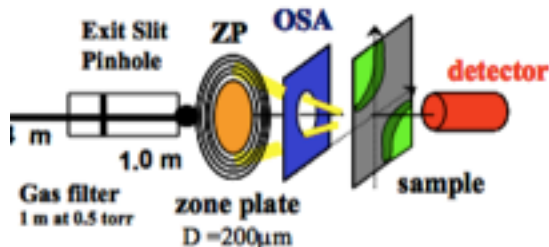
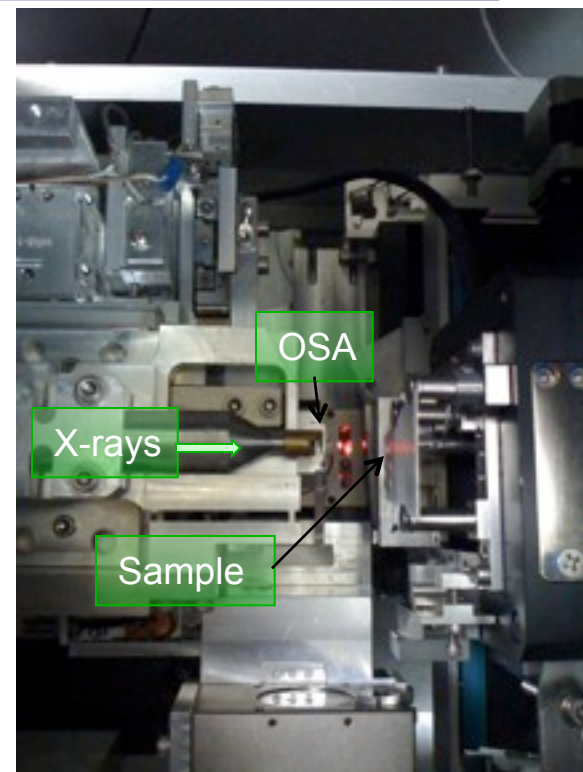
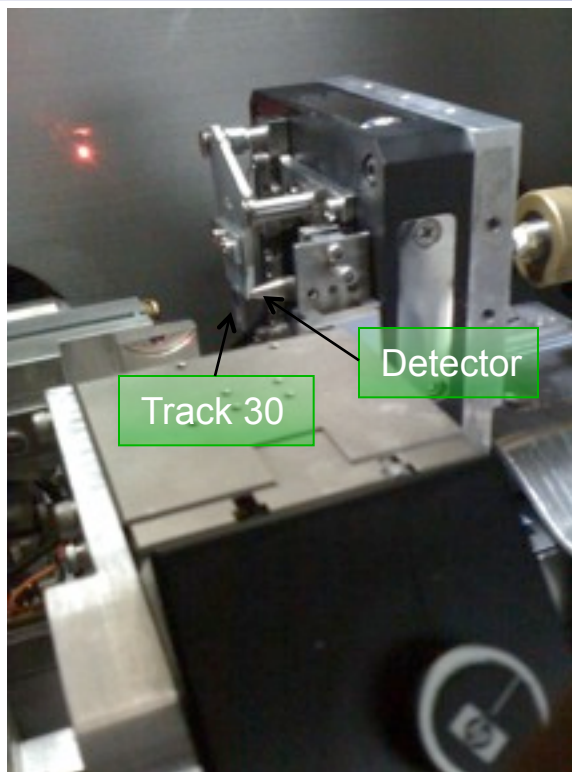
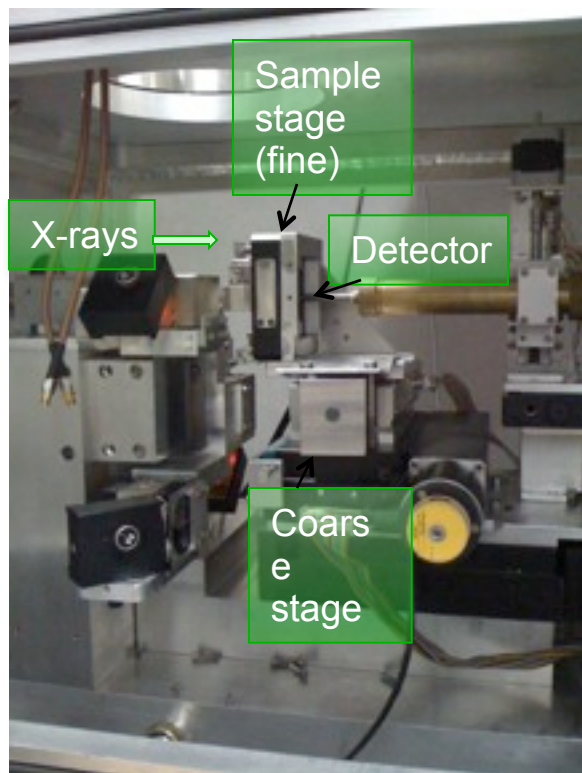
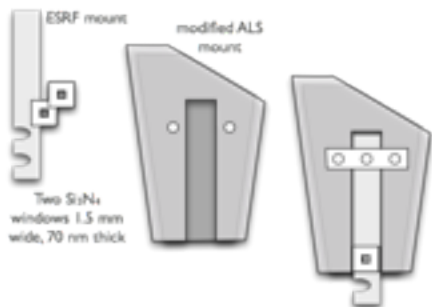

