Machine learning tools in analytical TEM

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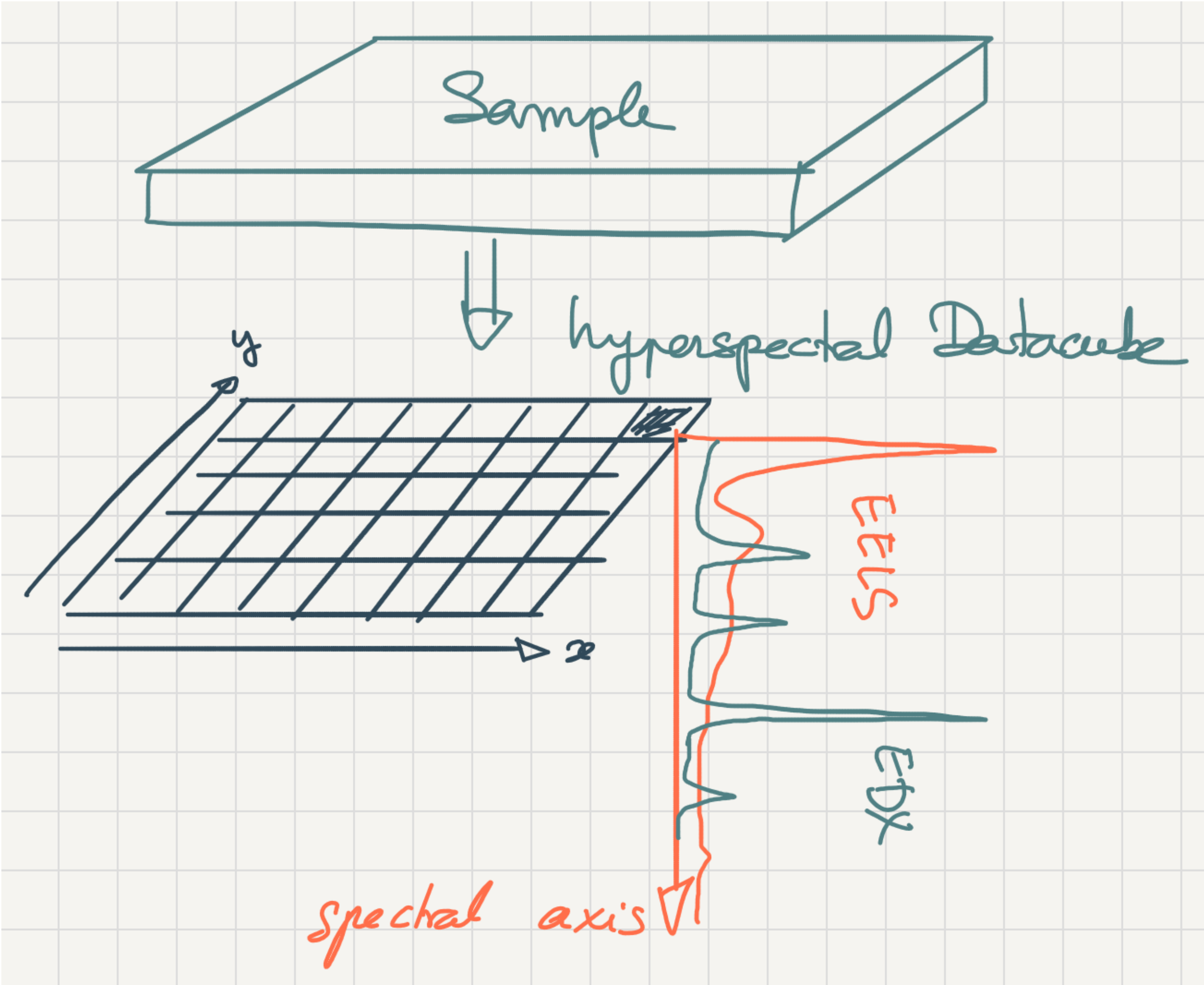
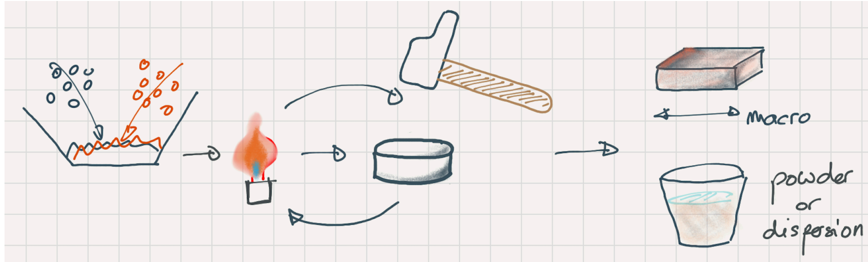
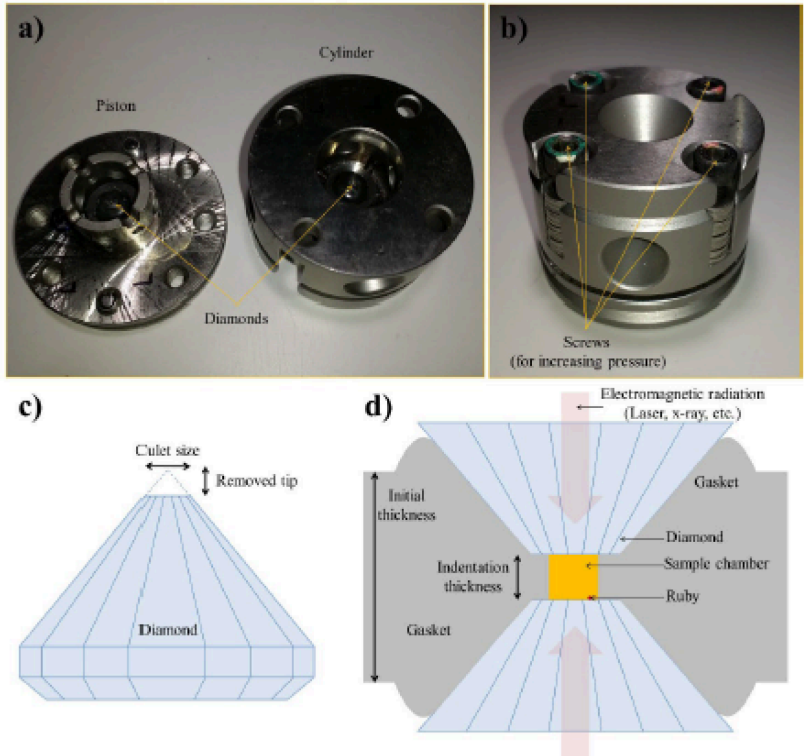
Analytical STEM: EELS & EDX



