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Avantage Datasystem *version 5*

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Chinese Users Meeting 2014

Contents

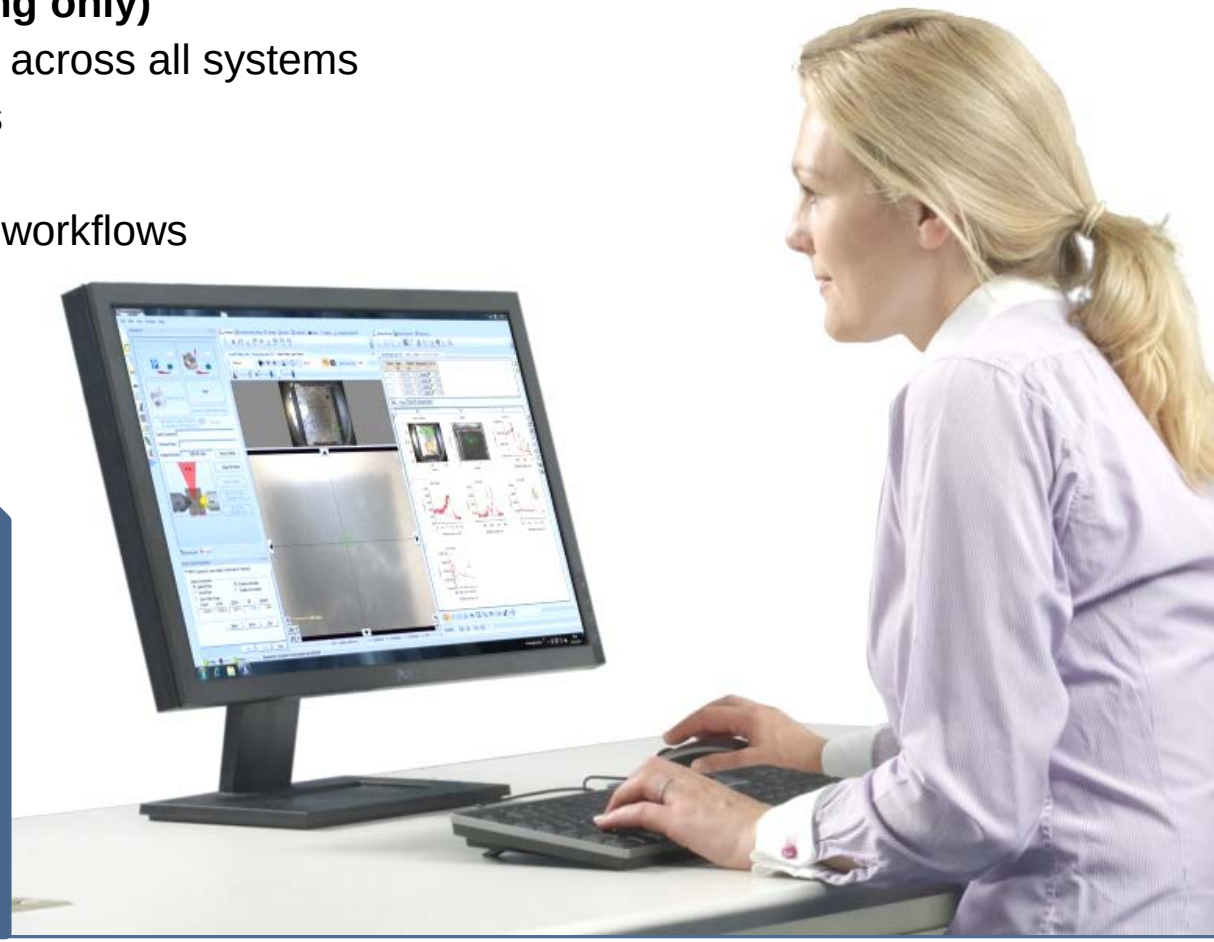
- Introduction
- Avantage version 5
  - Interface
  - Features and Functions
- Data processing examples
  - Multilevel data sets
  - XPS Images
  - Depth profiles
  - ARXPS
- Language Support

The Avantage datasystem

- A modern Windows application
  - Covers entire Thermo Scientific XPS product range
  - ***NEW!*** **Version 5:**
    Windows 7 (acquisition & processing)
    Windows XP (processing only)
  - Familiar and consistent interface across all systems
  - Designed for multi-level datasets
  - Multi-technique support
  - Advanced Sample – to – Report workflows
  - Automated calibration tools

AVANTAGE **:**

- **Control**
- **Acquire**
- **Process**
- **Report**



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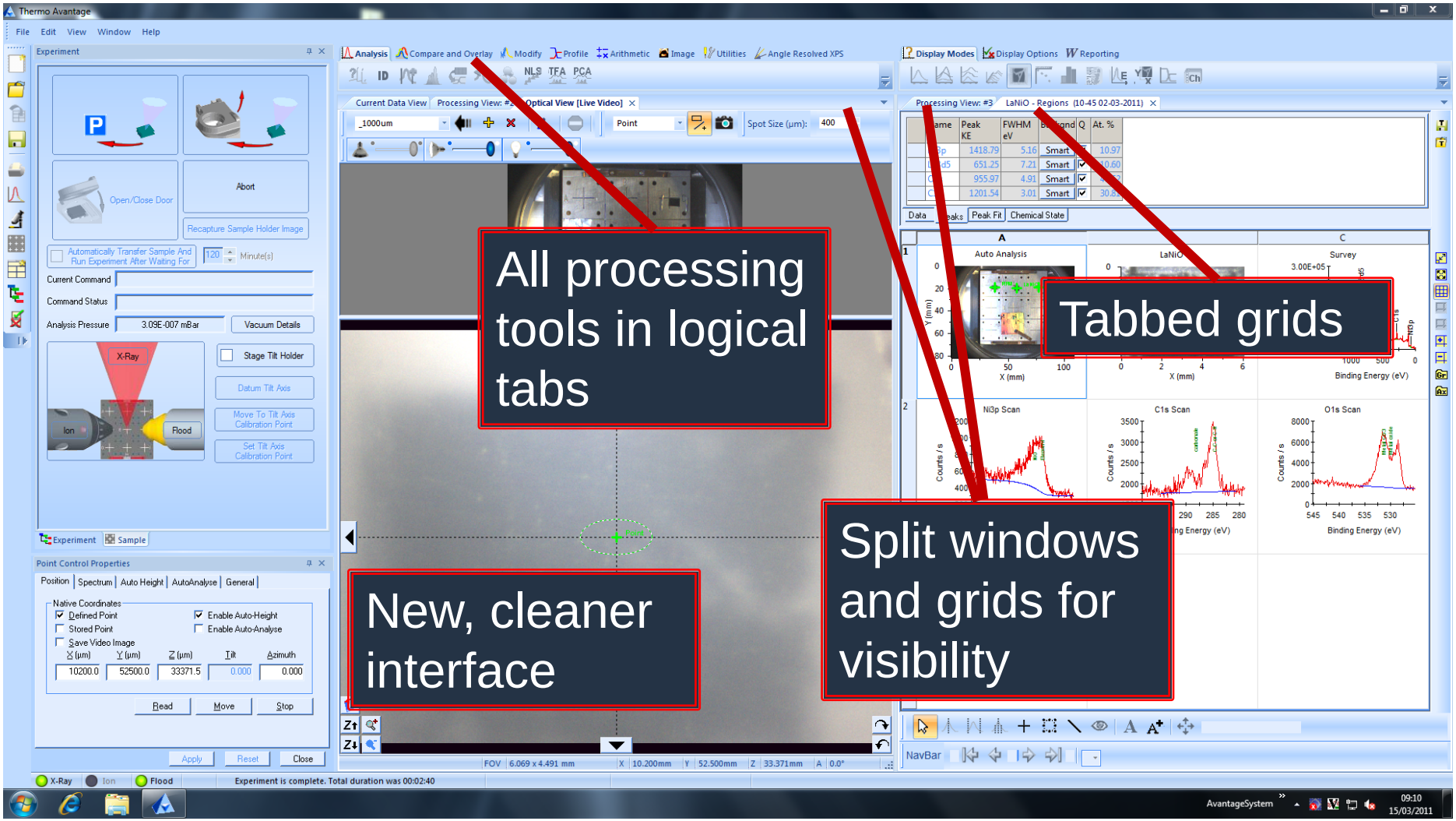
Avantage v5

Avantage v5 – Powerful Features, Easier to Find



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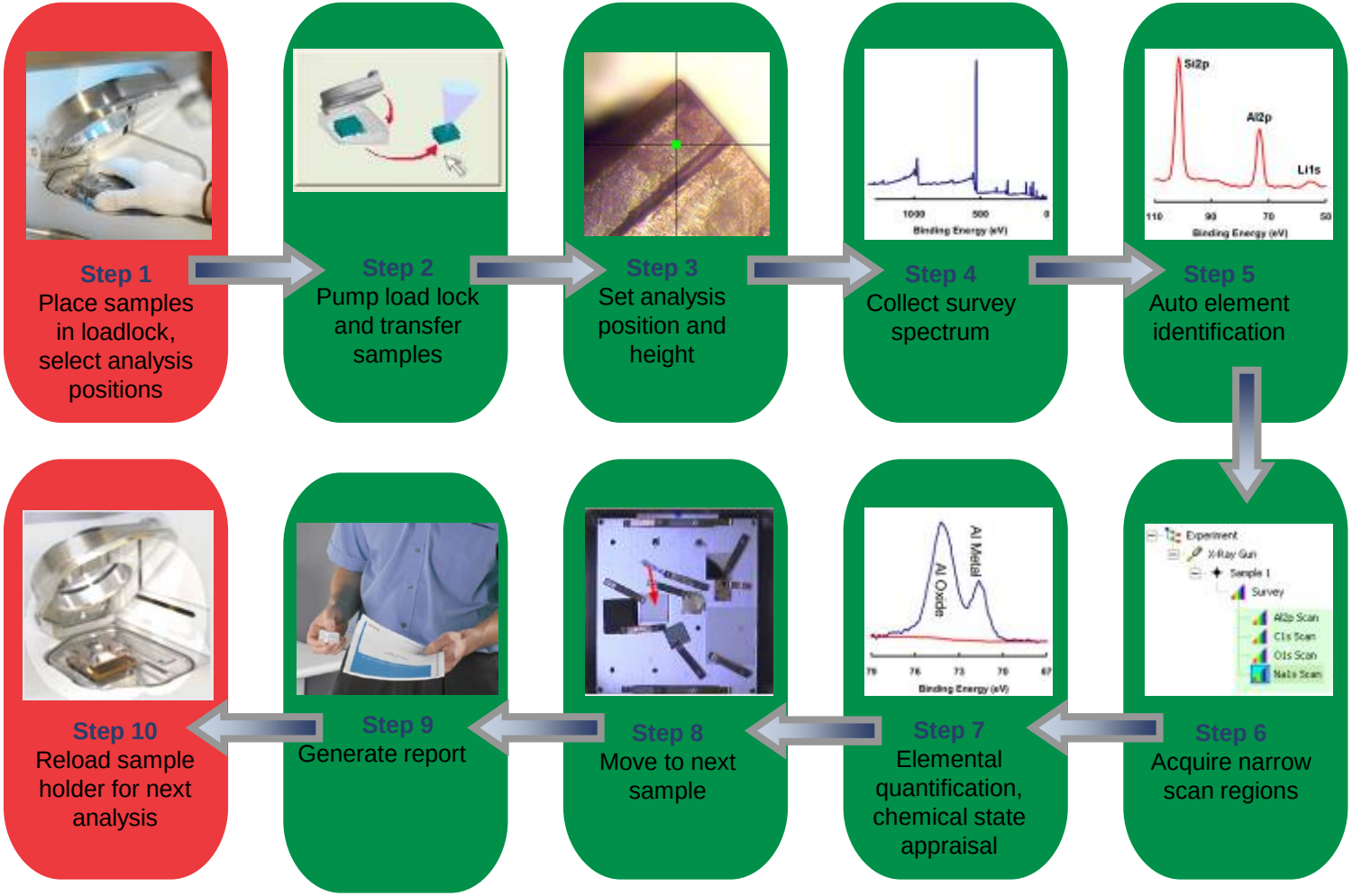
Auto analysis

- Generation of 1st pass reports
  - Based on auto ID of survey spectrum
  - Automated report compilation
    - **Survey & narrow scan data**
    - **Experimental acquisition parameters**
    - **User comments**
    - **CCD image and analysis position information**
    - **Peak ID and quantification table**
    - **Chemical state appraisal table**
- ***Now available for E250Xi & ThetaProbe***

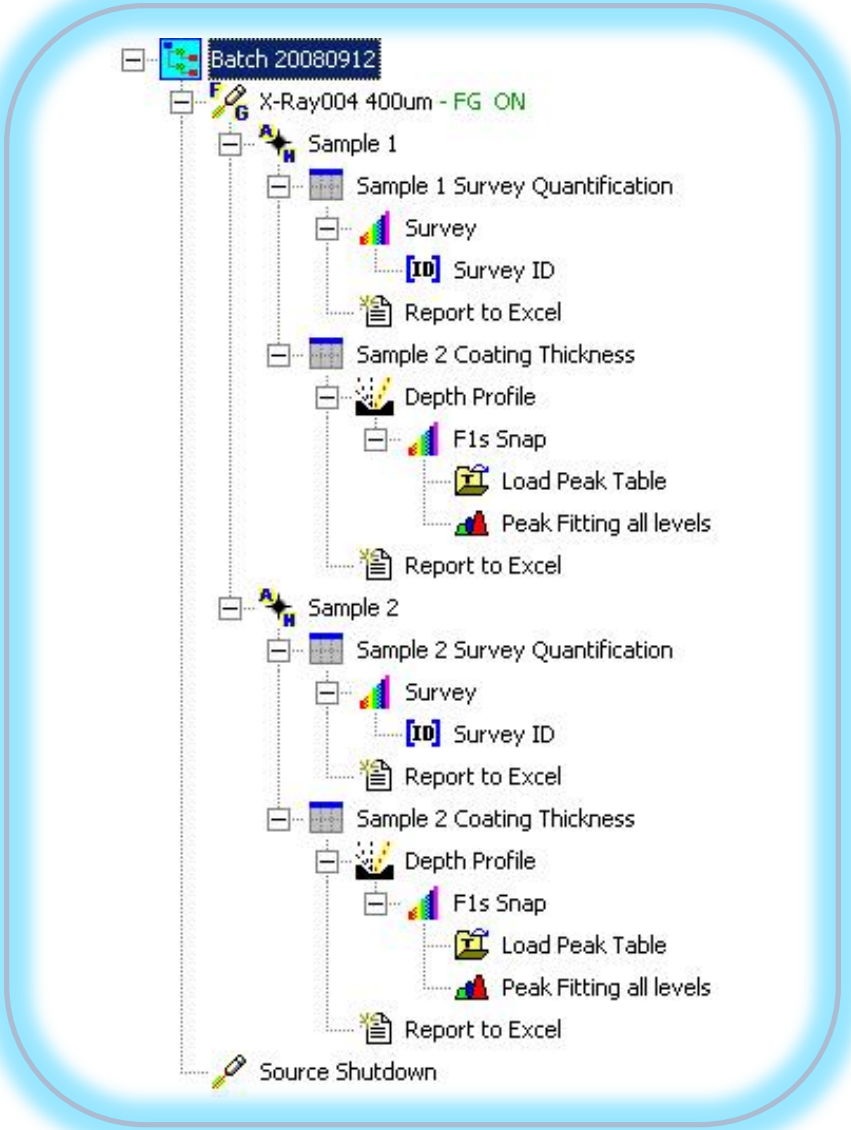
Auto Analysis



Full recipe generation

Experiment Setup

- Advanced experiment object
  - Acquisition
    - Predefined acquisition of
      - Points
      - Linescans
      - Iterations
      - Mapping
      - Depth Profiles
  - Processing
    - Full processing options
      - Peak ID, Add and Quantification
      - Peak Synthesis
      - Multi- sample point & level batch processing
      - Re-use tried and tested parameters
  - Reporting
    - Export to 3rd party software
      - Word, Powerpoint, Excel etc.



Full recipe mode of operation

The user

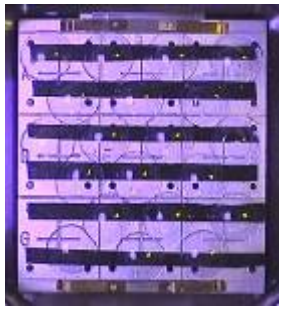
Loads samples into pre-defined positions

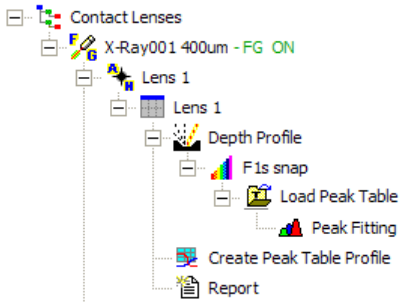
Selects recipe

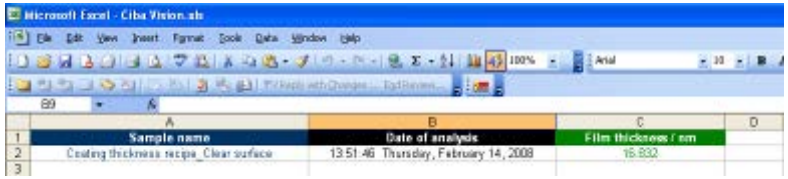
K-Alpha

Automatically acquires coating XPS data

Exports composition and depth information







Recipe output in Excel

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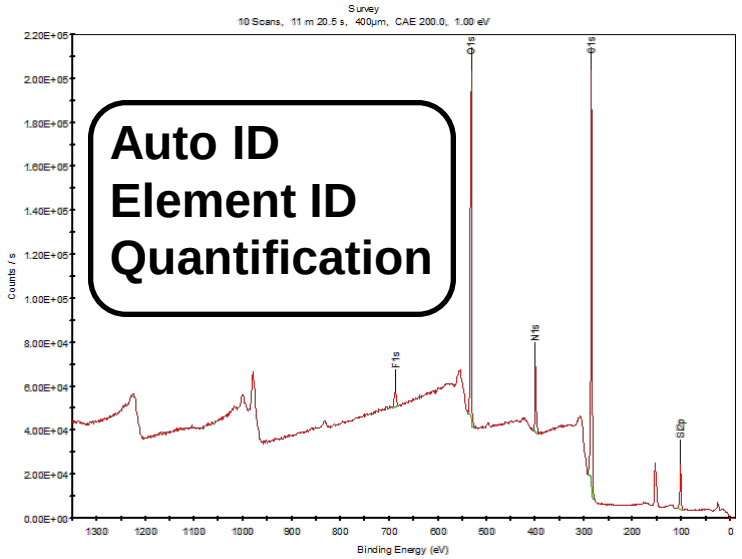
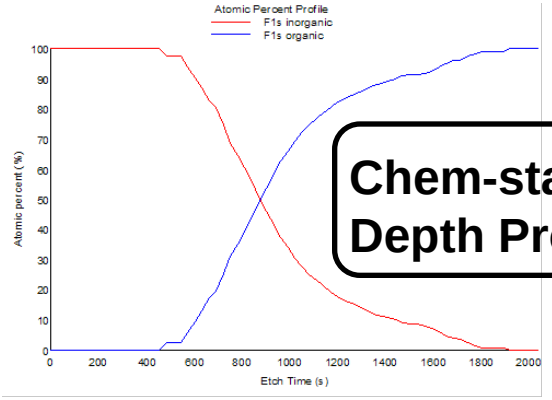
Full recipe mode of operation

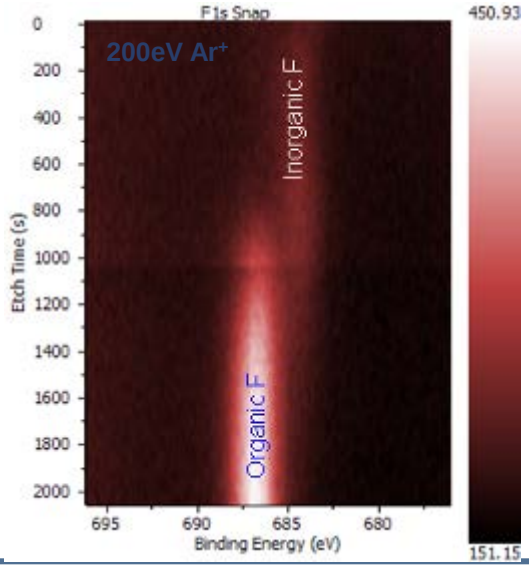


Auto ID
Element ID
Quantification

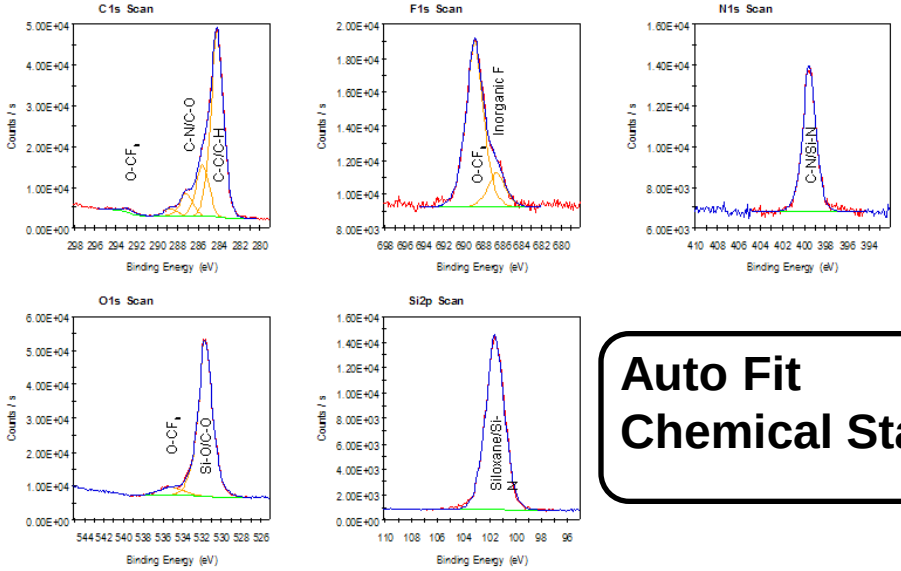
Recipe calculates the coating thickness From DP