Track 30: I1043,1,30,0,0

ALS STXM 11.0.2 13 Jan 2010

**Anna Butterworth, Tolek Tyliszczak,
Zack Gainsforth, Andrew Westphal**

Track 30 mount in STXM 11.0.2

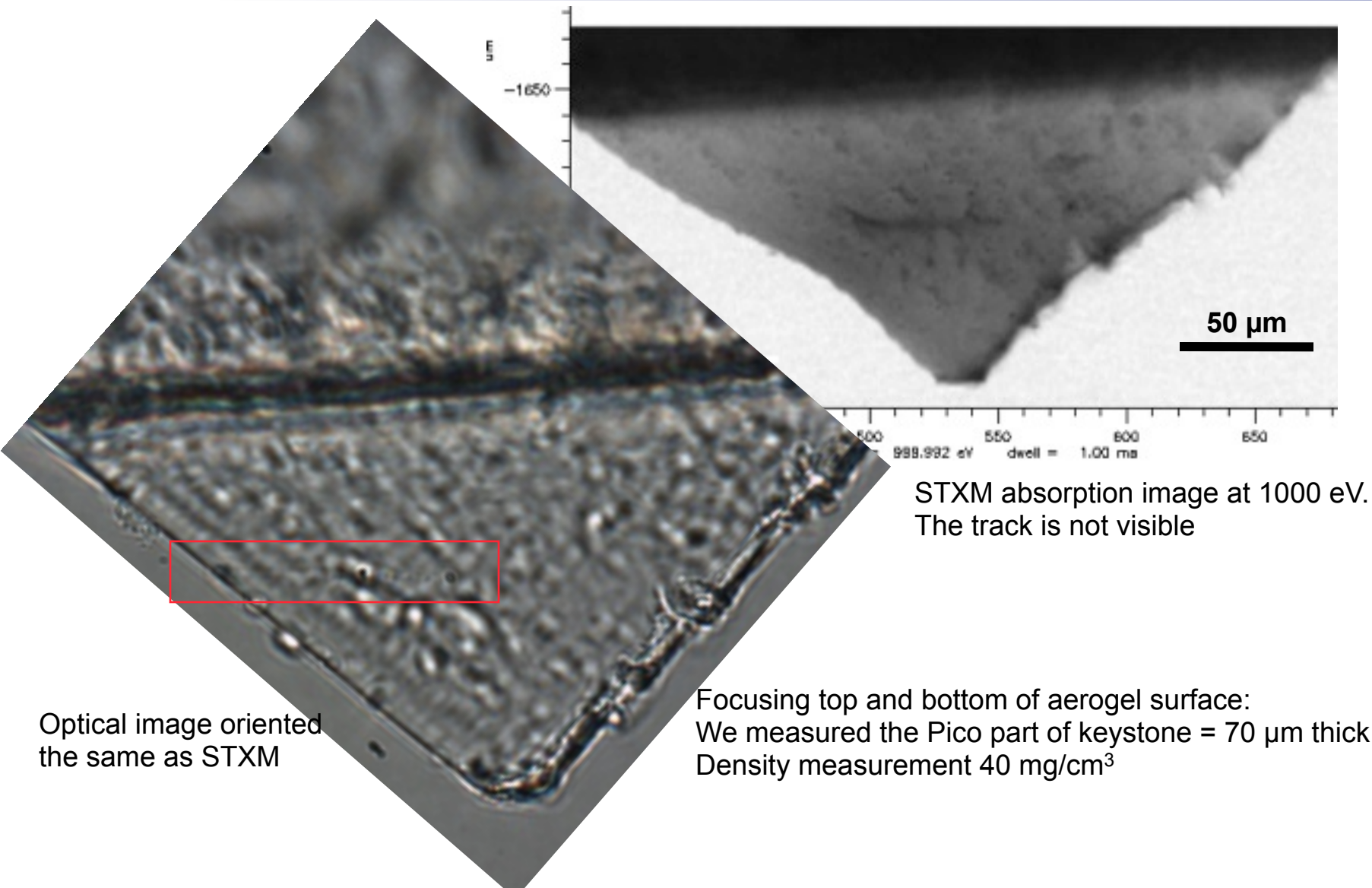


Loading sample mount into STXM with sample stage moved back from the order sorting aperture (OSA) and zone plate (ZP). For Fe L₃ XANES sample - OSA distance is ~1 mm (25 nm zone plate)

Track 30 mount in STXM 11.0.2

- Our cautious approach to handling Track 30 sample mount involved:
 - The Si₃N₄ sandwich is (unintentionally) sealed air tight. We purged the STXM with He while pumping out air to minimize pressure difference across the membranes.
 - We tested our procedure on Track 26, before successfully loading Track 30.
