

The Maia Detector System: A New Way to do Scanning Probe Fluorescence Imaging

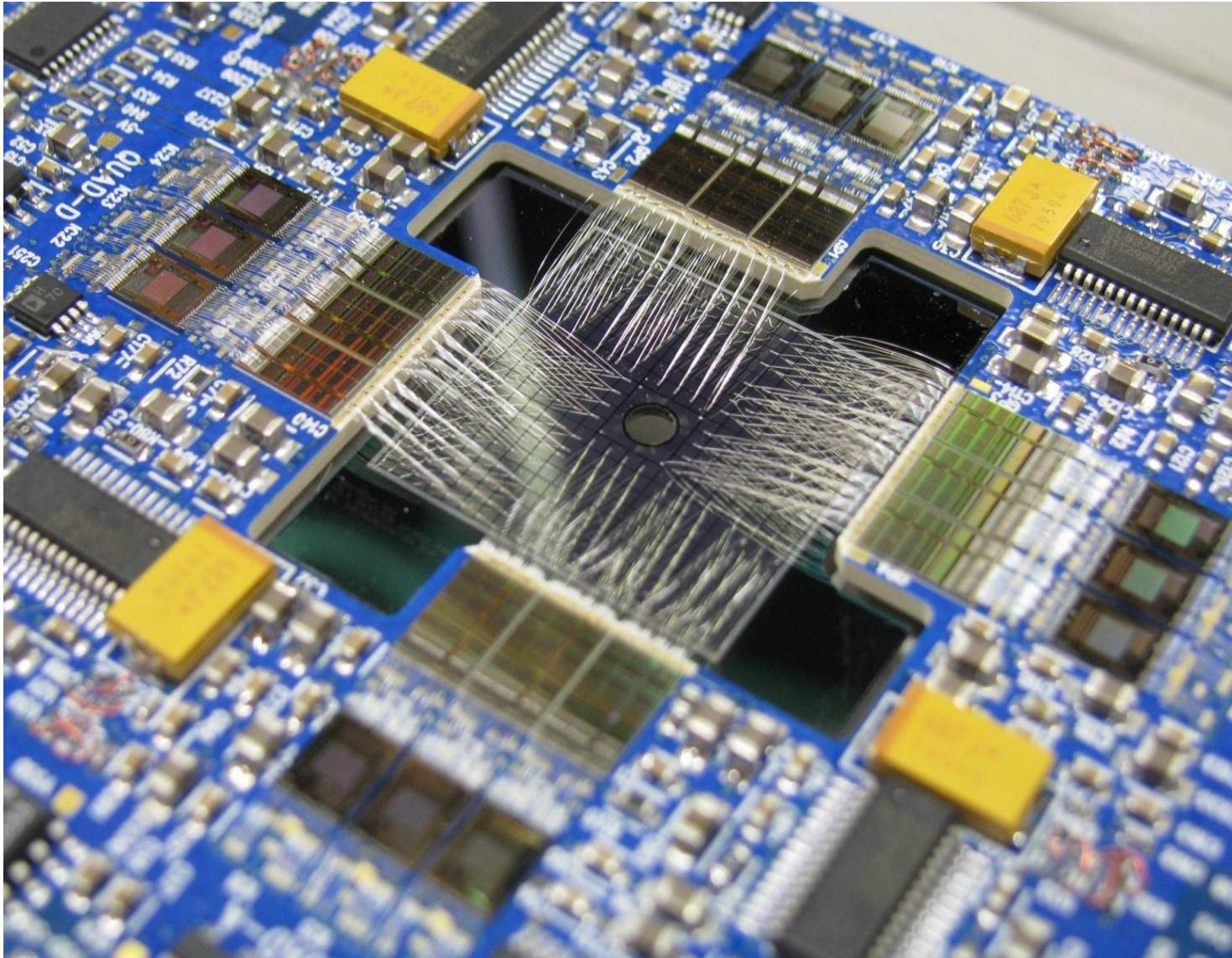
D. Peter Siddons,
NSLS-II
for the Maia Collaboration

Brookhaven National Laboratory
Upton, NY 11973
USA

What's new?

- When we started the Maia project, fly-scanning was not common. First demonstration in 2005 (not yet Maia!)
- New acquisition model; “Event-based”, photon-by-photon, with embedded scan information
- New fitting method: “Dynamic Analysis” from Chris Ryan, again, photon-by-photon.
- Very large detector array: 384 independent detectors with independent readout chains.
- ASIC-based high density readout
- FPGA-based fast, on-the-fly computation of elemental maps.
- R&D100 Award in 2011.

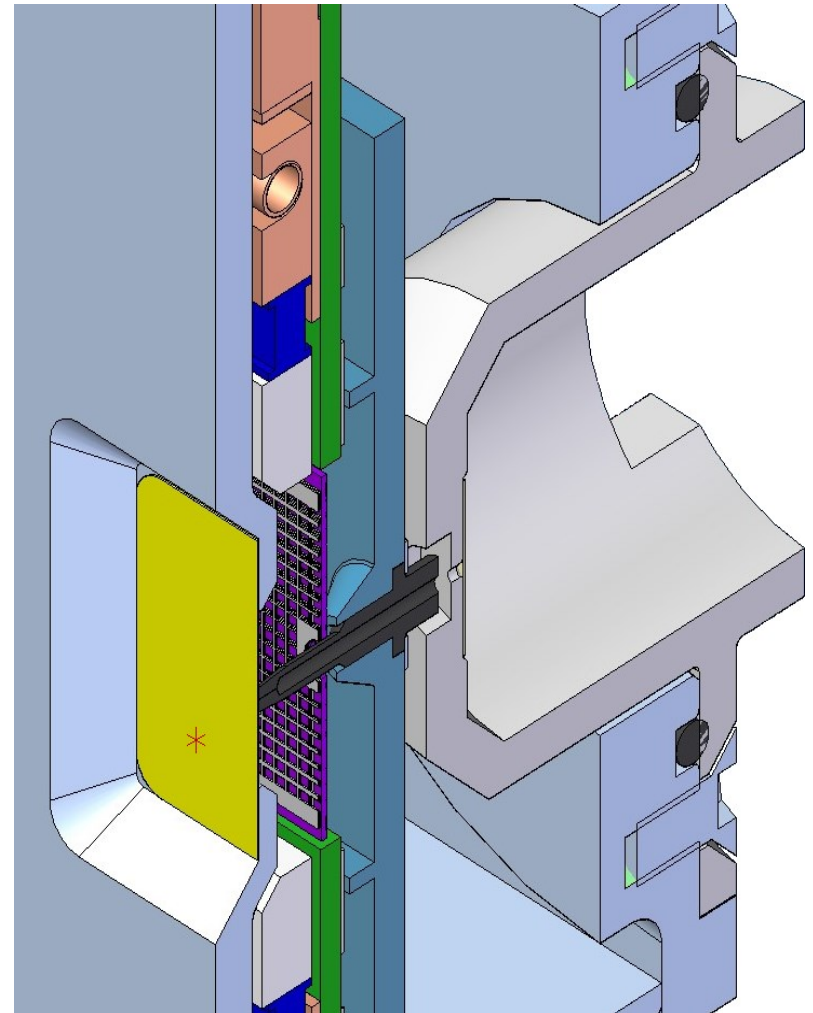
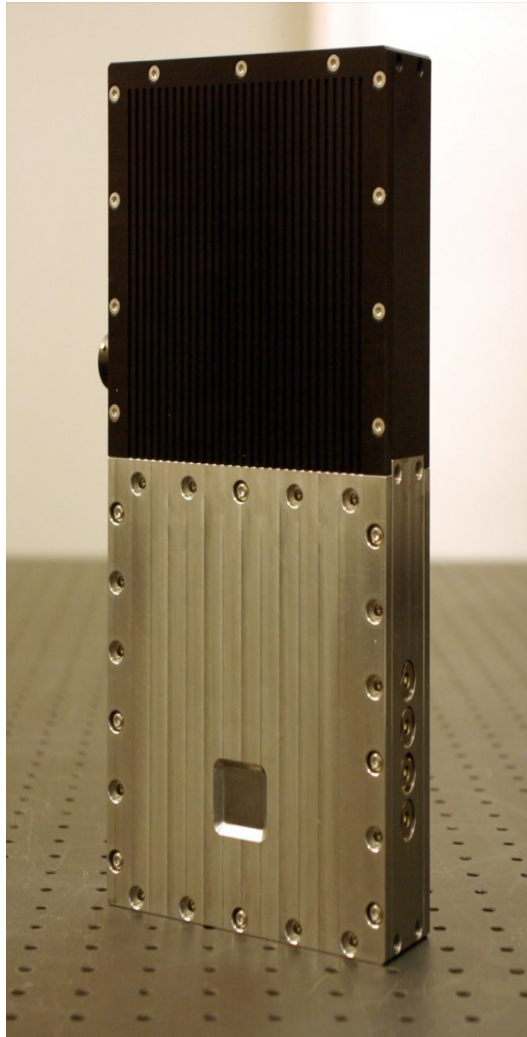
Maia Sensor + ASICs



Siddons et al., Journal of Physics:Conference series 499
(2014) 012001

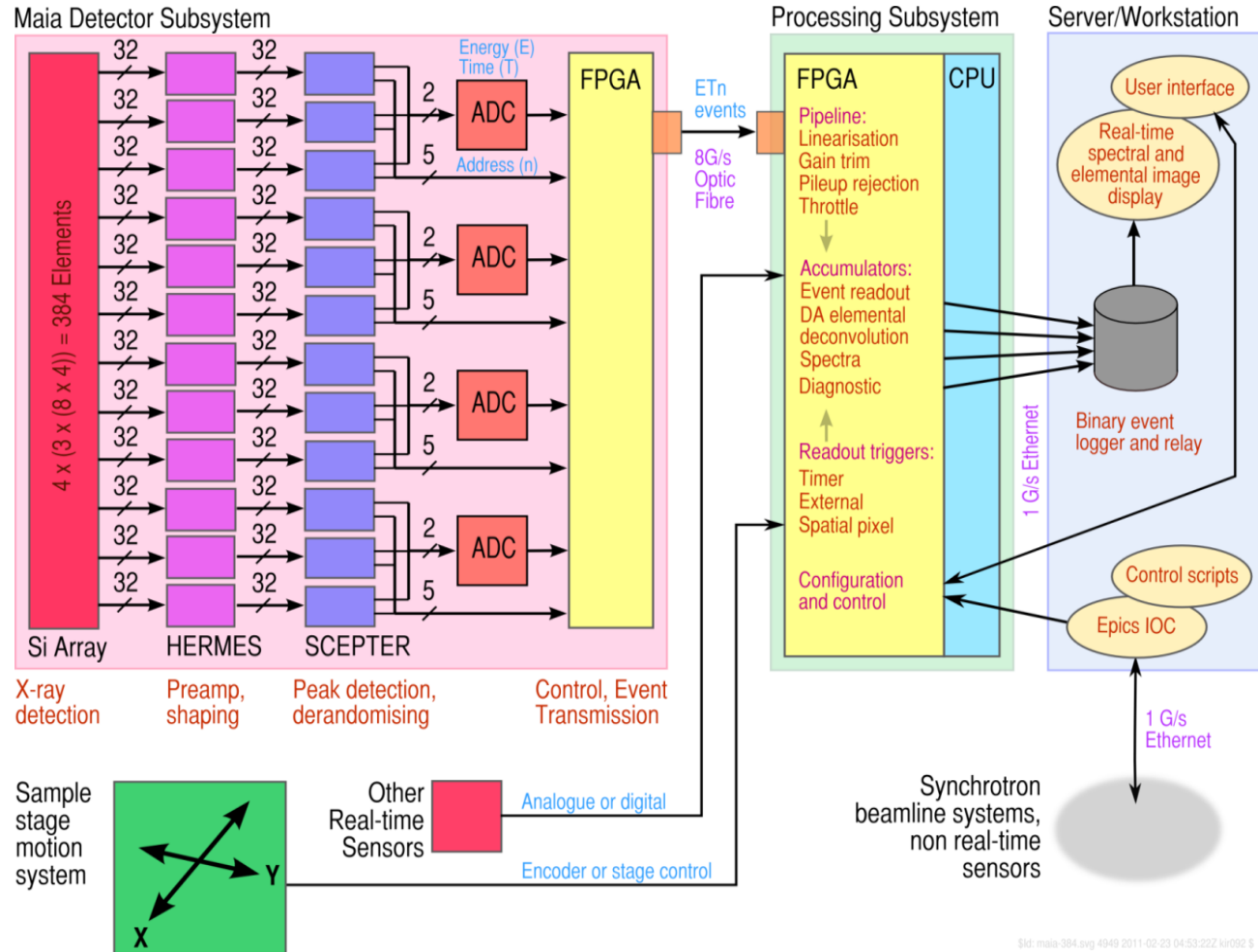
IFDEPS 2018, Annecy, France

Photo and cross-section



Block diagram of Maia processing chain

- HYMOD controls stage and reads detector
- Each photon tagged with energy, XY position and pileup status
- Initial coarse scan generates 'average' spectrum which makes DA matrix
- DA technique then presents elemental map as acquisition proceeds.



Spectral deconvolution → Dynamic Analysis method

PIXE/SXRF spectra are linear combinations of pure element spectral signatures.

