KRATOS ANALYTICAL
A SHIMADZU GROUP COMPANY

NEWS 08 2022

News from the Applications Lab.
Questions, highlights and collaborations

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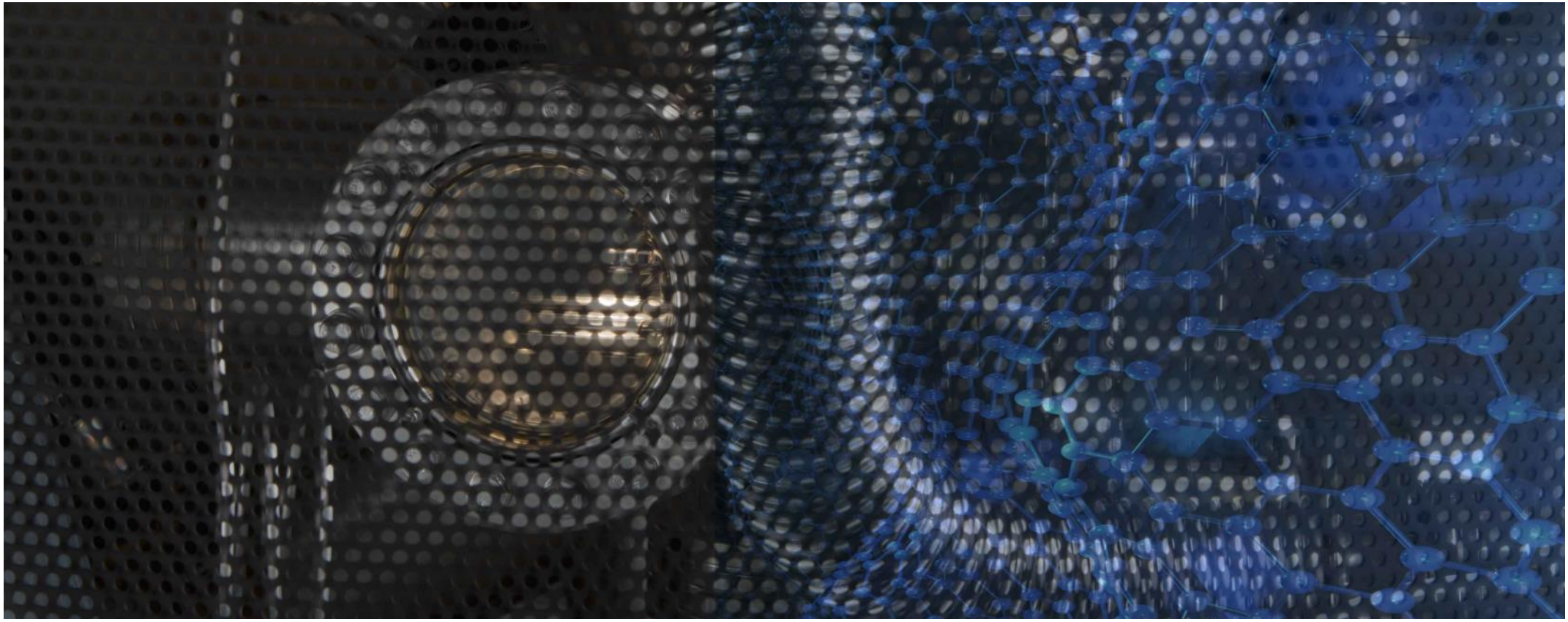
Exhaustive Identification
Similarities of Wordle and XPS peak ID

Meet our Users
Martin Moos, 3M Deutschland GmbH

Review of the European Users' Meeting
Hybrid meeting, July 2022



WELCOME TO THE AUTUMN KRATOS NEWSLETTER

Best for our customers

Whoops! We didn't publish a Summer Newsletter. With organisation of the European Users' Meeting and other commitments writing the Newsletter slipped down the 'to-do list'. But we're back with the autumn edition.