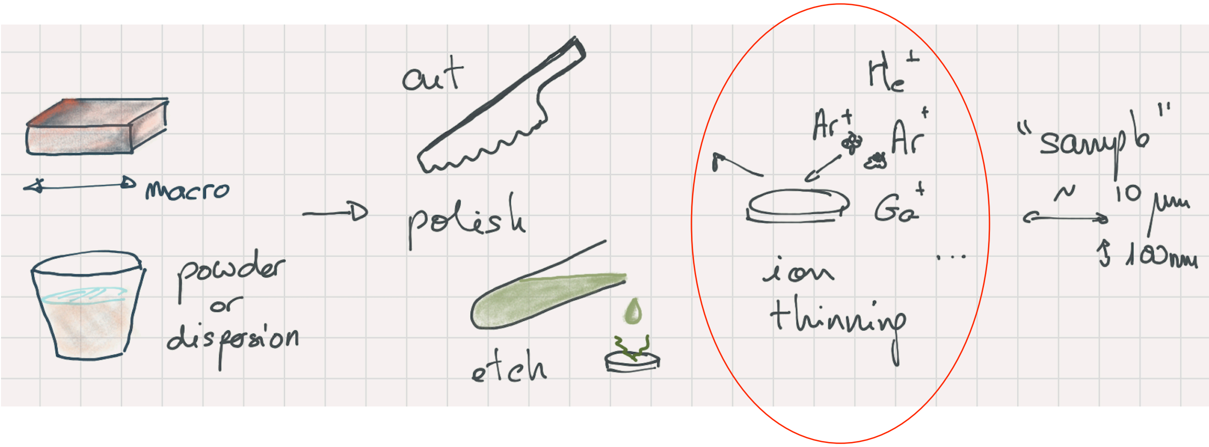
Sample used as an example:

- Starting material: a synthetic pyrolite glass doped with Nd, Sm, Hf, Lu, and U (0.3 wt.% for each).
- Samples were first compressed in symmetrical diamond anvil cells (DAC) at 46 Gpa and melted by double-sided laser heating, and then slowly cooled down below the solidus temperature before quenching.

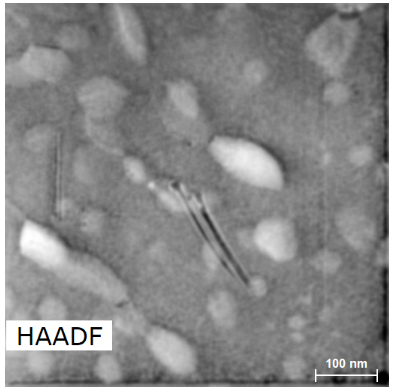


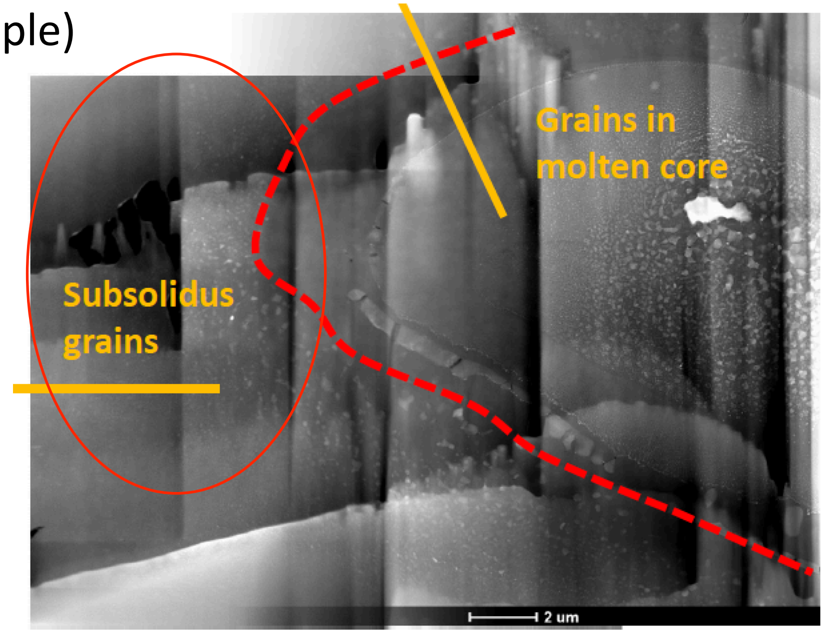


From materials to TEM sample

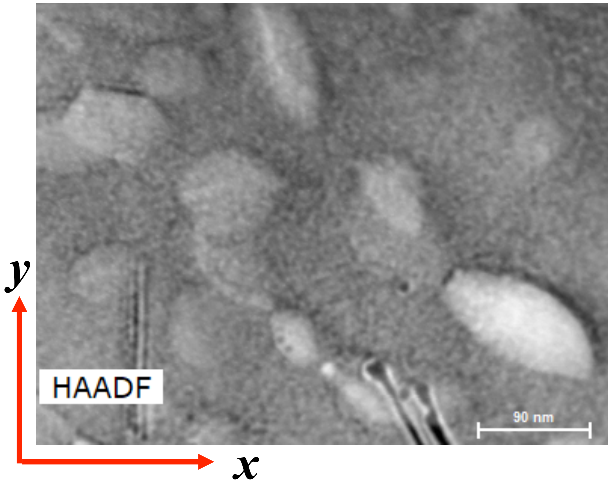


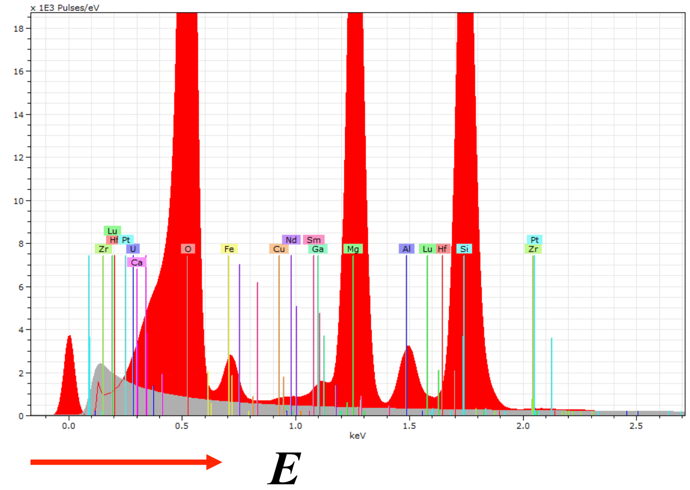
FIB sample preparation (1 DAC exp = 1 TEM sample)