 - We installed a hard stop on the stage, as well as a software controlled limit, to prevent the sample from touching the OSA.
- There were no mistakes. The entire experiment performed extremely well.

Track 30: Navigation



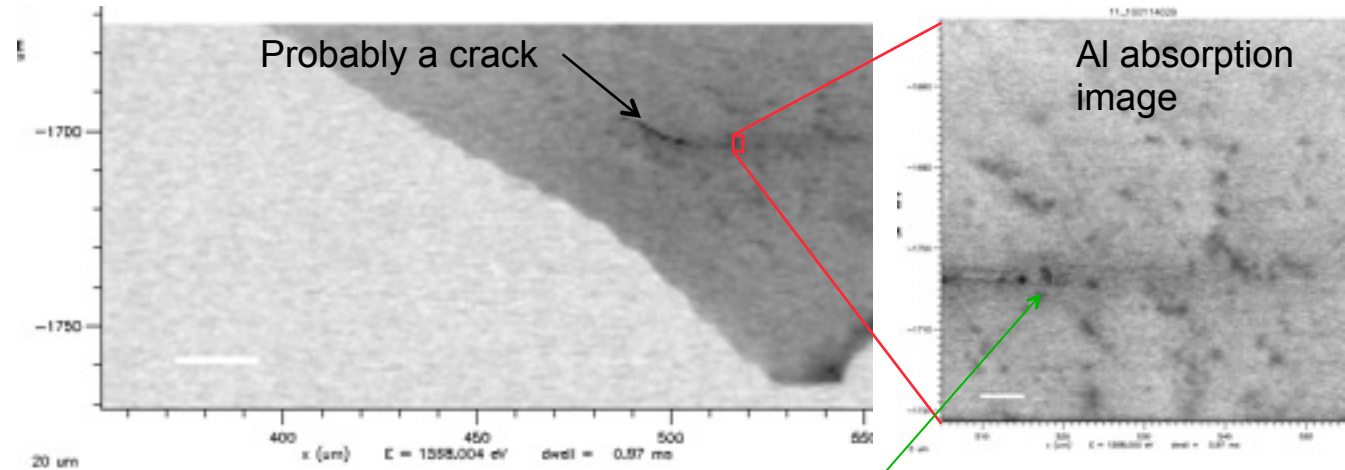
Optical image oriented the same as STXM

STXM absorption image at 1000 eV. The track is not visible

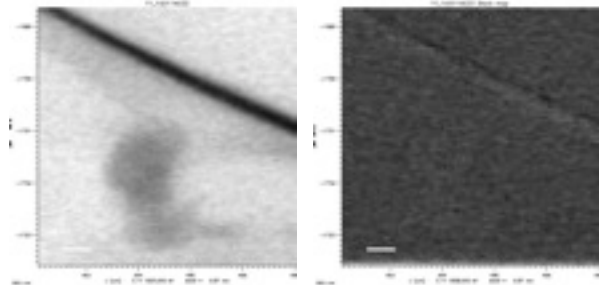
Focusing top and bottom of aerogel surface:
We measured the Pico part of keystone = 70 μm thick
Density measurement 40 mg/cm^3

I1043,1,30: Aluminum

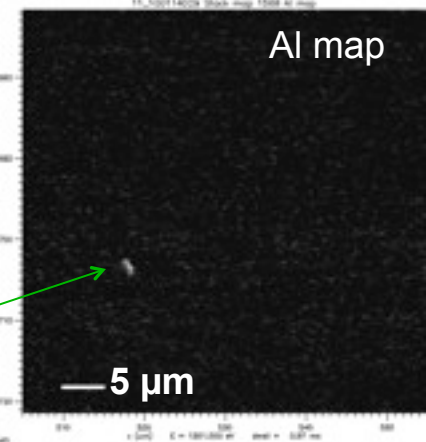
We could not verify the track at higher energies (low contrast). We located the terminal particle by matching features with the optical image and looking for a dense particle.



