Atomic Scale Elemental Analysis with Spherical Aberration Corrected STEM

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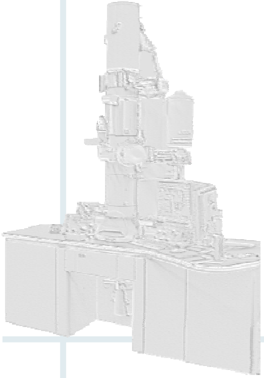
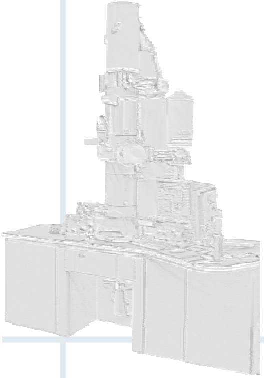


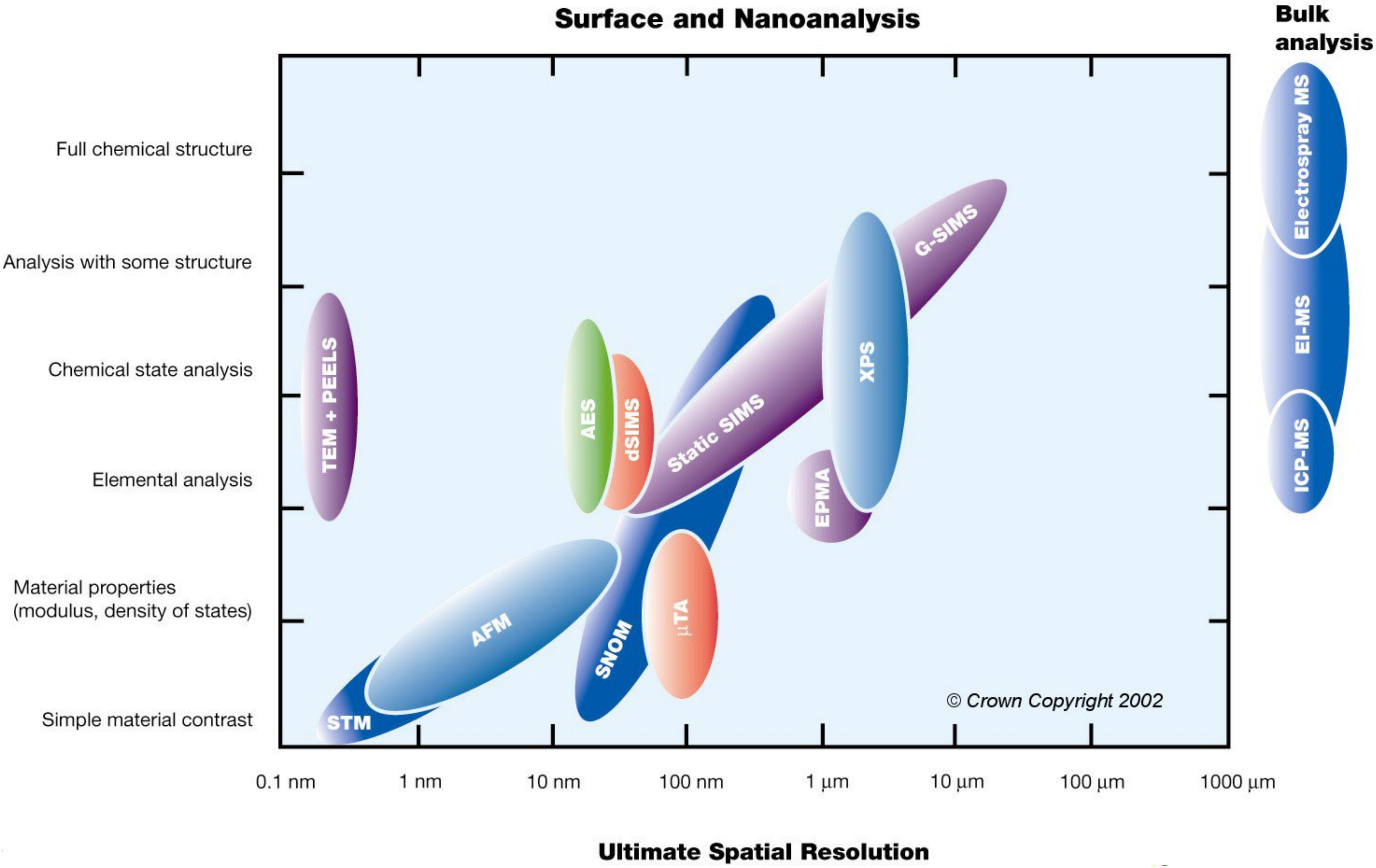
JEM-2100F with CESCOR

Spherical Aberration corrector for Probe forming Lens

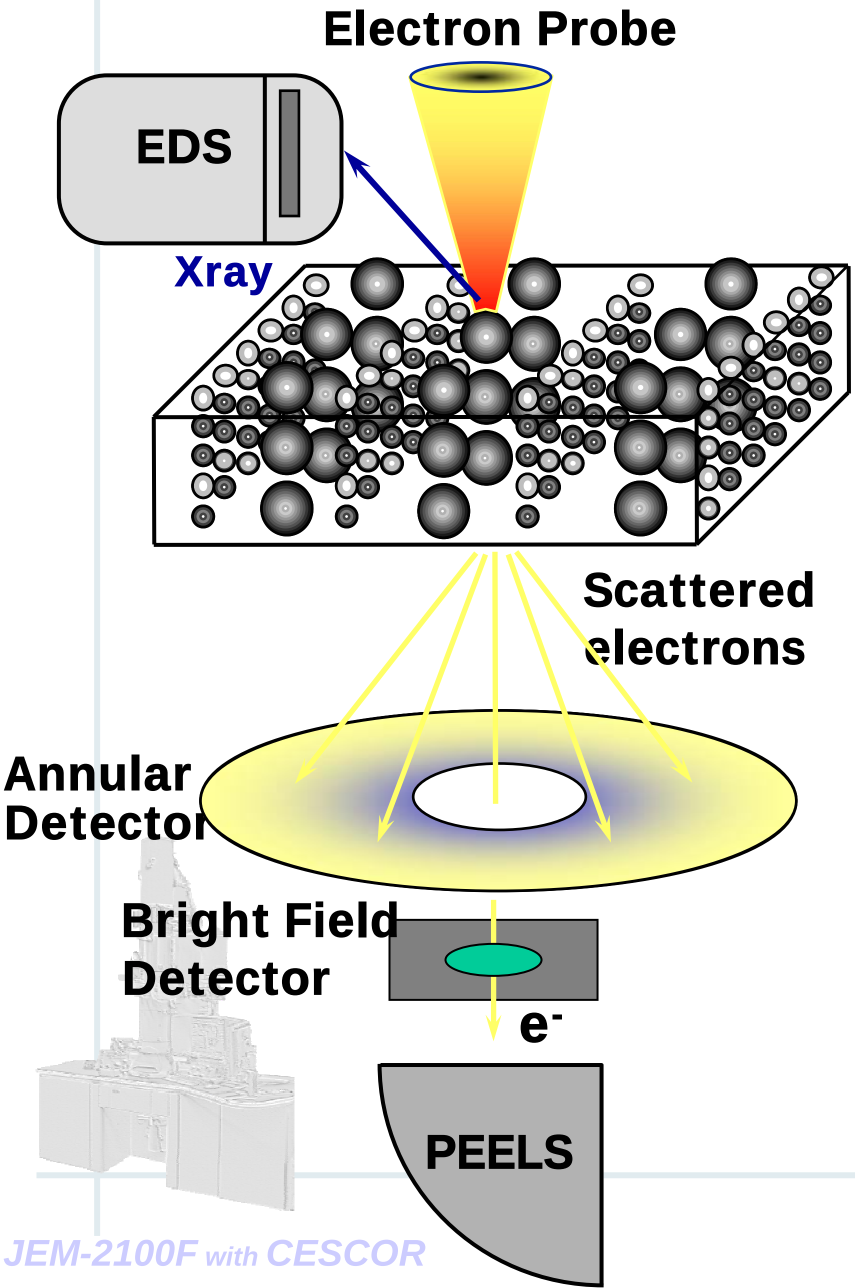
Why Scanning Transmission Electron Microscopy?



Techniques for Surface and Nanoanalysis

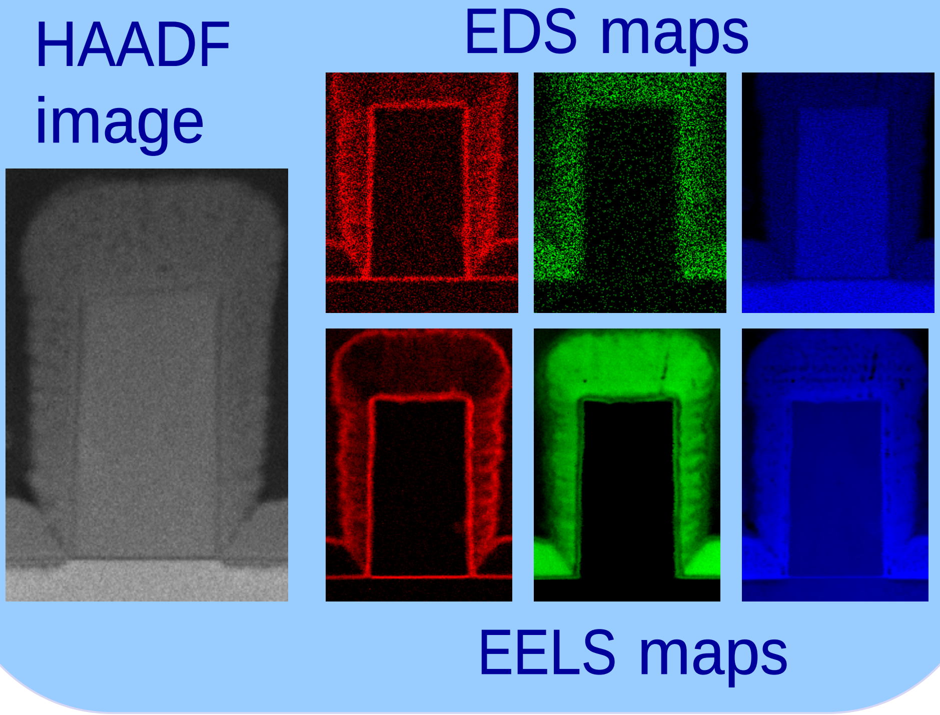


STEM is a very powerful analytical tool

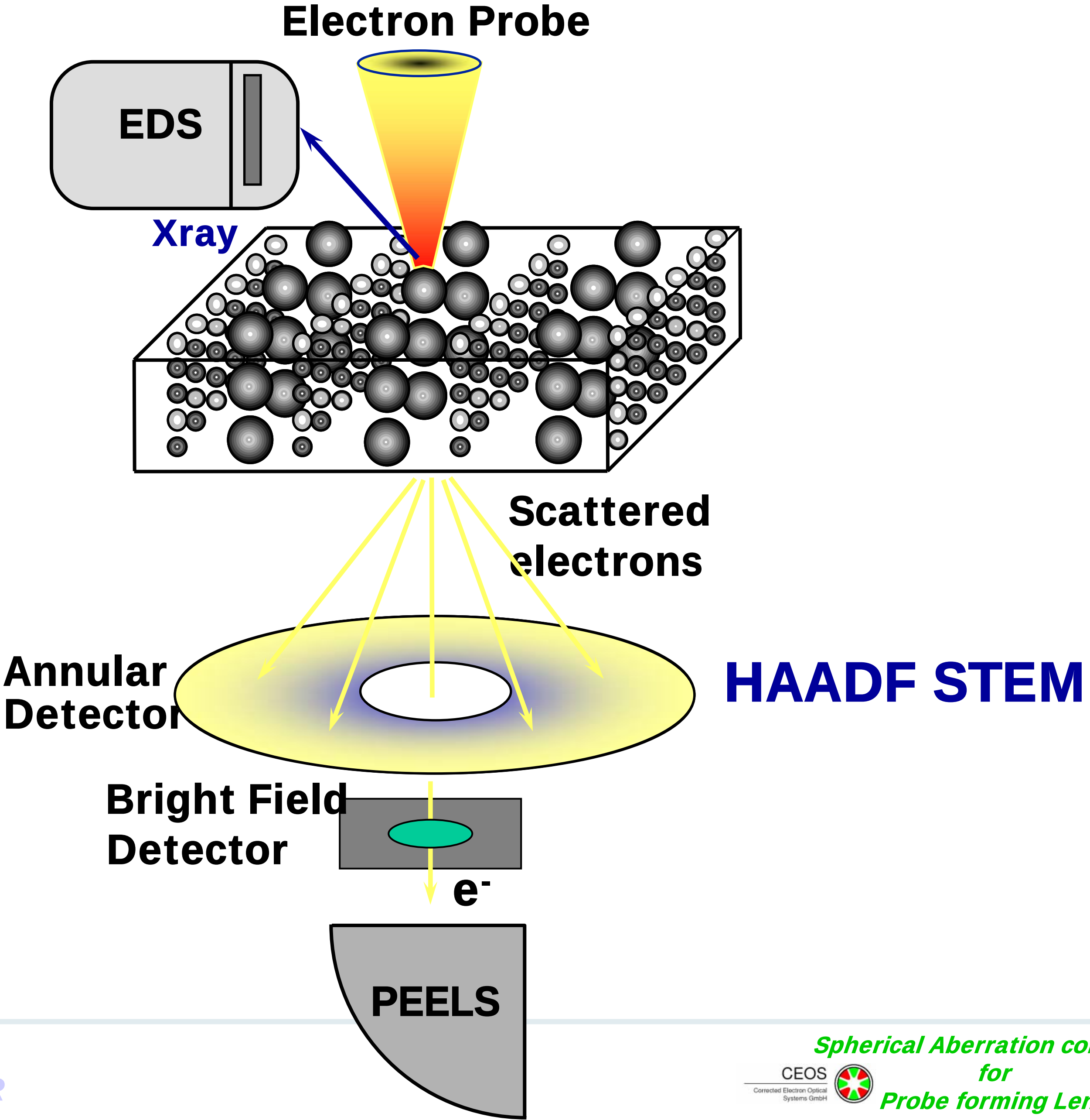


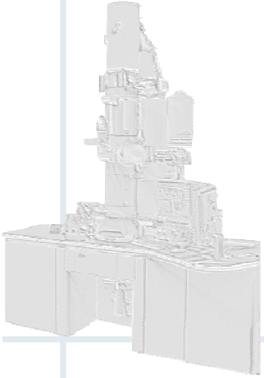
STEM uses a scanning electron probe for combined imaging and analysis

STEM image, EDS and EELS data are corrected simultaneously

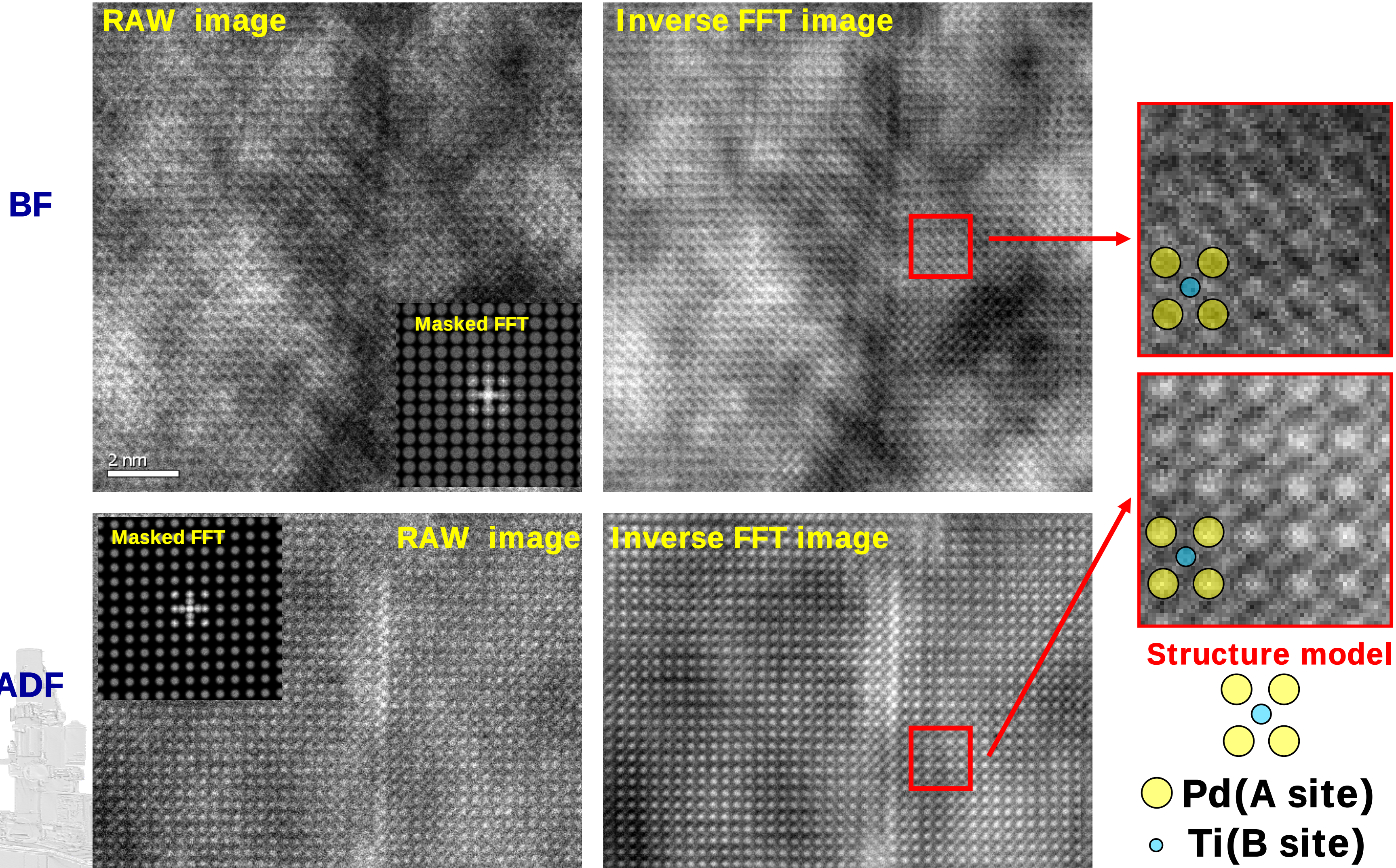


HAADF STEM


Electron Probe
EDS
Xray
Scattered electrons
Annular Detector
HAADF STEM
Bright Field Detector
e-
PEELS