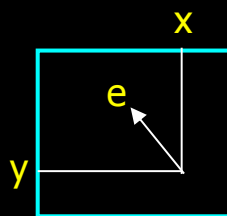
Deconvolution cast as a matrix transformation:

$$C = Q^{-1} \Gamma S$$

Concentration vector C = Transform matrix Q^{-1} Γ Spectrum vector S

PIXE/SXRF Imaging:

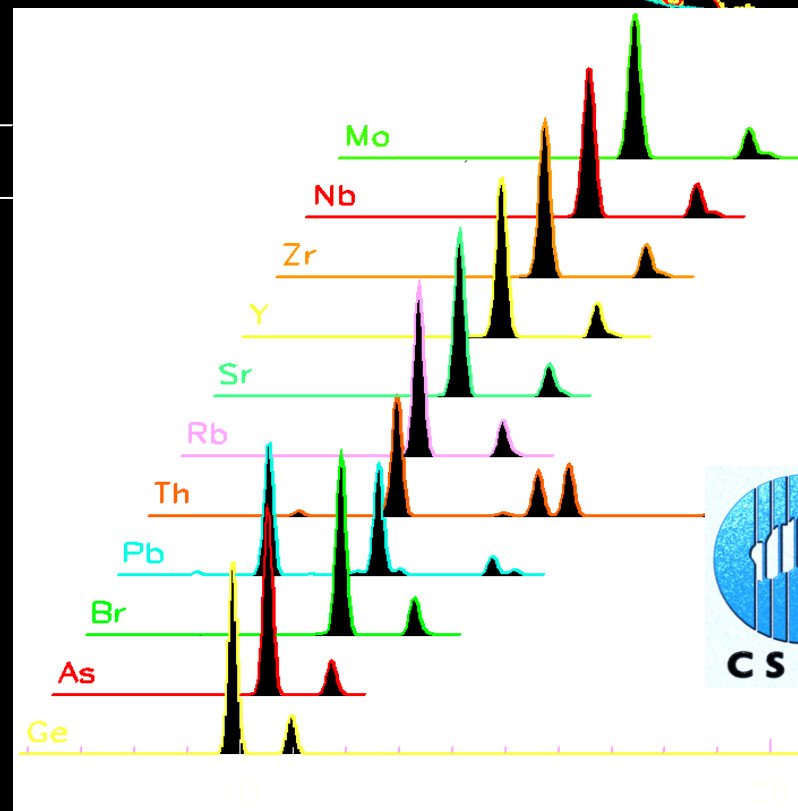
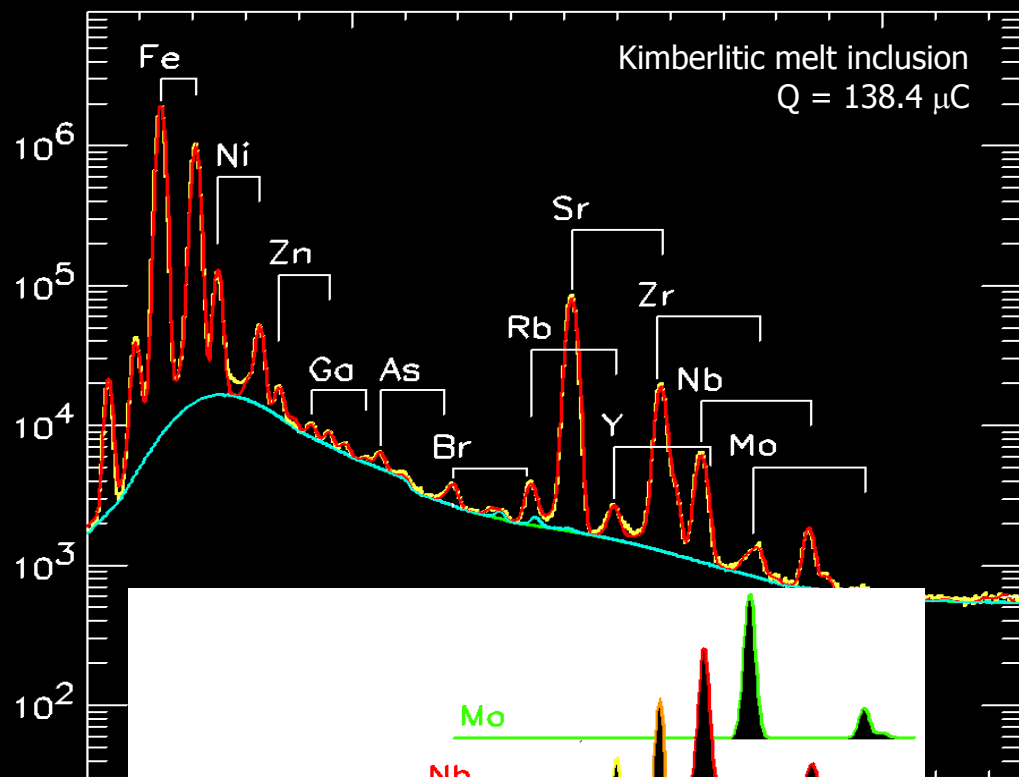
X-ray event: Energy 'e'
Position 'x,y'



The elements of the Γ matrix for column 'e' are the increments to make to all images at 'x,y'.

GeoPIXE software

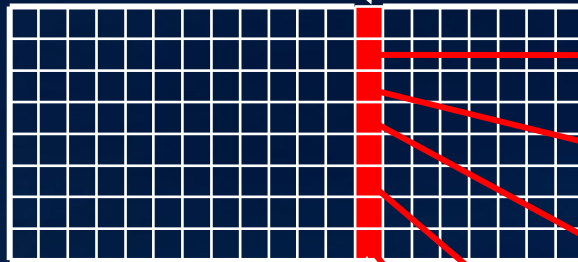
Now at APS, NSLS, CLS, 19 NMP labs



Real-time Elemental Imaging ...

Matrix column

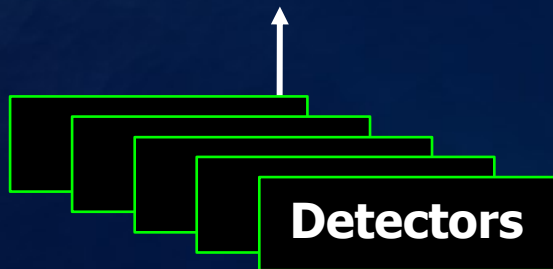
Dynamic Analysis
 Γ matrix



N:



Event: Detector **N**, ADC count **i(E)**, Position **X,Y**



X

Y

As

Fe

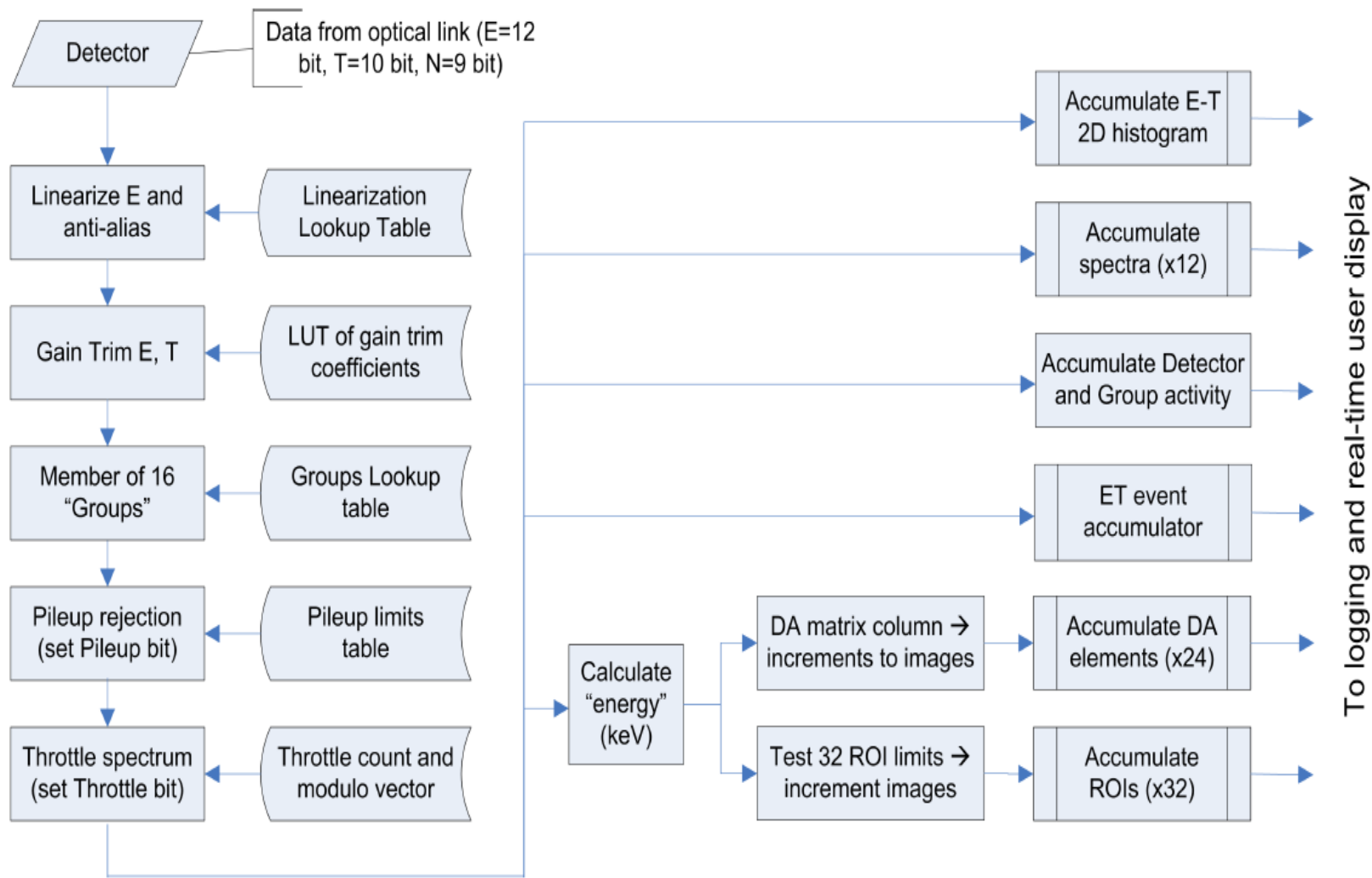
Cu

Zn

Cd

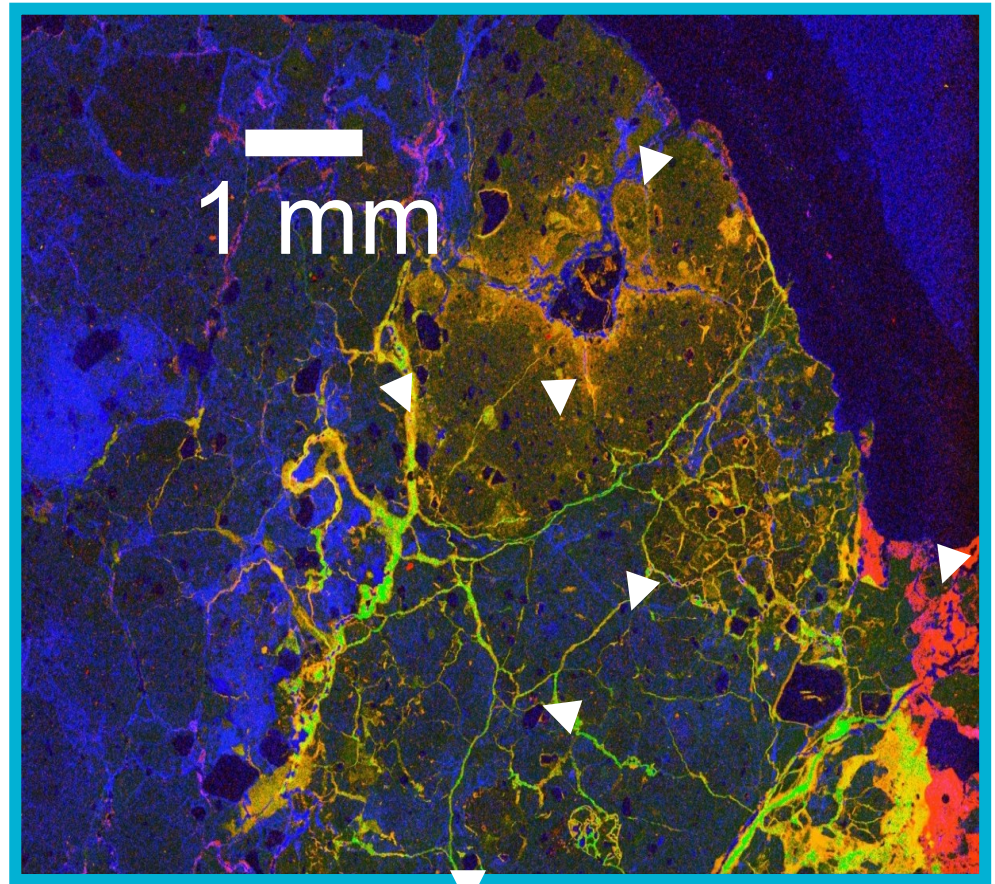


Maia data flow



Extremely inhomogeneous samples

- 9600 x 8000 pixels
- 0.6 ms/pixel
- 1.25 μm pixels
- Arrows point to micron-scale gold particles

