With hundreds of AXIS photo-electron spectrometers around the world it's hard to imagine the contribution that they must be making to scientific endeavour. A simple Google Scholar search for "Kratos AXIS" shows that there have been over 3500 publications since 2021 alone.

With this in mind we'd welcome the opportunity to highlight data generated and published from your Kratos instrument.

Do you have a result or publication which you'd like us to feature? If so, please get in contact and we'll work out the best way to highlight your research!

Collaboration opportunity

Much of the work published by Kratos Applications specialists is as a result of collaboration with one of our Users, a prospect or former colleague. Whilst we have access to our own AXIS Supra$^+$ in the applications lab. we do not have easy access to complementary materials analysis techniques beyond those that we sell with our spectrometers.

Similarly, we rely on collaboration with surface and materials scientists to provide new and interesting samples that require surface analysis. Often collaborations occur with Users who have an older generation of Kratos XPS instrument and don't have the current state-of-the-art capabilities of the latest generation instrument. An obvious example of this is the availability of a gas cluster ion source (GCIS). Although this accessory has been available for almost a decade, budget or age of spectrometer could mean that older instruments do not have this capability. Although Users are keenly aware that their samples would significantly benefit from the ability to sputter depth profile using the GCIS this capability may not be available to them.

Similarly the monochromated Ag Lα excitation source available on our Applications lab. instrument provides the potential for experiments with greater sampling depths, higher binding energy core level spectra or 'removal' of Auger feature overlaps with core level spectra, greatly simplifying data interpretation.

As highlighted in last quarter's Newsletter, we have recently added a basic glovebox to the applications lab. AXIS Supra$^+$. This gives us greater capability to mount and analyse air and moisture sensitive samples.

If this is beginning to sound a little like a sales-pitch, perhaps that's because it could be just that. We're seeking opportunities to collaborate that would lead to publication of results in the form of applications notes or even better, peer reviewed journal articles. If you think your materials characterisation research would benefit from collaboration with our Applications Specialists and use of the AXIS Supra$^+$ we'd encourage you to e-mail us with an outline of how we could collaborate.

So whether it's cluster depth profiling through a multilayer material, studies of buried interfaces with the higher photon energy Ag X-ray source or characterisation of air sensitive samples we'd like to hear from you.

Meet our Applications Team

Dr. Sarah Coultas Applications Manager

Dr. Jonathan Counsell Applications Specialist

Dr Nikki Gerrard Applications Specialist

i-work

Interview with an employee

Name Craig Allerton

Job title Purchasing Manager

How long have you been at Kratos?

Over 8 years

How would you describe your job at Kratos?

I manage a team of Buyers who purchase all the parts the company needs to manufacture our instruments. We manage the many suppliers based all around the world buying as diverse a group of products from labels to lasers, 1000's of differing parts, both simple and very complex.

Best part of your job?

Working in an excellent team, providing a purchasing service that makes a significant contribution to Kratos that helps produce final products that make a difference.

How did you end up at Kratos?

I've always worked in a purchasing team. I fell into a Purchasing role straight from university in 1997. I started working for Fujitsu as a Buyer of computer equipment. I worked there for 7 years, got a couple of promotions, completed my professional purchasing diploma (CIPS). I then worked in a manufacturing company making security camera's and video telemetry equipment for a couple of years. Before moving to Kratos my previous role was at a semiconductor manufacturer. I was there for a number of years buying some pretty nasty chemicals and gases amongst many other things. Having to wear "space suits" in the class 100 cleanrooms whilst auditing the wafer fabrication process was interesting. Then I got the opportunity to come and work for Kratos as a Strategic Buyer in 2013. I got promoted to Senior Buyer before being offered the Procurement Manager role in 2019.

What have you learnt working at Kratos?

