Thermo K-Alpha XPS Standard Operating Procedure

Draft v.0.1

Quick Guide

Procedure overview

1. Vent the loadlock
2. Secure your sample to the stage using clips, check the height of the final assembly.
3. Place the stage in the loadlock with the letters facing into the chamber. Ensure that the stage is mechanically secure against the pins.
4. Close and pump the loadlock. Wait until the loadlock pressure is below 5e-7 mbar.
5. Transfer the sample to the analysis chamber.
6. Build your experiment tree and set the sample position for each analysis point.
7. Wait until the chamber pressure is below 8e-8 mbar, and then start the experiment.
8. When the experiment has finished, check that the guns have ramped down, and transfer your sample to the loadlock.
9. Vent the loadlock and retrieve your sample. Close the loadlock and leave it under vacuum.

Critical precautions and common mistakes

- Wait for base pressure to start your experiment. Do not load the sample into the main chamber until the loadlock pressure is below 5e-7 mbar. Do not ramp up the X-ray gun until the chamber pressure is below 8e-8 mbar.
    - Always wear clean gloves when handling the stage. A single fingerprint will extend your pumpdown time by hours
    - Do not place carbon tape or other adhesives directly on the stage
    - If the chamber pressure does not recover because your sample is outgassing, ask the staff about how to prepare your sample appropriately.
- The most common cause of downtime is getting the stage or sample stuck in the transfer mechanism

Tool overview

XPS has been widely used for surface analysis. The X-ray depth penetration is about 10 μm, but only photoelectrons from within 1-10nm of the surface are able to reach the detector. Typical uses of XPS are:

I. Identification of elements present at the surface (qualitative analysis)
II. Concentration of elements at the surface (quantitative analysis)
III. Chemical bonding state of elements (chemistry)
IV. Distribution of elements across the surface (elemental map)
V. Change in composition with depth into the surface (depth profile)

This tool has a monochromatic aluminum Kα X-ray gun, which is generated by a 12 kV electron beam, resulting in a photon energy of 1.4866 keV and line width of 0.85 eV. The X-ray spot size can be changed from 400 μm down to 30 μm with 5 μm increments, which is controlled and adjusted by changing the e-beam spot size. The analyzer pass energy can be varied from 5 eV to 200 eV. For this system, the sensitivity for most elements is around 0.05 to 0.1 At%.

The system is equipped with an argon sputtering gun, which can be used for surface cleaning or depth profiling. The argon sputtering size is about five times of the X-ray spot size with sputtering energy varies from 200 eV to 3000 eV.

Full procedure

1. Sample restrictions

(1) Ideal sample size is about 1 cm x 1 cm, but the smaller the better. Hence multiple samples can be loaded at one single run, with reduced pumping time
(2) Maximum sample size including the sample clips CANNOT go beyond the stage area.
(3) NEVER use those 4 clips on both ends of the stage to mount your samples. These 4 clips are designed to hold the sample plates rather than the samples. Make sure that the "back" side of the clips does not go over the edge. (*Add picture of wrong clip mounting)*

(4) ALWAYS use clean gloves to handle the sample stage, plate, and clips. A single fingerprint can prevent the chamber from reaching base pressure.
(5) NEVER load wet samples. If your samples contain Zn, As, F, Cl, Br, I and other toxic/volatile materials, contact IAC staff for advice.
(6) ALWAYS use the pre-built Teflon sample height measurement gadget to check the height of your sample before transferring the stage into the load lock. NOTHING including the clips should hit the stainless steel bar. Ideal sample height is about 3 to 5 mm below the bar. If your samples are too low, you should check and adjust the stage height. Try to load samples with similar height for each experiment run.

2. System status checking

(1) Make sure all system status lights are on with green LEDs only. If orange color is shown for any status light, STOP your work and contact IAC staff immediately.

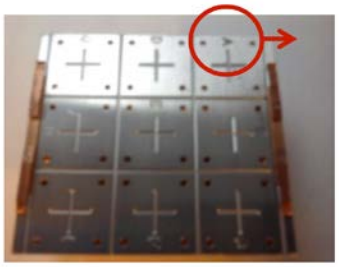
System Status Lights

(2) Check all guns status (X-ray gun, ion gun and flood gun) on the lower left-hand-corner of the control software panel and make sure all guns are all off before you start anything. If any guns are on, you need to manually shut them off from the control panel before loading the samples.

3. Sample loading

(1) Place your sample holder on the sample stage.
(2) Click on the venting load lock button. The button can be found within the Sample section on the top left-hand-corner of the control software panel.

(3) Load the sample holder with the letter "A" facing the analysis chamber.




(4) Make sure the holder is leveled and placed with right position. It should be secure in its position; if it is not, it has probably been inserted the wrong way.

(5) Click on the pump button and let the stage pumps in the load lock until the pressure reaches 5E-7 mBar.

