Extended Abstract of PSA-19 (review)

O-33

Quantitative HAXPES

Alexander G. Shard,[1],*
[1]*National Physical Laboratory, Hampton Road, Teddington, TW11 0LW, UK*
*corresponding author's e-mail: alex.shard@npl.co.uk

(Received: May 01, 2019; Accepted: Aug 16, 2019)

An overview of the lab-based HAXPES is provided, along with progress in making this technique quantitative. This is necessary to enable it to be used to its full potential. The use of HAXPES and inelastic background analysis demonstrates excellent prospects for elemental depth-profiling of ~100 nm thick multilayered films.

1. Introduction

Technologically important materials systems involve buried layers and interfaces on depth scales of tens to hundreds nm below the surface, well beyond the traditional information depth of XPS. It is often necessary to measure such materials without using a destructive process such as sputter-depth-profiling. This may be enabled using photoelectron spectroscopy with photons having a higher X-ray energy than traditional XPS instruments, so-called 'hard X-ray photoemission spectroscopy', HAXPES). For most elements this enables a modest increase in information depth for core level photoelectron peaks, scaling somewhat less than linearly with electron kinetic energy. However, much greater depth distributions may be probed using the inelastic background, arising from photoelectrons originating much deeper into the sample, having undergone some energy loss [1-3].

Many manufacturers are equipping their instruments with higher-energy X-ray sources, examples include Kratos with an Ag Lα source [4] which offers twice the X-ray energy (2984.4 eV) of the Al Kα source and uses the same monochromator. Much higher energies are now available in a laboratory system using an intense Ga Kα metal jet X-ray source at 9.25 keV [5] from Scienta Omicron. Such instruments offer better accessibility to HAXPES by industry. Previously, the technique was only available at national-level synchrotron facilities.

There are many challenges ahead before such instruments achieve the usefulness and reliability of traditional XPS instruments.

2. Analyser transmission function

In traditional XPS instruments the transmission function is typically determined using one or more reference spectrum appropriate to the instrument configuration. To ensure that the data can be interpreted using physical theory, it is necessary to have accurate reference spectrum, and this was provided by NPL many years ago [6]. It is not clear how this may be achieved with HAXPES instruments without considerable effort, however for the Ag Lα source, where the X-ray spot size and geometry is identical to the Al Kα source in the same instrument, this can be addressed through extension of the NPL method [4]. For other instruments and, particularly for synchrotron beamlines, this is a challenge that requires a solution before quantitative HAXPES measurements may be made.

3. Physical data

Thanks to the efforts of many researchers, data for photoionisation cross-sections [7,8], inelastic mean free paths [9,10] and single scattering albedos [11] are now available. These enable us to provide useful relative sensitivity factors and algorithms for data interpretation that can be applied to HAXPES data. In the case of the Ag Lα source, these are available online as supporting information [4].