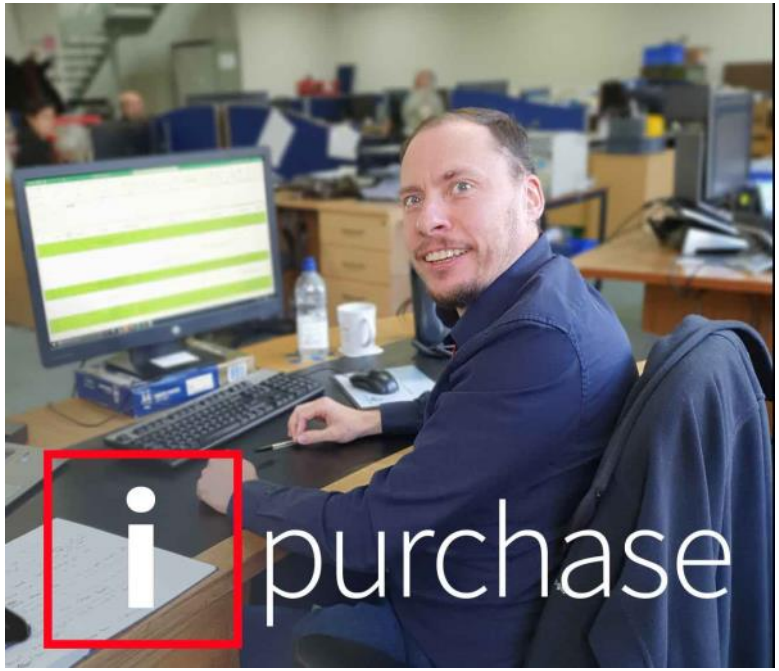
You don't have to know the intricacies of what you are buying but it does help. I've learnt what X-ray photoelectron spectrometers do and the importance of creating vacuum conditions amongst many other things.

What are the biggest challenges of ensuring parts are available for colleagues in production?

The impact of Covid on the global supply chains is unprecedented. Both in my experience and people that have been in manufacturing longer than I have they agree that its tough at the moment. Global markets have been really shaken by Covid as has most aspects of life. The initial lockdown during the start of the pandemic saw suppliers shutting as Covid cases rose. Markets such as the car industry stopped as demand dropped and materials became more and more difficult to find as production fell and staff were furloughed. In the second year of Covid that we find ourselves in, we have seen lead times extending on certain products to over a year as suppliers struggle to switch back on to increasing demand. Scarcity of materials and rising energy costs are pushing prices up. All this and we had to navigate the disruption brought about from Brexit too. Its been two years of having to be determined and persistent in ensuring materials arrive as production needs them. Given that Kratos had a record output year for both MALDI and Surface in the midst of all this was testament to how hard the Purchasing team had to work to make sure material supply disruptions were kept to a minimum.

Your favourite quote from a book?

"Even the smallest person can change the course of the future". Tolkien



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

I'm lucky to have a garden where I can potter about tending to plants, shrubs & flowers and grow fruit & veggies.

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

I've been quad biking on three different continents through deserts, forests and around lakes & mountains.

What is your motto or personal mantra?

It'll soon be Friday.

You don't have to know the intricacies of what you are buying but it does help.

Five decades of journal publications from Kratos

Leading surface analysis

The 'sales not science' philosophy is not something that we have ever adhered to at Kratos Analytical. Decades of experience in developing our instruments has taught us that it's embracing science that generates sales. This is why our scientists, engineers and applications specialists have always sought to collaborate and conduct novel research which is published as peer-reviewed journal articles, patents or contributions to international meetings.

This philosophy is well demonstrated in reviewing two publications authored by Kratos colleagues separated by over fifty years.

One of the very early publications presenting results from a Kratos (then AEI) spectrometer was published in Journal of the Chemical Society D: Chemical Communications in 1970 (received 31st October 1969) by Micky Barber and Dave Clark. At the time, the authors were affiliated to the Consultant Lab., AEI Scientific Apparatus Ltd and Department of Chemistry at The University of Durham respectively. Titled 'The theoretical interpretation of molecular core binding energies as measured by X-ray photoelectron spectroscopy', the paper stands out as one of the few physical chemistry contributions to the journal with most publications focussed on organic and inorganic chemistry.

In their paper Barber and Clark summarise that 'a theoretical interpretation of molecular core binding energies as measured by XPS is given and it is shown that simple relationships between "shifts" in binding energies and "charge distributions" can be misleading. They highlight the example of acetonitrile (H_3C-C≡N) where the calculated electron population is much higher on the methyl carbon than on the carbon attached to nitrogen. There is a one electron difference in population for the two atoms, the net atomic populations being 5.40 and 4.69 respectively. Based on correlations between shift and charge density, it might reasonably be assumed that there would be a substantial shift between the C 1s levels for acetonitrile and that the carbon bonded to the nitrogen should have the highest binding energy. The Al Kα excited C 1s X-ray spectrum was measured with 'the prototype of the AEI Scientific Apparatus ES 100 Spectrometer' and showed 'single line which is slightly broadened due to overlap of the two components'. The shift between these two components was 'estimated from a computer analysis of the line shape' and reported as <0.5 eV.