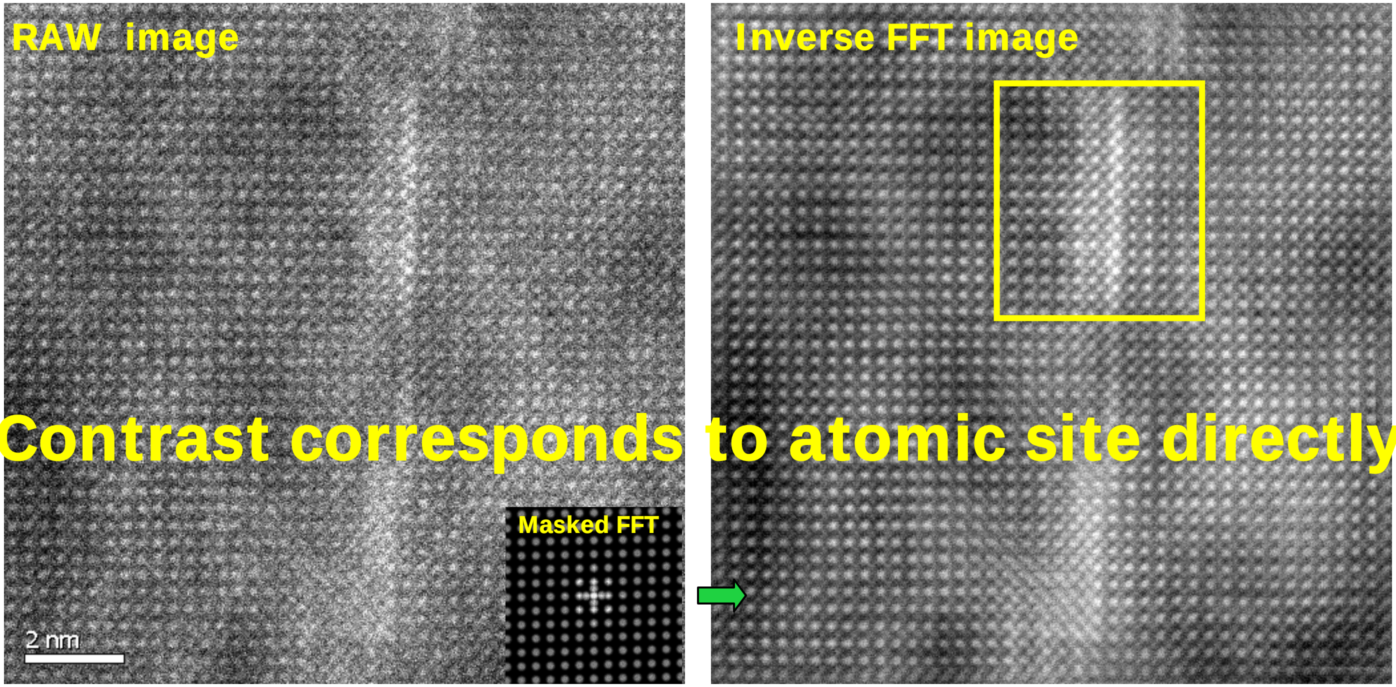
HR-HAADF and BF Simultaneous Image Acquisition of TiPd shape memory alloy



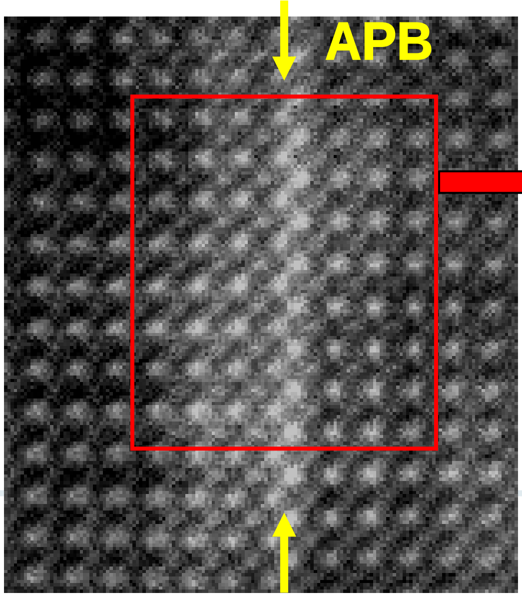
HR BF image : dark contrast corresponds to atom sites

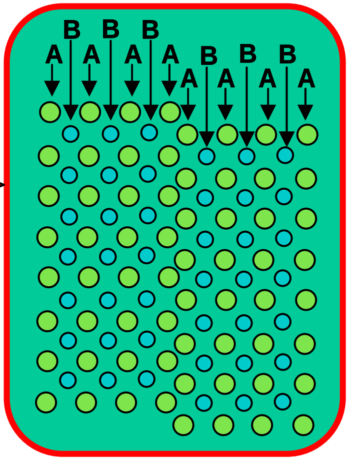
HR HAADF image : bright contrast corresponds to atom sites

HAADF image of APB(Anti Phase Boundary) in TiPd shape memory alloy



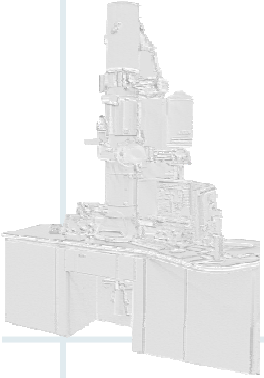
Contrast corresponds to atomic site directly





- ○ **Pd(A site)**
- ○ **Ti(B site)**

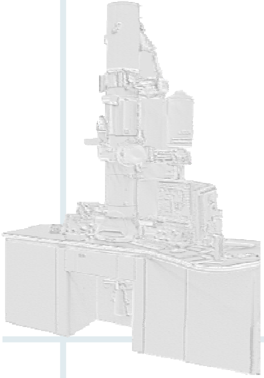
Data courtesy of prof . M.Nishida (Kumamoto univ)



JEM-2100F with CESCOR

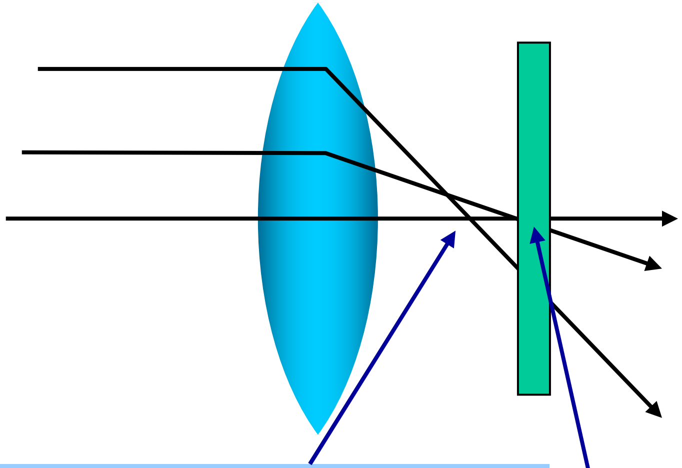
Spherical Aberration corrector for Probe forming Lens

Why correct Spherical Aberration Cs?



Spherical Aberration Cs

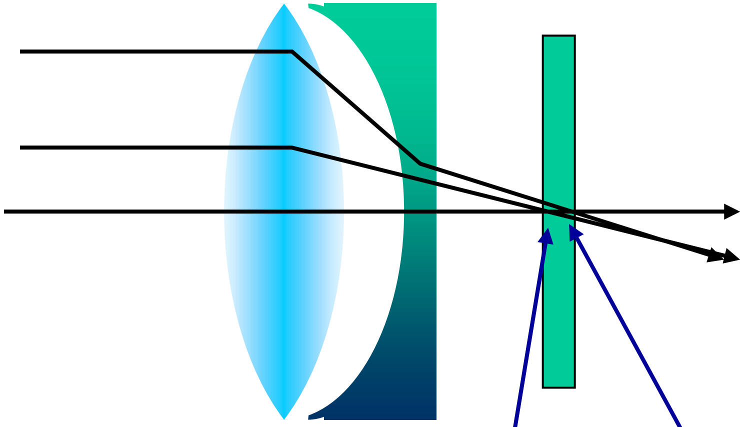
Convex lens



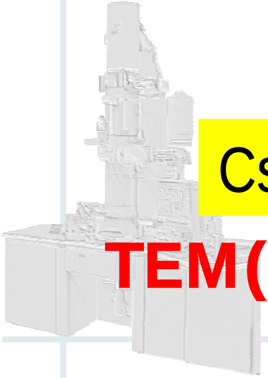
beams through the outside focus on a point outside the specimen

beams through the inside focus on a point on the specimen

Convex lens + Concave lens



beams through the inside and outside focus on the specimen



Cs is not corrected

Cs is corrected

TEM(electromagnetic lens)

Spherical aberration Cs

In STEM, the beam diameter on the sample is influenced by Cs.

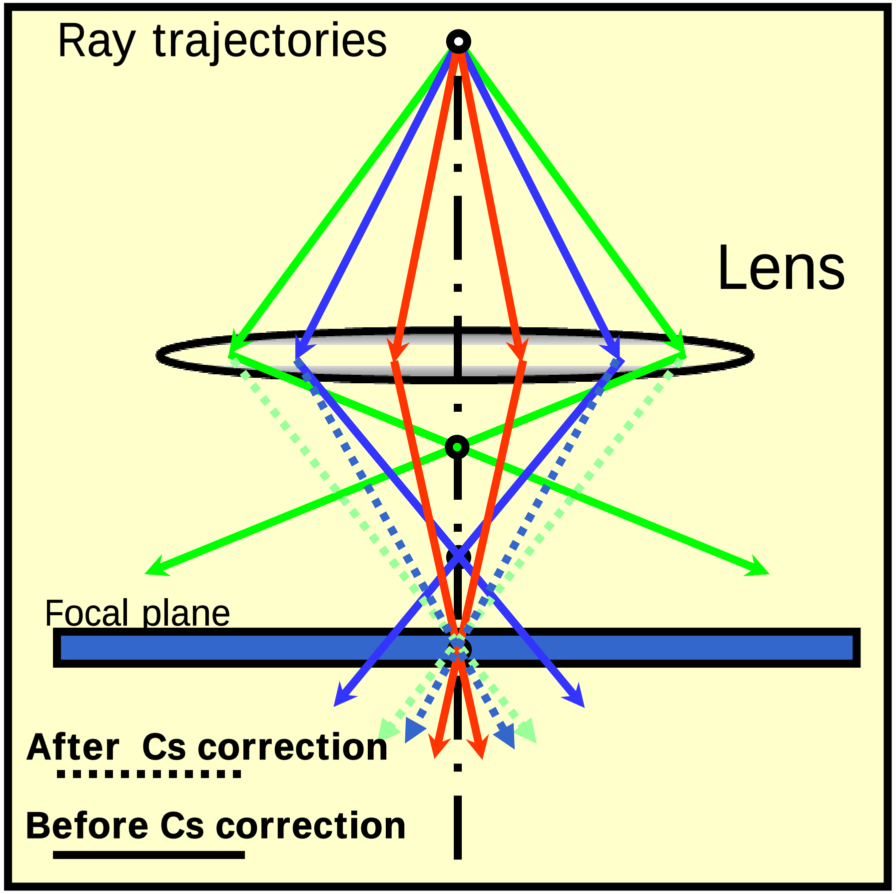
The electron beam that passed several routes is not able to focus at same point

In case of OM, optical lens:
Cs is corrected by combination of convex lens and concave lens

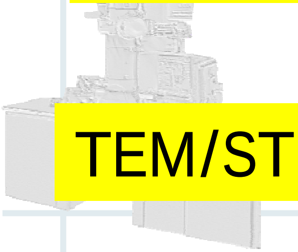
In case of TEM / STEM: electromagnetic lens, only convex lens.

As a consequence: Cs !

Recently, Cs-correctors were developed. Cs-correctors are constructed by combination of multiple-pole lenses.

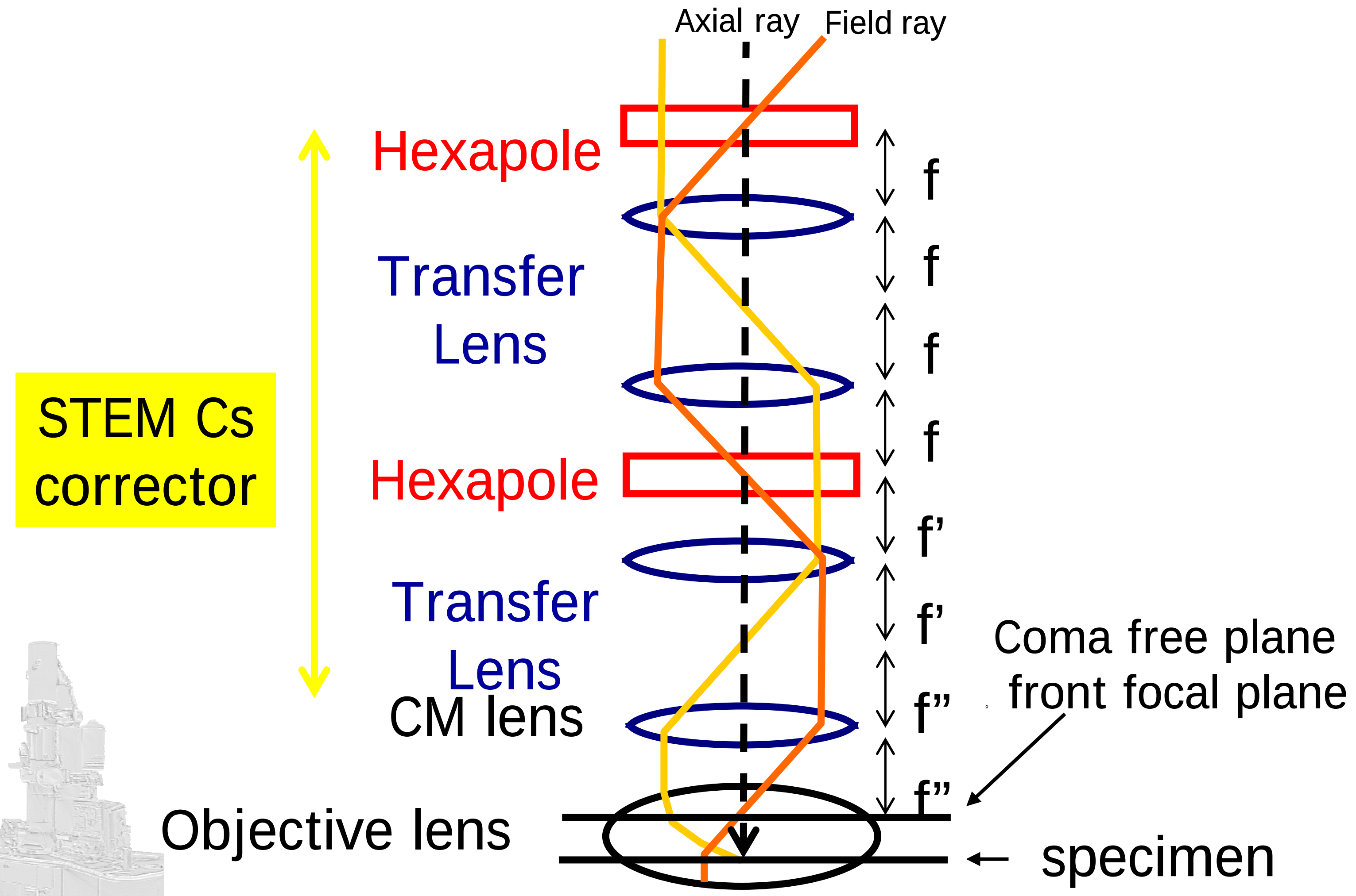


TEM/STEM performance is improved after correction of Cs



Hexapole type Cs Corrector

M.Haider, H.Rose (CEOS GmbH)



