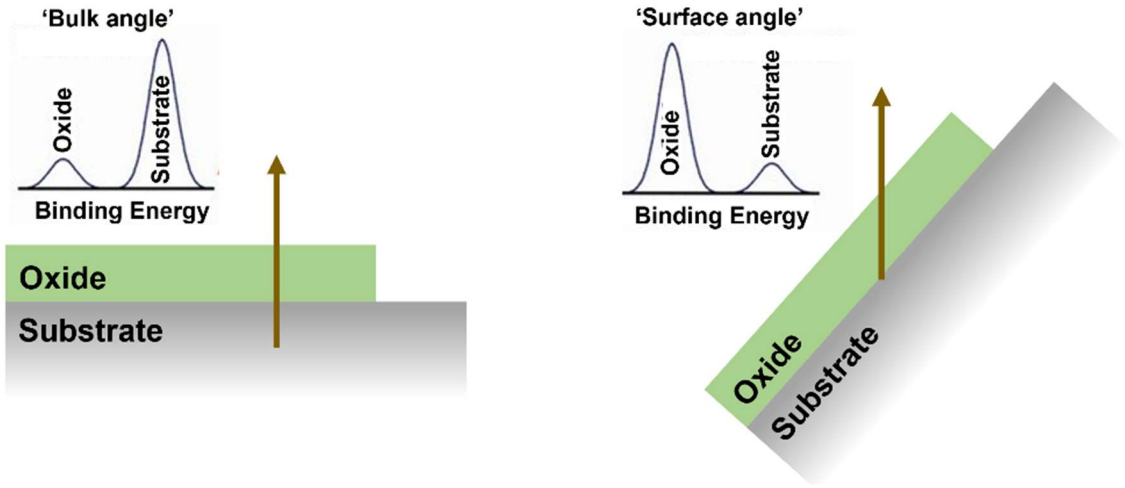
XPS2 Angle Resolved XPS (ARXPS) Operating Procedure

Angle Resolved XPS (ARXPS) is a technique that varies the emission angle at which electrons are collected, in order to alter the probing depth within a sample and generate information on the thickness and composition of thin films. This is achieved by tilting the sample with respect to the analyzer's position. The image below is a schematic representation of ARXPS for a thin oxide on a substrate, whereby increasing the tilt angle results in increasing the relative amounts of oxide signal in the spectrum.



Help Resources

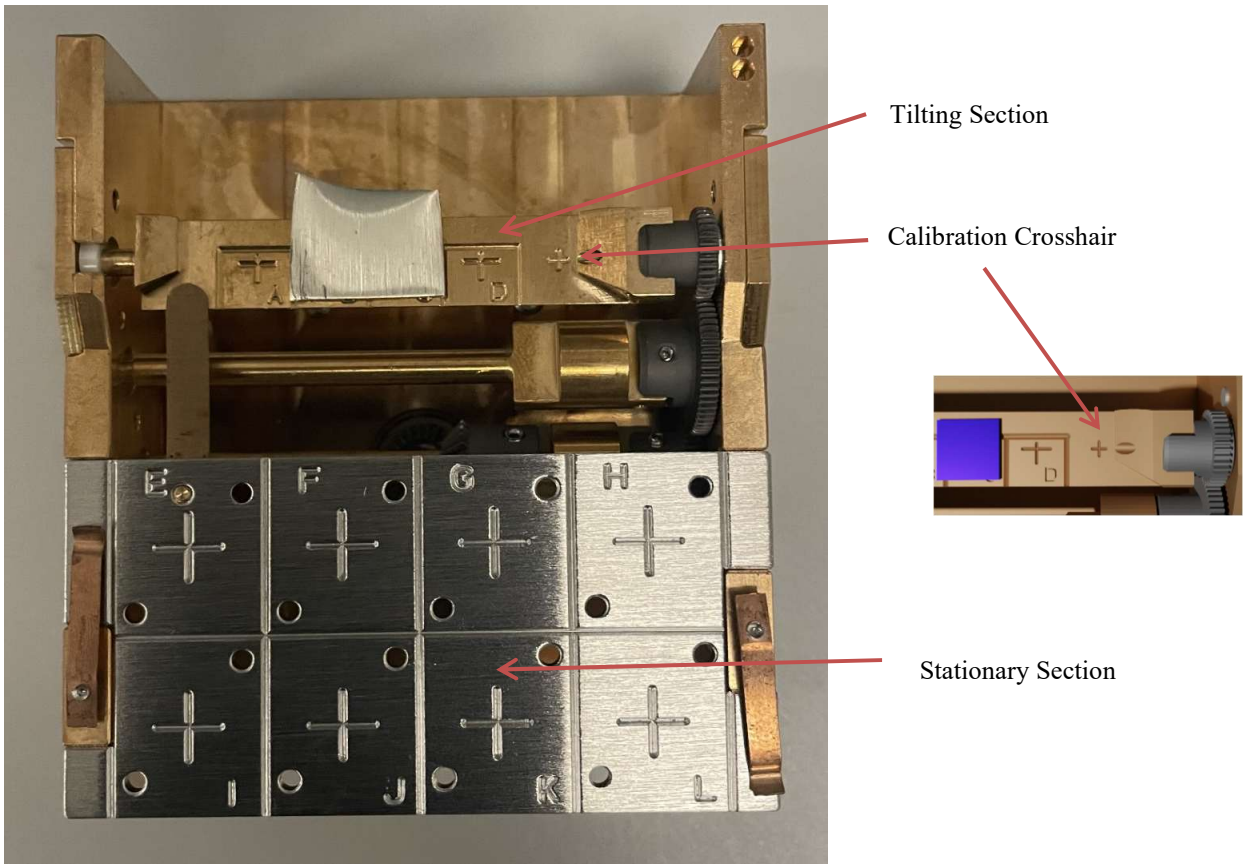
XPS knowledge viewer – XPS Experimentation (Page 2) – Angle Resolved XPS