It is important to remember the context of Barber and Clark's publication. Kai Siegbahn had only published his seminal paper two years earlier. The technique was still very much in its infancy and spectra for this Chem. Comm. paper were recorded on a prototype spectrometer. Fast forward five decades to the latest collaborative publication from one of our applications specialists; High-throughput design of Na–Fe–Mn–O cathodes for Na-ion batteries.

Now a mature technique, XPS often plays a supporting role in materials characterisation. In this paper high throughput XPS analysis is combined with X-ray diffraction and cyclic voltammetry in a systematic study of an entire pseudo-ternary Na_xMO_2 system.

The high-throughput chemometric approach used a sol-gel method to synthesise 448 samples, each weighing 3-5 mg. XPS was used to systematically screen stability in air across the phase diagram and determine the oxidation states of Fe and Mn, including survey and narrow region scans. The electrochemical performance of these cathode materials is significantly limited by the material's stability in air. In this study, the carbon chemistry is also reported, with two surface chemistries identified for the C 1s. A low binding energy peak is assigned to aliphatic hydrocarbon contamination common for air exposed samples with a higher binding energy component indicating carbonate formation. Interestingly the molar fraction of carbonate varies from 22 to 216 % showing the dramatic impact that composition has in the surface reactivity. It is further noted that the carbonate content shows a clear trend with composition across the three phases of the materials studied. Discussion of the XPS results allow the authors to conclude 'the air/moisture stability and the mechanism of degeneration are substantially determined by the structure and compositions of the layered oxides and not all Na-Fe-Mn-O materials are equally prone to air sensitivity'.

The inclusion of XPS data for combinatorial chemistry and high-throughput materials discovery is testament to the major advances made in instrument automation, sensitivity and energy resolution in the preceding fifty years. Easy acquisition and reduction of vast datasets as well as output and visualisation facilitate the understanding of the XPS data. These are all attributes and expectations of a modern spectrometer and its associated data system. They are what allow our applications specialists to collaborate and learn what is required of a modern materials characterisation tool. But it is important that we do not forget the pioneering work of our colleagues and what that has taught us in the intervening 50 years.

The Theoretical Interpretation of Molecular Core Binding Energies as Measured by X-Ray Photoelectron Spectroscopy.
M. Barber and D. T. Clark
Journal of the Chemical Society D: Chemical Communications, (1), 22–23.
doi:10.1039/c29700000022

High-throughput design of Na–Fe–Mn–O cathodes for Na-ion batteries.
Shipeng Jia, Jonathan Counsell, Michel Adamič, Antranik Jonderian and Eric McCalla
DOI: 10.1039/D1TA07940A (Paper) J. Mater. Chem. A, 2022, 10, 251-265

MEET OUR USERS

Dr. Stuart Leadley, Dow Silicones Belgium, srl

What is your role at Dow Silicones?

I am a Senior Research Scientist in Dow's Core R&D Analytical Sciences organisation. I support Dow's different businesses and R&D groups with surface and interface character-isation. This can be new product development, catalyst research or customer support. In short, I help to answer questions.

Can you describe a typical day at work?

Fortunately, there is no typical day, which keeps the job interesting and provides new opportunities to learn. Because I help the businesses with customer support, urgent requests can arrive that have to take priority over the longer-term research and product development work. Part of the job is determining what is the true level of urgency and balance priorities. Whatever the request, I work with the partners to determine the questions that need to be answered. We then develop the hypotheses and the experiments required. Sometimes this does not include surface analysis and I then need to work with my colleagues to get their assistance. If we don't have the analytical tools in Belgium, I use my global network within Dow to have the work done.

You recently replaced your AXIS Ultra with an AXIS Supra$^+$. Since you've been using the new instrument, what can you identify as the biggest improvements?

Because Covid 19 has forced us to work from home a lot of the time, the biggest improvement is the level of automation. This means I only need to be in the lab to prepare samples and load them into the Flexilock. The ability to connect to the instrument via Dow's intranet means I can operate the instrument from anywhere. I have calculated that the sample throughput with Supra$^+$ is more than three times greater than Ultra. The addition of the gas cluster ion source means we can depth profile through silicones, which are Type I polymers. We are only really scratching the surface of its potential in our applications. Also, having an automated silver x-ray source, which can be used to access the silicon 1s chemistry and measure silicon Auger parameters in a more routine way.