Chem-state
Depth Profile



Rapid
Snapshot
DP



Auto Fit
Chemical State

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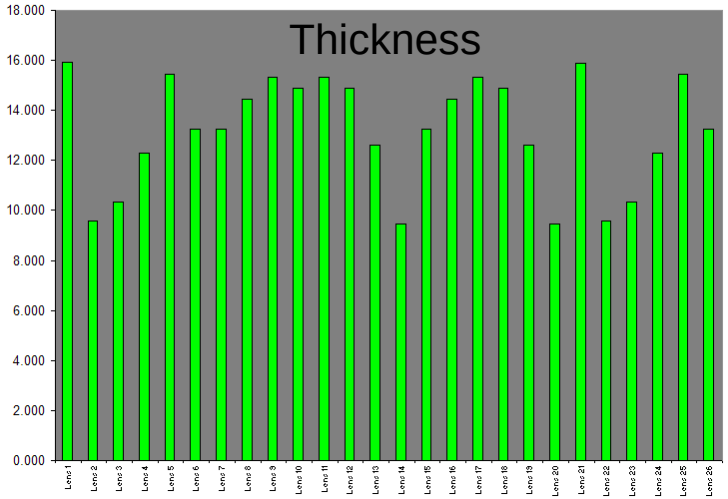
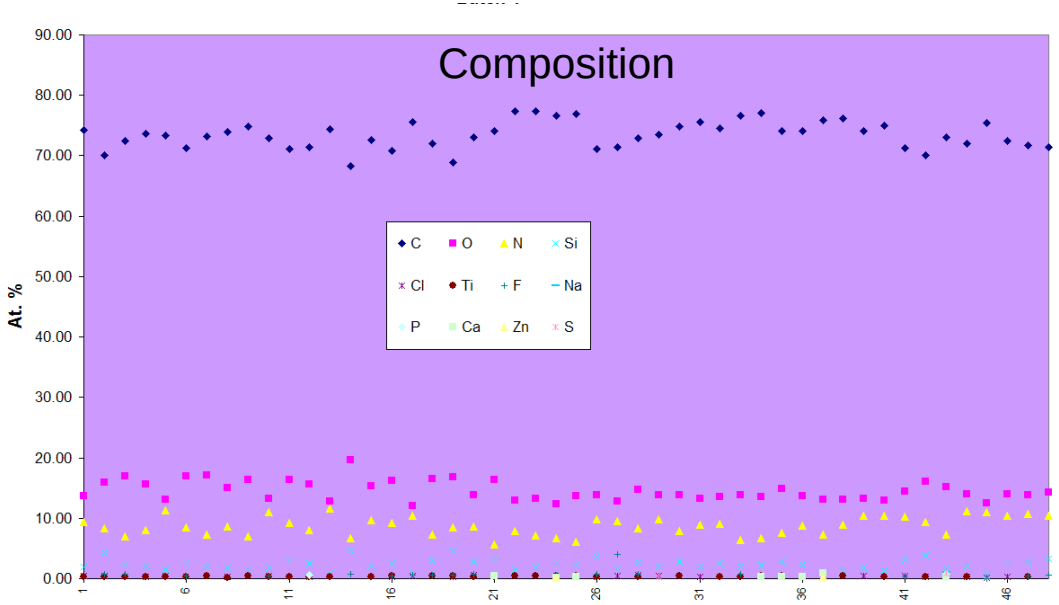
Full recipe mode of operation

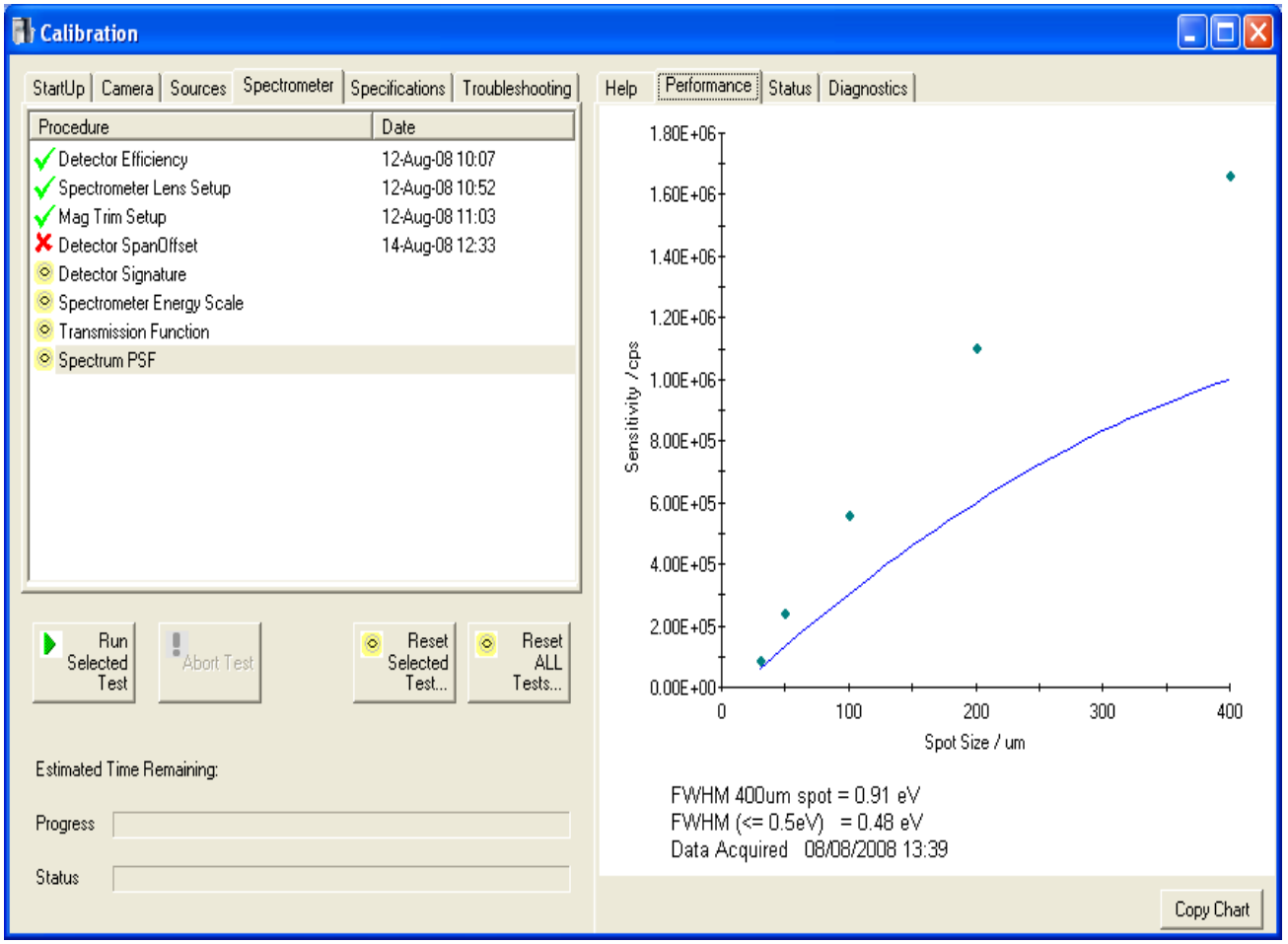
- Output format
  - Excel, word etc..
  - Export to predefined template documents
  - Additional processing from via excel macros





Automated Instrument Calibration

- Avantage
  - supports automated instrument calibration
- Performance confidence
  - completely calibrated and ready for use
- Ease of use
  - Sequential steps ensures calibrations are completed in correct order
- Traceability
  - Full calibration and performance history available
  - Date and status of last calibration recorded



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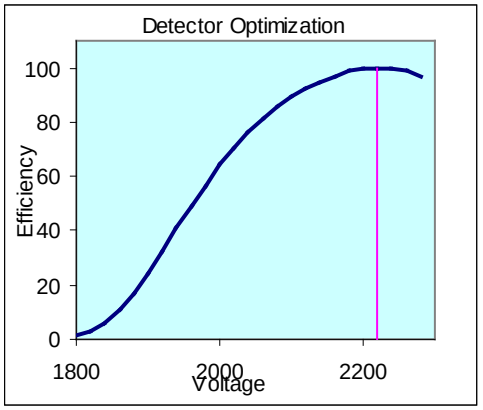
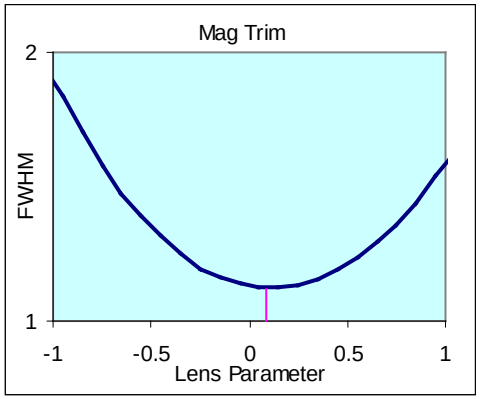
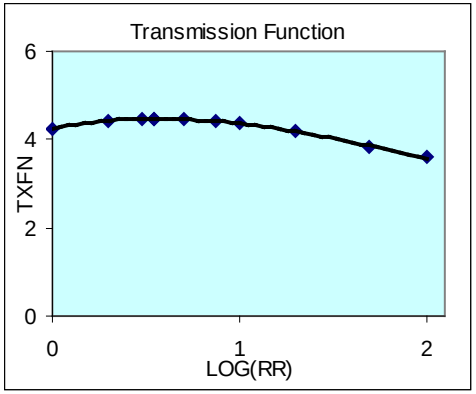
Automated Instrument Calibration

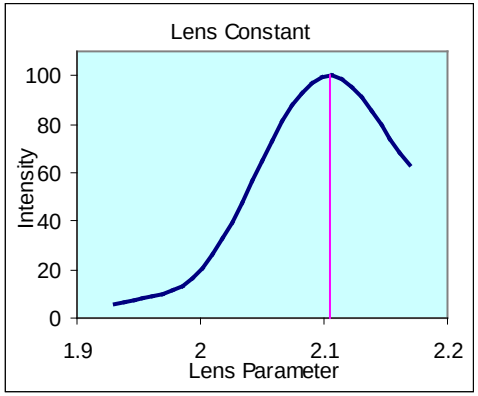
Detector, Lens, Analyser optimisation

- Detector
  - Optimum operating voltage
  - Efficiency signaturing









- Spectrometer
  - Lens constants for optimized performance
  - Energy Scale
  - Transmission function
  - Energy deconvolution parameters

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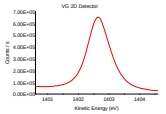
Automated Instrument Calibration

Energy scale calibration

Move to Au ref

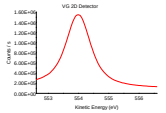
Sputter 20s



Acquire Au4f 7/2 spectrum

Move to Cu ref

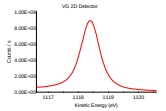
Sputter 20s



Acquire Cu2p 3/2 spectrum

Move to Ag ref

Sputter 20s



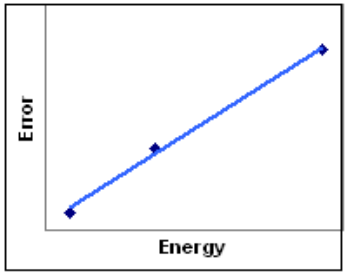
Acquire Ag3d 5/2 spectrum

- Calibration standard reference materials kept under vacuum in K-Alpha, and on standard block for 250Xi
- Single click for entire energy scale calibration
- Rapid, completed < 10 mins
- Traceability calibration history log recorded

Determine peak energies

Calculate error from reference values

Adjust electronics energy scale calibration



Repeat process to confirm within tolerance

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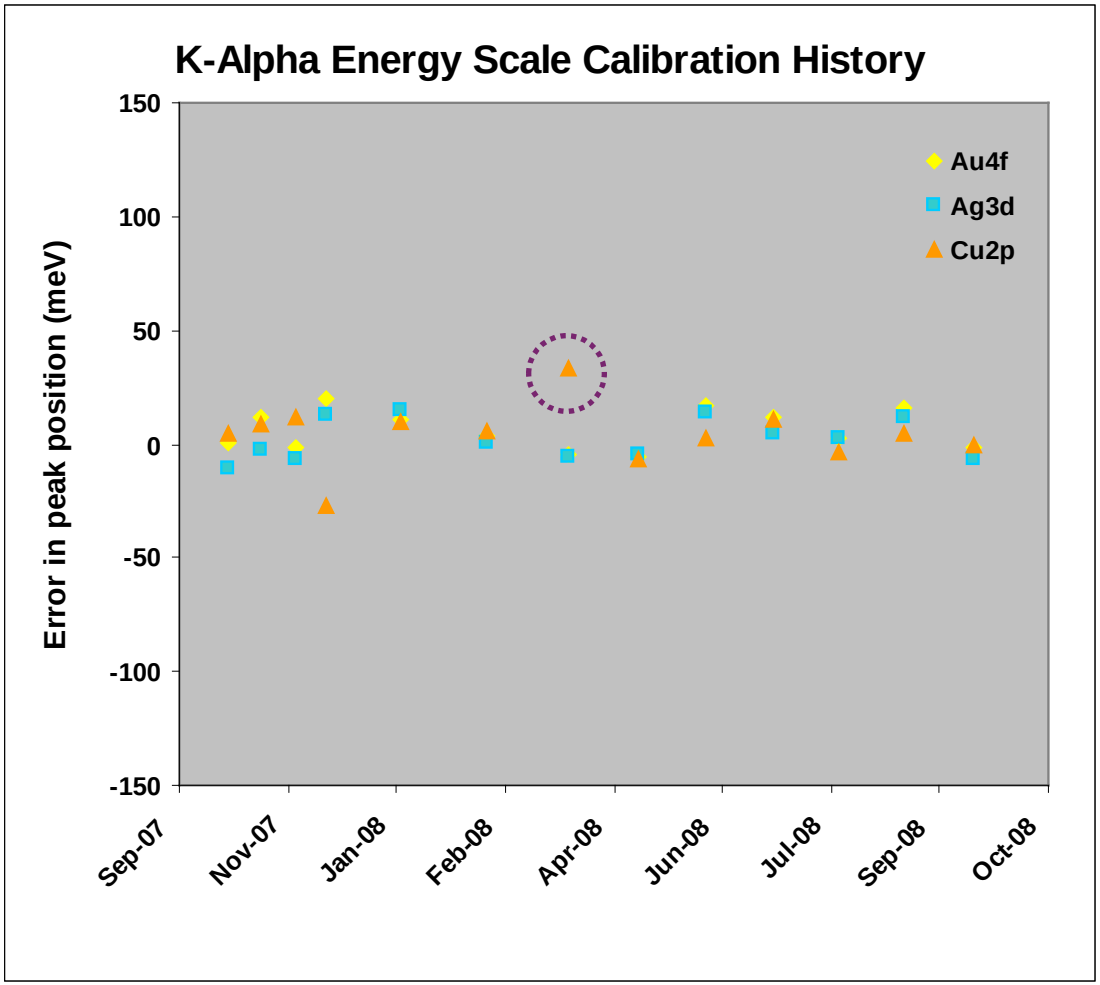
Automated Instrument Calibration

Energy scale calibration history

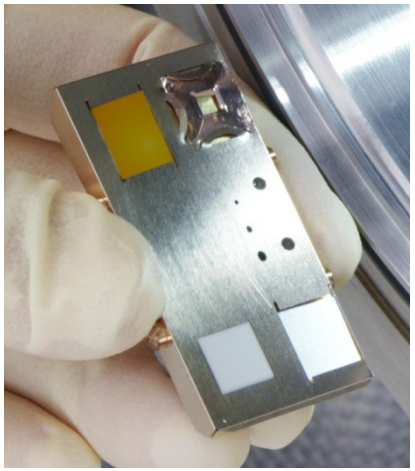


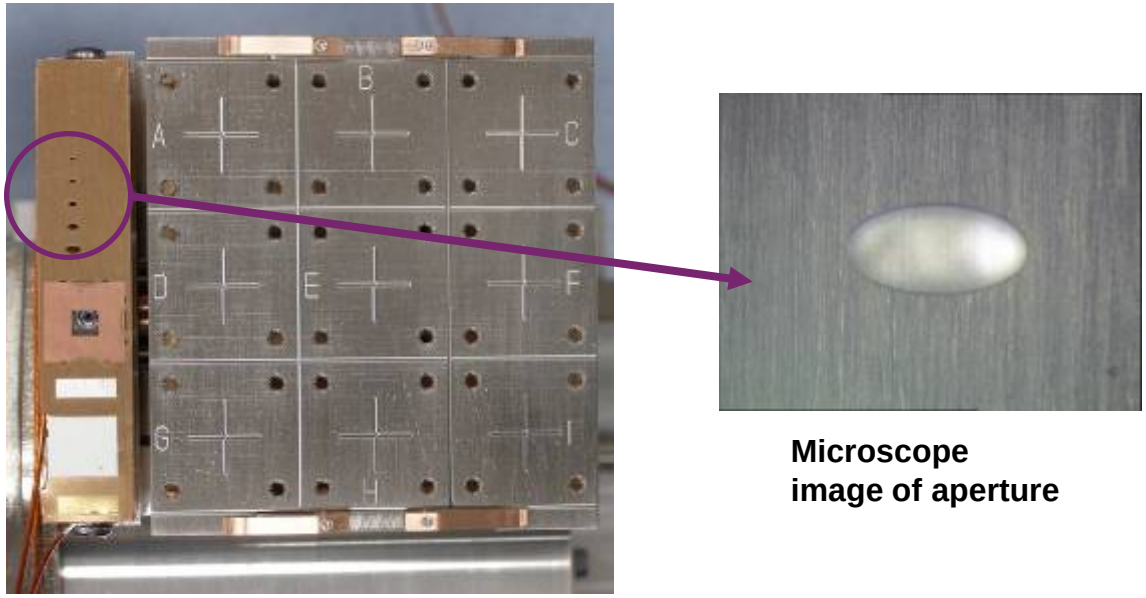
- Maximum error in 9 months 33meV
- Full calibration record available on each tool

Automated Instrument Calibration

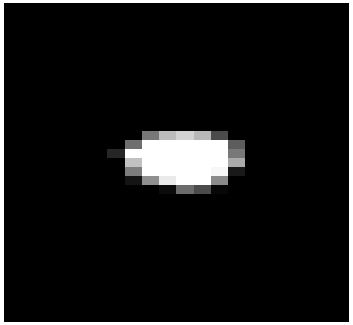
Ion gun setup

- Ion gun auto set-up
  - Optimum and calibrated beam current and focus
  - Optimizes all modes 100-3keV
  - Auto-detect centre of current map
  - Excellent Alignment with XPS analysis position for depth profiling
  - Standard size apertures permanently mounted on stage in vacuum
  - Excellent tool matching repeatability
  - Consistent sputter rates





Microscope image of aperture



Sample current map through aperture

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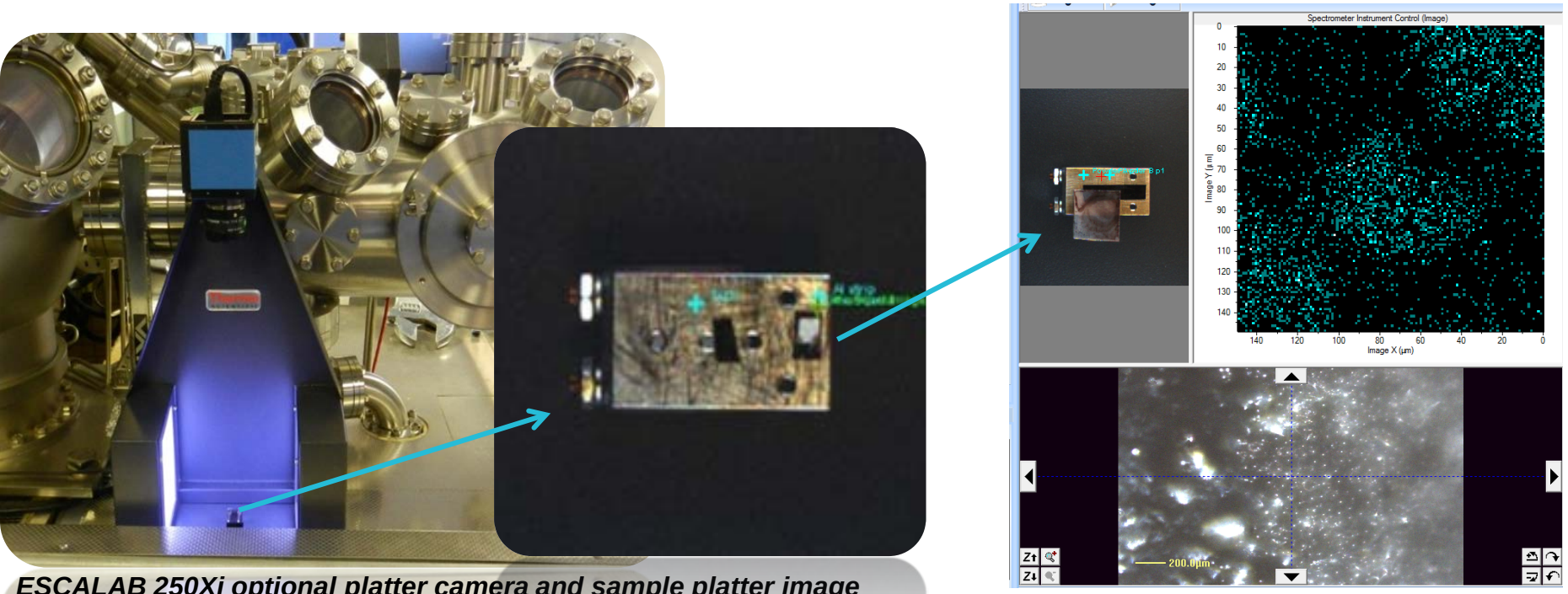
- E250Xi platter camera (option)
  - Sample platter camera option on E250Xi allows users to locate samples/features quickly and with confidence. Improves efficiency and ease-of-use
    - Photographic record of sample analysis
    - Captures an image of sample block prior to loading into the system
    - Calibrated sample image can be used to navigate from sample to sample, with simple mouse clicks