Nexsa G2 User Manual (Page 122) and Processing Manual (Page 203) provided by Thermo Scientific is located in the Manuals folder on the desktop

Sample Loading

You must use the Sample Tilt Holder for ARXPS Measurements.

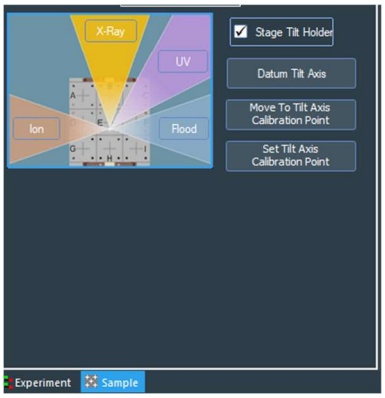
- This sample tilt holder has two sections for sample mounting: the tilting section for ARXPS and a stationary section for other samples
- Always adhere samples to the tilting section using double side copper or carbon tape, and ensure the sample is securely held in place.
- Ensure the sample does not overhang to the top or bottom of the tilt arm
- Ensure the sample is not mounted over the crosshair at the far-right side of the tilt arm, which is used to calibrate the tilt axis



Procedure

Tilt Axis Calibration

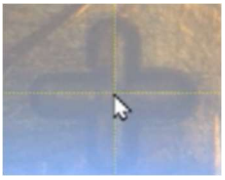
This ensures that the tilt state 0 position is horizontal and the correct center of rotation is defined



1. Select the Stage Tilt Holder box in the Sample Tab
2. After sample transfer, move the sample position close to the tilt axis calibration point (the cross on the right hand side of the tilt arm) and get the surface in optical focus using the Z controls
3. With the top down light on full, use the tilt controls in the bottom right corner of the optical view to find the angle at which the flat surface appears brightest

4. Click “Datum Tilt Axis” in the Sample tab. Click OK on the message box that appears.
5. To confirm the new 0 degrees position is horizontal, move to the top edge of the sample and use the Z controls to get it into optical focus. The move to the bottom edge of the sample and confirm it remains in optical focus.

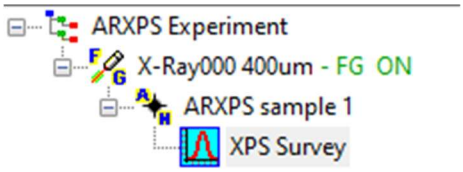
6. Click “Move to Tilt Axis Calibration Point” in the Sample tab and ensure the top surface of the plate at the edge of the cross is in optical focus using the Z controls.
7. Move the optical cross hair position to the center of the engraved cross.



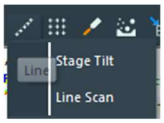
8. Click “Set Tilt Axis Calibration Point” in the Sample tab, then click OK in the message box that appears.