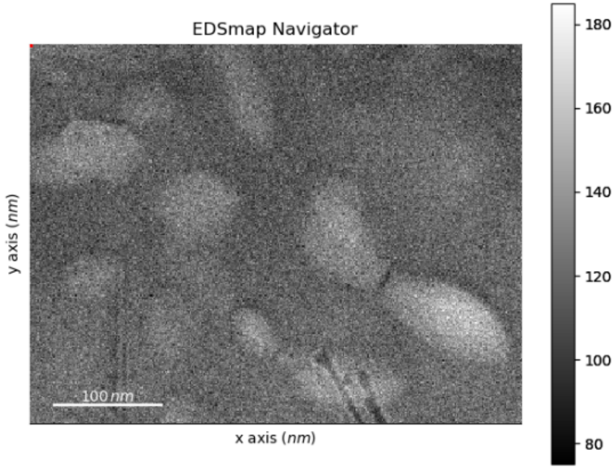
- **Theintegrated EDS spectrum**

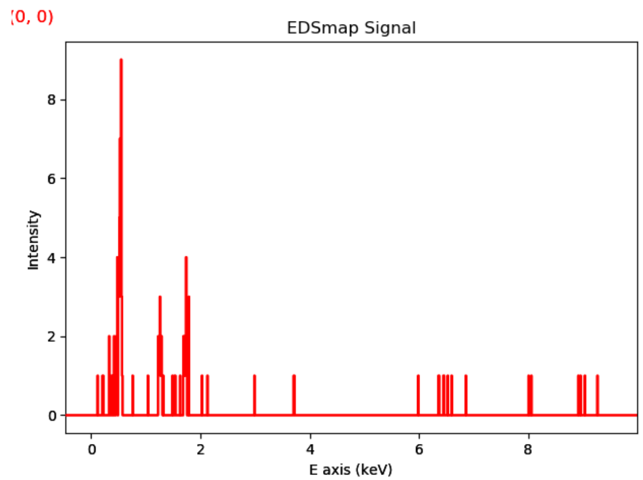




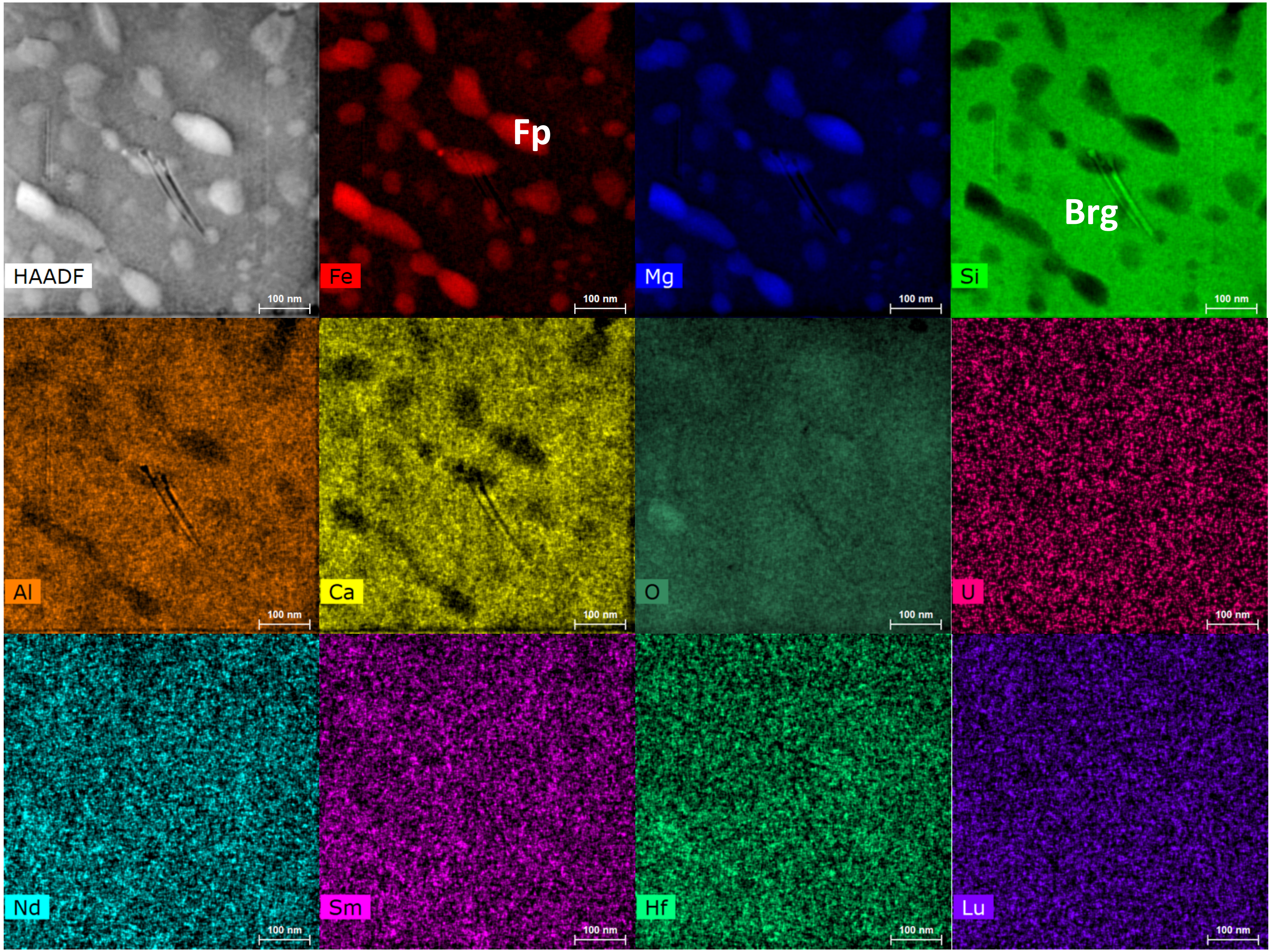
Spectrum = **background signal** + **sets of Gaussian peaks** (each specific element in the sample yields a unique set of Gaussian peaks, like a signature in ***E*** axis) + noise

- **A representative single pixel EDS spectrum**





EDS maps of the subsolidus region from Esprit software



First use of PCA in analytical TEM

EELS elemental mapping with unconventional methods I. Theoretical basis: image analysis with multivariate statistics and entropy concepts