ESCALAB 250Xi optional platter camera and sample platter image

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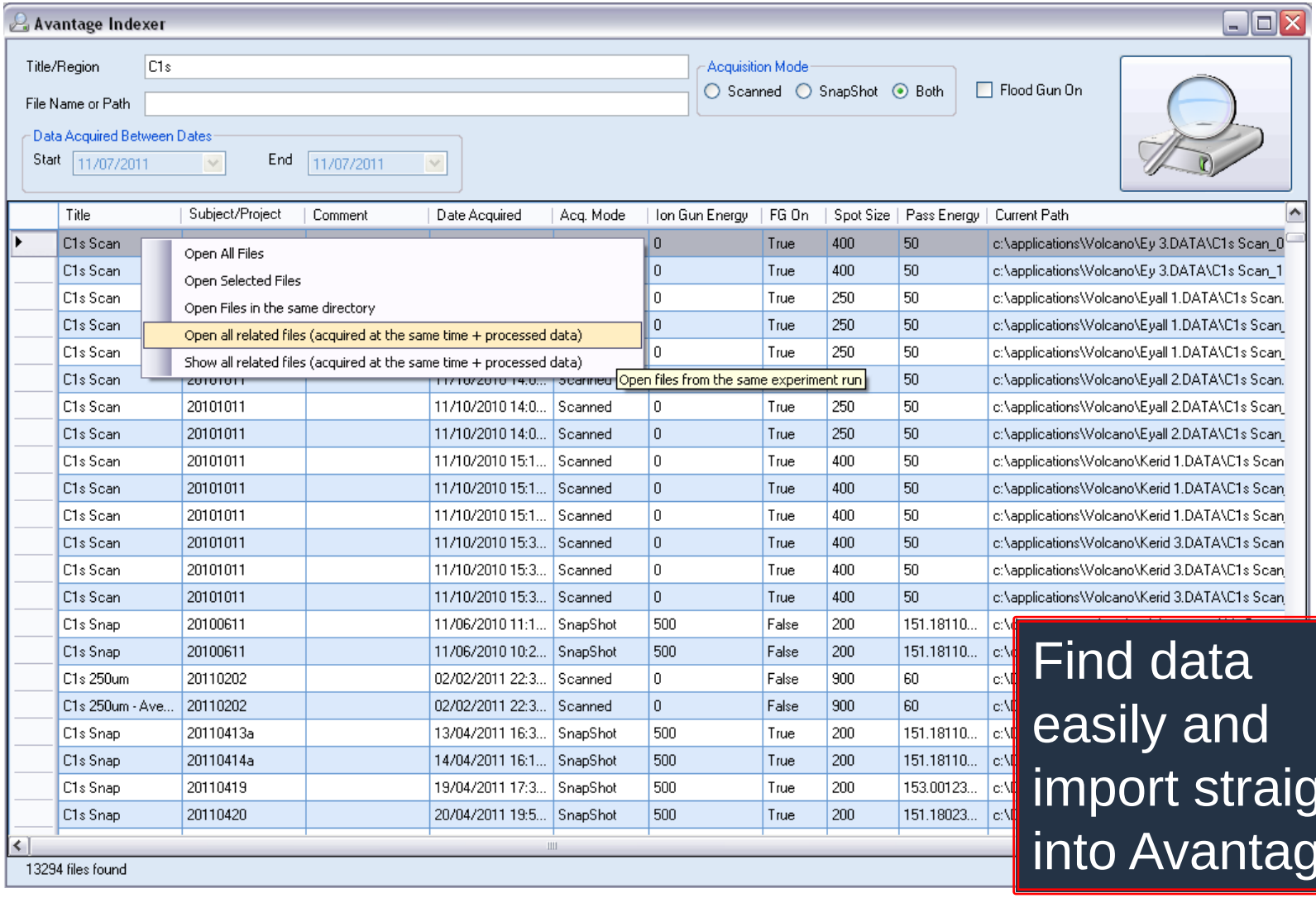
Remote Access

- *Another room…*
- *Another city…*
- *Another country…*

- Acquisition
- Progress monitoring
- Data processing
- Applications support
- System optimisation / calibration
- Online collaboration
- System support

Avantage Indexer



Find data easily and import straight into Avantage

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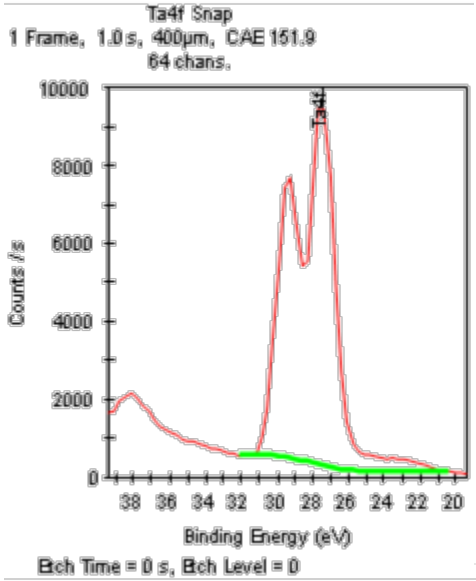
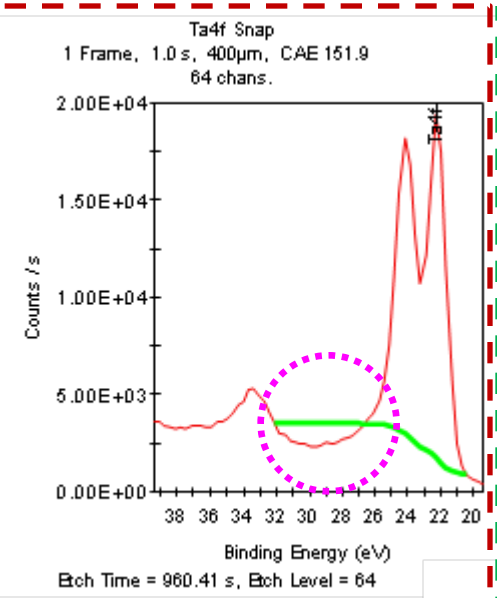
Data processing functions

Backgrounds & Spectral Improvements

Smart backgrounds

Ta4f Depth Profile Data



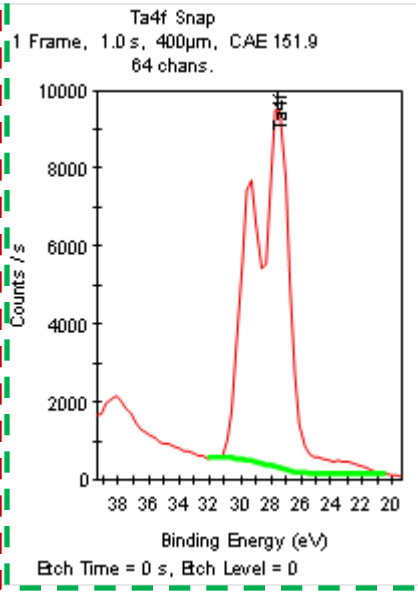


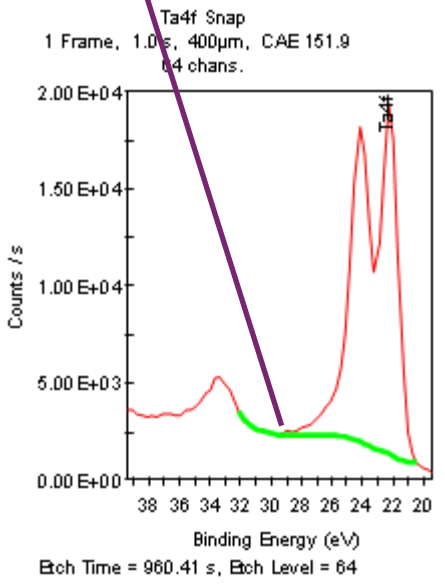
Shirley background

- We want to apply a single Bkgnd range
- Shirley background must end horizontally
- Peak moves during depth profile
- Interference from another peak
- Negative peak area contribution – inaccurate quantification

- We can apply a single Bkgnd range!
- Peak area is consistent – accurate quantification
- Line follows spectrum background
- Resistant to interference
- Ideal for depth profiles etc

Smart background





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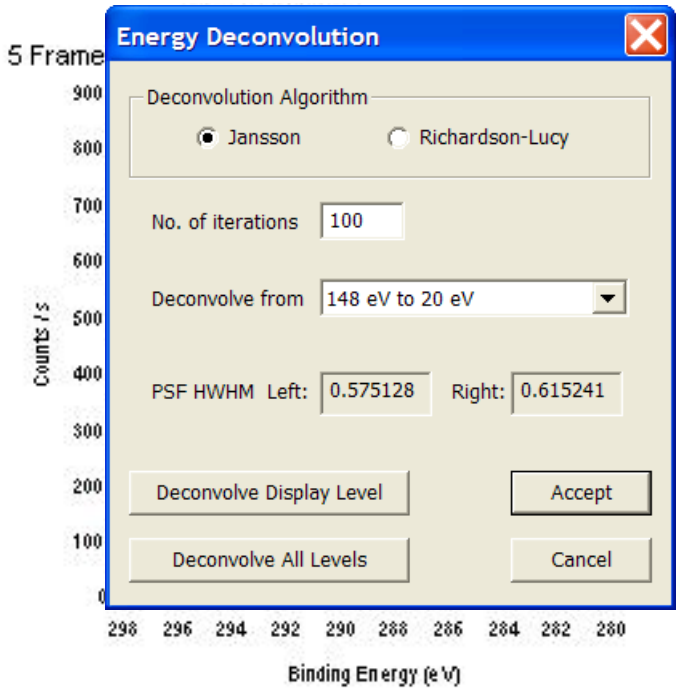
Spectral Deconvolution

- Spectral Deconvolution
  - Removal of analyser contribution
  - Jansson's method
  - PSF calculated from Ag standards
- Benefits
  - Reduced acquisition times
  - Reduced sample degradation
  - Improved signal to noise
  - Improved chemical state determination



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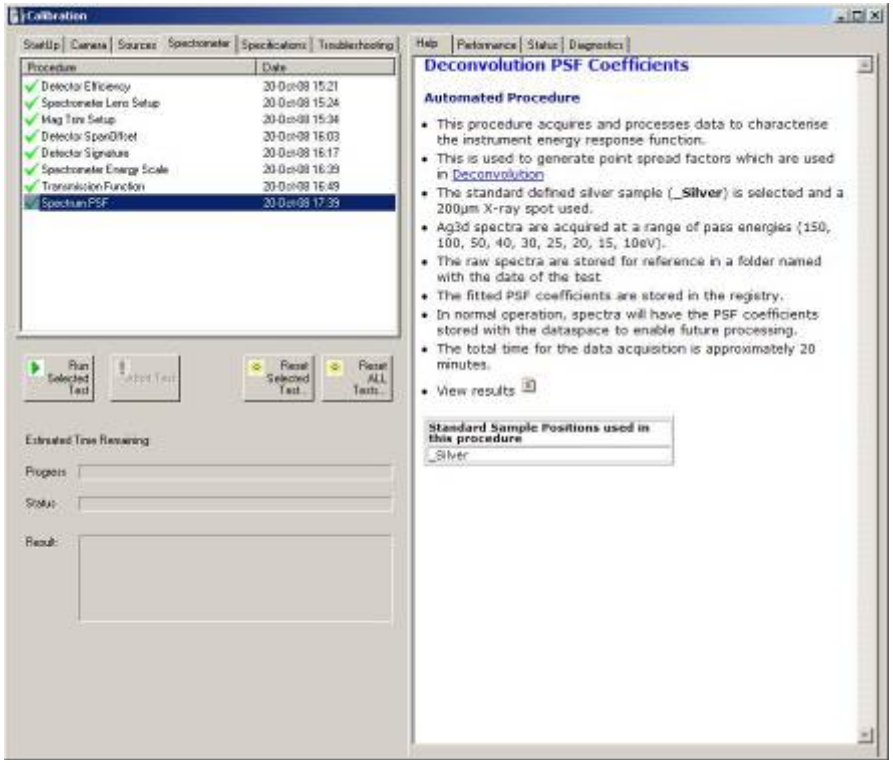
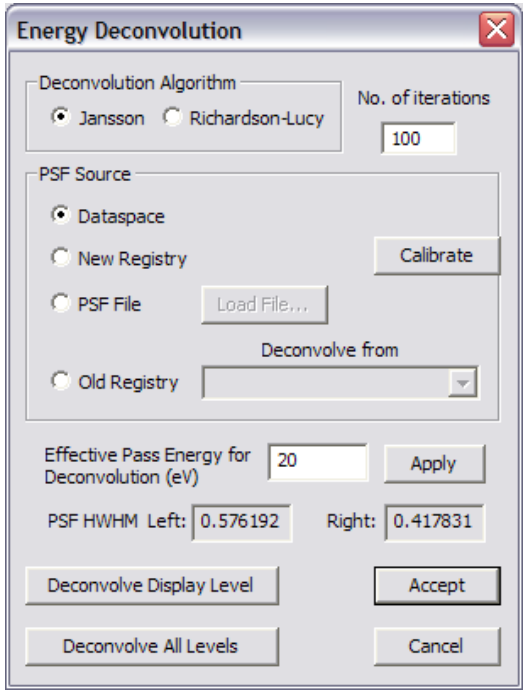
Spectrum deconvolution

PSF acquisition

- PSFs for spectral deconvolution are calculated from spectra acquired from a standard (e.g. Ag3d) at a range of pass energies.
- These are used as the basis of an equation describing the response at any given pass energy
- These can then be applied to scanned or snapshot data to improve energy resolution.





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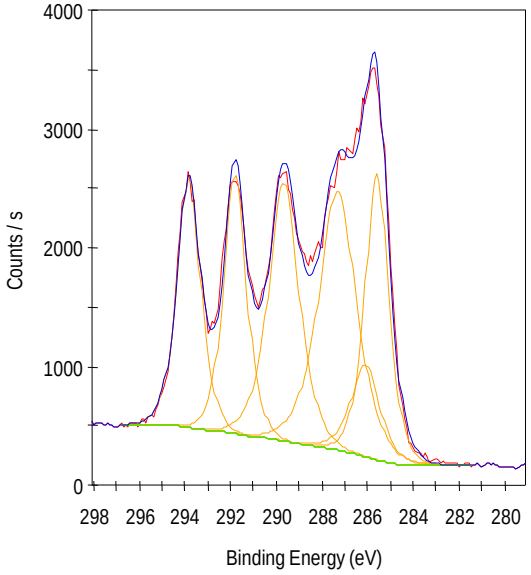
Spectrum deconvolution

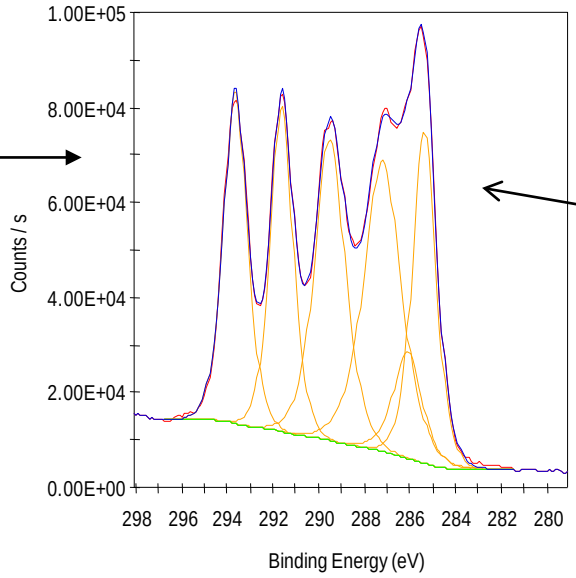
Fluoro-polymer data

- Aim to reduce total acquisition time
- Acquire snapshot data at high PE
- Convert high PE data to simulate low PE data
- Factor of 10 improvement in acquisition time
- Ideal for mapping, depth profiling and delicate samples

Hi-res acquired



Deconvoluted



Deconvolution

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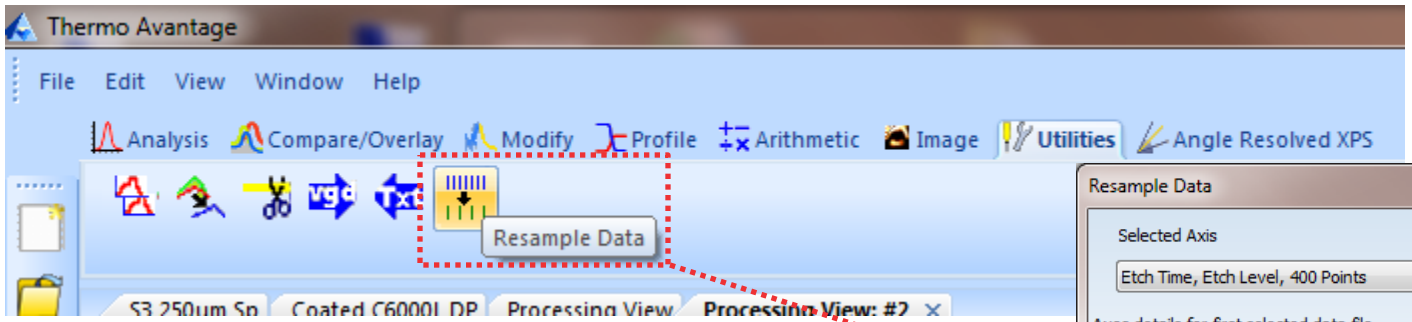
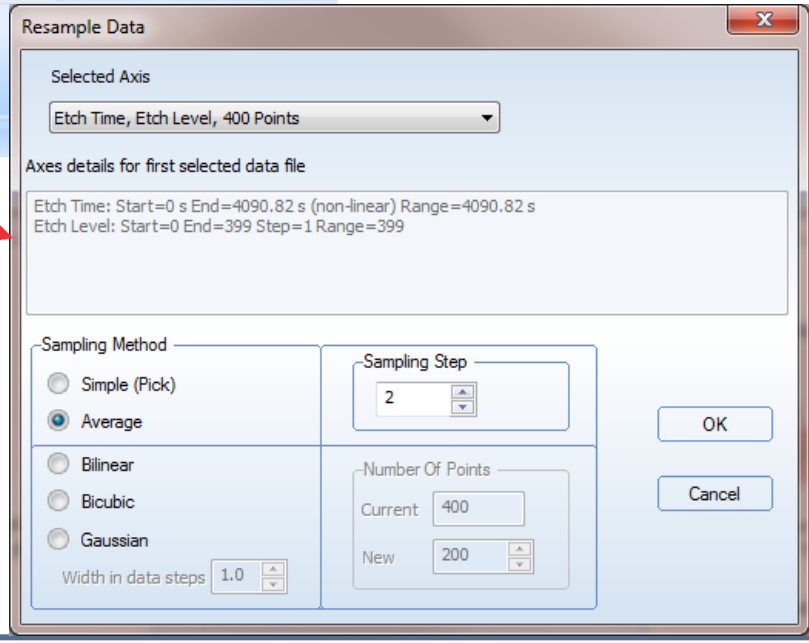
Data Resampling

- Sometimes we acquire too many data points – oversampling.
- Resampling allows us to correct oversampling
- Also permits interpolation of data sets in case of undersampling
- Works for spectra, images, profiles…





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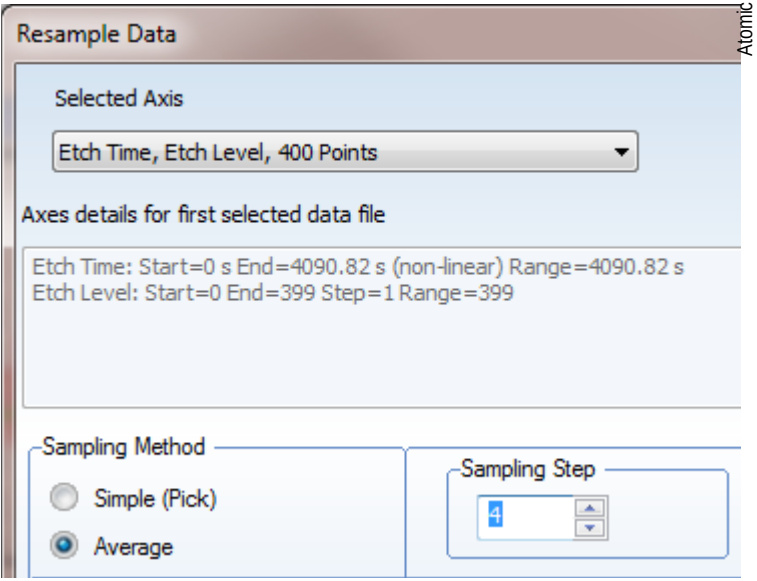
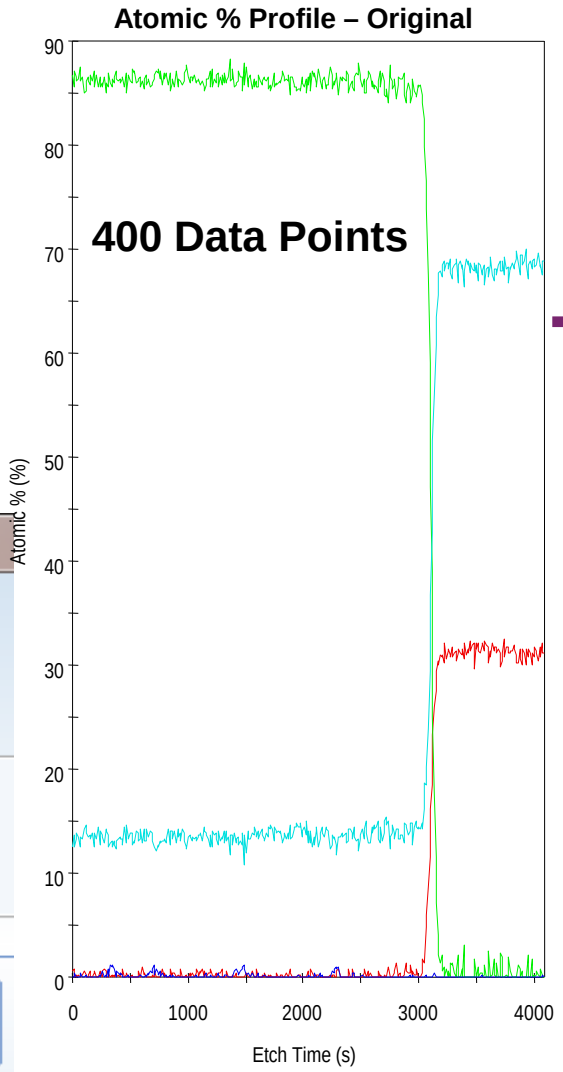
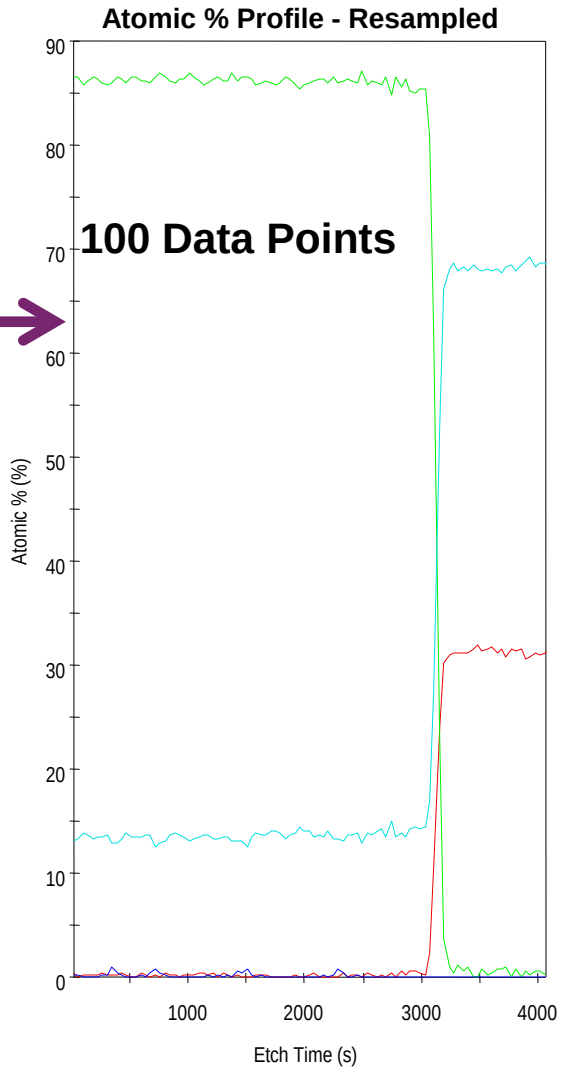
Data Resampling – Depth Profile

- Depth profile acquired with 400 levels – too many, noisy, slow processing
- Resample to 100 levels by averaging groups of 4 levels
- Clearer, less noisy profile. Easier to process.