Data Collection

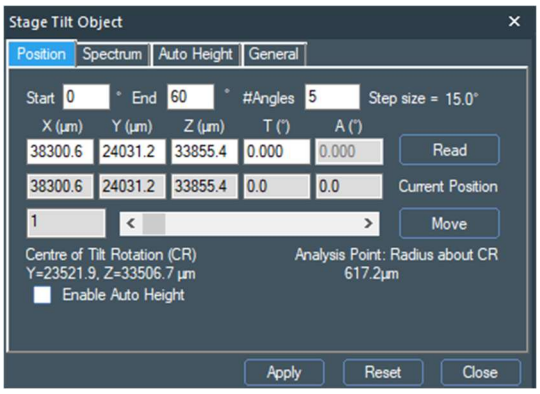
1. Navigate to the sample position you want to measure and focus the optical image using the Z controls
2. Select the Experiment tab and create the measurement program
3. Add an X-Ray source and Point object and collect a Survey Spectrum with Auto-Height enabled

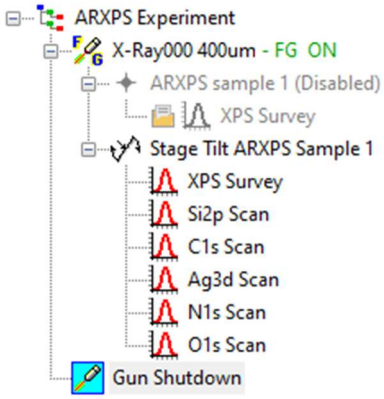


4. Disable the survey spectrum point and add a Stage Tilt Object from the Line Scan menu
    1. Input the Start angle, end Angle, and Number of Angles. A typical experiment would be 0-60 degrees with 5 to 10 angles.



2. Add survey and region scan spectra based on analysis of the previously acquired survey spectra





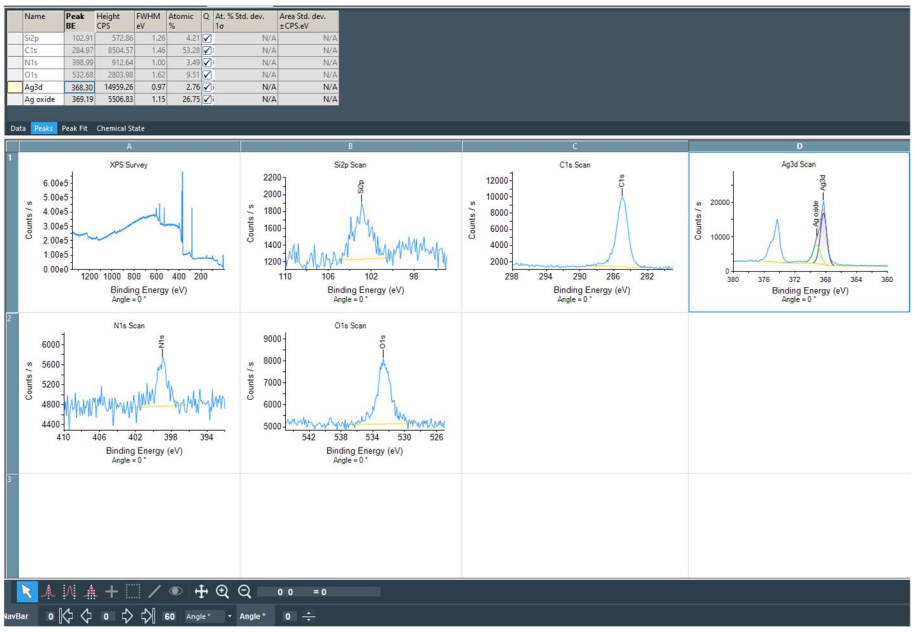
5. Run the experiment

Observe the data in the processing grid. The count rate should be steadily decreasing with increasing tilt angle. If the count rate is erratic, increasing, or not changing, then repeat the Tilt Module Calibration steps.