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

We use the Supra$^+$ to understand different surface related phenomena. Here are a few examples from the last 12 months. Deposition of materials onto different surfaces, e.g. hair samples after treatment with shampoo and conditioner. Understanding root causes for loss of adhesion or surface contamination. Surface segregation of a constituents in adhesive and paint formulations. Changes in the surface elemental composition of catalyst materials

What do you see as the value of surface analysis?

Because a material's surface adapts to its environment, to truly understand how a material or product functions you need to understand its surface properties.

Silicones are often thought of as contaminants – where can they be useful?

Silicones have unique physical properties. They have very low surface energy (which is why they can become contaminants). They are water repellent, but they allow water vapour to pass through them. They are stable at very low temperatures and higher temperatures. They can be in different forms from liquids to resins structures. There are many thousands of different silicone products used in many different applications. Without silicone antifoams your laundry room would be full of foam. Silicone adhesives can be used to apply the screen protector to your smart phone screen and transdermal drug delivery patches. Self-adhesive labels would not be possible without a silicone release liner (the bit you throw away). Silicone sealants stop your bath from leaking and help hold together architectural glazing on glass fronted buildings. Silicone coatings on airbags can help you reduce injury in a car accident.

What has surface analysis taught you?

You can't beat entropy! A material's surface will adapt to its environment to reach the lowest energy state. This can be highly advantageous. It can also provide challenges due to contamination.

Any tips or tricks for surface analysts?

Good sample preparation can save hours of wasted time.

Kratos in Trafford Park

The city of Manchester is famous for being the cradle of the industrial revolution, with its factories and warehouses, many now converted into desirable apartments. But its less well-known neighbour, a few miles southwest of the city centre, has perhaps made a more significant contribution to the regions scientific and engineering developments. This area, known as Trafford Park, has been home to Kratos Analytical and its predecessors for over 50 years.

Trafford Park was built on land sold by one of the region's oldest families, the De Traffords. Towards the end of the 19th Century, Manchester's cotton barons became increasingly unhappy with the taxation by the Port of Liverpool so constructed the Manchester Ship Canal along the northern perimeter of then "beautifully-timbered deer park". The 36-metre-wide waterway allowed seafaring boats a direct route to the heart of "Cottonopolis" effectively opening a sea-port 30 miles inland.

1924 map showing Trafford Park almost entirely enclosed by the Manchester Ship Canal and the Bridgewater Canal

In June 1896 the entire Trafford Park was sold for £360,000 (2022 equivalent £42.6 M) leading to the creation of the world's first industrial estate. Showing foresight, Trafford Park Estates Ltd encouraged diversity in the goods manufactured on the estate in contrast to textile-dominated central Manchester. Initially Trafford Park embraced steel foundries, biscuit factories, and oil works.

The first American company to establish itself in Trafford Park was the Westinghouse Electrical Company. In addition to the factory, Westinghouse built a village for his workers on the American grid system of avenues and streets – still in evidence today. It is also possible to see the last remains of what was one of the UK's largest private railways around Trafford Park. In 1911 Ford opened its first factory outside the US. The well documented innovative production-line method was introduced and by 1920 output of 26,000 cars was achieved, with all parts also locally manufactured.

During the second world war Trafford Park's production was almost entirely turned over to supporting the war-effort. During this period employment figures on the estate reached 75,000. As highlighted in our last newsletter, our Company history can be traced back to this period with the development and subsequent manufacture of the magnetic sector mass spectrometers of AEI.

The end of the war marked the start of a slow decline in the fortunes of Trafford Park. It's documented that by the mid-1980s Trafford Park had virtually come to a standstill. The

Continued on next page

Kratos in Trafford Park *continued*

closure of some of Trafford Park's larger employers saw the emergence of a number of mini estates accommodating smaller companies. The establishment of an Urban Development Corporation provided a much-needed boost to the industrial estate. Improvement of the transport infrastructure and development of a show-piece site adjacent to the Quays lead to considerable success.