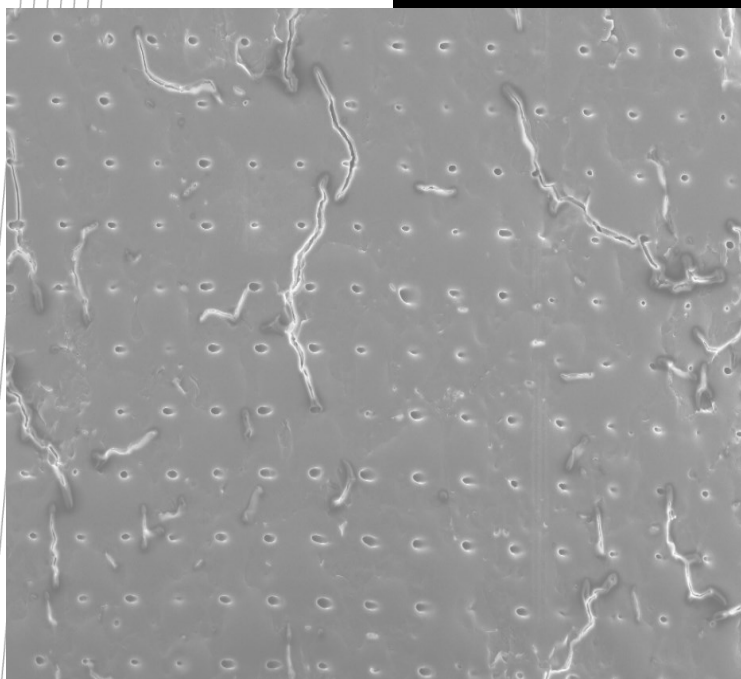


Rob Hough, James Cleverley,
CSIRO, private communication

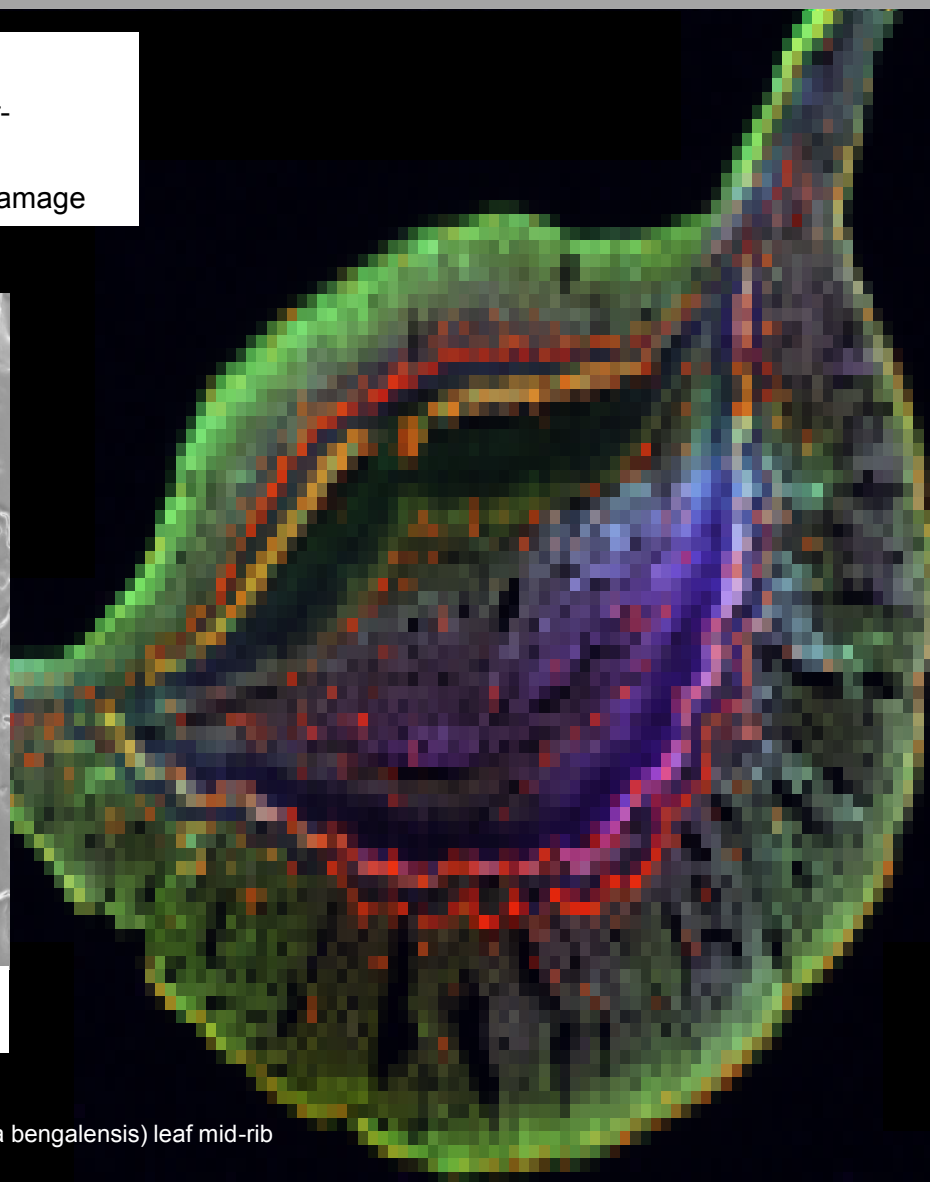
Why high definition imaging?

Minimize damage

- **Avoid step-scans and long pixel dwell**
 - ✓ Long dwells → few pixels → under-sampling
 - ✓ Concentrated dose → increased damage



SEM of wheat sample post-SXRF showing grid of damage holes (2 μm beam, 15 μm steps for 0.5 sec)



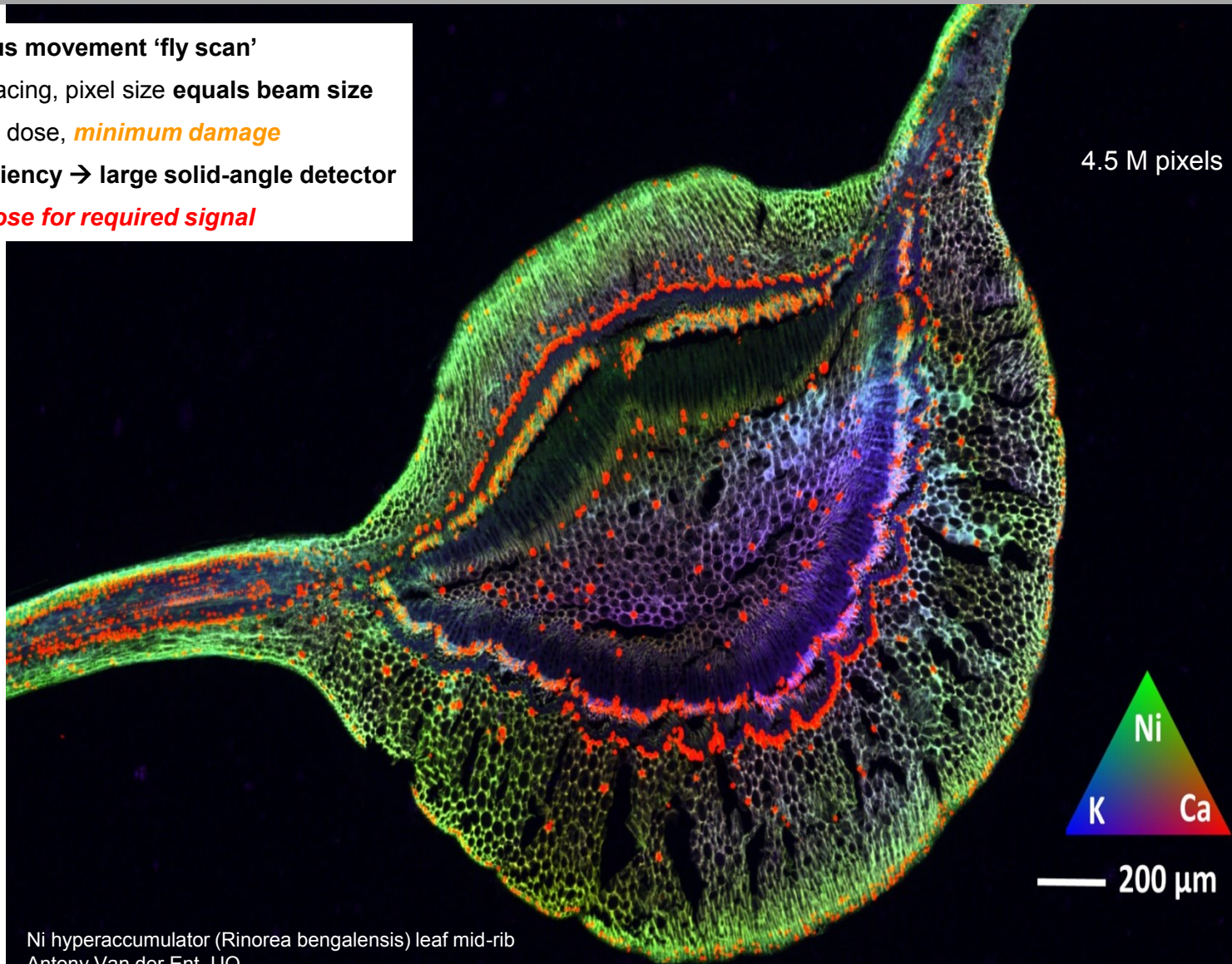
40k pixels

Ni hyperaccumulator (*Rinorea bengalensis*) leaf mid-rib
Antony Van der Ent, UQ

Why high definition imaging?

Minimize damage

- Use continuous movement 'fly scan'
 - ✓ Line spacing, pixel size equals beam size
 - ✓ Uniform dose, *minimum damage*
- Maximize efficiency → large solid-angle detector
 - ✓ *Less dose for required signal*



Ni hyperaccumulator (*Rinorea bengalensis*) leaf mid-rib
Antony Van der Ent, UQ

Maia 384
3D data-sets:
XANES imaging

Dynamic Analysis method - XANES

SXRF Spectrum - linear combination of element spectra.

... cast as a matrix transformation:

$$C = Q^{-1} \Gamma(E_b) S$$

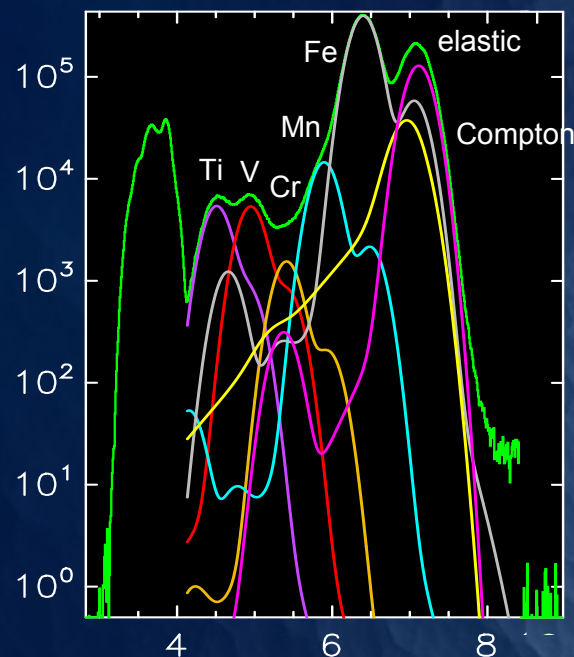
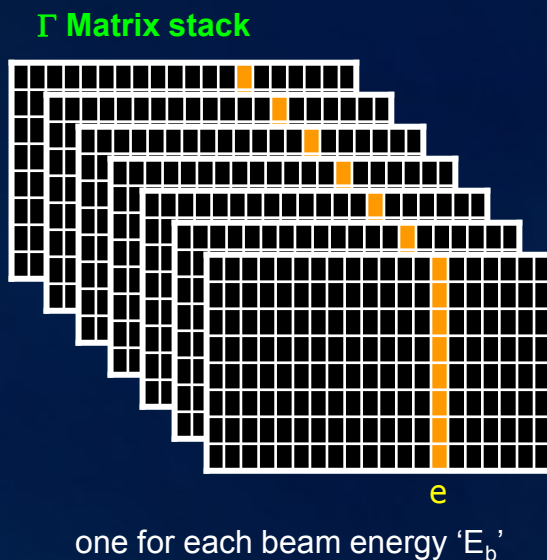


XANES Imaging:

X-ray event: Energy 'e'
Position 'x,y'
Beam energy 'E_b'

'E_b' selects matrix Γ in stack \rightarrow

'e' selects column of matrix $\Gamma(E_b) \rightarrow$ the increments to images at 'x,y'



Metal speciation in biosolids

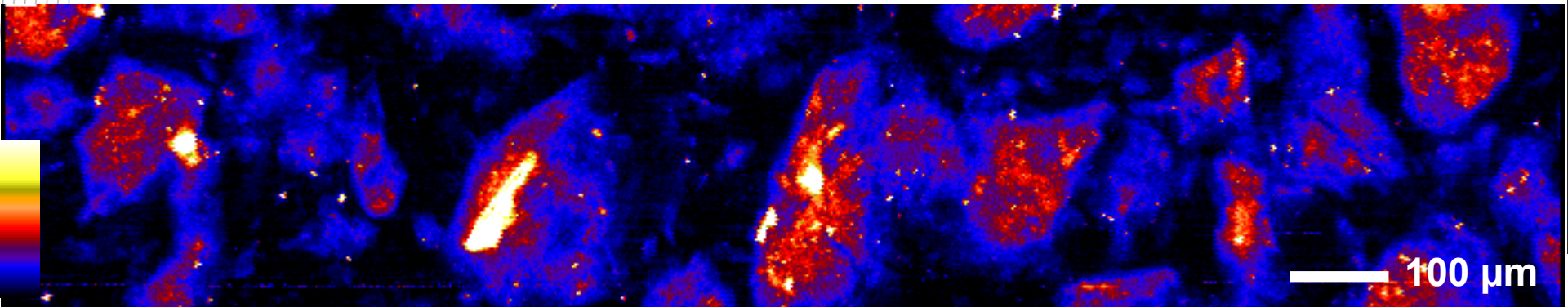
Contaminant time bomb?

