



Nexsa Surface Analysis System Operator Manual

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Introduction

This manual provides the basic introduction to operation of the Nexsa XPS instrument. For additional information, see the *Avantage* on line help and electronic manuals.



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Avantage Introduction

Each part of the software is described in the relevant section in this manual. Further information can be found in the *Avantage* help file, in the **Help** menu.

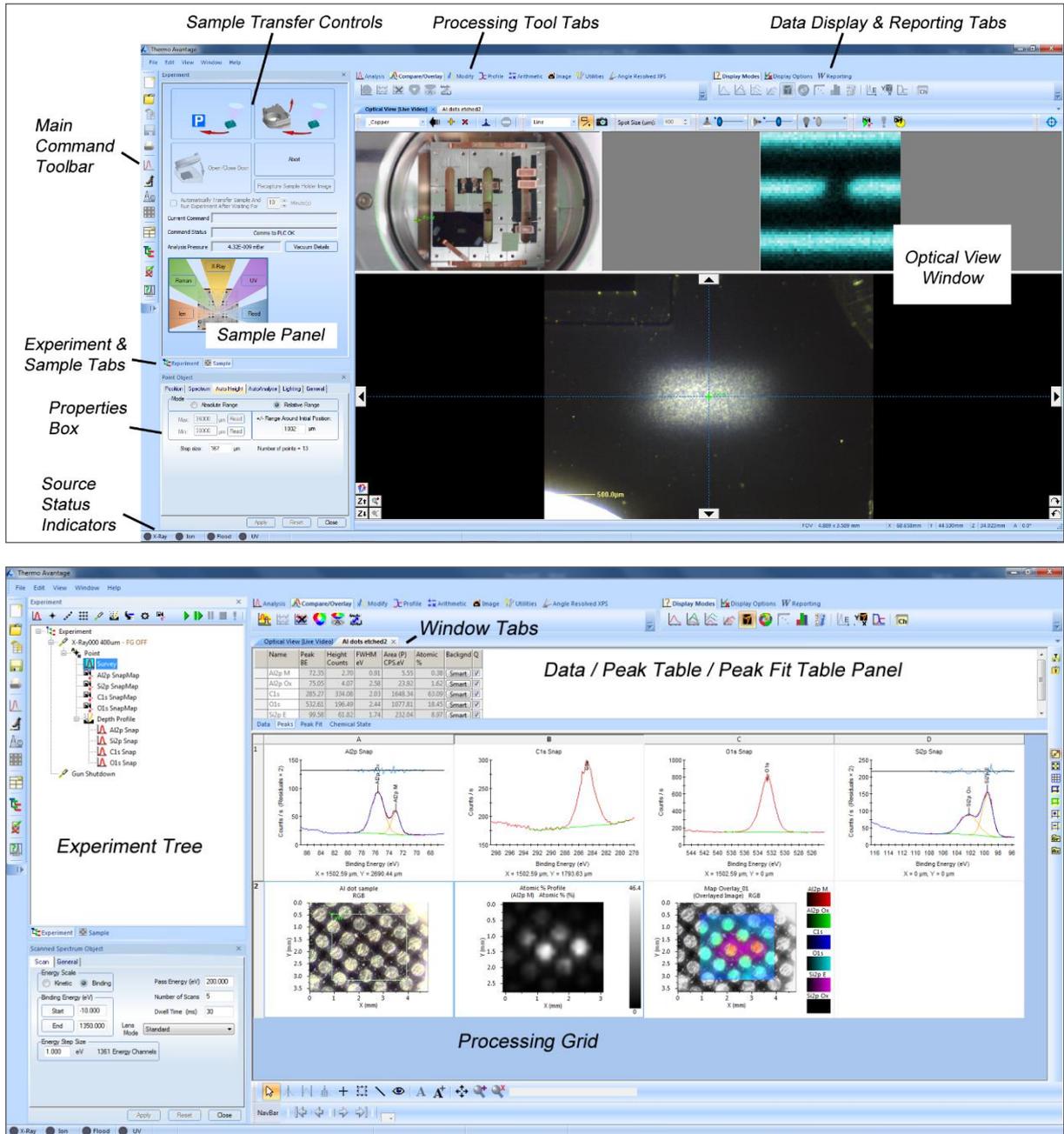


Figure 1: Main Screen Layouts

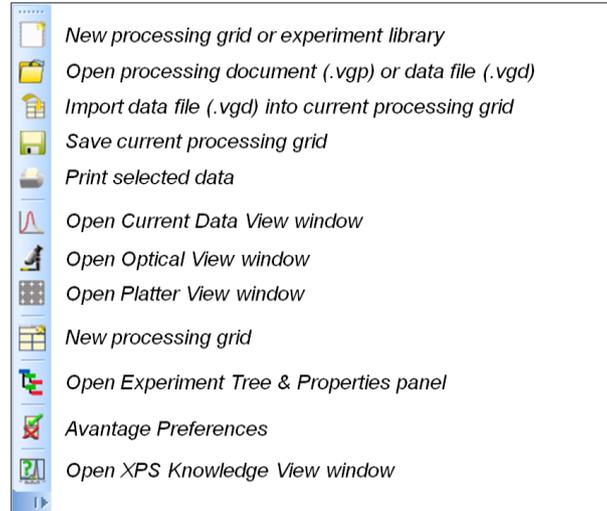


Figure 2: Main Command Menu Toolbar Icons

Status Indicators

The status of the sources and the sample bias is shown by the indicators at the bottom left of the *Avantage* screen.

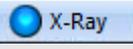
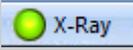
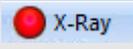
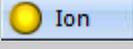
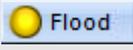
Indicators	Description
	No colour and a grey text label indicates that the gun is off or the sample bias is not being applied.
	Flashing blue indicates that a source is ramping, or running a degas/conditioning routine.
	Green indicates that a source is on and running (In the case of the ion gun – unblanked and etching) (For sample bias – bias is being applied).
	A red indicator indicates no communications to source hardware. See “troubleshooting” section
	Ion gun only: Yellow indicates source is on and blanked (not etching)
	Flood gun only: Yellow indicates filament conditioning

Table 1: Status Indicators

System Status Panel

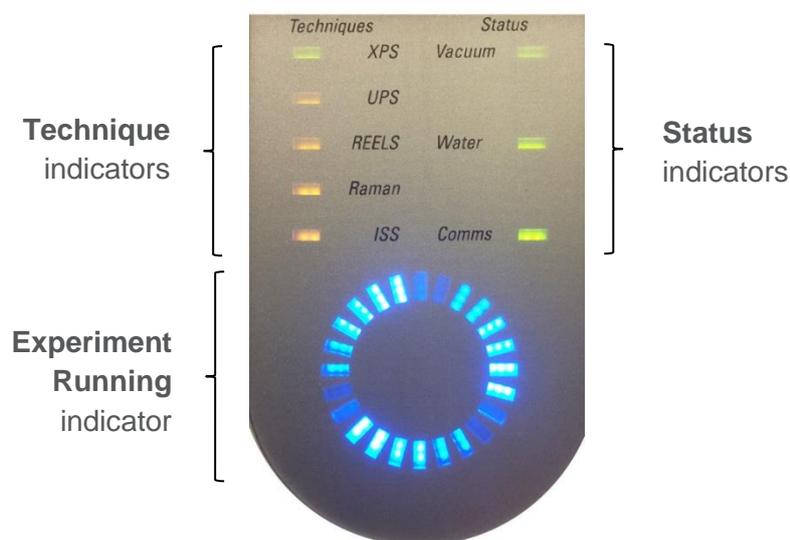


Figure 3: System Status Panel

The instrument status panel indicates the current condition of the instrument.

Technique indicators	An orange indicator shows that a technique is available on the system. A green indicator shows which technique is currently being used to acquire data. If a technique is not installed, the indicator is not lit.
Status indicators	These indicate the instrument status. A green light shows that the status is OK, an orange LED indicates that the system is not OK for data acquisition. Vacuum indicates if the vacuum level is OK to start the sources. Water indicates that the water flow to the instrument is good enough to allow the X-ray source to be used. Comms indicates that <i>Avantage</i> is open and communicating with the instrument
Experiment running	A blue light scans when the system is acquiring data.

Table 2: System Status Panel Description

Sample Holders and Sample Mounting

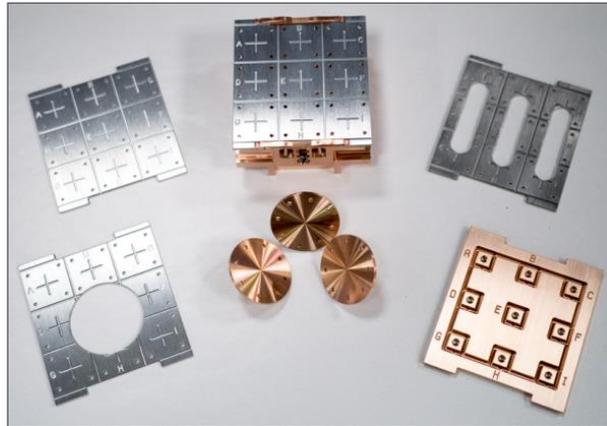


Figure 4: Sample Holders

- Always use gloves and tweezers to avoid contamination of the sample surface. Try to avoid touching areas that you wish to analyse.
- Samples are usually mounted onto a standard sample holder using the spring clips provided.

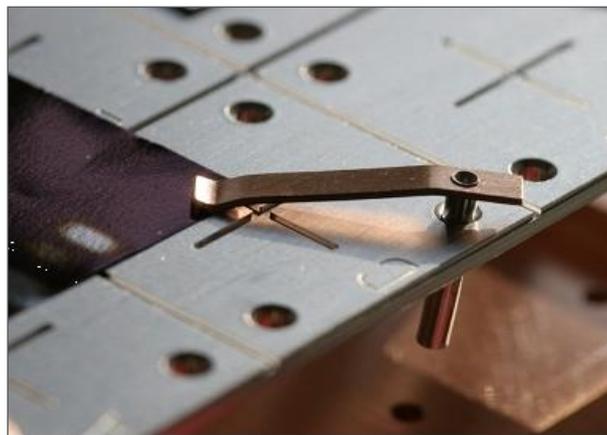


Figure 5: Spring Clip

- Alternatively, conducting carbon adhesive tape may be used.
- Samples must not go outside the area of the top plate (must not overhang the edges of the holder). This is to prevent samples hitting surfaces inside the system during transfer, which could cause internal damage to the transfer mechanism.

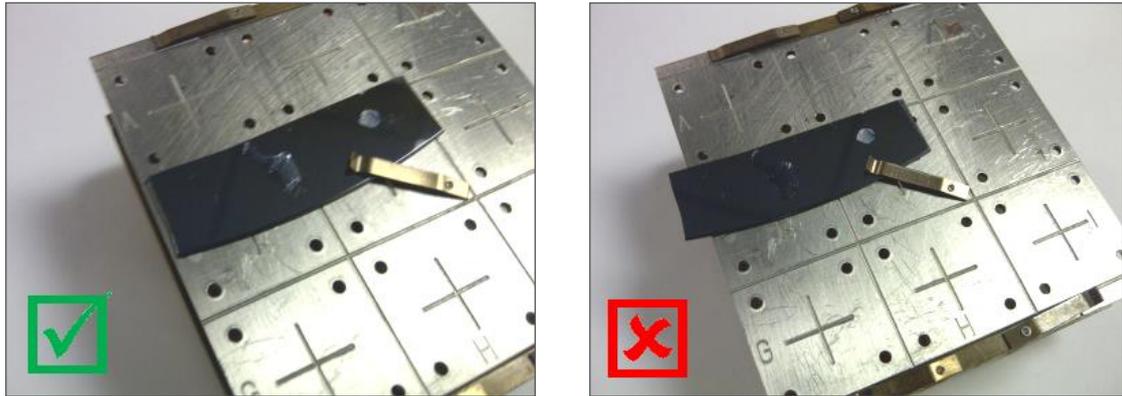


Figure 6: Good Sample Mounting (Left) and Bad Sample Mounting (Right)

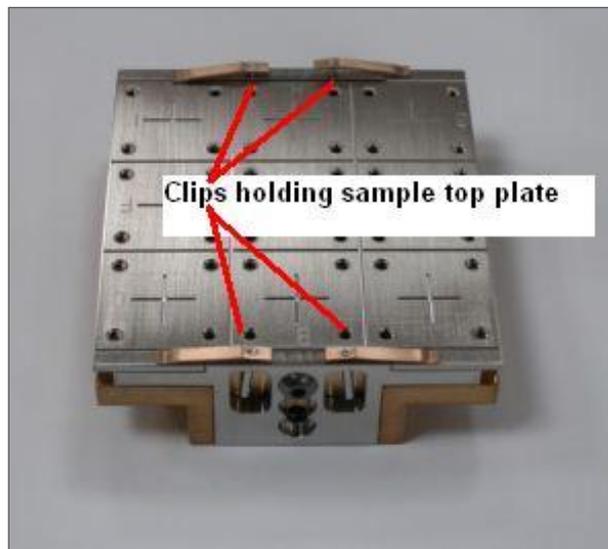


Figure 7: Clips Holding the Sample Top Plate

- The top plate can be replaced by removing the four clips at the edge of the holder. The top plate can then be lifted out.

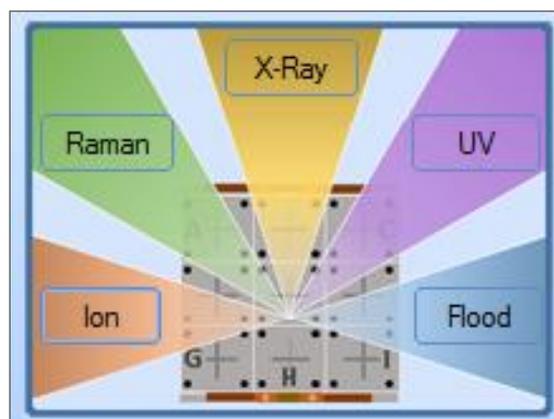


Figure 8: Source orientation around the sample holder
(N.B. The diagram displayed is determined by the options fitted)

- Care should be taken where different samples of different thickness are to be analysed to avoid shadowing of the X-ray, ion or flood gun by the thicker samples. The diagram above shows the source orientation as seen by the system cameras.
- Depending on the sample thickness, the height of the top plate may need adjusting; this is done using the two screws on either side of the sample holder. The table below shows the screw positions and the sample thickness that can be analysed. Note that only samples of a thickness within the range can be analysed at a particular setting. For example, if the top plate is set to the middle position, for thicknesses of 5-10 mm, then samples of 0-5 mm would have to be attached to supports to bring them into the 5-10 mm thickness range.

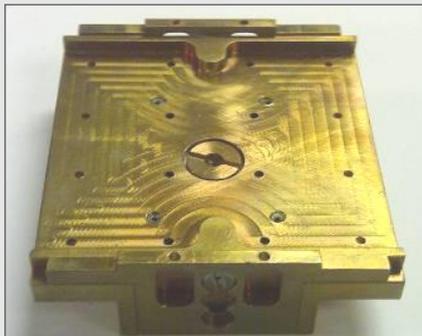
Screw Position	Image	Sample Thickness Range (mm)
Lowest position (default)		0-5
Middle position		5-10
Top position		10-15
Top plate removed		15-20

Table 3: Sample Thickness and Screw Position

Rotation Holder

To set up the holder for rotation depth profiling, first remove the top plate from the holder as described above. Choose the correct rotation stub depending on the sample thickness (largest stub – 0-5 mm sample thickness, medium stub – 5-10 mm sample thickness, smallest stub – 10-15 mm sample thickness).

The stub screws into the hole in the base of the sample holder; it is best to hold the gear underneath the holder still whilst doing this to ensure that the stub is fixed tightly in position.

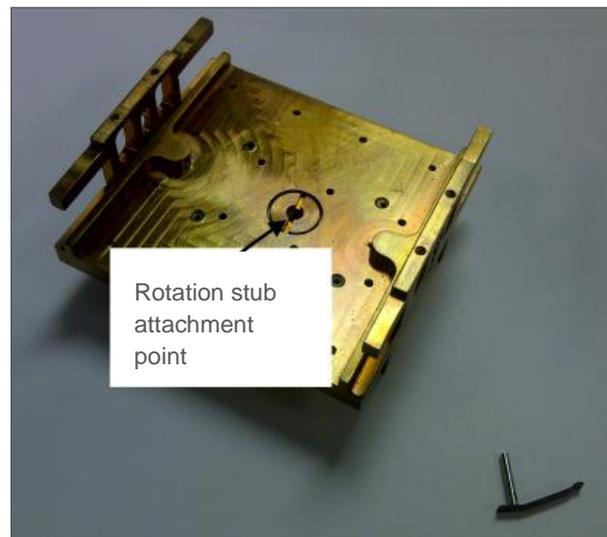


Figure 9: Rotation Stub Attachment Point

The optional top plate with a hole can be used to mount additional samples when a rotation stub is used. Remember that the height of the top plate may need adjusting down to prevent the samples being shadowed if the medium or small stubs are used.



Figure 10: Rotation Holder

Powder Holder

Powders can be mounted either by pressing onto tape and using the standard top plate, or by pressing into the recesses on the powder top plate. To use the powder top plate, first remove the top plate from the holder as described above, and then attach the powder top plate using the four spring clips.

Powder should be pressed into a recess, ensuring that the powder surface is level with the surface of the top plate. Care should be taken to minimize the amount of loose powder on the holder, as this can become mobile in the system during pump down.



Figure 11: Powder Holder

Fibre Holder

Fibres can be mounted using the supplied top plate with channels. To use the fibre top plate, first remove the top plate from the holder as described above, and then attach the fibre top plate using the four spring clips. Fibres should be stretched horizontally across the channels and either clipped or taped into place. This ensures that the XPS signal is just from the fibre, and there is no contribution from the sample holder. It also allows large X-ray spot sizes to be used, to maximise the signal.

Care should be taken to ensure that no fibre overhangs the sample holder, which could get caught during the sample loading process.

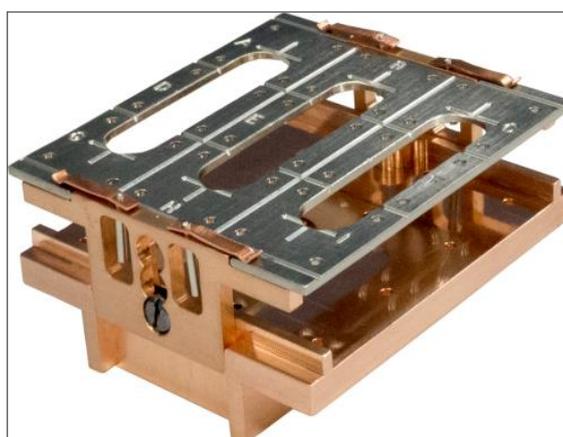


Figure 12: Fibre Holder

Additional Sample Holders

Details of the optional sample holders can be found in the section "[Optional Sample Holders](#)" (Page 124).

Sample Loading

Before loading samples into the system always check that the maximum height of the samples is suitable by checking with the loading gauge arm. It should move across the sample holder without touching the top of the samples.

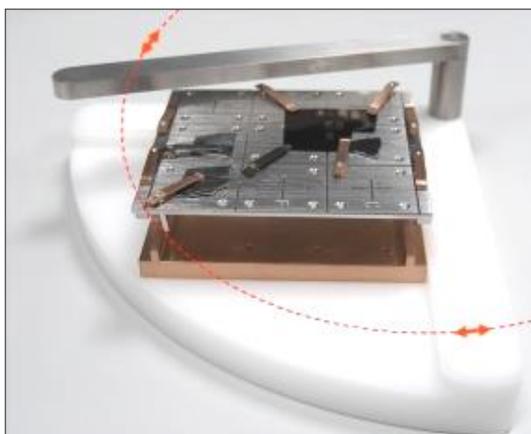


Figure 13: Checking Maximum Height

CAUTION

Do not load samples that are touched by the loading test gauge. Do not attempt to load samples that are outside the edges of the top plate

The sample is loaded onto the plate in the entry-lock as shown, ensuring that the two holes on the bottom of the holder (white arrows) engage correctly with the pins on the carrier plate (black arrows).

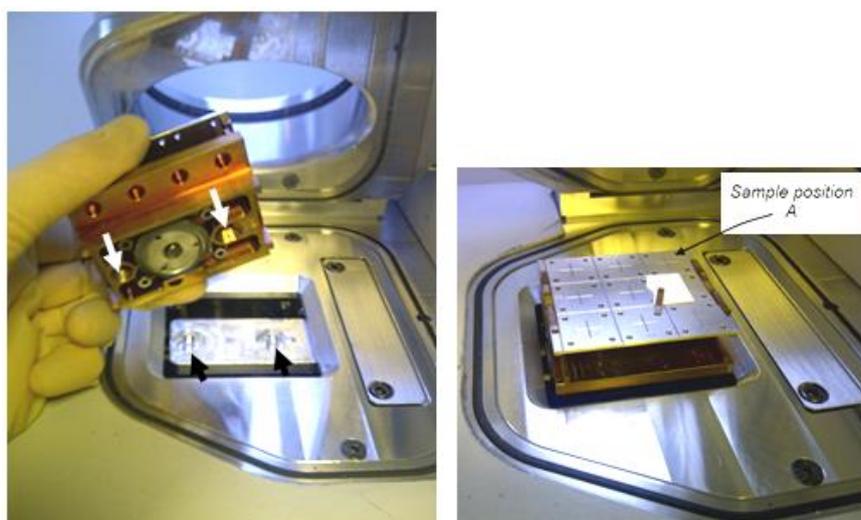
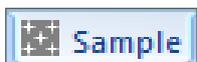


Figure 14: Loading a sample

The Sample Page

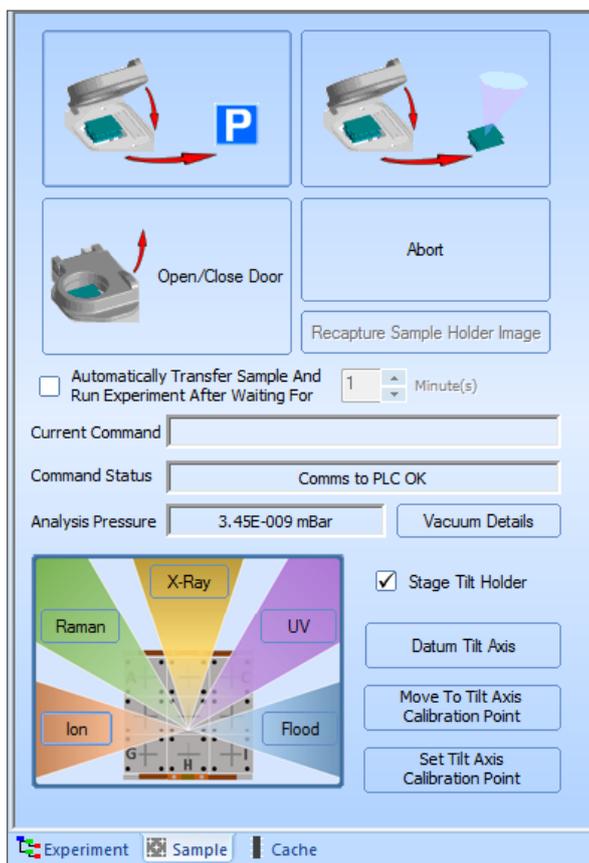


The **Sample** page is used to load and unload samples. It also provides buttons to access the Engineer Details Pages for Vacuum Control, X-ray, Ion and Flood Guns, and displays the pressure in the analysis chamber.

A diagram indicates the orientation of the fitted sources (X-ray, Ion source & Flood Gun, plus optional UV lamp and Raman system) relative to the sample holder and provides access to the source details windows (for engineer use only). If the sample tilt module has been purchased, the check-box control to inform the system that it is loaded should also be displayed (please see the Optional Sample Holders section, page 124, for more information about the tilt module).

PLC Command
Status & Analysis
Chamber Pressure

Source
Arrangement &
Open Details
controls



Sample handling
controls

Open Vacuum
Details

Tilt Module
Controls*

Figure 15: Description of the Sample Tab
(* Tilt Module Only)

Sample Handling Controls

The buttons at the top of the sample page control the movement of the sample in and out of the analysis chamber, and allow the entry-lock to be vented. The buttons are context sensitive, and the control displayed depends on the location of the sample holder.

When the entry lock is vented the following buttons are enabled:

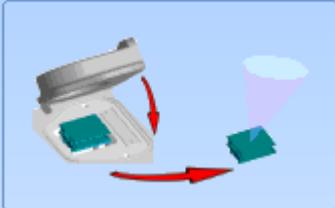
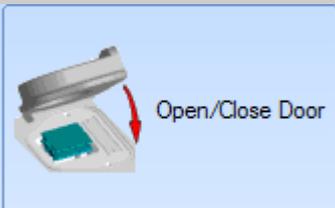
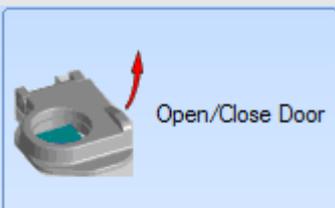
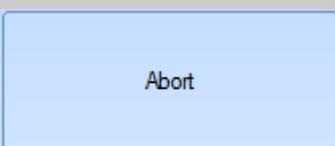
	Pump and transfer to the analysis position.
	Pump and park in the entry lock. The park position may be useful for allowing samples to outgas for a longer period.
	Close the entry-lock door. This does not start the entry-lock pump. It is recommended to use this button when changing samples to keep the chamber and door o-ring seal clean. (Visible if door is open)
	Open the entry-lock door. (Visible if door is closed)
	Abort the current operation. Depending on the process running this may take a few minutes to complete.

Table 4: Buttons Enabled when Venting the Entry Lock

The entry-lock chamber pressure is monitored, and the sample will not be transferred into the analysis chamber until the pressure is below a threshold level.

When the entry lock is pumped down, and the sample holder is in the park position the following buttons are enabled:

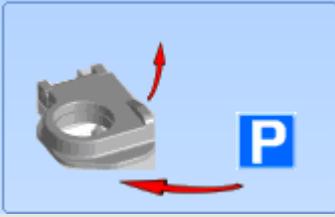
	<p>Transfer to the analysis position. If the pressure in the entry-lock is not below the threshold level, then the system will wait until the vacuum is good enough.</p>
	<p>Vent the entry-lock and open the door</p>
<p>Recapture Sample Holder Image</p>	<p>Collect a new platter view image.</p>
<p>Abort</p>	<p>Abort the current operation. Depending on the process running this may take a few minutes to complete.</p>

Table 5: Buttons Enabled when the Entry Lock is Pumped Down

When the sample holder is in the analysis position the buttons are enabled:

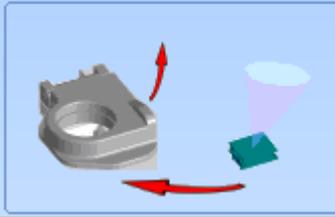
	<p>Transfer to the entry lock, vent to atmosphere and open the door.</p>
	<p>Transfer to the entry lock and park the sample block.</p>
<p>Abort</p>	<p>Abort the current operation. Depending on the process running this may take a few minutes to complete.</p>

Table 6: Buttons Enabled when Sample Holder is in the Analysis Position

When any transfer process is completed, the buttons are re-enabled. The entire vacuum and transfer sequences are automated. After transfer to the analysis position, the sample holder is raised to approximately the normal analysis position (Z~35 mm).

PLC Command Status

When a vacuum sequence is in progress, the status and details of current command are displayed below the sample movement buttons. The **Current Command** window indicates the routine that is running; the **Command Status** window displays the step that is currently in progress.

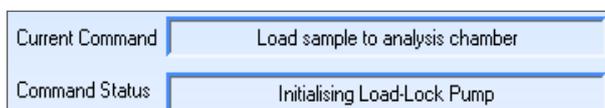


Figure 16: PLC Command Status

Automatic Running of the Experiment

A predefined experiment file can be run automatically after the sample has been transferred and a defined time has elapsed. This function is usually used with samples that are known to out-gas significantly during pump-down. To use this feature, first pump-down the samples using the **Pump and park in the entry lock** button. Click the checkbox to enable the wait, transfer and run experiment mode, and type the required wait time (in minutes) into the adjacent text box.

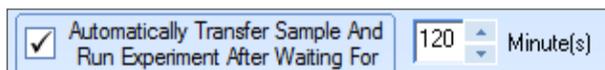


Figure 17: Automatic Running of the Experiment

Next define the experiment or load an existing experiment (see later sections in the manual for how to define experiments). Remember that points, lines or areas will have to be defined from the Platter View, and that auto-height should be used in the **absolute** mode. When the experiment is ready, press the **Transfer to the analysis position** button; a small window will open indicating the time until the transfer will be attempted.

If the pressure is not good enough at this time, the system will wait until the pressure is acceptable before loading the samples and running the experiment. This procedure is most suitable for large area analyses.

Details Pages

All users may view the Details pages but only users that have *Avantage* Engineer access level will be able to change settings. See ***Avantage Help* > Index > Add User** for further information about this restricted access.

Many of the operations on the Details pages are for engineers only, and in normal operation they are not required. Please see the Maintenance & Troubleshooting section, and the relevant Option sections for information on use of the details pages.

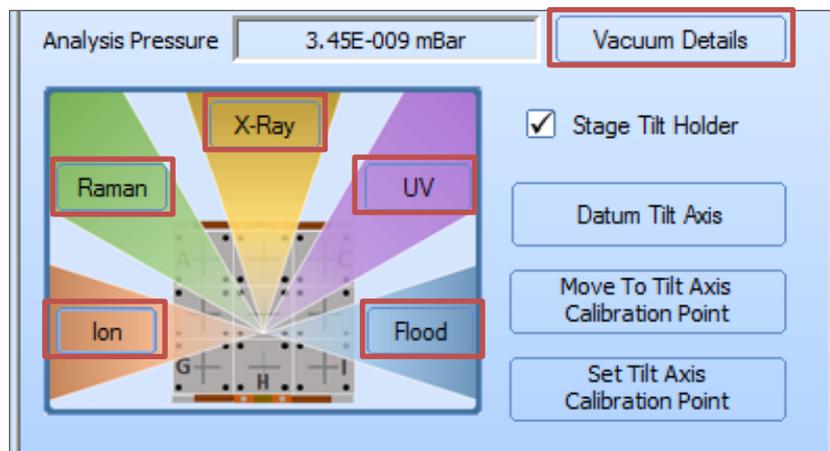


Figure 18: Buttons to open details windows (layout seen depends on the options fitted)

The Optical View: Sample Navigation and Lighting

The **Optical View** is used to navigate around the sample and select features for analysis by using the following:

Platter view

Photograph of the sample holder taken whilst it is in the load-lock during pump-down. Used to move from sample to sample, and to define experiment positions.

SnapMap

Rapid XPS mapping for feature identification & alignment.

Live optical view

Switchable between the through-the-lens (TTL) camera view and the height-set camera view. Used to move the stage, and to define experiment positions. The stage can only be moved up and down (along the Z-axis) when the height-set view is displayed.

Lighting controls

Used to control the three light sources on the system

X-ray spot size

Control to adjust the X-ray spot size of a selected point, line, or area.

Experiment insert controls

Used to insert points, lines, areas, depth profile experiments and auto-analysis experiments.

Defined positions

Allows quick movement to the standard positions for calibration, or user defined positions.

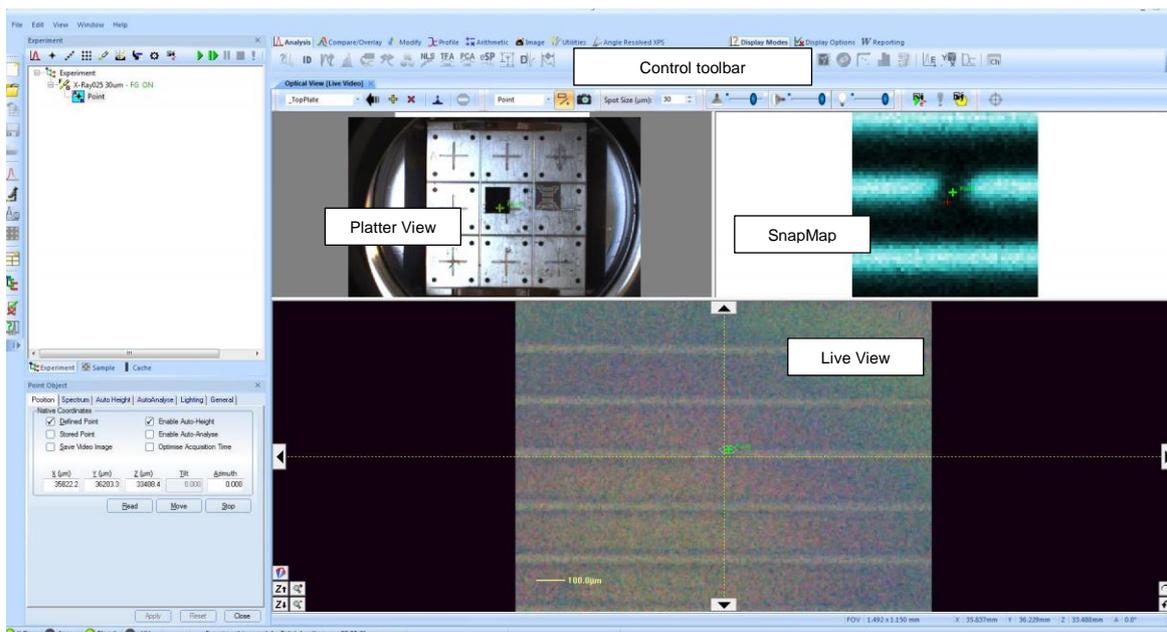


Figure 19: Optical View Window



The **Optical View** window is started from the toolbar button or from the main *Avantage* menu **Window > Open > Optical View**.