Our User interview is with Martin Moos who operates the AXIS Nova at 3M´s Corporate Research Analytical Laboratory in Neuss, Germany. We also interview one of Kratos Analytical's Technical Authors, Wendy Coy. If you've ever taken time to read one of our User Manuals you will be familiar with Wendy's work. We learn a little more of what is involved in her role within Kratos.

In news from the Applications Lab. we highlight some recent publications using the monochromatic Ag Lα source which not only demonstrate the capabilities of this accessory but also provide useful reference spectra for elemental and polysiloxane materials. We're also excited to highlight that we have shipped our 200th AXIS Supra^{+} to its new Users in China.

In the article titled *Exhaustive Identification* we highlight the similarities in solving the word-game Wordle to identifying the elements in a survey spectrum—see if you agree.

Our final article is a review of the European Users' Meeting. There are links within the article to recordings or the presentations, allowing you to learn what both colleagues at Kratos and our Users have been using their instruments for.

Enjoy the read.

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UPDATES FROM THE APPLICATIONS LAB.

Any questions?

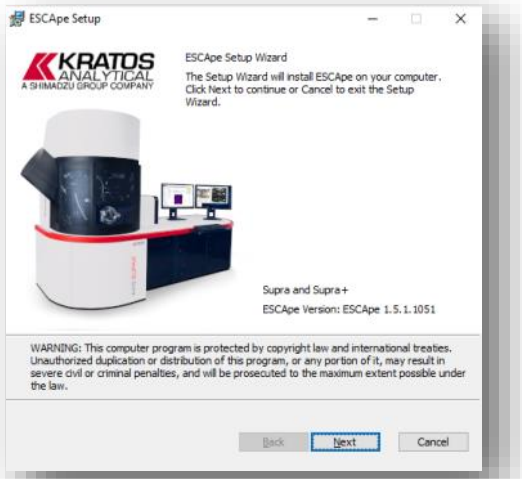
Have you ever wondered how something on your Kratos instrument works? Want to know the best way to process your data? Chances are that you're not alone. We'd be keen to provide answers to your questions in a new 'ask the experts' feature that we'll publish in the next Newsletter.

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

ESCApe 1.5 released

The latest version of our acquisition and processing software is now available for AXIS Supra / Supra$^+$ instruments.

The software and installation instructions can be downloaded from the members' area of the Kratos website.



The upgrade is configured to work for instruments running ESCApe 1.2 or ESCApe 1.4. If your instrument is running an earlier version of software, it is essential to install one of the intermediate versions as there were some significant changes to the firmware that must be implemented. Contact your service centre for specific instructions in this case.

Whilst this version of software has a few new features available to Users, the bulk of the changes are 'behind the scenes'. Based on instrument logging over extended periods we have optimised a number of key operating parameters.

To benefit from these changes it's important that your instrument is running the latest version of software. Using ESCApe 1.5 software also ensures that our Service Engineers have the full capability for remote support of the instrument if an intervention is required. **Please ensure that you upgrade to ESCApe 1.5 as soon as possible.**

Ag Lα excited reference spectra published

Between sample analysis for and instrument demonstrations for prospective customers, our colleagues in the Applications Lab. have been busy generating reference spectra using the monochromated Ag Lα excitation source.

This work has resulted in 4 publications in Surface Science Spectra (SSS) since our last Newsletter. Dr Gerrard was lead author in publishing XPS analysis of group 4A elements including Si, Ge, Sn and Pb). Dr Counsell published reference spectra for coinage metals (Au, Ag and Cu) and molybdenum di-sulfide (MoS_2) and Dr Coultas published spectra from a number of first row transition metals (Fe, Co, Ni, Cu and Zn). As highlighted elsewhere in this Newsletter, the publications are open access and data can be downloaded and read in the free eSpectra plotting, comparison and sharing tool developed and supported by the American Institute of Physics.

We're also pleased to highlight the recent publication from Dr Stuart Leadley who was our featured User in the last Newsletter. In the publication 'Applying monochromated silver X-rays to the surface characterization of silicon-containing materials' Dr Leadley and co-authors report the binding energy shifts in Si 1s, 2s, and Auger spectra, which will allow the identification of silicon chemistry from polysiloxane materials.

