Venting Kratos

Make sure filament and high voltage have been off for ~30 minutes Make sure UPS, sputter gun, heating stages have also been off for 30 minutes (any heated components) Open SAC-STC valve (flap valve) Open manual line between load lock and STC Turn CCG's off Press "Stop HV" on ion pump (bottom left box on rack) Turn off STC Turbo Close STC turbo backing valve (V3) Turn off load lock pumping station Open N2 vent (V5) to assist slowing of STC turbo Wait for load lock arm to open (will open when pressure is equilibrated, be sure that clips are unclipped) Access STC through window on top (6" copper gasket) but it is necessary to also remove flange on sputter gun (2 ¼" copper gasket)

Putting Kratos under vacuum and baking

Remove samples that may degas from chamber Close valve between load lock and STC, bring load lock under vacuum normally Ensure N2 vent (V5) is closed and open turbo backing valve (V3) to small scroll pump and wait for pressure to reach the -2's Start STC turbo, wait for it to spin up (30 min), and turn on CCG's Before bake, all control boxes can be on EXCEPT the stage control unit - make sure this is off Ensure that the protected mains switch is also off Remove all parts that cannot withstand high temperatures (cables, RGAs, camera, CCG on STC, etc.) - don't forget cable on bottom of the back side of the stage Put blanket over main instrument chambers, leave high voltage post in back, CCG on SAC, and turbo pump on SAC outside of blanket Put heating tape around load lock, cover in foil, and open valve to suitcase loading port Ensure flap valve is open, automatic baking sequence should open ion gauge differential pumping valve (V2) Apply thermal tape to load lock arm and stage manipulator, cover in foil, use variac to heat (stage manipulator to 90 deg, and UPS differential pumping to load lock line to 125 deg, use

manipulator to 90 deg, and UPS differential pumping to load lock line to 125 deg, use thermocouples to measure temp)

Using EELS bake rack, turn on Mains, set time to desired hours on back panel, press start, monitor temp

On Kratos Bake, set time to match EELS bake time. Allow several hours to cool to around 60 deg before degassing filaments

VCU Engineer's tool: Parameters: if necessary, can change limit of flow rate drops higher than 10 (try 10,000?)

Messages window: VME slave messages: type 220 to see flow rate status (note: magnet controller and protected mains must be ON)

Degassing filaments on Kratos

After bake, let cool to temperature reading around 60 C on the annealing stage temperature controller

Take off blanket, plug everything in, don't move any of the mechanical components until completely cool

Turn on all panel boxes and protected mains

X-ray gun - check degas button, turn on degas, wait for automatic sequence to finish lon gun - check degas button, turn on degas, wait for automatic sequence to finish Charge neutralizer, start at 1 A current, increase slowly over 15 min to 2 A (0.1 A increments), turn off

RGA - open Process Eye Professional, double click Easy View, click squiggle button (filament), after 10 min or so, click <1> to change it to <2> and wait for another 10 min or so, then click filament button again

TSP - do this one after all the others have finished. Make sure flap valve is open. VCU Engineer's tool: Parameters: Set HVSTCoff limit to 1 E -5 and set HVSACon limit to 1 E -6 in VCU engineer's tool in software. On touchscreen panel under menu, TSP config, select degas, run it at 40 A, then again at 45 A, then again at 50 A. Change back pressure limits. For the second time degassing, put in some liquid nitrogen to cool it. Run it just at 50 A, making sure to change pressure limits in the software.

Repeat degassing for all filaments a second time, this time the charge neutralizer can just be left at 2 A

Realigning Kratos camera after bake

Aperture and iris - open tabs in control manual, click initialize, then confirm, then calibrate, then confirm and wait.

For stage, (don't have sample on stage) click calibrate and then confirm, wait.

Align camera - pump in puck with gold patterns on it, find the coarse gold grid, zoom in with camera

Focus on Au 4f like normal

In XPS imaging - lens: Field of view 1, aperture: low res imaging, set energy to 84.5 eV, AI (mono) source, click "on"

When a line appears, move to the fine Au grid, or go to the alphabet grid and find the capital delta in the center

Determine what letter is in the circle on the alphabet grid, align the camera with the letter, and turn off imaging

How to fix random Kratos problems

If the STC SAC pressures are not updating on the computer or VCU, and the flap valve is not opening...

Try restarting the VCU: unscrew panel on back of tower

Turn off turbo pump in STC

Ensure the turbo backing valve is closed [V3]

Turn off Ion pump

Ensure all other valves are closed

On VCU unit behind panel, flip power switch.

Note: when VCU is powered off, the turbo pump will turn off, so must make sure STC is isolated.

The VME/Slave messages window with message level 220 will only display meaningful information if the control boxes on the instrument tower are switched on. To get useful flow rate information, the magnet control box must be on (top right).

Water flow problems can sometimes be corrected by running a wire through the aperture in the manifold on the side of the instrument (top left hose). If this doesn't work, try switching the inlet

and outlet water at the manifold for 2 h with the X-ray running (turn the gun on and off several times). If this still doesn't help, replace the flow meter on the circuit in question. If this also doesn't help, yell at Kratos.

Contacts: Kratos

Chris Moffitt: cmoffitt@kratos.com - helpful and prompt, your best bet for an answer (in Houston, TX)

Paul Keenan: paul.keenan@kratos.co.uk - Chris' boss, also helpful and good to include (in Manchester, UK)

Larry Newman: Inewman@kratos.com - generally doesn't respond, though he does our site visits, he's the Larry we tell stories about (Phoenix, Az)

David Surman: dsurman@kratos.com -, sometimes chimes in on emails (Chestnut Ridge NY, President of Kratos USA)

Chris Finch: cfinch@kratos.com - sends parts

Contact: HREELS

Larry Kesmodel: lktech@lktech.com - this will be LK himself, the guy that made the EELS, our only contact at this point, but very helpful (Bloomington, IN) lkesmode@bluemarble.net - Larry's old email, I don't know if he still uses it

Email to possible XPS Trainees

Hi _____,

One requirement to be trained will be to submit short (~2 page) research proposal (instructions here:<u>http://mmrc.caltech.edu/Safety/New_Users.html</u>). This is so we know what kinds of samples you will be putting into the instrument and gives us an idea how often you plan on using the instrument. If you only plan on using it a couple times, we urge you to find a collaborator to run the samples for you.

The trainings usually take anywhere from 3-6 h depending on how much data collection people want to do. The instrument requires that new users complete two full training sessions, so this would count as your first. If you don't have time to draft a proposal before the first training, you can still participate in a training, but we won't do your second training until you have completed this requirement.

Best,