Axial ray

Field ray

STEM Cs corrector

Hexapole

Transfer Lens

Hexapole

Transfer Lens

CM lens

Objective lens

f

f

f

f

f'

f'

f"

f"

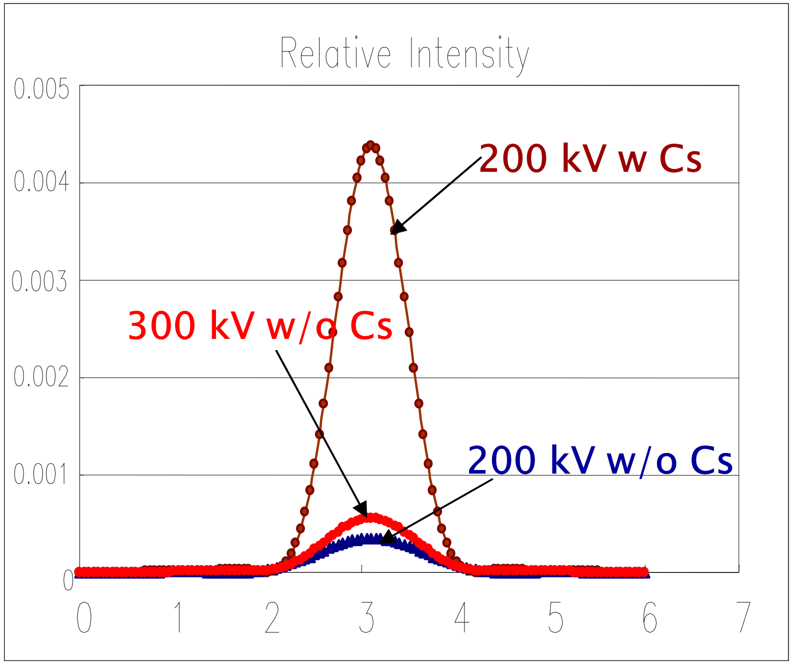
Coma free plane

front focal plane

specimen

Effects of Cs correction for STEM

Probe calculation



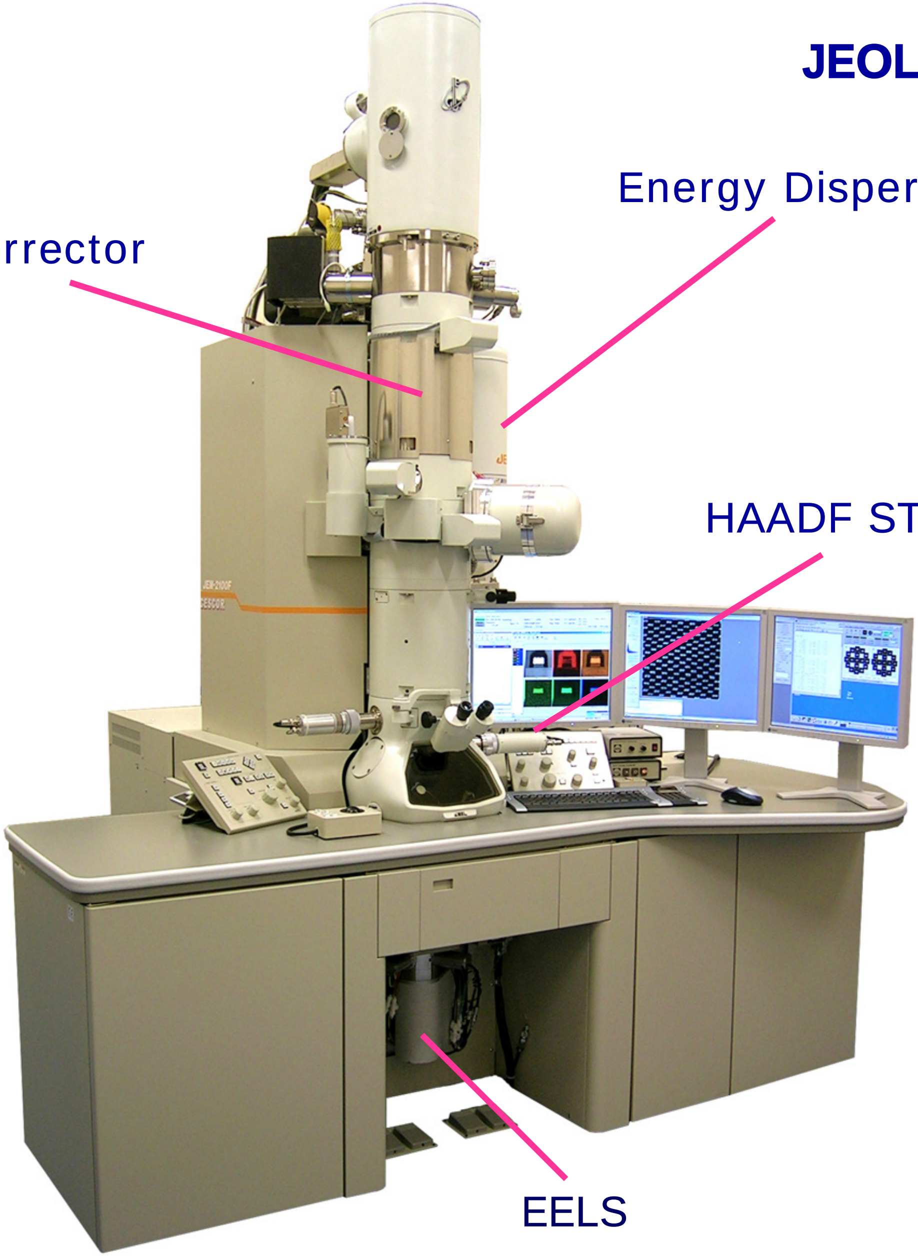
1 Finer probe is made: from 0.136nm to less than 0.1nm probe (FWHM)

2 Finer probe has higher current: from 500pA 1nm to 500pA 0.2nm probe

Cs corrector allows sub Å imaging and elemental analysis?!

200 kV FEG-TEM with STEM Cs corrector

JEOL JEM-2100F Cescor

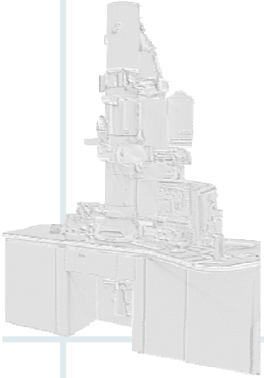


Spherical Aberration corrector for Probe forming Lens

CEOS Corrected Electron Optical Systems GmbH

JEOL Serving Advanced Technology

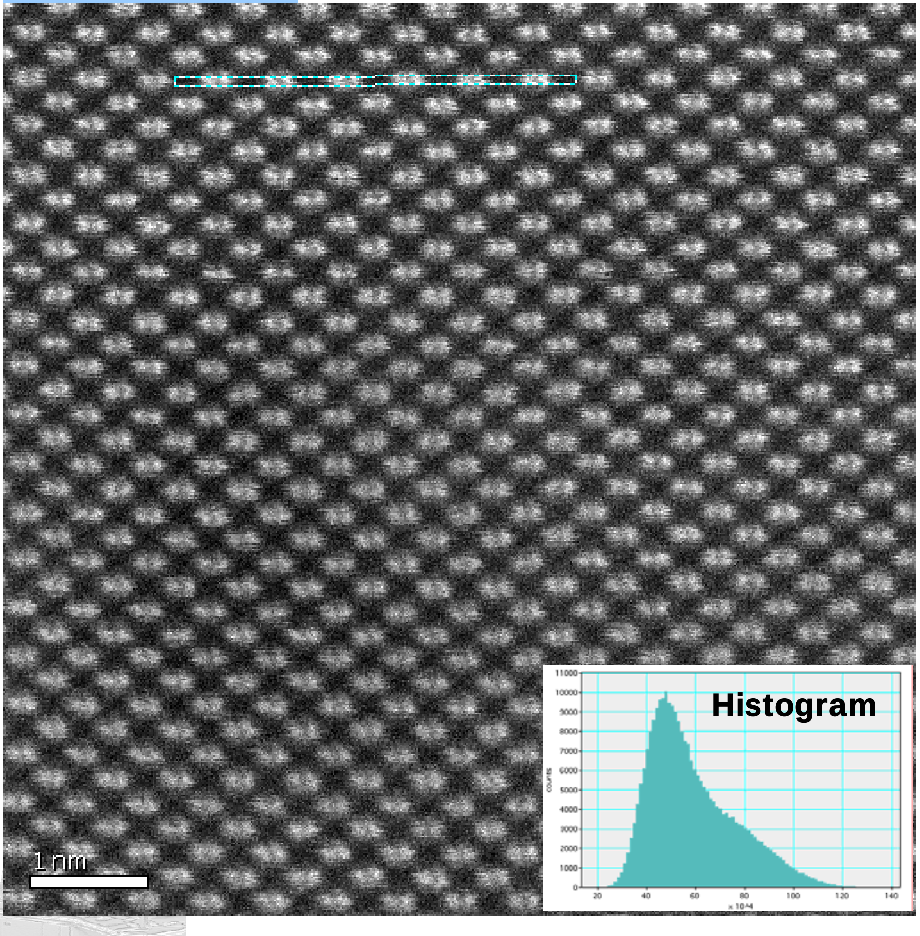
Imaging capability of Cs corrected STEM



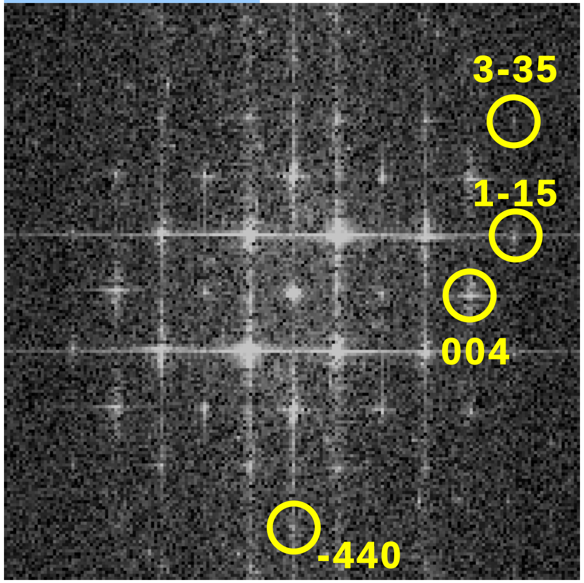
EM Art
EM Application & Research Team

HAADF STEM resolution)
Si[110] dumbbell image

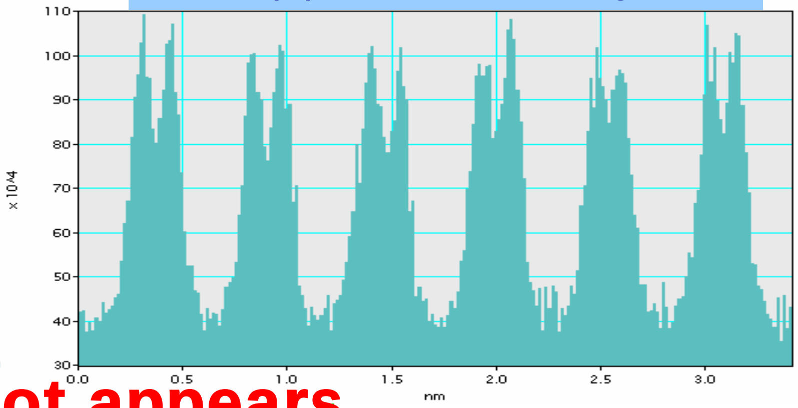
HAADF image



FFT pattern



Intensity profile from rectangle area



Resolution better than 0.09nm

0.083nm(335) spot appears

JEM-2100F with CESCOR

CEOS
Corrected Electron Optical Systems GmbH

for
Probe forming Lens

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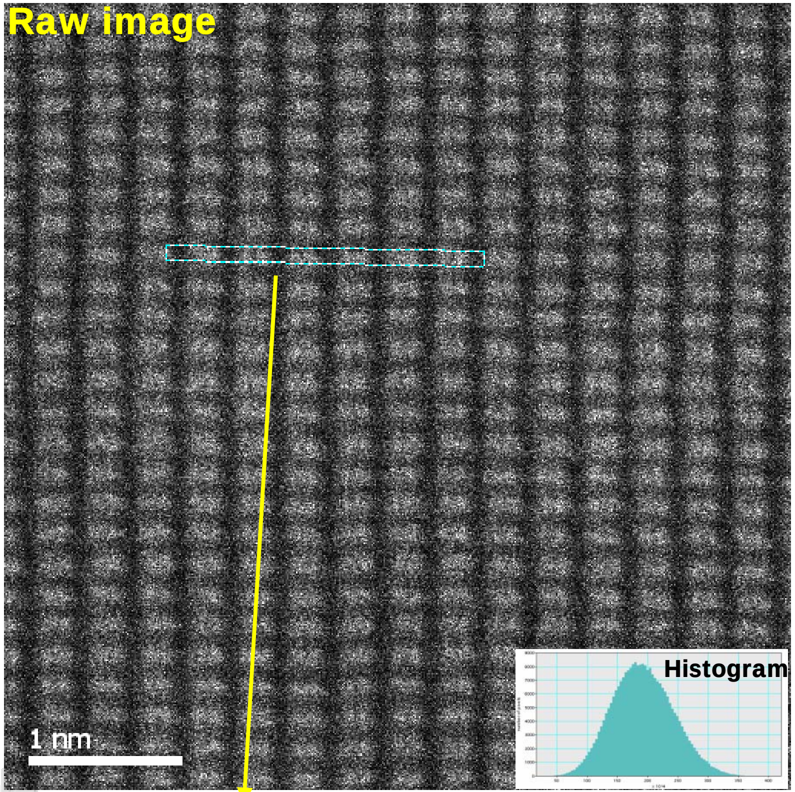
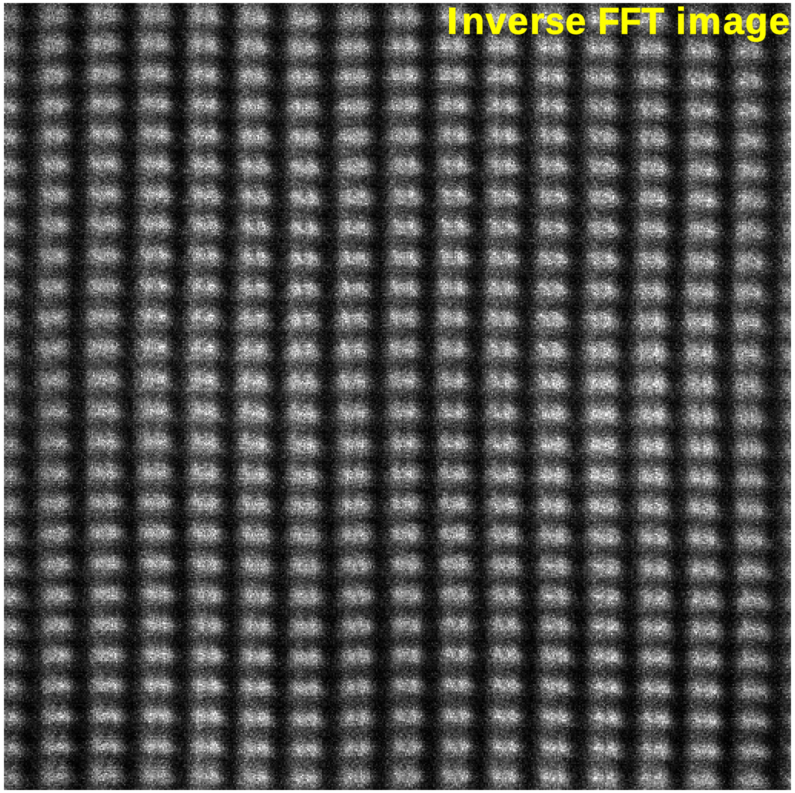
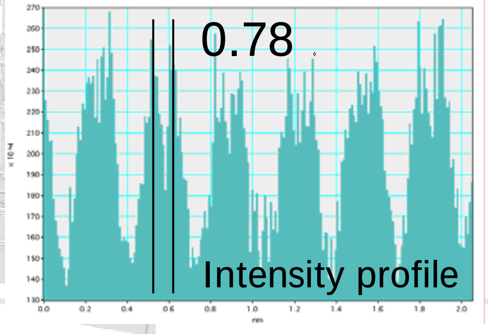
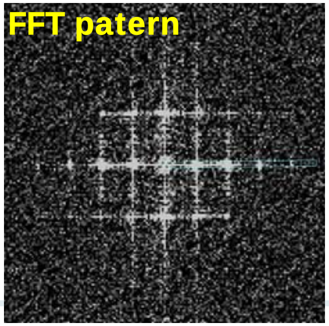
Image Resolution by Cs-corrected HAADF imaging