AVS 68th International Symposium & Exhibition

Kratos are Bronze sponsors of the annual American Vacuum Society symposium and exhibition. We are also presenting 2 posters and 2 oral contributions at the meeting which runs from the **6th - 11th November 2022** in Pittsburgh, PA, USA.

We appreciate that many reading this will not be attending AVS so we'll share the presentations after the meeting.

200th AXIS Supra leaves the factory

This month the 200th AXIS Supra/Supra$^+$ completed test and was signed off by our Quality Department, ready to ship to its owner. Kratos' Production Manager, Billy Hughes, comments 'the 200th instrument is a significant milestone for Kratos. Building and testing an AXIS Supra+ requires large and dedicated team. Great credit goes to colleagues involved in the production and test of this and every surface analysis instrument.'

i-work

Interview with an employee

Name Wendy Coy

Job title Technical Author

How long have you been at Kratos?

Just approaching 11 years.

How would you describe your job at Kratos?

I write the documentation for Kratos. The documents can be anything from instructions on how to build the instruments through to how best to use them. Sometimes I'll have to write the documents from scratch, other times it will be editing other people's work.

Best part of your job?

I enjoy the breadth of the documents that I work on. It means that I get to interact with a wide range of colleagues within Kratos. If I'm working on a manufacturing instruction for one of the sub-assemblies of the spectrometer I will work closely with the fitters and wiremen on the shopfloor. In parallel to that I could be writing a User document for an accessory and learning and documenting how it is used from our Applications Specialists. I do find it important to understand how the instrument works, including most aspects of the acquisition software, so that I can write a manual that will be useful for the User. I'm probably one of the few people at Kratos that sees the products from initial build through to its use in the lab. I enjoy the fact that this job provides insights into the instrument that most people don't get.

How did you end up at Kratos?

I graduated with a Physics degree from Leeds University and then gained a teaching qualification. After teaching for 3 years I decided that it wasn't a career I wanted to pursue. I fell into the job of technical author not really knowing that such a job existed. I learned that Ferranti had a vacancy, applied and I got the job. I ended up in a similar rôle at Kratos after hearing about it from a former Kratos colleague, Bill Stevens, when we were both at a monthly Institute of Scientific and Technical Communicators meeting in Manchester.

What have you learnt working at Kratos?

I've learnt a lot from the people on the shop floor, especially the manufacturing processes and what is required to build the instruments. At various times, I've also been reminded of some of the physics I studied and haven't used for some considerable time.

Wendy collaborating with physicist, Dr. Boxford, on drafting a User manual.

What is the most useful gadget you own?

Last year while I was laid up after a nasty break to my leg I really relied on 'Alexa'. It was great to be able to play music, listen to the radio, switch lights on or off all without getting up! It's just a shame she can't cook and wash-up!

What is the best or worst manual that you have ever used?

In the past the worst manuals were those that were poorly translated, usually from Chinese. You still see them now and again. But the most recent one was for a multi-mode kitchen blender. There were no words, just hundreds of tiny pictures so small you needed a magnifying glass to be able to see them. I guess it solved the issue of translation to different languages but it was particularly useless. I also had 'fun' with an IKEA manual last year when I was helping my son build a wardrobe. Not quite as bad as the blender, but I think you need to 'get your eye in' to follow IKEA instructions. It was frustrating having to backtrack and disassemble because we'd missed a component.

I asked a colleague the same question and we both agreed that Lego instructions are probably the best, although it's a long time since I've built a Lego model.

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

I enjoy reading and keeping my mind ticking over with puzzles such as Sudoku. I also enjoy heading out for walks—nothing too strenuous, I'd consider 5 miles about long enough. I love being by the seaside or moving water, finding it relaxing. A walk around Salford Quays during my lunch break recharges the batteries.