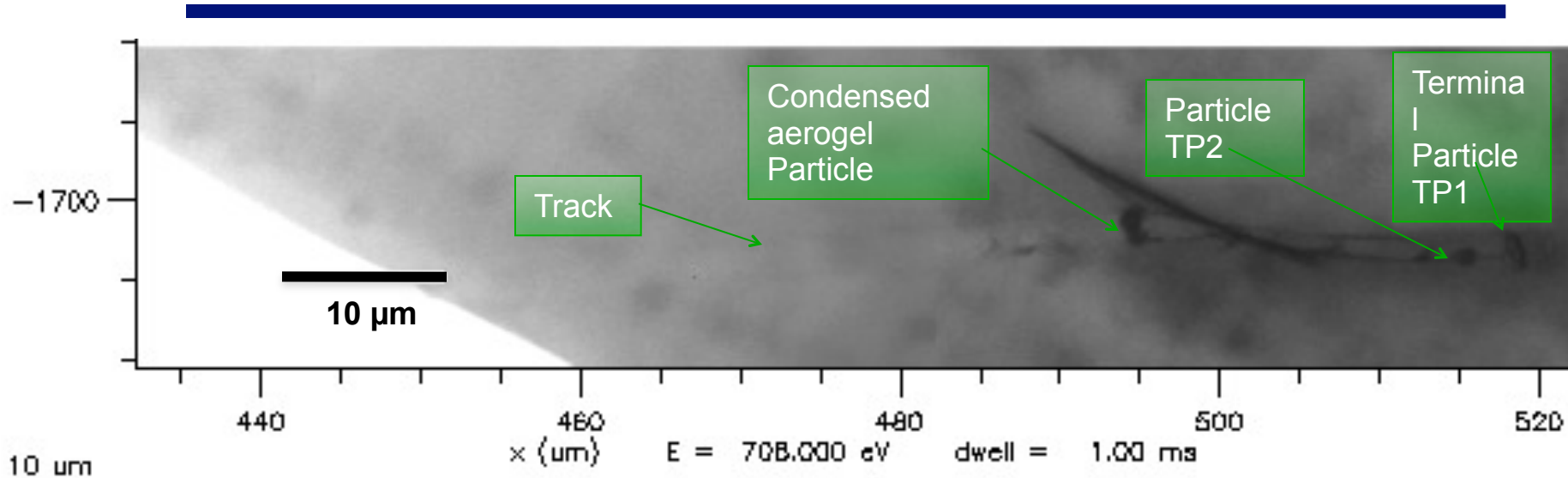
Al map of dense particle in track. This is most likely condensed aerogel



- Aluminum image (1568, eV) of small region shows a particle at the end of a track
- Aluminum map of same region shows the particle contains Al.
- 1568 eV peak is higher than 1561 eV peak, which means Al is non-metallic.