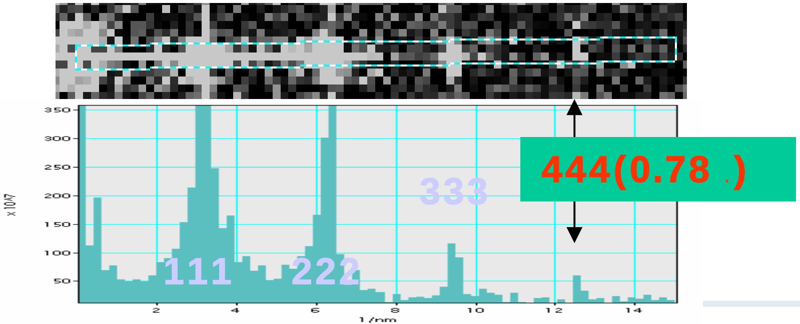
0.78 resolution by Si[112] direct observation and FFT analysis











0.078 nm from Si[112] dumbbell structure

JEM-2100F with CESCOR

Spherical Aberration corrector
for
Probe forming Lens

CEOS
Corrected Electron Optical Systems GmbH

JEOL
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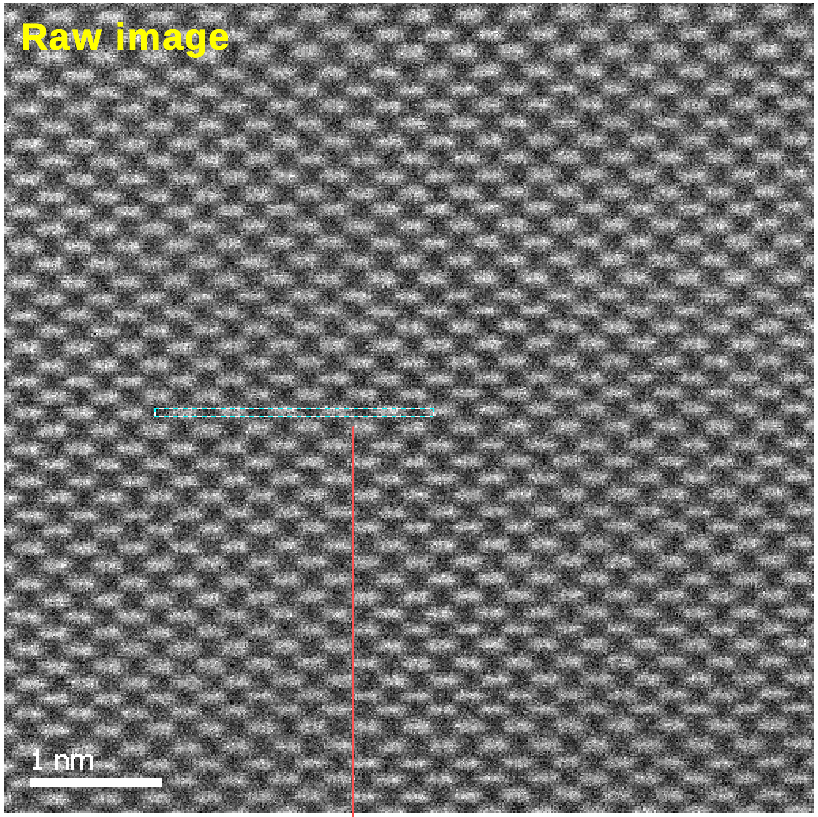
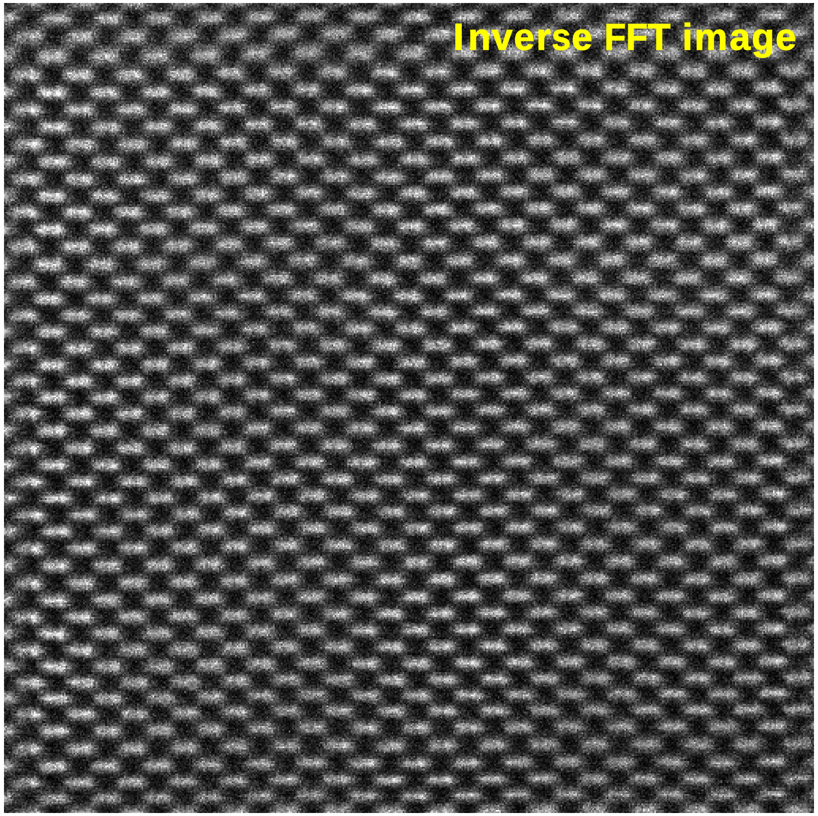
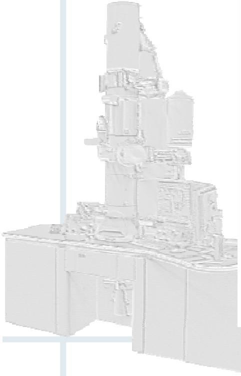
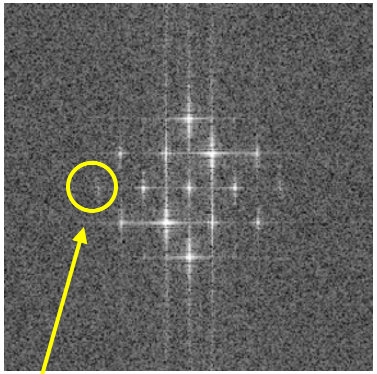
HAADF observation of Dumbbell structure from Diamond<110>



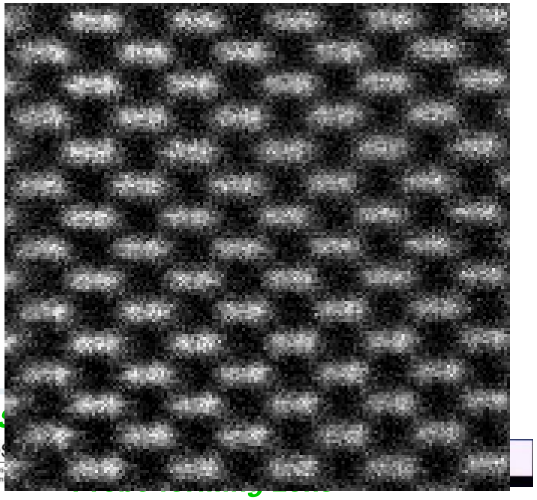




0.089nm



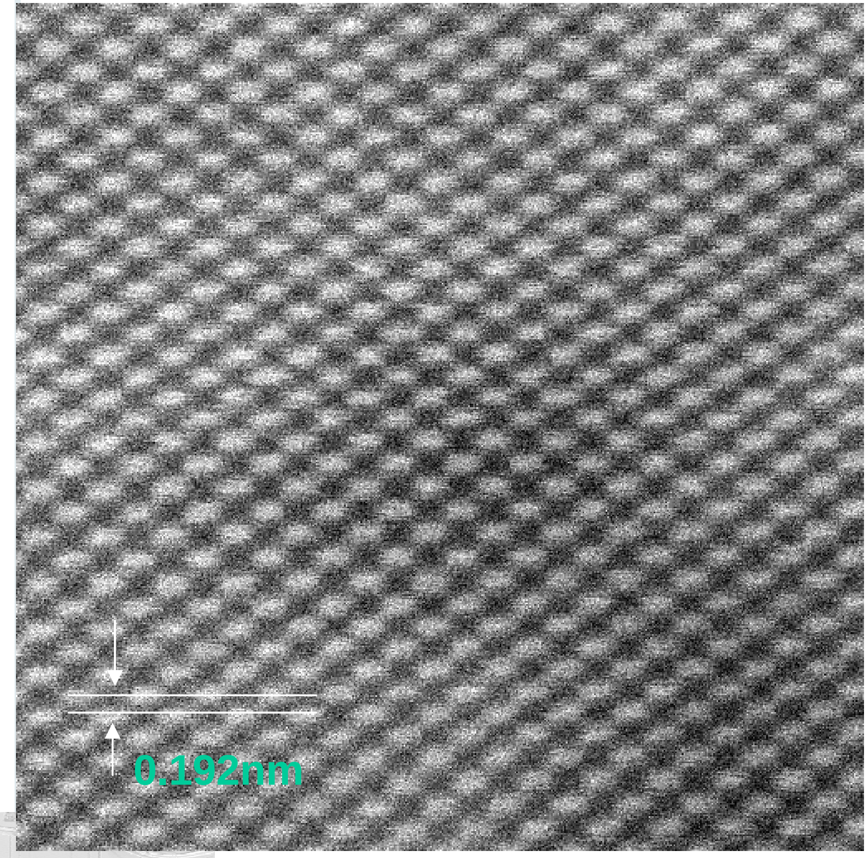
004 spot (0.089 nm)



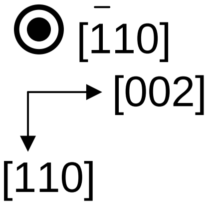
CEOS

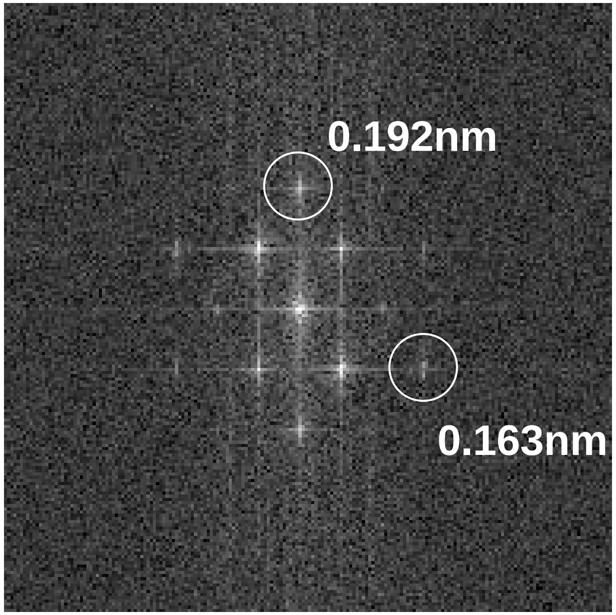
Analytical probe size measured from FFT pattern

HAADF image



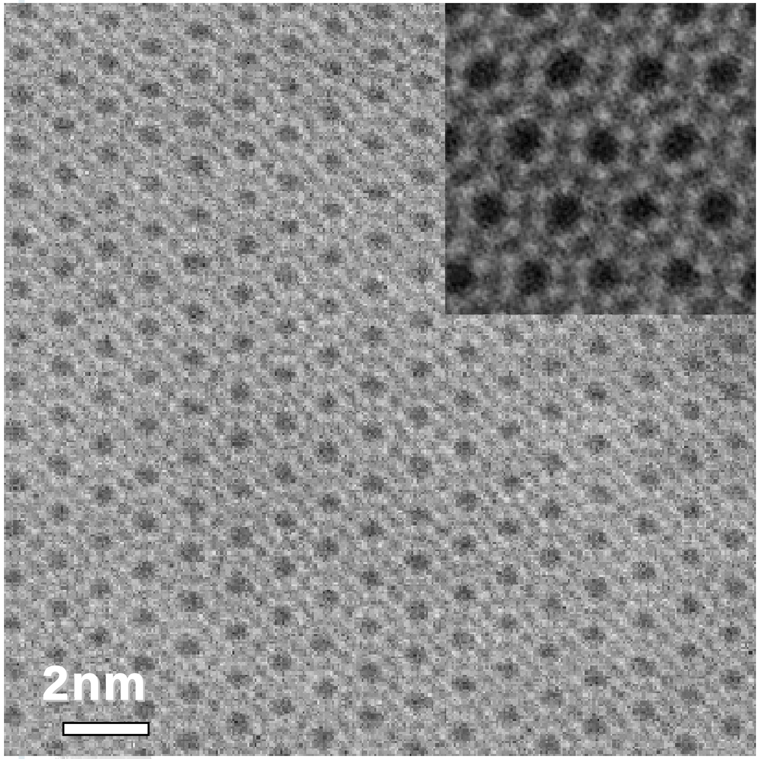
Analytical probe current : 500pA



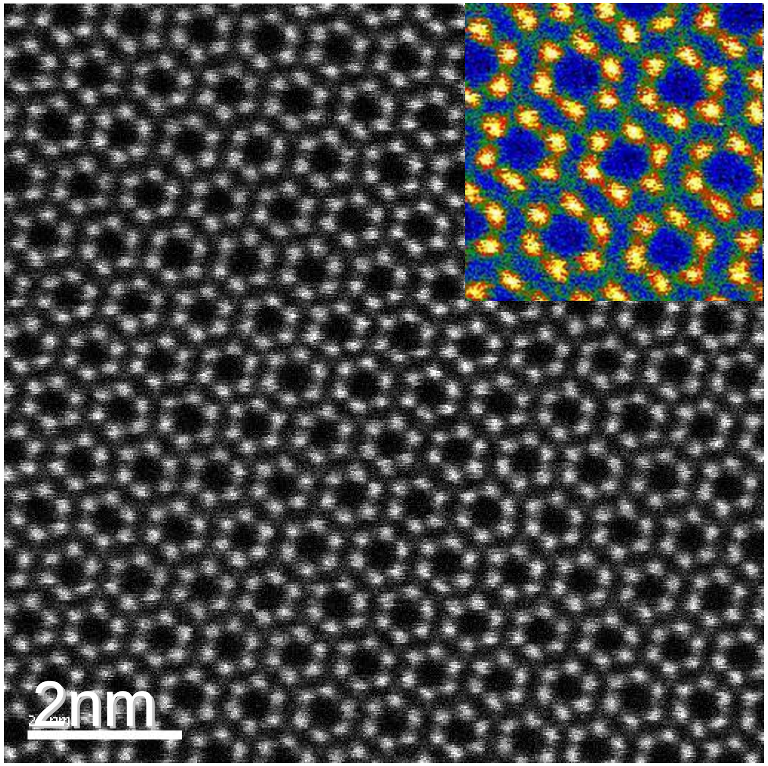


Example: β-Si3 4

HAADF images



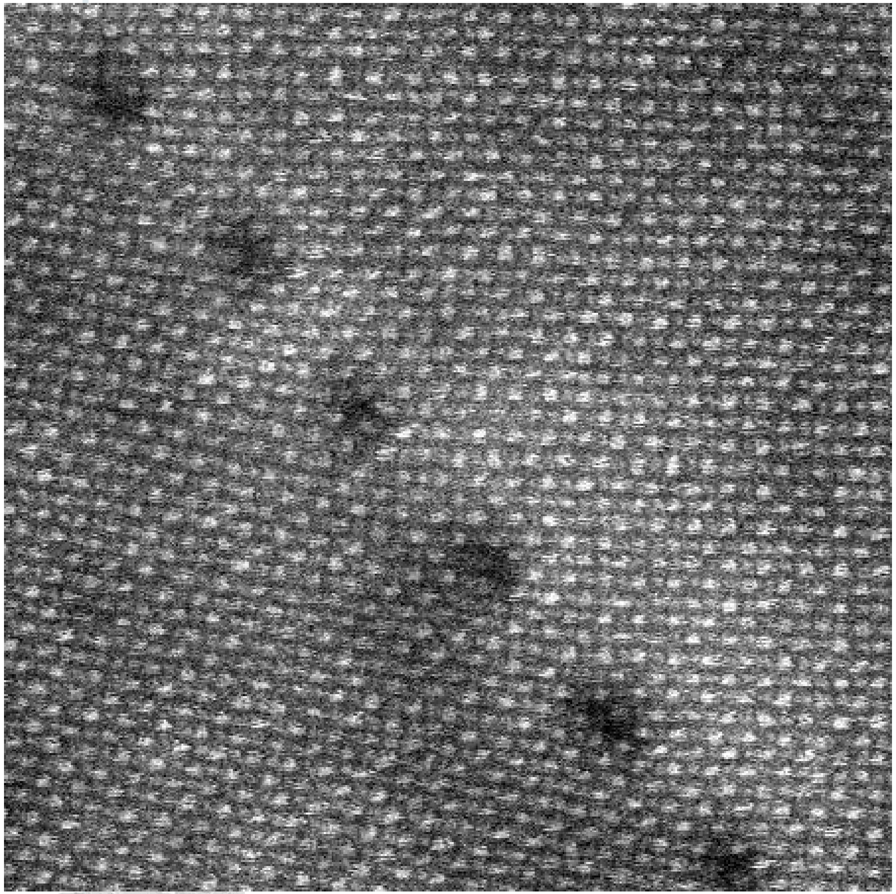
Without Corrector
(Cs:1.0 mm)



