

Improved spectroscopy instrument generates \$2m in export sales

For 50 years, Manchester-based Kratos Analytical has been a leading manufacturer of X-ray Photoelectron Spectroscopy (XPS) instrument, and a prominent player in surface analysis. It is a UK advanced machinery exporting success story, with 75% of its customers outside the UK, mostly in the United States, EU, China, Japan, and Korea.

Its instruments are sought after for cutting-edge materials research and testing, from studying the surface chemistry of battery materials to improve efficiency, to developing surface treatments for the miniaturisation of semiconductors, to researching nano- and bio-materials with novel surface properties. As such, it plays a critical role in enabling some of the world's most important, yet complex, innovations.



Challenge

As a world leader in surface measurements, Kratos is comfortable mapping any surface chemistry. But increasingly, customers want to go deeper, probing beyond the surface, to 50-100 nm below. Many new electronics and biomaterials involve combining thin layers of materials with different properties to create unique functionalities. The companies behind these nanoscale innovations need to measure the surface properties of each of these layers.

On paper, Kratos' had the capability to do this. Its AXIS Supra+ XPS surface analysis instrument has two X-ray sources that can measure surface properties up to 10 nm and 20 nm deep respectively. To go deeper, it can employ a technique called Ar-ion sputtering, which fires ions at a surface to remove material in thin layers, allowing the XPS instrument to measure beneath the surface. Repeating this process again and again, alternating sputtering and analysis, gives the scientist an understanding of the material's chemistry at depth.

The problem is sputtering can damage the material below or leave residue from the cleared layer on top of the newly exposed layer. "It's like sandblasting, but on the atomic scale, using ions instead of sand grains." says Dr Benjamen Reed, Higher Scientist at NPL, who led the NPL team for this project. "And on both scales" he continued, "it's an aggressive method for removing material. It's often unavoidable that in trying to 'sandblast' your way to a buried layer to measure its chemistry, you end up damaging it."

That affects the integrity of the resulting measurements in ways that Kratos did not fully understand, and so they were unable to guarantee high-resolution measurements beyond 20 nm using Ar-ion sputtering. That was becoming a blocker to sales, with potential buyers deciding not to go ahead with a purchase because of a lack of confidence in the measurement quality at this depth.

Solution

Kratos understood that removing the contribution of the damage layer from the data was possible but lacked the expertise to overcome the interference caused by surface removal. So, they approached NPL through the A4l programme.

NPL brought a deep understanding of the physics of how electrons – such as those generated by the different X-ray sources of the Kratos XPS instrument – move through materials. That allowed them to model the impact of Ar-ion sputtering on the buried surfaces, and remove these effects from the measurement data, leaving just the data from the buried surface measurements.

"After you have 'sandblasted' your material", says Dr Reed, "you will have almost certainly damaged it, changed its chemistry, roughened it up, or left some 'grains of sand' behind. This damage is usually at the surface of the material, and forms in a layer. Our analysis method is able to remove this damage layer from the final measurement data, so that we end up with a 'pristine' measurement of the buried material, i.e. what we would have measured had the damage not taken place."

A physics-based model that uses data collected using two X-ray energies was developed by NPL into an algorithm that analyses Kratos measurement data, and removes the damage caused by the sputtering process. That gave users of Kratos instruments accurate surface chemistry data on each layer of the sample, even after sputtering.

Impact

This advancement not only solidifies Kratos' position as a global leader in the XPS market, but also enables its clients to improve product development and quality control processes. "That should lead to better manufacturing techniques for semiconductors, batteries, and other novel materials," says Dr Jonathan Counsell, Applications Manager at Kratos Analytical Ltd. A publication is also being worked on.

"We are lucky we have NPL on our doorstep," he adds. "They have played a key role over the past decades in developing XPS as a technique, and their surface analysis team is head and shoulders above other metrology groups in this area. Our customers also recognise that, so being able to say that the algorithm is NPL-approved gives our buyers a lot of confidence."

"We were also lucky to have access to the A4I funding. Without A4I and NPL, we probably couldn't have done it", he adds.

"Having this improved capability has removed a big blocker to sales of our leading XPS instrument, particularly in the semiconductor and device manufacturing industries" says Dr Counsell. "That has directly translated into around \$2m additional annual revenue that we just wouldn't have had without the NPL algorithm".