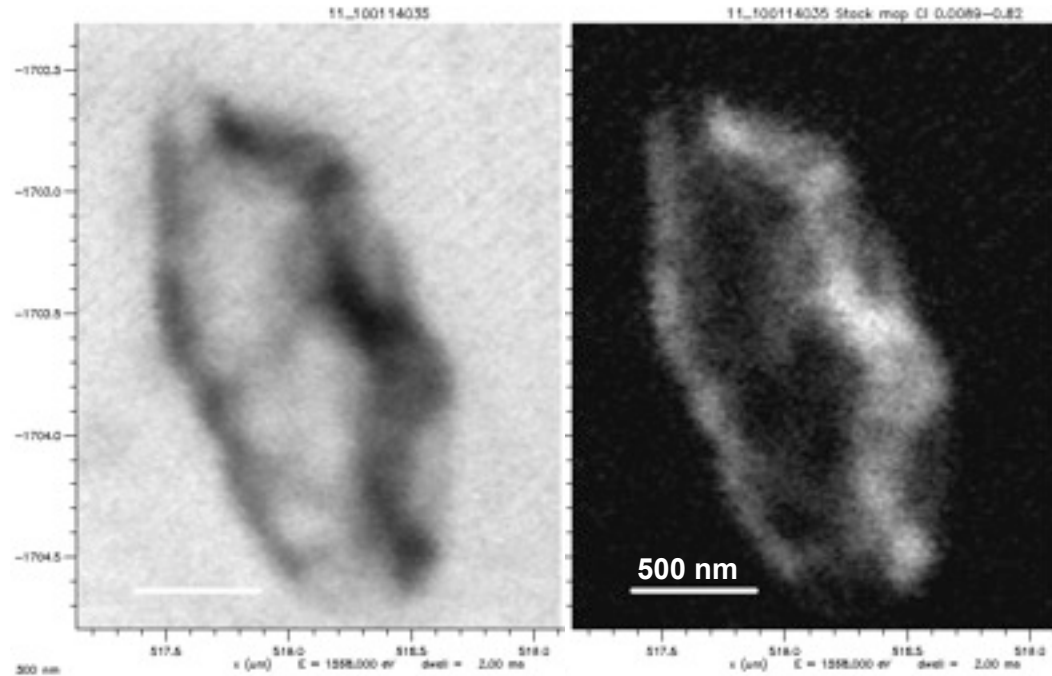
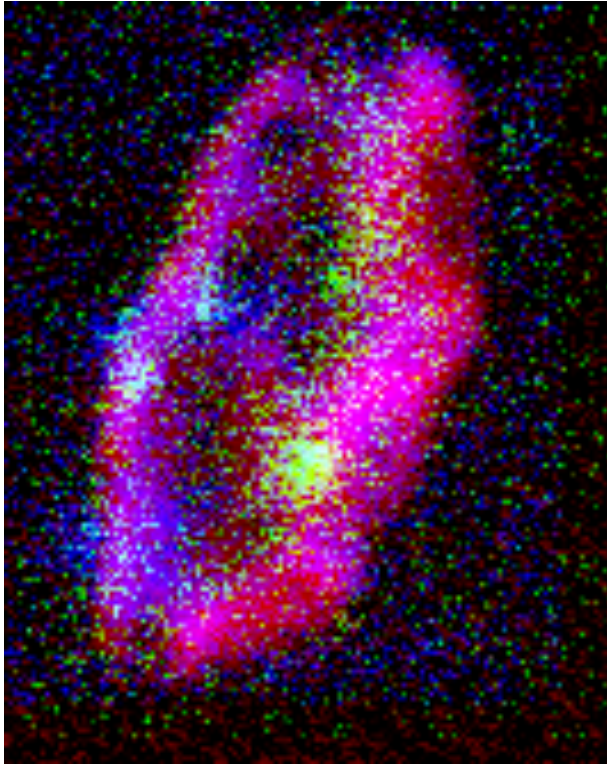
I1043,1,30 track image



- High resolution absorption image at 708 eV shows the whole track.
- We did not use any energy lower than 700 eV, as this would require a sample-OSA distance < 1 mm.

Terminal Particle Characterization

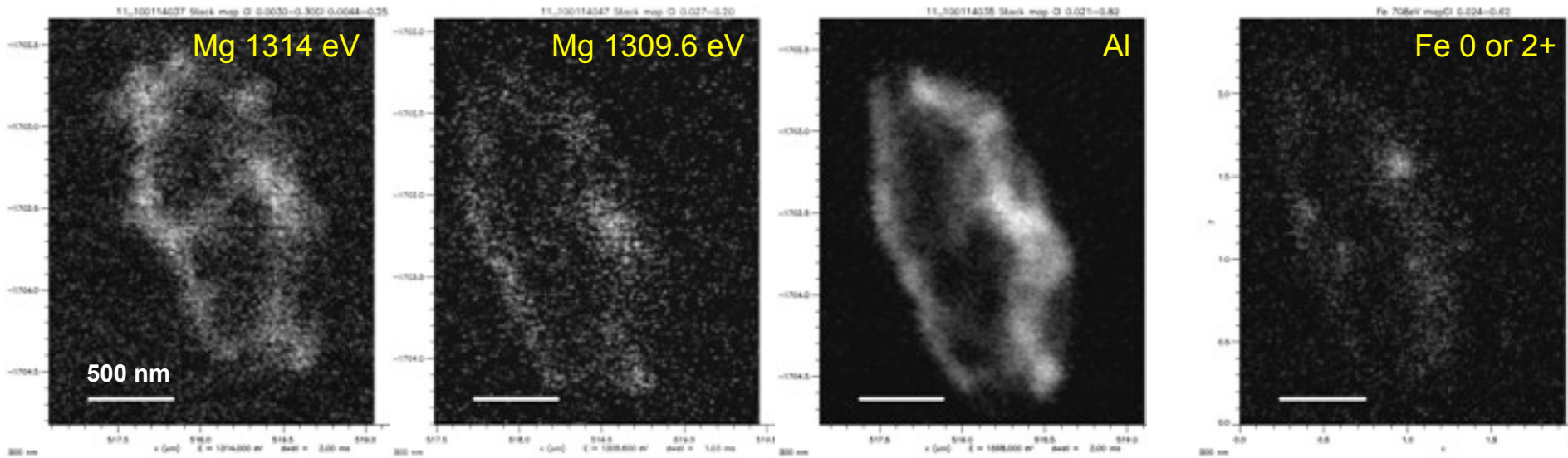
- Minimal photon dose used in making element maps:



Fine

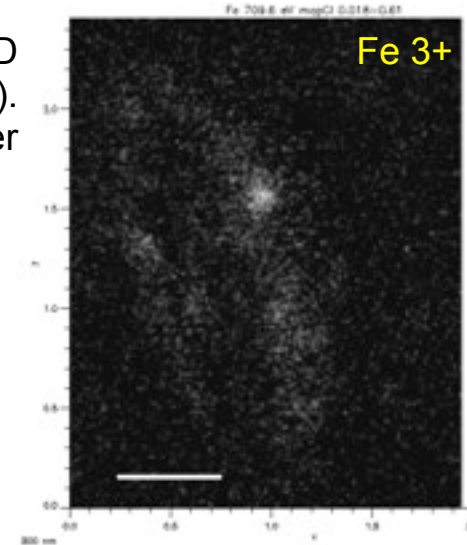
RGB overlay of Aluminum (red),
Iron (green), Magnesium (blue)
maps.

I1043,1,30 TP1 maps



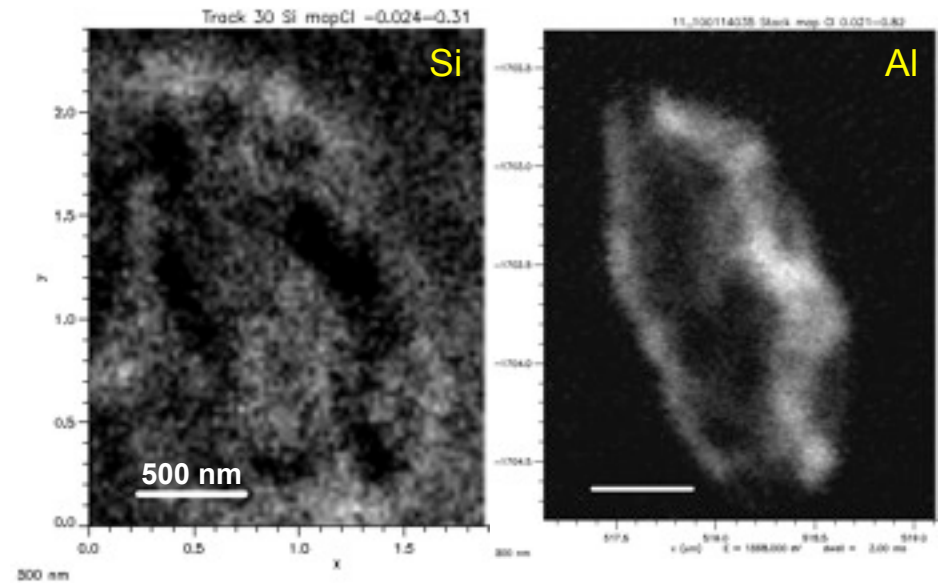
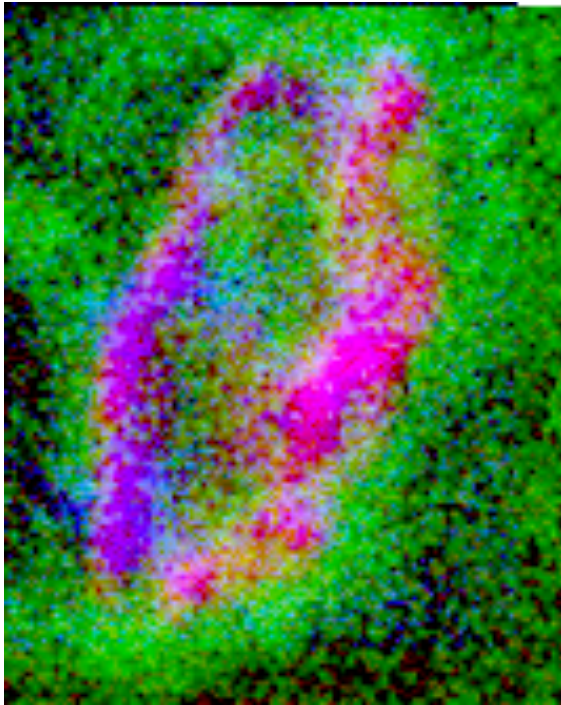
1314 eV peak is higher (OD 0.244) than 1309.6 eV peak (0.191) means Mg is not spinel. We would need XANES to narrow further.