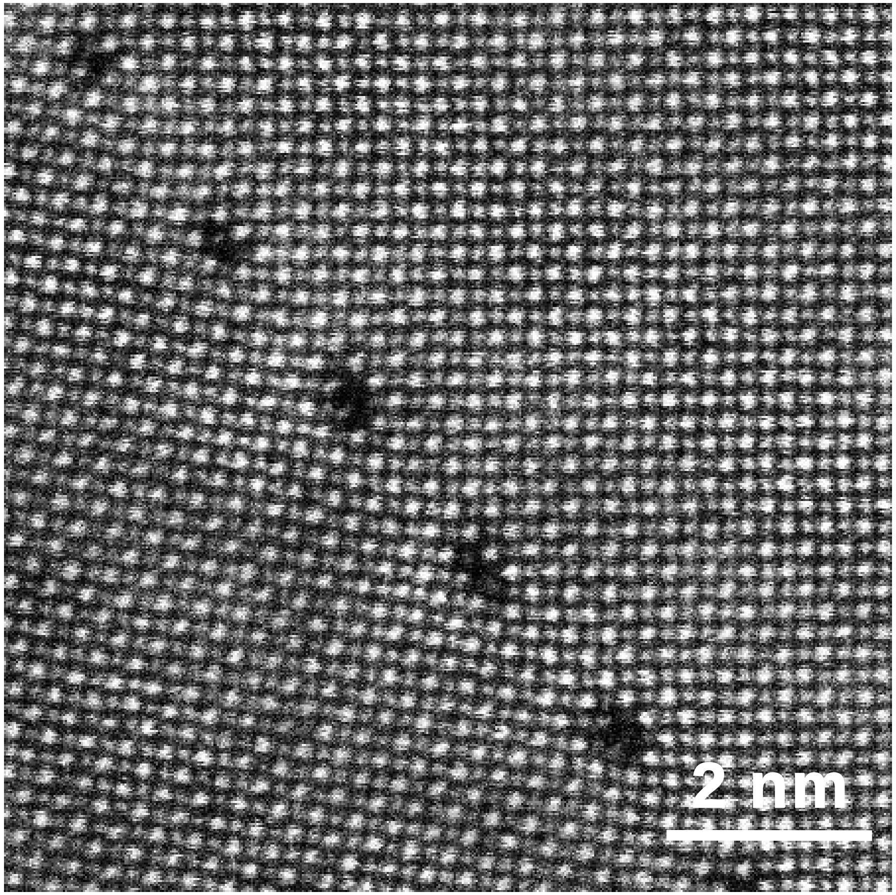
With STEM Cs corrector

Example: SrTiO$_3$ Grain Boundary

HAADF images



Without Corrector

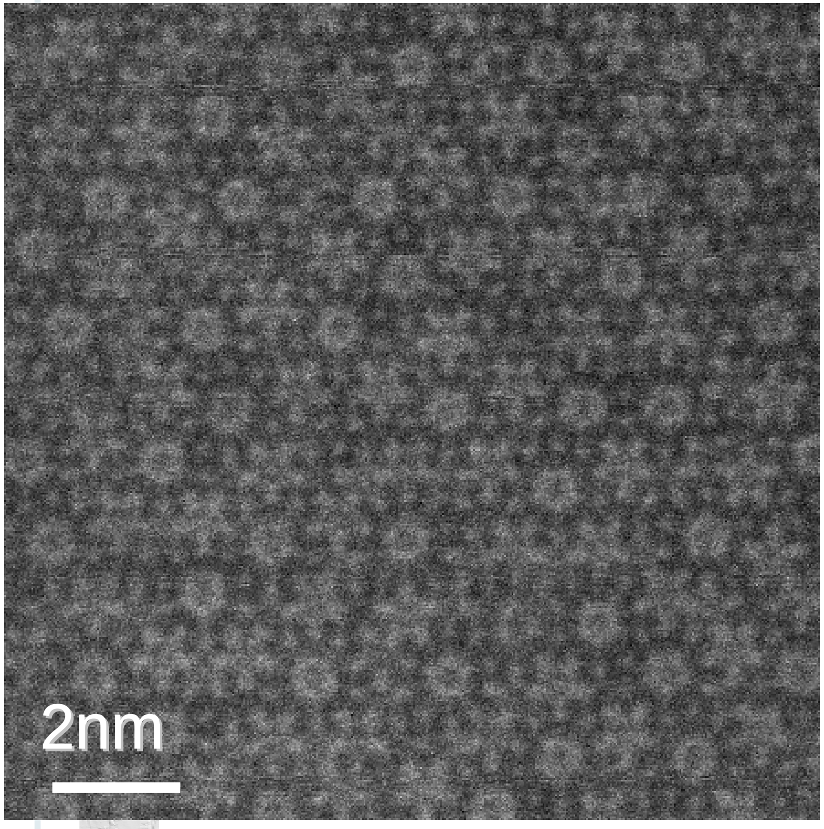


With STEM Cs corrector

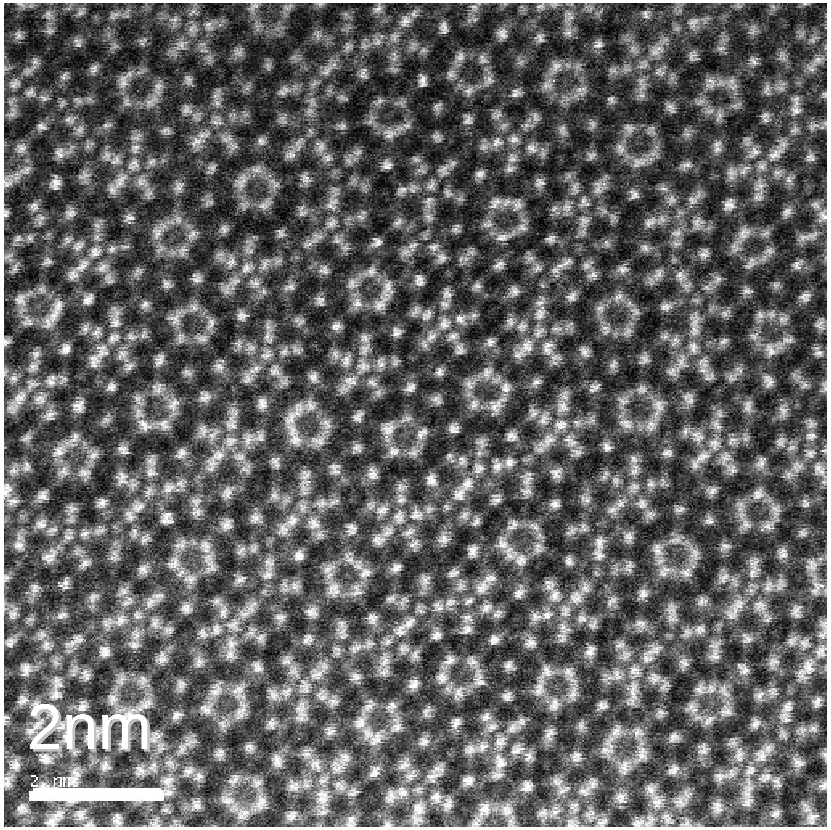
Specimen: courtesy of Prof. Ikuhara/Prof. Yamamoto, The University of Tokyo

Example: Quasicrystal(Al Cu Co)

HAADF images



Without Corrector

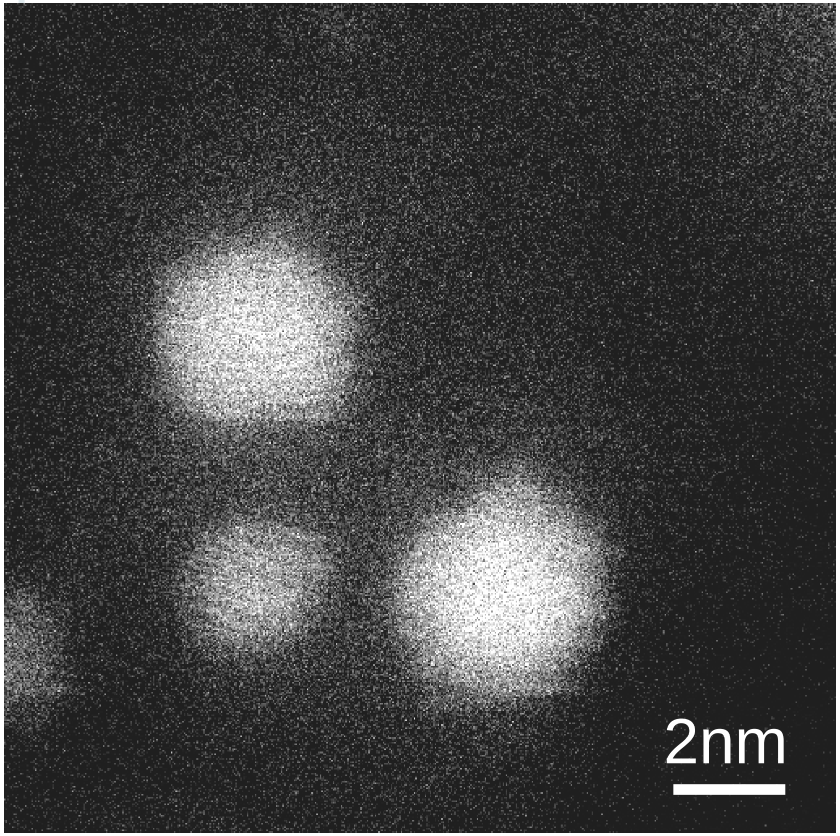


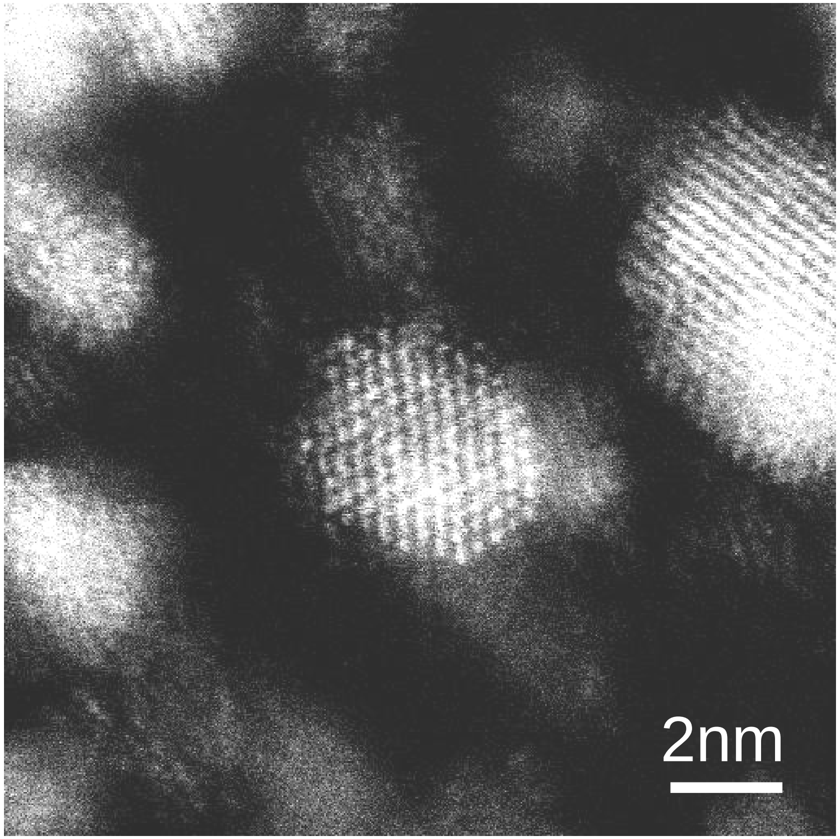
With STEM Cs corrector

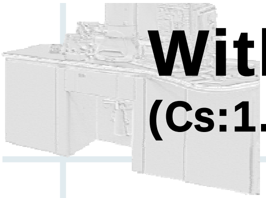
Specimen: courtesy of Prof. Eiji Abe,
The University of Tokyo

Example: Catalyst Pt particles

HAADF images



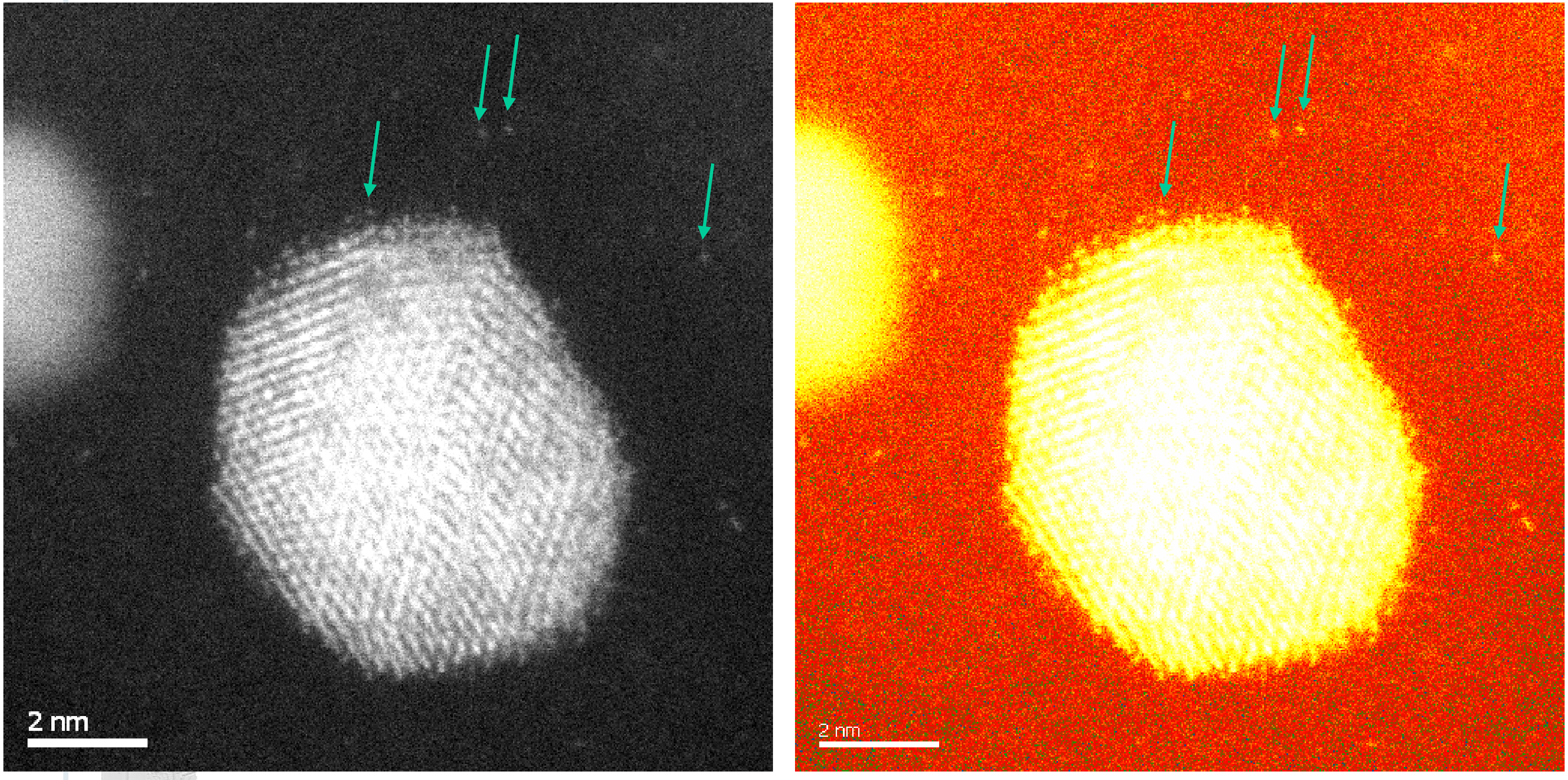




Without Corrector
(Cs:1.0 mm)