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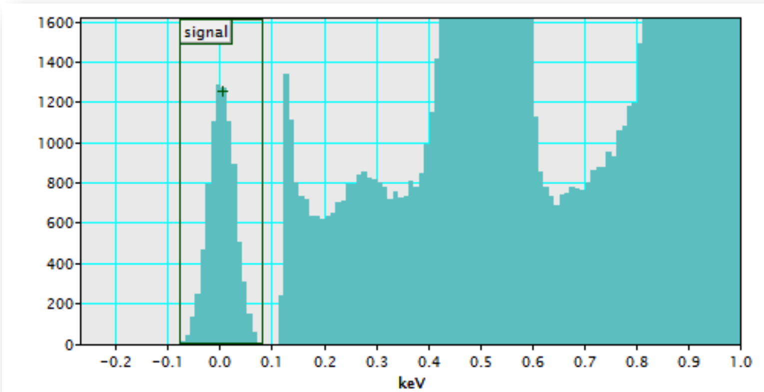
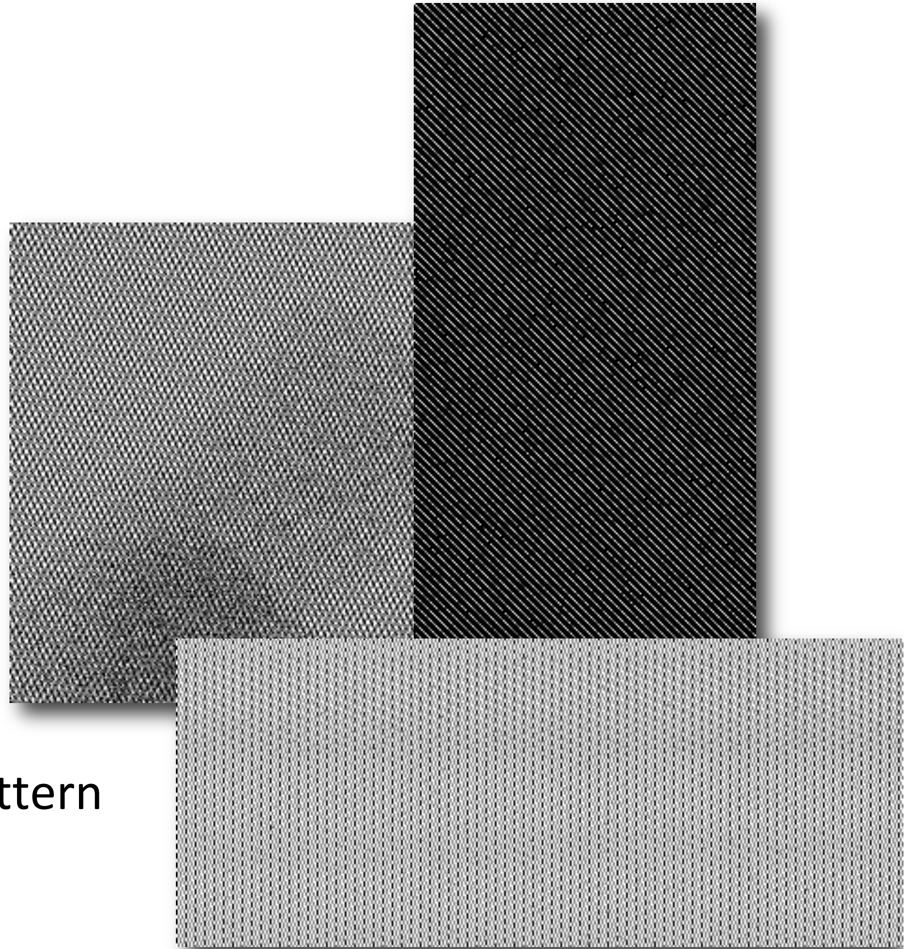
HyperMap data processing: the "0 eV peak"

- With raw data cube PCA gives component(s) with regular patterns of intensity
- This is the 0 eV peak, whose integrated signal shows regular fringes
- Must be removed for PCA to work





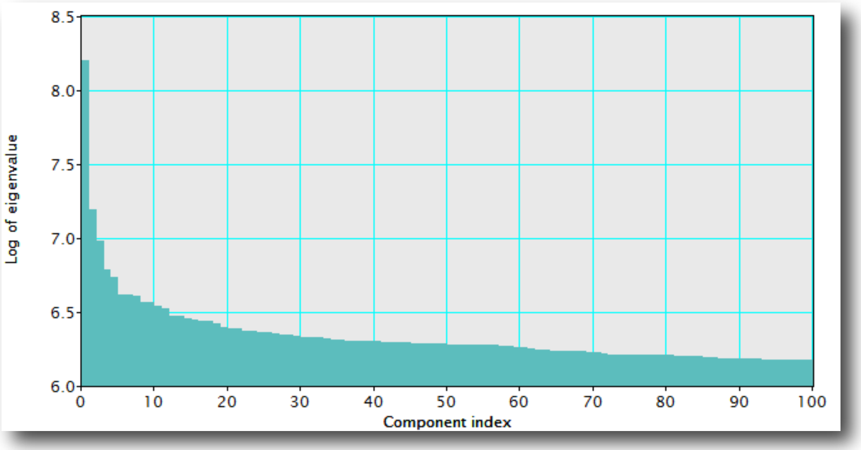
- Every data cube has its own 0 eV peak pattern
  What is it?

Solution: cut of “zero eV peak” and move on

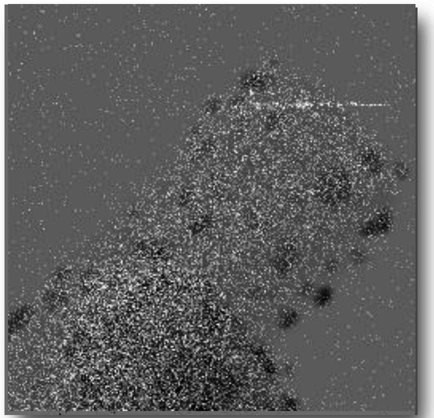
Guillaume Lucas and Duncan Alexander, LSME&CIME

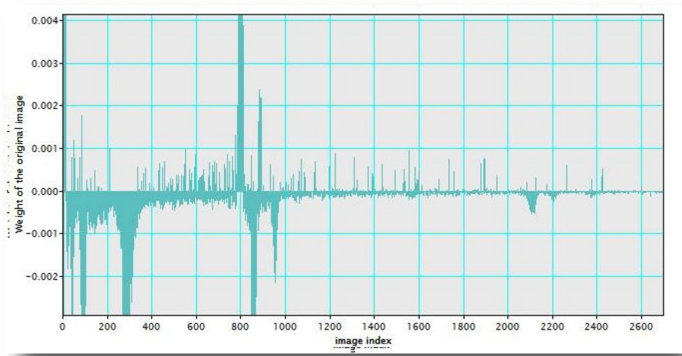
EDX HyperMap data processing: non-random noise components in low signal regions

- PCA decomposition gives scree plot which does not taper off rapidly
- “Physical” components appear mixed up with noise components
- Noise components seem to show strong, non-random speckle
- => Non-random (e.g. non-Poissonian) noise - readout noise?



