Application Note: 31068

Energy Resolution in X-ray Photoelectron Spectroscopy (XPS)

Key Words

- ESCALAB 250
- Surface Analysis

Introduction

The ultimate energy resolution of an XPS spectrometer is usually measured using the full width at half-maximum intensity (FWHM) of the Ag $3d_{5/2}$ peak from sputter cleaned silver. This allows a comparison between instruments supplied by different manufacturers. However, on modern instruments, the ultimate energy resolution of the instrument is significantly smaller than the natural line width of the $Ag_{5/2}$ peak from pure silver (0.33 eV). This means that a relatively large improvement in instrument resolution will provide only a small change in the FWHM of the peak.

Assuming that the peak shapes can be described as gaussians, the FWHM of an XPS peak (ΔE) can be calculated using the equation:

$$\Delta E^2 = \Delta E^2_{peak} + \Delta E^2_{instrum} \quad (1)$$

Where ΔE_{peak} is the natural line width of the XPS peak and $\Delta E_{instrum}$ is the instrumental resolution and is affected by the analyzer pass energy, slit widths and the line width of the incident X-rays. The use of a microfocusing monochromator is likely to provide better instrumental resolution than if a large spot monochromator were used.

An alternative method for determining the instrumental resolution is to measure the width of the Fermi edge. In document AN31007, it has been shown that the width of the Fermi edge for silver is 0.26 eV (using the 12% to 88% measurement) when measured using Thermo Scientific ESCALAB 250. The width of the Fermi edge is given by kT (k is Boltzmann constant and T is temperature in Kelvin). At room temperature, kT is approximately 0.025 eV. Using equation (1), this means that the instrument resolution is 0.258 eV.

Using this instrumental resolution and the Ag $3d_{5/2}$ natural line width and applying equation (1) again, the experimental peak width can be calculated to be 0.42 eV. This is exactly the ultimate resolution observed from ESCALAB 250.

It can be shown that a 20% deterioration in the instrumental resolution will only increase the FWHM of the $Ag_{5/2}$ peak to 0.45 eV (a 7% increase). This means that an instrument having a much inferior ultimate energy resolution will only show a small difference in the ultimate FWHM of the Ag $3d_{5/2}$ peak.

To demonstrate the resolution of an XPS spectrometer using an XPS peak, a sample other than silver should be selected.

Choice of Sample

The material must have inherently narrow XPS peaks. To minimize the natural peak width the sample must:

- Be a single crystal
- Be easy to prepare without sputtering
- Form a reproducible surface following sample preparation
- Preferably have a flat surface
- Have only one chemical state present

The surface of the material must be clean but should not be sputtered because sputtering can increase the line width of the XPS peak, due to co-ordination and disorder effects.

In this work, a single crystal of tungsten selenide was chosen and, to get a clean surface, the material was cleaved immediately before the analysis. The cleaving process reproducibly provides a clean flat surface.

The microfocus monochromator was operated at a spot size of 500 µm and the analyzer with a pass energy of 2 eV. Under these conditions, the spectrum shown in Figure 1 was obtained.

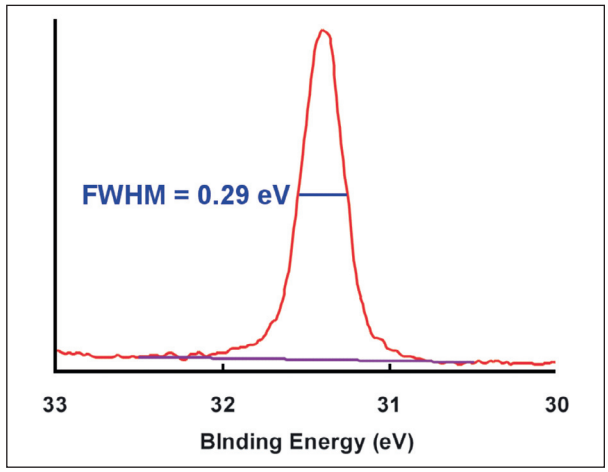

Figure 1: W $4f_{7/2}$ region of the XPS spectrum from WSe_2 showing a FWHM of 0.29 eV

Under these conditions, the W $4f_{7/2}$ FWHM was shown to be 0.29 eV, from which the natural line width of the peak can be calculated to be 0.13 eV.

To date, a FWHM figure of 0.29 eV has not been matched or surpassed by any other, current commercial XPS instrument.

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