With Cs corrector

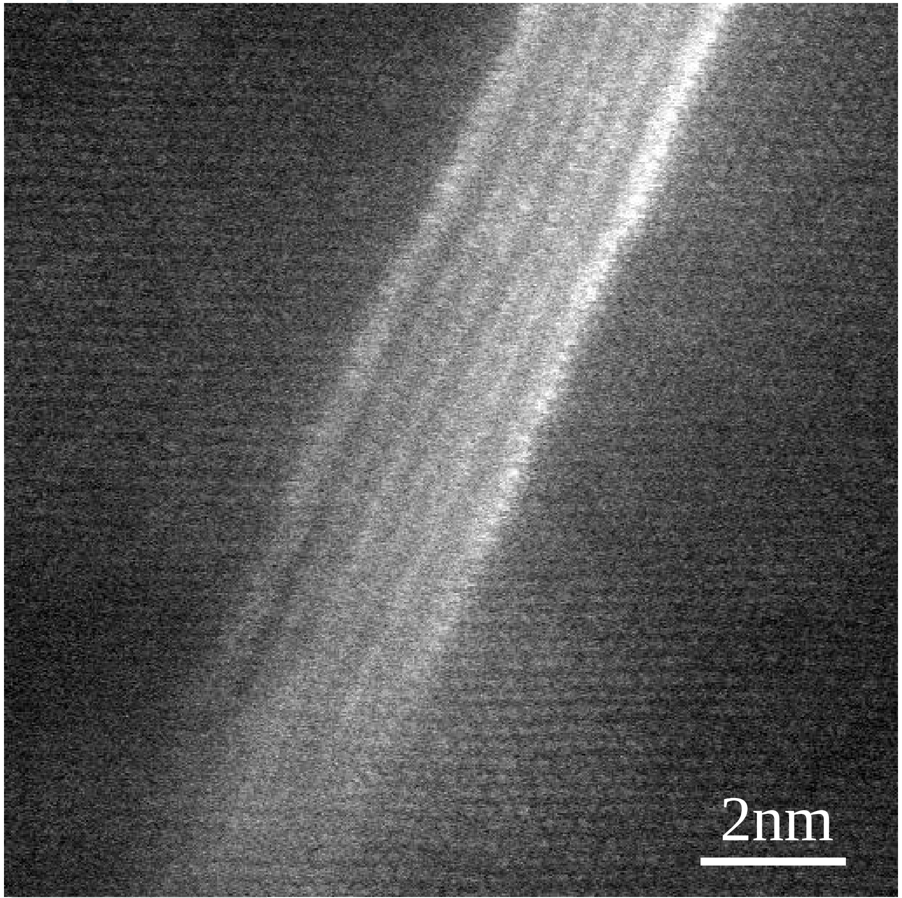
Single atom observation in Pt-C



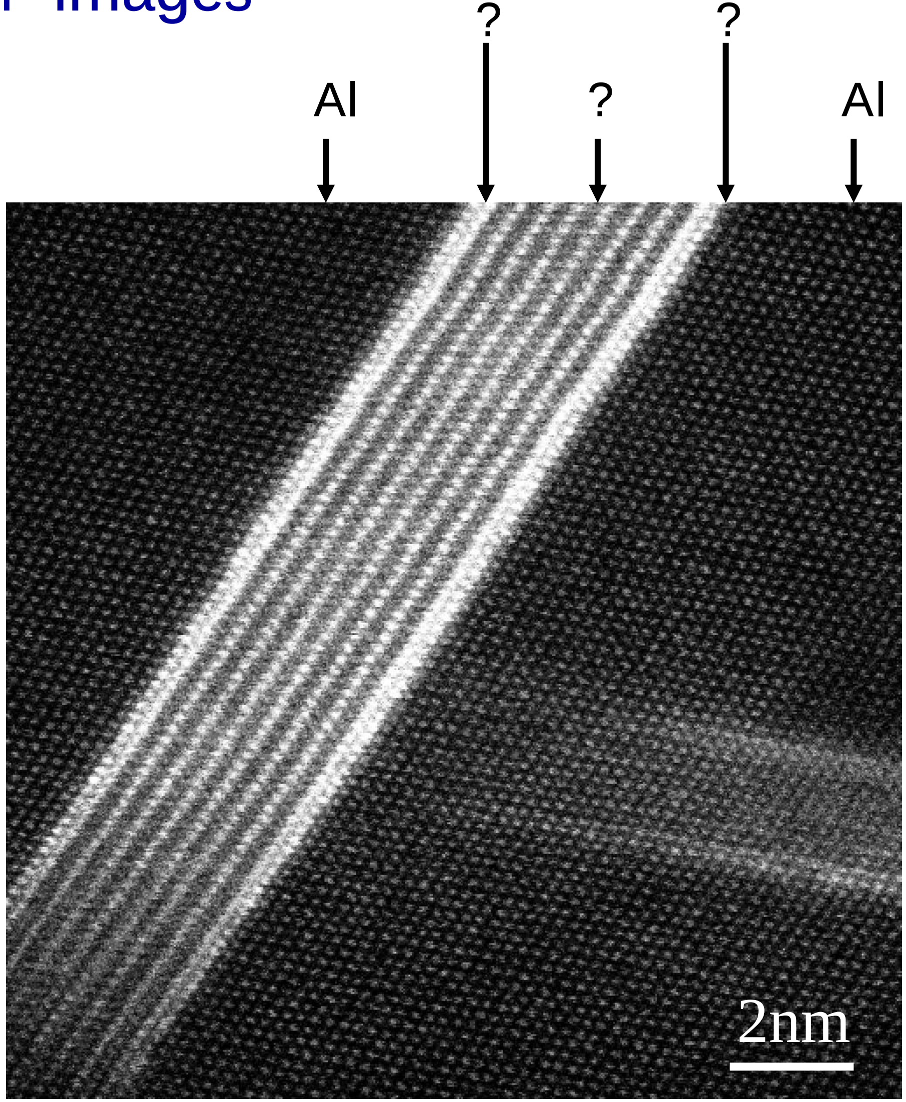
Single Pt atoms can be observed very clearly

Example: Precipitate in Al-Cu alloy

HAADF images

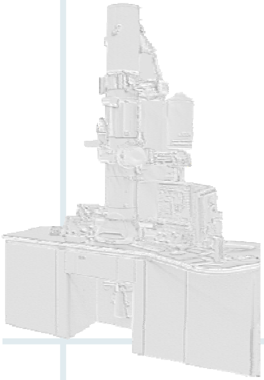


Without corrector



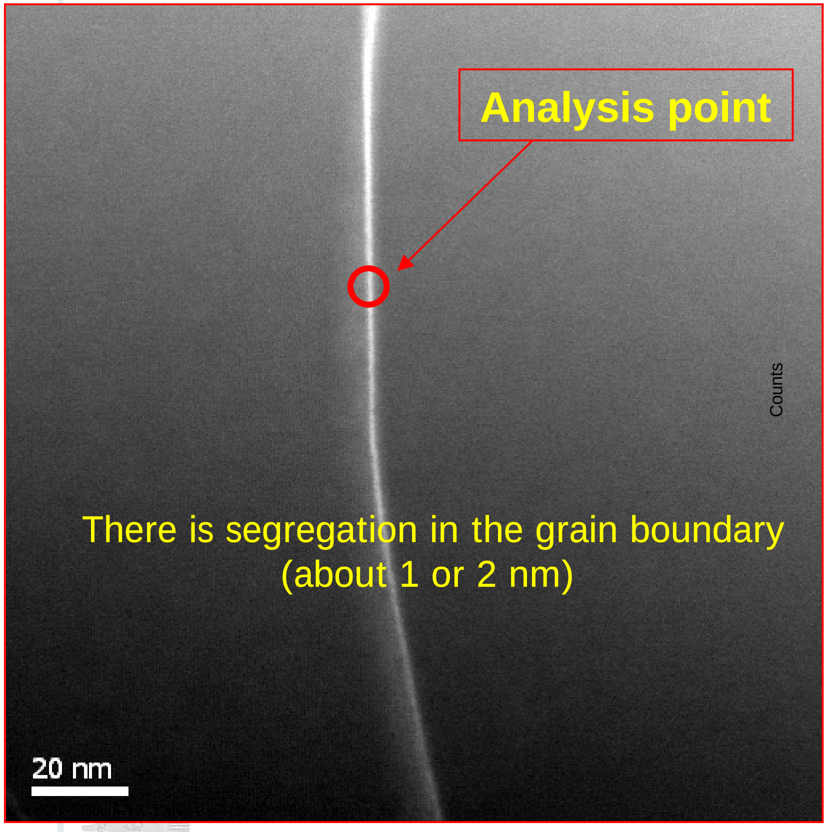
With Cs corrector

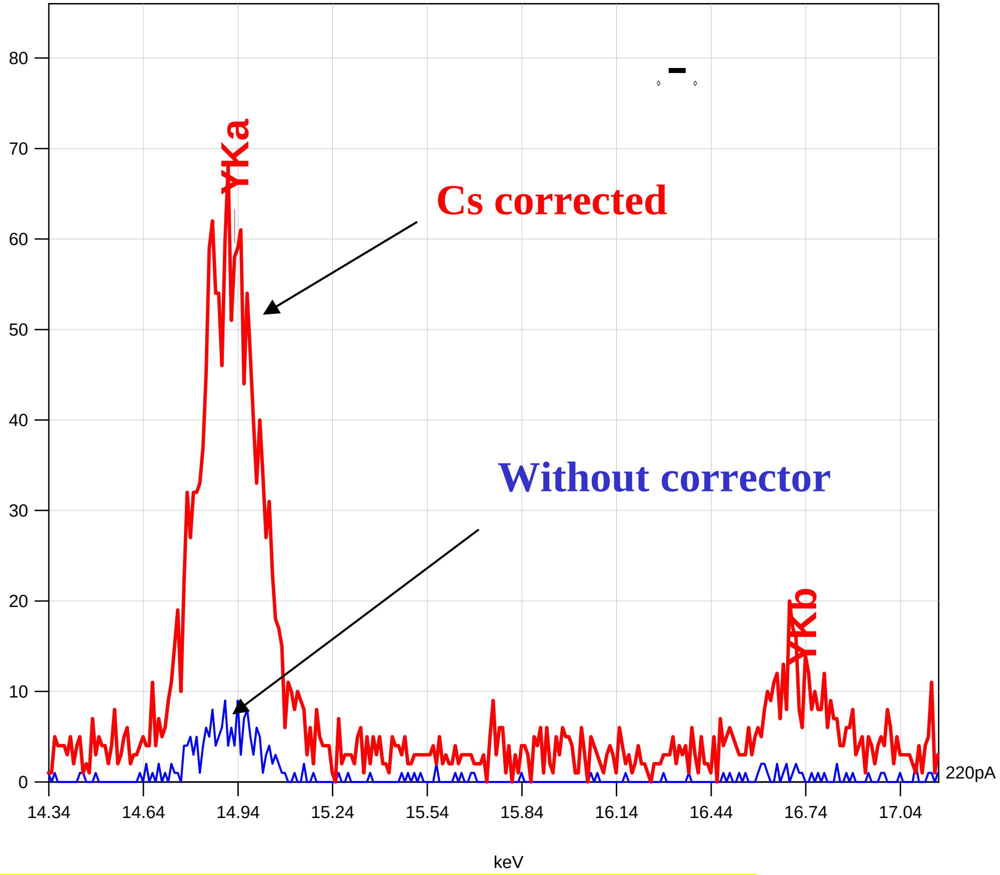
Elemental analysis capability of Cs corrected STEM

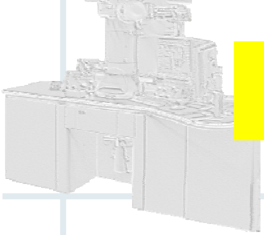


Comparison of EDS spectra (Y-Ka)

0.5nm probe
30s acquisition





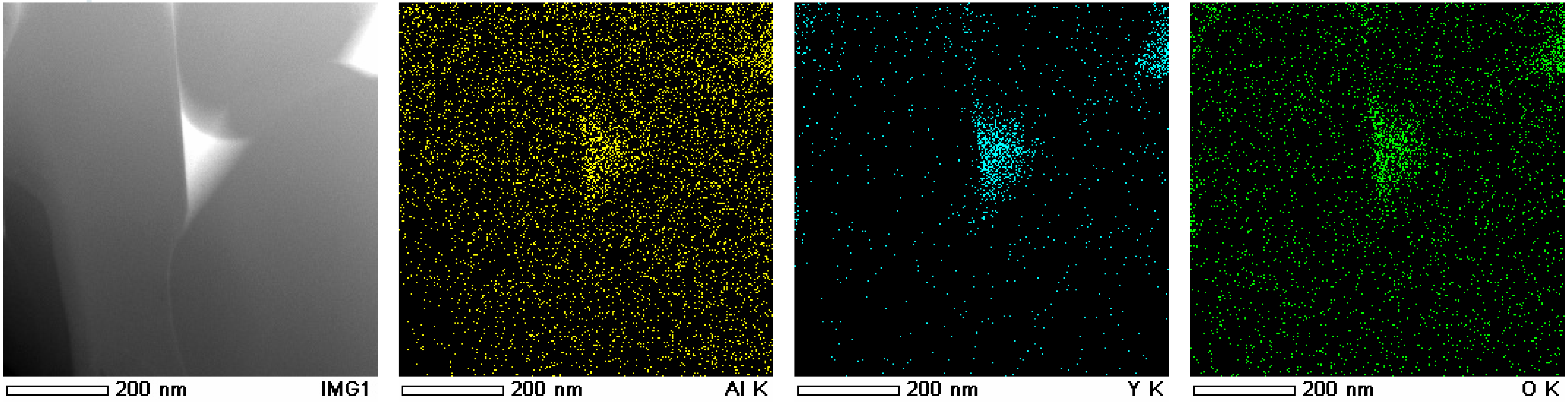


EDS signal is 6 times higher with Cs corrector

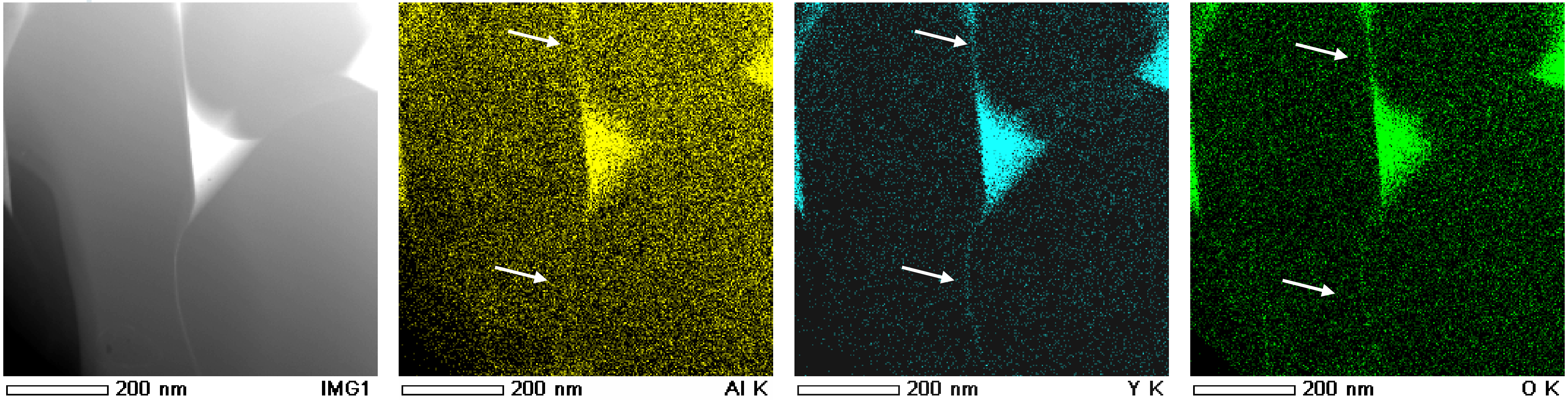
Specimen: SiAlON

EDS mapping of segregated elements (Al,Y,O)