Lombi, Donner, Etschmann *et al.*, U. South Australia



**PUTTING
WASTE
TO WORK**

What is the speciation of Cu?



Cu elemental map

Metal speciation in biosolids

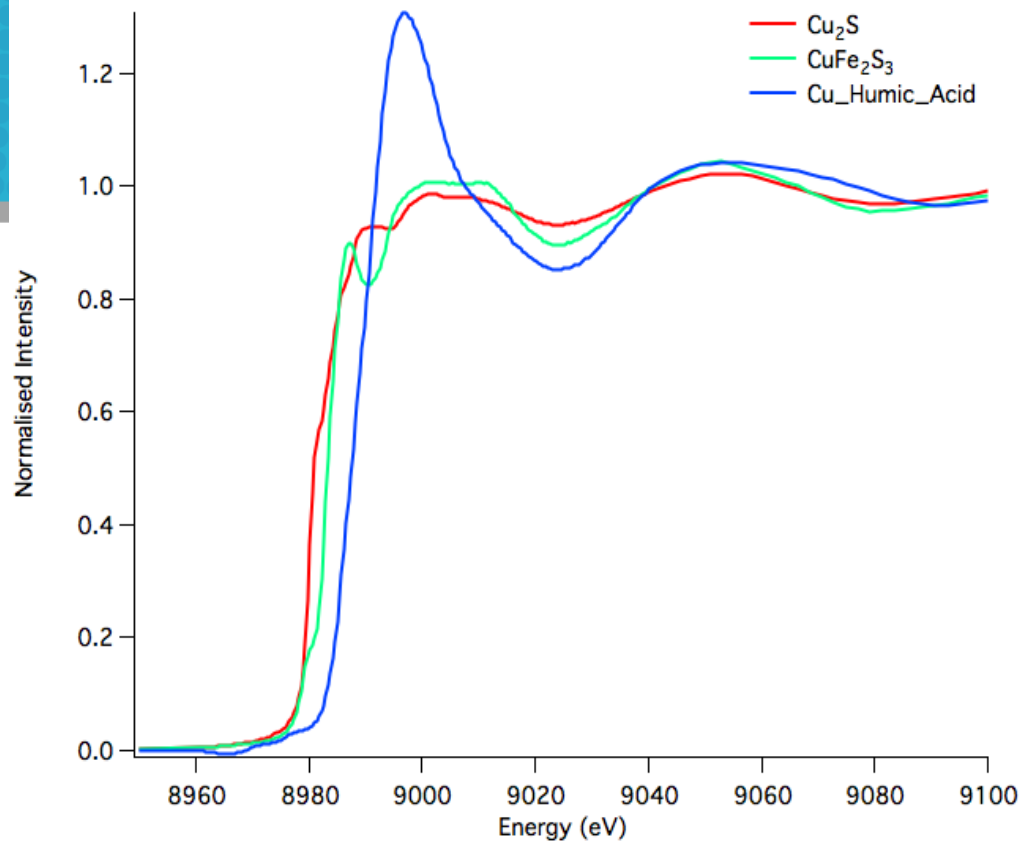
Lombi, Donner, Etschmann *et al.*

● XANES imaging

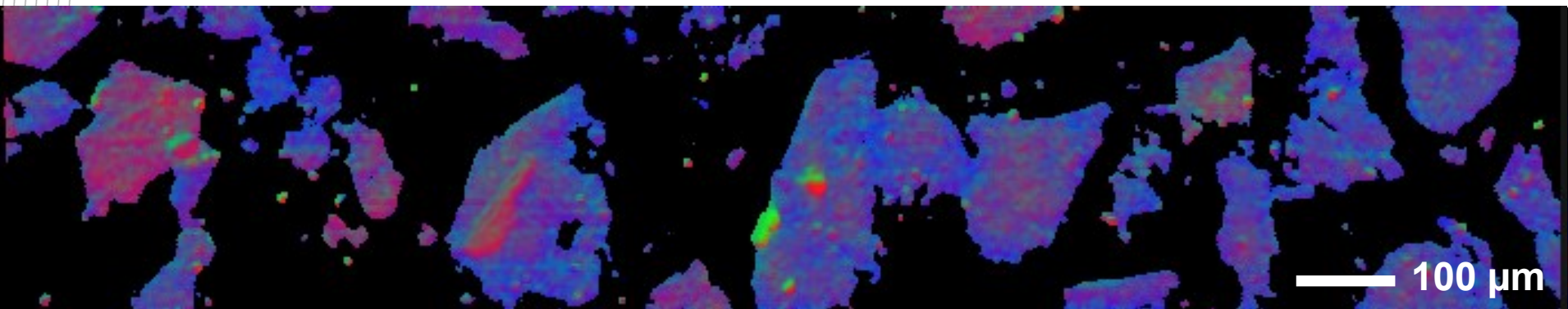
- ✓ 1.6 x 0.27 mm (800 x 136 pixels)
- ✓ 1.9 ms/pixel
- ✓ 80 energies across Cu K edge

● Component fitting

- ✓ from bulk XAS and PCA analysis
- ✓ PCA analysis indicated 3 significant components

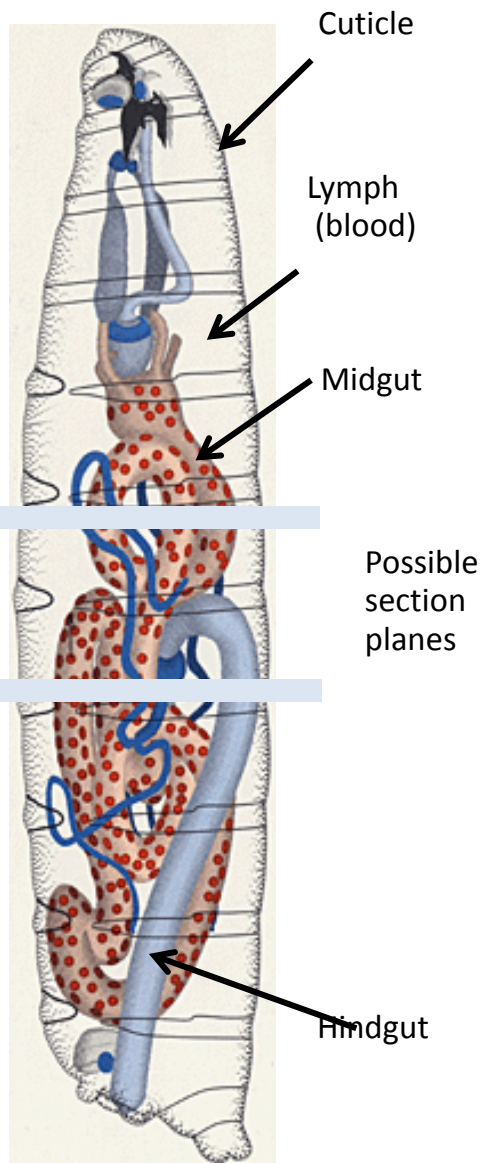


XANES imaging: Cu speciation map



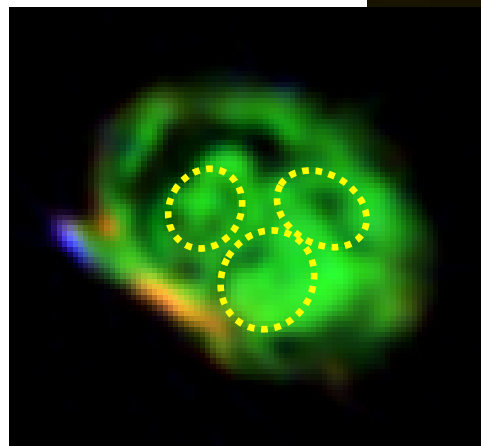
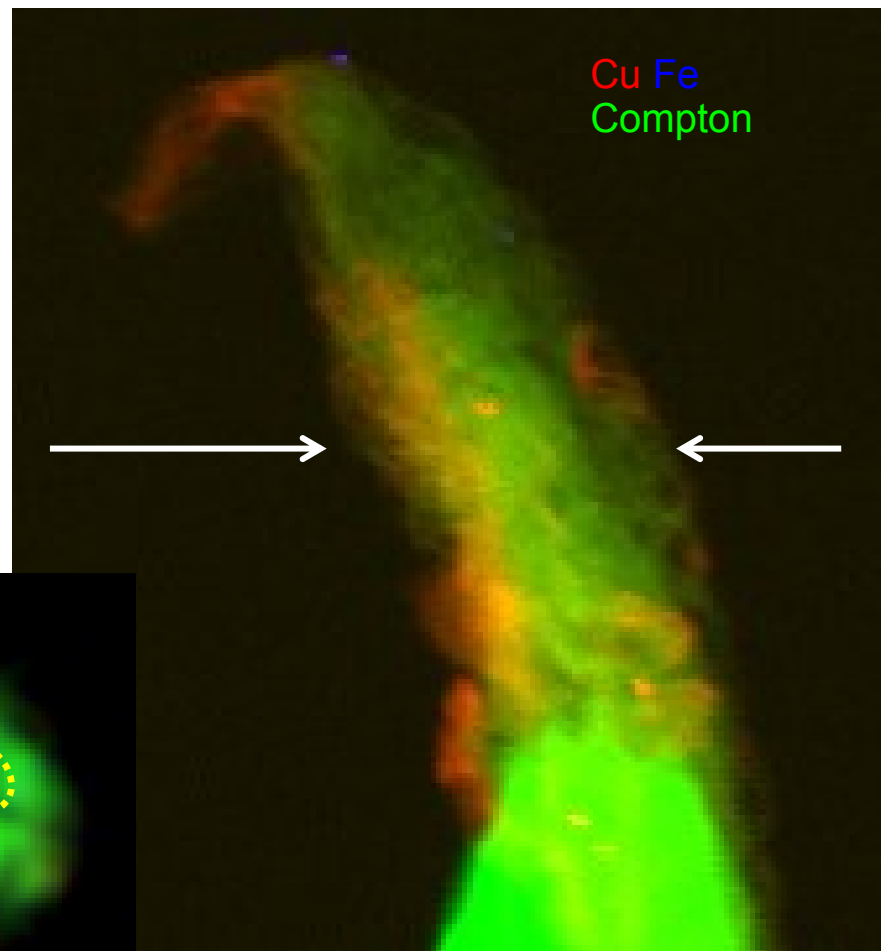
Red= Cu_2S , Green= CuFe_2S_3 , Blue= Cu-HA

Cu oxidation state mapping in Drosophila (Cu efflux transporter ATP7 knockout)



2D XFM

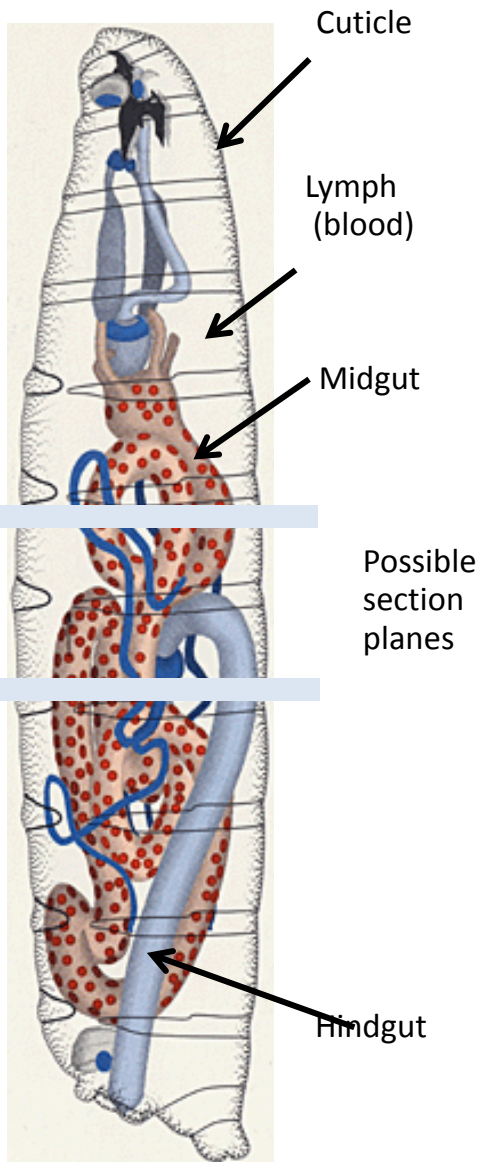
2D Fluor.
Tomography
section



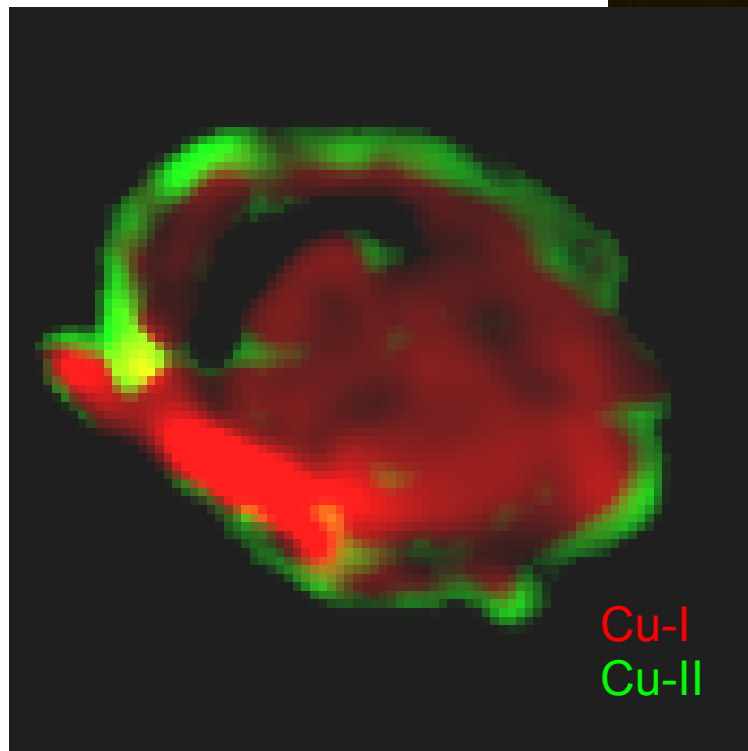
0.4 x 0.55 mm; 200 x 275 pixels, 16 minutes