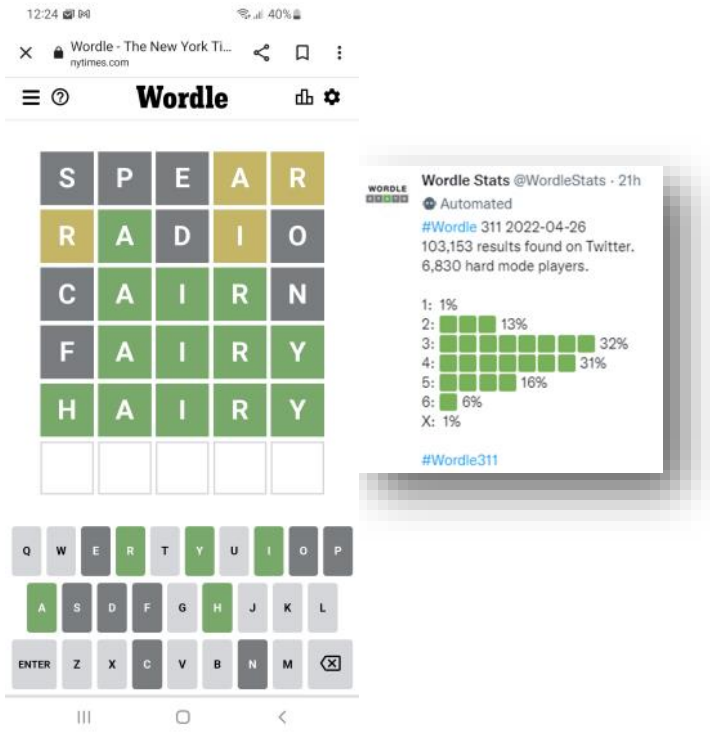
Tell us one thing that we don't know about you?

During lock down I started making cheese. I had particular success with ricotta and a Canadian curd cheese which can be added to fries and gravy to make poutine. Cheese making is quite a laborious process, needing bursts of attention throughout a day, so I've been less productive since lock-down finished. It's quite a straightforward process and you can get good starter kits online.

EXHAUSTIVE IDENTIFICATION

Thoughts on the similarity of Wordle and peak identification in an XPS survey spectrum

I'm sure that you must have tried Wordle – the online word puzzle? You get 6 goes to guess a 5-letter word. It's quite addictive. Probably more so because everybody does the same puzzle each day. We may have now passed peak-Wordle, but a quick look online shows that over 103,000 people have shared their Wordle score on Twitter today. There's likely to be millions more Worldling in private and not sharing their score.



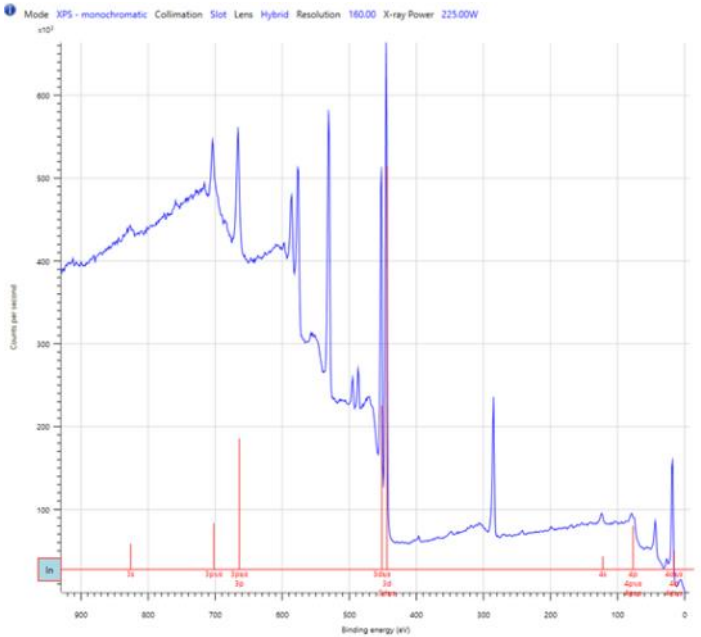
Like many word puzzles, Wordle is really a numbers game dressed-up as a letters puzzle. The crucial strategy is one of "exhaustion" explains Alex Bellos, a mathematician and puzzle compiler for the Guardian newspaper. 'You have a finite number of solutions, and you have to exhaustively look at every permutation and combination. It's a natural instinct for mathematicians'.