PCA score





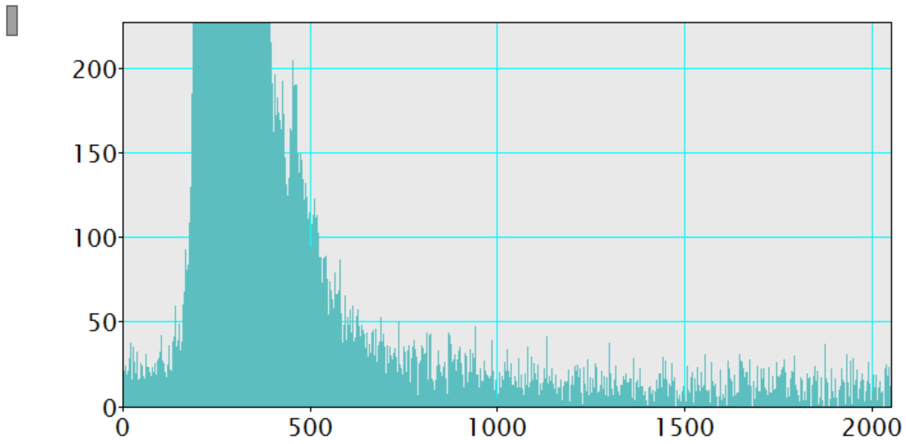
PCA loading

Avoid holes and low intensity EDX region (or mask them out for treatment), and move on

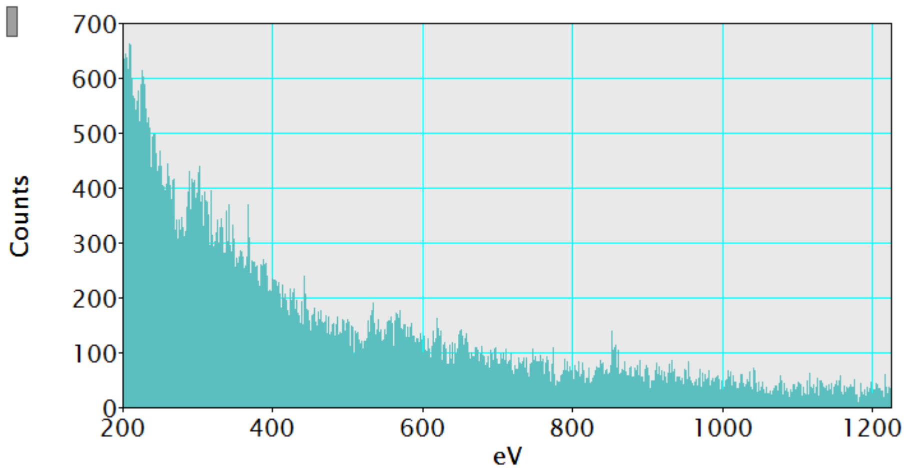
EELS is not spared…

- “Raw” spectrum image data: example single px spectra:

Low-loss:



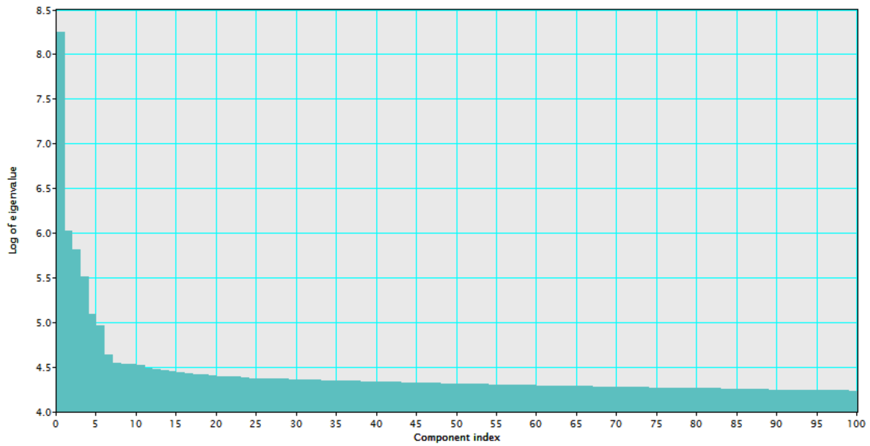
High-loss:

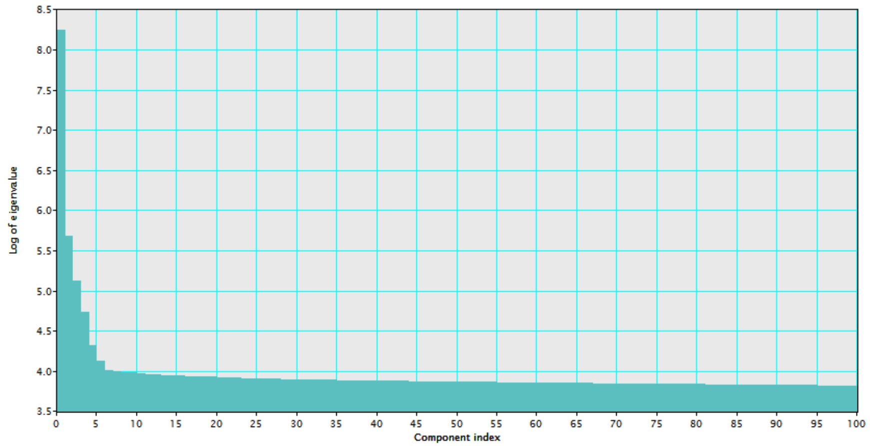


- Spectra look correct.

Dual EELS: camera response

- Perform MSA; scree plots look correct
- However many components show distinct difference between the two detector quadrants across which the spectrum has been recorded, both for low-loss and core-loss

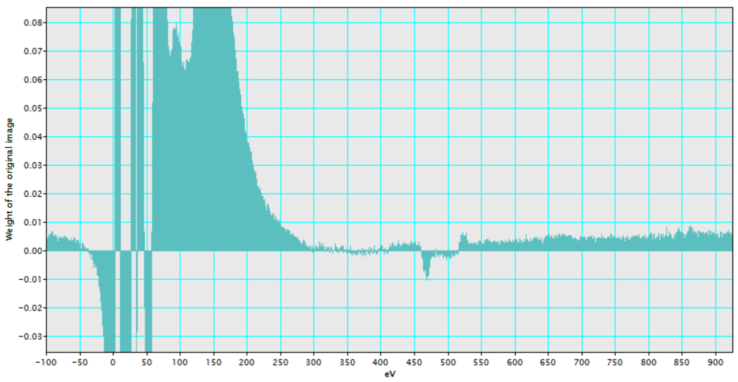




Dual EELS: camera response

Low-loss loadings:

1



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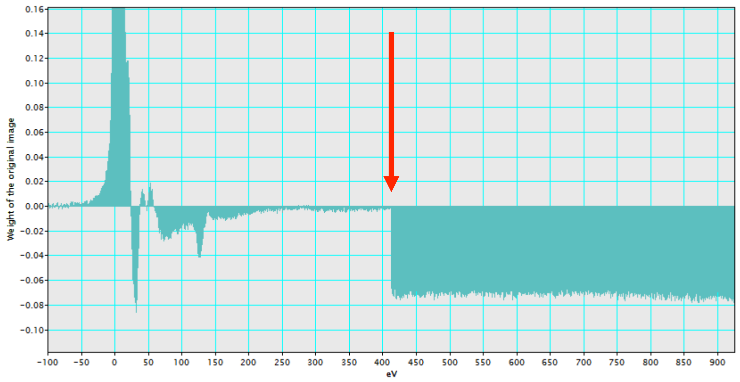