0.3 mm x 360°, 400 angles; 150 x 400
pixel sinogram, 18 minutes

Cu oxidation state mapping in *Drosophila* (Cu efflux transporter ATP7 knockout)

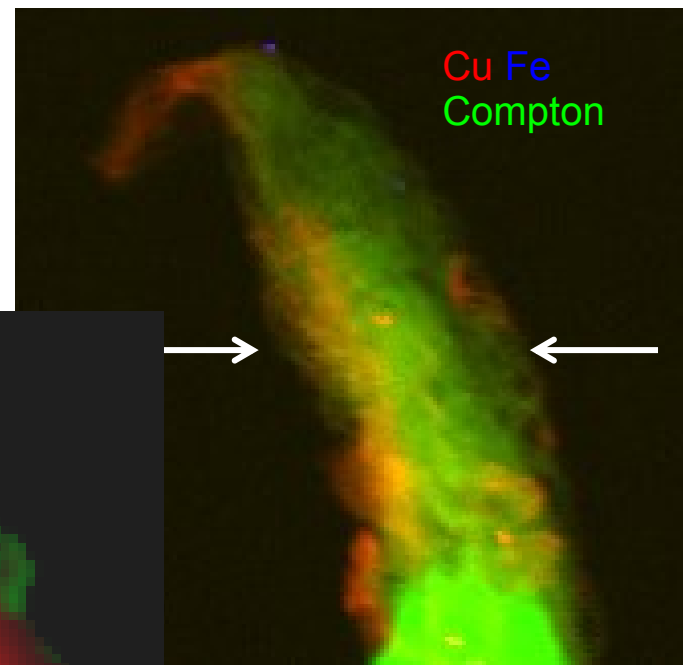


**XANES
Tomography
section**



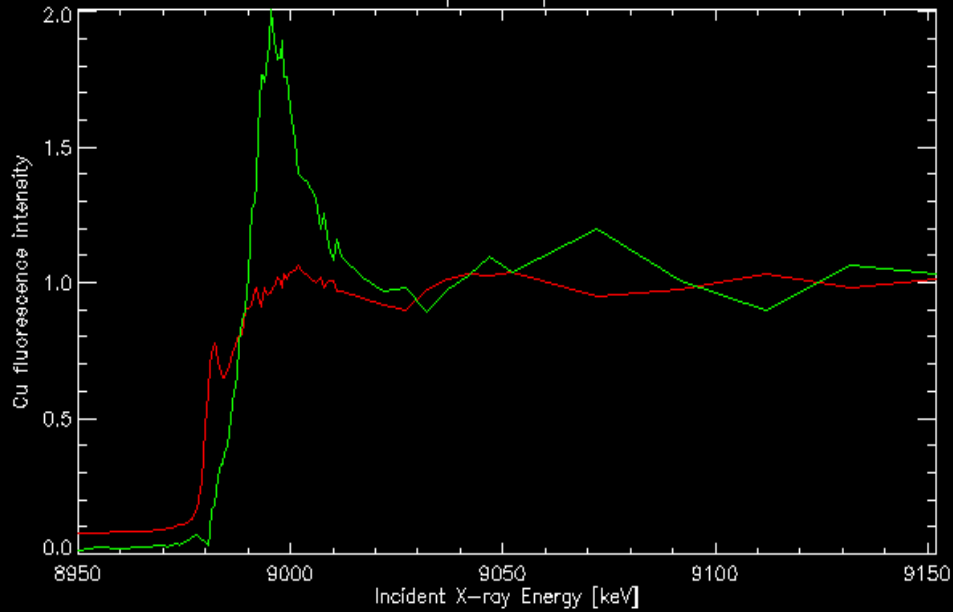
0.3 mm x 180°, 100 angles; 150 x 100 pixel sinogram, 4.7 minutes
per energy x 80 energies

2D XFM



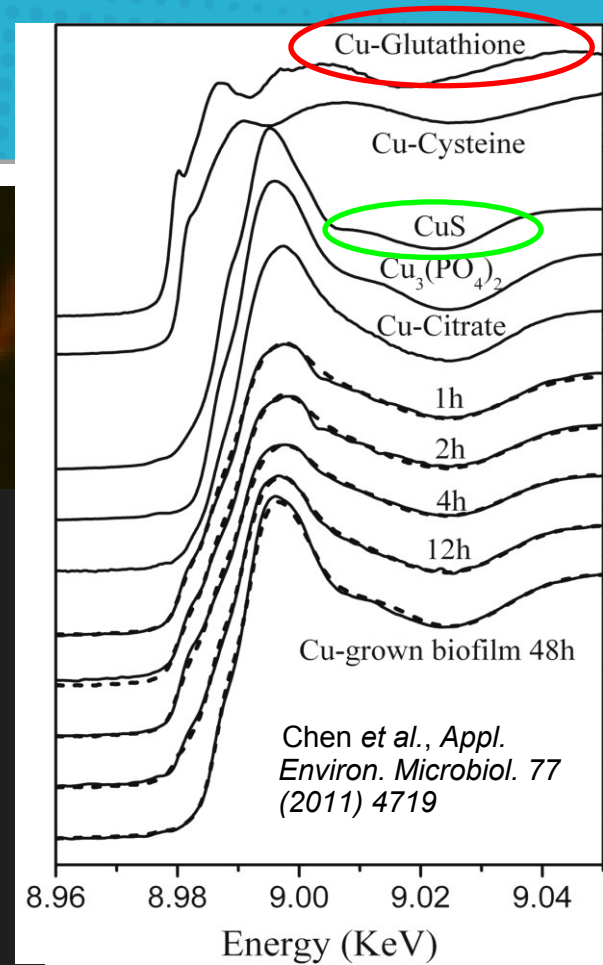
0.4 x 0.55 mm; 200 x 275 pixels,
16 minutes

*Cu speciation maintained in
Drosophila...*
de Jonge *et al.*, in prep.



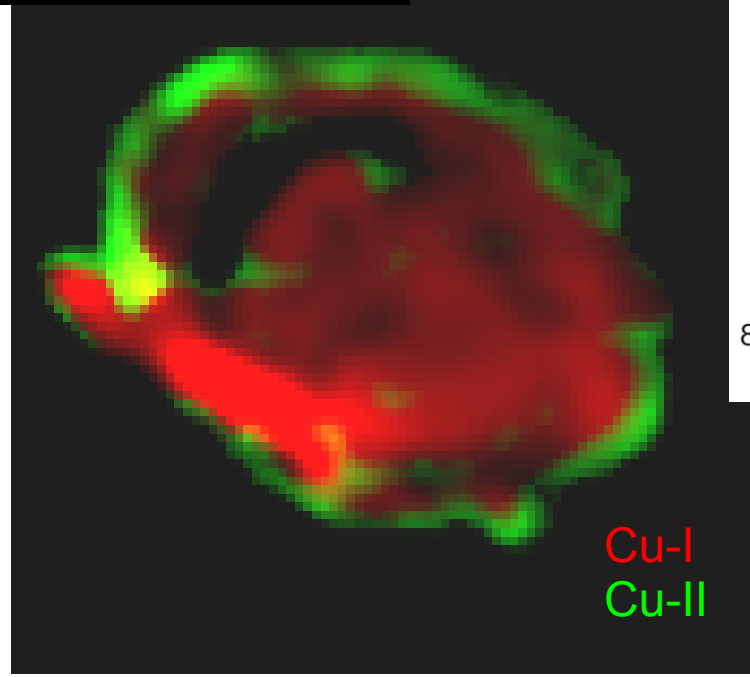
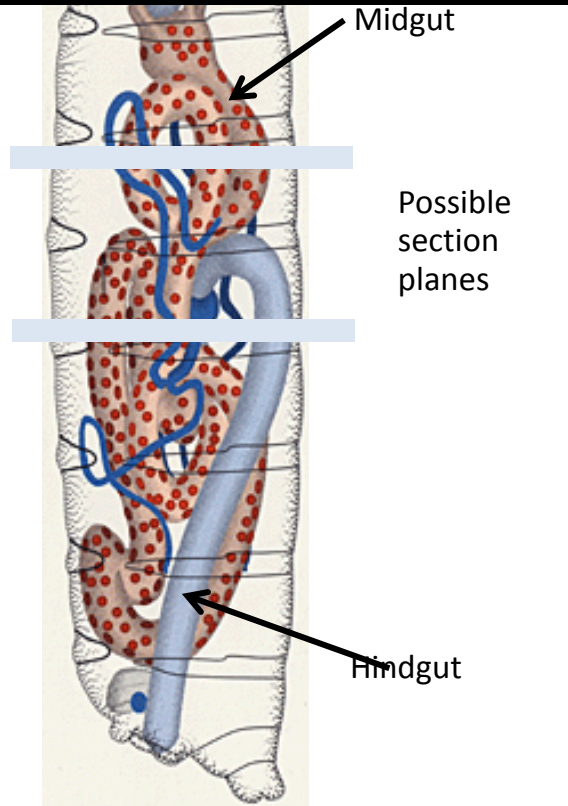
sophila
(out)

2D XFM



Chen *et al.*, *Appl. Environ. Microbiol.* 77 (2011) 4719

Cu speciation maintained in Drosophila...
de Jonge *et al.*, in prep.

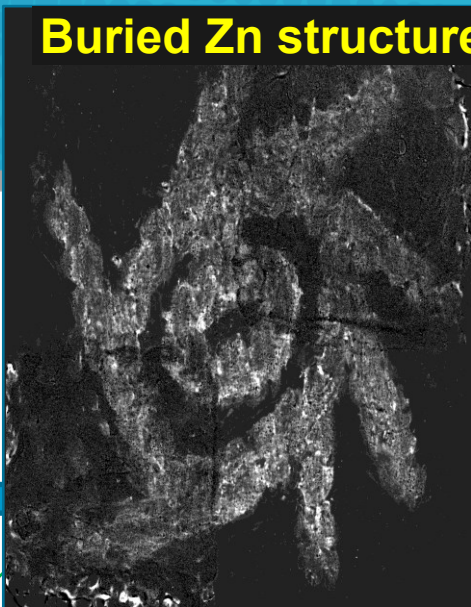


0.3 mm x 180°, 100 angles; 150 x 100 pixel sinogram, 4.7 minutes per energy x 80 energies

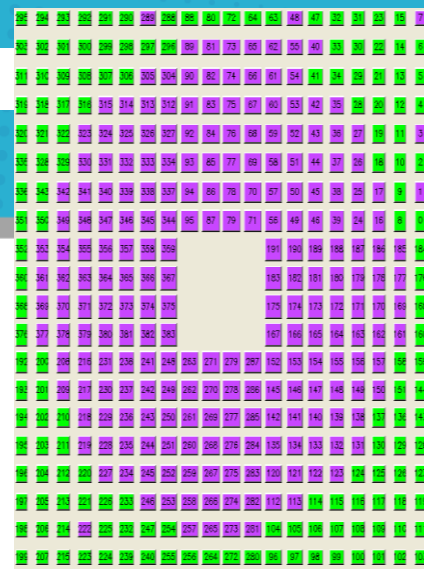
Maia Depth Sensitivity

Depth contrast

Buried Zn structure



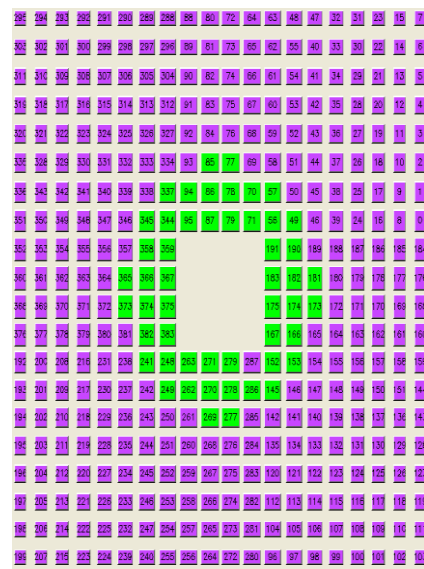
outer



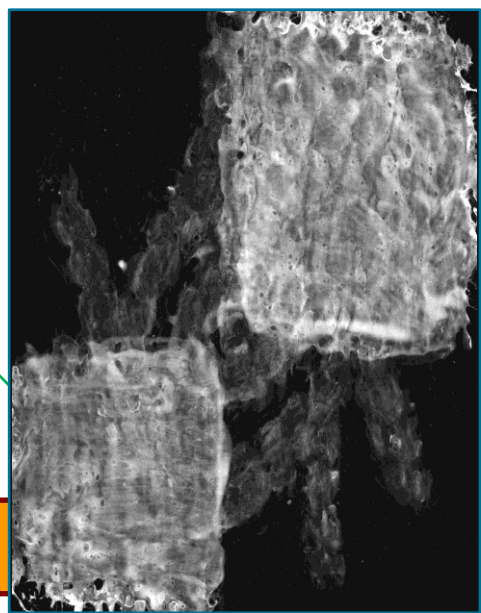
More "surface" sensitive

Zn map ("inner" minus "outer" scaled)