Which lead me to think that it's not dissimilar to identifying peaks in an XPS survey spectrum. Like Wordle, you will have your starting elements. Carbon – ubiquitous to just about all samples - will have a 1s core level around 285 eV binding energy. Oxygen, also extremely common, with its 1s peak around 530 eV and a corresponding Auger series first peak maximum around 975 eV binding energy. As well as being commonly measured, both carbon and oxygen 1s core levels have reasonable cross sections, meaning that they're relatively easy to measure. Just as with the first 5-letter word in Wordle, the first identification of elements for the survey spectrum can give a confident start in determining the elemental composition of your sample.

After carbon and oxygen, for an unknown sample, the next elements to look for would be either nitrogen (its 1s is around 400 eV BE) or silicon (2p is around 100 eV BE). Silicon is an easy one to confirm as its 2s component will be obvious around 150 eV BE, with similar intensity to the 2p.

However, if there's aluminium in the spectrum as well as silicon, peak identification can start to get a little more exciting. Permutations and combinations need to be considered. Aluminium's primary 2p peak is around 72 eV, the 2s at 119 eV and Auger KLL features centred around 99 eV – these transitions possibly overlapping with the Si 2p.

From there, the move is to less-common elements. Some will have instantly recognisable peaks, or combinations of peaks. But this is only true if you are familiar with the photoelectron survey spectrum of that element. What happens for the novice spectroscopist, or even the experienced one analysing a new element? Prior knowledge of sample composition is invaluable in guiding and confirming elemental identification. Thankfully, there are also tools available to help elemental peak identification both in the data processing software and online databases.



ESCApe software has a useful tool that superimposes the photoemission lines associated with an element in their relative intensities. This allows a quick visual comparison of peaks in the survey spectrum with those of the element, as shown in the figure. ESCApe software will go a step further, with the ***Peak ID** data processing* tool. This clever algorithm runs through the survey spectrum and identifies the binding (kinetic) energy of every peak. The positions of the peaks in the spectrum are then compared to a database of elemental core-level transitions, allowing the assignment of each peak to an element. It works

EXHAUSTIVE IDENTIFICATION (continued)

Similarity of Wordle and elemental peak identification

well with 'well behaved' survey spectra and is especially good at mathematically identifying very low intensity peaks that are not easily seen by eye. Auto peak identification can begin to struggle if individual core-level peaks have a large chemical shift, or worse still when sample charging has caused the whole survey spectrum to be shifted in energy.

Even with such tools available to help peak identification it's a case of 'spectroscopist beware'. There is still the necessity for the analyst to confirm assignments. What confidence would you place in assignment of the peak at binding energy 102.8 eV to Ga $3p_{3/2}$, is it more likely to be Si 2p shifted to higher BE? We return to the need for an exhaustive evaluation of every permutation and combination again!

There are several other extremely useful resources available. The Handbook of X-ray Photoelectron Spectroscopy [1] is a valuable reference book of standard spectra for identification and interpretation of XPS data. The handbook presents survey spectra and high-resolution scan of the most intense core-level peak excited with monochromated Al Kα and non-monochromated Mg Kα x-rays. It also provides tables of energy shift data for commonly analysed compounds for which the specific elements are found sourced from available literature when the handbook was published in 1992.

More recent, peer-reviewed, reference data is published in Surface Science Spectra. The published archive extends to over 1300 specimens of interest with downloadable data, providing usable reference spectra in unsmoothed, original format giving confidence that data is not dependent on someone else's interpretation. Spectral analysis can be taken a step further by using eSpectra [2]. This interactive tool allows you to plot and visualise XPS data against the SSS database, with the added confidence that the XPS database has been peer reviewed by subject experts. Uniquely, eSpectra also allow registered Users to upload their data to the site and share it with the eSpectra community – making more data from industrial, government, and university labs. readily available.