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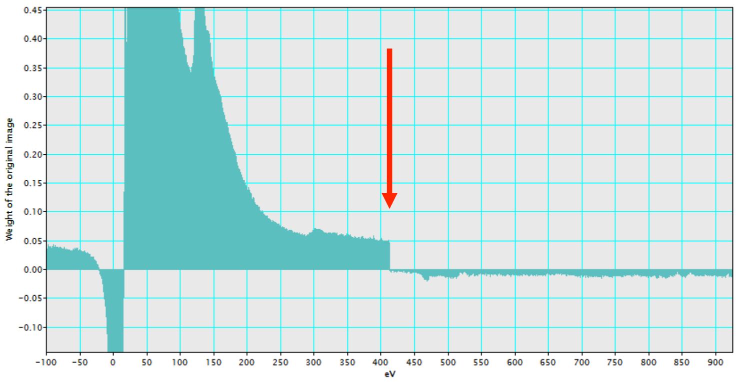

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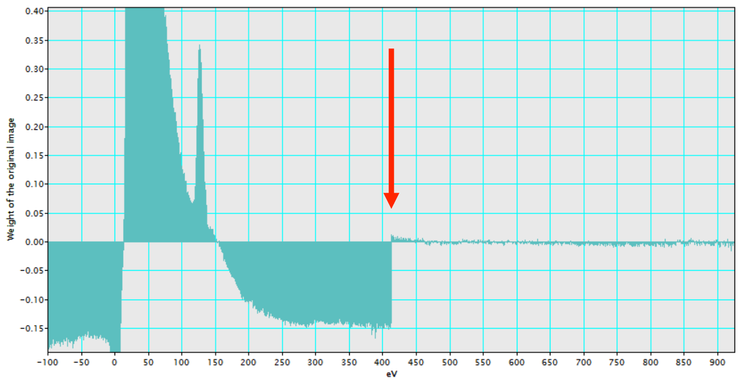
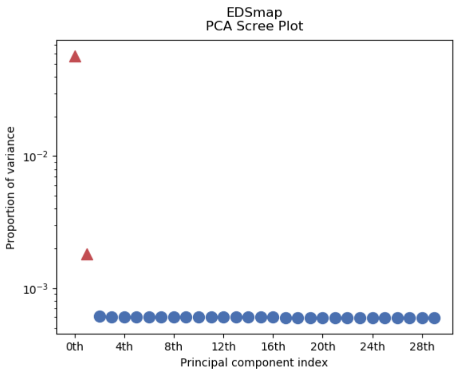
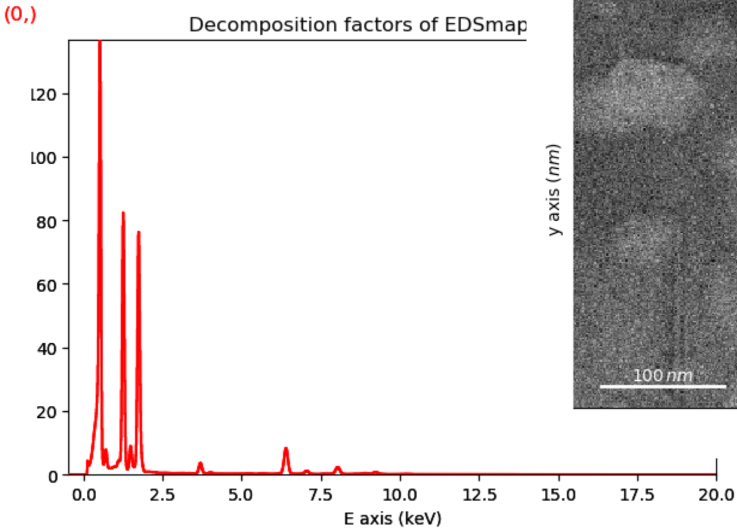


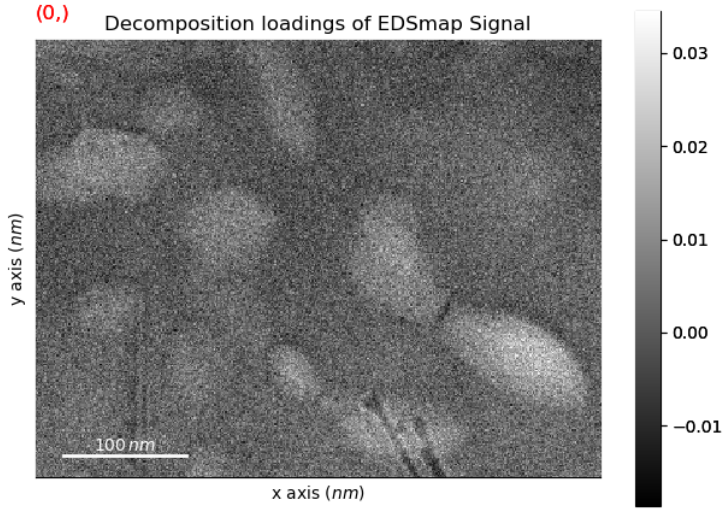
MSA facts

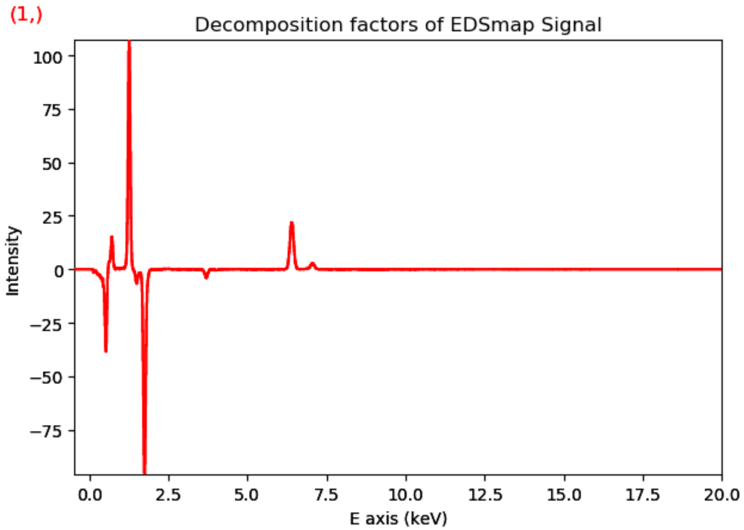
- PCA is damn good at spotting detectors artifacts…