"See" deeper



inner



Zn map (all detectors)

beam

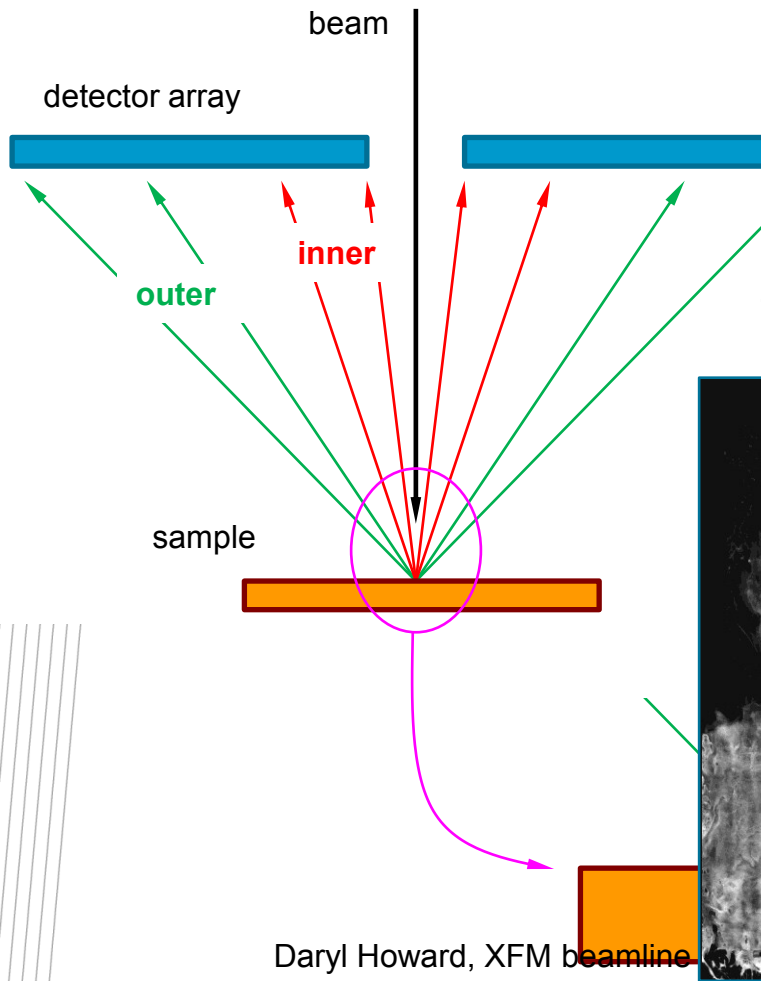
detector array

outer

inner

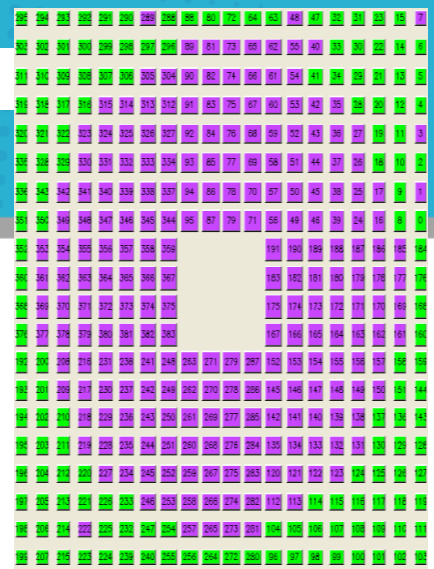
sample

Daryl Howard, XFM beamline



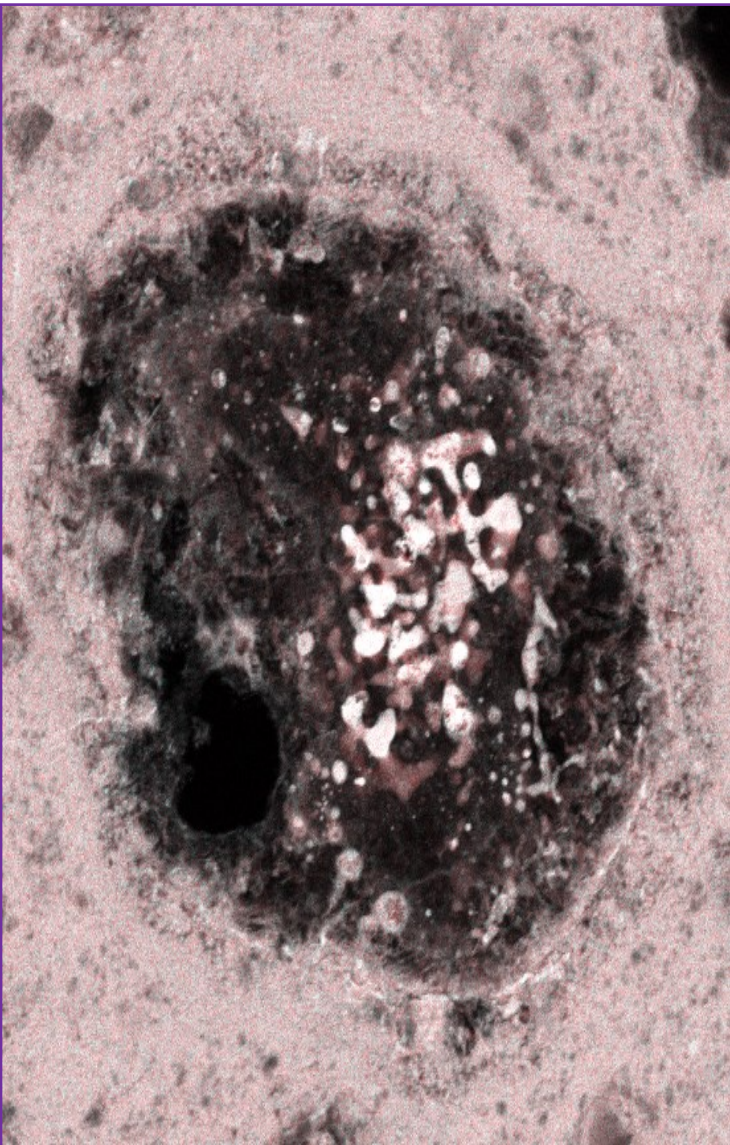
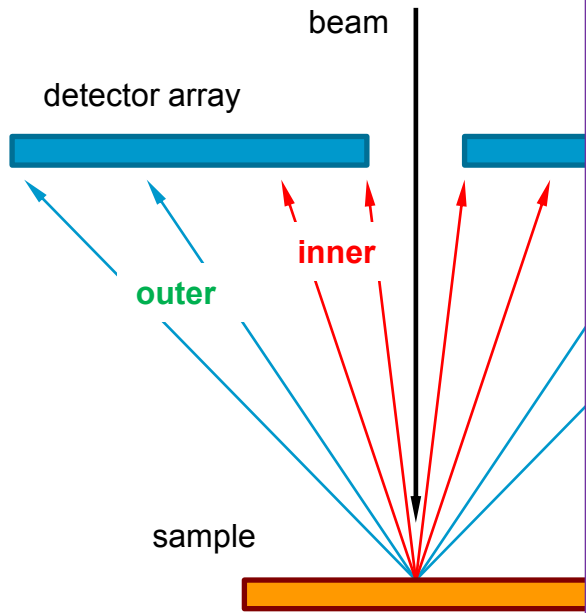
Depth contrast

outer



Blue +
Green

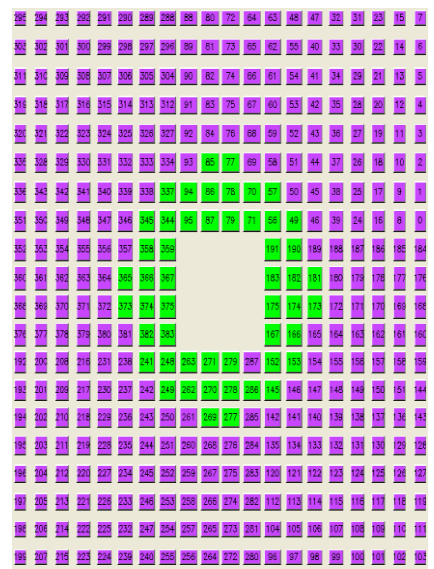
more "surface" sensitive



deeper

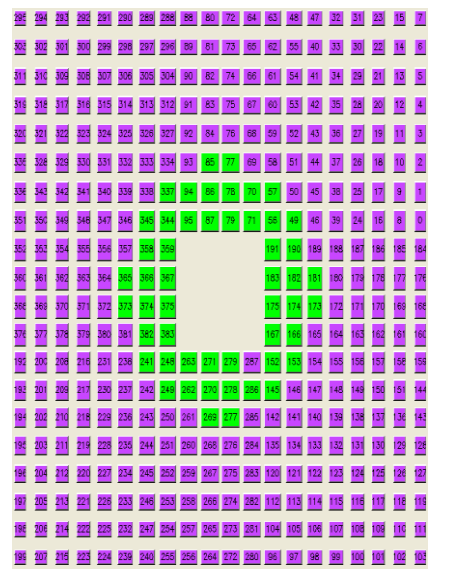
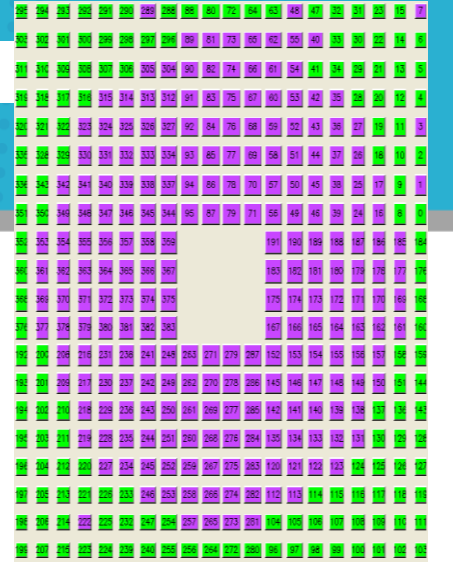
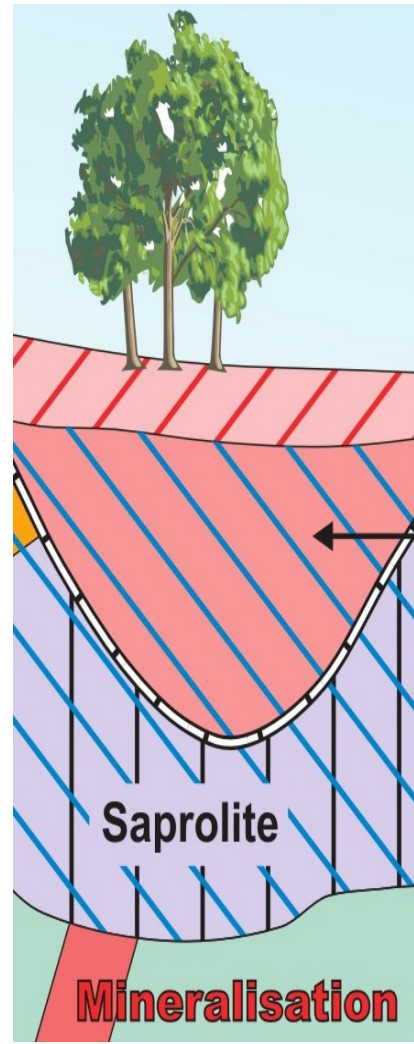
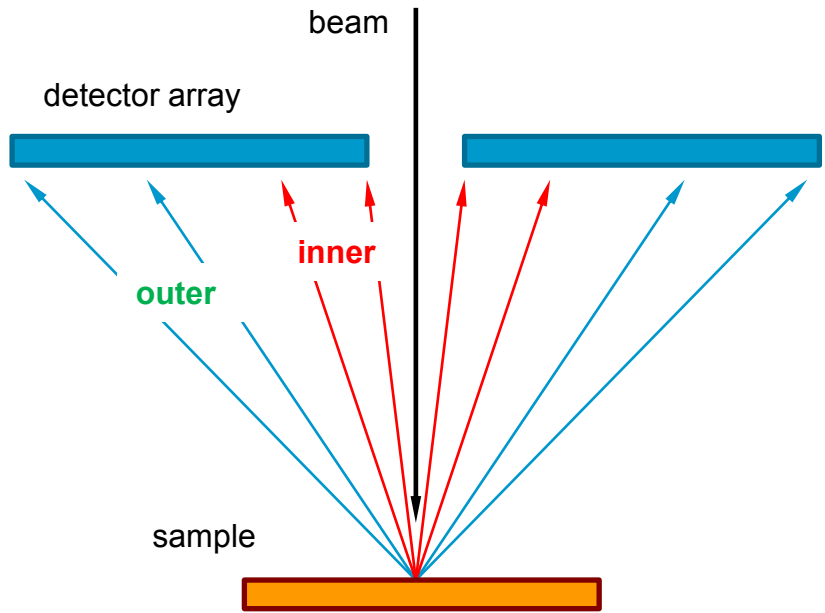
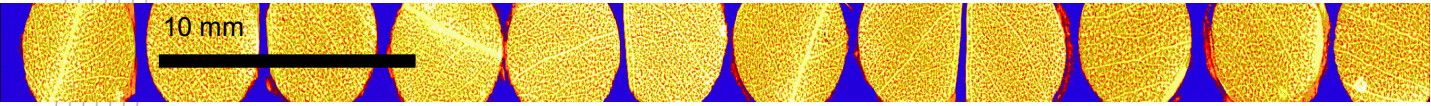
Red