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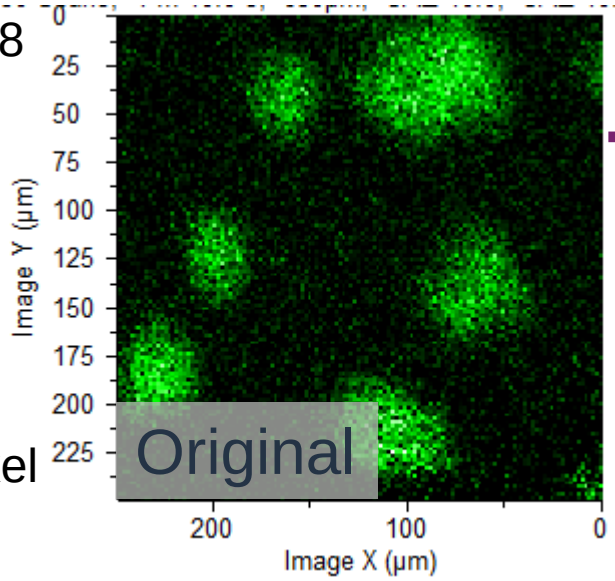
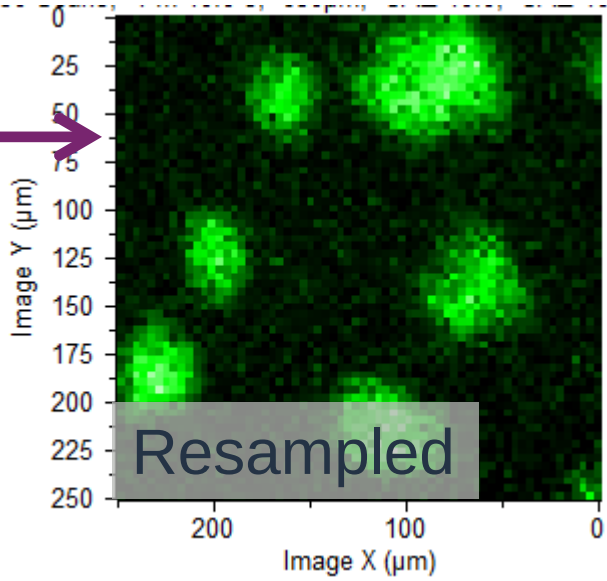
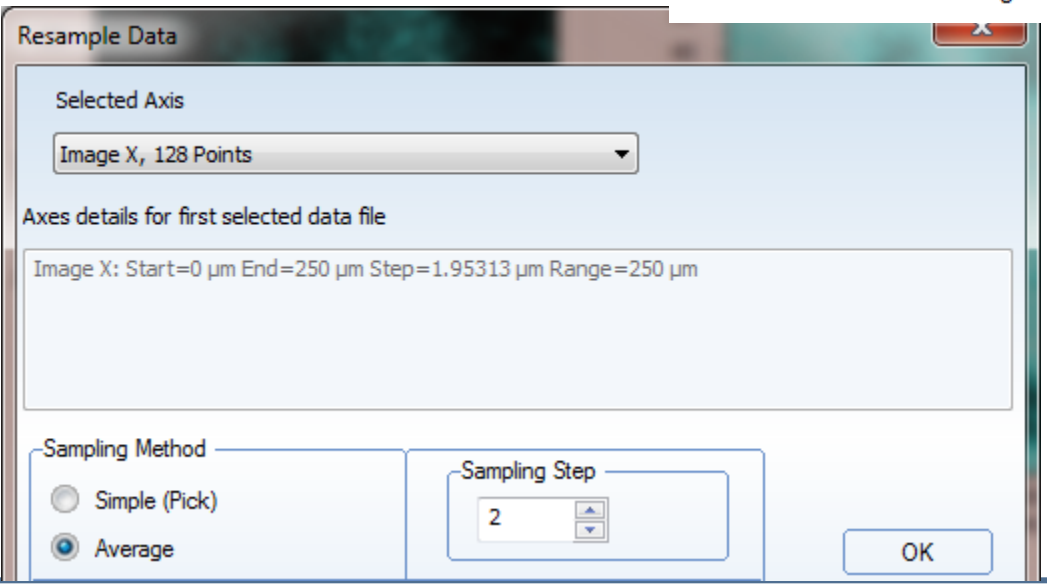
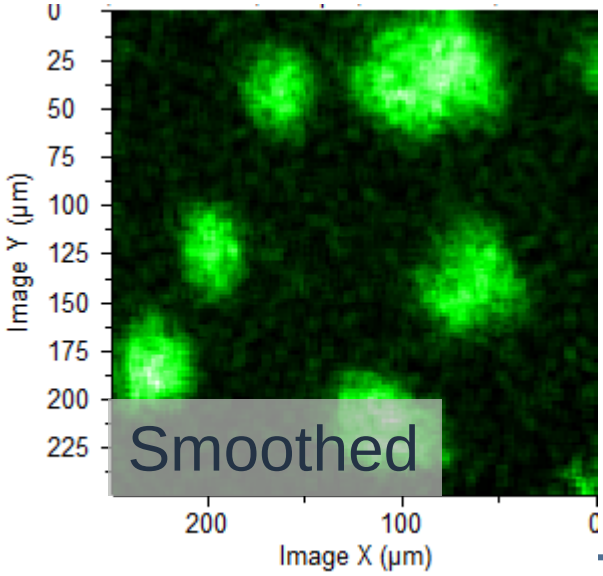
Data Resampling – XPS Image

- Spectral images with 128x128 pixels
- Resample to 64x64 pixels
- Clearer, less noisy images.
- Sharper than smoothing
- Better for quantitative data
- Improved spectra at each pixel









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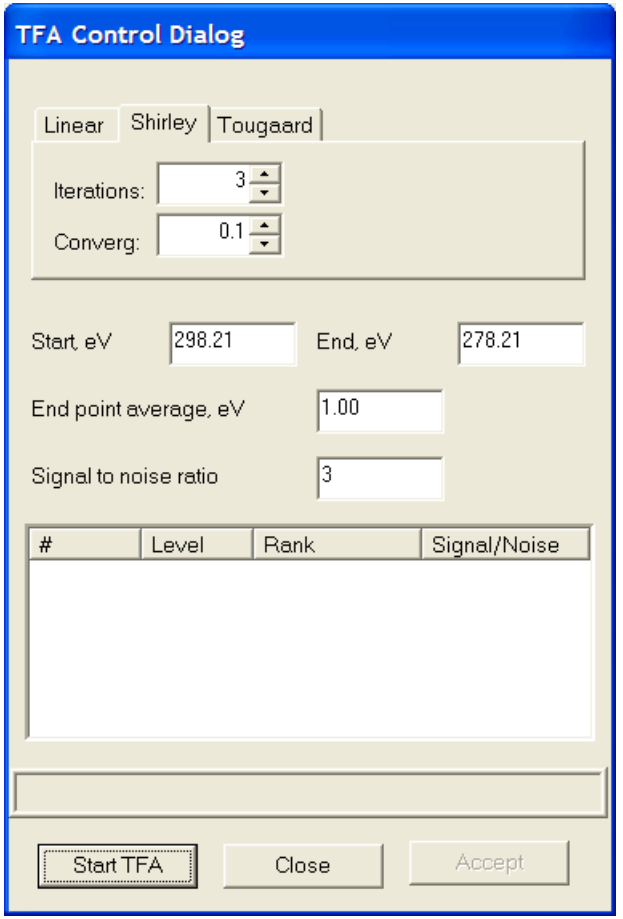
Data processing examples

Reviewing multi-level datasets

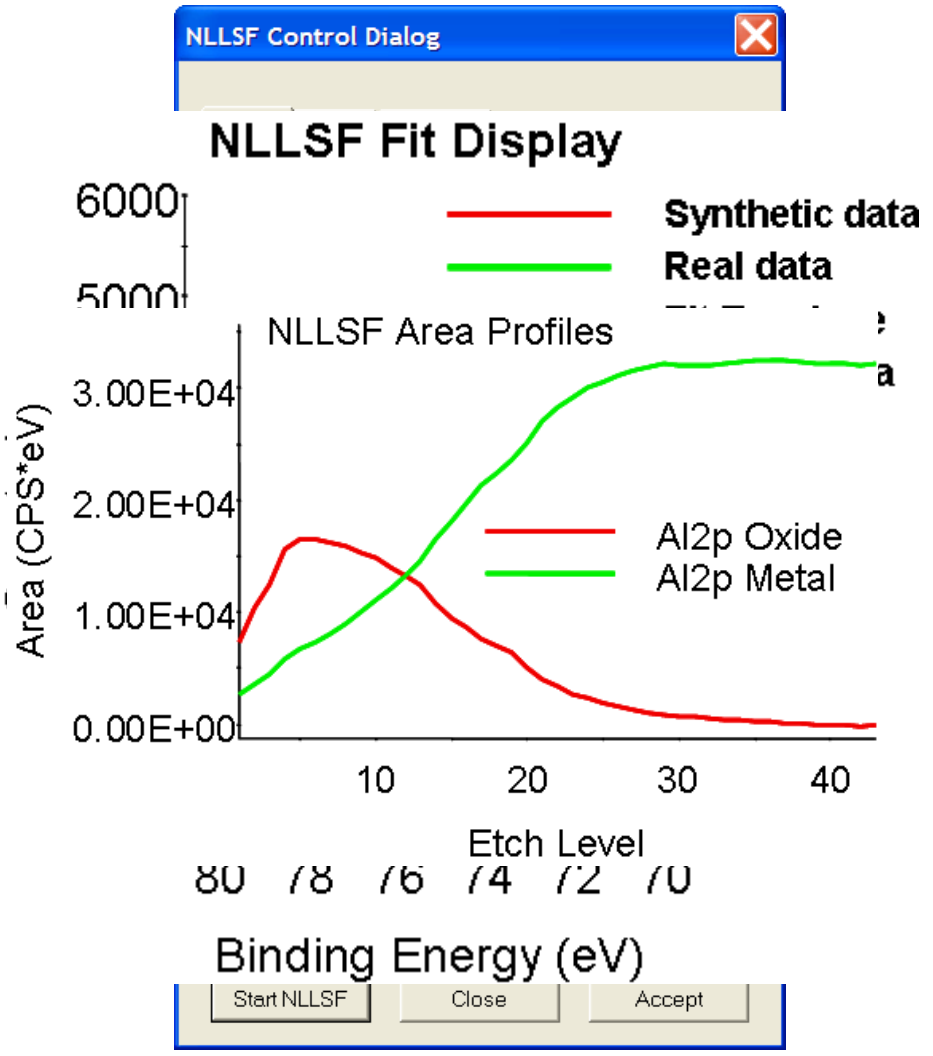
Target factor analysis

- Target factor analysis
  - Most significant components selected using TFA
  - Data set fitted with significant levels
  - Poorly fitted levels added as significant components
  - Process continues until all levels fit below signal to noise limit
- User defined parameters
  - Fit range
  - Background shape
  - Signal averaging for background points
    - Improves background selection for noisy data
  - Signal to noise ratio
    - Adjust the sensitivity of the process



Non linear least squares fitting

- Non Linear least squares fitting
  - User can select reference levels
  - Entire data-set fit with reference levels
  - Reference levels can by
    - Synthetic Peaks
    - Data from within the data set
    - Library reference spectra
  - Possible to combine synthetic and real references
  - Merged references for doublets
- User Defined parameters
  - Fit range
  - Background shape
  - Signal averaging for background points
    - Improves background selection for noisy data
  - Non linear components
    - Total shift
    - Shift increment
  - Merge selection



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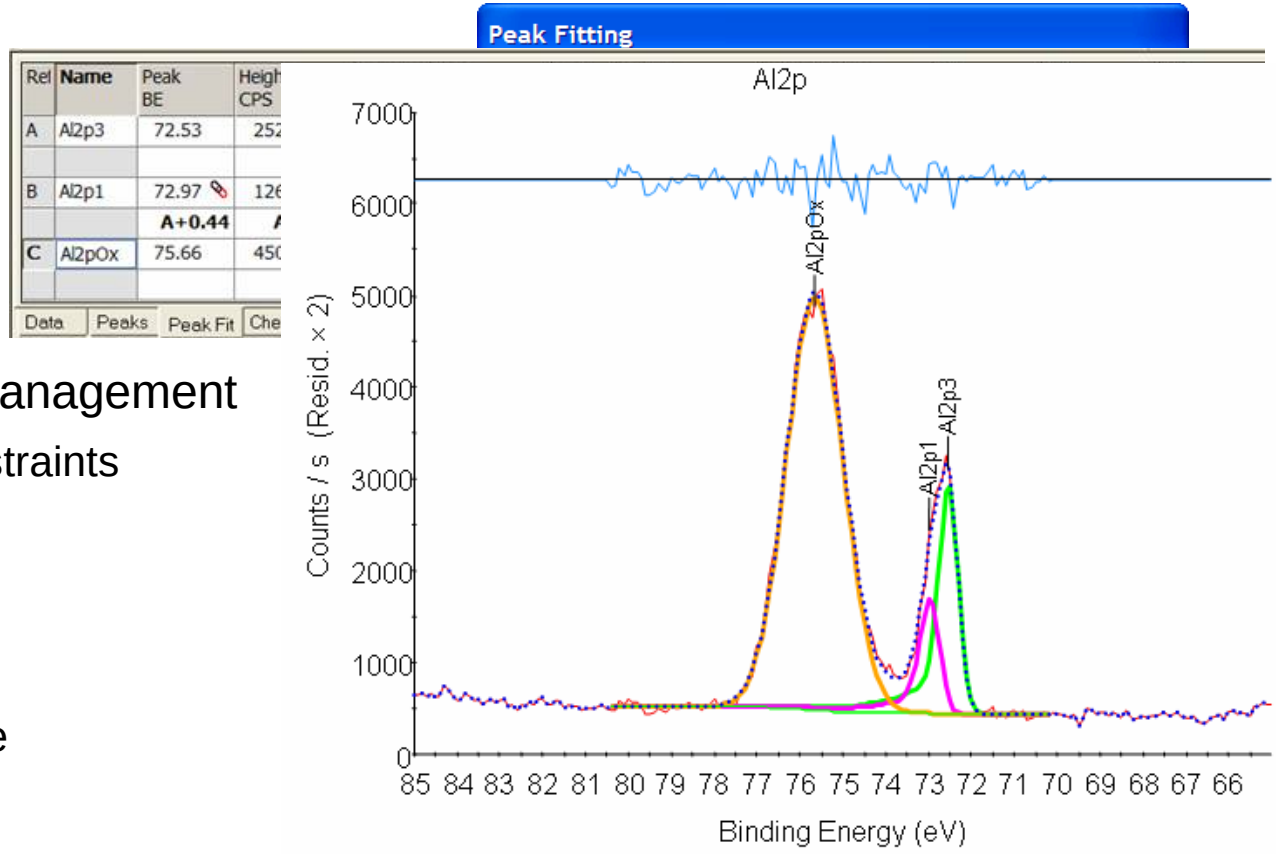
Peak fitting at every level

- Full fitting parameter control
  - Fix values
  - Link parameters
  - Set ranges
  - Asymmetric peaks
  - Add doublets
- Parameter & constraint management
  - Propagate values and constraints
  - Define data space ranges
- Recipes and automation
  - Save and load peak tables
  - Peak fitting within expt. tree



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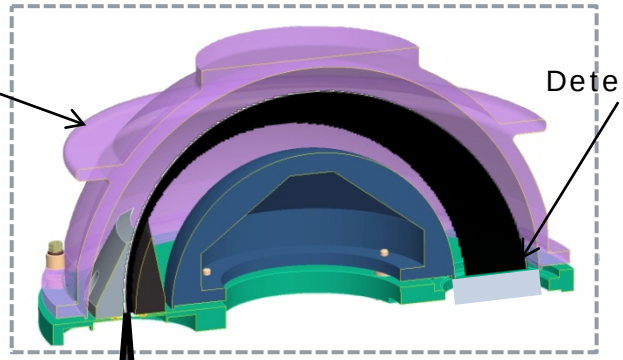
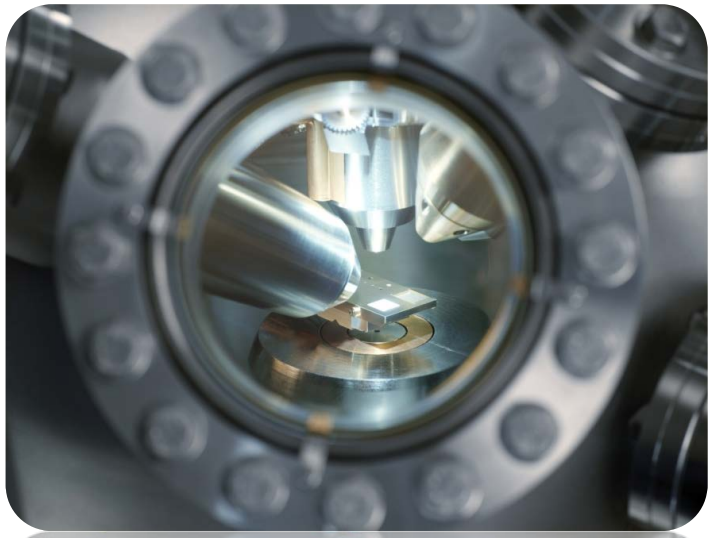
Data processing examples

Parallel XPS Imaging

Point XPS Analysis





2. Photo-electrons from the surface are collected and focussed into the energy selector

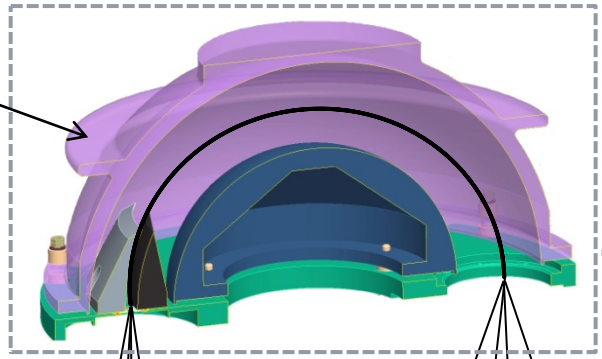
3. Energy filtration is applied, picking out a single energy

X-ray source

4. An energy spectrum is obtained by stepping through a large range of energies in turn

1. Aluminium Kα x-rays illuminate a large area on the sample, freeing electrons from the surface

Parallel XPS Imaging



3. A band-pass filter is applied, selecting a single photoelectron energy to be focussed onto the detector.

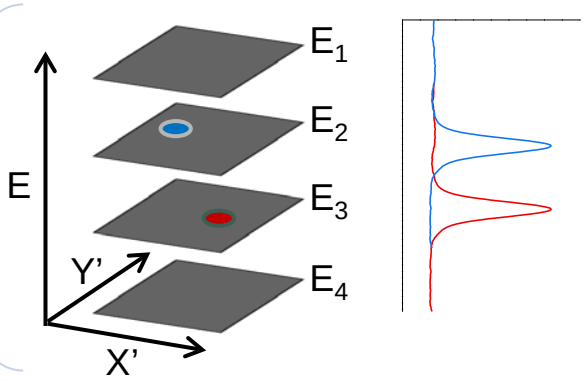
Detector

2. Photo-electrons from the surface are collected and focussed into the energy selector, while preserving positional information

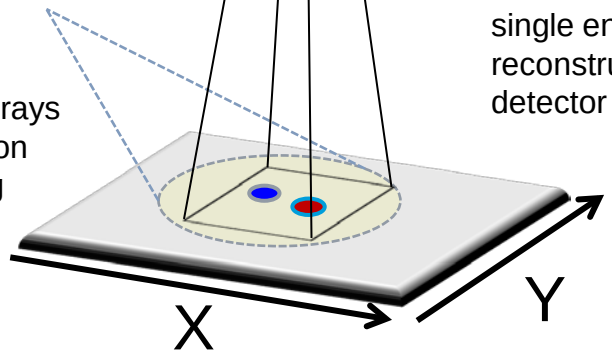
X-ray source

4. The 2D positional information for a single energy is reconstructed at the detector

5. By stepping through different energy values, a three-dimensional dataspace is formed.



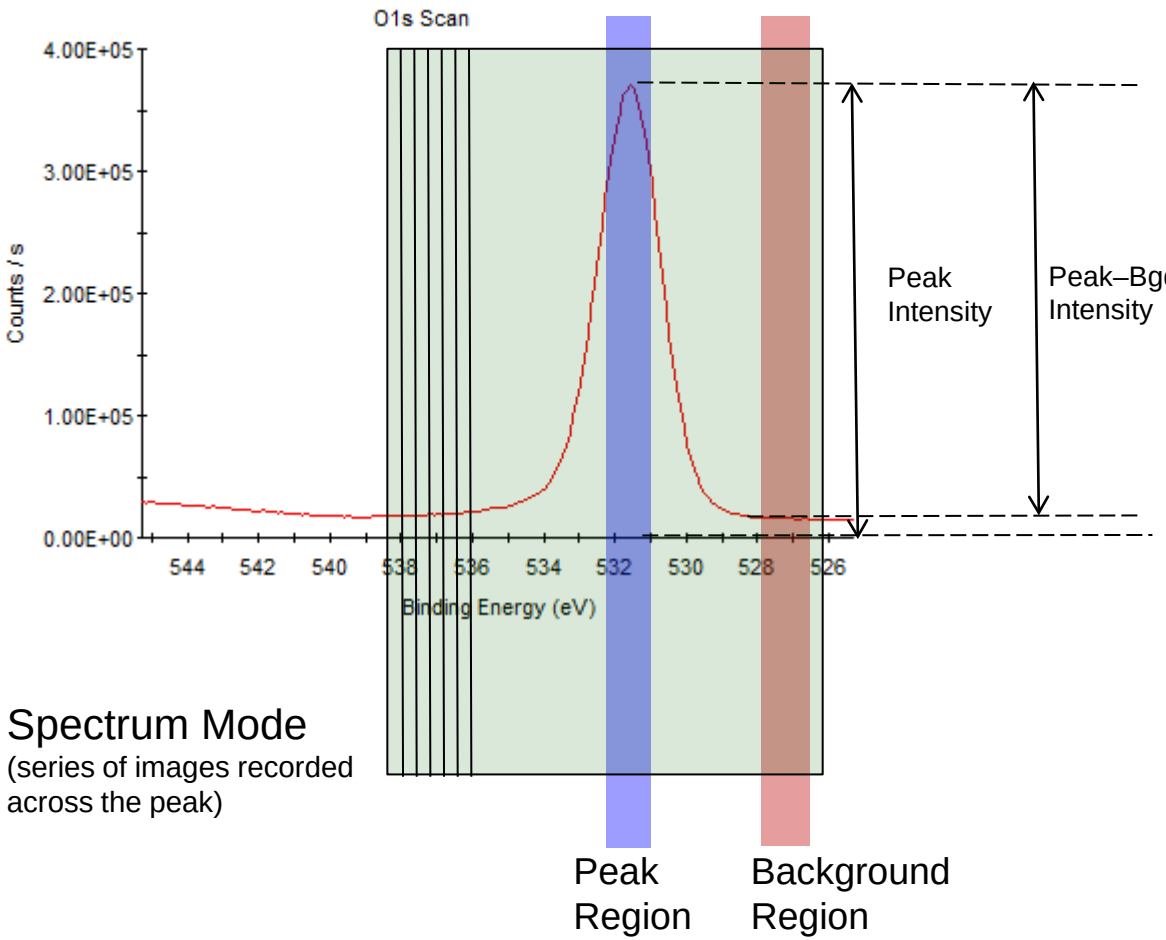
1. Aluminium Kα x-rays illuminate an area on the sample, freeing electrons from the surface