Light Controls

There are two in-system light controls, one for the **co-axial** light in the Reflex camera view, and one mounted at an off-axis angle on the chamber (**side light**). Lights can be used individually or in combination. The third control adjusts the bench light above the entry lock door. (the system internal lights – “cladding lights” – are controlled through the vacuum details page, see Figure 67).

Different types of sample may benefit from adjusting the lighting as shown below, generally flat samples are better with the co-axial light, and rough samples work better with the side light.

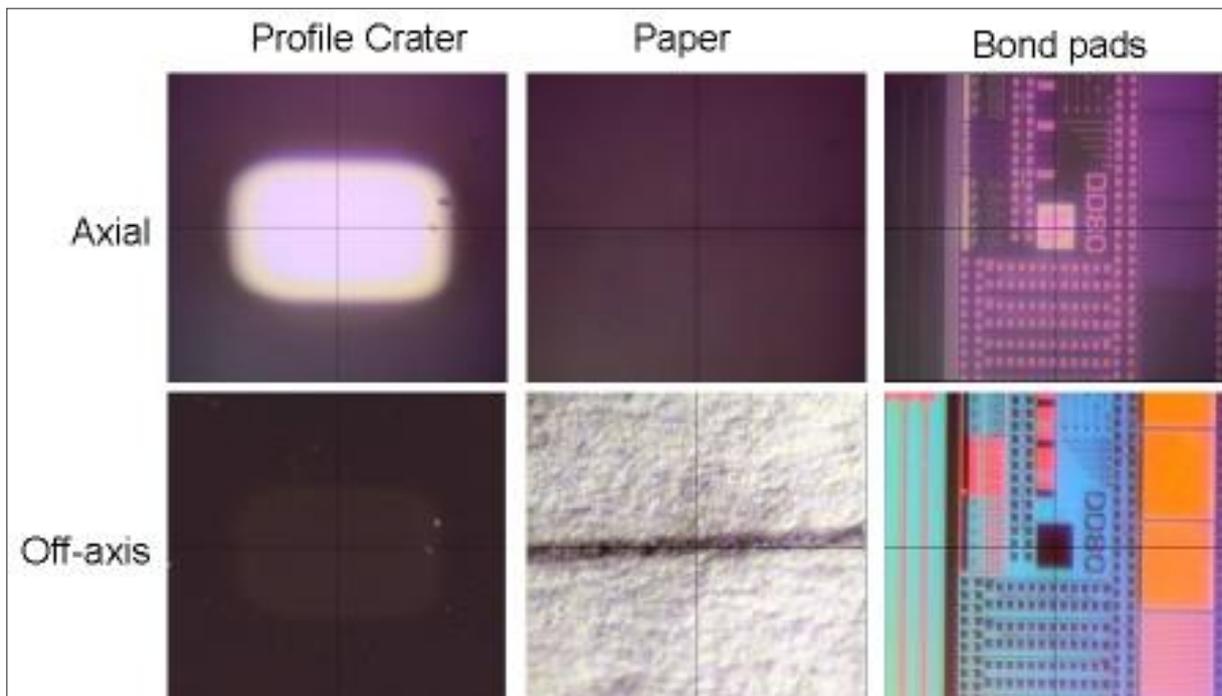


Figure 20: Different Samples

Each light has an On/Off button (the lamp icon on the left) and a level control.



Sample Navigation

- Double-click with the left mouse button on a feature in either the platter view, SnapMap or the live optical view (TTL view only) to move to that feature.
- The buttons around the image can also be used to move the sample in X, Y and Z directions and also Rotation or Tilt on applicable sample holders.
- The digital zoom facility allows the field of view to be selected to help when viewing small features
- The camera view can be switched to the height setting camera using the red and blue icon in the lower left corner of the live optical view.

SnapMap (Optical View)

SnapMap in the optical view is used to collect a total counts XPS image at a specified binding energy for feature visualisation and alignment. SnapMaps can also be collected as part of an experiment, which allows the data to be processed and used analytically, and is described later in this manual. SnapMap is controlled from the SnapMap section of the optical view toolbar.



Run SnapMap. Collects an image using the current settings, centred on the current position in the Live View window



Abort SnapMap. Stops acquisition of a SnapMap.



Open SnapMap settings. Used to configure the acquisition parameters (peak position, scan size, scan mode).

SnapMap Settings

Enter centre energy or pick element from the periodic table

Energy Scale: Kinetic Binding

Map Size in mm: 1.0

Image Quality: Coarse Fine

Centre Energy: 534.384

Image Resolution: 50x50 100x100

Flood Gun: Turn On

H																	He
Li	Be											B	C	N	O	F	Ne
Na	Mg											Al	Si	P	S	Cl	Ar
K	Ca	Sc	Ti	V	Cr	Mn	Fe	Co	Ni	Cu	Zn	Ga	Ge	As	Se	Br	Kr
Rb	Sr	Y	Zr	Nb	Mo	Tc	Ru	Rh	Pd	Ag	Cd	In	Sn	Sb	Te	I	Xe
Cs	Ba	**	Hf	Ta	W	Re	Os	Ir	Pt	Au	Hg	Tl	Pb	Bi	Po	At	Rn
Fr	Ra	**	Rf	Db	Sg	Bh	Hs	Mt	Ds	Rg	Cn						
La	Ce	Pr	Nd	Pm	Sm	Eu	Gd	Tb	Dy	Ho	Er	Tm	Yb	Lu			
Ac	Th	Pa	U	Np	Pu	Am	Cm	Bk	Cf	Es	Fm	Md	No	Lr			

OK Cancel

Figure 21: SnapMap settings window

Before running a SnapMap, the acquisition parameters need to be set. Open the SnapMap settings from the control toolbar. The centre energy position for the SnapShot spectrum acquired during the SnapMap needs to be selected first, either by choosing an element from the periodic table, or typing a value into the Centre Energy dialog box. The map size (0.1, 0.25, 0.5, 1, 2, or 3 mm²) and the pixel resolution (50 x 50 or 100 x 100) should then be set. The image quality determines the speed of acquisition. “Coarse” mode uses an “S” raster with no backlash compensation, which is fast, but may give a noisier image than “Fine” mode, which uses a fly-back raster and has backlash compensation enabled. Finally, the check box should be set for the flood gun.

SnapMap acquisition is started by pressing the “Run SnapMap” button. The centre of the SnapMap is defined by the centre position in the Live View window. Once the SnapMap is acquired, it can be used to define experiment positions in exactly the same way as the other views in the Optical View window.

Acquisition can be aborted using the Abort SnapMap button.

Saving the Optical Image

- Click right in the **Optical View window** you wish to save and select **Save Image**.
- The image may be saved in several standard formats or as an Avantage (.vgd) data file for further viewing and processing within Avantage. When saved as .vgd format, the field of view information is also saved.
- It is also possible to automatically save an optical image at any defined **Point** during an **Experiment**. Check the **Save Video Image** in the **Point** property page (see **Figure 23**).

Defining Named Points

The **Optical View** allows multiple sample stage coordinates to be given a name and stored for future use, independent of an Experiment. This can be useful when analysing a number of areas on a sample, or when a number of different samples are to be analysed.

NOTE:

Standard Test Sample positions start with an underscore (e.g. *_Silver*) and users should not create new names beginning with an underscore. Names starting with underscore character can only be edited by users with Avantage Engineer access.



Figure 22: Named Points Controls

Adding Named Points

- Move the stage to the desired position.
- Type a new name in the list box.
- Press the  control to add the new point.

Moving to a Named Point

- Select the named point from the list box
- Press the  control.
- Whilst the sample is moving, the stage can be stopped by pressing the  control
- A named point may also be selected in the Point Property page.

Deleting Named Points

- Select the named point that is to be deleted from the list box.
- Press the  control.

Optical View Properties Page

The **Optical View** has one **Property** page which is displayed by selecting **Properties** from the right click menu in the main display window.

This allows adjustment of the contrast, colour and brightness of the camera generating the displayed image but it is usually preferable to adjust the quality of the image by changing the setting of the light source.

The Experiment Tree & the Properties Panel

The **Experiment** page contains the experiment run buttons, the experiment tree (the area where the experiment objects are displayed), and the properties panel, where parameters for a highlighted experiment object can be viewed and edited. It is displayed by clicking on the  **Experiment** tab.

Generally **Experiment** objects are inserted from the optical view, as described in the section **Choosing an Analysis Position** (page 38), but the object insert buttons are also available for this purpose. The spectrum insert button is always used to add spectra to an experiment.

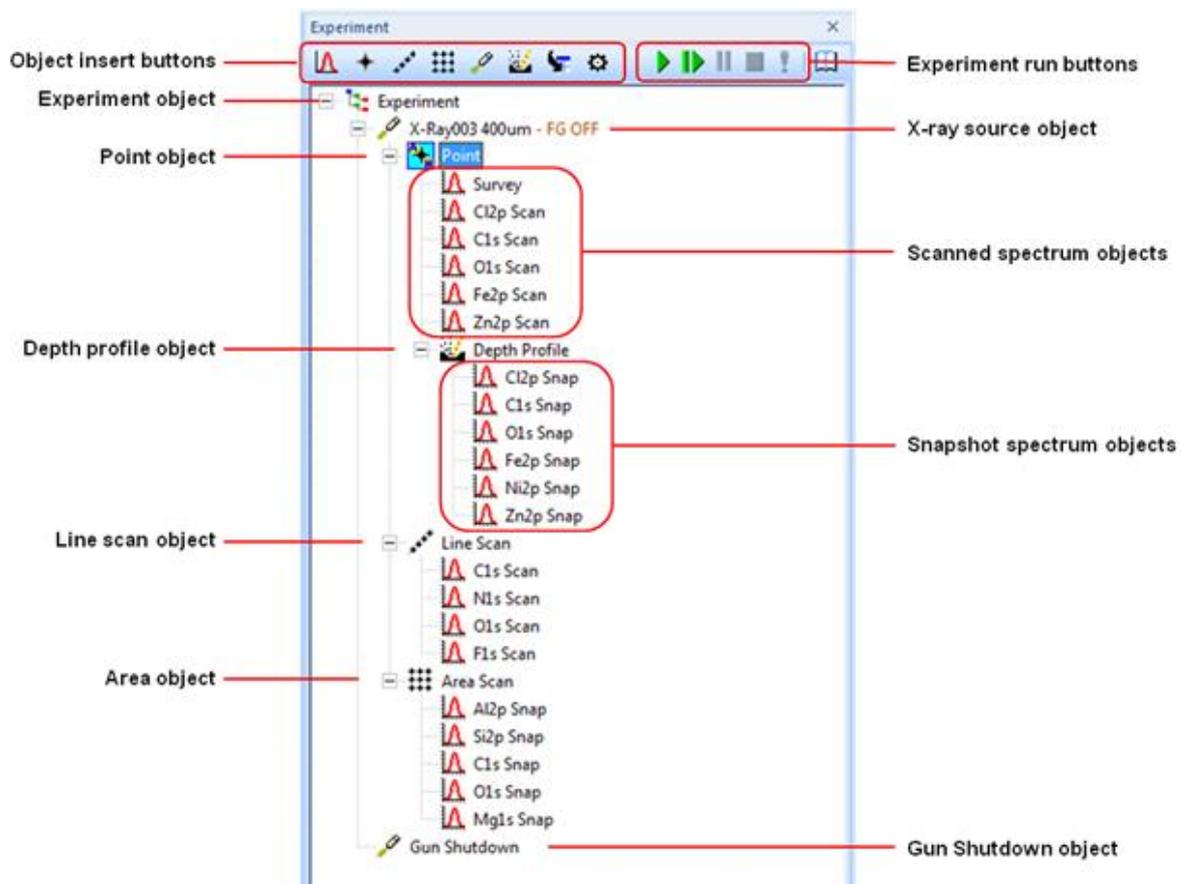


Figure 23: Experiment Panel

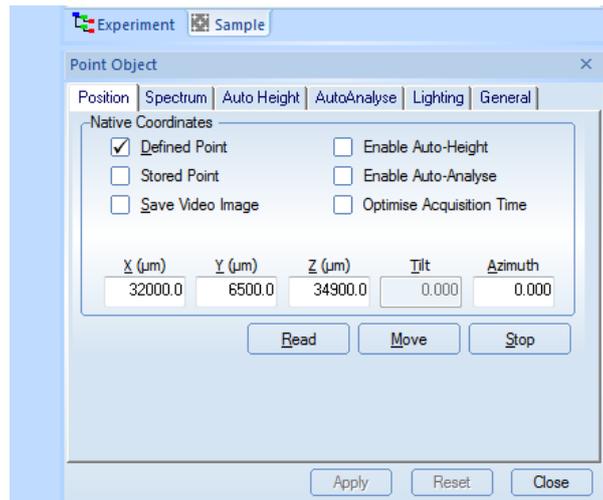
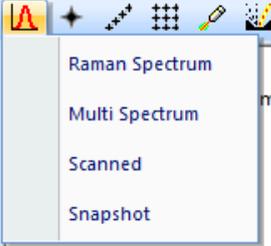
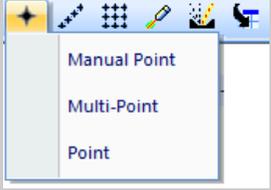
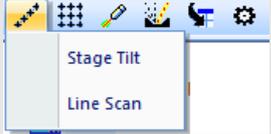


Figure 24: Object Properties Panel

	Run complete experiment
	Run experiment from selected step
	Pause experiment
	Stop experiment (at the end of current scan)
	Abort experiment (stop immediately)
	<p>Spectrum insert icon</p> <ul style="list-style-type: none"> • Multi Spectrum – open periodic table to insert regions, surveys, snapshots using default parameters • Scanned – insert scanned spectrum • Snapshot – insert snapshot spectrum • Raman Spectrum – insert Raman spectrum (available if iXR Raman option has been purchased)
	<p>Point insert icon</p> <ul style="list-style-type: none"> • Manual Point – undefined position • Multi-point – insert multi-point object • Point – insert point object
	<p>Line insert icon</p> <ul style="list-style-type: none"> • Stage Tilt – insert tilt object for ARXPS (needs tilt module option) • Line Scan – insert line scan object

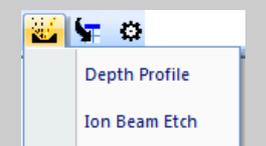
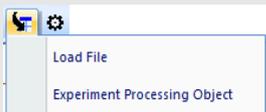
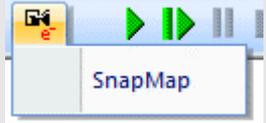
	<p>Area insert icon</p> <ul style="list-style-type: none"> • Area Control – insert area object • Circular Area Control – insert a circular area
	<p>Source insert icon</p> <ul style="list-style-type: none"> • X-ray Gun • Gun Shutdown – enables all sources to be turned off at the end of the experiment • REELS Source – available if REELS option purchased • ISS Source – available if ISS option purchased • UV Source – available if UPS option purchased • Manual Source Gun – mainly for legacy instruments
	<p>Ion Gun insert icon</p> <ul style="list-style-type: none"> • Depth Profile – for etching and acquisition • Ion Beam Etch – for etching only
	<p>Processing insert icon</p> <ul style="list-style-type: none"> • Load file – load a data file • Experiment Processing Object – allows addition of processing steps to the experiment (peak fitting, peak table profile, etc). See Avantage help for more details
	<p>Miscellaneous insert icon</p> <ul style="list-style-type: none"> • Group – group together objects for easy copying, management etc. • Instrument Iteration – engineer use only • Iteration – multi-level data collection, e.g. for time-resolved experiments. • User Message – pause the experiment to display a message • Wafer Registration – Theta300 system only • Wait Object – pause the experiment. This can be programmed to allow the TSP to fire (if required before a depth profile for example)
	<p>Image insert icon</p> <ul style="list-style-type: none"> • SnapMap – insert default SnapMap object

Table 7: Experiment Controls

Choosing an Analysis Position

Moving the Stage

The stage can be moved to a position of interest by double-clicking on either the platter view or the TTL view. Fine positioning should be done from the TTL view. Align the cross-hairs with the feature of interest. This sets the X & Y co-ordinates. Use the Z+ and Z- buttons to bring the surface into focus.

Getting the sample at the correct height is crucial to ensure that the sources and analyser are pointing at the correct feature. There are two ways of doing this, automatically and manually.

Setting the Sample Height: Auto-Height for Point Analysis

For large area analysis on larger uniform samples e.g. > 5 mm in size, using the 400 μm X-ray spot, it is recommended to use the **Auto Height** option in the **Point** object after the analysis position has been defined. This sets the sample position on the z-axis using the XPS signal at a particular energy (the default is 530 eV binding energy, as defined in the **Spectrum** tab).

There are two modes of operation of the auto-height option; **Full** or **Relative** range. **Full** mode measures the signal across all possible sample positions and is best used when points are defined whilst the samples are still in the load-lock. **Relative** mode uses the position defined when the point was inserted, and measures the XPS signal +/- a given value above and below this position. Good default values for these two modes are shown below. The default settings can be defined by accessing the **Stage Auto-Height** page in **Avantage preferences > Experiment Preferences**.



Figure 25: Auto Height Absolute Range

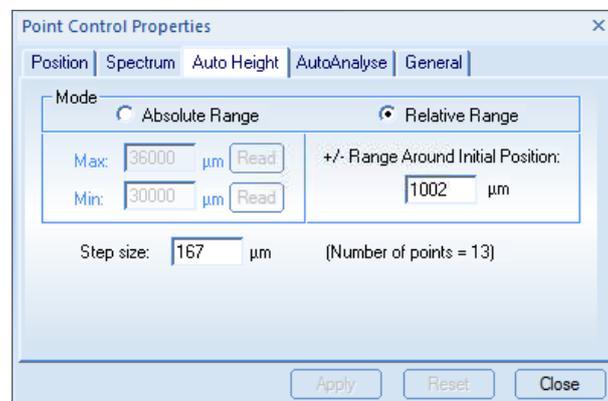


Figure 26: Auto Height Relative Range

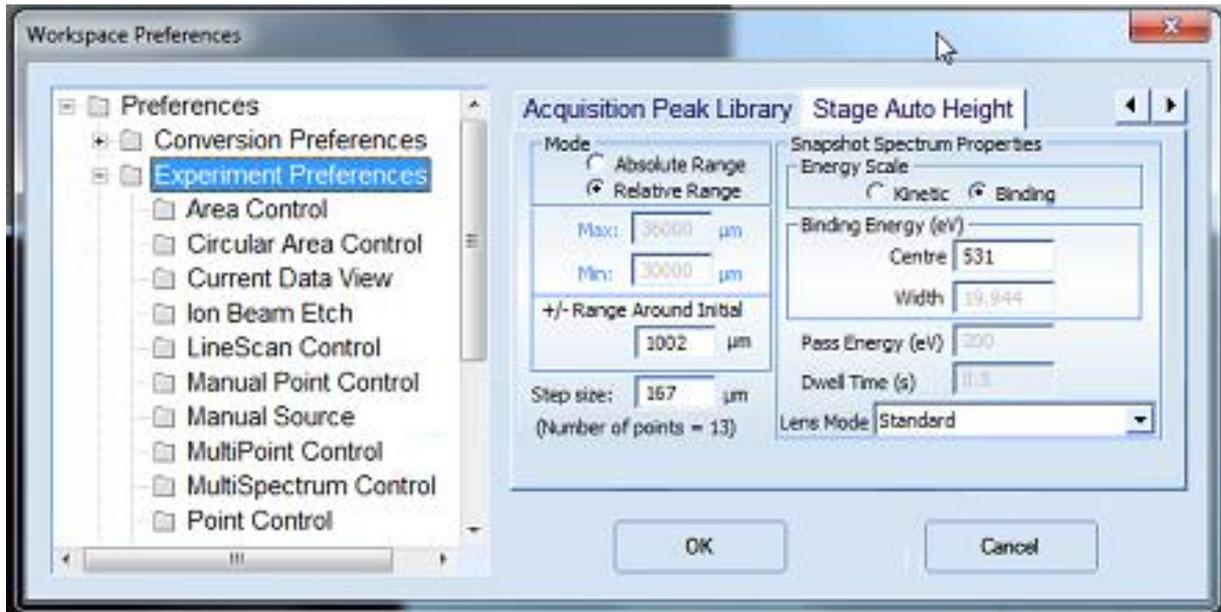


Figure 27: Experiment Preferences

Setting the Sample Height: Manual Height Setting for Point Analysis

- For small feature analysis, the height may be optimised by using the **Height Adjust** camera.
- Locate the feature using the **Platter** and **Sample View** images.
- Adjust the sample height so **Sample View** image is approximately in focus.

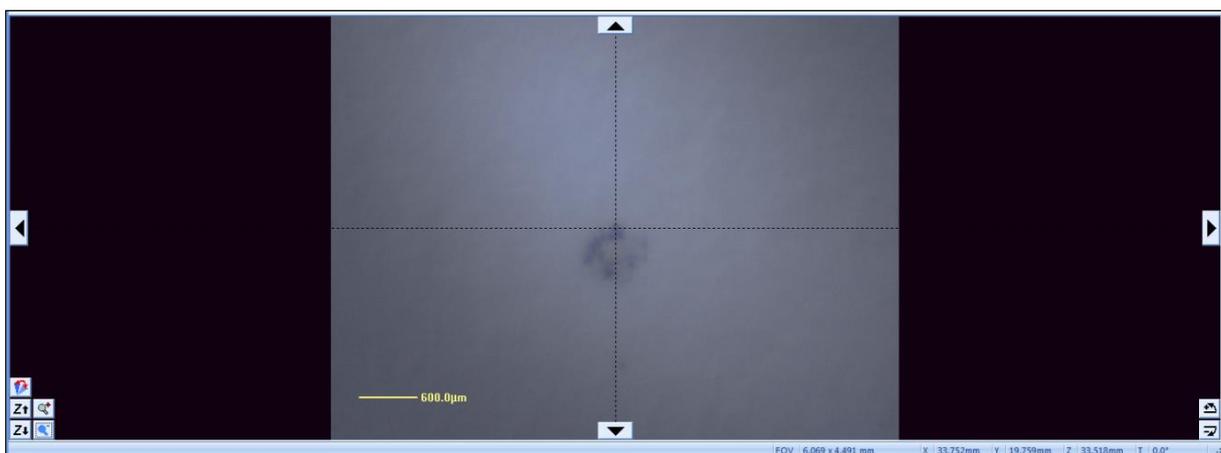


Figure 28: Sample View Image

- Switch to **Height Adjust** image 

- The different camera angle may change the appearance of the feature and you may need to adjust the lighting to get a clear image.



Figure 29: Adjusting the Lighting Gear

- Adjust the height using the Z+ and Z- buttons until the cursor lies on the feature.
- In the **Height Adjust** image, only the Z+ and Z- controls are enabled.



Figure 30: Height Adjustment

The diagram below shows how this procedure operates. The two cameras will only view the same feature when the sample is at the correct height.

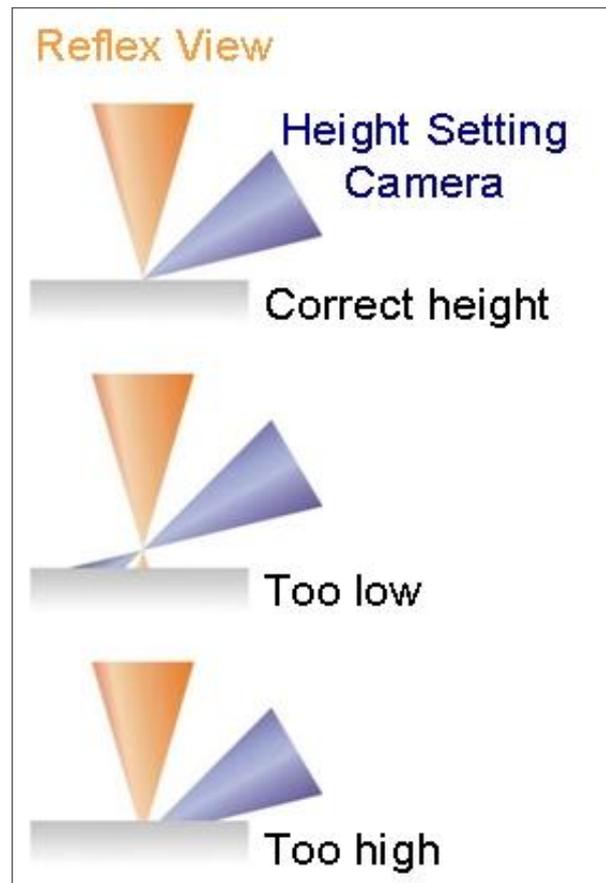


Figure 31: Camera Height Setting

Setting the Sample Height: Auto-Height for Line Scans

For line scans auto-height is enabled by clicking the checkbox on the **Position** tab of the properties panel. The mode can be set using the **Auto Height** tab. This is whether the height is measured just in the centre (1 point), at each end (2 points – the line will then have a calculated height at each point which fits a line between the two end heights), or at every point (good for rough surfaces).

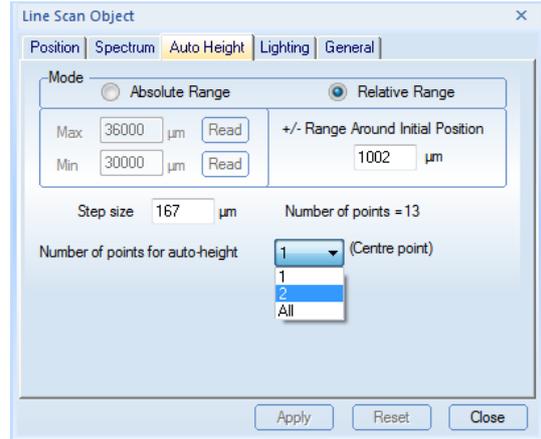
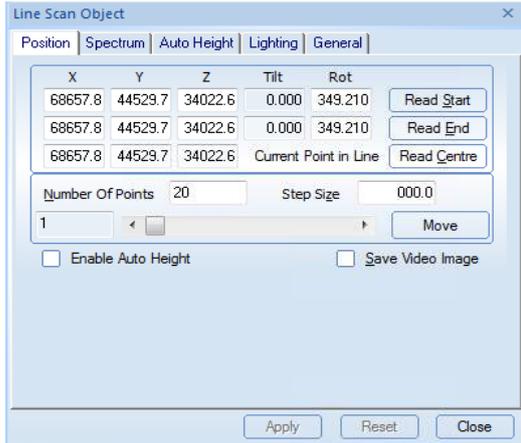


Figure 32: LineScan Control Properties

Setting the Sample Height: Auto-Height for Areas

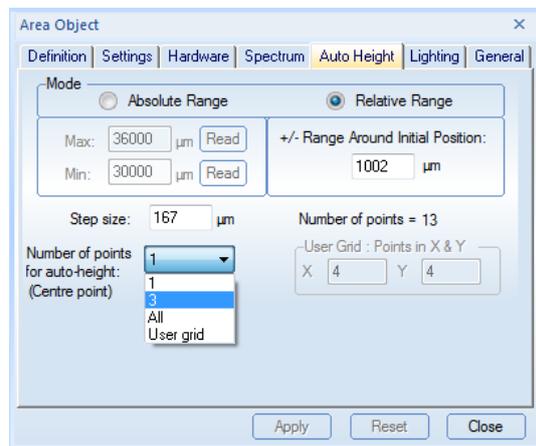
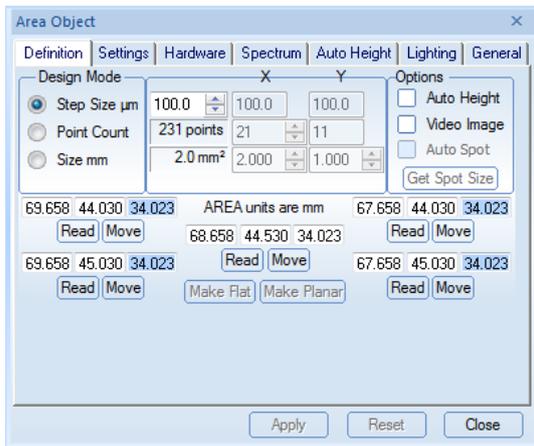


Figure 33: Area Control Properties

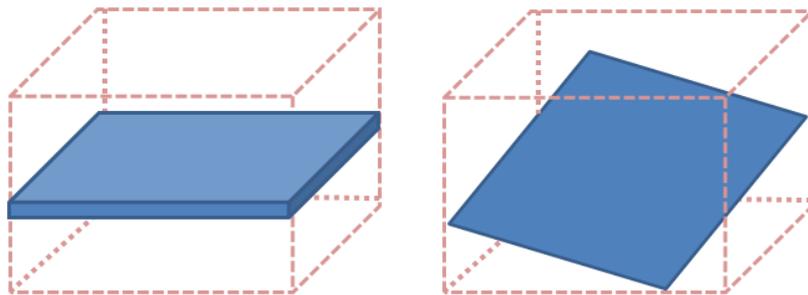


Figure 34: Suitable Sample for 1 Point Auto-Height (Left). Suitable Sample for 3 Point Auto-Height (Right)

Adding Analysis Areas to an Experiment

Select the analysis type from the drop-down menu in the optical view toolbar. There are 6 options available:

Point	A single point. This is the standard mode of operation.
Auto-analysis	A single point enabled for auto-analysis mode.
Multi-point	Allows several points to be added. The same spectra will be collected at each point and will be stored in the same set of data files for easy comparison.
Depth-profile	A single point with a depth profile object.
Line	A line scan object. This is added by clicking and dragging to define the start and end of the line on the optical view or platter view
Area	An area is used to create an XPS image of the surface. It is created by clicking and dragging a rectangle on the optical view or platter view.

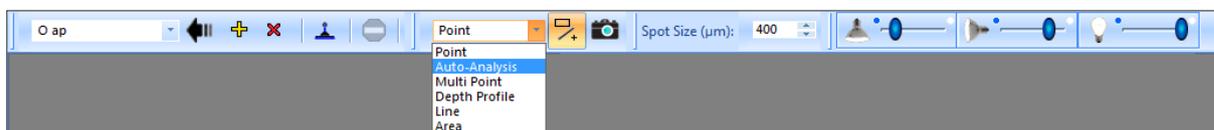
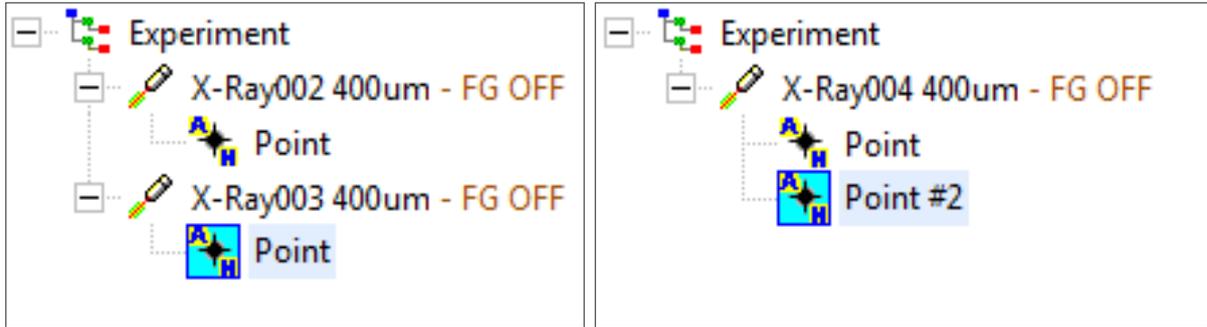


Figure 35: Analysis Types

Once the analysis position has been found, the actual analysis area needs to be defined. This is done by holding down either the Ctrl or Shift keys and clicking with the left mouse button at the point on the optical view, SnapMap view, or platter view you wish to analyse (for areas or lines, click and hold the left mouse button at the start point, and then drag to the end point and release the mouse button).

- Use **Ctrl** left click to insert an analysis object with a new X-ray spot size.
- Use **Shift** left click to insert an analysis object under the currently selected X-ray spot size in the experiment window.



2x ctrl clicks – points under 2 sources

2x shift clicks – points under the same source

Figure 36: Inserting an Analysis Object

Moving Points, Lines and Areas

Click on the point and drag it to the new position.

Click and drag on the end or corner of the Line or Area to resize a line of area.

Click and drag away from an end of edge of a line or area to move without resizing.

Deleting Points, Lines and Area

Select the object in the Experiment tree and press the <Delete> key.

Point Settings

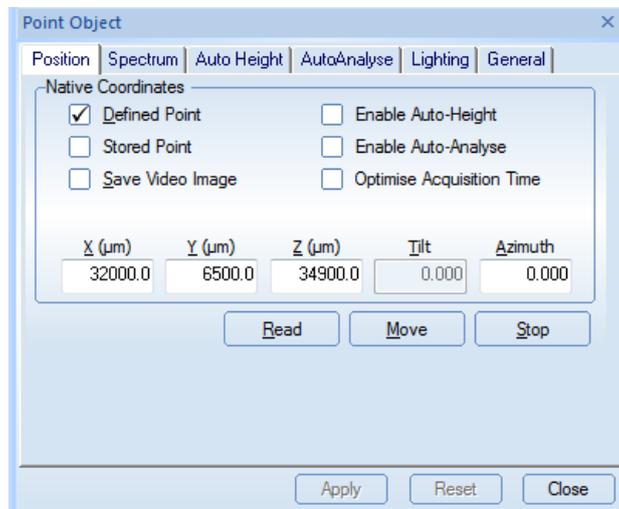


Figure 37: Point Control Properties – Position Tab

Defined Point	Use X, Y, Z, Angle co-ordinates defined below for the analysis.
Stored Point	Use a stored position from the Optical View preset list for the analysis (e.g. “_Silver”).
Save Video Image	Stores a time-stamped .vgd of the optical view with the data when collected. Note that this happens every time the experiment is run, so multiple images will be stored.
Optimise Region Acquisition Time	<p>Apportion the total experiment acquisition time between regions, in proportion to a function of the current measured Signal : Noise Ratio (SNR) for each region. The SNR evaluation is carried out after all regions have completed a scan / snapshot.</p> <p>As the experiment progresses, the (remaining) regions with the highest SNR sequentially have no further scans or snapshots acquired. Near at the end of the experiment only the lowest SNR regions are still acquiring until all the original ‘total acquisition time’ has been used.</p> <p>Not available for multi-level experiments.</p>
Enable Auto-Height	Use the XPS signal to determine the best Z-position for acquisition.
Enable Auto-Analyse	Activate Auto-Analysis for this point. No spectra need to be defined if this is clicked. Automatically activated if the point is defined as “Auto-Analysis” from the Optical View.