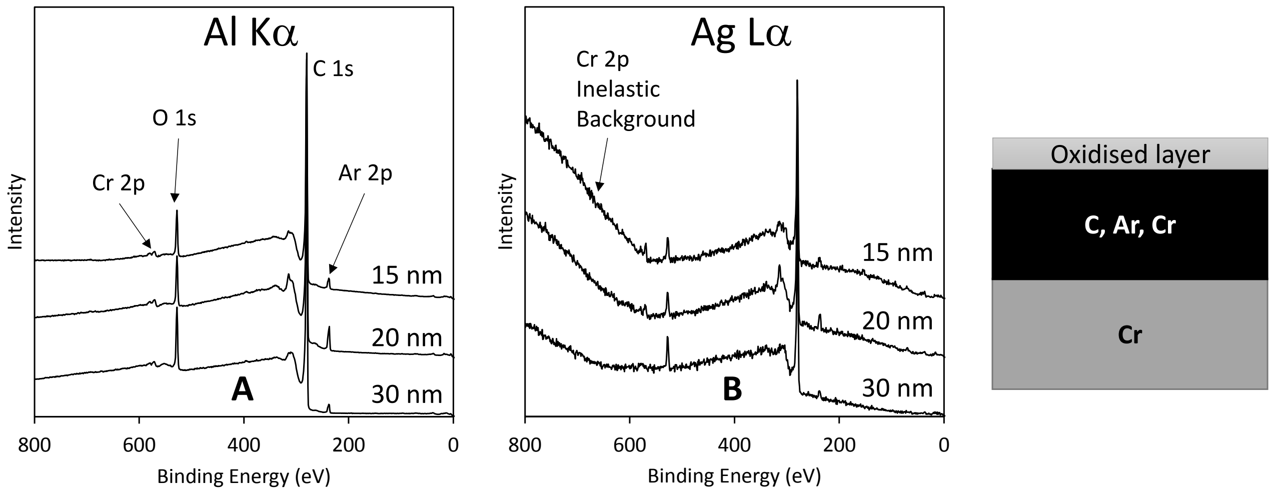

Fig. 1 Three samples of silicon wafer coated with chromium (40 nm) and carbon (with thicknesses 15 nm, 20 nm and 30 nm) by magnetron sputtering and analysed using Al Kα (A) and Ag Lα (B) radiation in XPS. Data are plotted as intensity against binding energy. Quantitative analysis reveals the presence of surface oxygen and trace chromium and argon throughout the carbon layer. The presence of the underlying chromium layer can be inferred from the inelastic background in the Ag Lα spectra [4].

4. Applications

As well as providing greater depth information in the peaks, HAXPES has the capacity to measure the structure of intricately layered structures using the inelastic background. In Figure 1, a silicon wafer coated by magnetron sputtering with chromium and then carbon is analysed using 1486.6 eV and 2984.4 eV X-rays. The higher energy Ag Lα XPS spectrum demonstrates the presence of the underlying chromium layer and this feature is barely visible even for the thinnest carbon film using the lower energy Al Kα source. The combination of HAXPES and inelastic background analysis has the capability to provide information from over 100 nm depth. This is demonstrated in Figure 2, which shows that gold is identifiable under 150 nm of organic coating and the overlying thickness can be established, in terms of the inelastic mean free path, to ~10% error by analyzing this data.

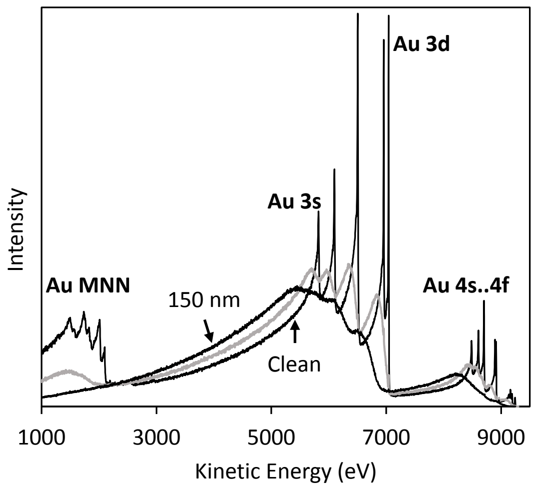

Fig. 2 Clean gold and gold coated with 50 nm (grey line) and 150 nm of organic material and analysed with Ga Kα HAXPES.

5. References

[1] S. Tougaard, *Surf. Interface Anal* **26**, 249 (1998)

[2] O. Renault et al., *Appl. Phys. Lett*. **109**, 011602 (2016)

[3] C. Zborowsk et al., *Appl. Surf. Science* 432, 60 (2018)

[4] A. G. Shard et al., *Surf. Interface Anal* https://doi.org/10.1002/sia.6647 (2019)

[5] A. Regoutz et al., *Rev. Sci. Instr*. **89**, 073105 (2018)

[6] M. P. Seah, *J. Electron Spectr. Rel. Phenom*. 71, 191 (1995).

[7] M. Trzhaskovskaya, V. Yarzhemsky, *Atomic data Nuclear data tables* **119**, 99 (2018)

[8] L. Sabbatucci, F. Salvat, *Radiat. Phys. Chem*. **121**, 122 (2016)

[9] S. Tanuma et al., *Surf. Interface Anal*. **43**, 689 (2011)

[10] M. P. Seah, *Surf. Interface Anal*. **44**, 497 (2012)

[11] A. Jablonski, *Journal of Physics D: Applied Physics* 48, 075301 (2015)