Fe³⁺ map has lower maximum OD (0.284) than other Fe (0.308 OD). Fe L-edge XANES would further distinguish Fe²⁺ from metal/sulfide



- Mg, Fe, Al maps were 15 nm per pixel
- Si map was 25 nm per pixel

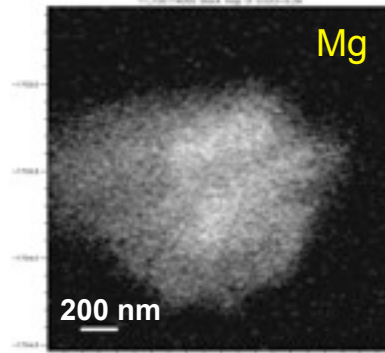
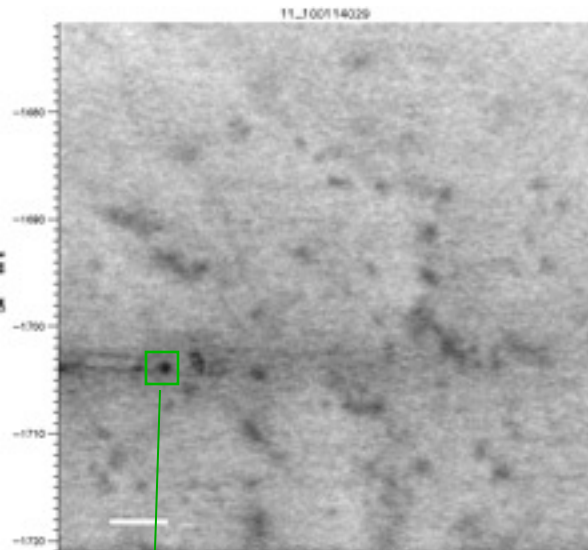
I1043,1,30 TP1 Silicon



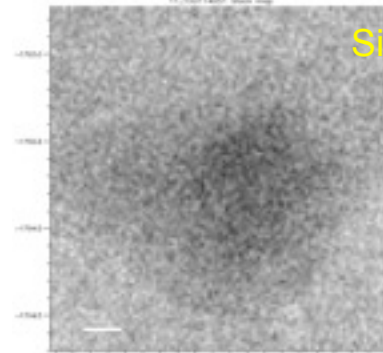
RGB Overlay Aluminum (red), Si (green), Magnesium (blue) maps

- Silicon poses a mystery. The 1846 eV, 1840 eV map shows condensed aerogel around the particle, but an apparent Si depletion associated with Fe, Mg, Al.
- Si-XANES may provide an answer; we have successfully performed Si-XANES on olivine in cometary aerogel.