inner



Depth mapping Gold precipitates in leaves

outer

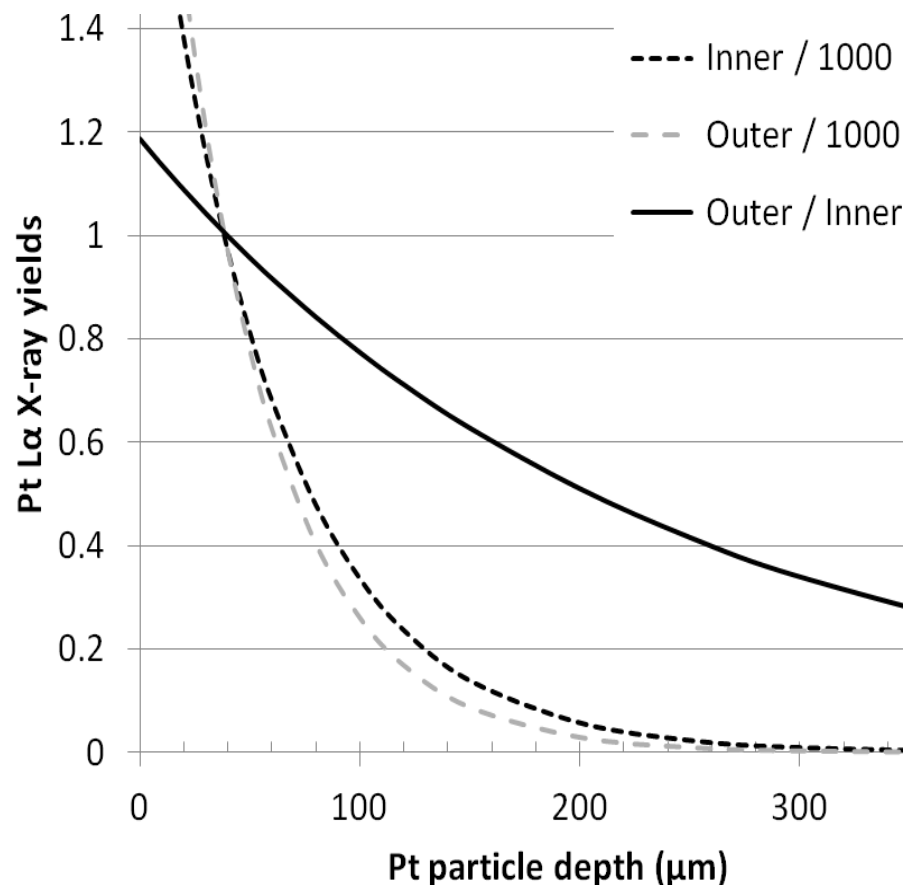
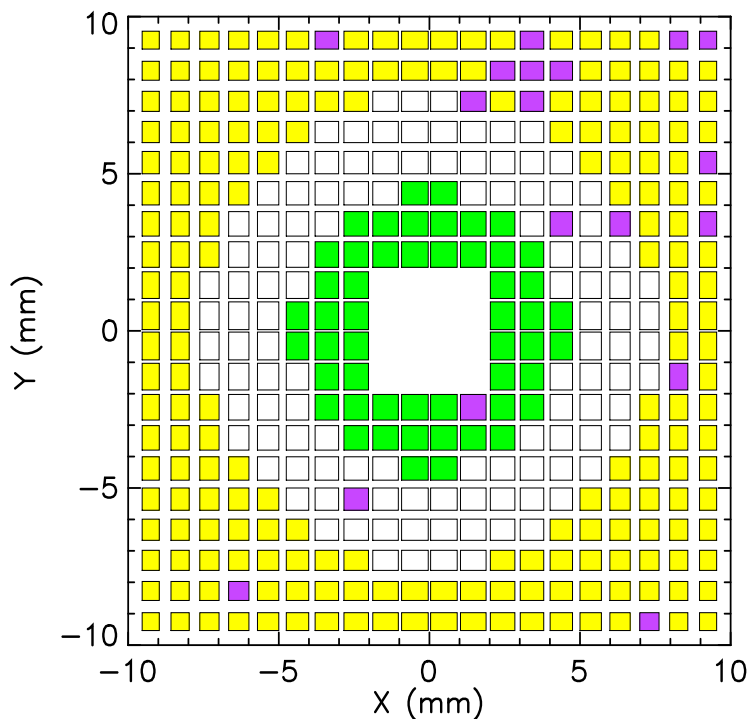


inner

Maia 384 array: Depth sensitivity and measurement

- **Depth sensitivity using large detector array**

- ✓ Angles from normal range from 13.9 ° to 52.6°
- ✓ Outer detectors “see” more self-absorption
- ✓ Inner detectors “see” deeper particles
- ✓ **Ratio “outer” / “inner” → depth measure**

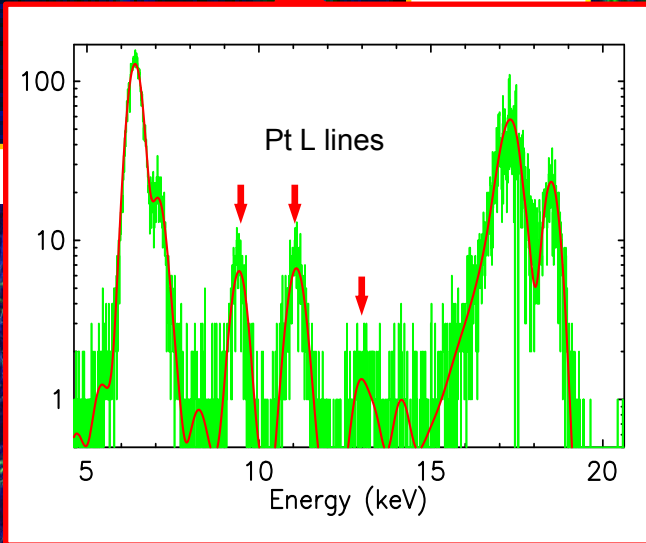
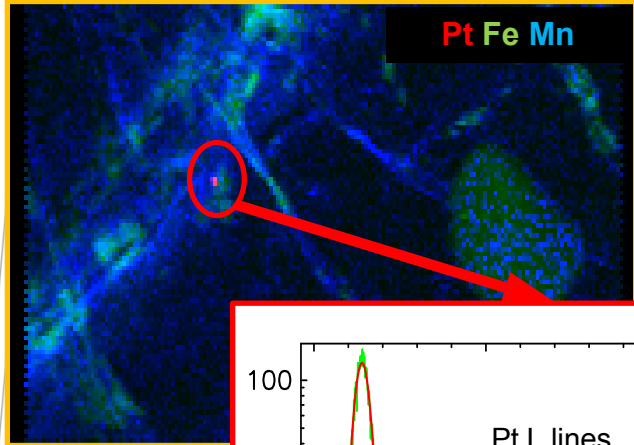


Pt L α yields, “outer”/“inner” ratio for Pt particle in olivine at 18.5 keV beam energy

PGM Search: Depth mapping

Pt Fe Mn

200 μm



: Barnes et al., Contrib. Mineral. Petrol. 171, 23

20 x 5 mm, 10002 x 2502 pixels, 0.49 ms

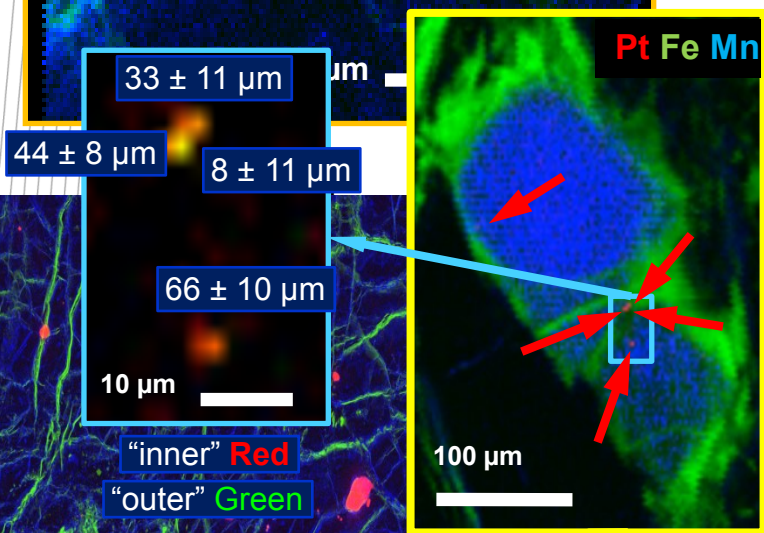
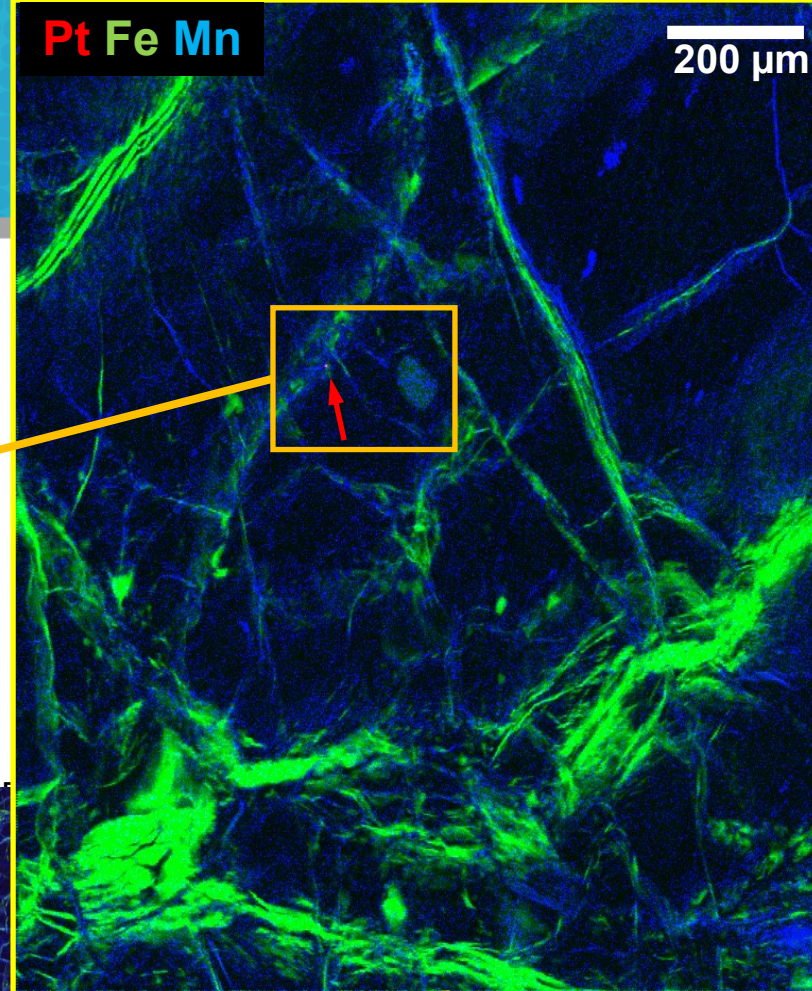
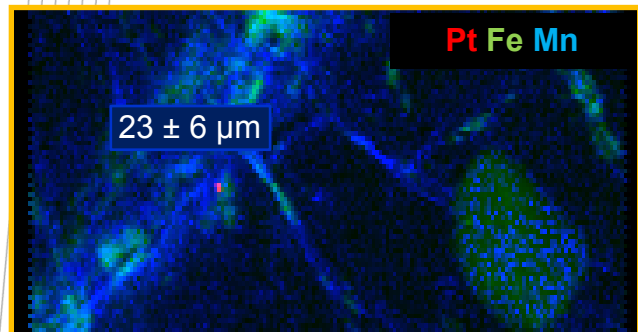
1 mm