6. Energy spectra can then be reconstructed from 2D areas in the images. In this example we can see an energy shift between the blue and red areas

Parallel Imaging – 3 Modes of Operation

- There are three possible imaging modes
  - Peak only
  - Peak - Background
  - Full Spectrum
- Each has distinct advantages
- Time vs. Information trade-off



Spectrum Mode
(series of images recorded across the peak)

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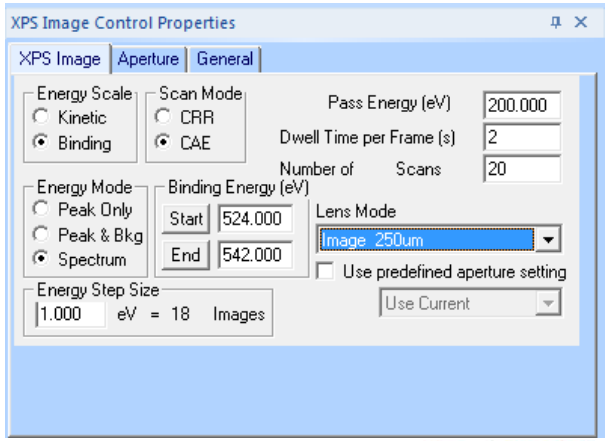
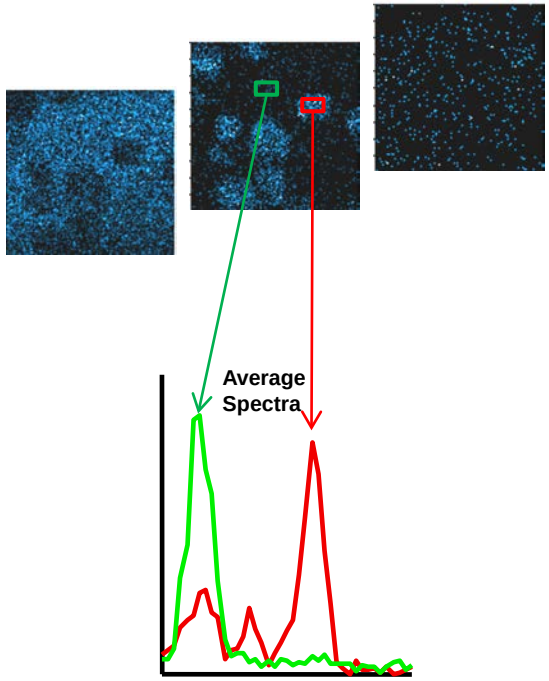
Image-Defined Area Selection

For image-defined area selection we acquire quantitative spectral image sets.



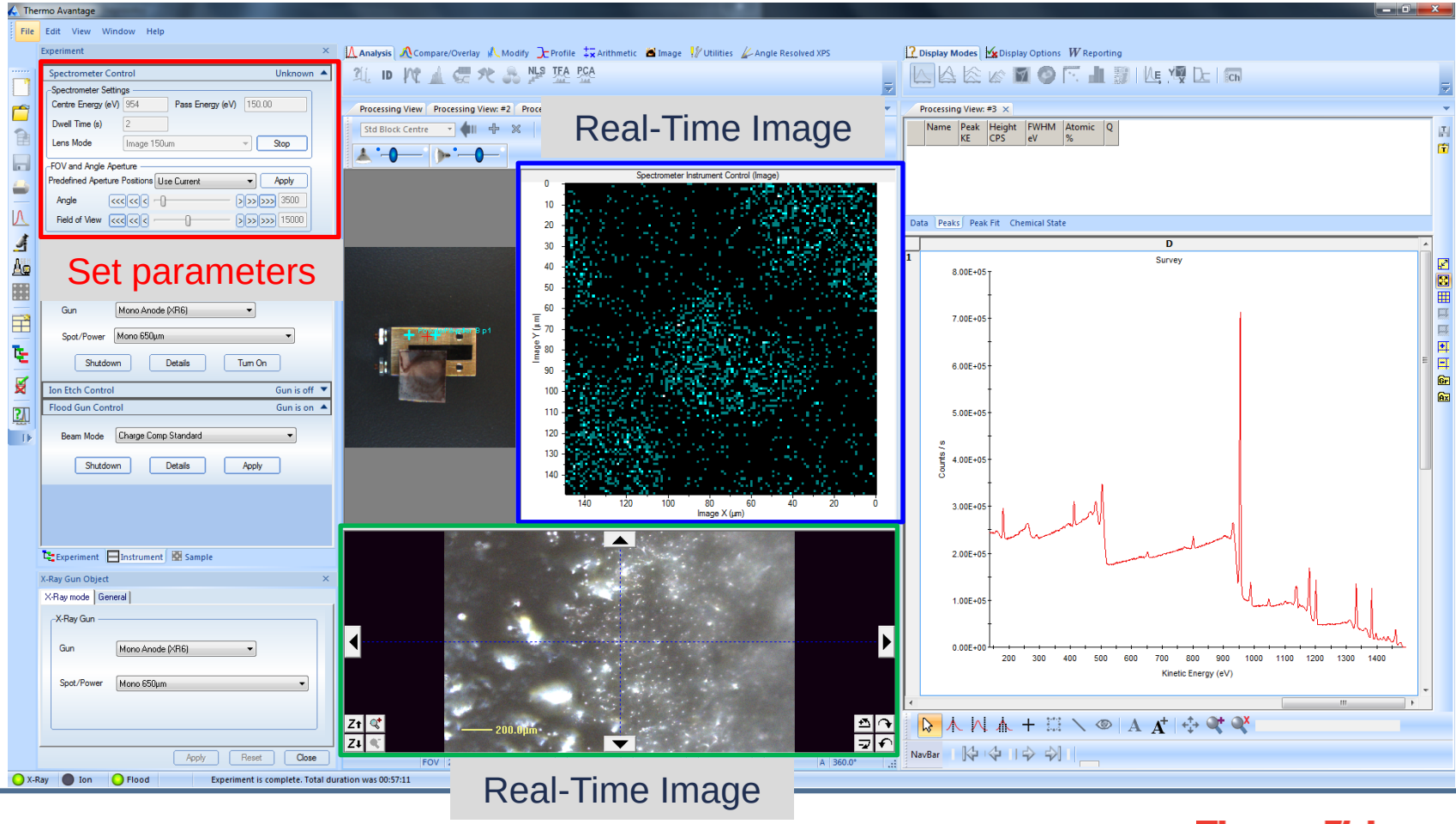
This is a sequence of images spanning a spectral region of interest. The result is an image with a spectrum at each pixel.



Any rectangular area can be selected from the image to generate average spectra from that area.

Real-Time Imaging For Alignment

- Alignment for selected area XPS using lens-defined analysis is straightforward with real-time parallel XPS imaging. The Spectrometer Control (red box) can be used to start imaging a peak of interest using any chosen instrument parameters. The Current Data View (blue box) then updates continuously (usually every 1-2 seconds) with a live XPS image of the sample. The live camera view (green box) allows fine changes in stage position to centre a feature of interest in the image, either by using the move arrow buttons or by double-clicking within the camera image.



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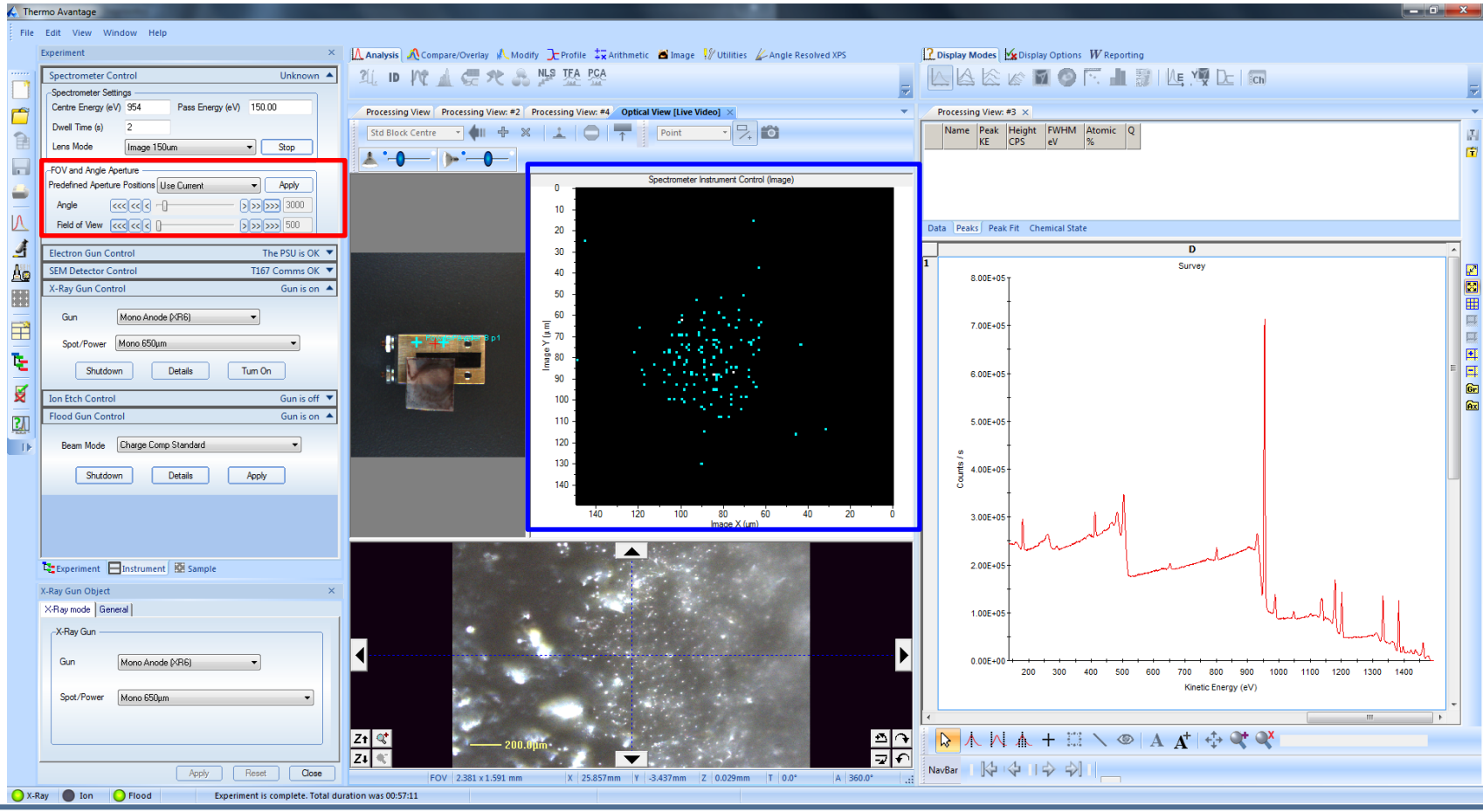
Real-Time Imaging For Alignment

Once a feature is centred, the analysis area can be chosen using the aperture control (red box). This lets the user close the field of view iris until the analysis area is within the feature of interest. The live image updates to show the iris closing, so that the size and location of the analysis area is precisely known. Small-area XPS can then be performed using the same iris settings, so that the analysis area is exactly the same as in the image.



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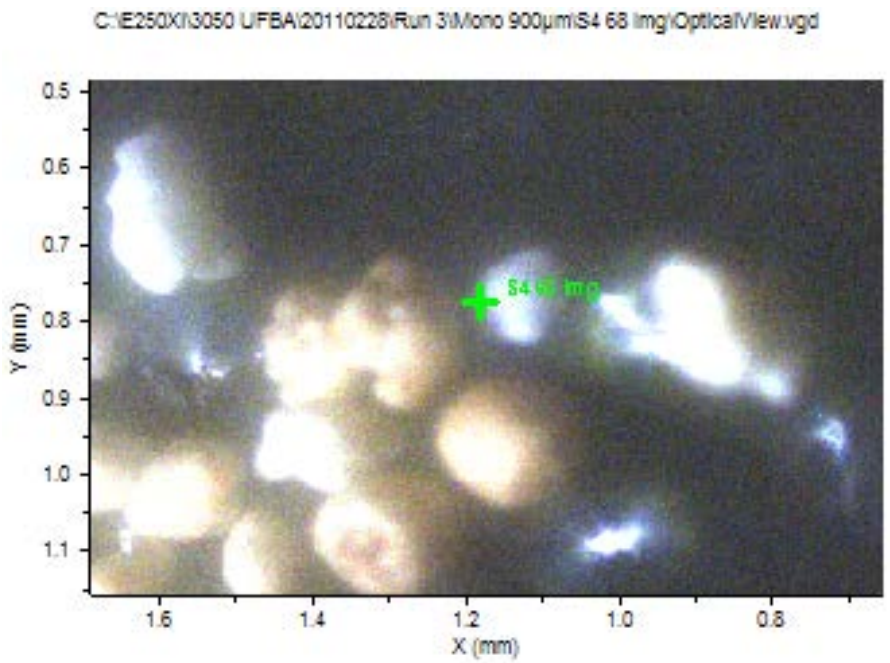
CO_2 Treated catalyst XPS Imaging Example

- Dispersed catalyst grains were studied
- Parallel imaging technique, 250 µm FOV
- Quantitative spectral imaging data
- Sequences of images in small energy steps across each peak.
- Images with a quantifiable spectrum at each pixel.



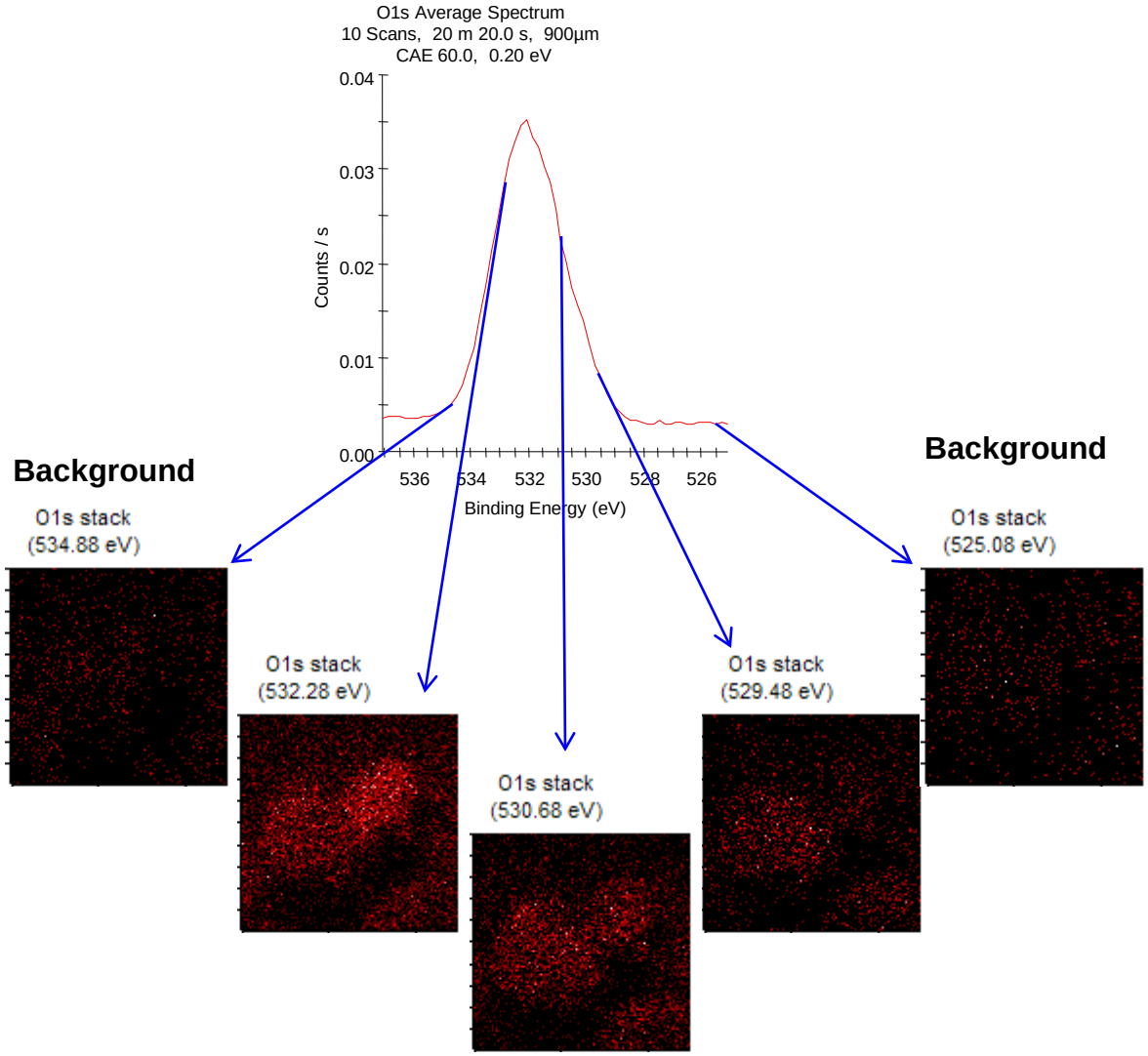
- The images were acquired with an analyser pass energy of 60 eV and an energy step between images of 0.2 eV. Each image sequence was acquired for 10 scans (8 for Si2p). Therefore each single-energy image was acquired for about 20 seconds.

- Acquisition Regions
  - **Si2p**
  - **O1s**
  - **C1s**
  - **Fe2p**
  - **Ni2p**

- A zoomed-in portion of the optical view shows the particles at the analysis position
- One of the particles at the centre is brown,
- The other is white.
- Do these particles have a different surface composition?

CO_2 Treated spent catalyst

- As an example, some of the O1s spectral image data
- 60 images were acquired in total, from 525-537 eV binding energy, in steps of 0.2 eV.
- The spectrum shown generated by averaging spectra from all 16384 pixels in the image set
- Represents the total O1s spectrum with no spatial information left.
- Five images from the set are shown below, with arrows showing the binding energy they correspond to. The middle three show varying intensities on the particles as the binding energy changes. This demonstrates that the two particles have different oxygen chemistries.



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•The data sets were quantified to generate atomic % images of the sample, shown here. These show two particles fully within the imaged area, one large and one small. The approximate outlines of the particles are shown on the Si2p image.

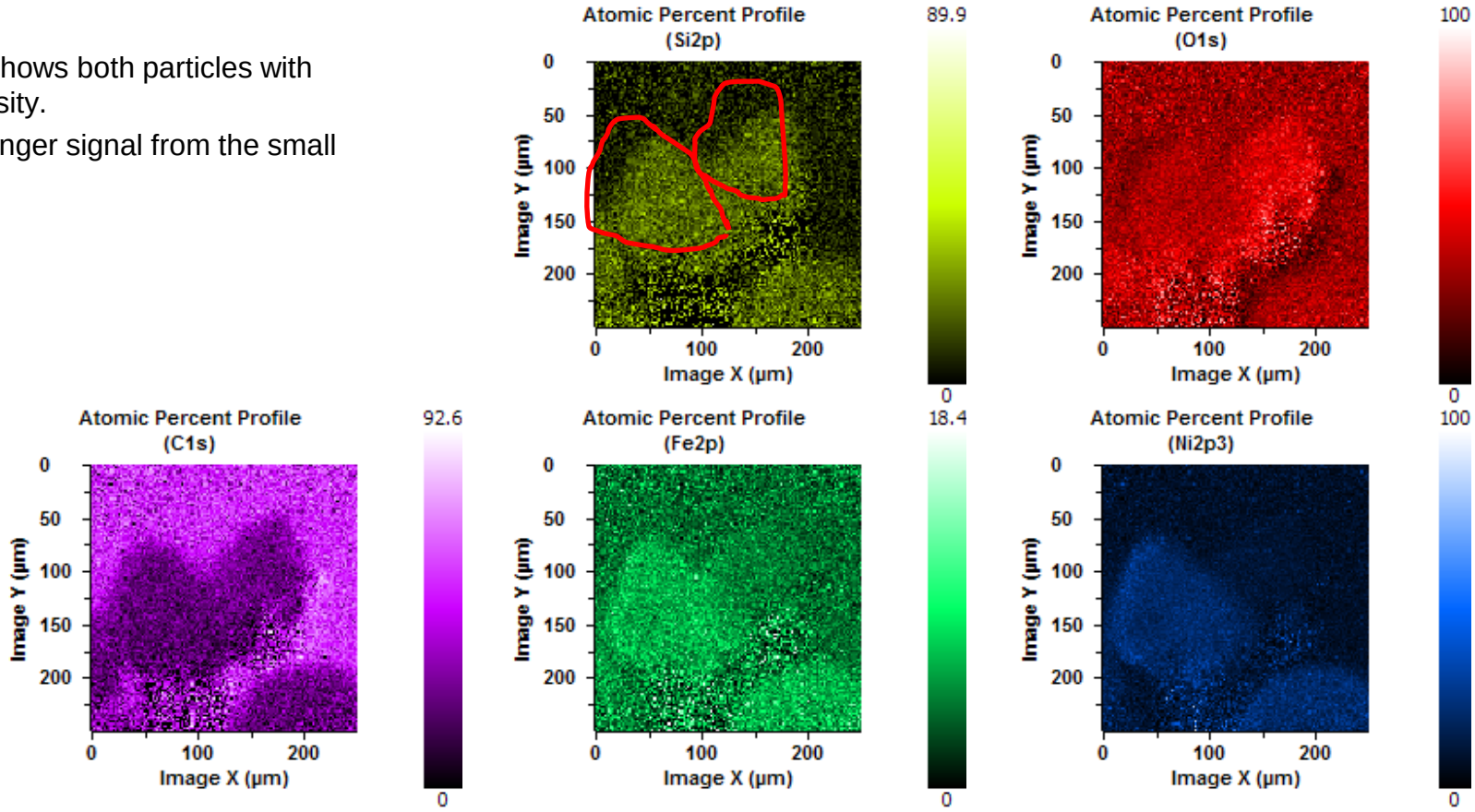
• The Si2p image shows both particles with similar signal intensity.