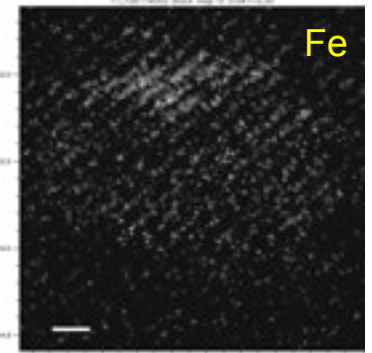
TP2



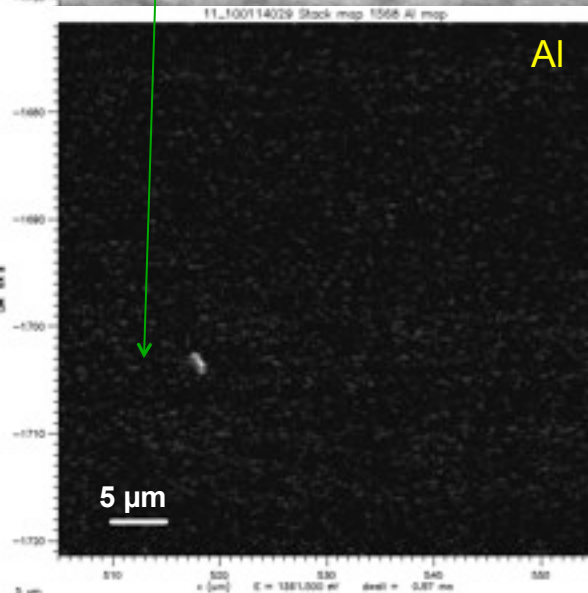
Mg map,
max OD= 0.368



Si map, 1846 eV, 1838 eV
Silicon depleted



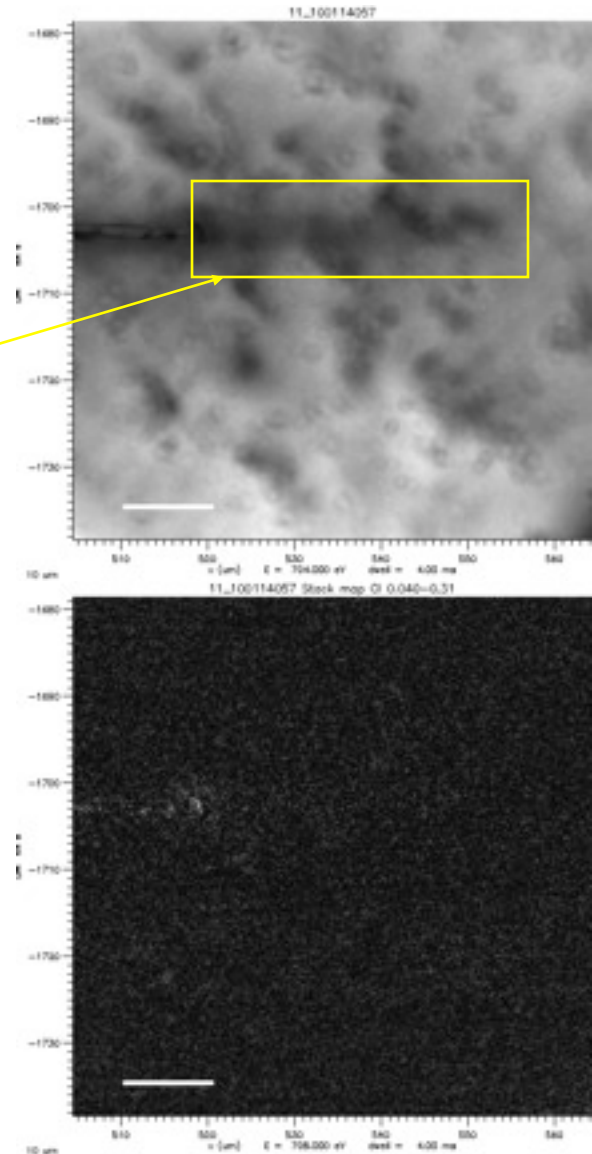
Fe map



- The second particle is Mg rich, minor Fe and without Al
- It appears to be Si depleted (1846 eV, 1838 eV map). Similar to TP1, Si XANES will distinguish between a real Si depletion and Si present in some other, non Si-O form.

Examining the rest of the keystone

- We examined the rest of the keystone to verify that STXM and ESRF analyzed the same particle.
- We found evidence for some beam damage to the aerogel extending beyond the end of the track. This is consistent with ESRF 45° angled beam
- We investigated the damaged area for C or other deposition, but found none. We do not understand yet the cause for the apparent denser aerogel.



Carbon test

- We changed to a longer working distance zone plate (without removing the sample and OSA) and re-purged with He.
- We performed a line scan at the edge of aerogel pico (away from the track) to test the viability of making a C measurement
- Though the aerogel is dense (40 mg/cm^3), we would be able to do full C-XANES to detect C above the high density and carbon background from the aerogel itself.

I1043,1,30,0,0 Summary

- Terminal particle contains non-metallic aluminum, magnesium, and iron.
- ESRF detected Fe plus Ca, Cr, Mn, Ni, Cu, Ga. We did not attempt to map these elements.
- Si appears to be non-correlated with Al, Mg, Fe. Si We see an Si-rich coating (probably aerogel) around the particle. Si XANES should resolve the mystery.
- The particle looks hollow at >700 eV. We have not attempted to map for low energy elements e.g. C
- We have verified elsewhere on this keystone that a carbon XANES map is possible.
- The second particle contains Mg and some Fe, but no Al or Si (but Si needs further investigation)