Cr Fe Mn

PGM Search: Depth mapping

Pt Fe Mn

200 μm



Barnes et al., Contrib. Mineral. Petrol. 171, 23

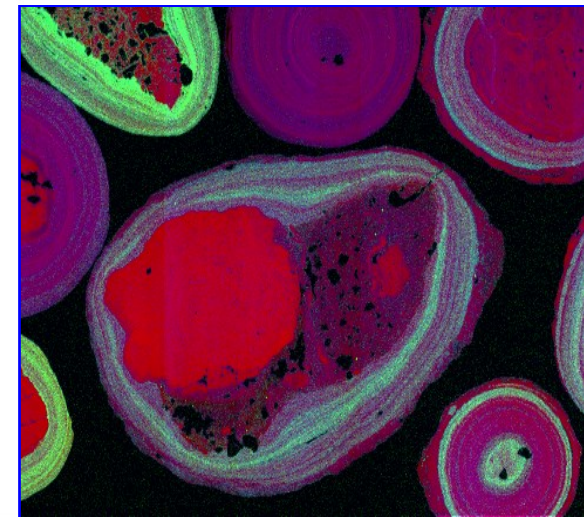
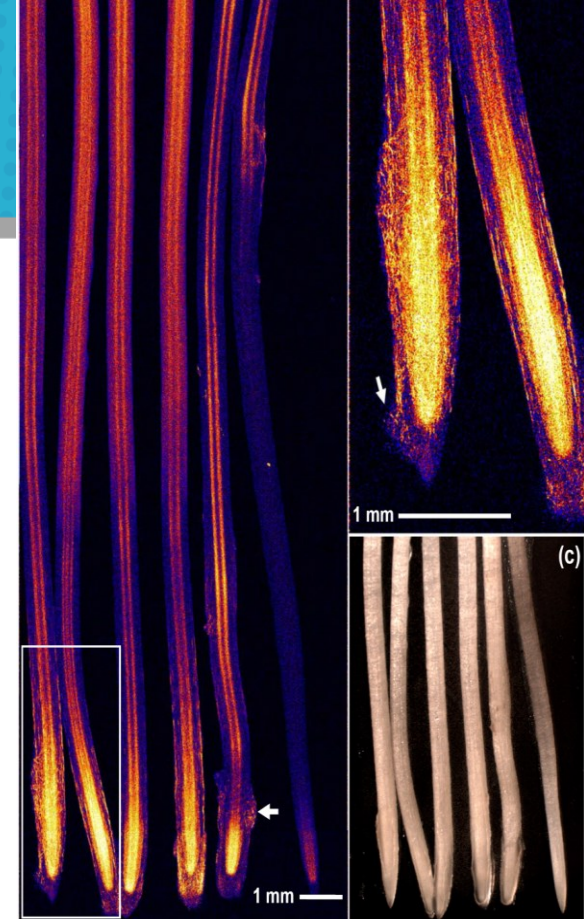
1 mm

20 x 5 mm, 10002 x 2502 pixels, 0.49 ms

Cr Fe Mn

Conclusions

- **Maia imaging system**
 - ✓ Large detector array with event-mode acquisition, real-time processing
 - ✓ **Enables high definition XFM with images up to 1G pixels (up to 100M typ.)**
- **Analytical challenge (~10⁸ spectra, composition contrasts)**
 - ✓ **DA → MPDA method**: quantitative images despite strong composition contrasts (now part of GeoPIXE s/w)
 - ✓ Still high throughput at **~10⁵ pixels per second** processing
- **2D ~10⁸ pixels → 3D imaging modes**
 - ✓ XANES imaging (~100 energies)
 - ✓ 3D Fluorescence tomography (~200 angles)
 - ✓ XANES tomography
- **User science: Maia @ XFM (AS), P06 (Petra III, DESY), SRX (NSLS-II), CHESS**
 - ✓ **Large area, high definition 2D and 3D mapping**
 - ✓ High throughput → More samples, less damage
 - ✓ **Greater user “freedom”** (sample regions, balancing fine detail and broader spatial context)

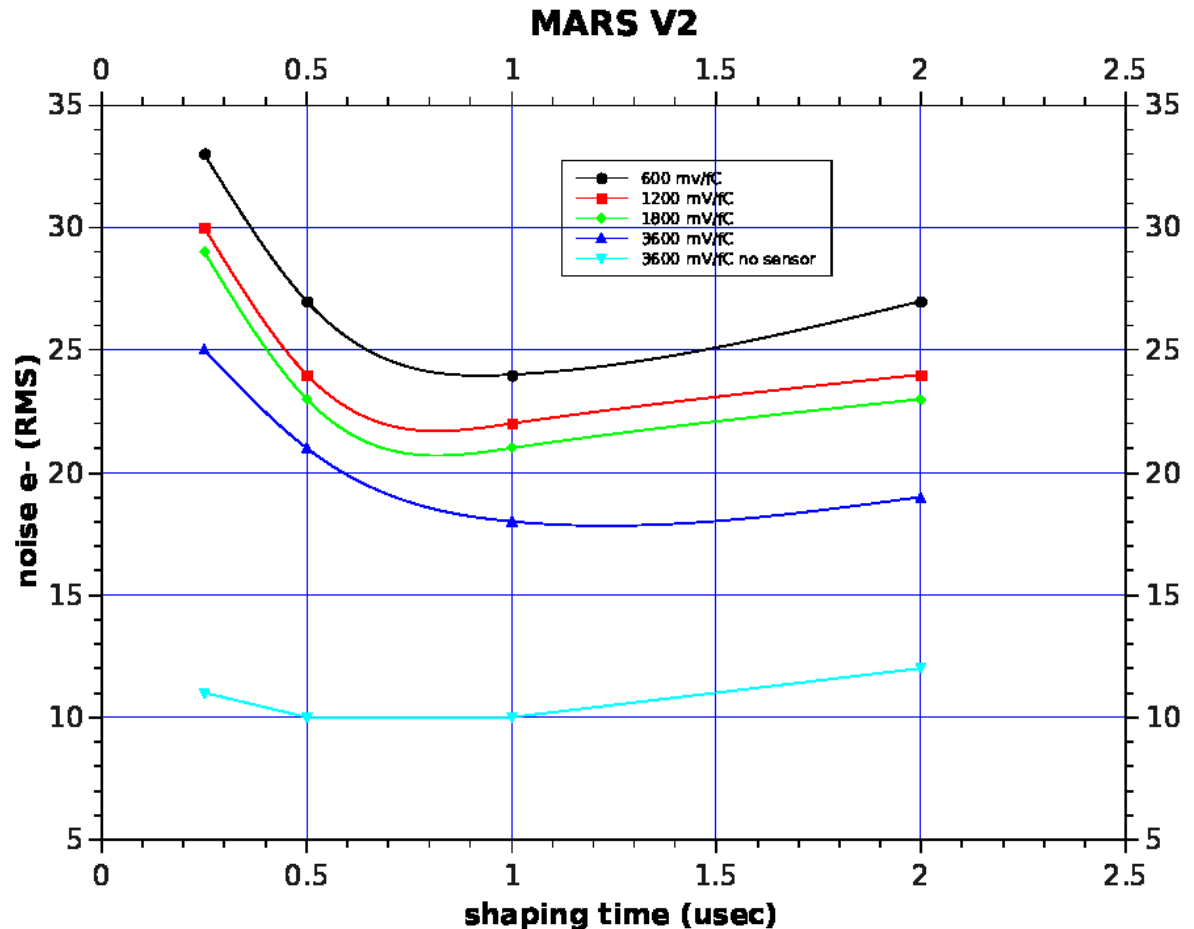


Future developments

- New ASIC (MARS) provides lower noise for low-capacitance sensors plus combining functions of both earlier chips
- New SDD sensor arrays

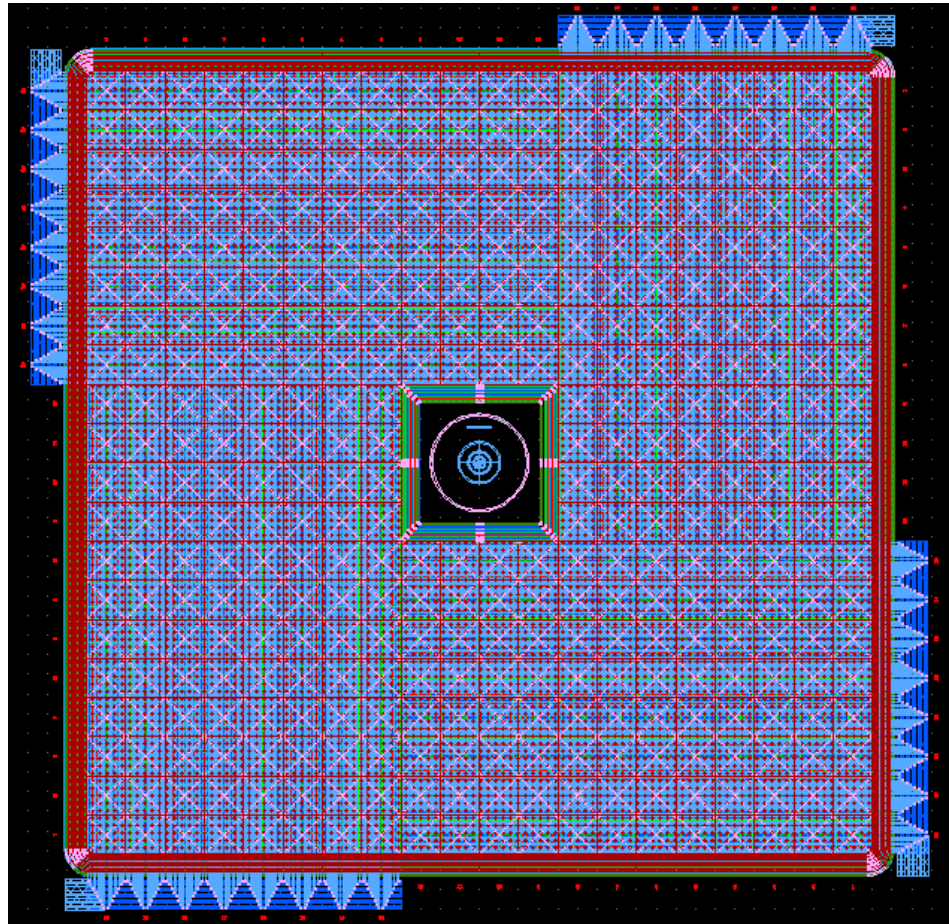
MARS noise performance

- Tested using standard Maia detectors
- MARS is designed for lower-C sensors, so no real improvement over HERMES.
- Lowest curve shows noise with no sensor.



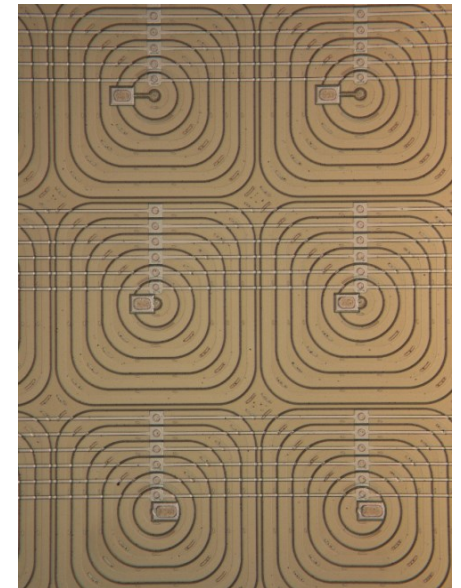
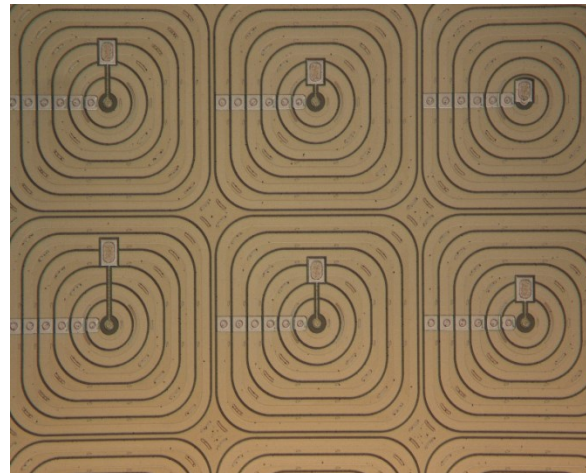
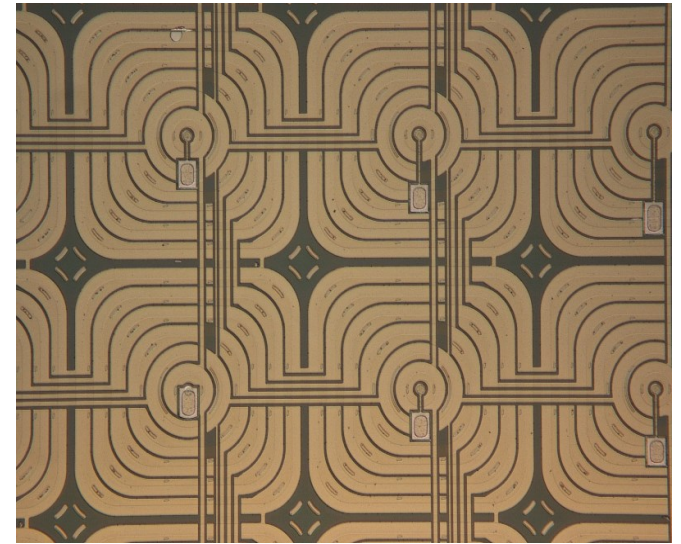
SDD arrays

- 20 x 20 array of 1mm devices (to match Maia's layout)



SDD arrays

- 20 x 20 array of 1mm devices (to match Maia's layout)



Summary

- Maia enables “Hi-res” fluorescence imaging, Megapixels rather than kilopixels
- “On-the-fly” scanning minimizes lost time due to motion system
- Higher dimensionality scans (XANES, tomography) become practicable
- Real-time data analysis provides prompt feedback to experimenters

The Maia collaboration

BNL:

D. P. Siddons, A. J. Kuczewski, A. K. Rumaiz, G. Giacomini, G. De Geronimo, D. Pinelli.

CSIRO

R. Kirkham, P.A. Dunn, C. G. Ryan, D. Parry, G. F. Moorhead, M. Jensen, R. Dodanwela