Kratos Analytical's fortunes had mirrored those of Trafford Park. The original Kratos buildings on Barton Dock Road were in poor repair and too large for the number of employees supporting our mass spectrometer and XPS instrument manufacturing. At the time that Kratos Analytical was purchased by Shimadzu Corporation it was decided to vacate the building and move to our current site at Wharfside. This coincided with the launch of the AXIS Ultra, our first spectrometer with parallel imaging capability. In his book, The first hundred years of Trafford Park, Nicolls [1] concludes that on the eve of the new millennium Trafford Park was now "a place of enterprise and optimism".

As you read this article, we have started assembling the 200th AXIS Supra/Supra^{+} XPS instrument. This is a considerable achievement, especially considering the national and international events of the last 24 months. It is also worth highlighting that, not only the current AXIS spectrometers, but every one of over a thousand X-ray photoelectron spectrometers that Kratos has manufactured has been designed and built in Trafford Park. We like to think that Kratos embodies the statement from 19th Century prime minister Benjamin Disraeli who famously suggested "what Manchester does today, the rest of the world follows tomorrow".

5 o'clock exodus from AEI. *from ref [1]*

Further reading

[1] R. Nicholls, Trafford Park: The First Hundred Years ISBN 1860770134 Phillimore & Co Ltd; UK ed. edition (1 April 1996)

https://manchesterhistory.net/manchester/gone/metrovicks.html

https://www.theguardian.com/cities/2015/apr/29/manchester-trafford-park-world-first-industrial-estate-history-cities-50-buildings

Looking forward ...

Meetings and exhibition attendance for 2022

Kratos Analytical and our agents are committed to supporting meetings throughout the coming year. Despite meetings being virtual we understand that they offer an ideal opportunity for us to learn what the surface analysis community needs and how XPS is applied to a wide range of materials characterisation. We look forward to 'meeting' during 2022.

UKSAF : Surface Physics at the Nanoscale
Wednesday 13th April 2022, Bristol University

Faraday Discussion on Photoelectron spectroscopy and the future of surface analysis
The meeting will focus on four main themes: 1) In-situ methods: discoveries and challenges, 2) Buried interfaces, 3) Time resolved surface analysis (kinetic timescale and molecular timescale) and 4) Future directions.
20-22 April 2022, London, UK & online virtual meeting

5th International Conference on Applied Surface Science (ICASS)
ICASS will report on and discuss current research on the role and use of surfaces in chemical and physical processes, related to catalysis, electrochemistry, energy, new/functional materials and nanotechnology. Also, the various techniques and characterization methods will be discussed.
25th - 28th April 2022, Palma, Mallorca, Spain

JSPE, Journées des Spectroscopies de Photoémission
The Photoemission Spectroscopy Days (JSPE, Journées des Spectroscopies de Photoémission) will be the first national event of the research federation « SPE » from CNRS, France.
16th - 18th May 2022, Dijon, France

48th International Conference on Metallurgical Coatings and Thin Films (ICMCTF)
AVS Advanced Surface Engineering Division. The ICMCTF is the premier international conference in the field of thin film deposition, characterization, and advanced surface engineering promoting global exchange of ideas and information among scientists, technologists, and manufacturers.
22nd - 27th May 2022, San Diego, CA, USA.

ECASIA : European Conference on Applications of Surface and Interface Analysis
The ECASIA biennial conferences since 1985 are aimed at, and intended for, those using surface analytical, surface-specific or surface-sensitive techniques for applied purposes.
29th May - 2nd June 2022, Limerick, Ireland

Kratos Users' Meetings
Save the date! Our Users Meetings for Asia, Europe and North America are still at the planning stage. It is likely that they will be on-line. We will be distributing further details in the next couple of months.
June 2022

Advanced Materials Show
The Advanced Materials Show will showcase the very latest in high performance materials technology for a range of applications including automotive, aerospace, energy and electronics.
28th & 29th June 2022, NEC Birmingham.
Kratos Analytical will be co-exhibiting with Shimadzu UK.

Microscopy & Microanalysis
Surface and Subsurface Microscopy and Microanalysis of Physical and Biological Samples
31st July - 4th August 2022, Portland, Oregon, USA

AVS 68th International Symposium & Exhibition
Theme "Imperfectly Perfect Materials"
Featuring Presentations on Emerging Topics Related to Materials, Processing & Interfaces
6th - 11th November 2022, Pittsburgh, PA, USA