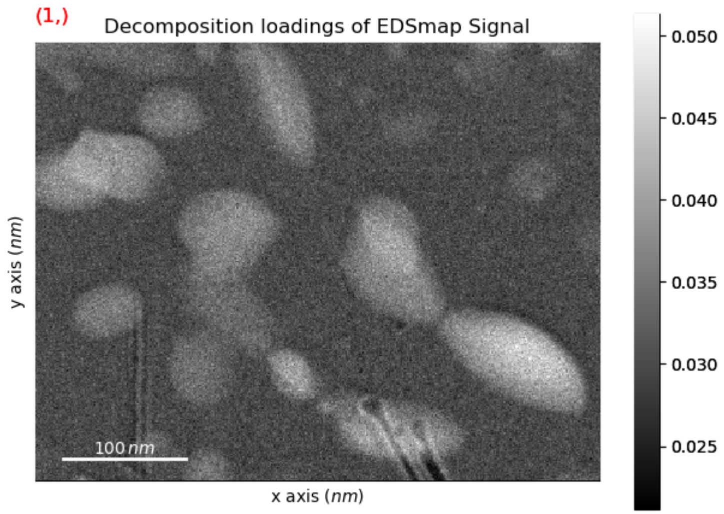
Data unmixing: PCA analysis











MSA facts

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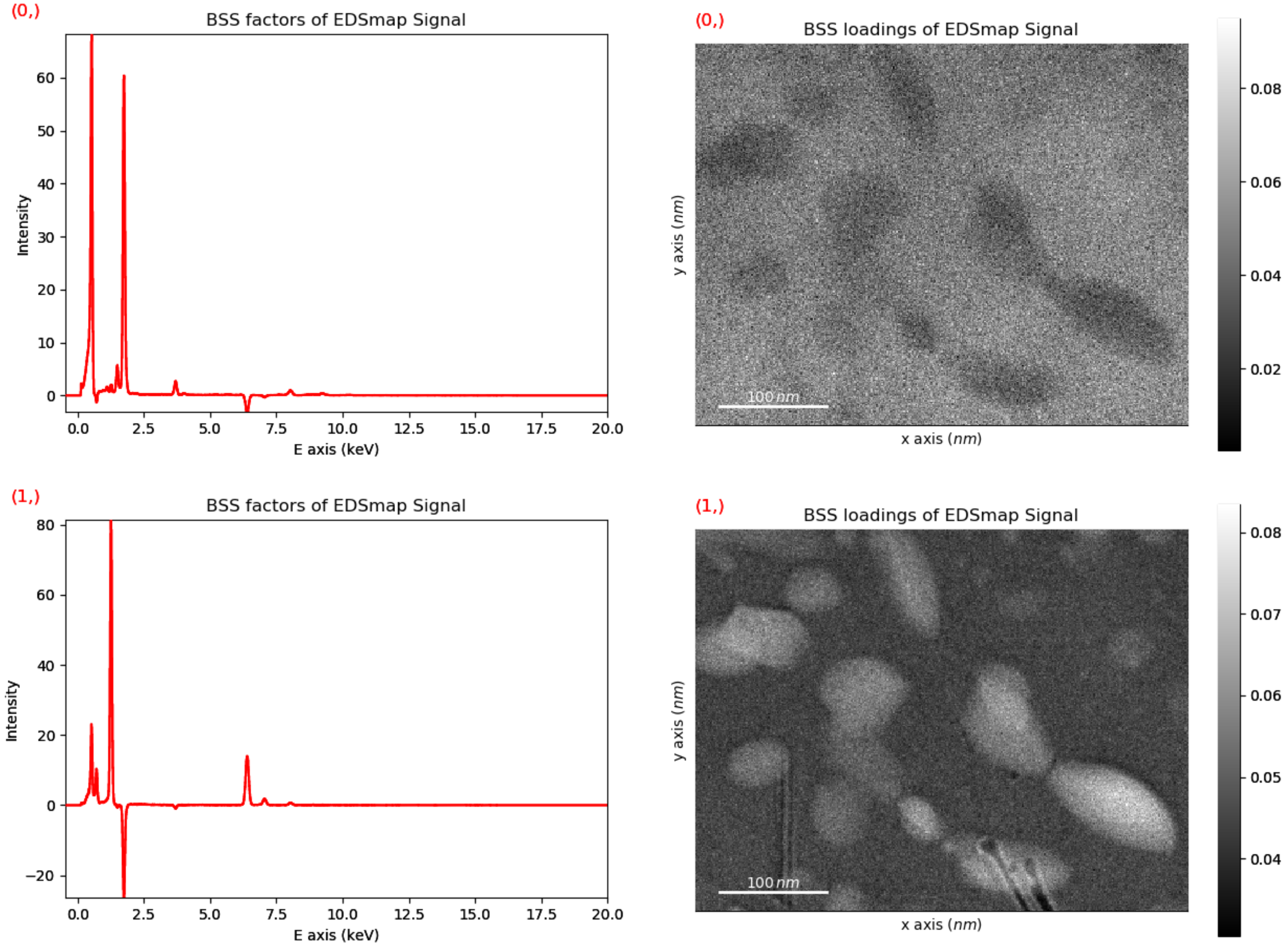
- PCA do *not* give components with physical meaning