So, if it's the deduction of a 5 letter word in Wordle, or the identification of the elemental composition of a specimen from its XPS survey spectrum, the challenge is similar. We're following that strategy of 'exhaustive identification'.

[1] J.F. Moulder et al Handbook of X-ray photoelectron spectroscopy – A reference book of standard spectra for identification and interpretation of XPS data. Published Perkin-Elmer Corp. ISBN 0-9627026-2-5

[2] eSpectra https://espectra.aip.org/app/#/home

MEET OUR USERS

Martin Moos, 3M Deutschland GmbH

Name and Company:

Martin Moos - 3M

Affiliation:

Product Development Specialist

What is your role in your company?

I am a surface analyst in 3M´s Corporate Research Analytical Laboratory in Neuss, Germany with responsibility for Scanning Probe Microscopy, X-Ray Photoelectron Spectro--scopy and wettability analysis.

How do you use Kratos instruments in your role?

We have a history of using advanced surface analysis techniques like XPS at 3M. Within the Corporate Research Analytical Laboratory in Neuss, Germany, we currently use a Kratos Axis Nova.

For my work large and versatile sample stages and automated loading options are very useful. I often face 'real world samples' which can be relatively large and at times have rather complex geometries. On a normal XPS analysis day, you may find all the instrument´s sample platen types in operation and that includes angle resolved and Zalar rotation platens.

Samples on XPS stage from a mechanical adhesion test.

In addition, the instrument is equipped with a multi-mode gas cluster ion source (GCIS) for sputter cleaning of samples or depth profiling experiments on metallic, organic or inorganic films.

It is not uncommon in our lab to schedule a run where things start with simple large area analysis, before doing imaging and selected area spectroscopy on a second platen and finally moving onto a Zalar platen to do depth profiling.

What do you see as the value of surface analysis?

The way in which materials interact with each other is through their surfaces and interfaces and hence this sample regime is crucial for their performance in any given environment.

Adhesives are a practical example. They can be so strong that they can hold a structural panel for the life of a building or so gentle they can be applied to human skin to secure tubing.

Applying an adhesive to an ill prepared or contaminated surface will lead to compromised adhesion and ultimately bond failure. That is a common example where surface analysis comes into play.

On a larger scale, surface analysis allows us to understand the complex physicochemical interactions happening at molecular level which translate into many products that we see and interact with around us. Due to the breadth of our surface analytical instrument-ation, we can approach scientific questions from different angles and thus offer a broader view on the surface.

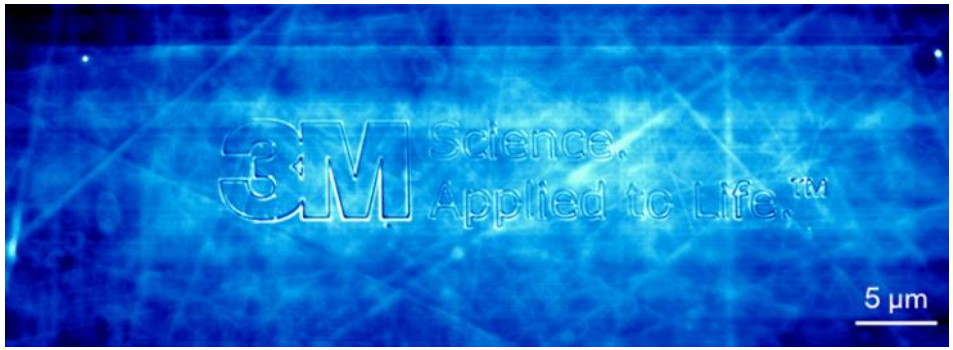
Any tips or tricks for surface analysts?

I would like to recommend the following collection of scientific papers:

Reproducibility challenges and solutions with a focus on XPS, [D. Baer et al., J. Vac. Sci. Technol. A 39, 021601 (2021)]

What has surface analysis taught you?