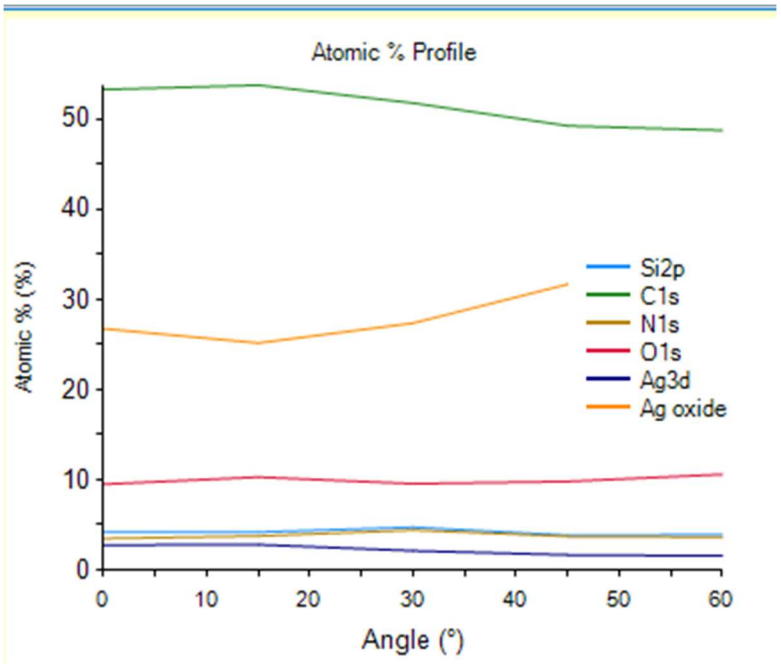
Data Processing

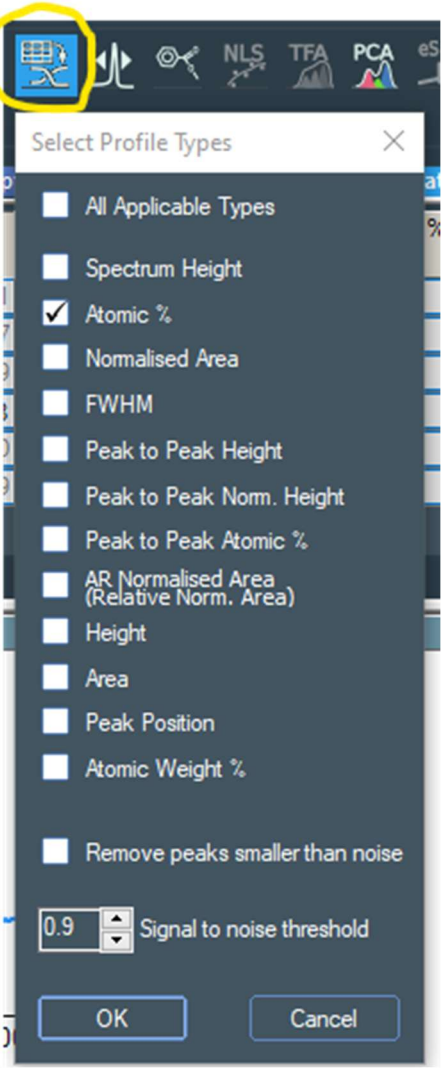
1. Generate Peaks Table

1. For peaks where elemental information only is required, use Peak Add (manual)
2. For peaks where chemical information is required (or for doublet peaks), use Peak Fitting



3. Add a Peak Table profile using Atomic % to give an initial indication of species distribution in the layer

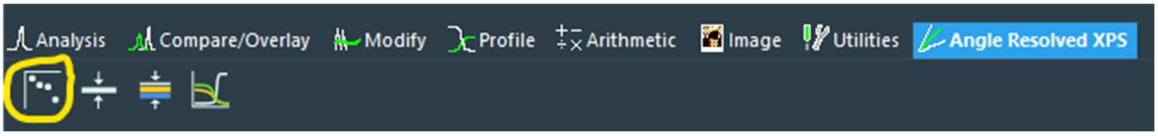




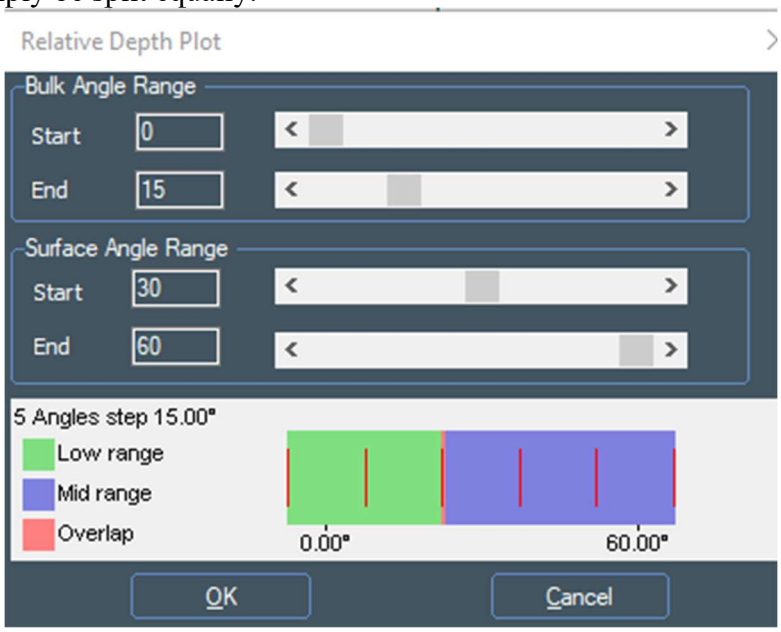
2. **Relative Depth Plot**

The Relative Depth plot tool gives a quick and simple qualitative indication of the relative depth of each species in the ARXPS data