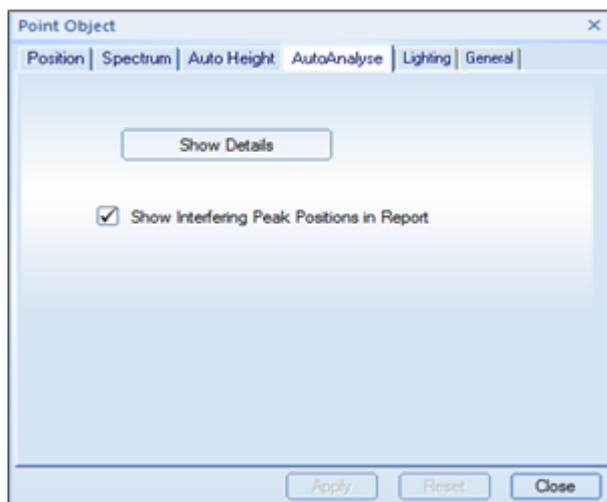


Figure 38: Point Object – Auto Analysis Tab

Show details

Configure the Auto-Analysis experiment (see Auto-Analysis section).

Show Interfering Peak Positions in Report

Display red markers to highlight overlaps in the auto analysis report.



Figure 39: Point Object – Lighting Tab

What to do with Analysis Lights?

Chooses whether to leave the lights as set in the optical view, recall a stored setting, or turn the lights off. Note: this setting is ignored if a Raman spectrum is acquired, as the lights must be turned off.

Read Light Levels

Reads the current light settings from the optical view.

Apply Light Levels

Applies the saved light settings.

Line Settings

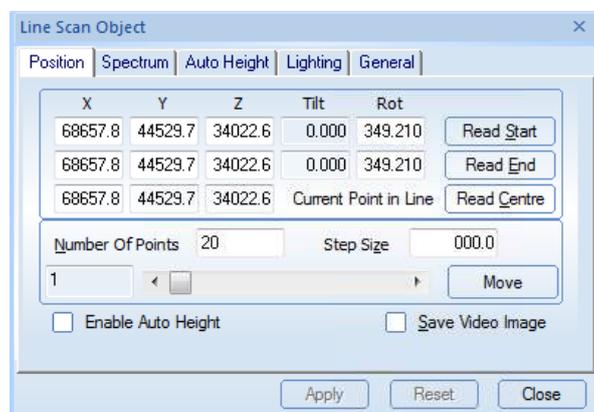


Figure 40: LineScan Control Properties

Read Start	Define the current stage position as the first point of the linescan.
Read End	Define the current stage position as the last point of the linescan.
Enable Auto-Height	Use the XPS signal to determine the best Z-position for acquisition. The settings on the Auto Height tab as described above determine how many points are used for the auto-height.
Number of Points	Number of points in the linescan.
Step Size	Distance between points in μm .

Area Settings

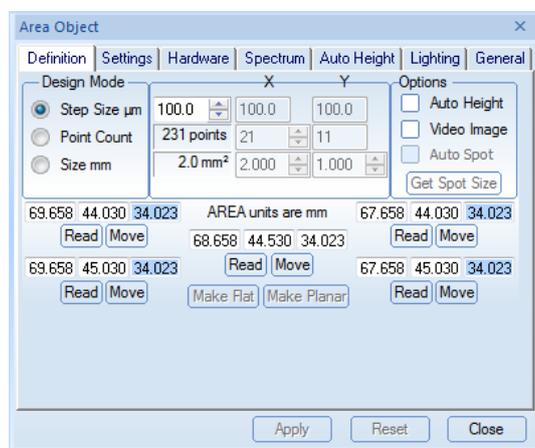
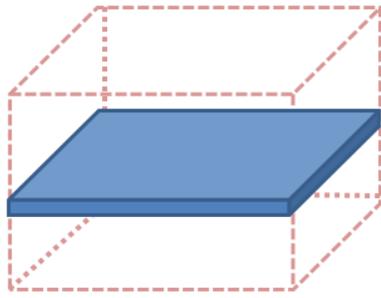
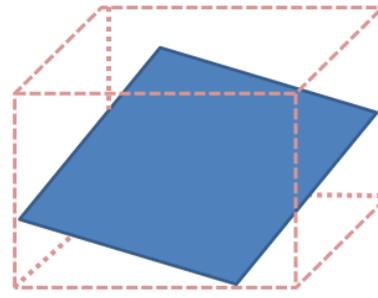


Figure 41: Area Control Properties

Design mode	Define the area by the step size between points, the number of points, or the area size.
Auto-Height	Use the XPS signal to determine the best Z-position for acquisition. The settings on the Auto Height tab as described above determine how many points are used for the auto-height.
Video Image	Save an optical image of the area to be mapped. If the area is larger than the camera field of view, a series of images will be collected and tiled to create an image of the area. The platter camera view is also saved in this case
Auto Spot / Get Spot Size	Optimises the X-ray spot size based on the step size.
Read / Move	Read in the current stage co-ordinates, or move to the defined position.



Make Flat sets all of the Z coordinates to the same value.



Make Planar takes three corner values and creates a sloping plane.

Figure 42: Area Settings

Multiplex Operation

The General settings for each object have an option called **Multiplex**. If this is selected, one scan for each element is collected, then the next scan is collected for each element until all requested scans have been acquired (unlike the standard mode of operation where all the scans for one element are collected before moving on to the next region). This can be advantageous if it is required to ensure that the X-ray dose is constant for all acquired data, rather than the first region acquired having minimal exposure compared to a long exposure for the last region acquired.

For X-ray sensitive samples it is probably better to collect low numbers of scans as a line scan or area and collapse the data, rather than operate in this way.

Configuring the Analysis Area by Setting the X-ray Spot Size

Once the positions are defined, the X-ray source size can be optimised to fit the analysis area. This is most easily done by selecting the Point (or Line or Area) object, clicking on the spot size control in the optical view, and using the mouse-wheel to adjust the X-ray spot to the desired size.

The X-ray spot is adjusted in 5 μm steps. The Nexsa is a source defined XPS instrument, and so the X-ray spot size defines the analysis area.

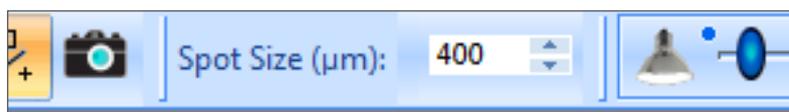


Figure 43: Spot Size

Enabling the Flood Gun for Insulator Analysis

For electrically insulating samples, activate the **Flood Gun** checkbox in the X-Ray property page to enable the use of the charge compensation system during the analysis. If you are unsure as to whether the source is required, it is best to turn the source on, as it will not adversely affect the analysis of a conductive sample.

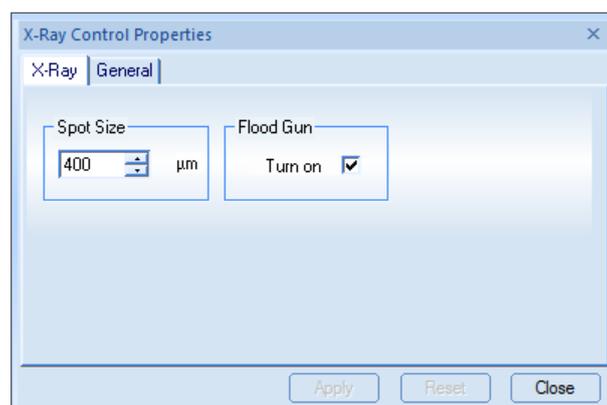


Figure 44: X-Ray Control Properties

Defining Spectrum Regions

For Auto Analysis, spectra do not need to be defined as this will be done automatically as the experiment is run. Otherwise, for each analysis Point, Line or Area, the spectra to be acquired need to be defined. This can most easily be done using the Multi-Spectrum tool. The Multi-Spectrum tool is context-sensitive to the source that is being used, and options change depending on if XPS, ISS, UPS or REELS are being used. See the relevant appendices for details

Using the Multi-Spectrum Insert Tool (XPS)

- Click on the Point object in the experiment tree that you wish to add spectra to and then select the **Multi-Spectrum** tool.

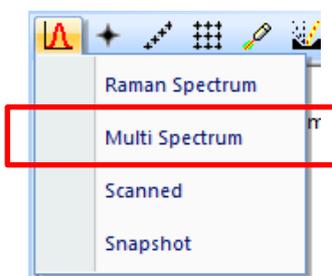


Figure 45: Multi Spectrum Tool

- The **Multi-Spectrum** tool allows the selection of the element regions and survey spectra from the Periodic Table. Right-clicking on an element allows you to choose transitions other than the major peak to be inserted into the experiment (to account for potential overlaps with other elements for example).
- The **Multi-Spectrum** tool detects the source object above it, so that default scans for use with the UV source (He1 and He2), ISS and REELS can be added using the check box that appears in the "Survey" position. Please see the relevant sections for UPS, ISS and REELS for more details.

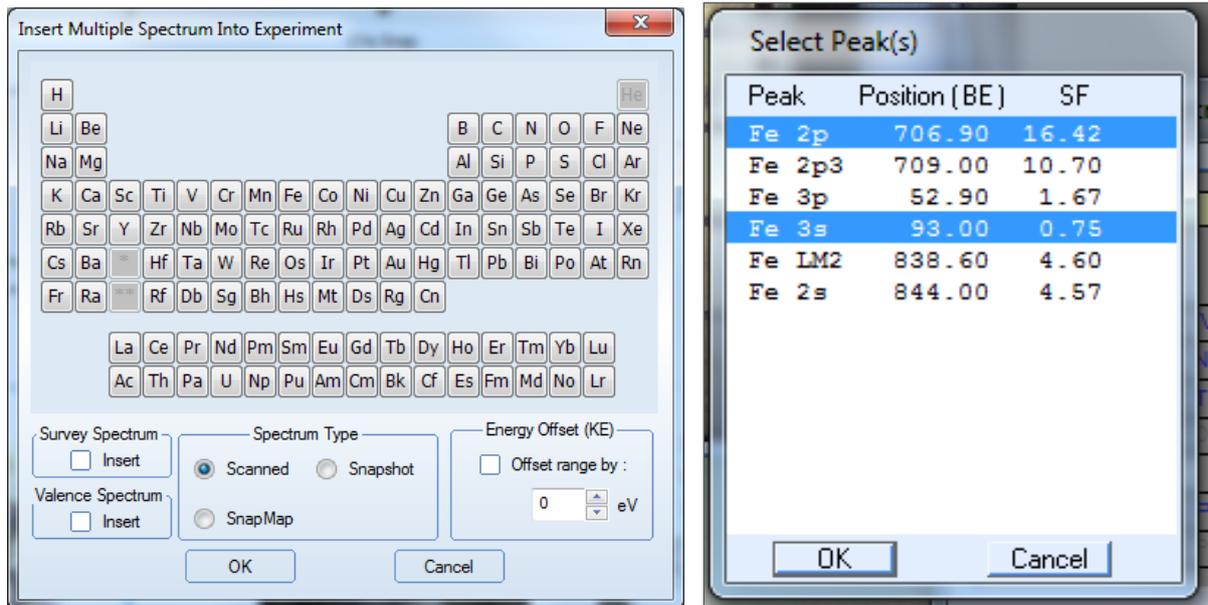


Figure 46: Inserting Multiple Spectrum Into the Experiment

- The spectrum type should also be selected from the Multi-Spectrum tool. Scanned spectra are best used for point analyses to get the best possible resolution. Snapshot spectra collect more quickly and are therefore best used for multi-level data sets such as line scans, areas, or depth profiles. SnapMaps are rapid images using snapshot spectra, scanned over 0.5, 1, 2, or 3 mm².

Selection of Pass Energy and Step Size

The pass energy determines the energy resolution - a higher pass energy will give increased XPS intensity but with increased peak width. Analysis is usually a compromise between energy resolution and sensitivity.

- Lens mode “Standard” should be used for XPS acquisition. Other lens modes are described in the relevant sections.
- For survey spectra, a pass energy of 200 eV and step size of 1 eV is suggested.
- For higher energy resolution, narrow scan spectra a pass energy of 50 eV with step size of 0.1 eV is suggested. Lower pass energies e.g. 20 eV or 10 eV may be required to get the ultimate energy resolution. Often, the natural linewidth of the photoelectron line combined with the width of the X-ray source mean there can be little benefit in using a very low pass energy. The intensity will be reduced but the observed peak width may not be significantly improved.
- For snapshot spectra, a pass energy of 150 eV is recommended to ensure that the spectrum width is approximately 20 eV. In snapshot mode changing the pass energy changes the width of the spectrum as these two parameters are linked.
- The acquisition parameters for each spectrum can be changed in the spectrum property page. **All scans that are going to be quantified together must be collected at the same pass energy.** It is best to set the parameters when the Multi-Spectrum tool is open so that they are applied to all region spectra inserted, but they can also be accessed by clicking on individual spectrum objects in the Experiment Tree.

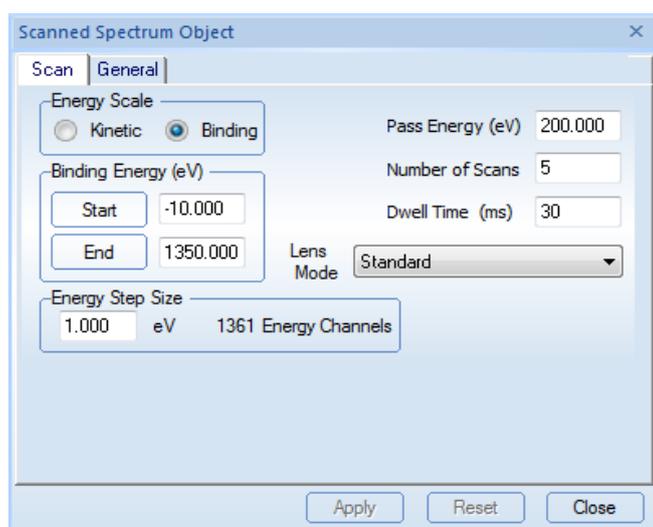


Figure 47: Scanned Spectrum Control Properties

- The defaults for the survey spectrum, and all the spectrum types can be set in **Avantage Preferences > Experiment Preferences > Multi-Spectrum Control**.

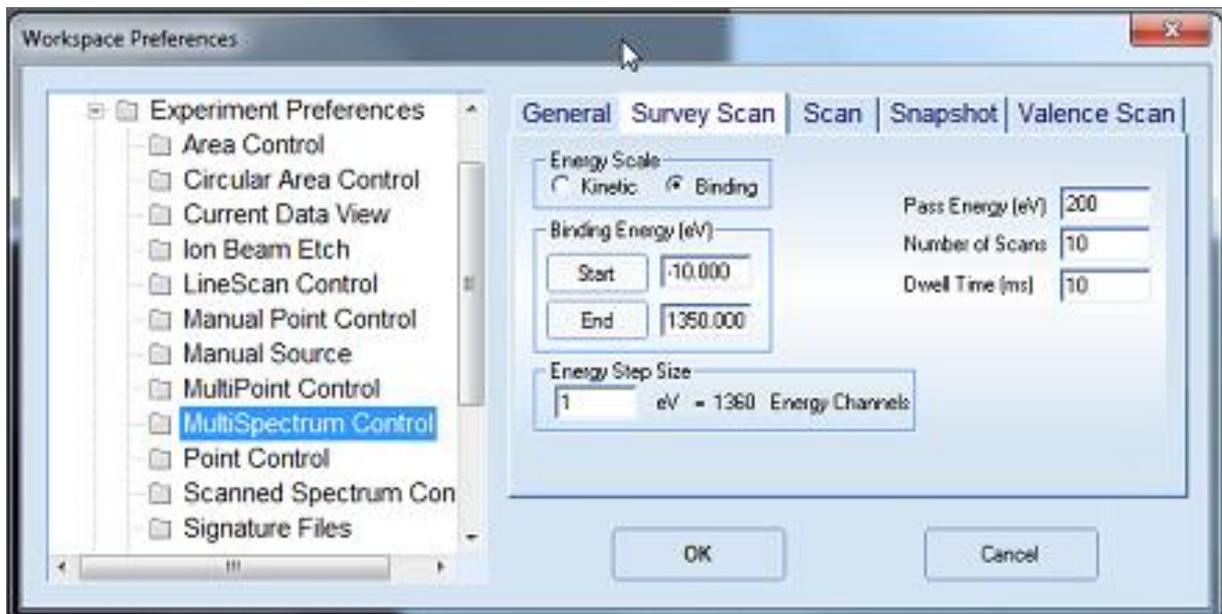


Figure 48: Workplace Preferences

Gun Shutdown Object

After completing the set-up for an experiment it is good practice to put a **Gun Shutdown** object at the end of the experiment. This ensures that the sources are only running when needed and prolongs the lifetime of consumable parts such as filaments.

The **Gun Shutdown** object is accessed from the **Sources** object button at the top of the **Experiment** page.

Depth Profile Analysis

NOTE: For information on MAGCIS, please see page 74.

- Depth profile analysis is set up in a similar way to other experiments.
- Use **Multi-Spectrum** to insert the spectrum regions.
- For electrically insulating samples, check **Flood Gun** in the **X-Ray** property page.
- The raster size of the ion beam sputtered area will be automatically optimised relative to the size of the X-ray spot. The raster area is rectangular in shape, with the short side in the y-direction, and the long side in the x-direction. The Raster Size control sets the short dimension; for example setting a 1 mm raster will generate an etch crater with dimensions 1 x 2 mm. By default the raster is automatically set to be 5x the X-ray spot size with a minimum value of 1 mm.
- Select the **Ion Energy** and **Current** in the **Depth Profile > Ion Gun** page.

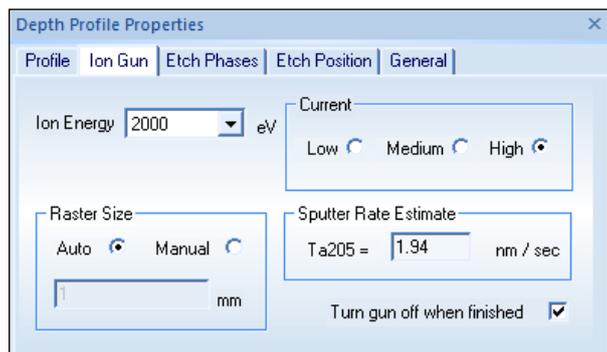


Figure 49: Depth Profile Properties. Ion Gun Tab

- Select the number and length of etch periods in the **Depth Profile > Profile** page.

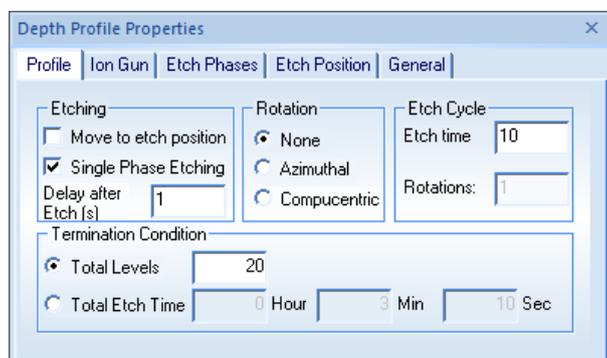


Figure 50: Depth Profile Properties. Profile Tab.

Delay after etch

If the sample is an insulator, this value is used to allow a delay for the charge compensation to rebalance after etching before acquisition. 10 s is a common value.

Rotation

If the rotation holder is being used, then the sample can be rotated during etching to improve the possible depth resolution. Azimuthal rotation requires acquisition to be at the centre of the axis of rotation (standard position **_Centre of Rotation** in the optical view), and allows minimum etch times of 10 seconds. Compucentric rotation allows profiling off the rotation axis. In this mode the minimum etch time is 30 seconds.

Multi-Phase etching

This allows the etch time per level and the number of rotations to be changed during a profile.

- Insert – adds a new line into the table
- Append – adds a new line at the end of the table
- Delete – removes the selected line from the table.

After changes have been made, **Apply** must be pressed to action the changes.

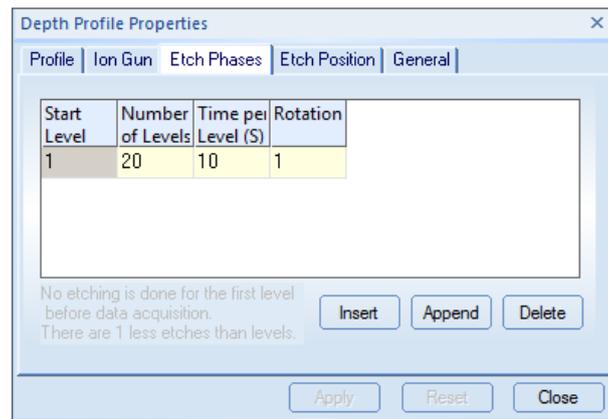


Figure 51: Depth Profile Properties. Etch Phases Tab

- When the experiment is run, the argon gas valve will be automatically opened, and the source will be turned on. It is left in **Blanked** mode (yellow status light) until required to etch (green status light).

Collecting Data

Select Where to Store the Data

Data is stored in the folders defined by **Project Root\Project Name\Experiment Name**. This is entered on the **Experiment** object properties page. The experiment name can be entered either on the **General** tab, or by clicking twice slowly on the **Experiment** object in the **Experiment Tree** (as would be done to rename a file in Windows).

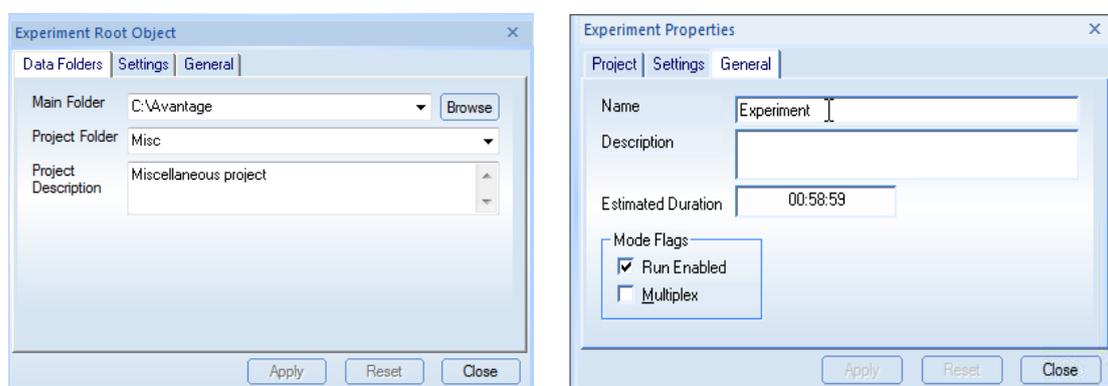


Figure 52: Experiment Properties Tabs

The data is stored in a file structure based on the **Experiment Tree** structure, with each object being a folder, and the spectrum data files being stored a folder based on the name of the object they are attached to. Typically this would be something like **C:\Project Root\Project Name\Experiment Name\Source\Point**.

Running the Experiment

The experiment is started using the **Run Experiment** icon, or from the **Run** menu. Either the entire experiment can be run or a part of the experiment from the currently selected object in the **Experiment Tree**.



Run complete experiment



Run experiment from selected step

- When the experiment is run, the required guns are switched on with the requested parameters. On rare occasions, this may take a few minutes to ensure the guns are providing the requested parameters. If the X-ray or flood gun does not produce the requested parameters in the usual time (~ 30 seconds), a gun conditioning routine may be automatically started, indicated by a yellow status indicator. When the gun is operating correctly, the experiment will continue.

Disabling Experiment Steps

While the experiment is running, steps can be disabled by selecting an object and either pressing the space key on the keyboard, or right-clicking on the object and deselecting **Enabled** on the menu. The object, and any child objects attached to it will change to a grey colour, and the legend (**Disabled**) will be displayed. It is not possible to re-enable objects while an experiment is running. This procedure can also be used before an experiment is run to prevent existing data being overwritten.

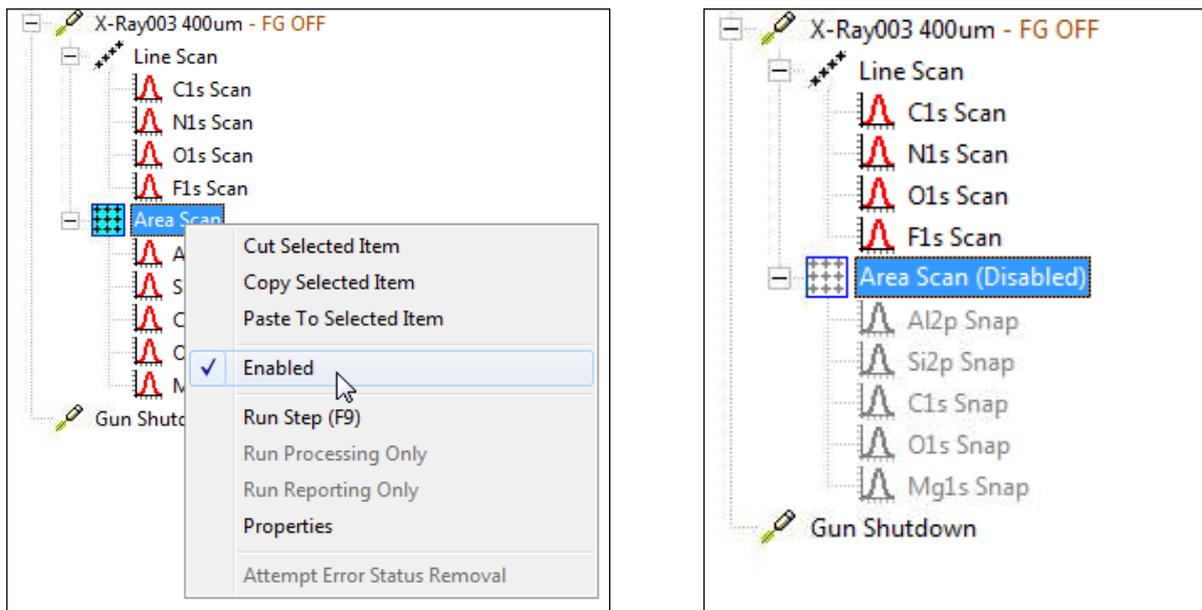


Figure 53: Disabling Experiment Steps

Auto Analysis

Auto Analysis is designed to be used to automatically acquire a survey spectrum, analyse to determine which regions are present and then acquire higher energy resolution spectra for those regions. It then analyses these regions to provide a simple chemical state assessment. The results are reported into a report document.

An Auto-Analysis experiment requires an X-ray source and Point object with **Enable Auto-Analyse** checked. It is recommended that **Enable Auto-Height** is also checked.

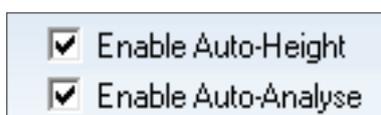


Figure 54: Enable Auto-Height and Auto-Analysis

Usually optimum analysis will use a larger X-ray spot and typically 400 μm for Nexsa.

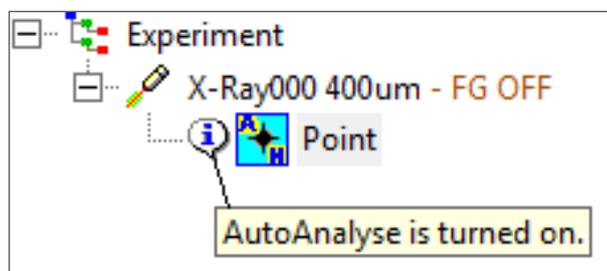


Figure 55: AutoAnalyse Turned On

When the experiment is run a survey spectrum is automatically inserted. The acquisition conditions are those defined for the Multi-Spectrum tool, defined in the **Edit > Preferences**.

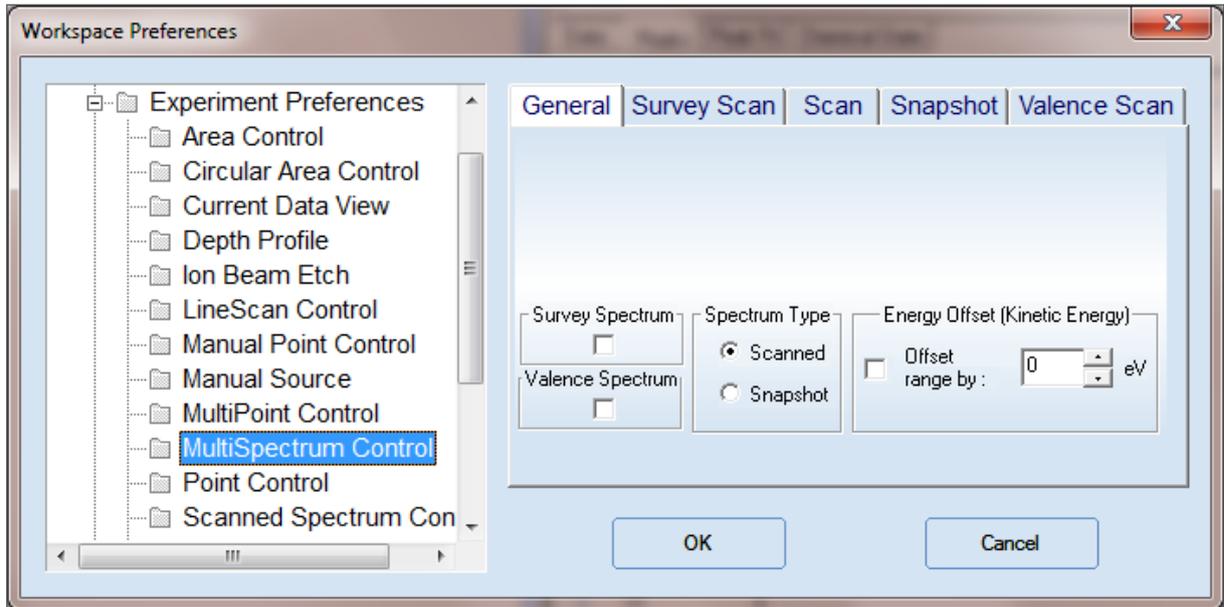


Figure 56: Workplace Preferences. MultiSpectrum Control

After analysis of the survey spectrum, the detected regions are inserted and the survey spectrum is disabled.

Display and Processing of Data

- Open a new processing grid .
- After the experiment has started to acquire data, Drag and Drop the data from the experiment tree into the processing grid.

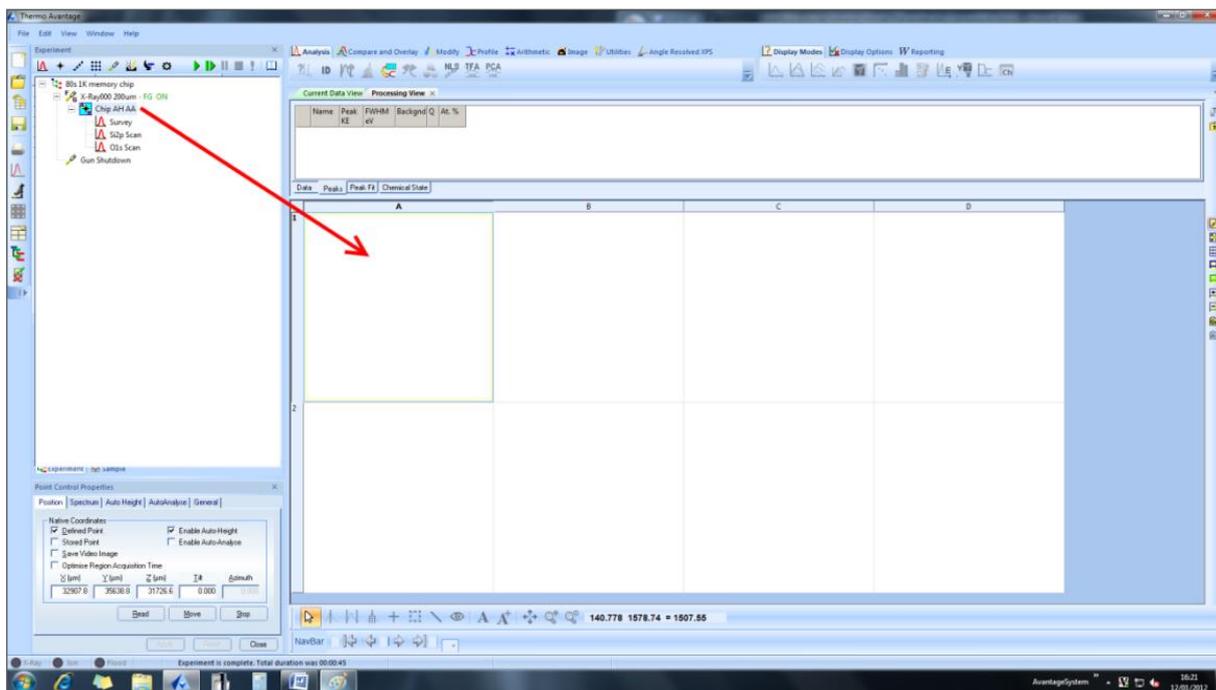


Figure 57: Dropping Data into the Processing Grid

- All of the data below a branch of the tree is copied into the processing grid.