(6) Once the loadlock has reached the desired pressure, click on the transfer button and move the stage into the analysis chamber.

(7) Wait until the stage comes to a full stop & let the analysis chamber pressure reach 8E-8 mBar or lower. Low pumping speed may be caused by sample outgassing or contamination of the sample plate and stage. If the chamber pressure does not reach 8e-8 mBar, abort the run and ask IAC staff for help.

After sample transfer, sample plate X, Y registries are recorded in a picture by the XXX camera. The image of the sample plate shows at the top half window. Double click on the sample plate picture to navigate to your sample position. Live video from the TTL camera shows in the lower half window. Adjust Z to focus on your sample. For any transparent sample, moving to the sample edge or marking at the sample surface helps you focus on the sample surface.

4. Setting Experiment

(1) There is currently a glitch that sometimes obscures sections of the experimental panel. To reset the interface, click the button with a check mark and a red x and navigate until you see a button to reset to the default interface.(This needs some screen shots)
(2) Go to Experiment section on the software control panel.
(3) Create a new experiment

- Source: X-Ray Gun
    - Set your desired spot size.
    - Turn on flood gun for nonconductive samples, e.g., semiconductors and insulators.
- Scan: Point / line / area

  To run experiments, an experimental tree needs to be constructed. Depending on your sample properties, you can set up to operate point scan, line scan, map scan and depth profile. All scans should include a survey scan and fine scans for each possible element.
    - Always do auto height unless working on small features.
    - For auto height, use relative range +/- 1000 μm with step at 50 μm.
    - Dwell time is the time staying at each channel of energy, Survey: 10 ms; Hi-res: 50 ms.
    - Set up pass energy and numbers of scans. Survey: 200 eV pass energy, 2-5 scans. Fine scans: 20-50 eV pass energy, 5-10 scans. For elements with low concentrations, further increase of the scan No. is helpful, or consult IAC staff for advice.
- Spectrum: Multi Spectrum
    - Check the Survey Spectrum box for survey scan.
    - Click on the elements you intend to do high resolution scans.
    - If necessary, to select secondary peaks for an element, right-click that element and choose the peaks to include.
    - Scan parameters dwell time, pass energy and scans) can be changed accordingly for each element.
- Depth profile
    - Set up the sputtering gun conditions: duration time, No of points (levels), low or high current, and spot size.
    - Build the depth profile into your experimental tree. This includes setting-up all parameters for both the survey scans and the fine scans.

- Gun shutdown
    - Always include this step at the end of your experimental process.
    - Check the Transfer to Park box to transfer samples to the load lock automatically after measurement, to minimize the time samples stay in the analysis chamber.

5. Height adjustment

(1) Navigate to your sample by double clicking on the optical images or use the 4 directions arrow keys to move the stage.
(2) Use the Z height up and down keys to bring the sample in focus for each point you are intended to do experiment.
(3) Read location for the experiment. Pressing "read" imports the current coordinates of the crosshair.
(4) Auto height function should always be selected to let the program assists and adjusts the optimal height.

6. Check the analysis chamber pressure and make sure it's at least 8E-8 mBar or lower.

7. Now you are ready to start your experiment by clicking on the big green arrow (Experiment Run) button!

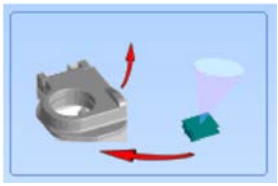
8. Data collection and Spectrum Processing

(1) To begin the analysis process, create "cells" and drag experimental steps into those cells.
(2) To zoom into a specific cell, click the top-left corner of that cell. To reset the view to all scans, click the gray box at the top-left of the cell. (More info can be found in the page below)

9. Finishing up

(1) DO NOT shut off the Advantage program.
(2) Make sure all guns are off before unloading your samples.

(3) Unload the sample (if you checked the Transfer to Park box when setting Experiment, samples are already in the load lock).



(4) Pump the load lock again. You always need to leave the load lock in high vacuum when you are done with your measurement!

(5) CLEAN UP the workplace, REMOVE all samples from the stage, and leave it ready for the next user.

(6) Sign up on the logbook. Report any comments you might have about your experiment.

Troubleshooting

If it takes longer than usual to pump down, try unloading the stage and mechanically wiping it with IPA.

If the guns will not ramp up despite normal system parameters, Try restarting the Avantage software.

Version history

Draft	*Date*	*Author*	*Notes on changes*
v.0.1	October 16, 2016	Nathalie de Leon	

Emergency Information:

Medical Emergencies: Contact 911 and Public Safety (609) 258-1000
Room / facility emergencies: Contact Public Safety (609) 258-1000
Issues related to the instrument:

1. Contact IAC Staff.
2. Leave system as is, Do Not disable vacuum system.
3. Try to shut off the Gun.

Audible/Siren Emergency Alerts:
Follow previous steps 2 & 3 and leave the building.

Emergency Contact Information:

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