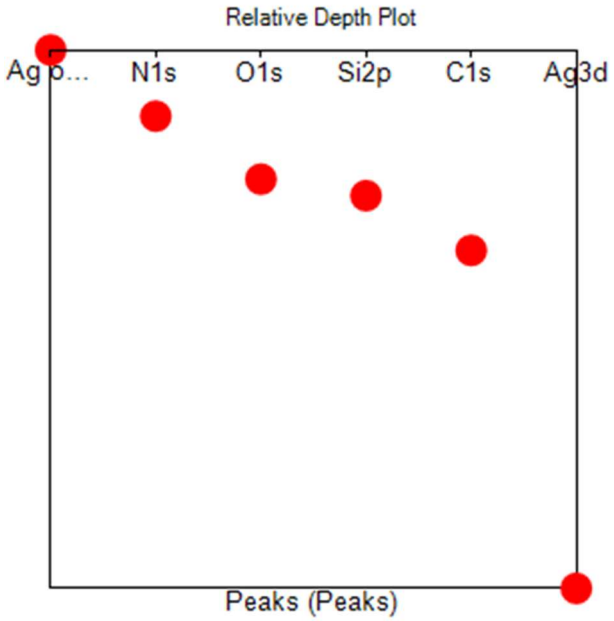
1. Make sure all species of interest are selected in the Peaks table
2. In the Angle Resolved XPS tab, click on the Relative Depth Plot icon



3. Default bulk and surface angle ranges are set by the software. These can be adjusted using the sliders. It is generally better to avoid having any overlapping regions. For most scenarios the surface and bulk angles can simply be split equally.



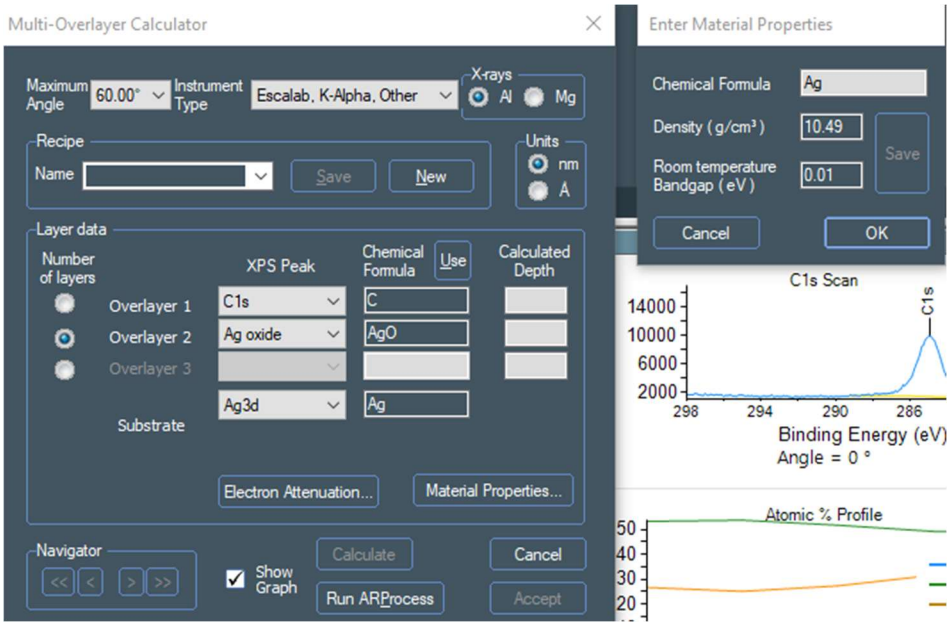
4. Click OK and a qualitative relative depth plot will be created.



Here the plot shows N, O, Si, and C contamination closer to the surface and the Ag elemental (substrate) at the bottom.

3. **Multiple Overlayer Thickness Calculator**
    1. In the Angle Resolved XPS tab, click on the Multiple Overlayer thickness calculator icon (even if the sample has only one overlayer)

    2. Select up to 3 overlayers and enter Chemical Formula and XPS Peak information for each layer (case sensitive)
        1. There is a small library of common materials for which the Band Gap and Density information is stored in the software
        2. If your material does not appear on the list, click "Material" properties and enter the relevant information. For metallic materials, enter 0.01 eV for the band gap. Click Save to add as a new material in the library.
    3. Enter chemical formula and XPS peak information for the substrate
    4. Select the maximum angle from the drop down. *Caution – for angles above 60 degrees the effects of elastic scattering can result in poorer fits to the data and less reliable results.*



    5. Click "Calculate" to show the thickness for each layer