• O1s shows a stronger signal from the small particle.

•Fe2p and Ni2p3 show a stronger signal from the larger particle.

•This indicates that the smaller particle has lower concentrations of metallic species at the surface

•Does this explain the colour change?



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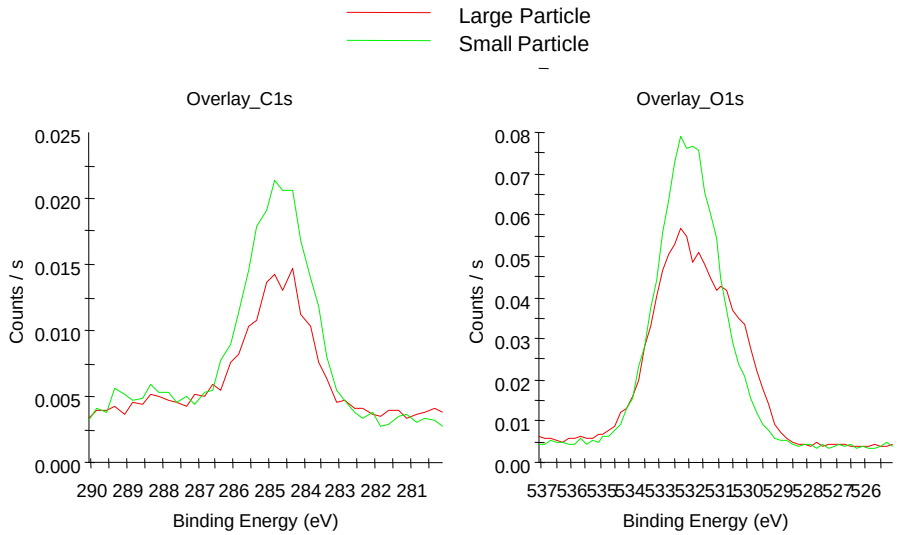
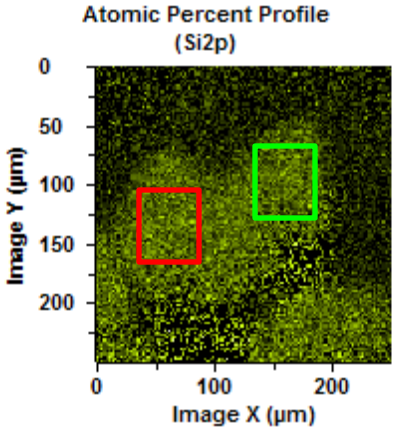
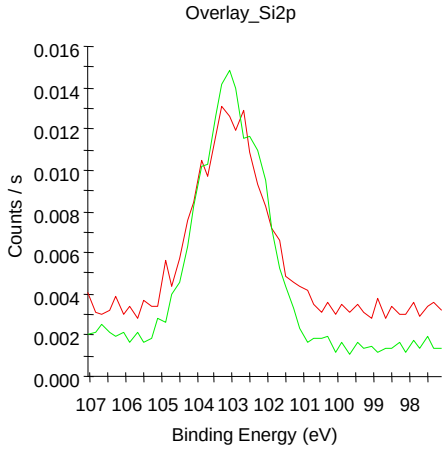
CO_2 Treated spent catalyst

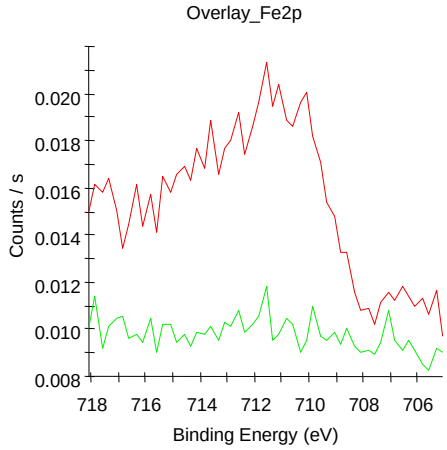
- Rectangular areas were defined on the at.% images (shown on the Si image below left), and the spectra from all pixels within those areas were averaged together. A comparison of spectra from the large particle (red) and small particle (green) shows that the small particle has virtually no Fe or Ni, and increased C and O (and a small increase in Si).

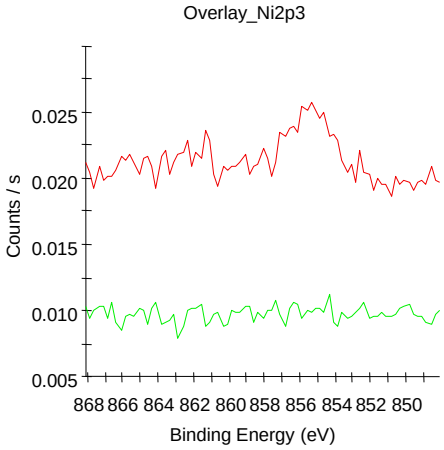
- On the large particle, the O1s spectrum shows a clear metal oxide peak to low binding energy, but that is weak or missing from the small particle.











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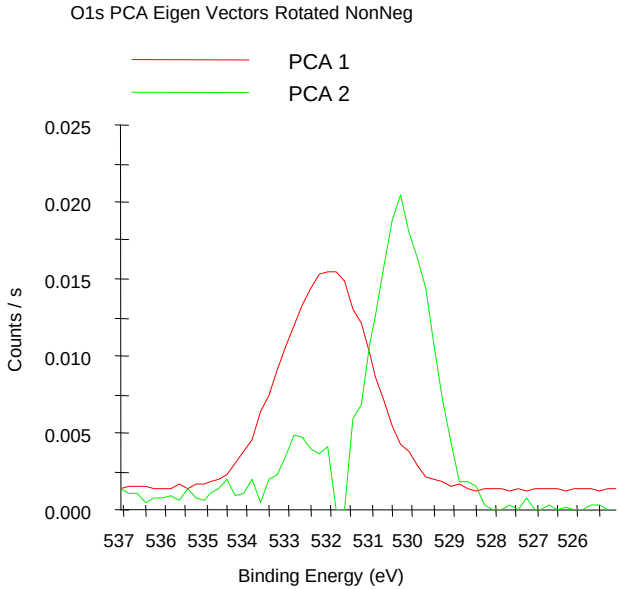
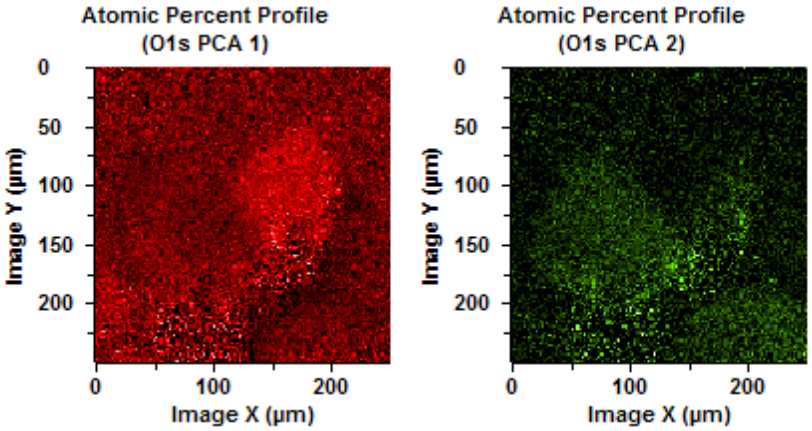
CO_2 Treated spent catalyst

- The principal component analysis (PCA) algorithm was applied to the O1s spectral image set. This identified two factors, shown right. The second (green) factor is dominated by metal oxide chemistry, whereas the first (red) is more typical of Al and Si oxides, and organic material.
- The two factors were then fitted back into the original data to plot the distributions of the two chemical states. An example of this is shown far right, with the two factors fitted to one of the spectra from a single pixel in the image set. One strength of PCA is clear here – the factors identified in the process have much better signal statistics than any single spectrum in the data set. This is due to the PCA function using the entire data set to identify signals and reduce noise.
- After fitting the PCA factors to the data, the atomic % images presented earlier were regenerated, including the two O1s states.
- The O1s images are shown below. PCA 1, the "other" oxygen, is on both particles but is stronger on the smaller one. PCA 2, the metal oxide, is only strong on the large particle. The different distributions of the two states are clear from the PCA analysis.





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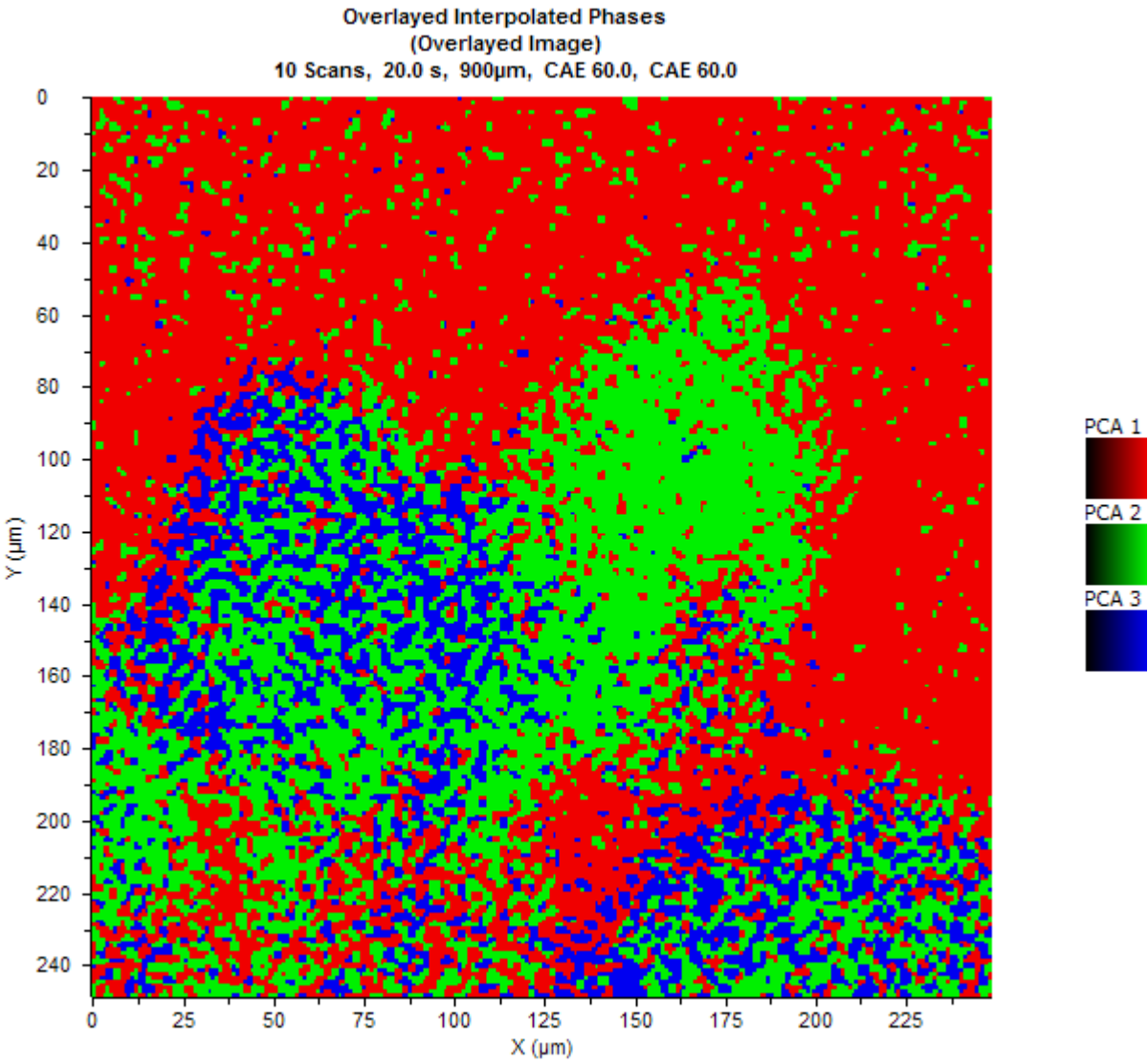
CO_2 Treated spent catalyst: Phase analysis Result

- For further detail, the Phase Analysis algorithm was used, which applies principal component analysis (PCA) to the quantified compositional information at each pixel. This effectively looks for areas of similar composition in the sample
- The phase analysis algorithm identified three compositional phases as shown in the phase image here.
- **The first phase (red) is the surrounding carbon tape.**
- **The second phase (green) is all particles within the image.**
- **The third phase (blue) highlights the brown coloured particles, but not the smaller white particle**.



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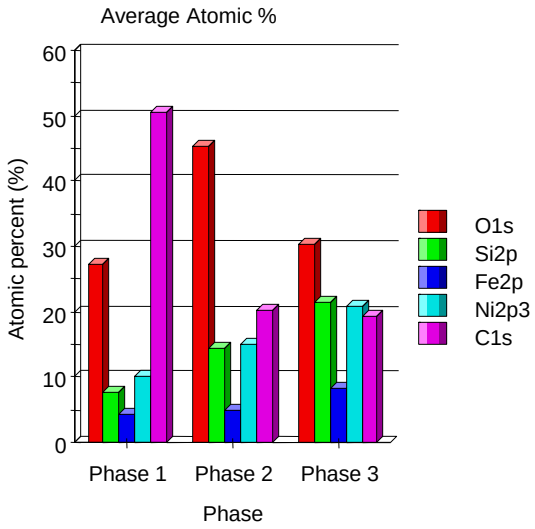
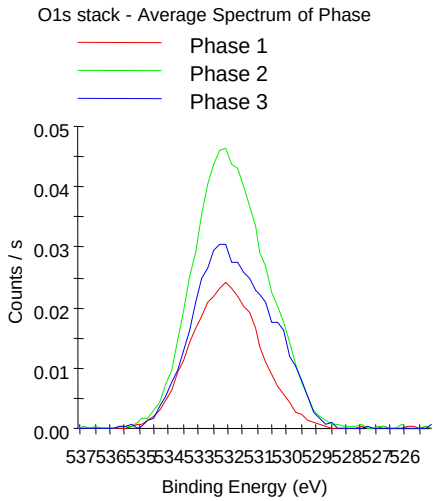
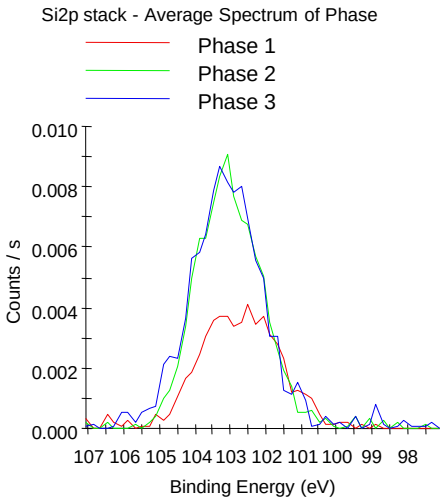
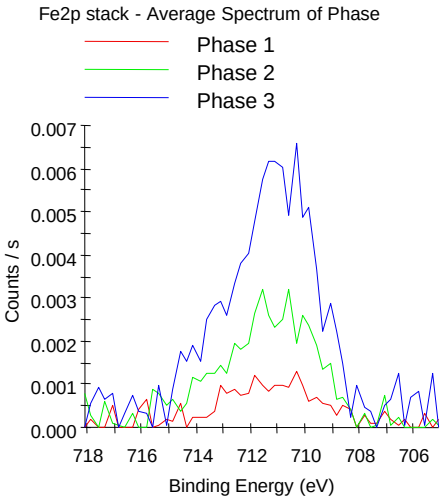
CO_2 Treated spent catalyst

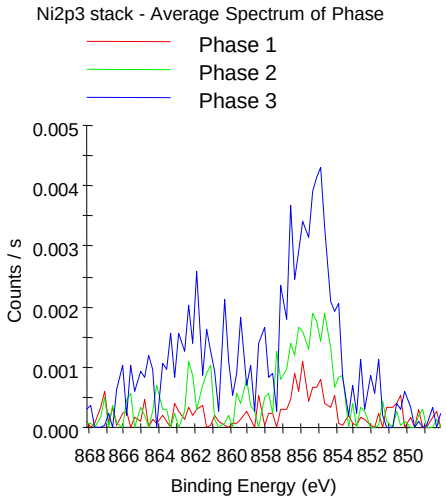
- The phase analysis routine generates average spectra from each phase, by combining the spectra from all of the red, green and blue pixels. These are shown here, together with the compositional information from these average spectra.

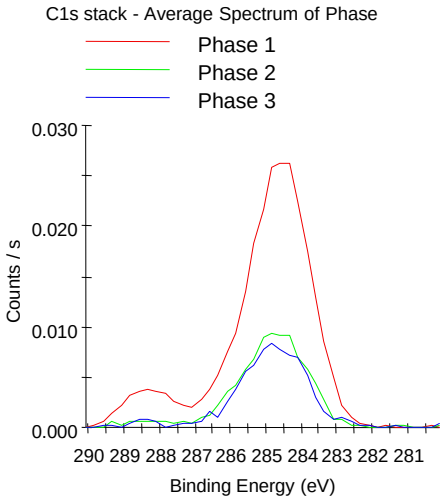










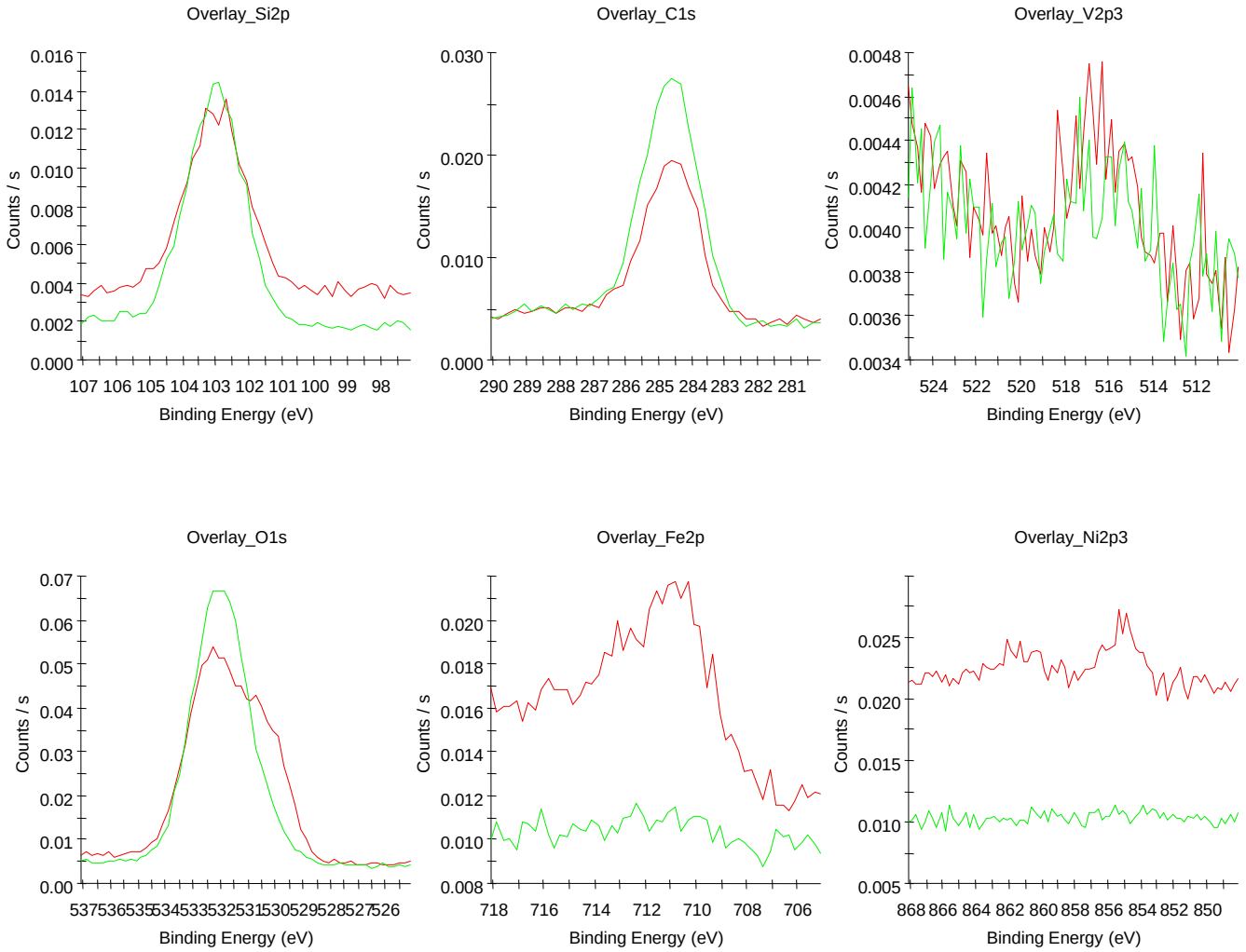


CO_2 Treated spent catalyst

- Average spectra were generated from rectangular areas on the large (red) and small (green) particles as before. The spectra are shown here. These have been quantified to give the composition tables below. This data shows that the white particle does have a small amount of Fe and Ni, and has a similar amount of V to the brown particle.