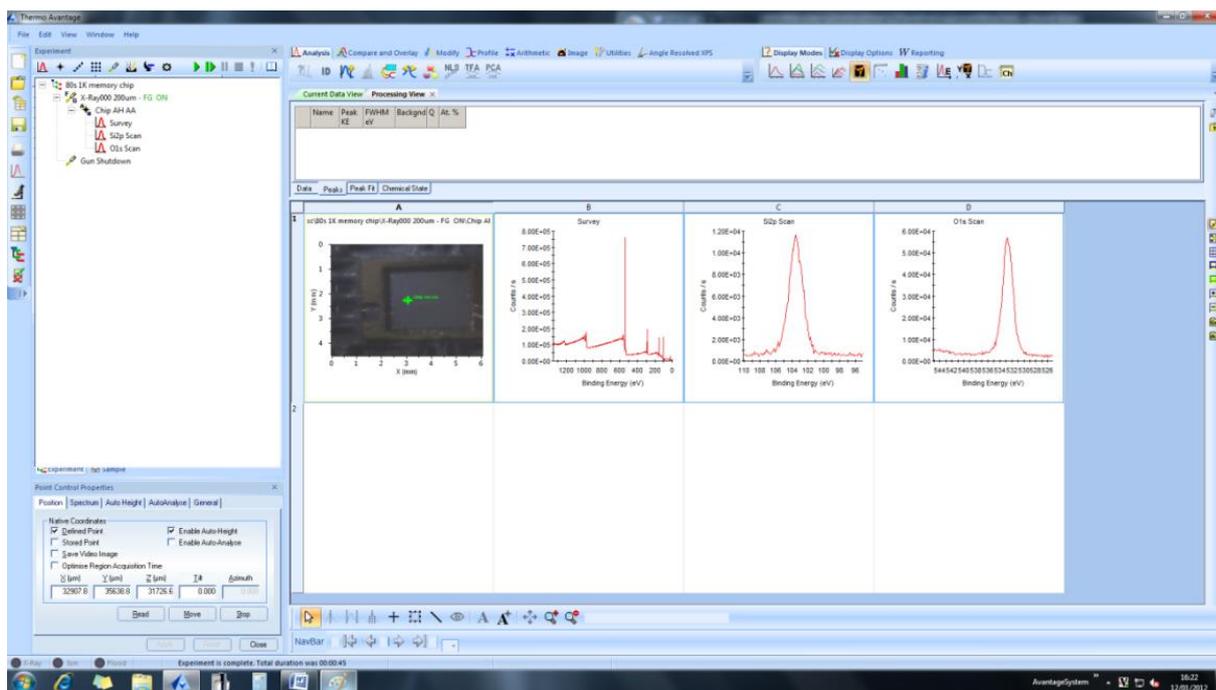


Figure 58: Data Copied to the Processing Grid

- As the data is acquired the spectra will be updated.
- For further information about data display and processing, see the Processing section of the Avantage Help manual (a glossary of the commands available is listed in Appendices 1 & 2, page 136).

XPS Knowledge View

The **Knowledge Viewer** provides useful information to aid analysis of XPS data. It can be started from the icon on the left side of the screen, from the menu bar select **Windows > Open > XPS Knowledge View**, or from a region spectrum and right click **Show XPS Knowledge for this spectrum**

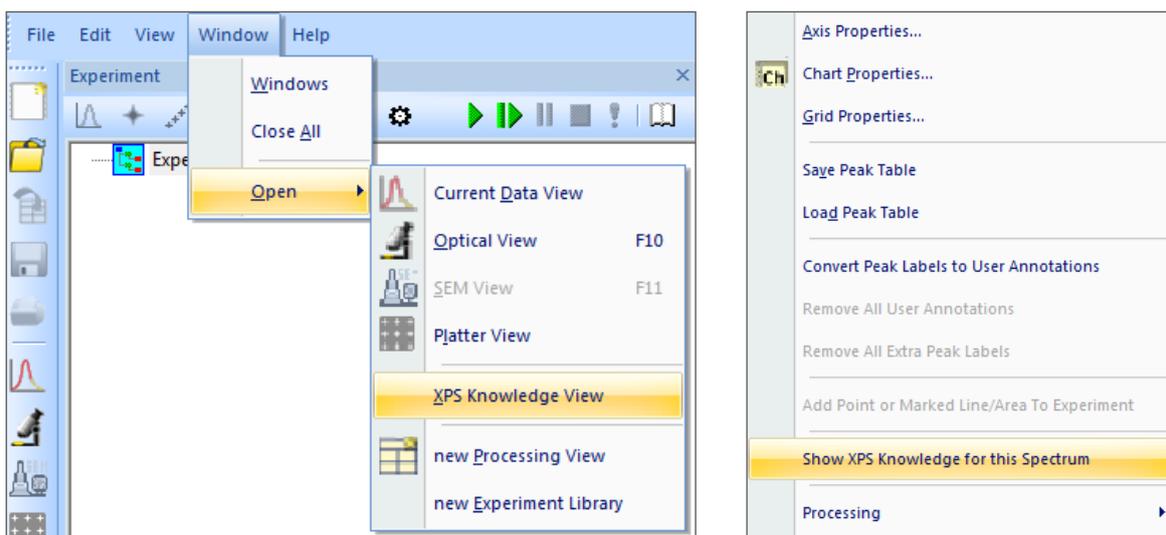


Figure 59: Showing XPS Knowledge View

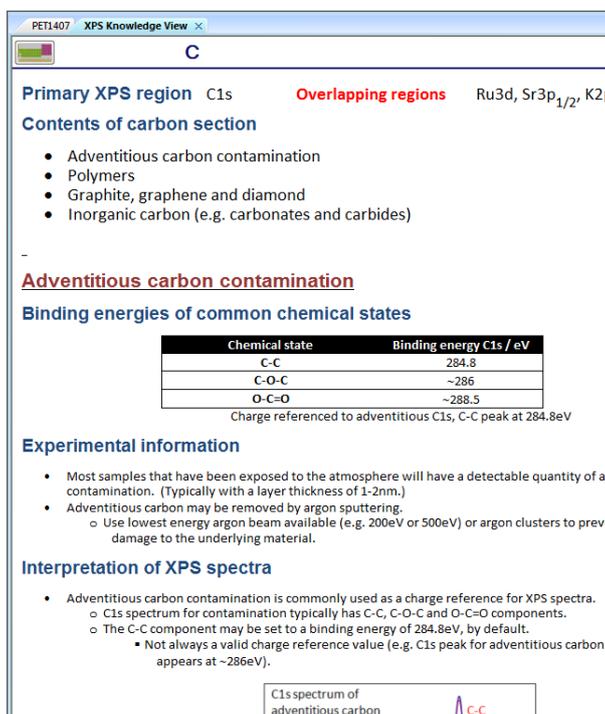


Figure 60: XPS Knowledge View

The **Knowledge Viewer** opens in a tabbed document. As for all tabbed documents, the tab may be dragged and dropped onto the document to split it into a separate pane.

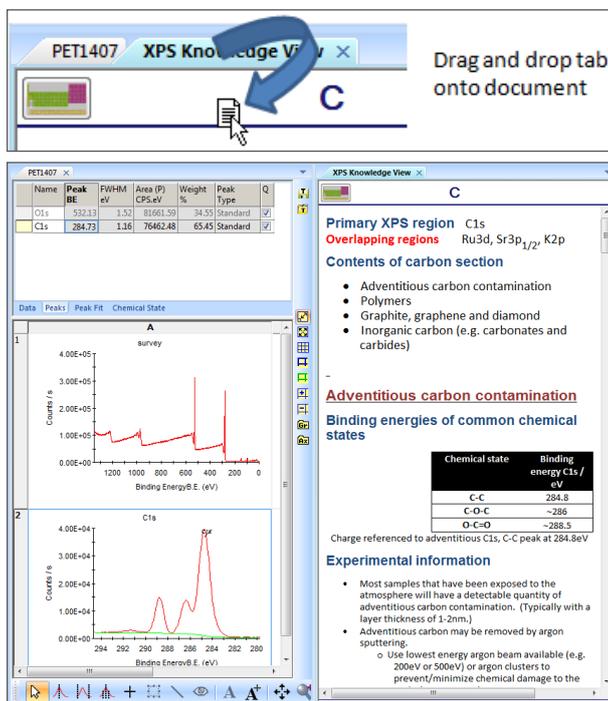


Figure 61: Knowledge Viewer



Many of the pictures in the document have links to vgp files. These are shown by the cursor changing to a hand pointer.

Click on the picture and this will open the *Avantage* processing vgp document.

The **Periodic Table** button can also be used to select an element.

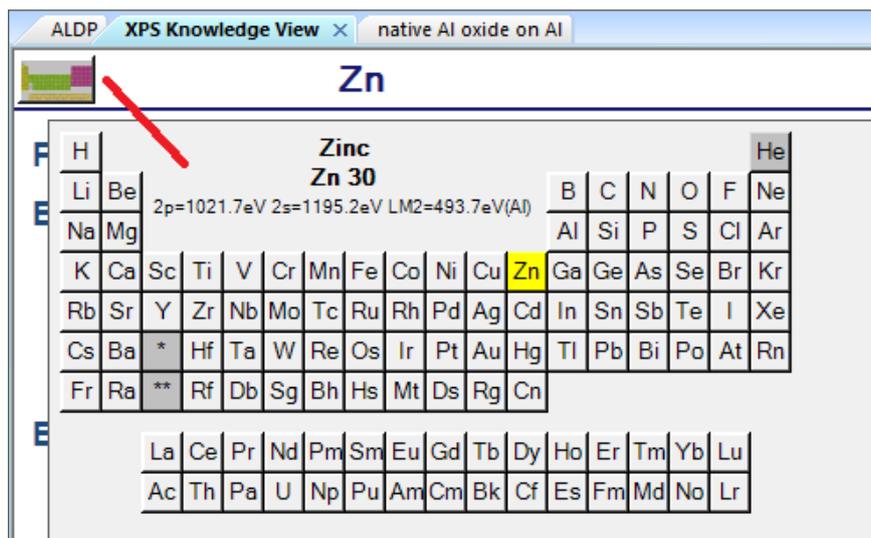


Figure 62: Periodic Table

Maintenance & Troubleshooting

IMPORTANT:

The Nexsa system has no user serviceable parts. If the instrument has a problem, please contact your local service support for advice. This section is primarily for information and should be used with the guidance of a service representative.

Vacuum Details Window

The vacuum details window shows the status of the vacuum system, and contains routines for flushing gas lines, and baking the system. Only routines that are required for regular use of the instrument are described here, and other controls should not be used unless guided to do so by a support engineer.

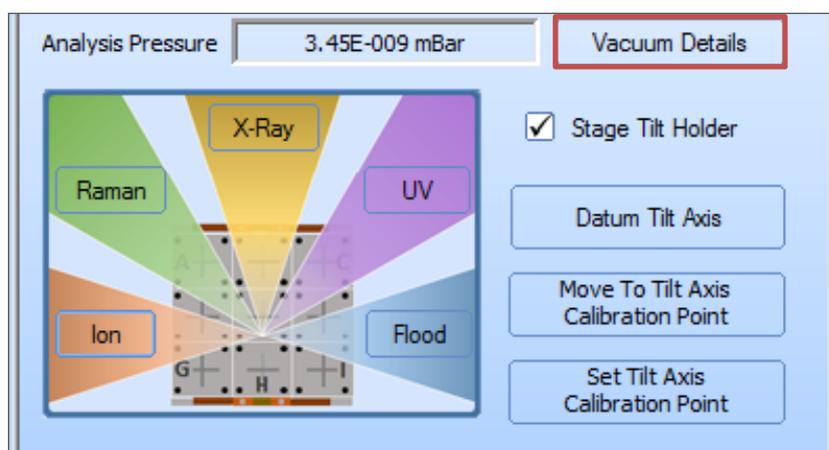


Figure 63: Vacuum Diagnostics Window button

The first page shows the vacuum schematic, the status of the valves and pumps, and the option fitted. The schematic is interactive, and it is not recommended to click on controls unless guided to do so by a support engineer.

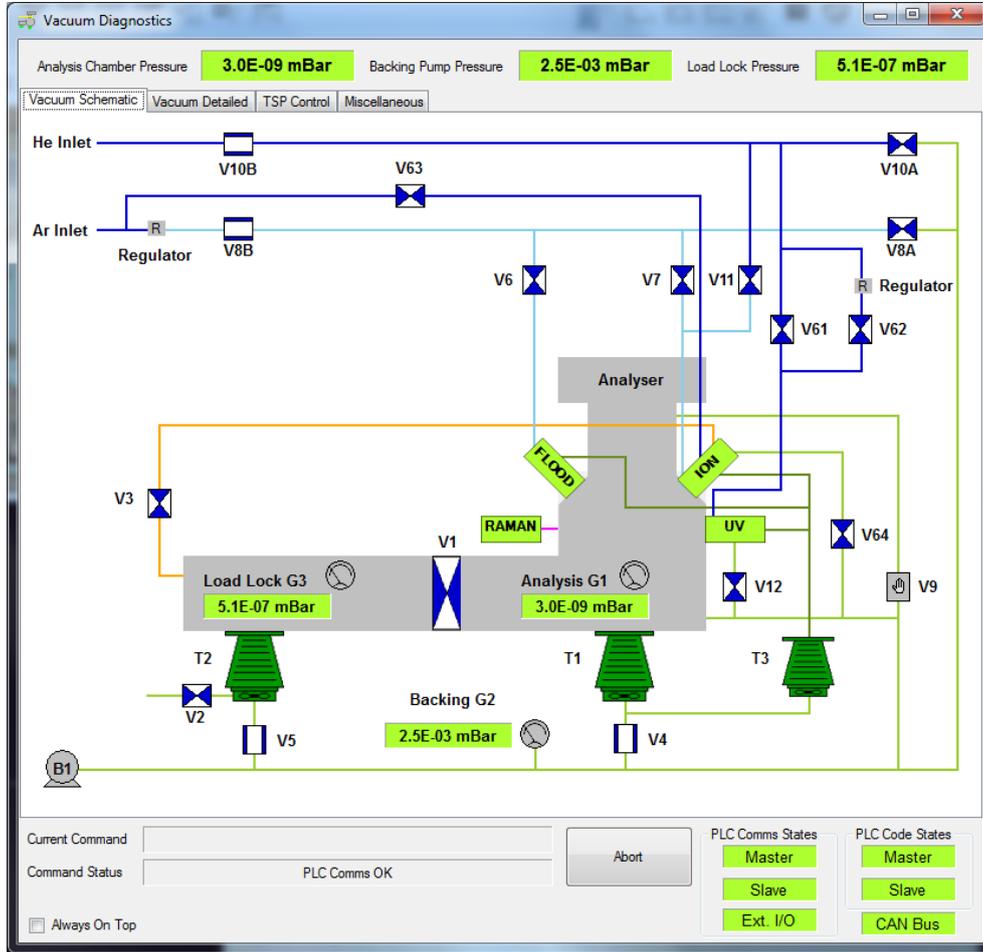


Figure 64: Vacuum Schematic Window (layout seen depends on the options fitted)

The second tab shows the detailed vacuum status. This page contains controls for pumping the argon gas line (and helium gas line if fitted). This should be done after a gas bottle is exchanged, to ensure that clean gas is used by the instrument and prolong the life of consumable parts.

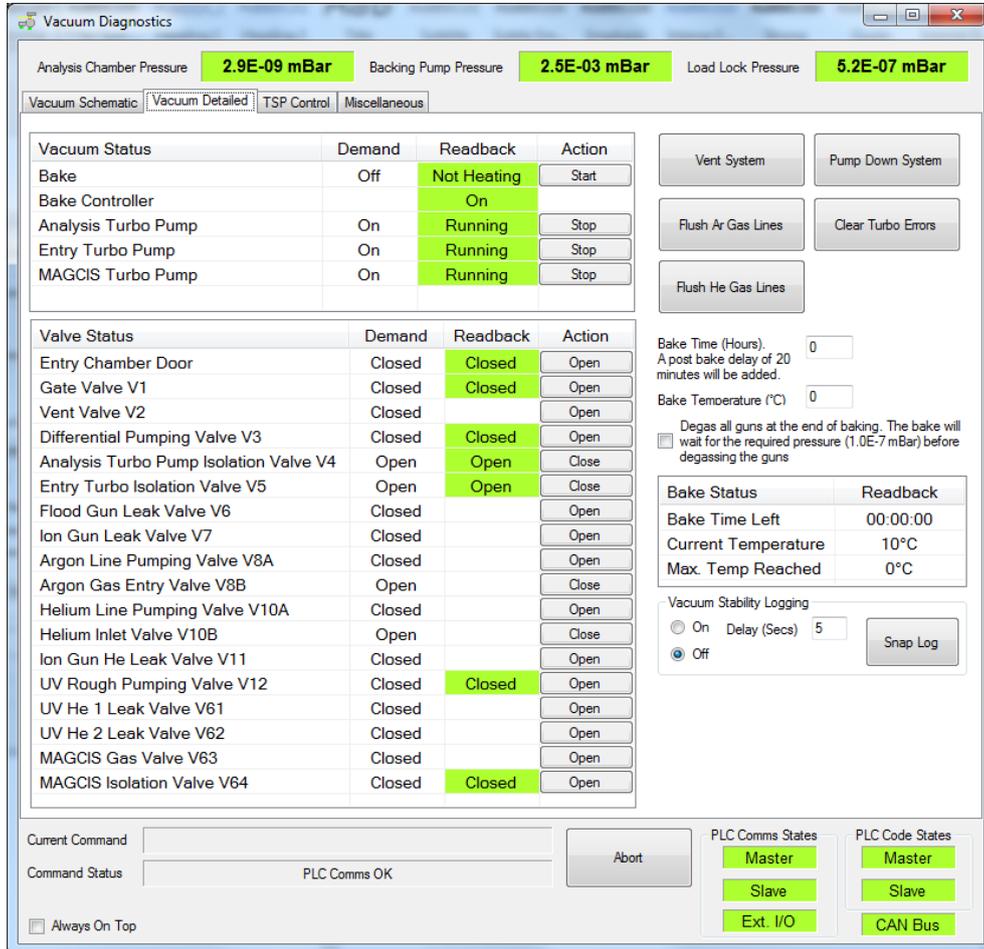


Figure 65: Vacuum Detailed Window
(layout seen depends on the options fitted)

The TSP Control page shows the status of the titanium sublimation pump. The pump runs automatically, and there is no need to operate the controls on this page in normal operation.

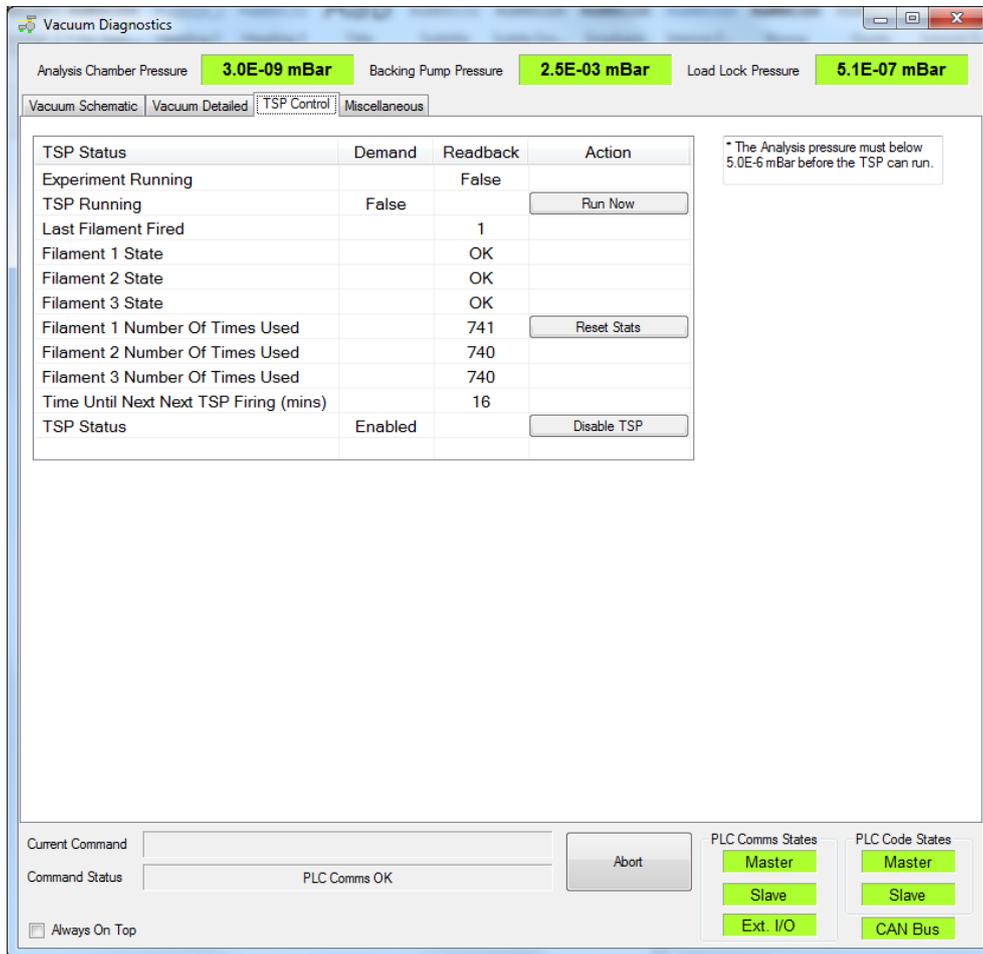


Figure 66: TSP Control Window

The Miscellaneous tab contains troubleshooting and diagnostic controls, and is for engineer use only.

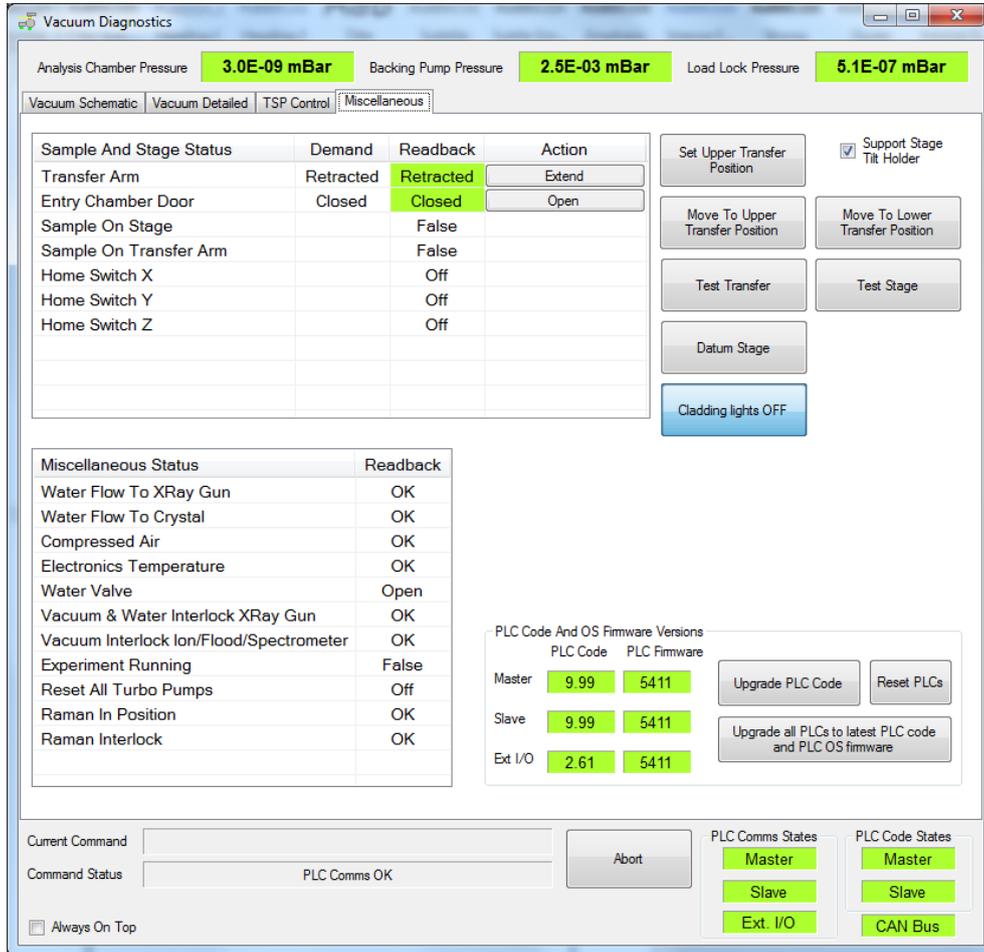


Figure 67: Miscellaneous Window

Ion Gun alignment and calibration

NOTE

Alignment of the ion gun should only be done if requested to do so by a service and support engineer. In typical use of the system it is not necessary to realign the ion source except after the system has been vented for maintenance.

Before starting the procedure, it must be checked that the standard aperture positions are correct, and that the gas lines are purged and connected to the gas supply. To align the ion gun, open the ion gun details page, and choose the **Filament** tab. From this page several options are available to align the currently selected mode, all monatomic or cluster modes, or all modes.

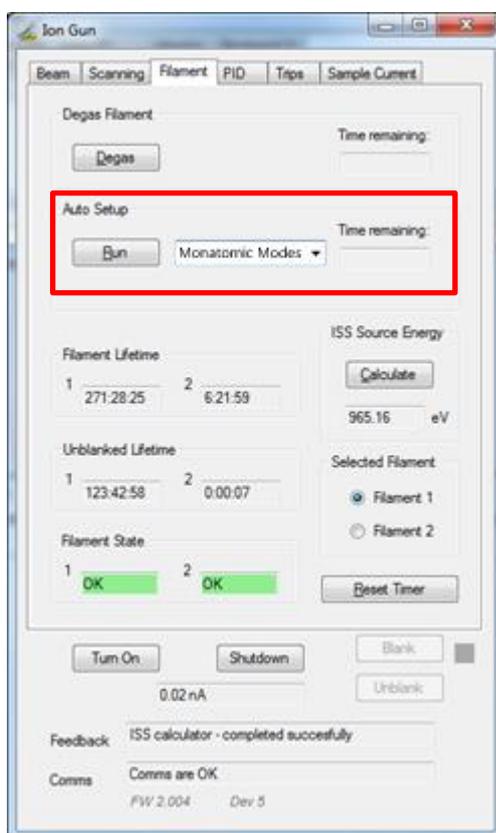


Figure 68: Calibration routine page on ion gun details window

Spectrometer Calibration



The Nexsa calibration tool contains full instructions on how to run procedures. Most routines are designed for authorised service personnel, but there are tools that are for routine calibration of the instrument and also for assisting with troubleshooting.

Spectrometer energy scale and transmission function calibration

To calibrate the spectrometer, open the calibration software, and choose the **Spectrometer** tab. Select **Energy Scale/Tx Fn/Performance Specs** and press **Run**. This will take around 30 – 60 mins to run, and will calibrate the spectrometer and measure the instrument performance. It is recommended to run this process once per month.

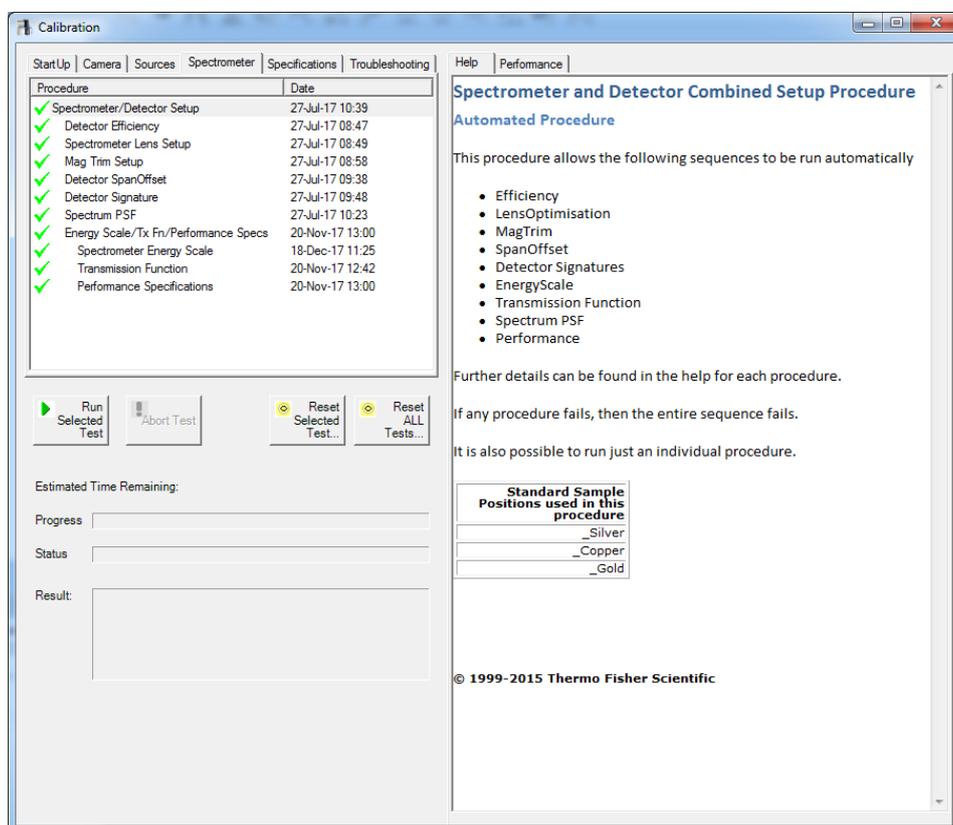


Figure 69: Spectrometer calibration page

Log files

The **Troubleshooting** tab in the calibration software contains routines to collect the logfiles if requested by a support engineer.

Software Reset



If Avantage fails to respond, the software should be closed using the Windows Task Manager. The **ServerStopper** program (found in the **Thermo Avantage** group on the Windows Start menu) should be run before Avantage is restarted.

Full System Reset

If it is necessary to restart the Nexsa system (as advised by your local authorised technical support team), the PC must be shut-down first.

The main power switch at the back of the Nexsa should be turned **Off**, and turned back **On** after a delay of at least 30 seconds. The system will automatically run the **PLC recovery** routine, which will ensure all the hardware is running correctly. The PC can then be restarted. When Windows has restarted, drivers may reload automatically. It is also advised to recalibrate the detector. This is done by loading the **1D Detector Toolkit** software from the **Thermo Avantage** group on the Windows Start menu, and clicking the **Calibrate** button on the lower left side of the screen. *Avantage* can then be restarted. The detector calibration procedure should also be used if the power has been interrupted to the instrument.

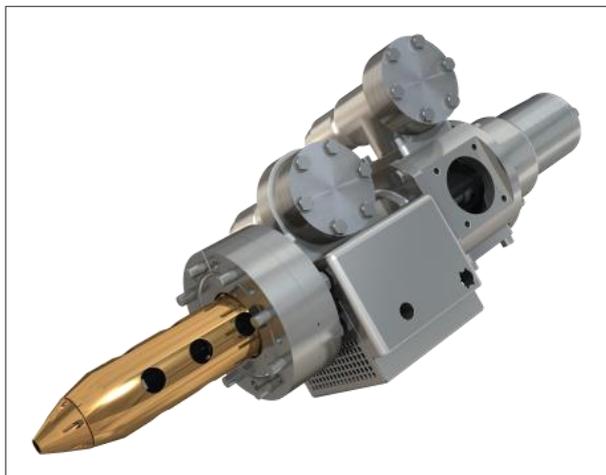


Figure 70: Main power switch

Optional Accessories

The following sections detail the use of the analytical options and the optional sample holders for the Nexsa instrument.

MAGCIS



Introduction

The MAGCIS gun (Monatomic And Gas Cluster Ion Source) is used as a dual purpose ion source for sputtering and depth profiling.

It can be used as a conventional ion gun, using Ar^+ ions, or to produce Ar cluster ions. Cluster ions are typically used for sputtering more delicate materials such as organic and polymer materials. The higher mass of the ion means the average energy per atom is lower and so less damaging.

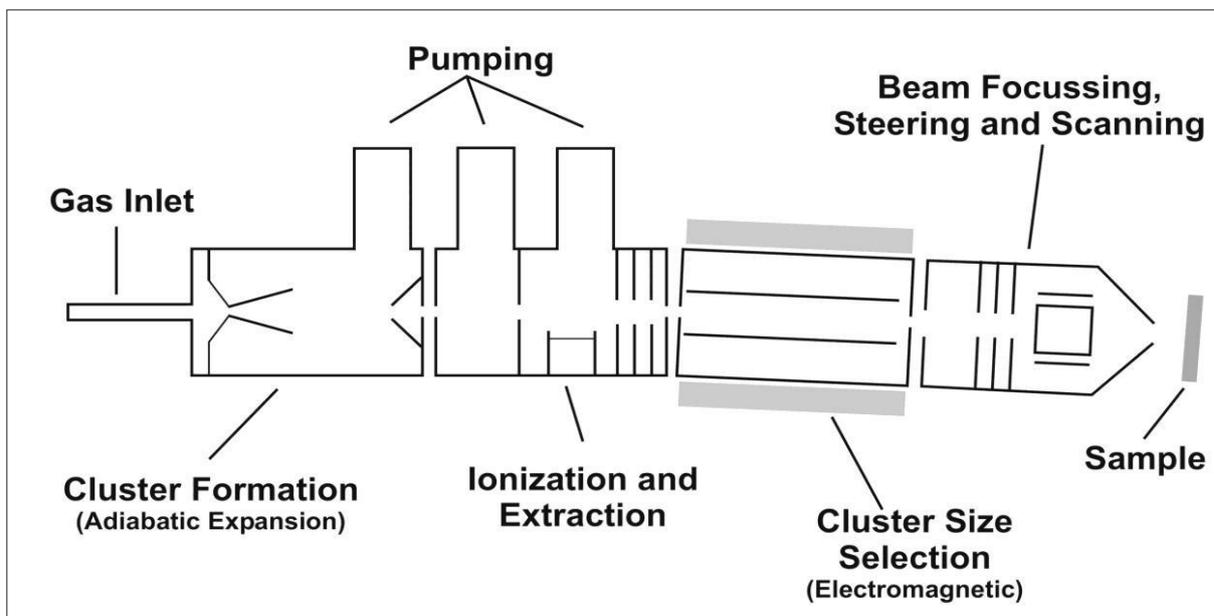


Figure 71: Schematic of MAGCIS

Inorganic materials are not effectively sputtered using the cluster source and usually require use of the monatomic source. Switching between operating modes is controlled through software.