No Cs correction



Cs correction



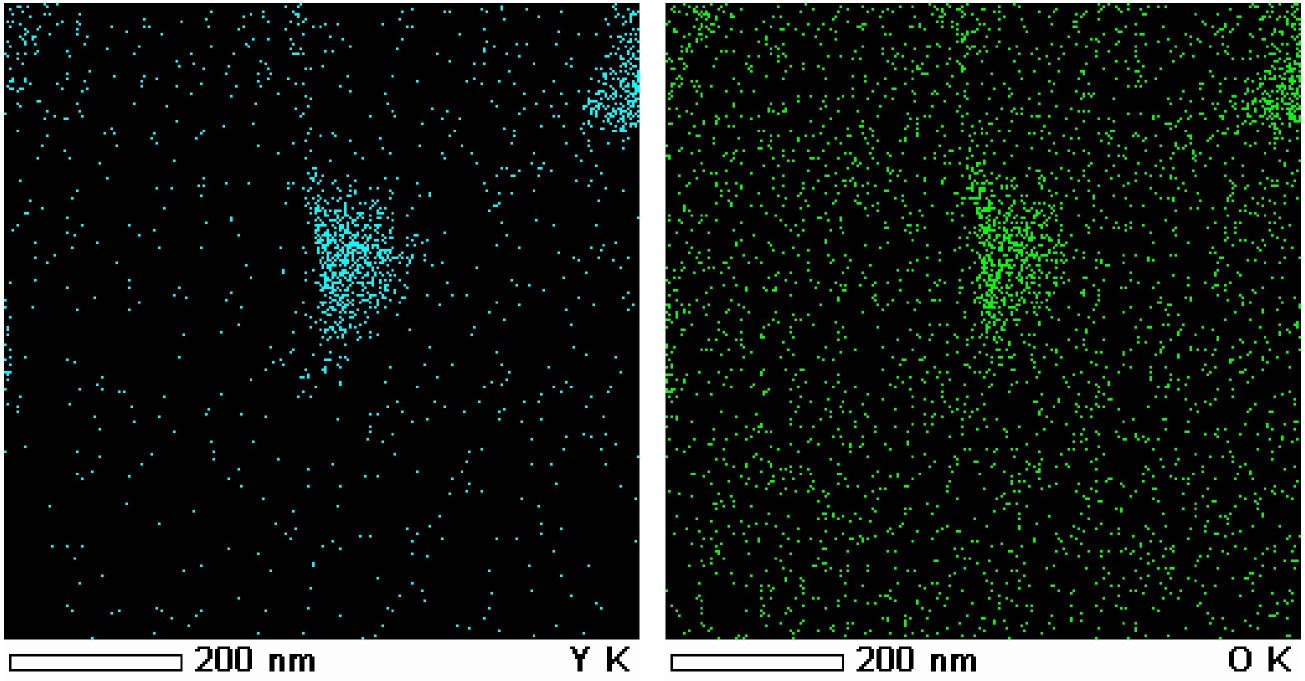
0.5nm probe
256x256 pixels
11 min acquisition

STEM image is same, but EDS map is quite different.

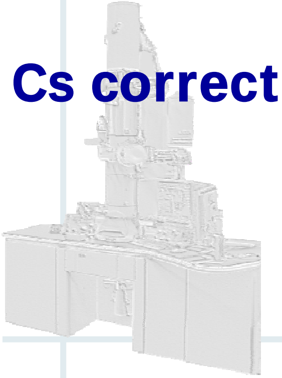
Good S/N ratio

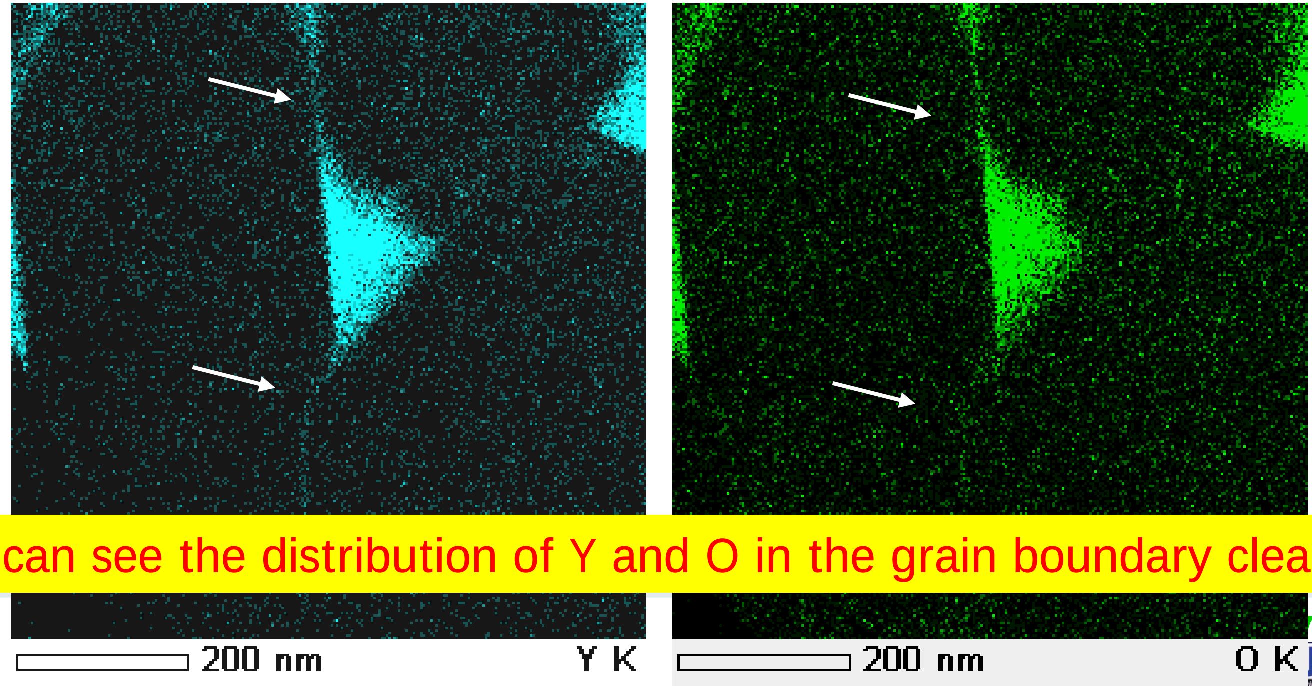
Spherical Aberration corrector
for
Probe forming Lens

No Cs correction



Cs correction

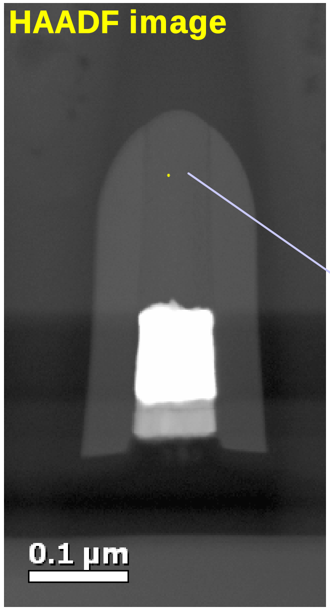




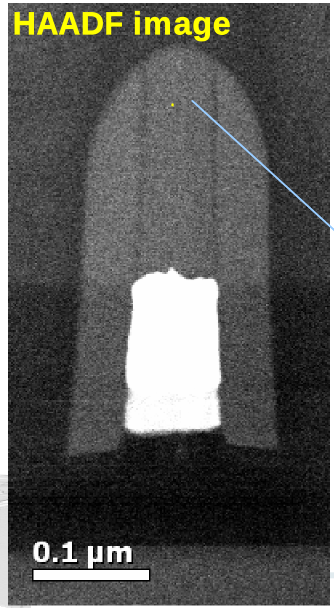
We can see the distribution of Y and O in the grain boundary clearly

EELS on semiconductor device

With Cs corrected



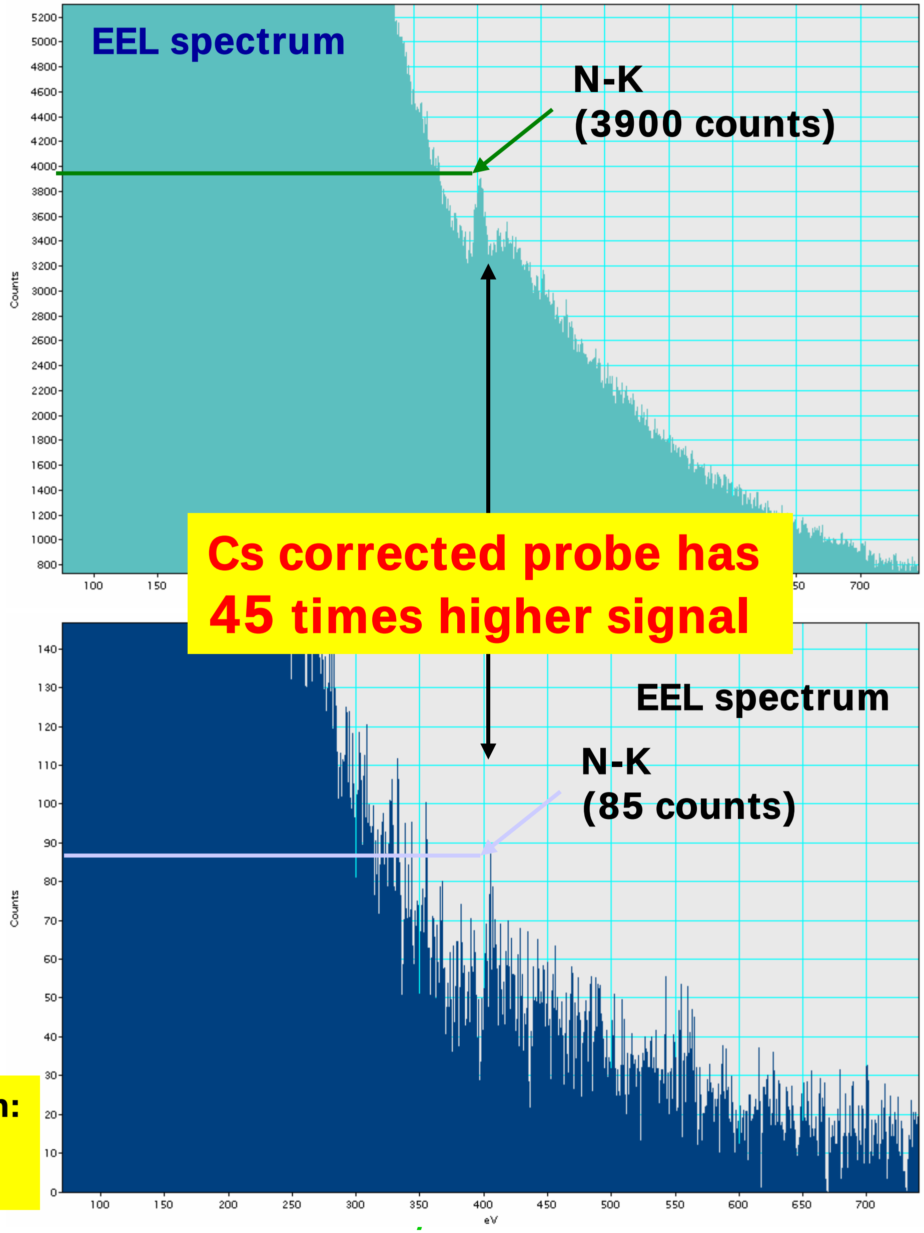
Without Cs corrected



0.5 nm probe

EELS Spectrum from 1 pixel

Si_xN_y area



Cs corrected probe has 45 times higher signal

Acquisition condition:
0.5 nm probe
0.001 sec / pixel

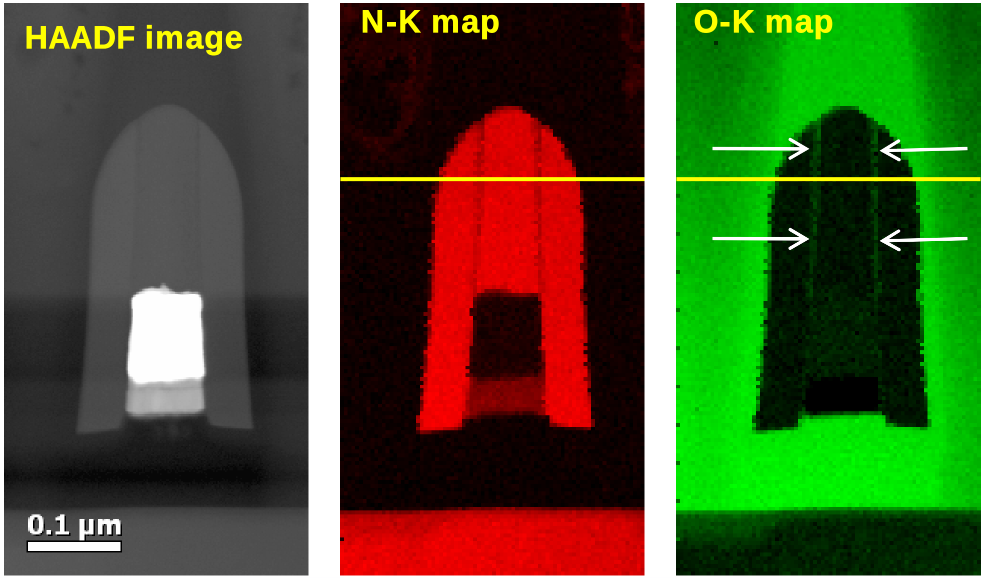
JEM-2100F with CESCOR

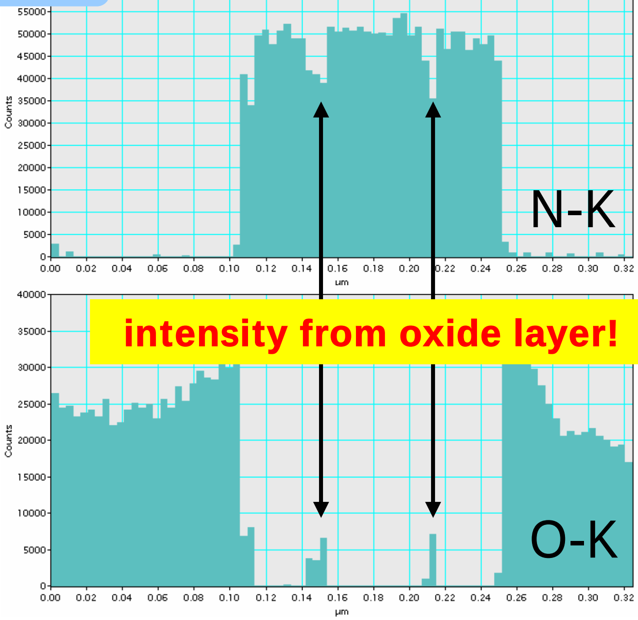
CEOS Corrected Electron Optical Systems GmbH *for Probe forming Lens* JEOL Serving Advanced Technology

Comparison of EELS mapping signal intensity

Intensity profiles

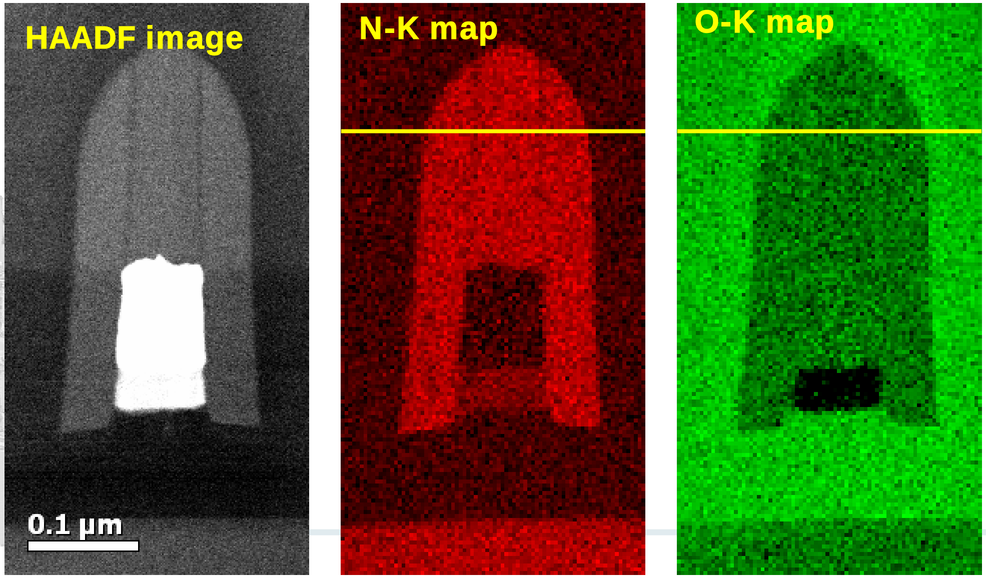
With Cs corrected

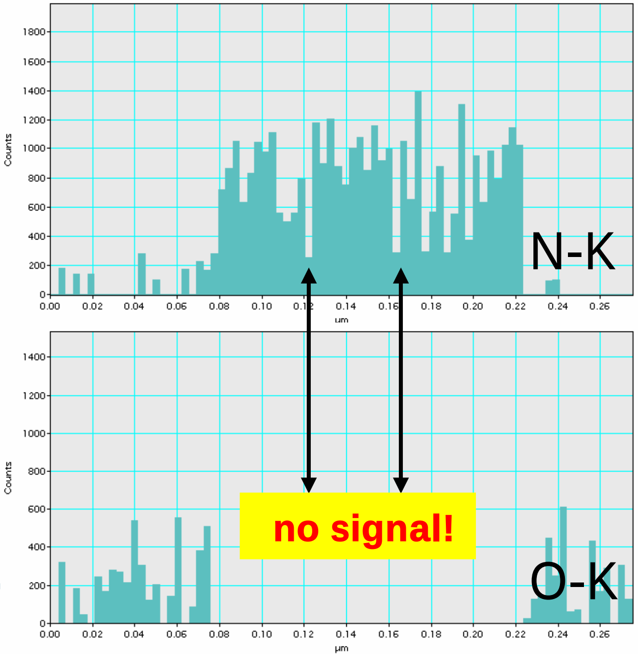




intensity from oxide layer!