Peak	BE / eV	At. %
Si2p	102.9	16.68
C1s	284.6	23.32
V2p3	516.3	0.24
O1s	532.0	55.93
Fe2p	710.7	2.26
Ni2p3	855.4	1.57

Peak	BE / eV	At. %
Si2p	103.0	17.42
C1s	284.6	32.72
V2p3	517.1	0.18
O1s	532.0	49.13
Fe2p	712.4	0.21
Ni2p3	853.6	0.34



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Data processing examples

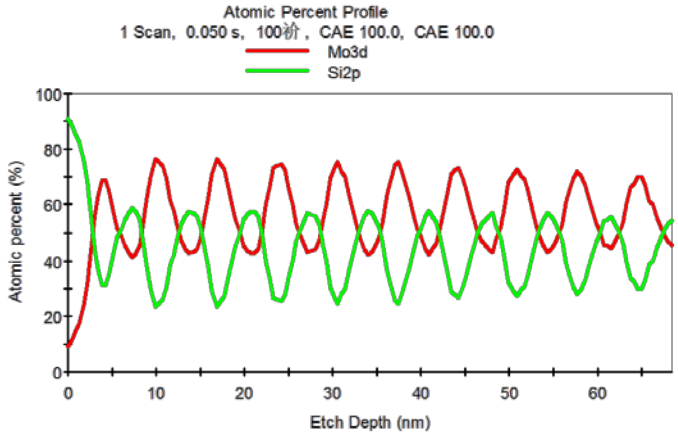
Depth profiles

Depth profile deconvolution

Escape depth correction

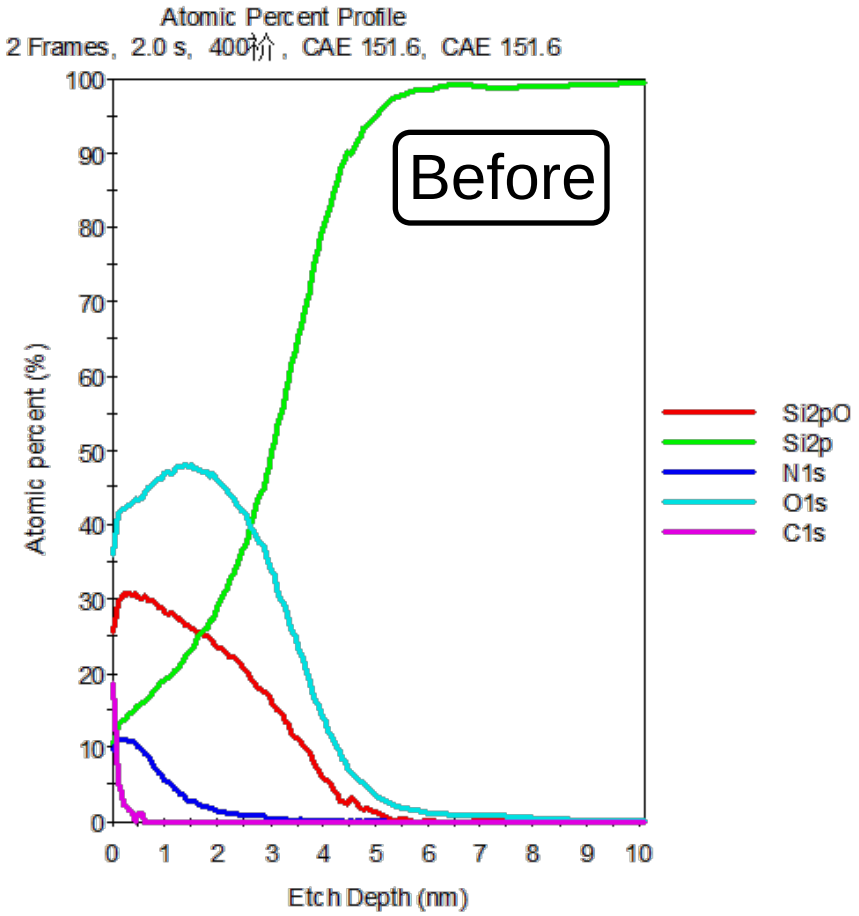
- Corrects for sampling depth
- Using Briggs and Seahs equation
  - $X(z) = I(z) - d(I)/dz * \lambda$

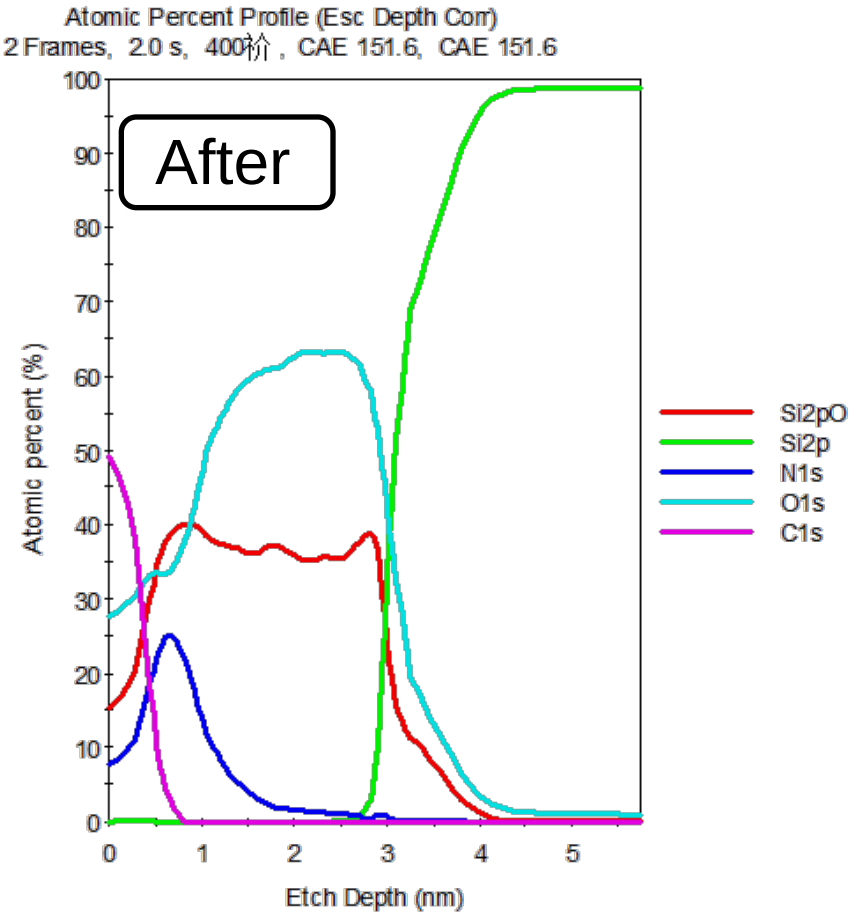
    eq. (4.9) on p155 of Briggs and Seah
  - Attenuation length is calculated using an “Average Matrix”
- Simple fast implementation
- Improves resolution of ultra shallow profiles
- Resolves discrete interface layers



Depth profile deconvolution

SiOxNy/Si Profile





- Resolves discrete layers
- Improves depth resolution

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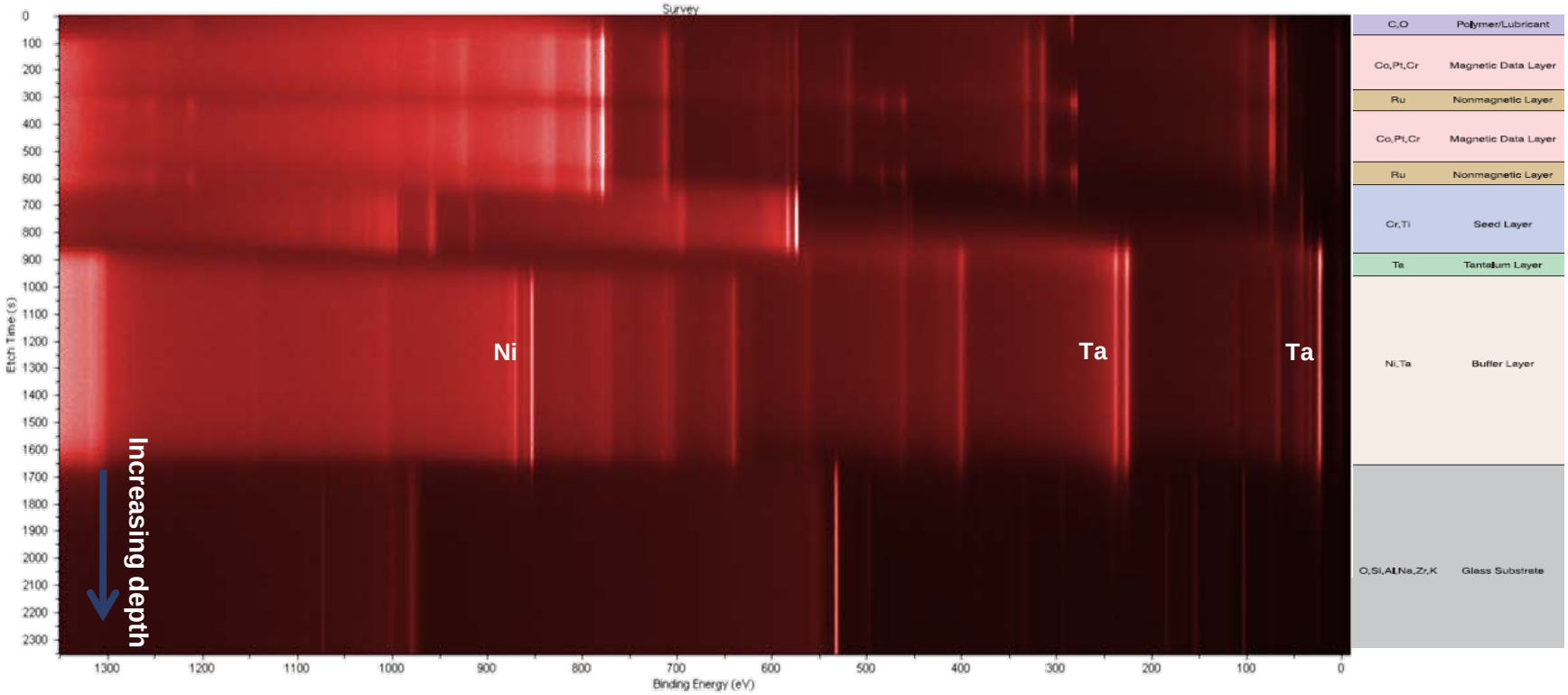
PCA depth profile analysis



- Survey depth profiling is a method which captures all XPS-detectable elements during a depth
  - It is not necessary to know all of the potential elements in the composition profile prior to analysis
- Only feasible with high sensitivity XPS system, combined with rapid-response electronics/software for data collection
- Above example is K-Alpha survey depth profile of a hard disk
  - Survey spectra at each profile level have only 9s acquisition time and total time to substrate is only 100 mins
  - All elements within the disk have been detected
  - Avantage datasystem allows collection of survey spectra to be viewed as an “image”, which can then be directly correlated with the layer structure of the hard disk

Data processing examples

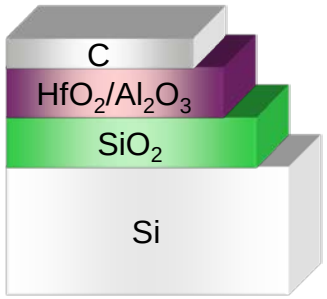
Angle Resolved XPS

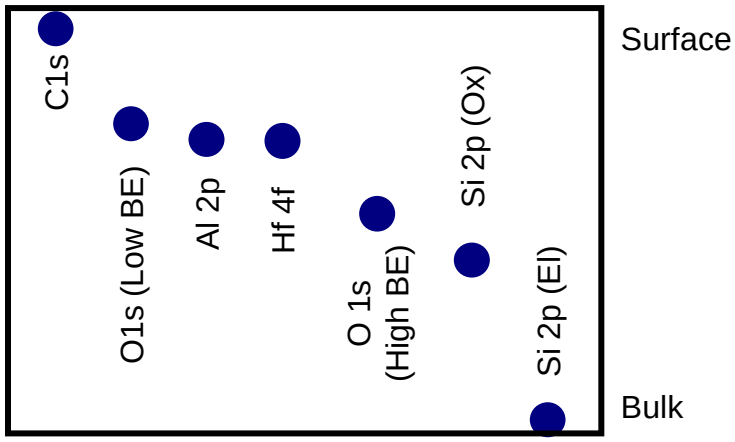
Relative Depth Plot

Provides Information about layer ordering

- Construction:
  - Collect ARXPS spectra
  - For each element, calculate:

$$\ln\left(\frac{I_{SurfaceAngle}}{I_{BulkAngle}}\right)$$



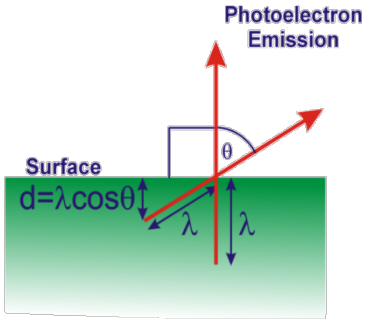


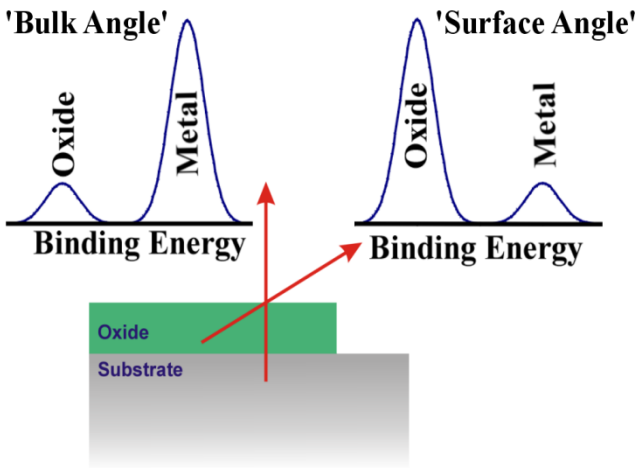
- Information
  - Reveals the ordering of the chemical species
- Advantages
  - Fast
  - Model independent, no assumptions
- Limitation
  - No depth scale

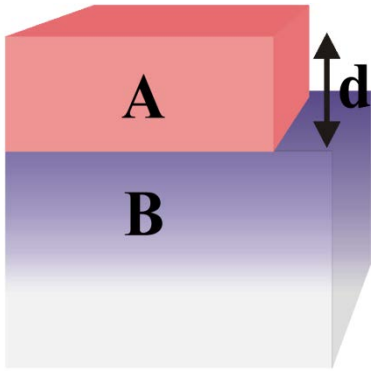
- Relative depth plot from silicon oxynitride shows:
  - C at surface
  - Two N species each with different depth distributions
  - Si substrate

Single Overlayer Model

- Information depth varies with collection angle
  - $I = I^{\infty}\exp(-d/\lambda\cos\theta)$

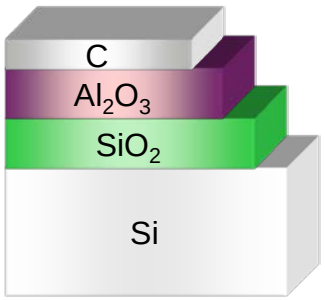






- **Signal from A**
  - $IA = I^{\infty}_{A}[1-\exp(-d/\lambda_{A,A}\cos\theta)]$
- **Signal from B**
  - $I_B = I^{\infty}_{B}\exp(-d/\lambda_{B},A\cos\theta)$
- **Ratio**
  - $$\frac{I_A}{I_B} = R = R_0 \frac{\left[1-\exp\left(-d/\lambda_{A,A}\cos\theta\right)\right]}{\exp\left(-d/\lambda_{B,A}\cos\theta\right)}$$
- **Simplify**
  - If $I_{A,A} = I_{B,A} = I_A$
  - Then **$\ln[1+R/R_0] = d/(\lambda_A\cos\theta)$**

Multi-Overlayer Calculator

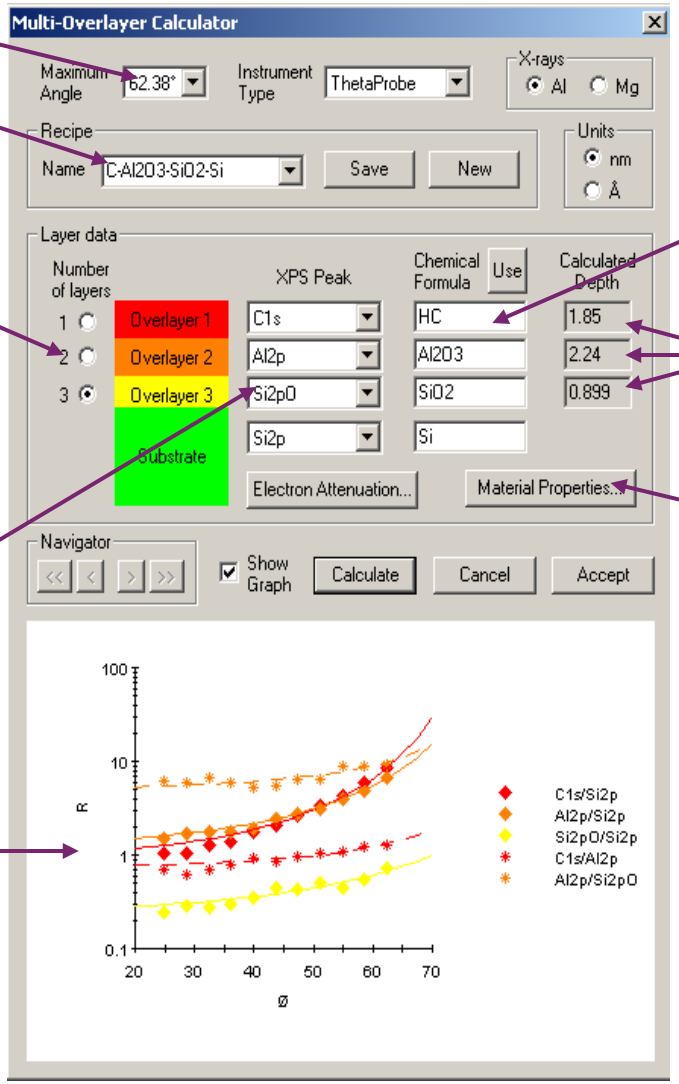


Set angular range

Save ‘recipe’

Select number of layers

Select peaks



Define layers

Calculated Thickness

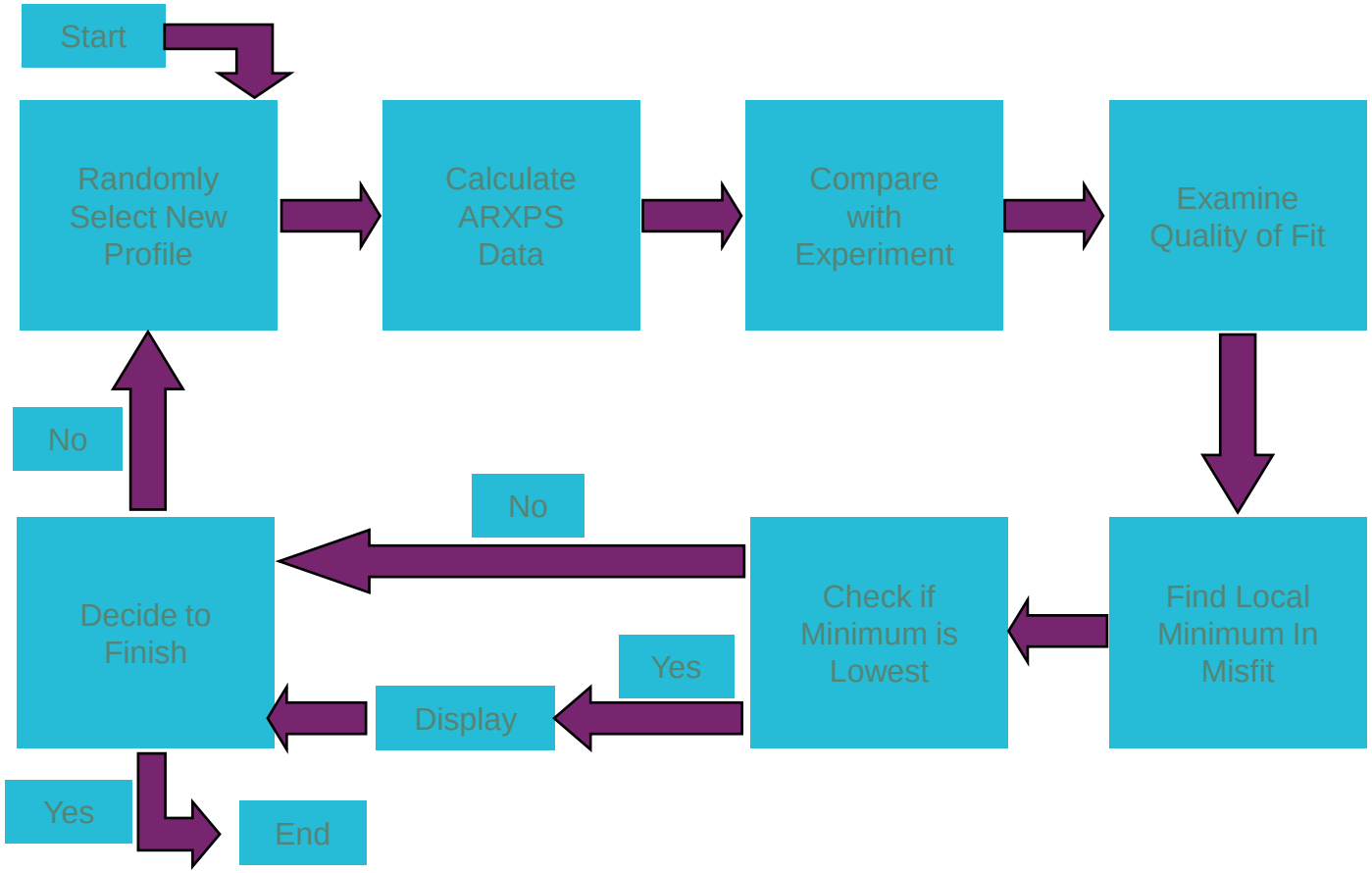
Enter material properties if not in data base

Compare calculated and experimental data to assess quality of fit

Operates on multi - dimensional data sets such as linescans and maps etc

Depth Profile Generation

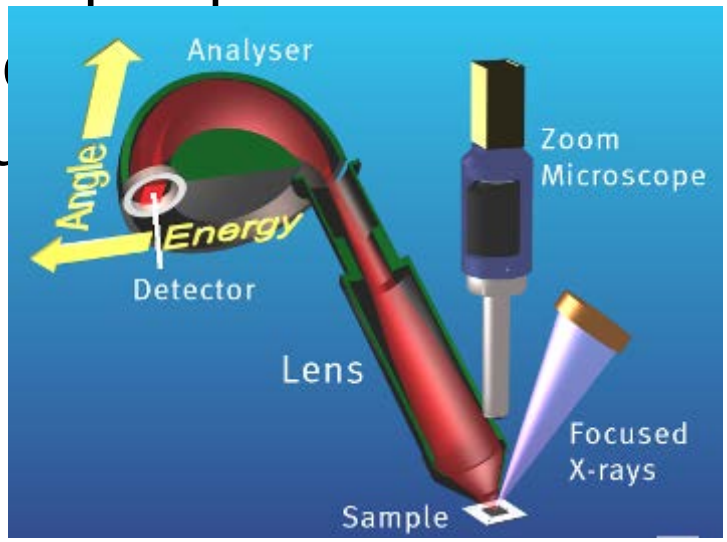
Non-destructive depth profiles can only be generated using an iterative method