Experiment objects provide controls to switch the mode between monatomic and cluster ion operation. Changing the operating mode to cluster changes the ion gun operating parameters and selects an increased pressure of argon gas through the supersonic nozzle, to enable cluster ion formation. The bend in the gun after the ionization and extraction region eliminates neutrals and requires use of an electromagnet to deflect the ion beam. This also provides a method to select cluster size.

In monatomic mode, argon gas is introduced into the normal ionization chamber. In cluster mode, argon gas is introduced through the nozzle.

Avantage is used to control the source, the gas valves, and differential pumping based on the selection of operating mode. In normal operation the **Depth Profile** or **Ion Etch** experiment objects will be used.

Selection of Operating Parameters

The source operates in exactly the same way as the standard EX06 ion gun (as detailed on page [55](#)). MAGCIS adds the extra control for using either monatomic or cluster mode.

Monatomic or Cluster?

Use of Ar clusters is usually restricted to polymer or organic based materials as Ar clusters do not effectively sputter inorganic materials where the bond strength is larger.

	Monatomic mode	Cluster mode
Energy applied	500 eV- 4000 eV	2000 eV - 8000 eV
Atom size	1	75- 2000
Energy per atom	500 eV- 4000 eV	1 eV - 100 eV
Typical samples for cleaning	Metals, metallic compounds, semiconductors	Glasses, ceramics, metal oxides, polymers, fibres, semiconductors
Typical samples for profiling	Metals, metallic compounds	Metal oxides, polymers, mixed oxide and polymer layers

Monatomic Mode

The monatomic modes are defined with ion beam energies in the range 500 – 4000 eV. When using monatomic modes, selecting a lower ion beam energy will be less damaging but the lower ion beam flux and lower energy will result in a reduced sputter rate.

The monatomic beam modes allow selection of a beam current – either Low or High. The “Low” setting is designed to be 3x lower than the “High” setting to maintain the same sputter rate when using copucentric rotation.

Cluster Mode

When using a cluster mode, the ion beam energies are higher than used in monatomic mode, in the range 2000 – 8000 eV. A lower ion beam energy will usually reduce sample damage but at low energies, there may also be very little sputtering. The cluster mode allows selection of a cluster size – 75-2000 atoms.

For a given cluster ion beam energy, the average energy per atom is higher for the small cluster mode and sputter rate will be higher. In the absence of other information, a typical operating condition for routine use is 6000 eV, 1000 atom cluster size.

Control in Avantage Experiments

Control of the ion gun in an *Avantage* Experiment uses the usual **Depth Profile** and **Ion Etch** experiment objects, as described in the section **Depth Profile Analysis**, page 55.

The **Mode** drop-down menu allows selection of **Monatomic** or **Cluster** mode. Users may then select the **Ion Energy** and **Beam Current** (monatomic modes) or **Cluster Size** (cluster modes).

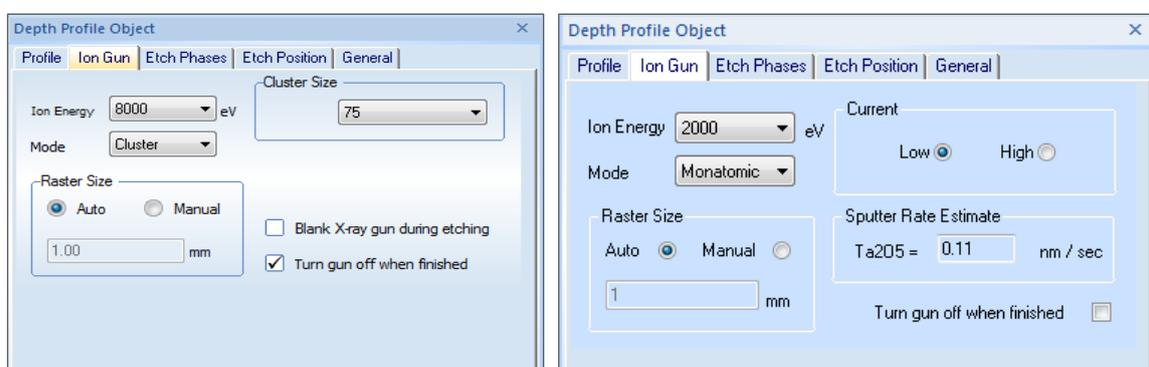


Figure 72: Depth Profile Object. Ion Gun Monatomic and Cluster Mode

Sputter rate for monatomic modes is calculated based on known sputter rates for Ta₂O₅ at different beam energies. The rate is adjusted using the actual optimized beam current obtained during the automatic beam mode setup sequence. Cluster etch rates are heavily dependent on the material being analysed, and so it is recommended to calibrate the sputter rate by analysing a standard of known thickness if possible.

The status of gas valves can be viewed from the **Vacuum Details** page. Users with *Avantage* Engineer access may also open and close valves.

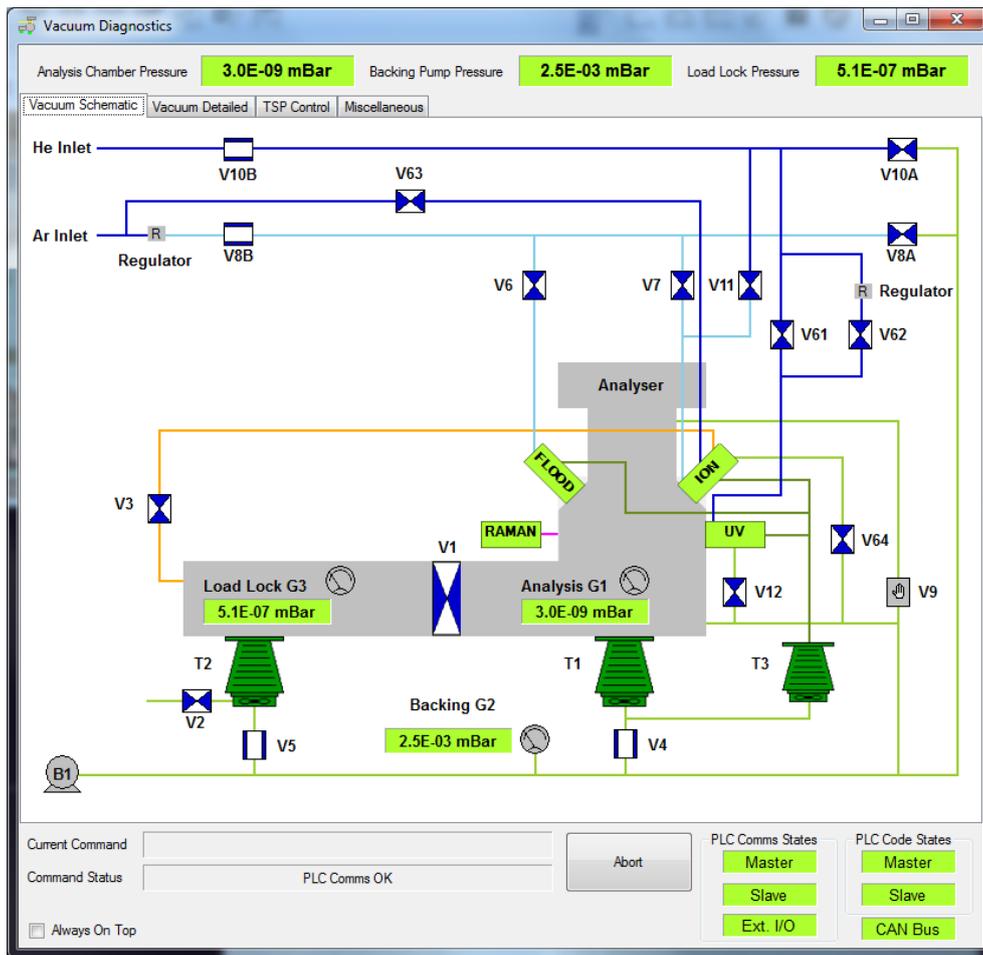


Figure 73: Example Vacuum Details Control with MAGCIS

When using cluster mode, argon valve V63 is open to allow a higher pressure of gas through the supersonic nozzle to allow adiabatic expansion and consequent cooling and condensation of the argon gas to form clusters. The above diagram shows the schematic layout on Nexsa instrument. V64 is opened to provide additional differential pumping. In monatomic modes, gas is introduced through a fixed leak valve directly into the ionization chamber. This mode is similar to conventional ion guns such as the EX06.

It is advisable to periodically check operation and alignment values to ensure optimum operation. This can be done using the ion gun details window, as described in the main body of the manual (Maintenance & Troubleshooting).

UPS

Overview

Introduction

UV photoelectron spectroscopy (UPS) is a related technique to XPS. Instead of X-ray photons, ultra-violet radiation is used to generate photoelectrons from the sample. These are in the kinetic energy range 0-50 eV, and correspond to the valence electrons, those that are involved in bonding.

As the process is the same as XPS, signal generated by the X-ray source will impact on the UPS data being collected, and so the X-ray source should be turned off when collecting UPS data. However, it is not necessary to turn off the UV source when collecting XPS data, as the UV photoelectrons are too low in energy to affect most XPS analysis.

More information on UPS can be found at www.xps-simplified.com.

Introduction for UPS Operation on Nexsa System

Flush/Prime the Gas Lines

It is important to prevent contamination of the UV source by using high purity research grade inert gas (99.999% pure helium, although other inert gases may be used) connected from the bottle regulator at approximately 1 bar (2 bar maximum) input pressure by clean stainless steel 6 mm pipe to the compression fitting on the service panel of the system (see [Figure 74](#)).



Figure 74: Service Panel Helium Input Connector Before and After Connecting 6 mm Metal Helium Line

It is recommended that the internal gas lines to the UV source are pumped and flushed with helium prior to operation, to prevent contamination of the UV source. This operation is automatically performed by pressing the **Flush He Gas Lines** button on the vacuum details page (**Figure 75**), which alternately opens V10A and V10B (**Figure 76**) to pump and recharge the gas supply line to the UV lamp; the pump and flush is repeated three times and takes approximately 2 minutes to complete. The final step flushes the backing line with argon to remove any remaining helium that may slow pumping performance. The UV source is then ready to use. It is recommended that another flush cycle is performed before using the source if it has not been used for more than a few days.

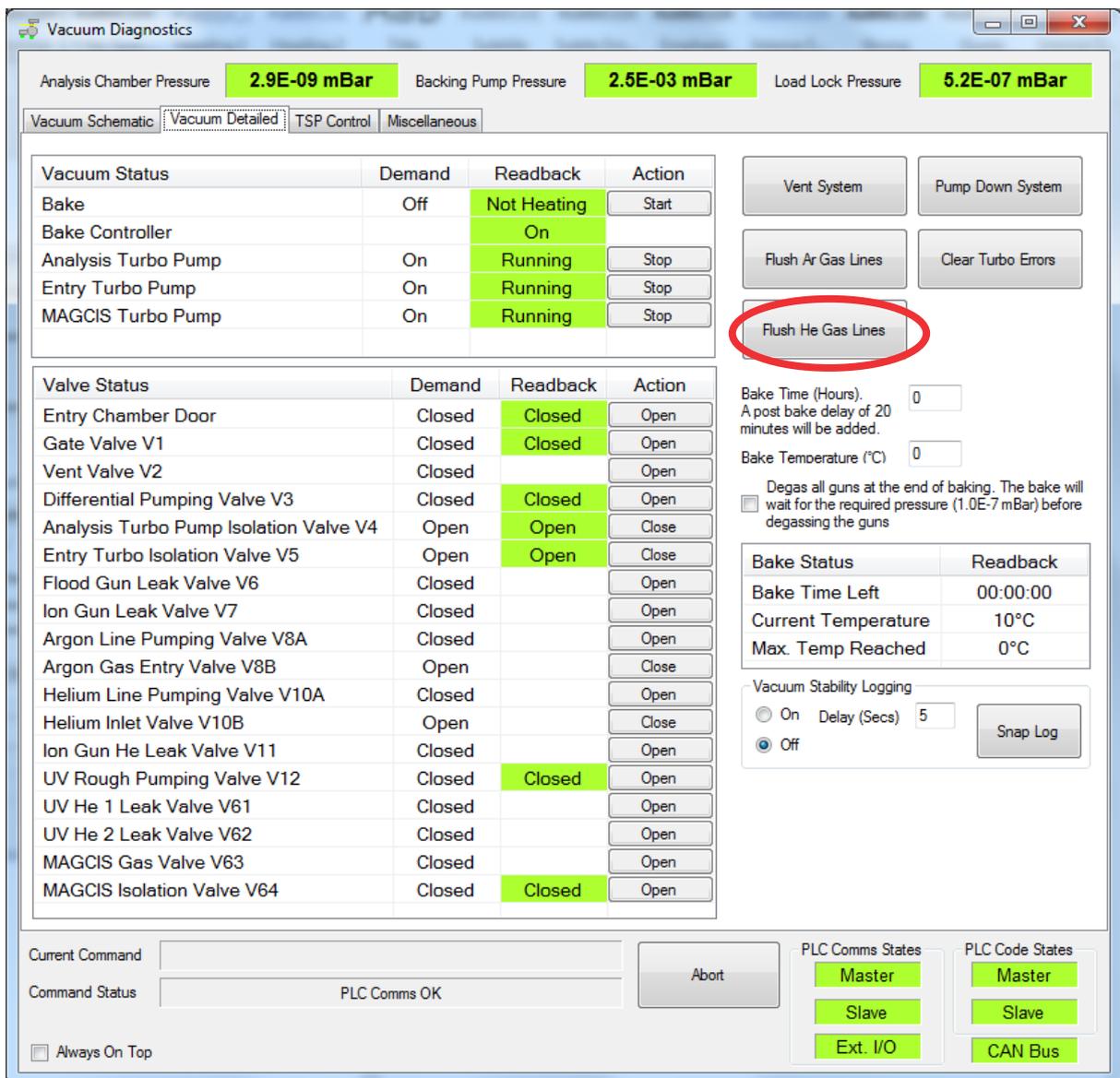


Figure 75: Avantage – Vacuum Diagnostics Page

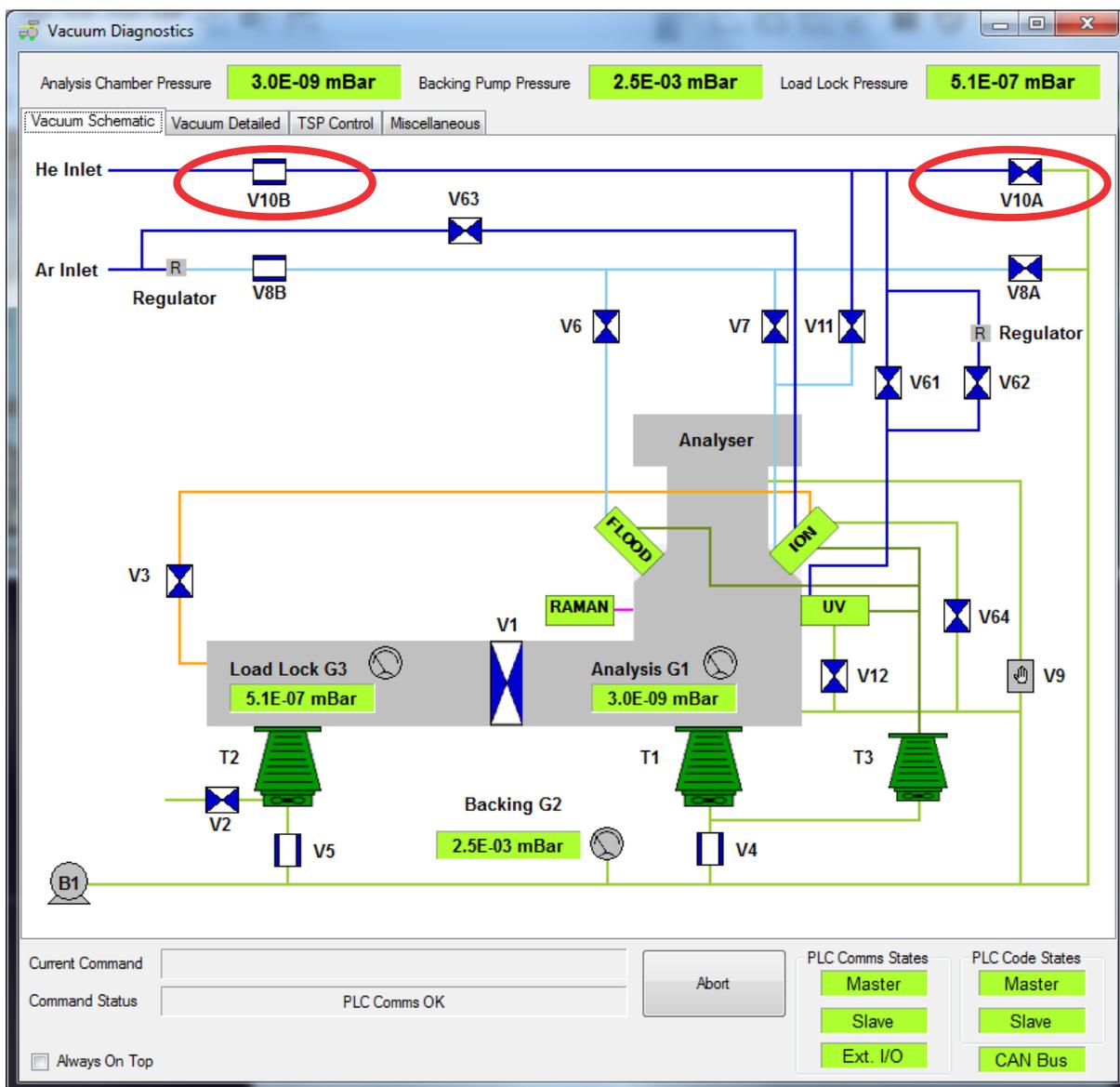


Figure 76: Gas Pumping V10A and He Gas Supply Valve V10B are Alternately Opened During Pump and Flush

Run the Source and Acquire UPS Data from the Avantage Experiment

UPS spectra may be acquired from the Avantage experiment/acquisition pane, with automated source control. This may be used as a series of XPS/UPS and depth profile experiment objects to allow programmed UPS control.

IMPORTANT

The X-ray source will produce photoelectrons that will impact the collected UPS spectrum. The X-ray source must be turned **OFF** when collecting UPS data. It is not generally necessary to turn off the UV lamp when collecting XPS data.

1. Start a new experiment from the Avantage file menu
2. Click on the Sources icon (**Figure 77**) and select **UV Source**. The source settings for **He I / He II** may be selected at the **Source Properties** page in the lower pane (**Figure 78**), together with enabling the flood gun for insulator analysis, and the sample bias if the sample bias stage is being used.



Figure 77: Sources icon

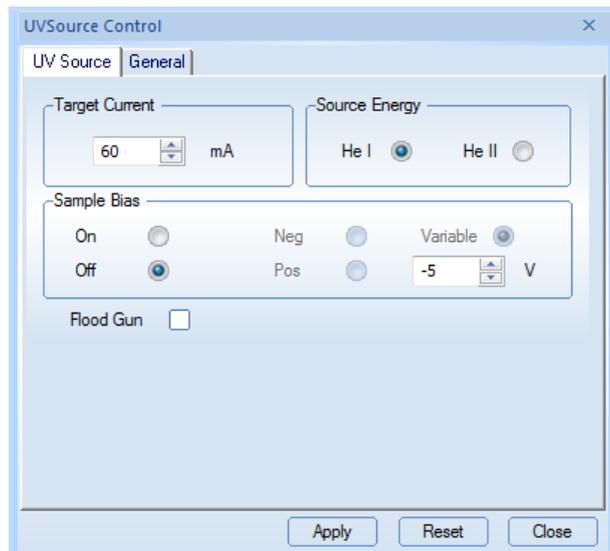


Figure 78: Sources Properties Page

3. Add a sample position 'Point' to the UV Source object (**Figure 80**), and a spectrum acquisition object using the **Multi-Spectrum Tool** (note only the **UPS He Survey** control is used for UPS), which will use the default parameters.

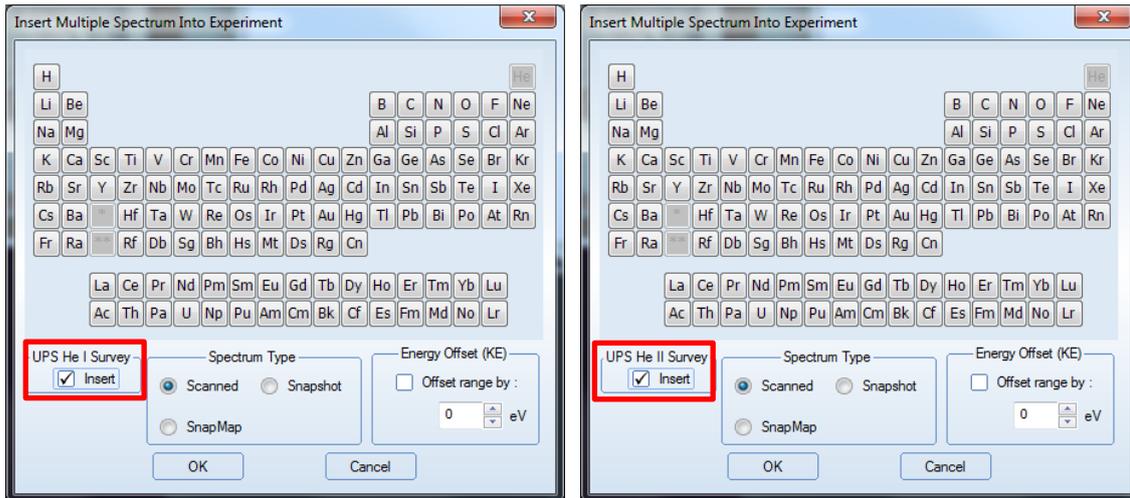


Figure 79: Multi-Spectrum Tool for He I and He II sources



CAUTION

Beware that using pass energy of greater than 4 eV when using He(I) may lead to extremely high count rates at the electron detector which can rapidly age the detector channel-plates and could require their premature replacement.

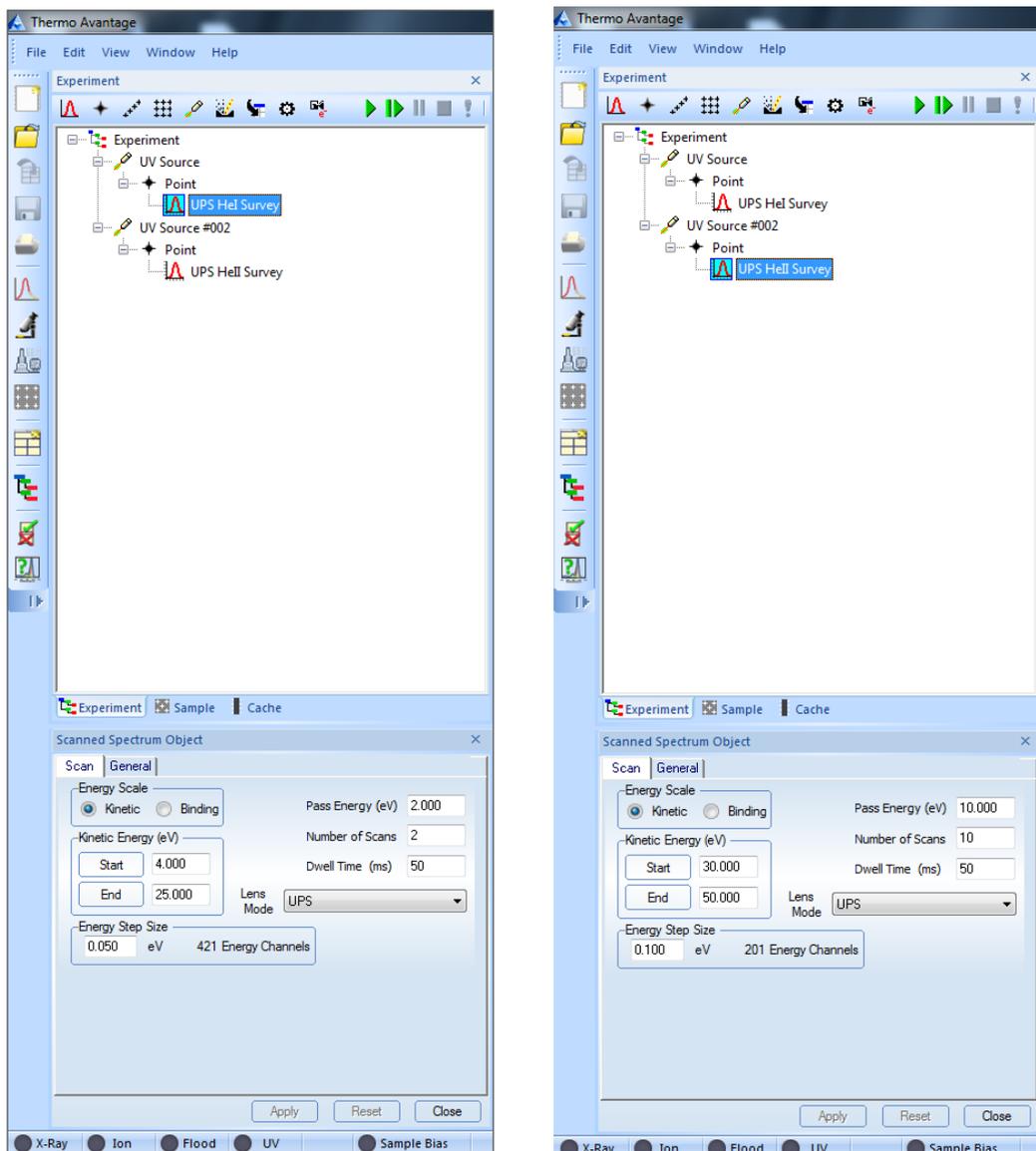


Figure 80: Example of UPS experiments with default parameters

Example data for standard samples can be found in the data collected during system installation and at www.xps-simplified.com.

The UV source will remain on at the end of acquisition unless a Gun Shut-down object is added to the acquisition tree, from the sources drop-down menu (**Figure 81**).



Figure 81: Adding Gun Shutdown Object

UV Source Details Page

The source control details page is accessed from the Avantage sample tab and clicking the “UV Source” icon on the drawing of the system: This is only required for troubleshooting / engineer usage.

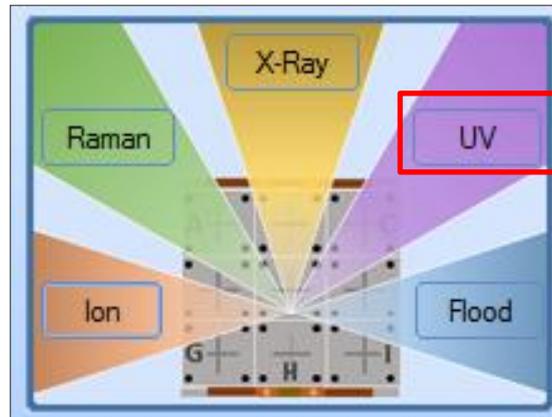


Figure 82: Avantage Source Control

Troubleshooting

If the source will not light, then it is advised that the following checks are made:

1. Communications between *Avantage* and the UV Power supply are good (no red indicator on the status bar).
2. The vacuum trip has been reset (**Details** page, **Trips** page and press **Reset**).
3. Pure Helium is connected to the system at 1 bar, with no leaks on the metal line.
4. The gas lines have been pumped and flushed using the **Flush He Lines** procedure.
5. The UV high voltage source cable is connected to the UV lamp.

If **He I** mode operation lights without problems but **He II** operation does not stay lit, then it is likely that the **He II** regulator is set too low. This is located behind the panel above the entry-lock, which can be removed by sliding upwards. Increase the regulator setting in steps of 0.01 MPa (0.1 bar) by lifting the black adjustment knob on the top of the regulator and turning clockwise and re-test **He II** operation. If this does not achieve stable He II operation then refer to Thermo Fisher Scientific support contacts.



Figure 83: He II regulator location

ISS

Introduction

Ion scattering spectroscopy (ISS, also known as LEIS – low energy ion scattering) is used to obtain elemental information from the first atomic layer of the surface. It uses noble gas ions, typically He^+ , which are directed at the surface from the ion gun, and the scattered ions are detected by the hemispherical analyser. To do this, the analyser runs in the opposite polarity to when collecting electrons, so that positively charged ions are collected at the detector. More information can be found at www.xps-simplified.com.

The extreme surface sensitivity means that samples must be clean to detect the actual surface and not any surface overlayers which may be present.

Ion scattering spectroscopy (ISS) operation on Nexsa is fully automated. This section describes preparing the system for ISS and how to collect ISS data. The same procedures apply whether the EX06 or MAGCIS ion sources are fitted. This section will be based around using helium, but ISS can also be collected using neon or argon by connecting to those gases to the helium input.

Preparing for ISS Operation on Nexsa System

Flush/Prime the Gas Lines

It is important to use high purity research grade inert gas (99.999% pure helium) with the ion source to prevent premature failure of the filaments. The gas should be connected from the bottle regulator at approximately 1 bar (2 bar maximum) input pressure by clean stainless steel 6 mm pipe to the compression fitting on the service panel of the system (see Figure 84).



Figure 84: Service Panel Helium Input Connector

The internal gas lines to the ion source need to be pumped and flushed with helium prior to operation. This operation is automatically performed by pressing the **Flush He Gas Lines** button on the Vacuum Detailed page of the Vacuum Details window (**Figure 85**) to pump and recharge the gas supply line to the ion source. The pump and flush is repeated three times and takes approximately 2 minutes to complete.

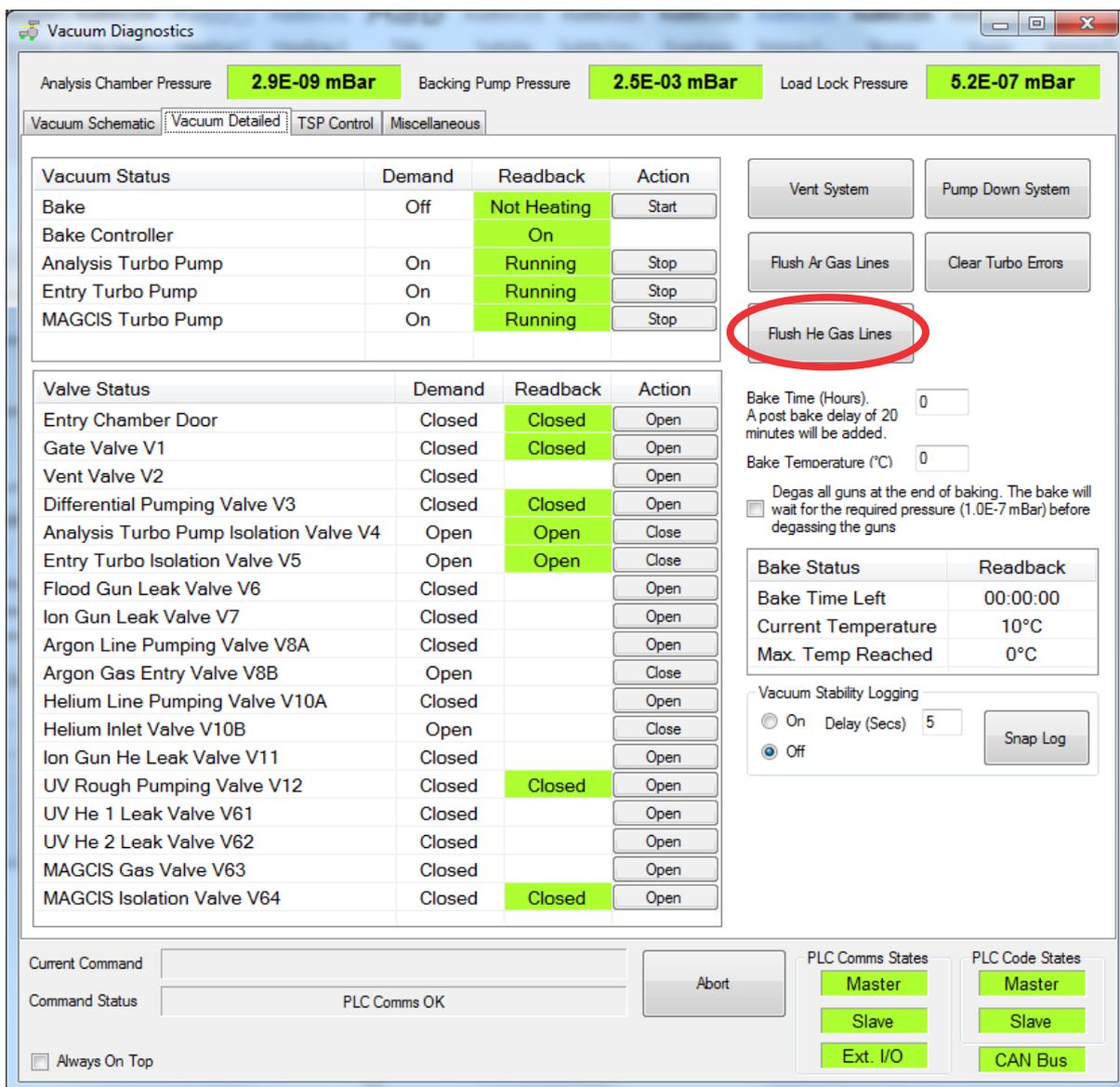


Figure 85: Vacuum Detailed Page

ISS system calibration

To ensure the best quality data it is advisable to calibrate the ion source if it has not been used for ISS recently, or if the system has been vented for a service. There are two steps to this procedure. The first is to align and focus the ion beam (as described in the Maintenance and Troubleshooting section), the second measures the ion beam energy to enable calibration of the peak positions. The second routine also checks the scattering angle, and both this parameter and the ion beam energy are stored with collected spectra for offline processing.