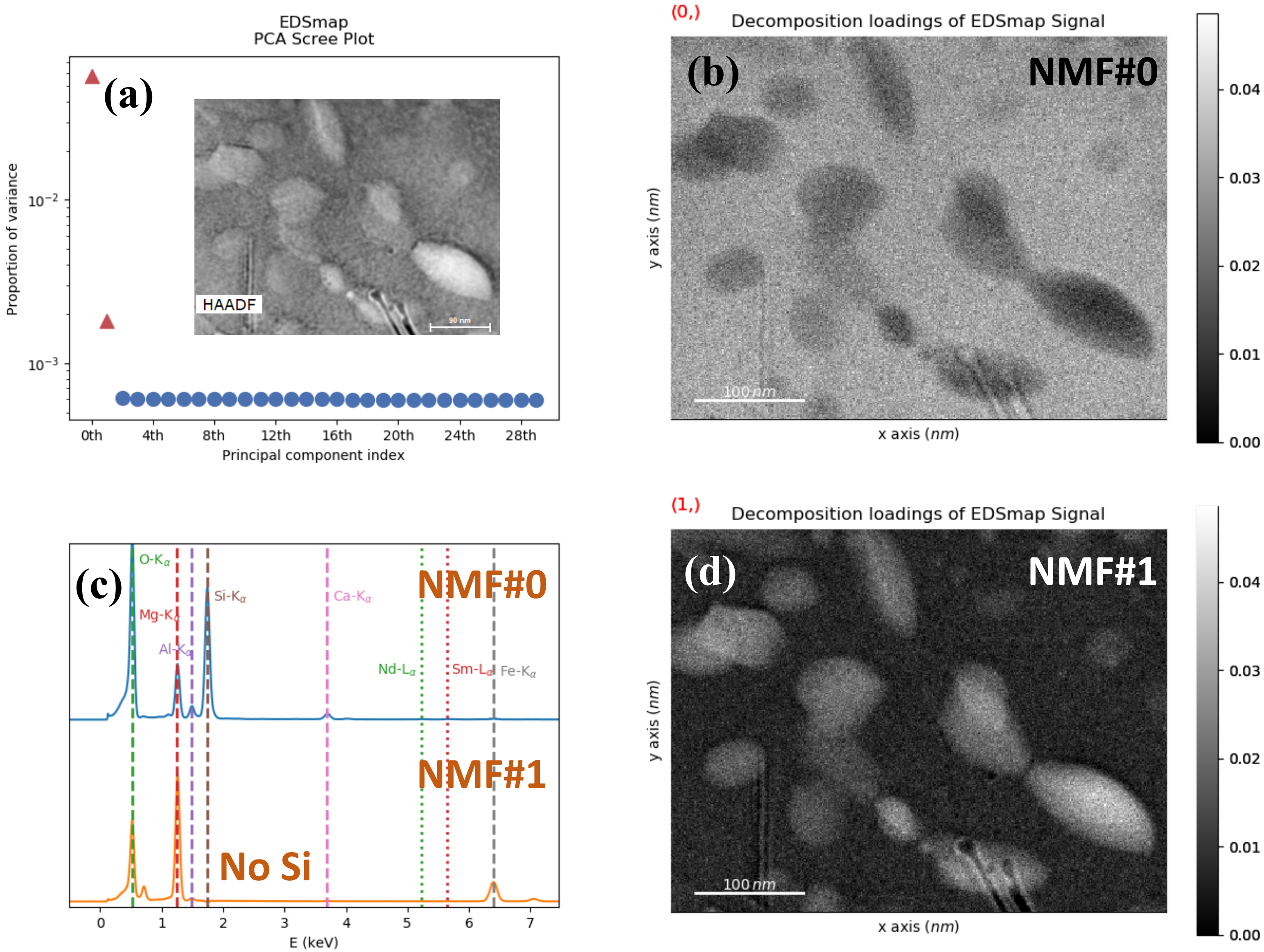
Independent Component Analysis



MSA facts

- PCA is damn good at spotting detectors artifacts…
- PCA do *not* give components with physical meaning
- ICA might give component with physical meaning

❑ Non Negative Matrix Factorization



MSA facts

- PCA is damn good at spotting detectors artifacts…
- PCA do *not* give components with physical meaning
- ICA might give component with physical meaning
- NMF give EDX components that looks like they have a physical meaning

❑ Brg phase segmentation via NMF mask

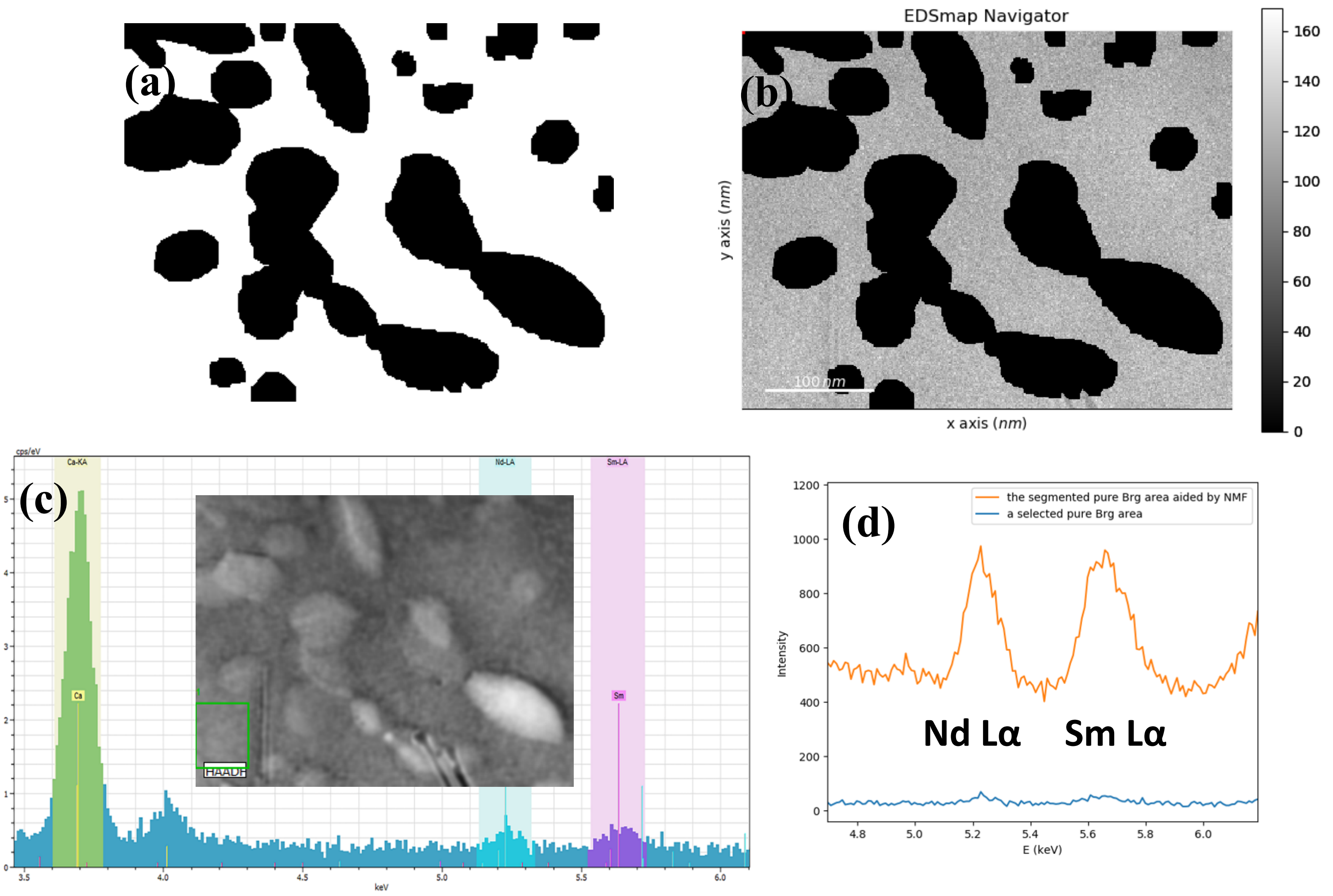


Fig. (a) mask of the pure Brg area generated via NMF#1 thresholding; (b) segmented pure Brg phase; (c) EDS spectrum of a selected Brg area; (d) EDS spectral comparison of a selected Brg area and the masked Brg area.

Quantification of the spectra

	Fe	Mg	Si	Al	Ca	Nd	Sm	O
NMF#0	0.17	9.3	24	2.2	1.1	0.066	0.071	63
NMF#1	8.2	40.5	0.0	0.87	0.076	0.0	0.0	50
Brg mask	1.7	15	19	2.0	0.93	0.048	0.070	60

Conclusion

- NMF gives components which are close to be physical, but are not. This is even worse (and more dangerous)
- Why is this method called “machine learning” if we cannot teach the machine ?
- Need to move to supervised learning. The microscopists has a lot of “knowledge” that can be transferred to the machine
- We can easily detect < 0.1% dopants
- Challenge : if there is a small particle in one corner of the sample