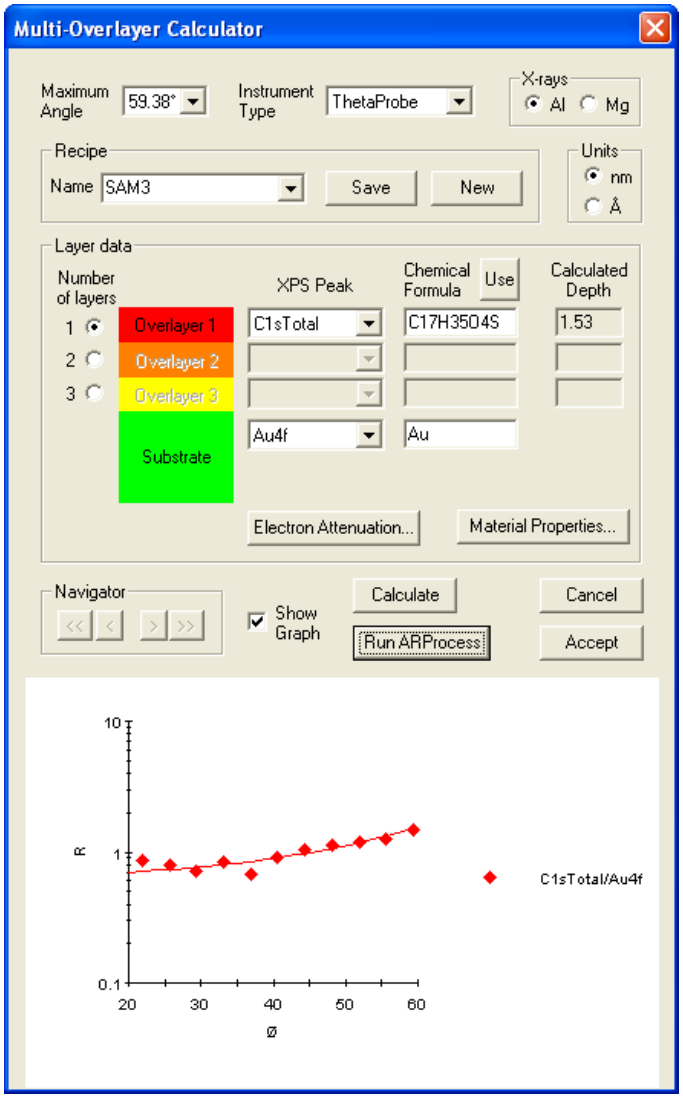
ARXPS Suite

Multi-Overlayer thickness calculator

- Built in TPP-2M calculator
- Fit up to three layers on a substrate
- Built in materials database
- Select angular range
- Recipe operation
- C
  su



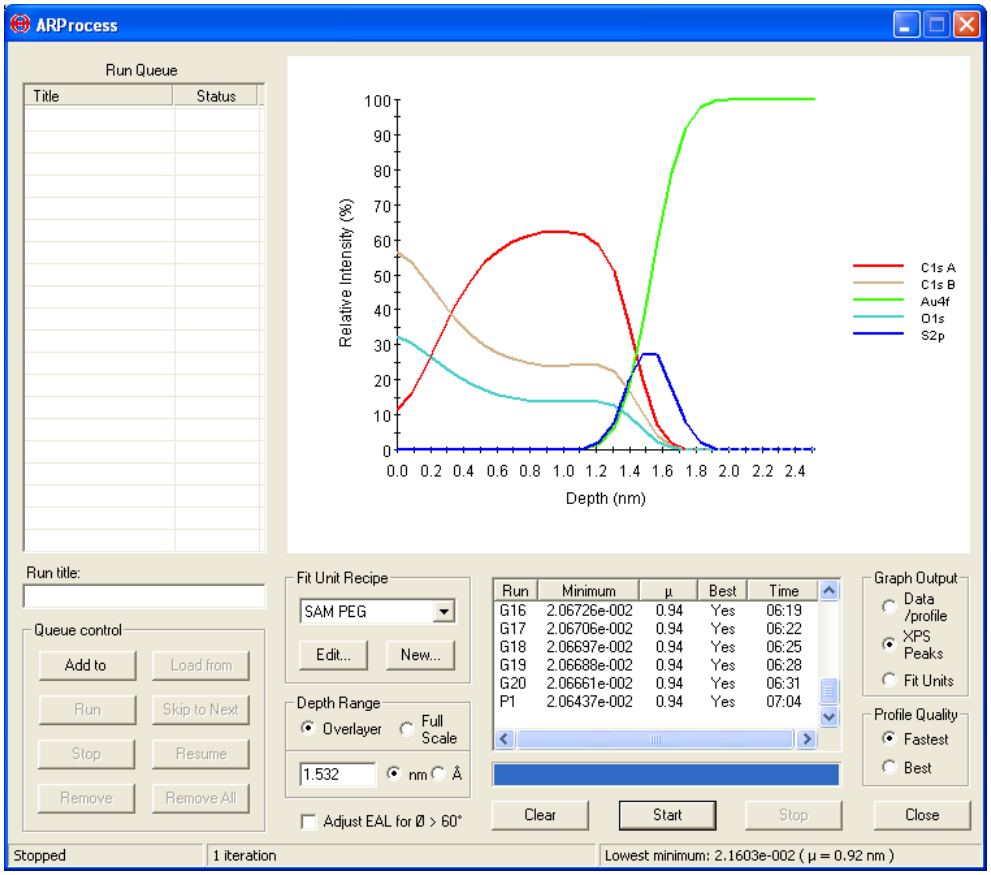
Theta Probe Technology

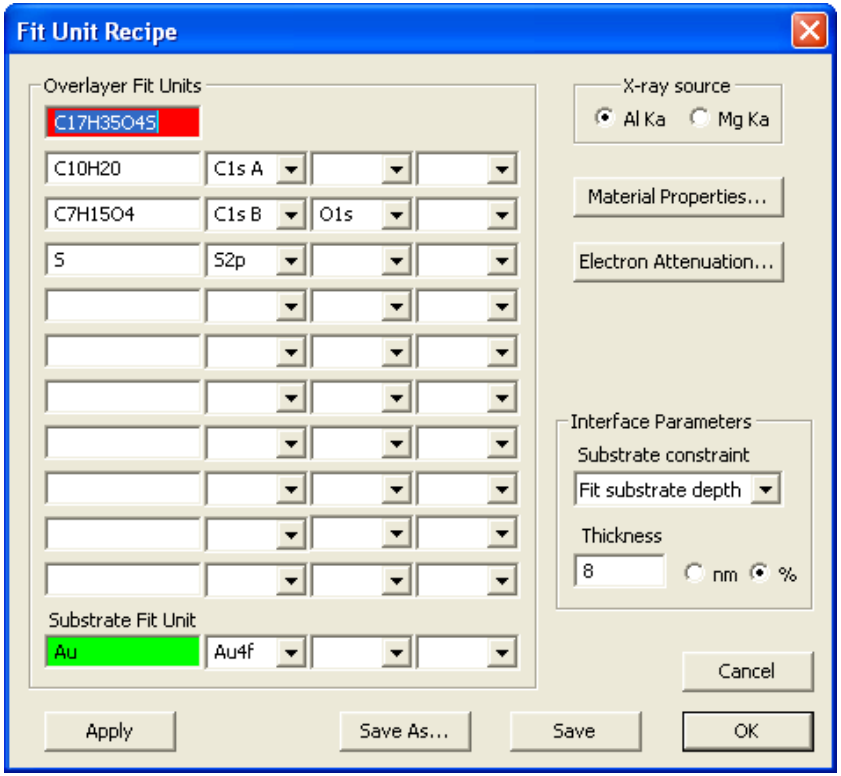


ThermoFisher SCIENTIFIC

ARXPS Suite

- Maximum entropy calculations
- Non-destructive profiles from ARXPS
- Fit with chemical units
- Recipe mode

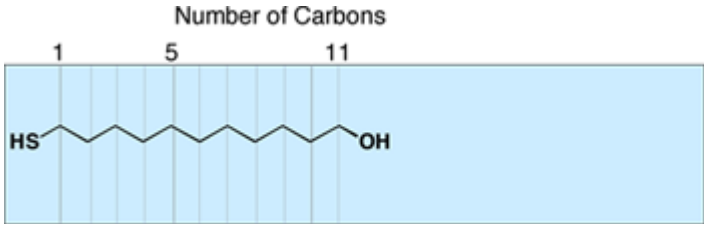


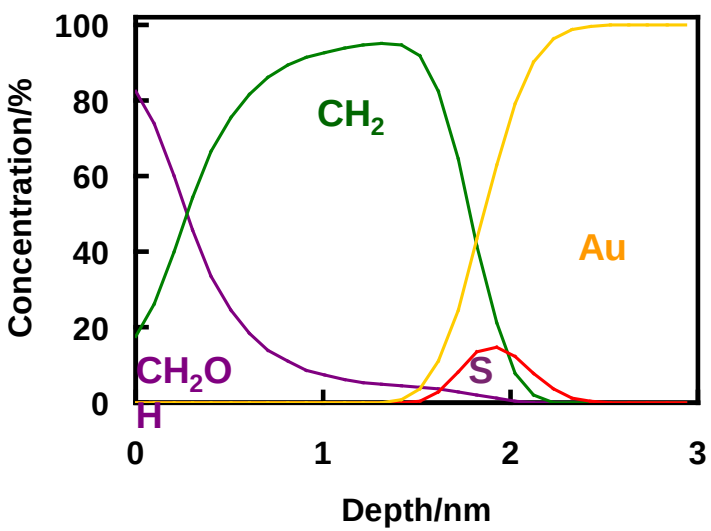


ThermoFisher SCIENTIFIC

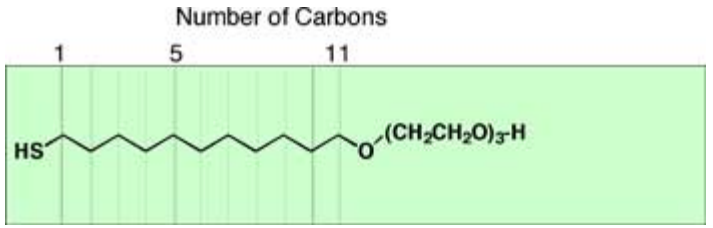
ARXPS Suite

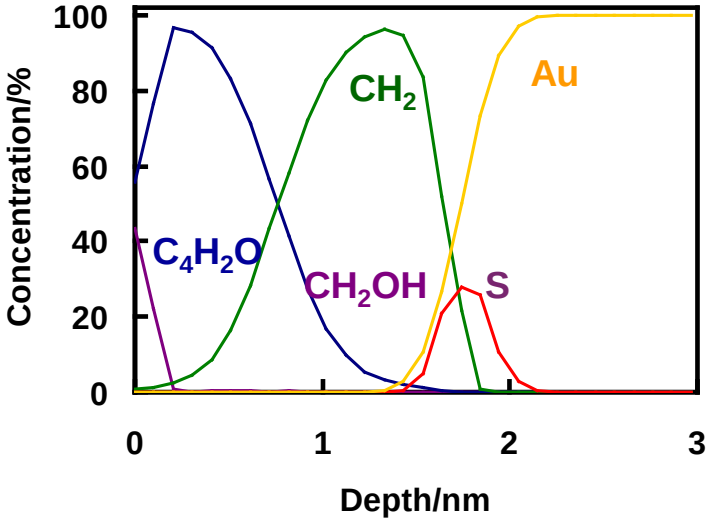
Hydroxy Undecanethiol





1-Mercapto-11-undecyl-tri(ethylene glycol)





- Samples and images provided by Asemblon Inc.

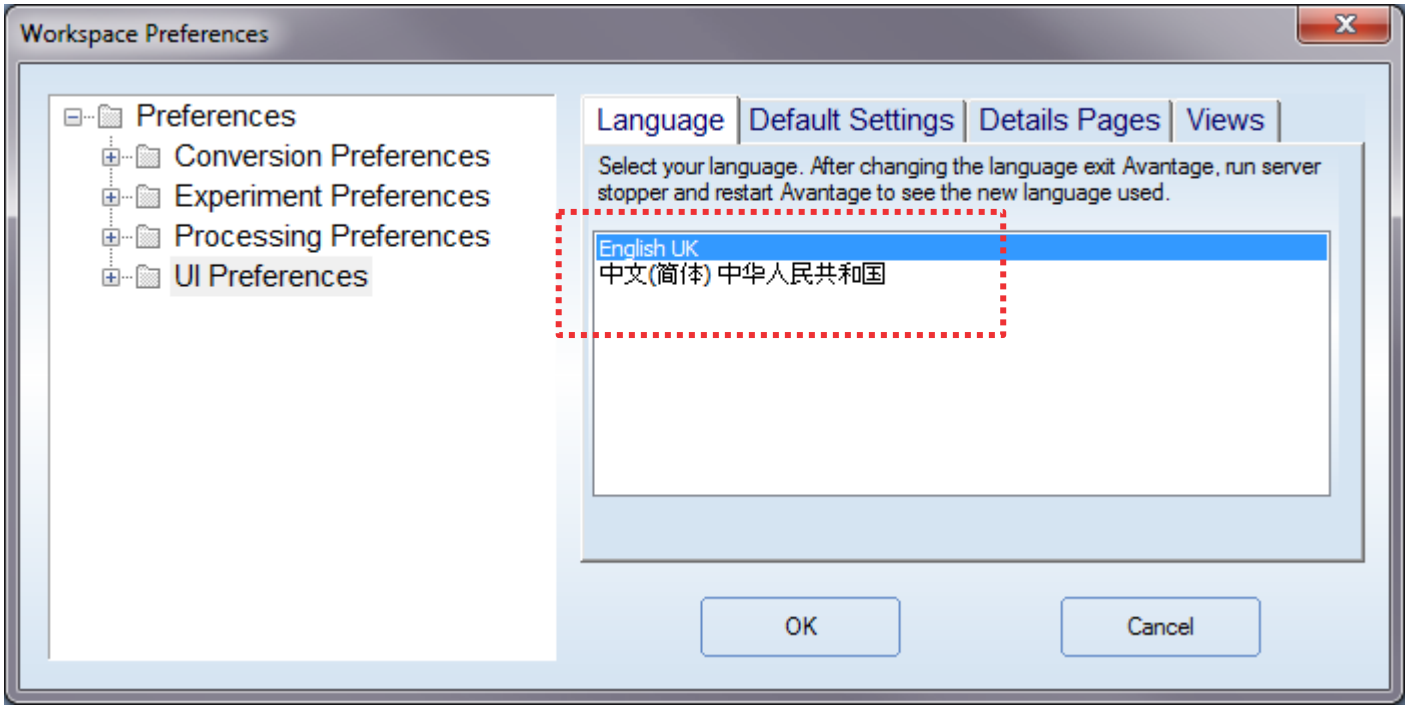
Upcoming Features

Multiple Language Support

Language Support

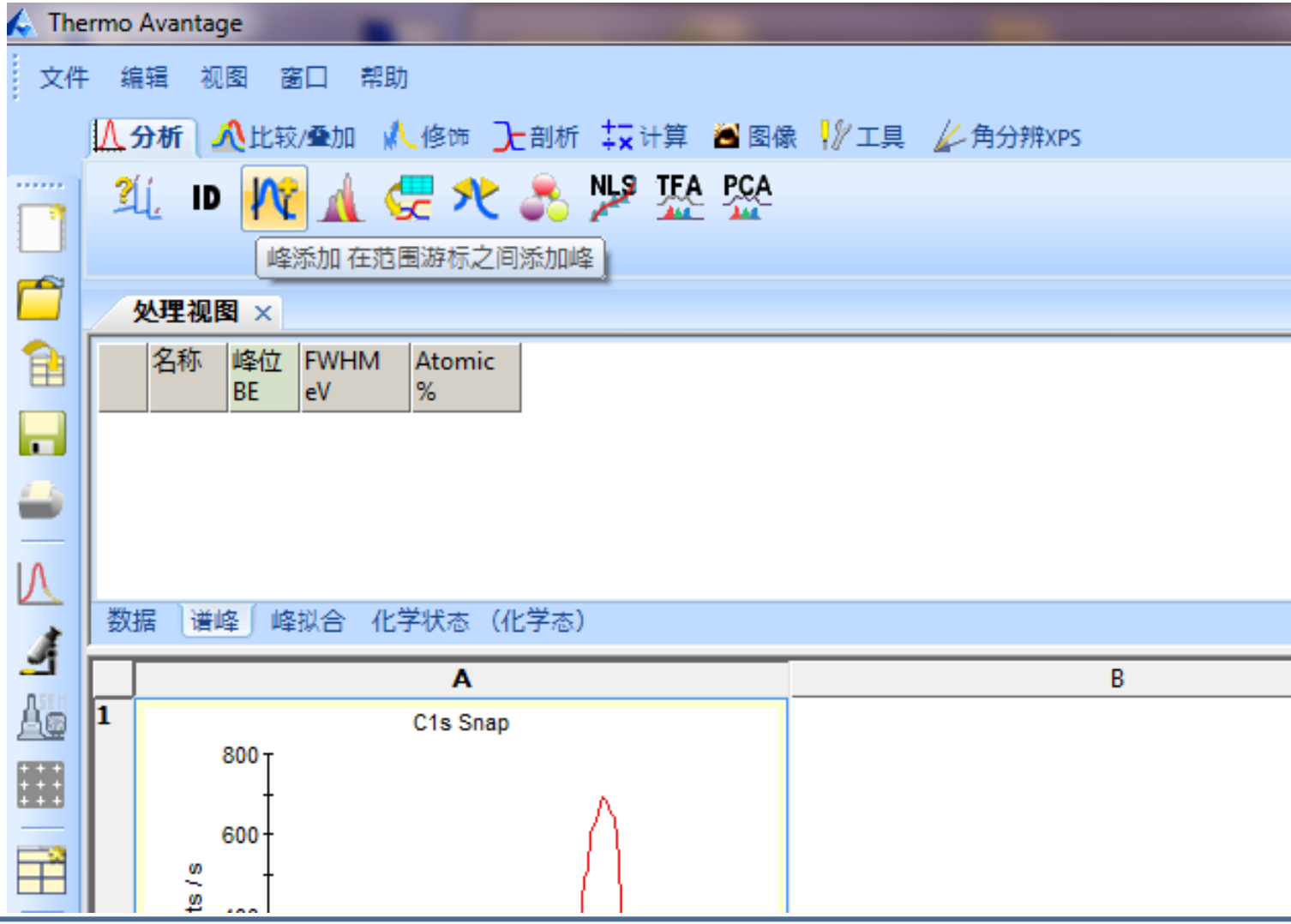
- Avantage v1 – v5 released in English Language only

- Coming soon – multiple language support



Chinese Language

- All text in the user interface can be translated



ThermoFisher SCIENTIFIC

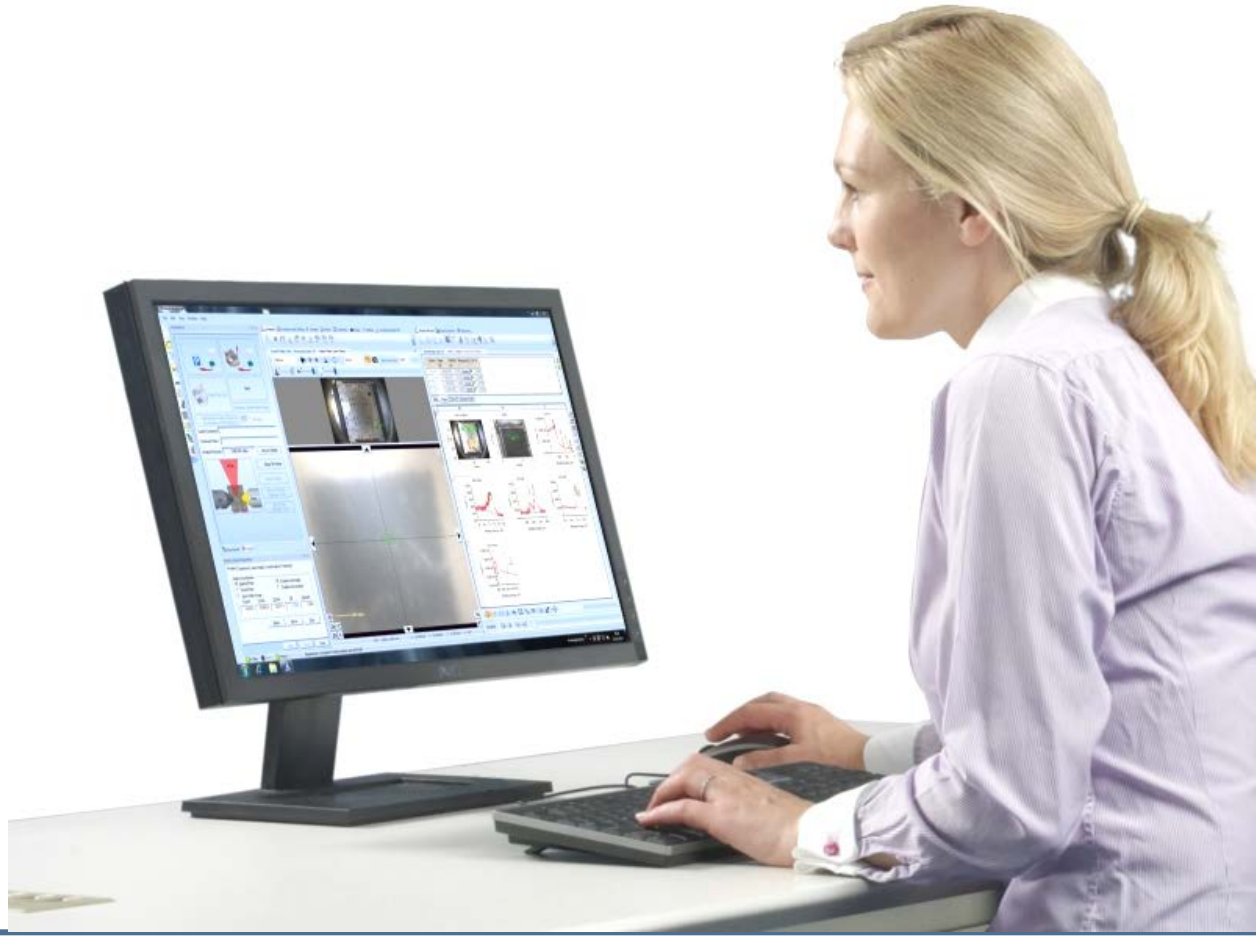
Chinese Language

- Translations currently in progress
- Shou Lin and Albert Ge are managing this

	A	B
331	Data must be an AR Normalised or Atomic Percent type profile	数据必须是AR归一化或原子百分比型分布
332	The sum of the limits must exceed 100%	极限的数额必须超过100%
333	The data has no O1s peak	数据没有O1s谱峰
334	The special processing selected is incompatible with this data	选定的特殊处理与该数据不兼容
335	Thread Did Not Terminate.\nClose and restart application	线程未终止。\n关闭并重启应用程序
336	Ready	就绪
337	Running	运行
338	Waiting to Stop	等待停止
339	Stopped	已停止
340	Stop Failed	停止失败
341	Fitting stoichiometry	拟合化学计量比
342	Solving(%d)	解决(%d)

Summary

- Avantage is the complete XPS software package
  - Design & run complex experiments
  - Interpret and process all types of XPS data
    - Images
    - Depth Profiles
    - ARXPS
    - Point analyses
  - Automate your workflow
    - Full auto-analysis
    - Batch process recipe mode
  - Report your data
    - MS Office application
    - Data export



Thank You For Listening!

Any Questions?