Align and focus the ion beam

Open the ion gun details page, and select the **Filament** tab. Choose **ISS** from the **Auto Setup** drop-down menu, and click **Run**.

Measure the ion beam energy

Open the ion gun details page, and select the **Filament** tab. Click **Calculate** in the **ISS Source Energy** section

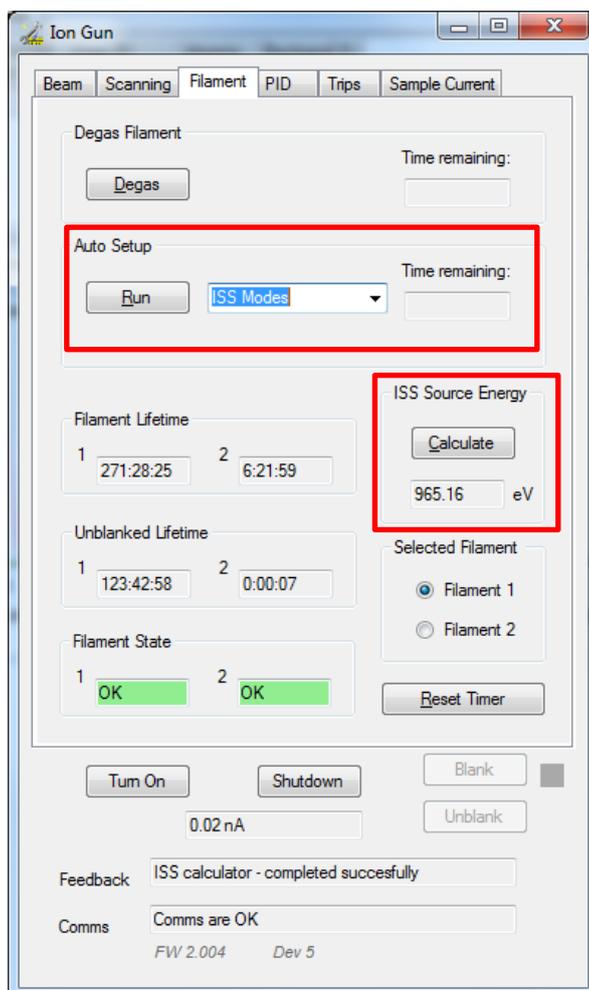


Figure 86: Ion Gun details page, Filament tab, with ISS controls highlighted

Collecting ISS Data using the Nexsa System

Collecting ISS data follows a very similar procedure to collecting XPS spectra. As with XPS, the structure is Source -> Position -> Spectrum.

To start, select **ISS source** from the **Source** menu above the experiment tree. This will insert the **ISS Source** object into the

experiment tree. The properties page has settings to enable the charge compensation system (if the material being analysed is an insulator).

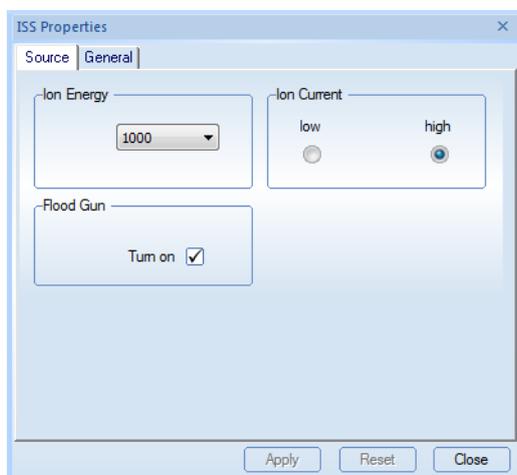


Figure 87: ISS Source properties page

Define the position for analysis by adding a point, and then select the **Multi-Spectrum Tool**. In an ISS experiment, the survey object changes to be an ISS survey, and this should be inserted (currently the periodic table will not insert spectra with the correct parameters and should not be used).

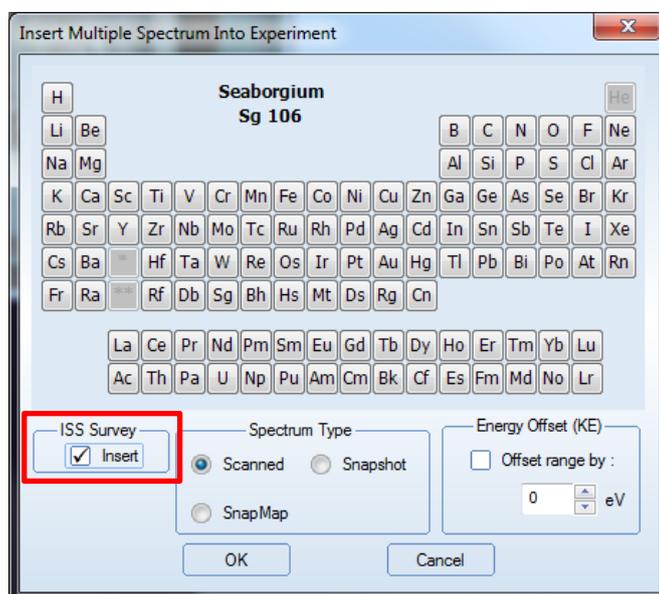


Figure 88: Multi-Spectrum Tool in an ISS experiment

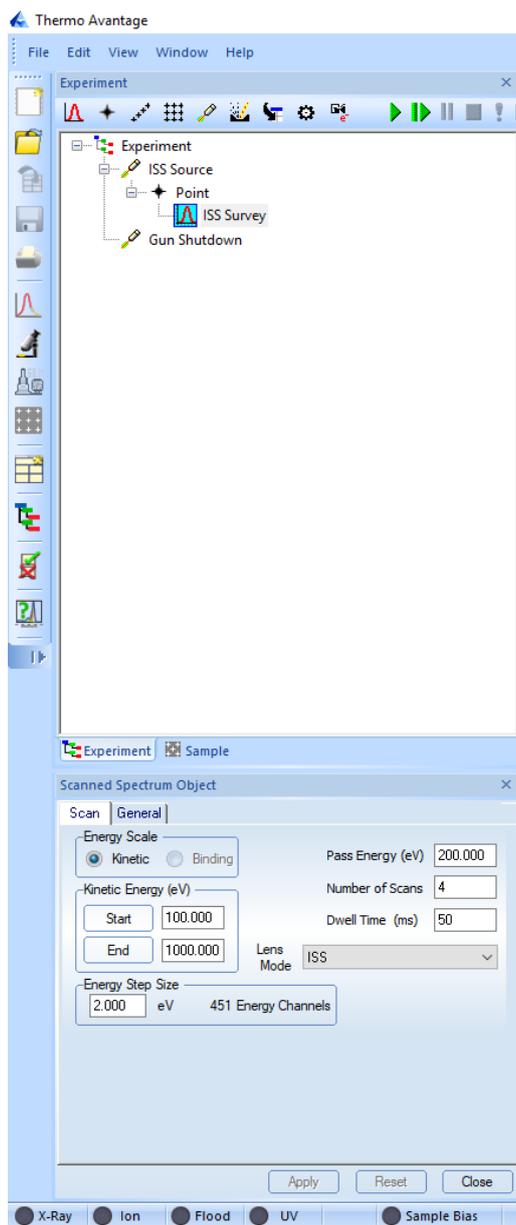


Figure 89: A typical ISS experiment

ISS is very surface sensitive, and the sample surface cleanliness is crucial in obtaining a good spectrum. It is also important to note that the He^+ ion beam used for the analysis will slowly etch the sample, and so changes may be observed from scan to scan if layers are very thin (this is most frequently seen if there is an incomplete carbon contamination layer on the surface).

Example data for standard samples can be found in the data collected during system installation and at www.xps-simplified.com.

After the acquisition has completed, the ion source is blanked. Turning off the source is controlled from a **Gun Shutdown** object. It is recommended that the ion source is turned off when not required.

REELS

Overview

Introduction

Reflected electron energy loss spectroscopy (REELS) operation on Nexsa is automated, under user instructions which are described in this section. REELS is a scattering technique, similar to ISS, but using an electron beam as the probe. It gives information on electronic properties, and can be a help in understanding the contribution of loss-features to an XPS core level spectrum. More information can be found at www.xps-simplified.com.

As REELS relies on detecting scattered electrons, it is important to turn off the X-ray source when collecting REELS data, as the XPS signal will be merged with the REELS results.

Collecting REELS Data using the Nexsa System

Collecting REELS data follows the same procedure to collecting XPS spectra. As with XPS, the structure is Source -> Position -> Spectrum.

To start, select **REELS source** from the **Source** menu above the experiment tree. This will insert the **REELS Source** object into the experiment tree. The properties page has settings to enable the charge compensation system (if the material being analysed is an insulator).

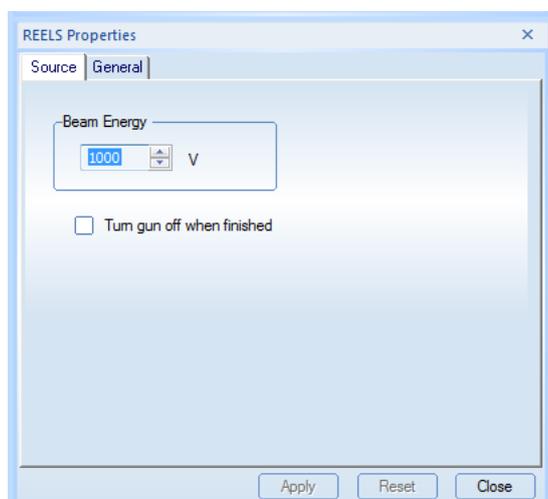


Figure 90: ISS Source properties page

Define the position for analysis by adding a point, and then select the **Multi-Spectrum Tool**. In an REELS experiment, the survey object changes to be an REELS survey, and this should be inserted (currently the periodic table will not insert spectra with the correct parameters and should not be used).

Note that the elastically scattered peak has a very high count rate and so pass energies of 15 eV or less should be used to prevent aging the detector. The default pass energy of 10 eV is recommended.

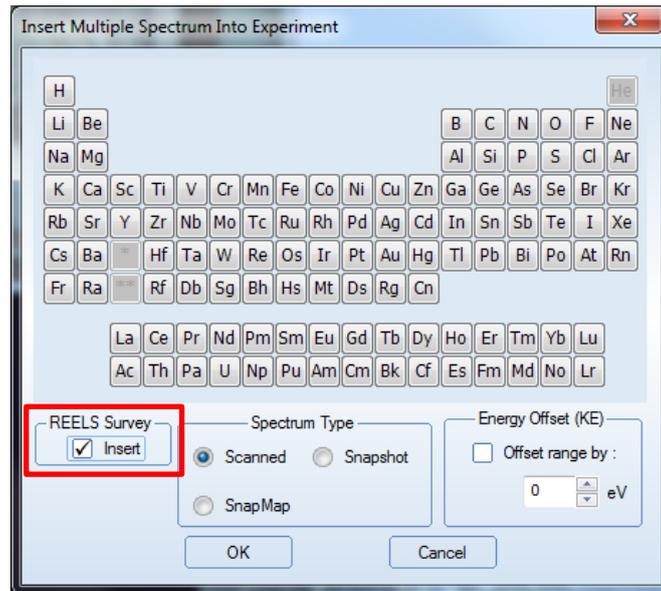


Figure 91: Multi-Spectrum Tool in an REELS experiment

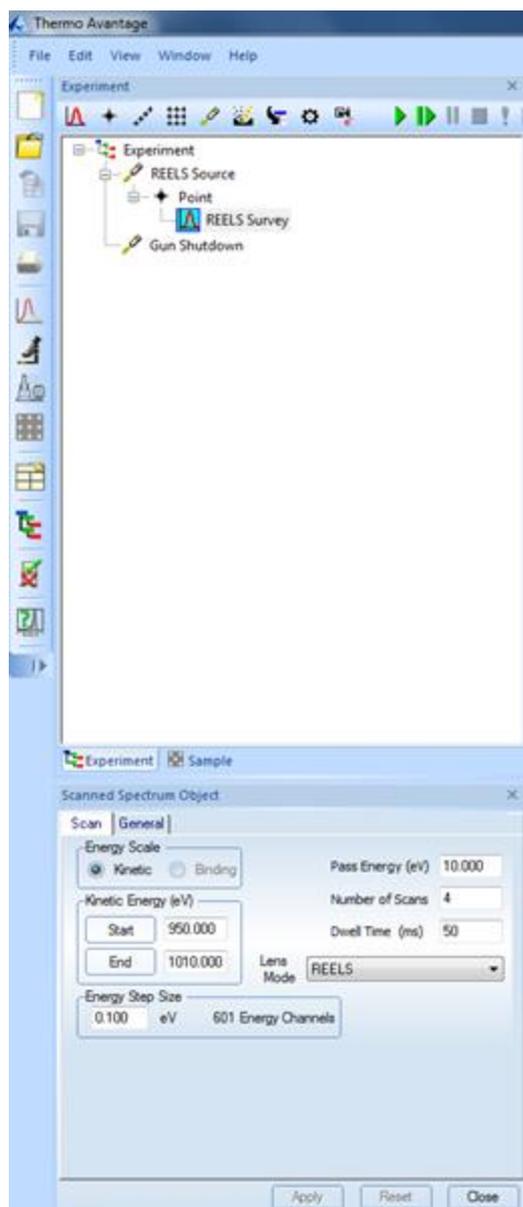


Figure 92: A typical REELS experiment

After the acquisition has completed, the electron source remains on. High energy electron beams may damage sensitive samples and so it is recommended that the source is turned off when not required. Turning off the source is controlled from the checkbox on the source object or by using a **Gun Shutdown** object.

Example data for standard samples can be found in the data collected during system installation and at www.xps-simplified.com.

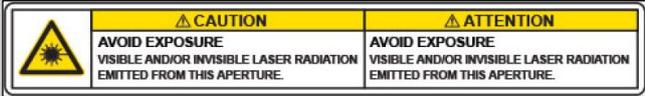
iXR Raman

General Safety Information

The iXR Raman on Nexsa has been designed and certified to have a Class 1 rating. No accidental exposure to the Laser beam is possible if the correct procedures are followed when operating and servicing this option. The iXR Raman unit has a classification rating of Class 3B, however suitable interlocking and tool access has been designed into the integration of this unit onto the Nexsa system to classify the safety status of the total integrated system as Class 1.

CAUTION **USE OF CONTROLS OR ADJUSTMENTS OR PERFORMANCE OF PROCEDURES OTHER THAN THOSE SPECIFIED HEREIN MAY RESULT IN HAZARDOUS RADIATION EXPOSURE.**

Product Labels

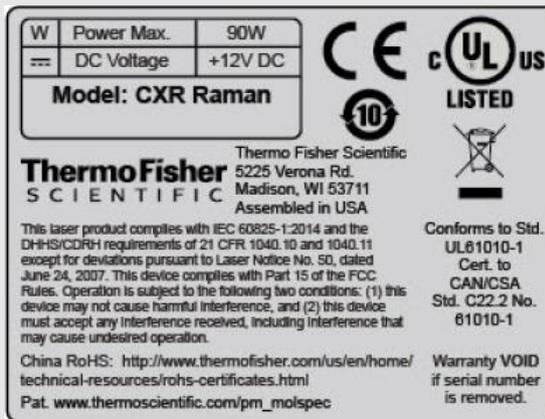
Letter	Label Image	Label Description
A		Avoid Exposure label for iXR unit
B		Power and Wavelength Description for iXR unit

C



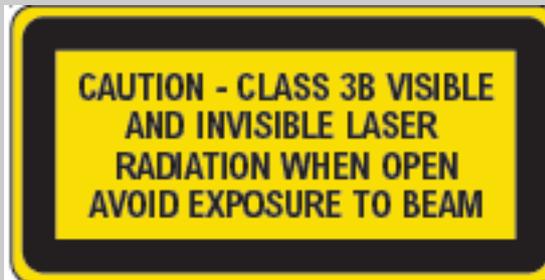
Exposure Caution label for iXR unit

D



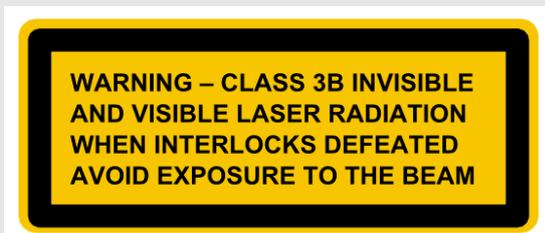
Rating Label

E



Exposure warning when removed

F



Interlock override warning

G



Class 1 LASER rating

Table 8: Product Labels

Product Label Locations

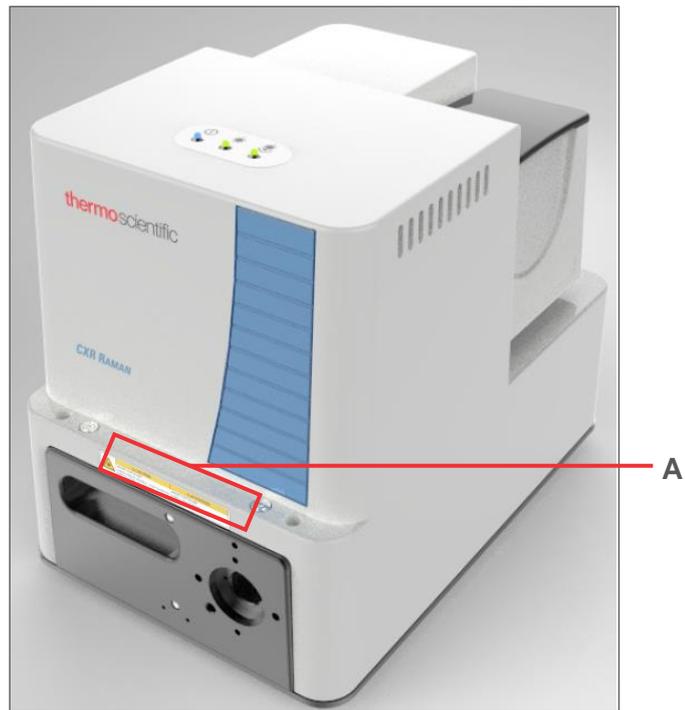


Figure 93: Safety Label Locations – A

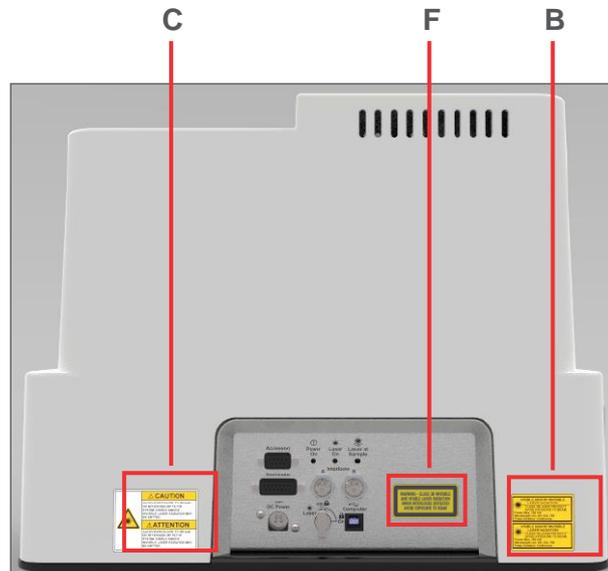


Figure 94: Safety Label Locations – B, C, F

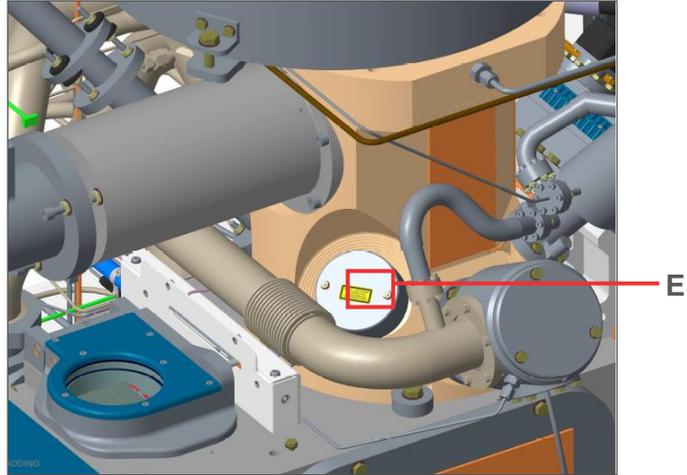


Figure 95: Safety Label Locations – E

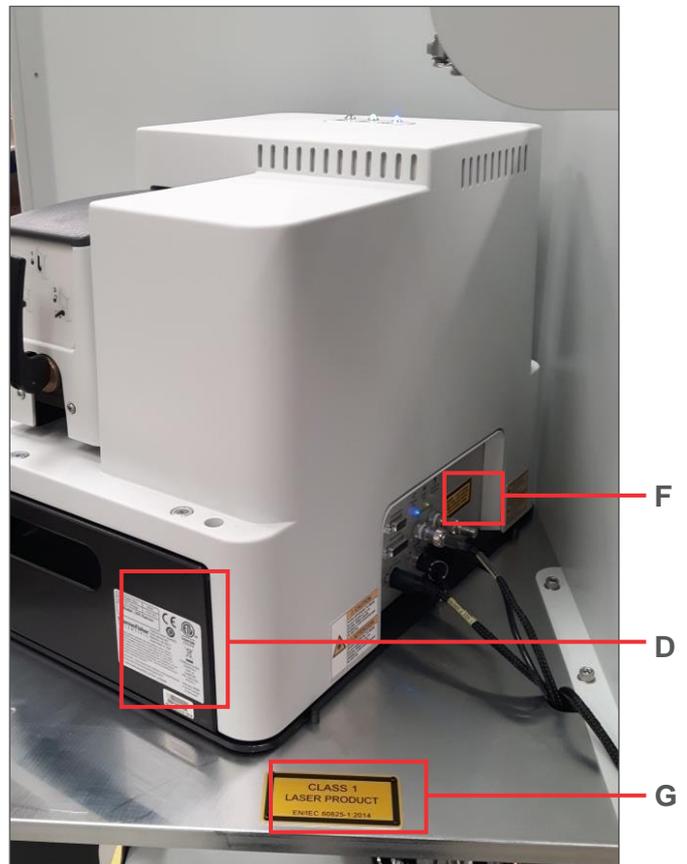


Figure 96: Safety Label Locations – F, D, G

Interlocks and Safety Covers

The iXR unit has a Class 3B Laser safety rating with internal interlocks present for this status, which can be found in the iXR Raman Spectrometer manual. This manual (iXR on Nexsa) details the additional interlocks present when integrating the iXR unit onto the Nexsa system. These additional interlocks increase the Laser safety rating of the combined item to Class 1, by preventing the Laser beam from the iXR system from exiting the Nexsa system and ensuring that the iXR unit can only be run once positioned in place on the Nexsa.

The additional interlocks take the form of both direct (in circuit) and indirect (via PLC) controls. In some cases double-redundancy is introduced via combination of direct and indirect switching.

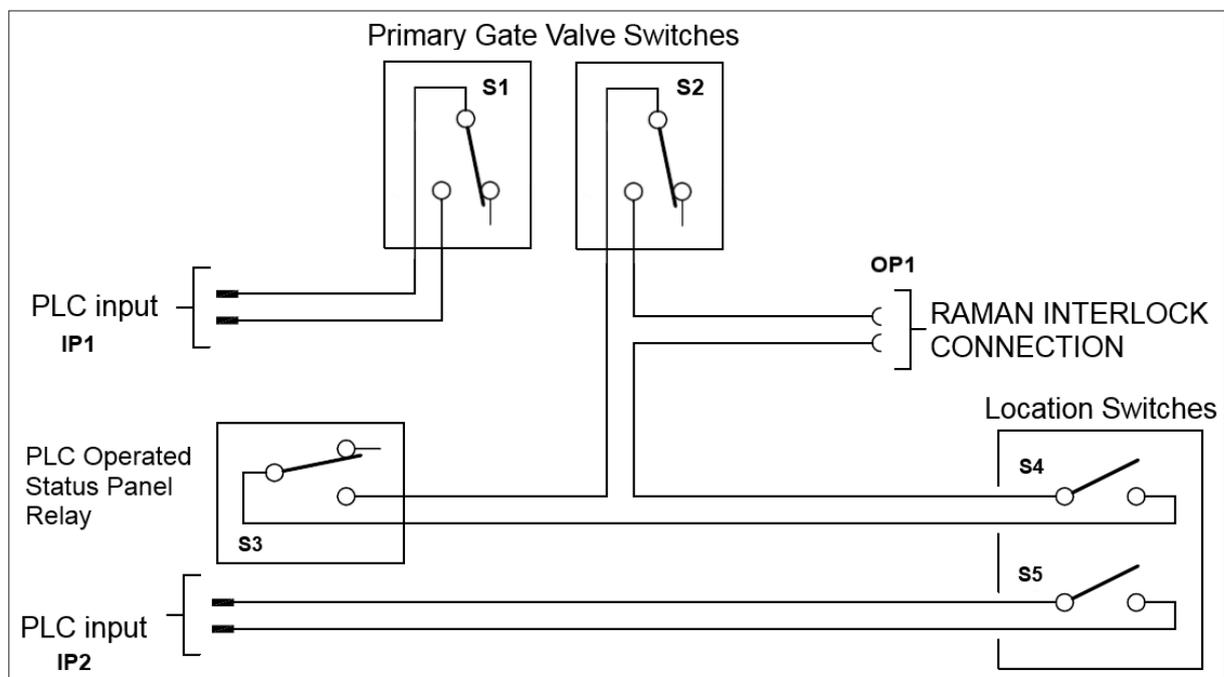


Figure 97: Interlock Wiring Schematic

Interlock Details

Switch	Type	Activation Details	Circuit Details
S1	NO	Physically activated contact switch. Closed circuit only when primary gate valve is closed.	Switches IP1 of PLC.
S2	NO	Physically activated contact switch. Closed circuit only when primary gate valve is in closed position.	Direct activation/deactivation of Raman interlock switch.
S3	NO	<p>PLC activated normally open relay, situated on NEXSA status panel.</p> <p>Activates to closed condition only when ALL of the following are all true:</p> <ul style="list-style-type: none"> • IP1 is closed circuit. • IP2 is closed circuit. • Vacuum system is pumped down (turbopump at speed). • <i>Avantage</i> software is open on the PC, and communicating with the PLC (Comms OK). • Status panel is connected to the PLC. 	<p>Direct activation/deactivation of Raman interlock input circuit based on these 4 conditions.</p> <p>Interlocks against turning on Laser if the unit is not located on the system or when the system is vented for engineer tasks.</p> <p>If the Status panel is disconnected from the PLC, power is lost to the relay and it will become open circuit automatically.</p>
S4	NO	<p>Coded magnetic switch.</p> <p>Closed circuit when Raman unit is correctly located in position.</p>	Direct activation/deactivation of Raman interlock switch.
S5	NO	<p>Coded magnetic switch.</p> <p>Closed circuit when Raman unit is correctly located in position.</p>	Switches IP2 of PLC

Table 9: Interlock Details

In the case of **any** of the above switches being open circuit, the Raman Interlock output (OP1) is also open circuit and the Laser is therefore deactivated and prevented from turning on, within the iXR raman unit. The combination of interlocks within this circuit prevents accidental exposure to the Laser beam, if all engineer procedures are followed correctly.

Introduction

The iXR Raman option on Nexsa is an assembly for recording Raman measurements on samples within the Nexsa system, consisting of four main parts:

1. Ultra-High Vacuum (UHV) lens assembly, mounted on the Nexsa system.
2. iXR Raman spectrometer.
3. Laser and Raman scatter beam path assembly.
4. Interlocks and Beam Exposure prevention measures.

The complete spectrometer assembly is aligned such that the Raman measurement area and position are matched to the smallest x-ray spot on the Nexsa system. This allows concurrent Raman and XPS measurements to be conducted if desired.

The iXR Raman unit parameters are controlled through Thermo Scientific OMNIC™ software, and data collection capability is provided through the Thermo Scientific Avantage™ software.

Electrical Connections



Figure 98: Electrical connections

1. Accessory connection
2. Illuminator output
3. DC Power Input (12V DC, 100W)
4. Laser lockout safety keyswitch
5. USB PC interface connection
6. Interlock input connections

NOTE: The Interlock input connector A must be connected to the Nexsa cable TG1420-2 in order for the user to be fully protected from Laser exposure. Do not override this interlock at any time.

Illuminators

There are three illuminators, located both on the top of the iXR unit on the main illumination display panel, and on the electrical connections panel on the side of the unit above the electrical connections.

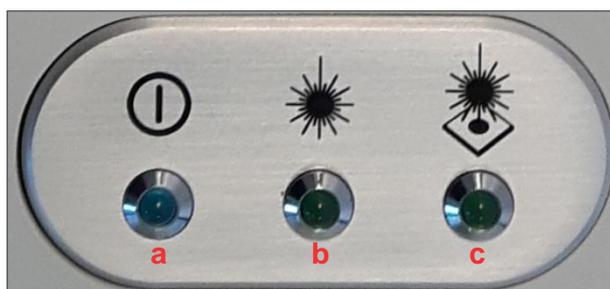


Figure 99: Illuminators

- a. Power
 - Illuminated when system is turned On.
- b. Laser On
 - Illuminated when Laser is operating (On).
- c. Beam at sample display
 - Illuminated when beam is being transmitted out of the Raman unit.
 - Off when beam is being blocked inside the unit.

Operation

All operations such as turning the Laser on/off and setting experimental parameters are completed using the Thermo Scientific *OMNIC for Dispersive Raman (OMNIC)* software.

Data collection can be completed using Thermo Scientific *Avantage* software, but the experimental parameters and Laser power must be set up in *OMNIC* prior to collecting via this method, and *OMNIC* must remain open during all data collection operations.

OMNIC For Dispersive Raman (OMNIC)

OMNIC for Dispersive Raman is started from the Windows™ Start Menu.



Figure 100: OMNIC for Dispersive Raman

Once clicked, *OMNIC* will open and the Raman unit will reset ready for operation. This involves resetting of some motors and other operations; as a result *OMNIC* startup may take up to 2 minutes.

If the Raman unit USB connection or power is not present during *OMNIC* startup, you will get an error stating that the unit is not available. If this is the case then no operation of the unit is possible until the connection is restored and the *OMNIC* software is restarted.

OMNIC Main Screen

OMNIC has one main screen which all functions can be accessed from, either through shortcut buttons or the **File** menu. Details of the individual functions can be found within the *OMNIC Help* file, but the most commonly used functions are highlighted in this section.

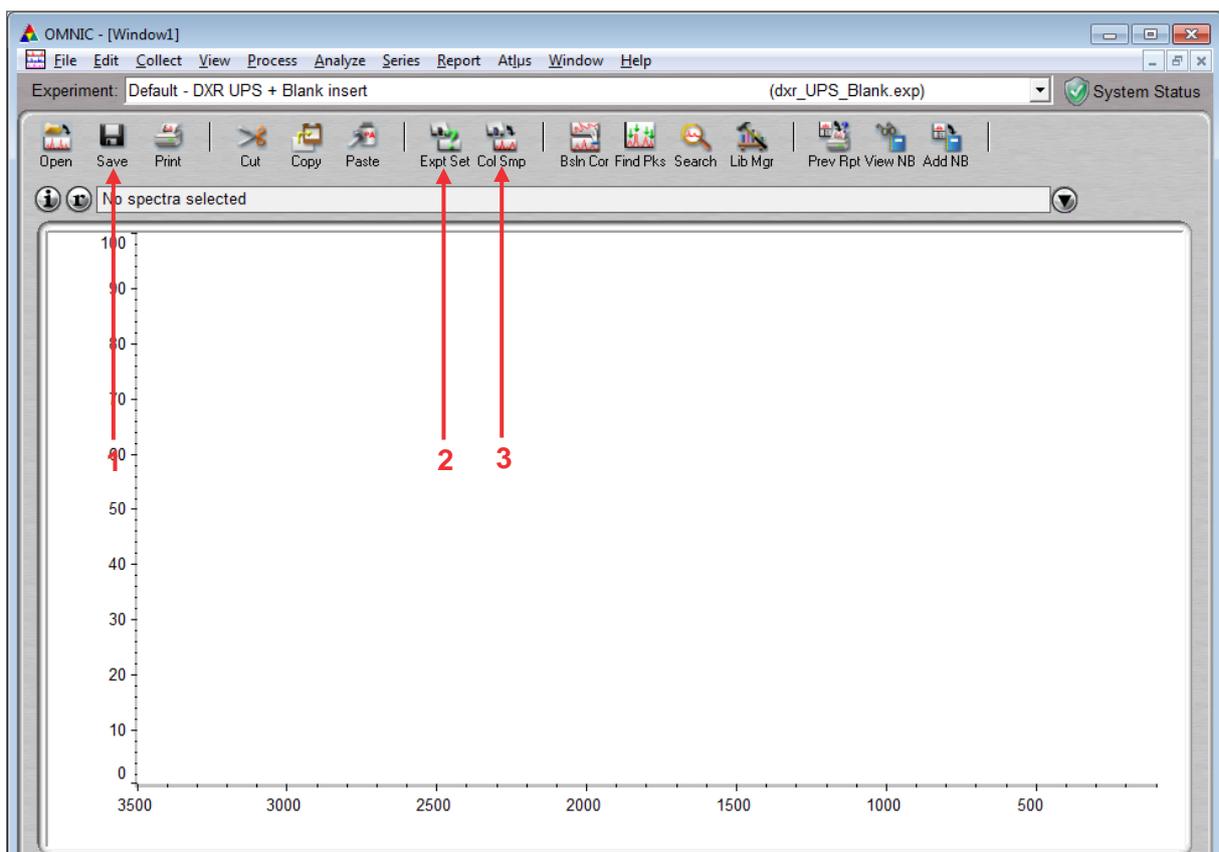


Figure 101: OMNIC Main Screen

1. **Save.**

Clicking this button saves the currently selected Raman spectrum, displayed in red in the active window.

