Ambient pressure XPS at MAX IV Laboratory and how it is used for atomic layer deposition research

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The discovery of novel materials and new chemical processes requires the detailed understanding of their constituent sub-processes. *In situ* techniques are invaluable in such fields of science and engineering, since they enable investigating the materials under realistic conditions which are close to those the materials are subjected to in their intended operational areas. Ambient Pressure X-ray Photoelectron Spectroscopy (APXPS) is one such *in situ* tool allowing one to obtain detailed elementally specific, chemically relevant and surface sensitive information from a wide range process and samples serving different scientific disciplines. APXPS is, in its simplicity, merely a way of conducting XPS measurements under higher pressure conditions. Typical tools, such as the ones presented here, allow measurements at pressure of tens of mbar and temperatures of several hundred degrees Celsius.

Typical XPS investigations are often limited to pressures in the ultra-high vacuum regime. Such pressures are far from the values that are found in real industrial chemical and materials manufacturing processes. Therefore, it is if utmost interest to study the materials in the conditions that are relevant for their manufacture in order to find routes of optimisation, energy efficiency, and other improvements.

One such area of research is atomic layer deposition (ALD), which is nowadays the most common tool used to create nanosized devices used in many different areas of nanoelectronics research. ALD relies on the cyclic exposure of the substrate surface to different precursor molecules. These precursor molecules will react with the surface and ideally create individual atomic layers. The desired layer thickness is realized by continuing to cycle the substrates over the surface until enough material has been deposited. Due to the self-limiting nature of the half- cycles, ALD creates uniform and chemically pure surfaces, in the ideal case. However, in many processes, it is not accurately known how the chemical reactions follow one another, especially in the details of the reactions occurring in the sub-cycle timescale.

In this manner, APXPS and ALD can together form a powerful combination that can provide information which has so far been inaccessible. Due to the pressure range of typical ALD processes being in the range that can be used with APXPS, one can truly use the power of XPS to obtain information from the surface layers as they are being deposited. Such a system has been built at the SPECIES beamline at the MAX IV Laboratory [1]. The setup has been used to investigate several different ALD processes to find out details on the ALD of TiO_2, HfO_2, platinum, and others, on many different substrates [2-5]. Information gained so far includes details about oxygen migration, roles of pressure and temperature, and the details of the individual half-cycles.

References

1. E. Kokkonen, *et al.*, Rev. Sci. Instrum. 93 (2022) 013905.
2. G. D'Acunto *et al.*, ACS Appl. Electron. Mater. 2 (2020) 3915.
3. G. D'Acunto, *et al.*, Surf. Interfaces 39 (2023) 102927.
4. R. Jones, *et al.*, Surf. Science 753 (2025) 122656.
5. E. Kokkonen, *et al.*, J. Vac. Sci. Tech. A 42 (2024) 062406.