The Development of Quantitative Analysis by AES and XPS via Four Experimental Data Bases and an Interlaboratory Study

by

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Quantification in Auger electron spectroscopy (AES) and X-ray photoelectron spectroscopy (XPS) is usually effected using sensitivity factors. This approach is for samples that are homogeneous over the analysis volume. Sensitivity factors were based on what are now called pure element relative sensitivity factors (PERSFs) and, early on, it was recognised that additional matrix factors were needed for accurate analysis. Without these matrix factors, errors of up to a factor of 7 could occur in AES and up to 3 in XPS. Various laboratories provided experimental sensitivity factors since the differential values required for AES were not easy to calculate, and manufacturers knew that their instrument transmission functions would modify the factors to be used.

As a result of a sustained programme of work at NPL, a new framework is in place that will be described. First, a spectrometer intensity calibration system was established [1] that is now available to all analysts [2]. Next, databases for AES and XPS were established to test the theoretical predictions [3,4]. To do this, a REELS database [5] was also established. Using the latter, the true background subtracted spectra may be determined and the peak areas compared with theory [6]. Checks and tests of different components of the theory allow the cross sections, etc to be selected, leading to an agreement between experiment and theory of approximately 10%. The correct parameters to use will be described. In the development of this approach, a more effective way of calculating quantities using average matrix relative sensitivity factors (AMRSFs) in place of the PERSFs, was formed. This led to linear equations that could be applied to any number of elements in the sample in a non-iterative manner and not requiring further matrix factors. These are currently the best, recommended, sensitivity factors for analytical use.

For samples that are not homogeneous, layer thicknesses are often required. For ultra-thin layers, non-destructive methods such as angle-resolved XPS (ARXPS) are used [7-9]. In a wide study of methods under the auspices of the Consultative Committee for Amount of Substance, the accuracy of measurements of the thicknesses of SiO_2 layers up to 8 nm thick on Si have been assessed. These lead to recommendations for XPS measurement for layer thicknesses[10,11]. For thicker layers, sputtering is generally used. Here a new method has been tested to generate a small sputter yield database that we have used to test and develop the accuracy of different theoretical predictions.

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