NOTE: **No spectra are saved unless specifically directed to by the user, unless the automatic file saving is activated in the *Collect* experimental parameters.**

2. **Experiment Setup.**

This button opens a dialog box which allows control and setting of all experimental parameters, including Laser power and ON/OFF control. There are multiple tabs on this dialog box, the number of which varies depending on the software components installed.

- **Collect** tab provides access to changing experiment collection parameters (see [Defining Collection Parameters](#) page **108**).
- **Bench** tab provides access to Laser parameters but also provides a real-time preview of the Raman spectrum from the sample using the current collection parameters.

Experiment Setup dialog can also be accessed through the **File** menu by clicking **Collect > Experiment Setup** or by keyboard shortcut, CTRL + E.

3. **Collect Sample.**

Clicking this button collects a Raman spectrum using the currently saved experimental parameters.

This method is also accessible from the **File** menu by clicking **Collect > Collect Sample**, or by keyboard shortcut CTRL +S.

Switching the Laser On

1. Open the **Experiment Setup** dialog.
2. Switch to the **Bench** tab (**Note: There may be a slight delay while the spectrometer adjusts ready for collection**).
3. Click the drop down bar in the **Laser** row of the parameter table. Select **On**.

NOTE: **If the drop-down bar button is not visible, double-click Off to display.**

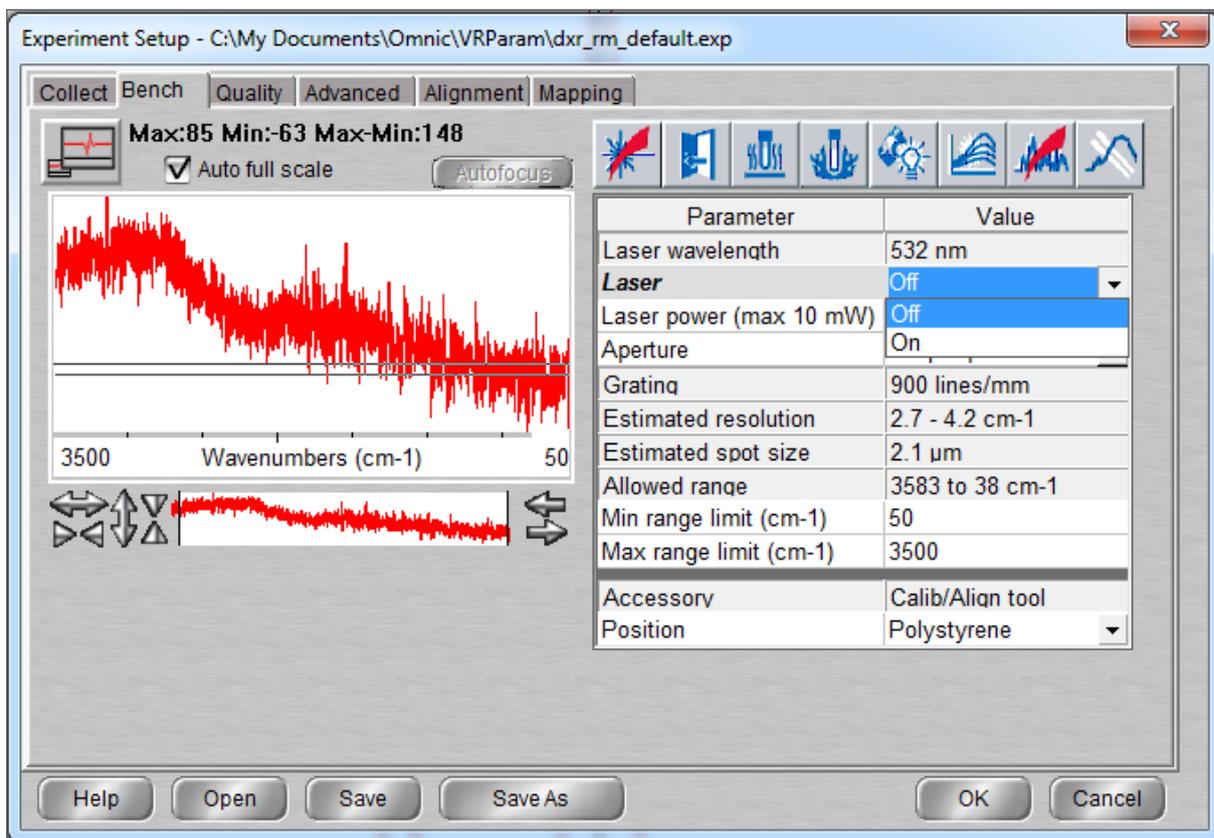


Figure 102: Experiment Setup Window

4. The Laser will turn on. A pop-up dialog will appear counting down the time to readiness. Once this has disappeared the unit is ready to use for data collection.
5. Close the **Experiment Setup** dialog by clicking **OK** (unless other experimental parameters, e.g. Laser power, are being setup at the same time).

NOTE:

The Laser will not turn on if any interlock is in the open state – interlock status is shown in one of the icons above the Parameter/Value table, as shown in the image below.

Switching the Laser Off

This operation is the same as switching the Laser **On**, but select **Off** from the drop-down bar. The Laser will switch off immediately once this is selected.

Parameter	Value
Laser wavelength	532 nm
Laser	On
Laser power (max 10 mW)	Off
Aperture	On

Figure 103: Switching the Laser Off

The Laser is a consumable component. It is recommended that the Laser be turned off whenever the spectrometer unit is not being used for data collection.

Setting the Laser Power

The output Laser power can be set to any value in 0.1 mW steps up to the maximum power specified for the Laser being used. The maximum power will be displayed within the *OMNIC* software, which will automatically detect the Laser type being used.

Laser name	Wavelength (nm)	Maximum power (mW)
455	455	6
532	532	10
785 High Brightness	785	24

Table 10: Laser Wavelength and Maximum Power

1. Open the **Experiment Setup** dialog and switch to the **Bench** tab.
2. Double-click in the **Value** column of the parameter **Laser Power**.
3. Type a Laser power and hit the return key. The stated Laser power will be set immediately.

Alternatively use single mouse clicks on the Up/Down arrows to the right of the Laser power number to adjust the Laser power in small steps. The power will change on each click.

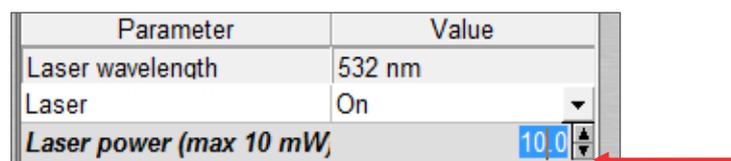


Figure 104: Adjusting Laser Power Using Arrows

Choosing an Aperture for Data Collection

The iXR Raman Spectrometer unit has four apertures at the entrance to the Raman spectrometer; two slits (25 μm and 50 μm wide), and two pinholes (25 μm and 50 μm diameter). Changing the aperture varies the amount of Raman light collected by the spectrometer from the sample, therefore changing intensity and resolution of the Raman signal processed.

For the Nexsa system, best results are usually obtained by using the 50 μm pinhole. This maintains a reasonable level of Raman signal

intensity while cutting out much of the scattered light from areas outside those desired from the sample surface.

To set the desired aperture:

- Open the **Experiment Setup** dialog and switch to the **Bench** tab.
- Click the drop-down arrow at the far right of the value column for the **Aperture** parameter. (**Note: If drop down arrow is not visible, double-click in the value box to activate**).
- Select the desired aperture from the drop-down menu.

Parameter	Value
Laser wavelength	532 nm
Laser	On
Laser power (max 10 mW)	10.0
Aperture	50 μ m pinhole
Grating	25 μ m slit
Estimated resolution	25 μ m pinhole
Estimated spot size	50 μ m pinhole
...	50 μ m slit

Figure 105: Selecting the Desired Aperture

Defining Collection Parameters

1. Open the **Experiment Setup** dialog.
2. Switch to the **Collect** tab.

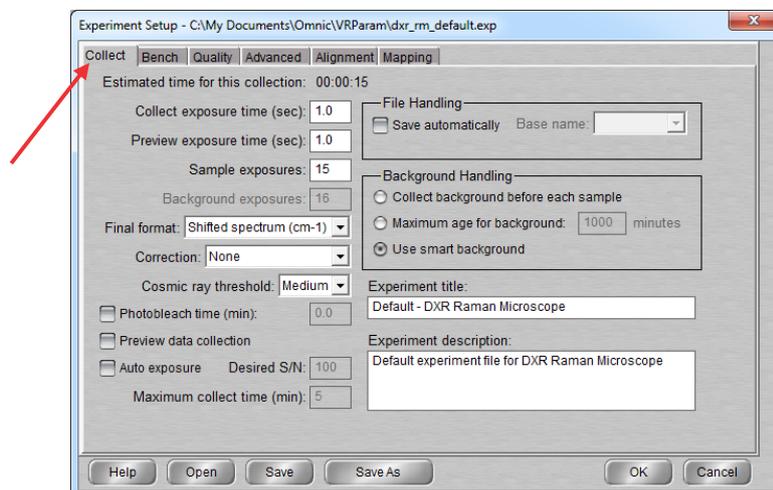


Figure 106: Experiment Setup Collect Tab

3. The following table outlines the parameters available for data collection.

Parameter	Explanation	Comments
Collect Exposure Time (sec)	Collection time for each full spectrum frame.	Shorter exposures of 1-2 reduce the chance of cosmic ray events in spectra.
Preview Exposure time (sec)	Collection time for each full spectrum in the Bench tab preview pane.	Minimum is 1s.
Sample Exposures	Number of times the same full spectrum is collected.	Total collection time is Collect Exposure Time x Sample Exposures. Shorter exposure times of 1-2s and multiple exposures recommended (<i>OMNIC</i> has automatic cosmic ray removal if occurring in frames after the first).
Final Format	X-axis scale for data display	Default is Shifted Spectrum (cm ⁻¹)
Correction	Indicates the type of spectrum correction applied prior to display.	None – no processing. Fluorescence – corrects for backgrounds due to sample fluorescence.
Photobleach time	Refer to <i>OMNIC</i> Help File.	Allows for stabilisation of samples prior to analysis.
Preview Data Collection	Refer to <i>OMNIC</i> Help File.	None
File Handling	Refer to <i>OMNIC</i> Help File.	Allows automatic saving of files.
Background Handling	Selects source for spectrometer dark-field correction.	Use Smart Background recommended – the dark-field background is collected and updated while the unit is on but in a passive state.
Experiment Title	Refer to <i>OMNIC</i> Help File.	Recommend not changing.
Experiment Description	Refer to <i>OMNIC</i> Help File.	Allows saving of description of experiment within the saved data file.

Table 11: Parameters Available for Data Collection

4. Click **Save**.

NOTE:

If **Save** is not clicked, then the parameters will be used for the current experiment run but will not be restored if **OMNIC** is restarted at any point.

5. Close the **Experiment Setup** dialog.

Data Collection

Data Collection Using *OMNIC*

1. Click the **Collect Sample** button within *OMNIC*.
2. A dialog will appear asking for the experiment title to be entered. Default text is Date and Time (including timezone). The **Spectrum Title** is the data description text that will appear in the display window when the data is displayed, and can be changed at a later time if desired.



Figure 107: Collect Sample Screen

3. Type the desired spectrum title and click **OK** to start data collection.
4. Once data collection is complete you will get a message asking if the data should be added to the active display window.

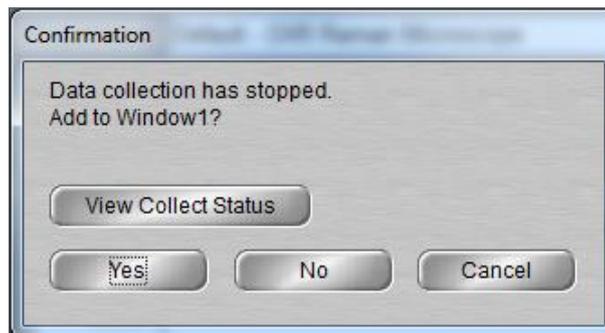


Figure 108: Confirmation Screen

5. Click **Yes** and the data will be displayed on the screen.

NOTE: Data is not automatically saved, so must be saved manually (unless the automatic saving has been set in the Collection parameters).

Data Collection Using *Avantage*

1. Set up all experimental parameters (Laser On and power set, exposure times etc.) using *OMNIC* software. Leave the *OMNIC* software open but all dialog boxes closed.
2. Ensure that *OMNIC* software has a data display window named "Window1". If not then create Window1 by selecting **Window > New Window** from the file menu (or keyboard shortcut CTRL + N) and entering the title for the window in the dialog as "Window1".

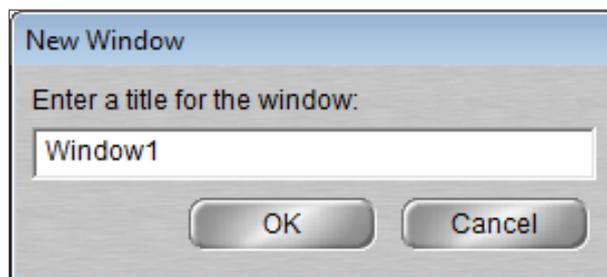


Figure 109: New Window Screen

3. Set up an *Avantage* experiment for XPS as normal.
4. Insert a **Raman Spectrum** object into *Avantage* at the point when the Raman spectrum is desired to be collected.

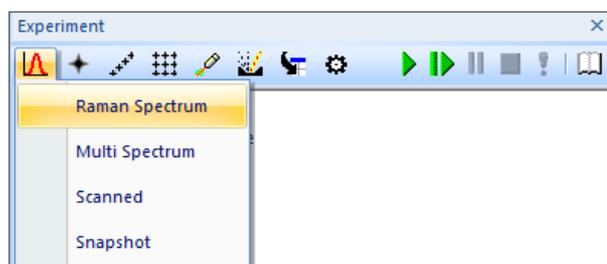


Figure 110: Raman Spectrum Object

NOTE: The Raman spectrum object must be inserted under a point object, as per XPS spectra.

5. Run the experiment. The Raman data collection will be undertaken at the specified point in the experiment.
6. Data will be automatically saved with a Time and Date filename, in the same folder as the *Avantage* data for the same point.

Potential Factors Affecting Raman Data Quality

Some aspects of the Nexsa hardware setup may lead to a detrimental effect on Raman spectral data quality, and therefore must be considered when setting up a Raman data collection experiment.

Factor	Action Required	
	<i>OMNIC</i> Data Collect	<i>Avantage</i> Data Collect
Light from charge compensation source	Turn off flood gun manually.	Insert Gun shutdown for flood source prior to Raman spectrum collection.
Fluorescence under X-ray illumination	Turn off x-ray source.	Insert Gun shutdown for x-ray source prior to Raman spectrum collection.
Fluorescence under UV illumination	Turn off UV lamp.	Insert Gun shutdown for UV source prior to Raman spectrum collection.
Light from Chamber LEDs	Turn off chamber LEDs manually. (Note there are 2 LED light sources which could be turned on).	No action required (LEDs will turn off automatically).

Table 12: Factors Affecting Raman Data Quality

Changing Laser Wavelength

From time to time it may be beneficial to use different Laser excitation wavelengths for collecting Raman spectra. Two alternative Laser wavelength kits, 455 nm and 785 nm, are available to purchase. Each kit consists of Laser assembly, grating assembly and filter assembly for the wavelength quoted. This section details how to change between the different Laser wavelengths on the iXR Raman unit.

1. Ensure that the existing Laser is turned off and the OMNIC software is closed.
2. Remove the right rear dark grey cladding panel to gain access to the Raman unit. To do this, lift the panel upwards by 2 - 3 cm then pull the panel towards the rear of the system to disengage from the mounting points, then lift further and pull away from the system in both directions.

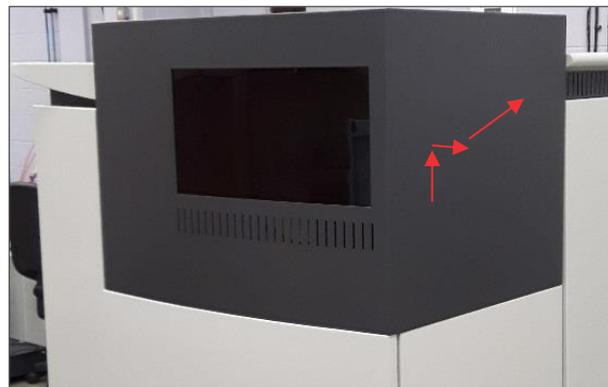


Figure 111: Removing the Grey Cladding

3. Undock the existing Laser and remove. Set to one side.

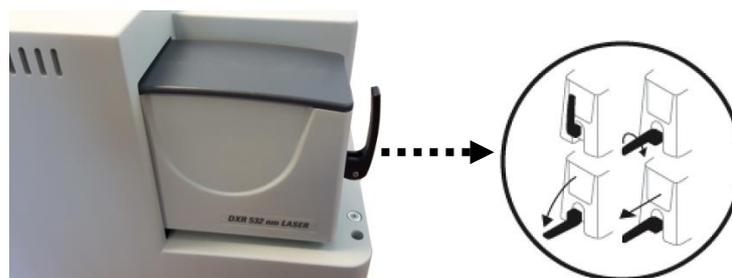


Figure 112: Undocking the Existing Laser

4. Remove the cover of the iXR unit by loosening the four silver-coloured fixing screws on the front and rear of the unit (note: screws are captive and sprung), then lifting the cover directly upwards.

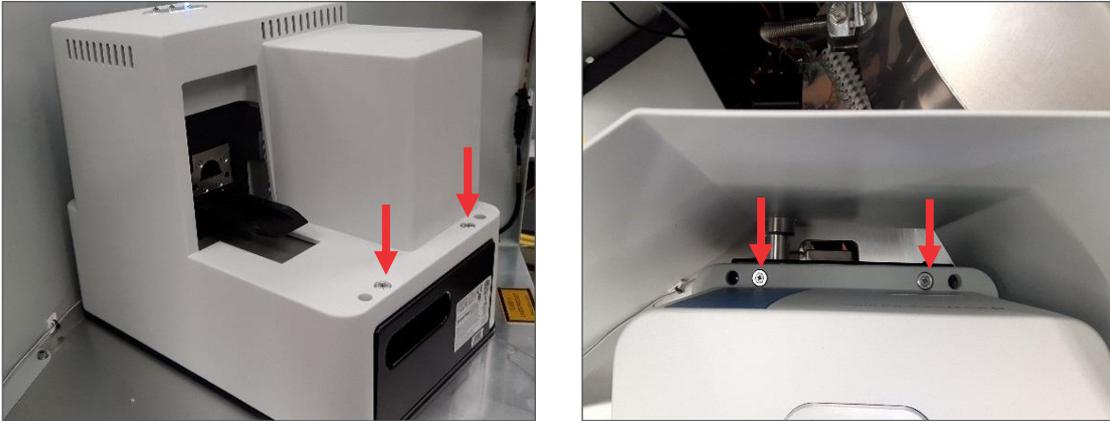


Figure 113: Removing the Cover

5. Close the aperture on the *existing grating*.



Figure 114: Removing the Existing Grating

6. Remove the existing grating from the unit by pulling gently directly away from the unit. Place in a plastic bag to prevent dust build up, and set to one side.



Figure 115: Remove the Grating

7. Remove the filter assembly from the unit, using the handle to pull directly outwards from the unit.

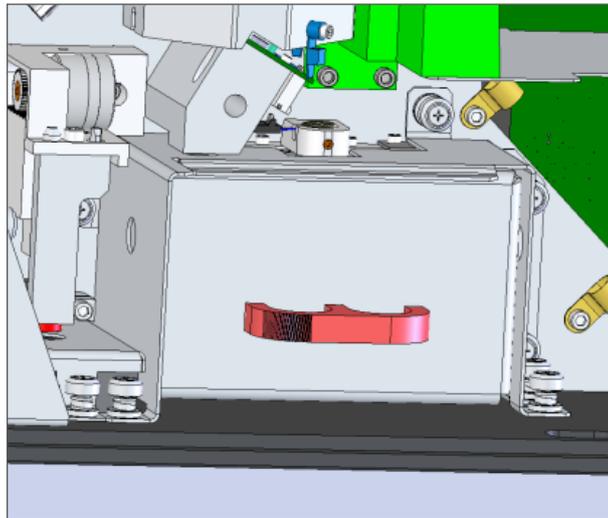


Figure 116: Remove the Filter Assembly

8. Unpack the alternative wavelength Laser kit and check all parts are present:
 - Laser assembly
 - Filter assembly
 - Grating
9. Fit the new grating to the unit, aligning the plastic tab with the recess in the housing. The grating will be pulled into place and held firmly by magnets



Figure 117: Fitting the New Grating

10. Open the grating aperture by turning the tab on the outside of the grating to the position shown with an open window.



Figure 118: Opening the Grating Aperture

11. Fit the new filter assembly into place, with the handle facing outwards and the magnet facing upwards. Like the grating, the magnets locate the filter assembly in the correct place.

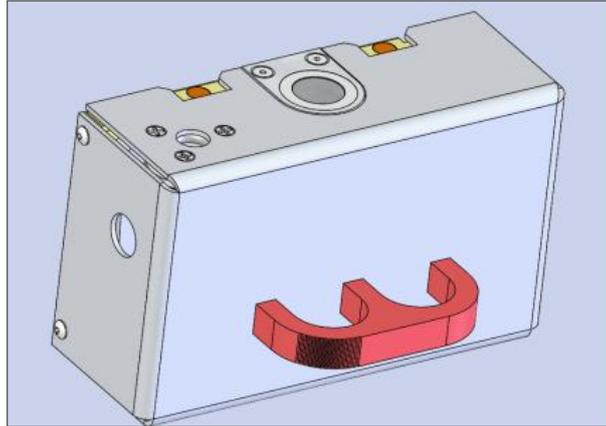


Figure 119: Fitting the New Filter Assembly

12. Replace the cover of the iXR unit and fix in place using the four captive screws.
13. Locate the new Laser assembly onto the slide at the rear of the unit, push in towards the unit and lock into place by twisting and rotating upwards the black handle on the rear.



Figure 120: Locking the Unit into Place

14. Open the OMNIC software
15. Align and Calibrate the unit following the instructions in the section iXR Unit Alignment and Calibration, page [121](#). Ensure that the Alignment and Calibration completes successfully.

NOTE: If Alignment/Calibration fails due to “Insufficient counts” then it is likely that step 10, opening the grating, was missed out.

16. Load a silicon sample into the Nexsa system.

NOTE: It is useful to locate the sample in the lower left portion of the sample holder, as it will then be closer to the phosphor standard

sample in the system and will minimise the stage movement required.

17. Move to the _Phosphor position. Confirm the correct analysis position (height) by using the x-ray source smallest spot, which will be visible on this sample.
18. Centre the Laser beam on the sample by varying the two adjusters on the mirror closest to the Nexsa (reducing the Laser power and/or adjusting the optical view settings may need to be modified to accurately view the Laser position). See section
19. **Analysis Position** Adjustment page **120** for more details.

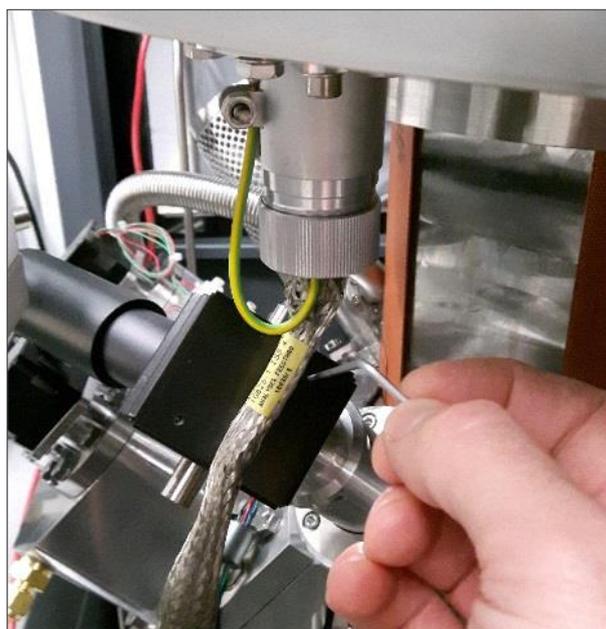


Figure 121: Centring the Laser Beam

20. Move to the silicon sample and locate the sample at the correct height (using the Laser beam if visible)
21. In the Bench tab of the experiment setup dialog, set the silicon peak range of $450 - 550 \text{ cm}^{-1}$. Take note of the **Max - Min** number on the display.
22. Turn the focus adjuster to maximise the sensitivity of the Raman signal (highest Max-Min number), noting that:
 - a. Some hysteresis may be present in the focus movement, so it is preferable to move in one direction only for fine movements
 - b. It is wise to average 2 - 3 measurements to ensure an accurate measurement of the signal level.



Figure 122: Turning the Focus Adjuster

23. Once the focus has been optimised, ensure that the spot position is still centred on the analysis position. If not, then adjust the spot position once more using the adjusters on the mirror closest to the Nexsa system.
24. The new Laser wavelength is then ready to use.

Analysis Position Adjustment

From time to time it may be necessary to make small readjustments of the Laser spot position on the sample, in to maintain a good match with the XPS spot position. This section details the best way to complete this adjustment.

1. Move to the ‘_Phosphor’ position.
2. With the Laser turned off (or blanked), ensure that the smallest x-ray spot and optical view crosshairs agree, as well as the crosshair on the height-setting camera. If necessary, either the x-ray spot position, TTL camera or Height setting camera should be adjusted such that the positions are coincident (such adjustment is detailed in the Nexsa setup procedure).
3. Turn the Laser on at low power (0.1 mW) and check that it is visible on the ‘phosphor’ sample.
4. Use the adjustment screws on the mirror closest to the Nexsa system to move the spot on the sample, as per diagram below.

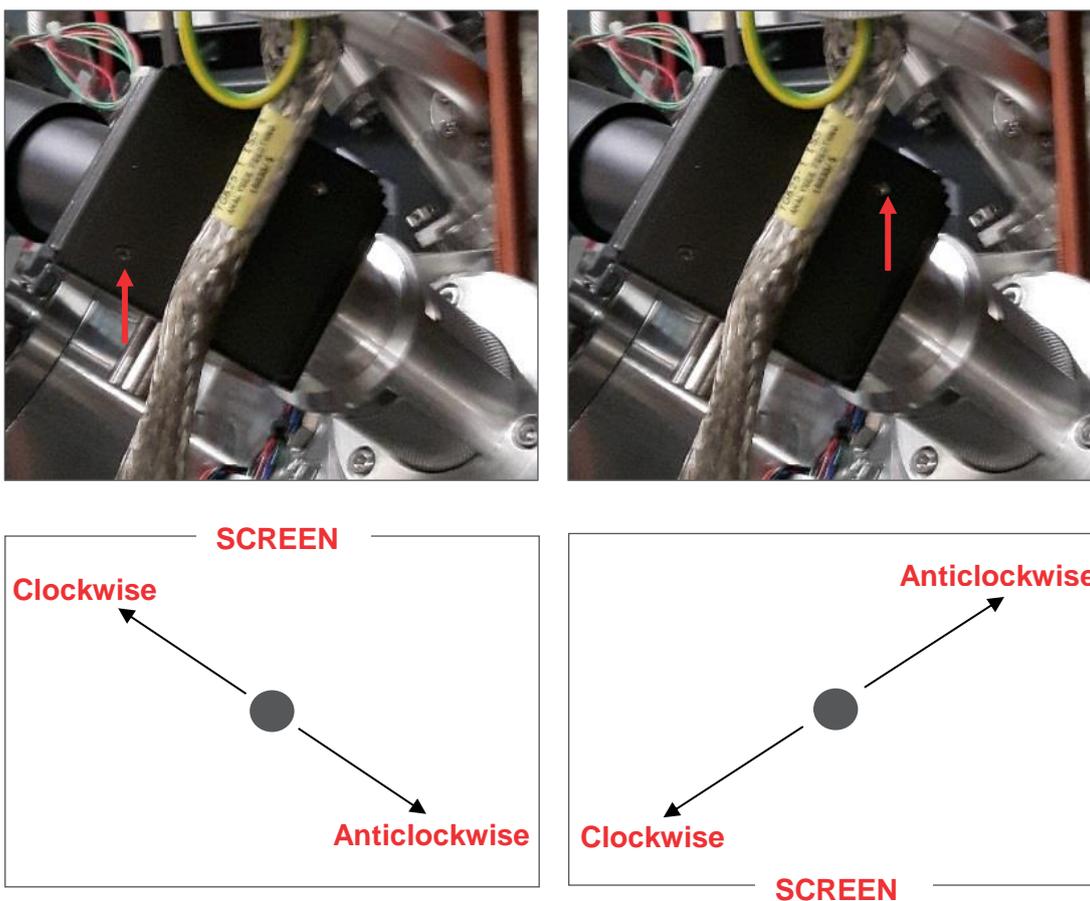


Figure 123: Screw Adjustment on Mirror

NOTE:

There is a limited range of movement before the spot will dim and/or start to reflect from the edges of the conduit leading to the system. If the spot cannot be centred then further adjustments will be required. Contact your local service representative.

iXR Unit Alignment and Calibration

If the operating environment (e.g. temperature etc.) does not change, then it is expected that the iXR Raman spectrometer will continue to perform at the optimum level with time. However, at least once per year the iXR Raman spectrometer will require calibration and alignment in order to maintain maximum performance, and calibration will be required if a different wavelength Laser and grating etc. has been installed (see **Changing Laser Wavelength** page **113**). This calibration can be undertaken by the user at any time through the *OMNIC* software generally takes less than one hour to complete.

The iXR unit has an internal calibration unit which is used for both Laser alignment and energy scale calibration.

Laser Fine Alignment

Fine adjustment of the Laser trajectory to best match the Raman collection path is necessary to ensure best performance from the unit. This is a software automated process and is run through the *OMNIC* software.

1. Open the **Experimental Setup** dialog box.
2. Switch to the **Bench** tab.
3. Turn the Laser on and set to 10 mW output power (532 nm Laser).
4. Switch to the **Alignment** tab. [It may take some time for the tab to become active].
5. Click on the **Align laser using Raman Signal** button.
6. The automated alignment process will begin after a short delay (during which the spectrometer is being prepared into the correct state to perform the alignment). If the automated alignment does not start, click the **Auto Align** button at the lower right corner of the dialog box to begin the process. You will observe the **Side to side** and **Up/Down Laser Fine Adjust** numbers changing.
7. Wait for the auto-alignment process to complete. At this point you should observe a **Max** number in excess of 10,000 in the top left corner of the spectrum display.
 - If a number less than 10,000 is observed then it is likely that there is a problem with the Raman spectrometer - contact your Thermo Scientific service representative.

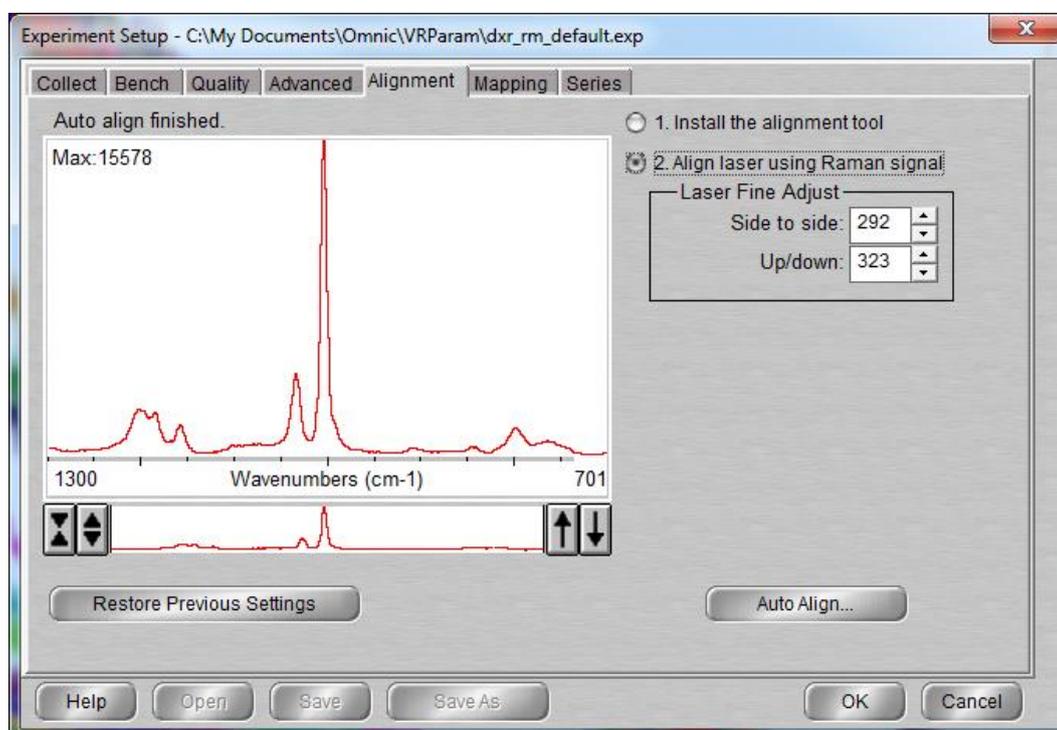


Figure 124: Experiment Setup Alignment Tab

8. Click **OK** to close the **Experiment Setup** dialog box.

NOTE:

At this point you may get a dialog asking if you wish to Calibrate. Clicking Yes will automatically kick off the unit calibration routine outlined in the next section. If it does not kick off the procedure, follow the procedure outlined in the Calibration section below.