The variety in conceptions of the term "surface" constantly reminds me of the variety in perspectives that exist when different people are talking about what they think is the same.



Scanning Probe Microscopy - ca. 50 nm deep scratching on a polycarbonate surface.

A REVIEW OF THE EUROPEAN USERS' MEETING

In July we hosted our European Users' Meeting at Emirates Old Trafford just a few days before England were comprehensively beaten by India in a one-day international cricket match. Thankfully, the Users' meeting was much more successful. As well as those attending in-person, we were joined by 25+ online attendees. In the review below we include links to recordings of the presentations where available.

Dr Chris Blomfield, Director of Kratos Analytical Ltd, opened the meeting with a welcome address and review of the Surface Analysis business over the last 2 years, highlighting the successes and record sales for the last financial year. The first presentation of the scientific sessions was given by Dr. Dave Morgan, Cardiff University titled Revisiting the Beamson and Briggs Degradation Index with Modern Spectrometers. He highlighted the care required when using high-power monochromatic X-ray sources and more importantly the effect of neutralisation method on polymer degradation. Next to present was Dr Alex Shard from NPL. In his presentation, Calibrating XPS intensities, Dr Shard discussed revisiting work from the 1990's and highlighted work that was undertaken to determine the transmission function of the Kratos AXIS spectrometers using the Ag Lα excitation source.

The theme of HAXPES was continued by Dr Sarah Coultas, Applications Manager here at Kratos. She presented **Ag monochromatic excitation - A practical guide to its use** in which the advantages of using the higher energy photon source were outlined using a number of applications examples. Dr Stuart Leadley from Dow Silicones, presented **Applying Monochromated Silver X-rays to the Surface Characterisation of Silicon Containing Materials**. This was an interesting talk presenting data from a number of model substituted silicones, materials that span the interface between organic and inorganic materials. The final contribution of the opening day was Characterisation of the SEI layer on a LIB anode by Dr Adam Roberts, presenting data generated by Shimadzu colleagues in our Kyoto applications lab.

Our social proved extremely popular. We spent an evening with mixologist Jamie at the Dirty Old Town Distillery, home to Salford Rum. We learnt about the history of rum and the spices used to give the spirit its unique flavour as well as trying the 3 rums produced by Salford Rum.

The second day of the meeting was started by Dr Lena Patterer and Eva Mayer, presenting Hydrogen-based reduction of iron oxide thin films studied by in-situ XPS. In their presentation they highlighted reduction of model catalysts in the form of iron-oxide films by back-filling the load-lock with an argon-hydrogen gas mixture. Our second presenter of the day was Dr Erdni Batyrev from Tata Steel, Netherlands who talked on a **NAP XPS concept for Kratos ULTRA DLD**. This was followed by an innovative presentation from Dr Marc Walker from the University of Warwick. In his presentation, 70 Shades of XPS - an Adventure in to the Unknown, Marc allowed the audience to nominate an element from a list of the 70 that he has analysed during his career. The nominations were drawn from a bag and Dr Walker presented a slide with data from that element with the motivation for the study. The final morning slot was filled by Dr Jacqueline van Veldhoven, TNO, Netherlands presenting Multi-technique surface analysis for the EUV-lithography industry. This was a great overview of the use of surface analysis to investigate how optical surfaces (mirrors and masks) are affected by exposure to EUV-environment.

After lunch, our applications colleague Dr Jon Counsell presented a compilation of applications and a recent publication which have utilised the array analysis functionality of the ESCApe software in his talk Array analysis - measuring lateral inhomogeneity. Another informative talk from Dr. Will Boxford, Physicist at Kratos Analytical highlighted the Principles and applications of the Gas Cluster Ion Source. His presentation was a good mix of theory, application and practical tips.

Feedback was very positive, with the ability for Users to meet Kratos colleagues as well as other Users the primary motivation stated for attending in-person. It will be interesting to see if there are any collaborations initiated after discussions between Users at this meeting.