Calibration

If the Laser fine alignment has been performed, or if the energy scale appears incorrect then it is likely the unit requires calibration. Calibration of the iXR Raman spectrometer is an automated process accessed through the *OMNIC* software:

1. From the **File** menu select **Collect > Calibrate Instrument**.
2. The automated calibration process will begin.
3. This process generally takes a few minutes to complete, but can take longer if the Laser wavelength has been changed. After completion you should see a message stating calibration was completed successfully.
If any step shows as failed or not completed successfully, contact your service representative.

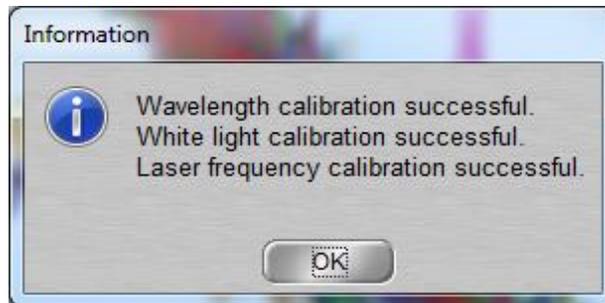


Figure 125: Information Dialog Box

4. Once the calibration is complete, turn the Laser off using the **Experiment Setup** dialog.

Optional Sample Holders

Tilt Sample Holder



Figure 126: Tilt Sample Holder

The optional tilt sample holder allows angle dependent XPS data to be acquired. Samples to be investigated must be no bigger than 26 mm x 5 mm x 5mm, and be mounted in the sections labelled A-D on the tilting bar. Samples must not over-hang the edges. It is recommended to mount samples using tape to prevent shadowing of sample areas. It is also necessary not to cover the calibration cross marking on the right side of the tilting bar.

After the sample is loaded into the Nexsa, the checkbox on the sample tab needs to be selected to change the rotation controls to the tilt controls. Once the sample holder is removed, this should be deselected so that the motor settings are returned to the default azimuthal rotation mode.

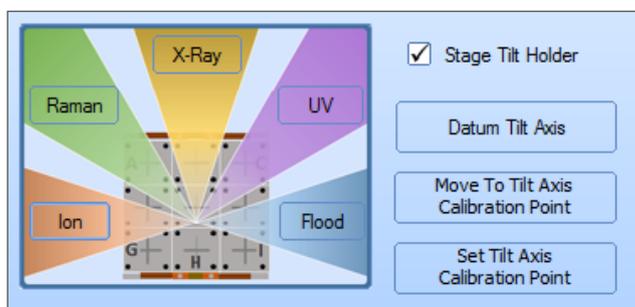


Figure 127: Setting Stage Tilt Holder

Once the sample holder has loaded into the analysis chamber, the tilt axis needs to be calibrated (full details can be found in the *Avantage* help file).

- Click **Move to Tilt Axis Calibration Point**.
- The flat position (0°) must then be found. There are two ways of doing this. Either adjust the tilt controls in the lower right of the optical view so that the top and bottom edges of the tilt bar are in focus at the same Z position. An alternative method is to turn on the co-axial light and adjust the tilt until the reflected light on the right of the calibration cross is at its brightest. At this point the tilt bar is flat. After using either of these methods, the **Datum Tilt Axis** button should be pressed to set the angle to (0°).
- The Z-position of the sample should then be optimised, either using the two camera views or the XPS signal.
- Once the height is set, move to the centre of the cross and press the **Set Tilt Axis Calibration Point** button. The tilt stage is now ready for use.

Experiments are set-up as described above, but use the **Stage Tilt** object from the **Line Insert** icon on the **Experiment** tab. The recommended angular range is 0-60°. Remember that the signal intensity will decrease as the angle increases, and so larger numbers of scans or snapshots may be necessary to obtain data with good signal:noise characteristics.

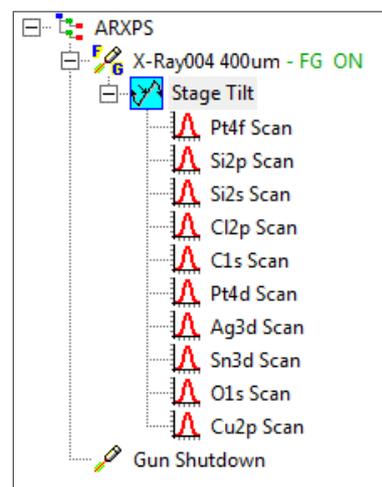
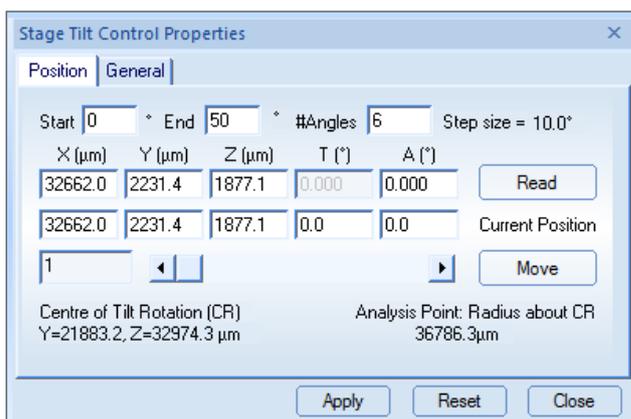


Figure 128: Stage Tilt

Bias Sample Holder

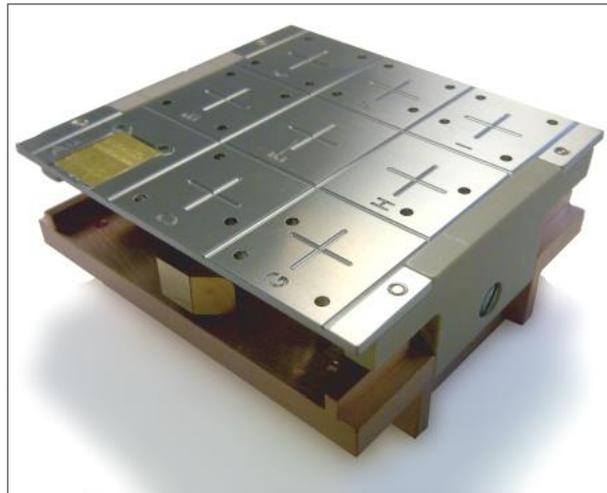


Figure 129: Bias Sample Holder

The optional bias sample holder allows work function measurements of samples to be performed. The work function is measured by extrapolation of the low kinetic energy spectrum cut-off, but this can be convoluted with the cut-off of the spectrometer. To separate the sample cut-off from the spectrometer cut-off, a negative bias voltage needs to be applied to offset the spectrum to higher kinetic energy. The module has an electrically isolated top-plate, which connects to the in-vacuum standards block using a spring-loaded contact, allowing a bias voltage to be applied.

Samples should be clipped to the sample block, and the electrical contact between the top surface and the top plate should be good. The sample block is loaded into the system in the usual way, although the position of the contact arm should be checked before loading to ensure that it is safely under the top plate.

Once the sample is loaded, the contact arm needs to be brought into contact with the samples block to allow the bias to be applied. To do this, the sample rotation controls are used. A rotation clockwise of 100° will make contact.

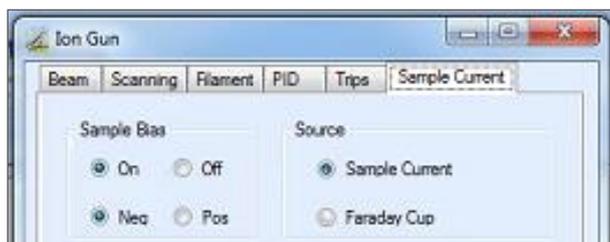


Figure 130: Ion Gun Details. Sample Current Tab

The sample bias is applied using the ion gun details dialog. Open the dialog, and turn on the sample bias. Set the polarity to negative, and the mode to **Sample Current**. This will apply a bias of -30 V to the sample. The sample bias indicator will be green to show that the bias is being applied, and can be clicked on to directly access the bias control. The sample bias can also be controlled from the UV source object in the experiment tree if a UV source is fitted..

A silver standard is supplied with the holder to allow testing of the system. Clean silver should have a work function of around 4.0-4.5 eV. As an example, this is the procedure to take a measurement on the silver standard.

- Clean the silver sample using the ion gun
- Collect two spectra
 - Cut-off: pass energy 3-10 eV, range 32 -> 38 eV (Kinetic), 10 scans
 - Fermi level: pass energy 50 eV, range -25 -> -35 eV (Binding), 10 scans
- Measure the position of the Fermi level, and shift both spectra so that the Fermi Level is at 0 eV. This determines the exact bias applied to the sample.
- Use the Work Function Measurement tool in Avantage to determine the work function. The work function is obtained by subtracting the cut-off position from the photon energy (on the Nexsa this is 1486.6 eV ± up to 200 meV; the error in the photon energy is determined during the energy scale calibration, and can be found in the calibration folder in C:\AvantageSystem for the best accuracy).

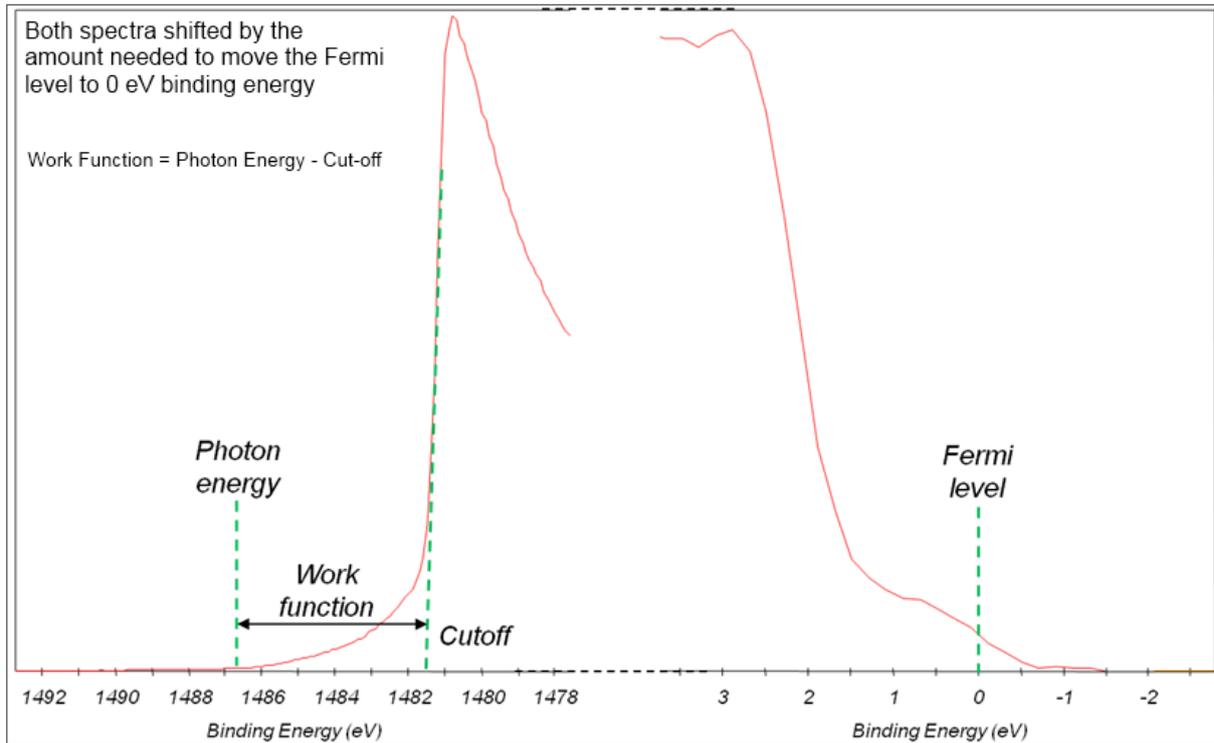


Figure 131: Work Function



Work function measurement tool, found on the Analysis tab

At the end of the experiments, it is important to remember to turn off the sample bias, and change the other radio buttons to “Pos” and “Faraday Cup”. The contact arm will automatically retract as the sample block is unloaded.

Vacuum Transfer Module



Figure 132: Vacuum Transfer Module

The optional vacuum transfer module allows samples to be transferred from a glove box environment into the Nexsa system without exposure to air. The device comprises three parts, the aluminium cover, the lower sample holder assembly, and the clamping bar.

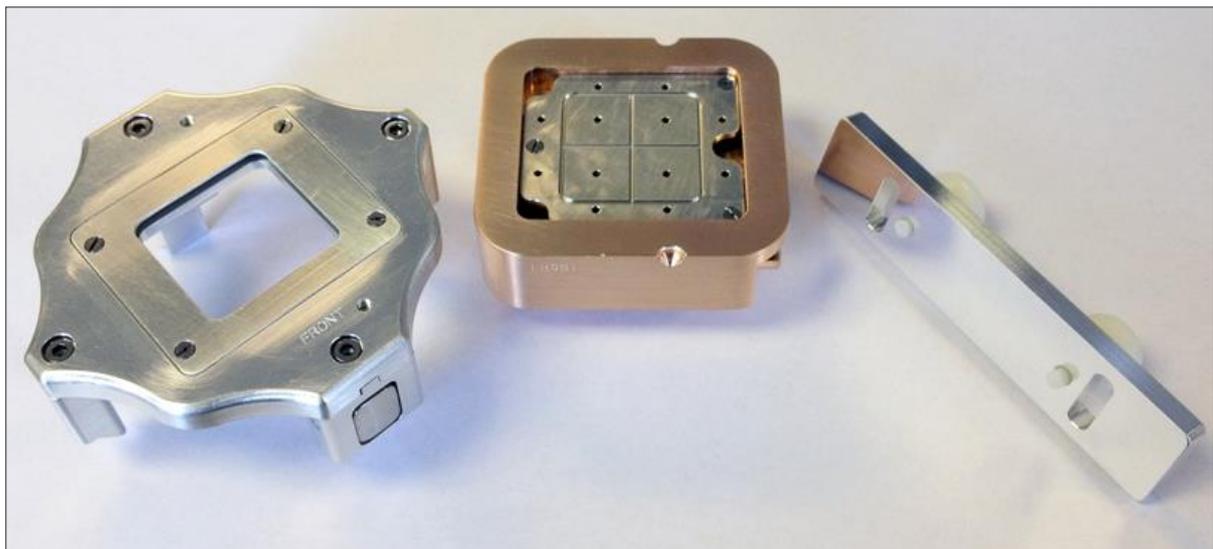


Figure 133: Vacuum Transfer Module Parts

Operation

The parts should be loaded into the glove box separately, to prevent the device sealing during the antechamber evacuation cycles. Samples can then be mounted onto the top plate. There are four positions for the sample mounting top plate, as shown in the following images.

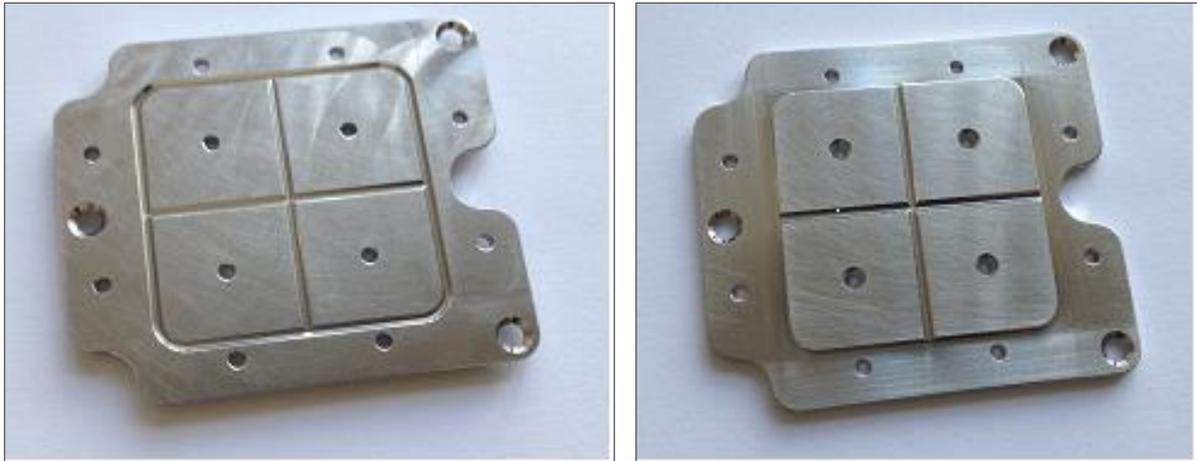


Figure 134: Sample Plate Flat Side (Left) and Raised Side (Right)

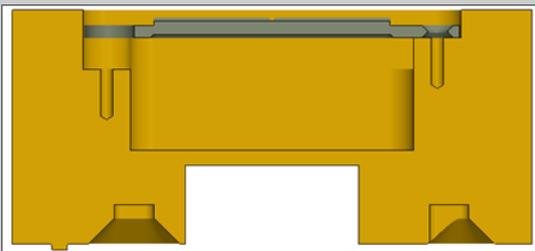
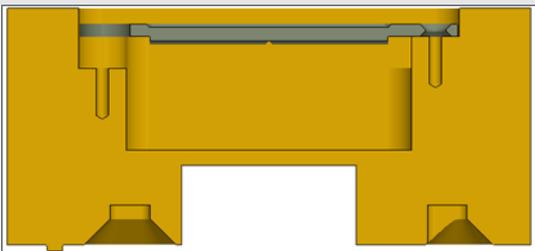
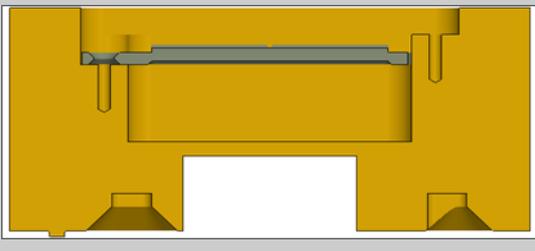
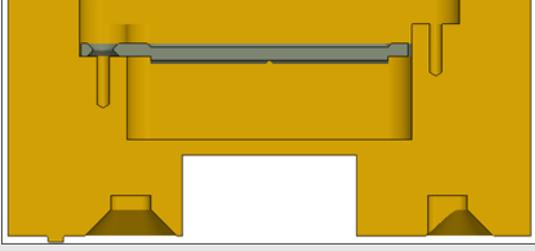
<p>Position 1</p> <p>Raised side up</p>		<p>Sample thickness range 0 – 1 mm</p>
<p>Position 2</p> <p>Flat side up</p>		<p>Sample thickness range 0 – 2 mm</p>
<p>Position 3</p> <p>Raised side up</p>		<p>Sample thickness range 2 – 5 mm</p>
<p>Position 4</p> <p>Flat side up</p>		<p>Sample thickness range 3 – 6 mm</p>

Table 13: Sample Plate Positions

Samples can be mounted using the clips in the standard Nexsa sample kit (supplied with the instrument) or using tape. Once the samples are mounted, the unit needs to be sealed. The upper section has two pins that engage with the two gaps on the edges on the holder.

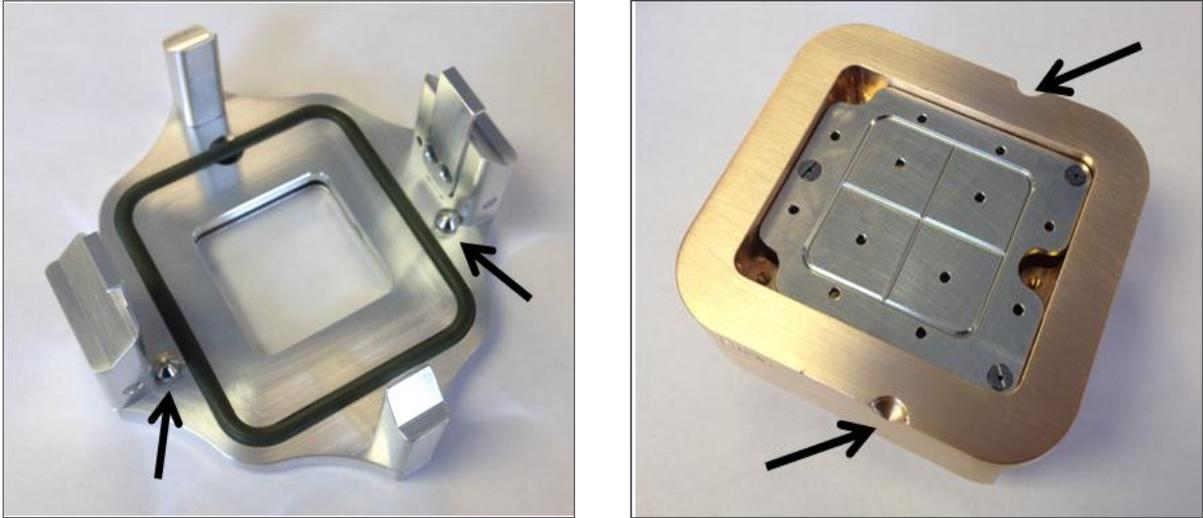


Figure 135: Transfer Module Pins

Once the unit is closed the clamp bar needs to be fitted, as shown below. This keeps the unit together until it is pumped down, maintaining the inert atmosphere. Care must be taken to ensure that the o-ring seal is clean before closing.

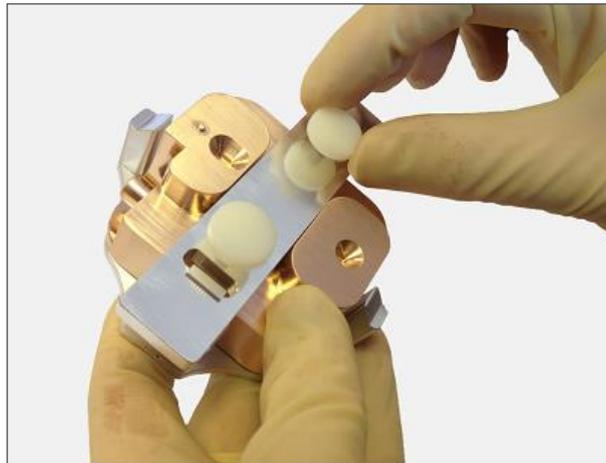


Figure 136: Clamping the unit for pump down (done inside the glove box).

Ensure that the clips engage before tightening the thumb screws.

The clamped unit must then be pumped out. It is recommended to use the glove box antechamber to do this as the sample holder is transferred out. Pump the unit for about 10 minutes to ensure that it is fully evacuated. If it is not possible to pump the unit in the glove box, then a pipe can be attached as shown below, and the unit pumped using a standard rough vacuum pump.

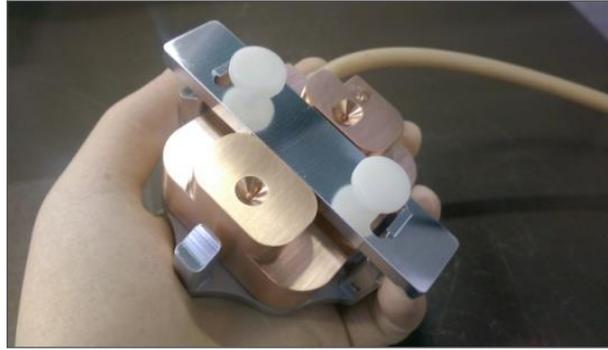


Figure 137: Pumping the Clamped Unit

To transfer the holder into the Nexsa system, the clamp bar must first be removed. External air pressure will hold the unit together once it is evacuated.

IMPORTANT:

The module should be loaded into the Nexsa system within 30 minutes of pump down. Longer delays risks the o-ring sticking and causing an unsafe release of the base in the instrument.

Fit the module into the Nexsa load-lock, making sure that the side marked **FRONT** is at the front of the instrument, and that the clips on the aluminium section engage correctly on the edges of the load-lock.

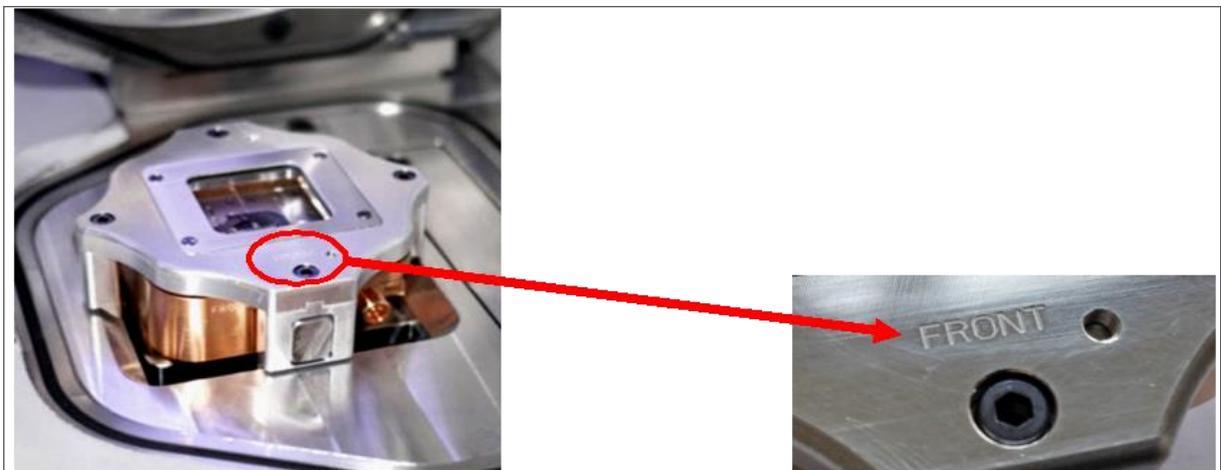


Figure 138: Fitting the Module into the Load Lock

The sample holder can then be pumped down using the **Pump and Park** button.

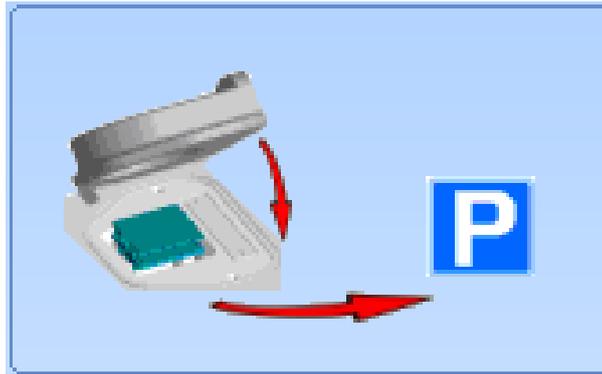


Figure 139: Pump and Park Button

The unit will separate during the pump-down, when the vacuum level in the load-lock goes below the level inside the holder. **To ensure separation it is important to allow the unit to pump for 15 minutes before transferring into the analysis chamber.**

After analysis, the unit can be unloaded from the system as usual. The two parts will stay separate when unloading from the system (i.e. the inert transfer is one-way only).

Troubleshooting & Maintenance

The vacuum transfer module needs little maintenance. The sealing faces need to be kept clean and free of scratches to ensure that the unit pumps down and seals correctly. The position of the ball valve is set with a small grub screw. This can be adjusted using a 2 mm hex wrench if required. Replacement parts for the valve are supplied with the module, comprising the O-ring, ball, spring and grub screw; the assembly is shown in the figure below.

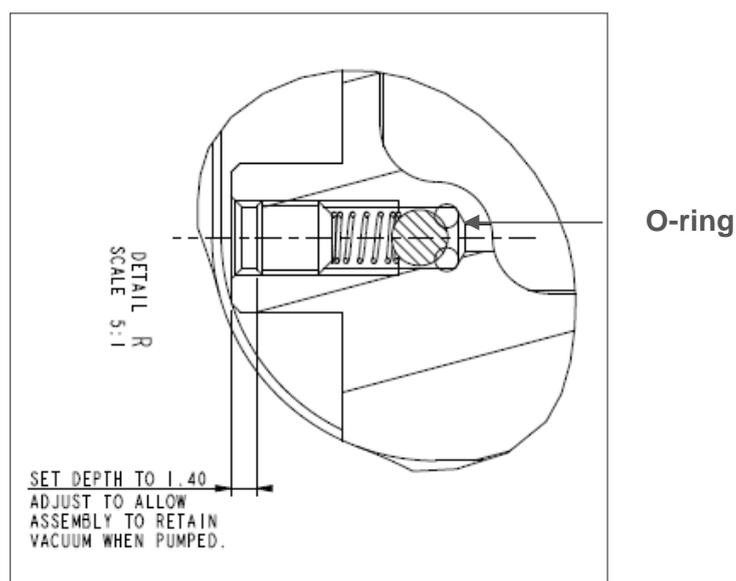


Figure 140: Replacement parts

Vacuum Transfer Module Operation Flow Chart

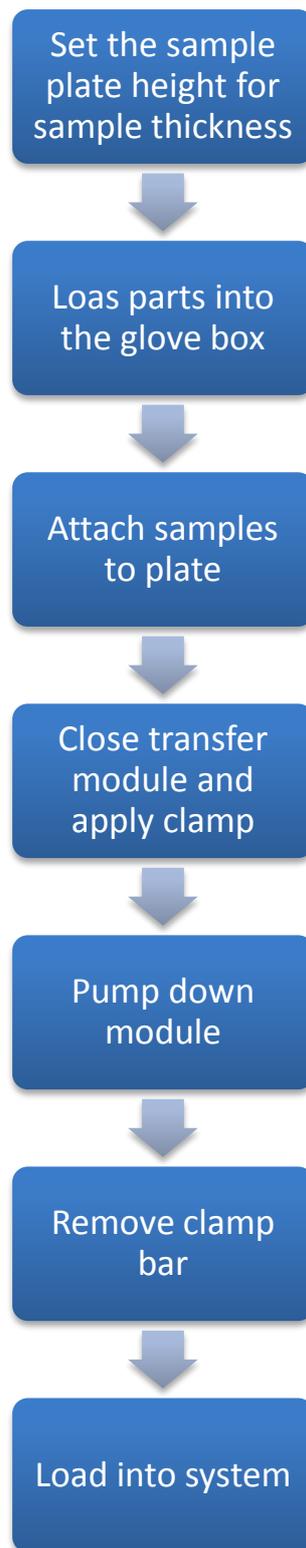


Figure 141: Vacuum Transfer Module Operation Flow Chart

Appendix 1: Processing Commands

For full information on the commands, please see the online *Avantage* help file.

Analysis

	Survey ID
	Manual peak ID
	Add peak
	Peak fitting
	Peak table profile
	Charge shift
	Chemical state assessment
	Non-linear least squares fitting
	Target factor analysis
	Principal components analysis
	eSP – Enhanced survey processing
	Band Gap Measurement
	D-parameter measurement



Work function measurement

Compare & Overlay



Lightbox compare



Overlay



Overlay manager



Image overlay



Overlay image on optical image



Combine

Modify



Smooth



Energy deconvolution



Differentiate



User background



PCA reconstruct



Spin-orbit subtract

Arithmetic



Add a constant

	Subtract a constant
	Multiply by a constant
	Divide by a constant
	Add spectra
	Subtract spectra
	Multiply spectra
	Divide spectra

Profile

	Smooth profile
	Interface width calculator
	Time to depth
	Split profile
	Escape depth correction

Image

	Average spectrum
	Smooth image
	Line / band scan



Interpolate scattered data



Phase analysis



Split image



Image deconvolution

Utilities



Clone dataspace



Crop / Collapse dataspace



Trim dataspace



Export data



Import data



Resample data

Angle Resolved XPS



Relative depth plot (requires tilt module)



Single overlayer calculator



Multi-overlayer calculator (requires tilt module)



ARProcess (requires tilt module)

Appendix 2: Display & Reporting Commands

For full information on the commands, please see the *Avantage* help file.

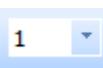
Display Modes

	Single trace
	2D chart
	Stacked chart
	3D chart
	2D image
	Wafer Map (Theta 300 only)
	Relative depth
	Bar chart
	Information
	Energy spectrum
	2D image X-Y
	Display as profile
	Chart properties

Display Options

	Toggle user annotations
	Toggle peak labels
	Toggle fitted peaks
	Toggle residual
	Toggle peak backgrounds
	Toggle user backgrounds
	Sum data series
	Display spectrum in binding energy mode
	Display spectrum in counts per second mode
	Reverse energy axis

Reporting

	Report to MS Word
	Report to MS Excel
	Report to clipboard (use for reporting to MS PowerPoint)
	Report preferences
	Number of spectra on one row of report output
	Include selected empty grid cells

Process Grid Icons

	Save peak table
	Load peak table
	Toggle constraint display (Peak fit table)
	Lock selected constraints (Peak fit table)
	Clear selected constraints (Peak fit table)
	Propagate constraints (Peak fit table)
	Resizable cells
	Maximise selected cells
	Tile cells
	Size all cells to current cell size
	Size all cells to default cell size
	Add cells
	Subtract cells
	Grid preferences
	Axis preferences