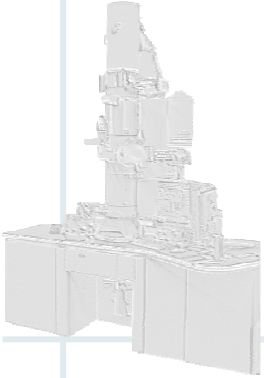
Without Cs corrected



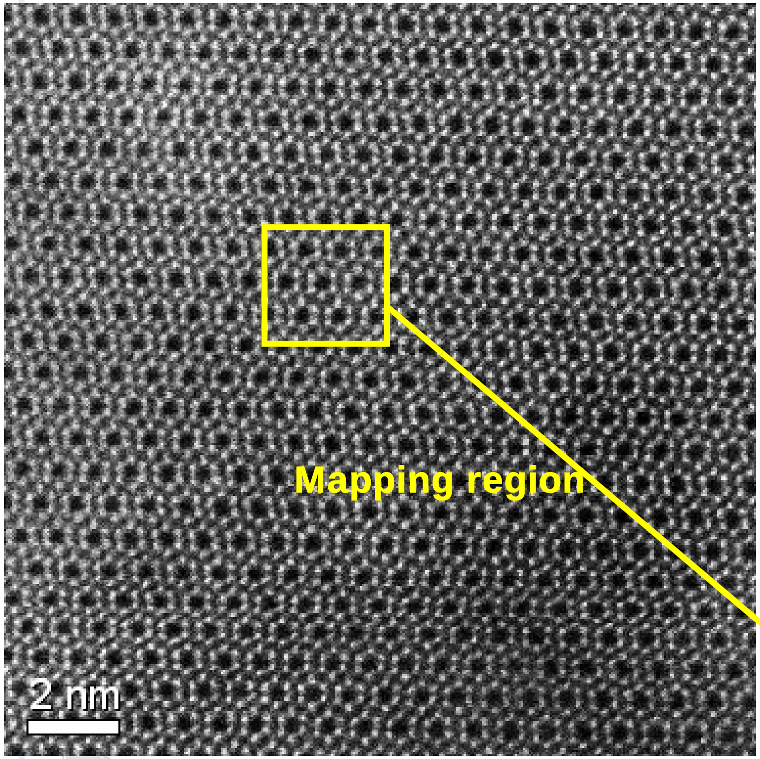


no signal!

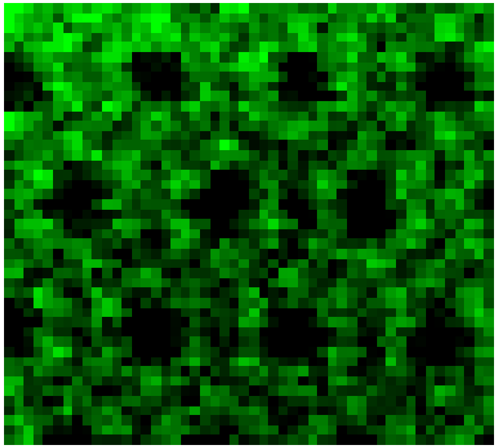
Atomic level analysis capability of Cs corrected STEM (Atomic column by column mapping)



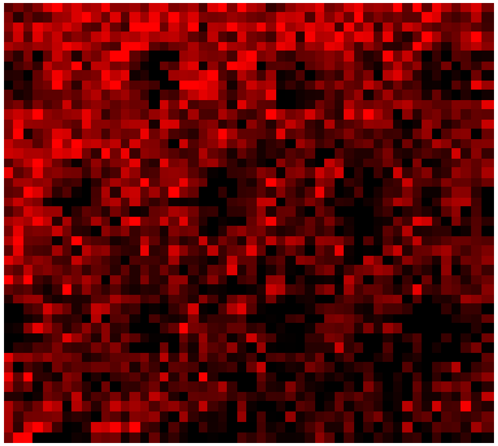
Atomic column by column EELS mapping of Si3N4



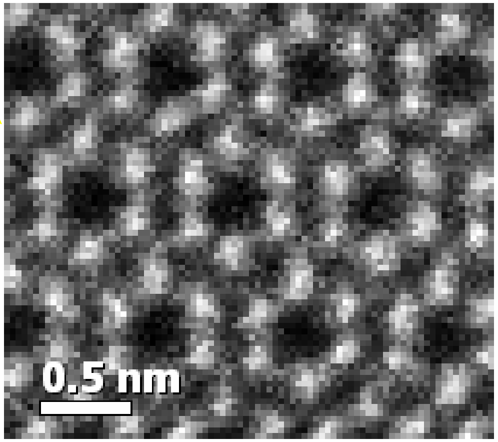
Si-L



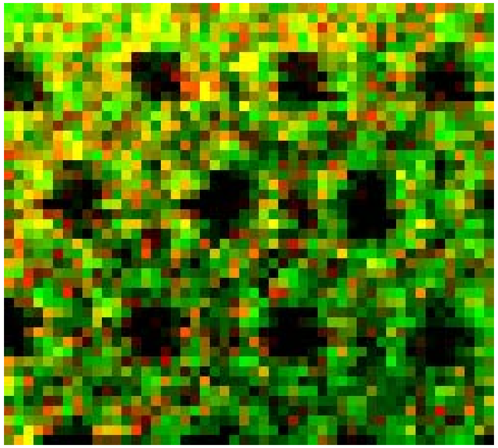
N-K

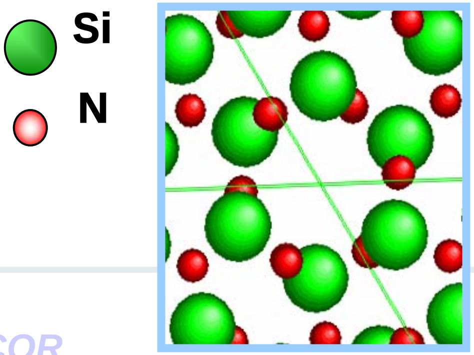


HAADF image

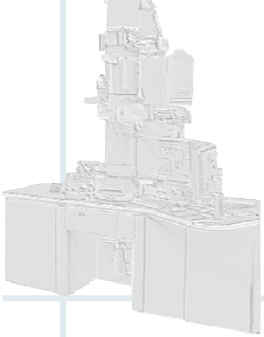


Overlap map





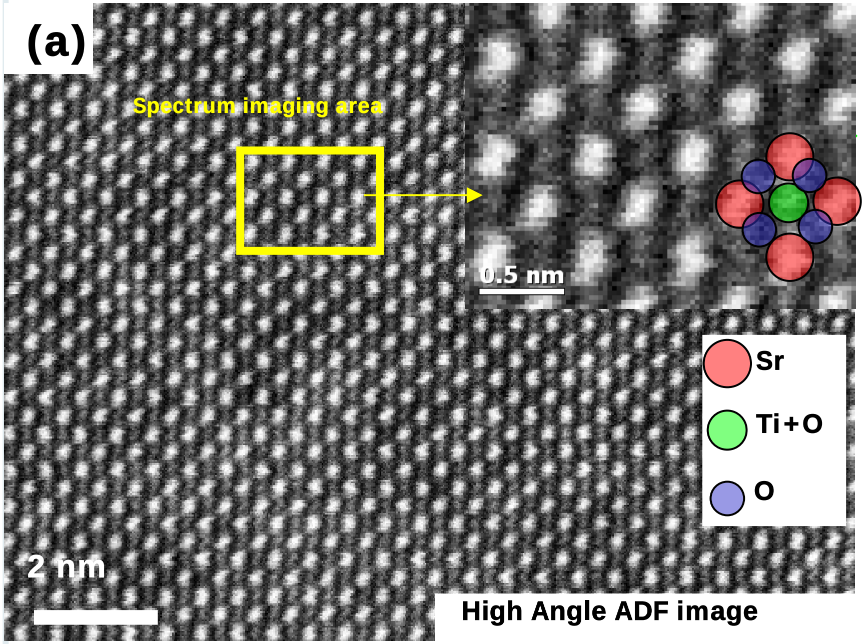
Atomic resolution maps are obtained !



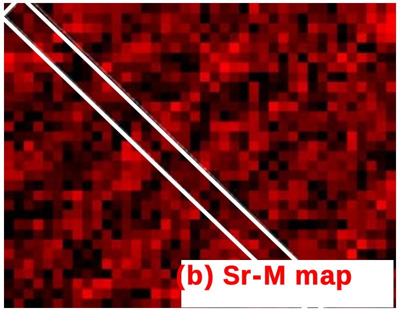
JEM-2100F with CESCOR

Spherical Aberration corrector for Probe forming Lens

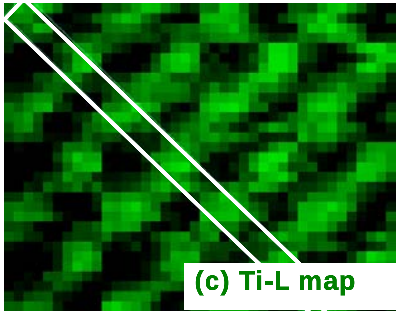
Atomic column by column EELS mapping of SrTiO3



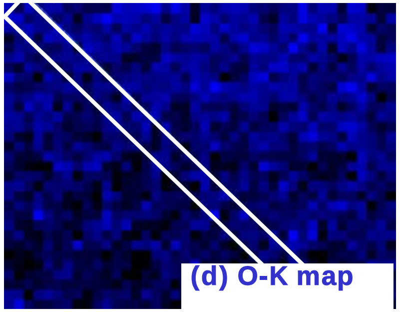
High Angle ADF image



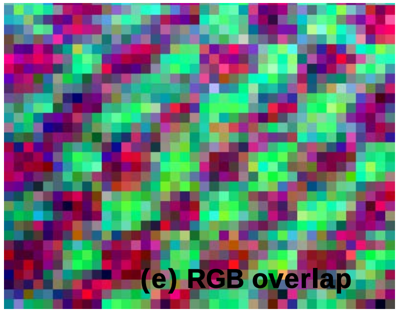
(b) Sr-M map



(c) Ti-L map

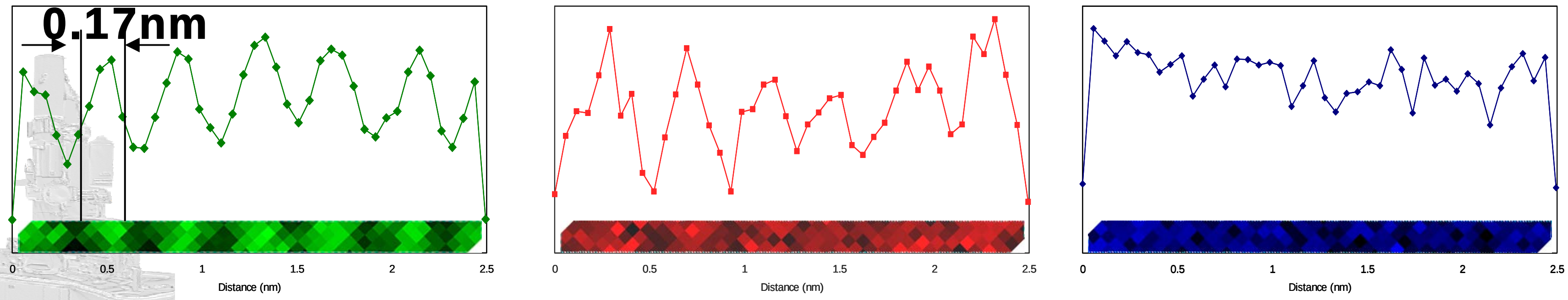


(d) O-K map



(e) RGB overlap

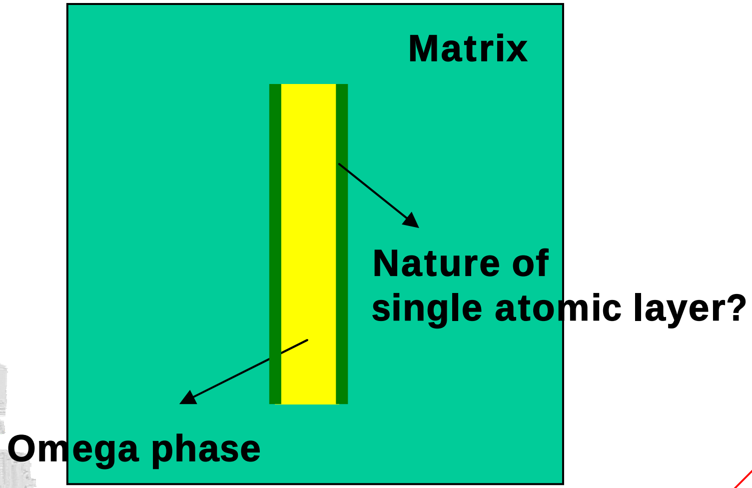
Intensity profiles from white rectangle area in EELS map

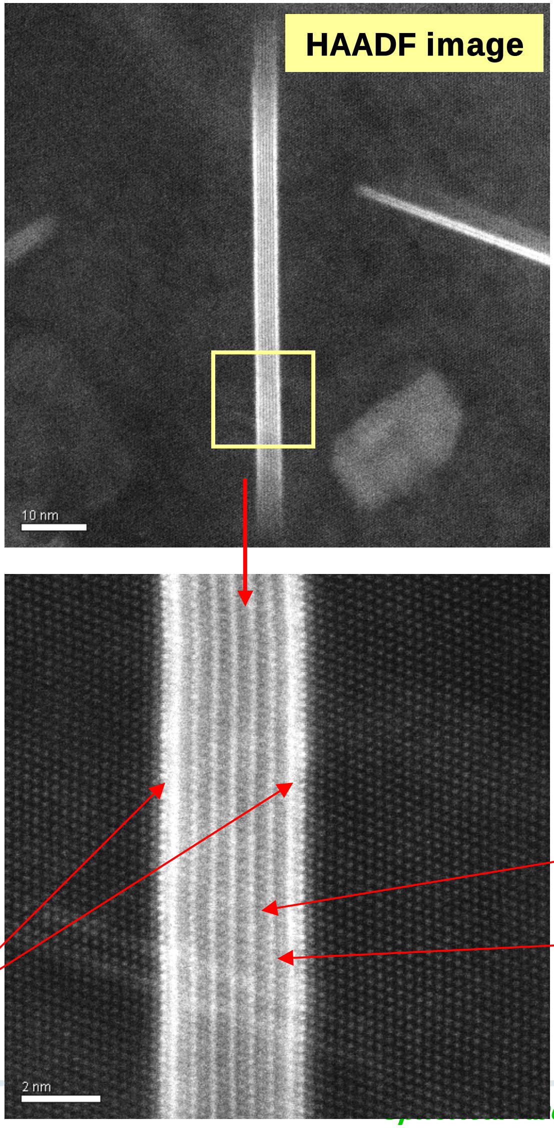


High-Resolution High Speed Mapping by EDS of omega phase in Al-Cu alloy

Mg and Ag contained Al-Cu alloy has precipitates called omega phase(structure is Cu_2Al).

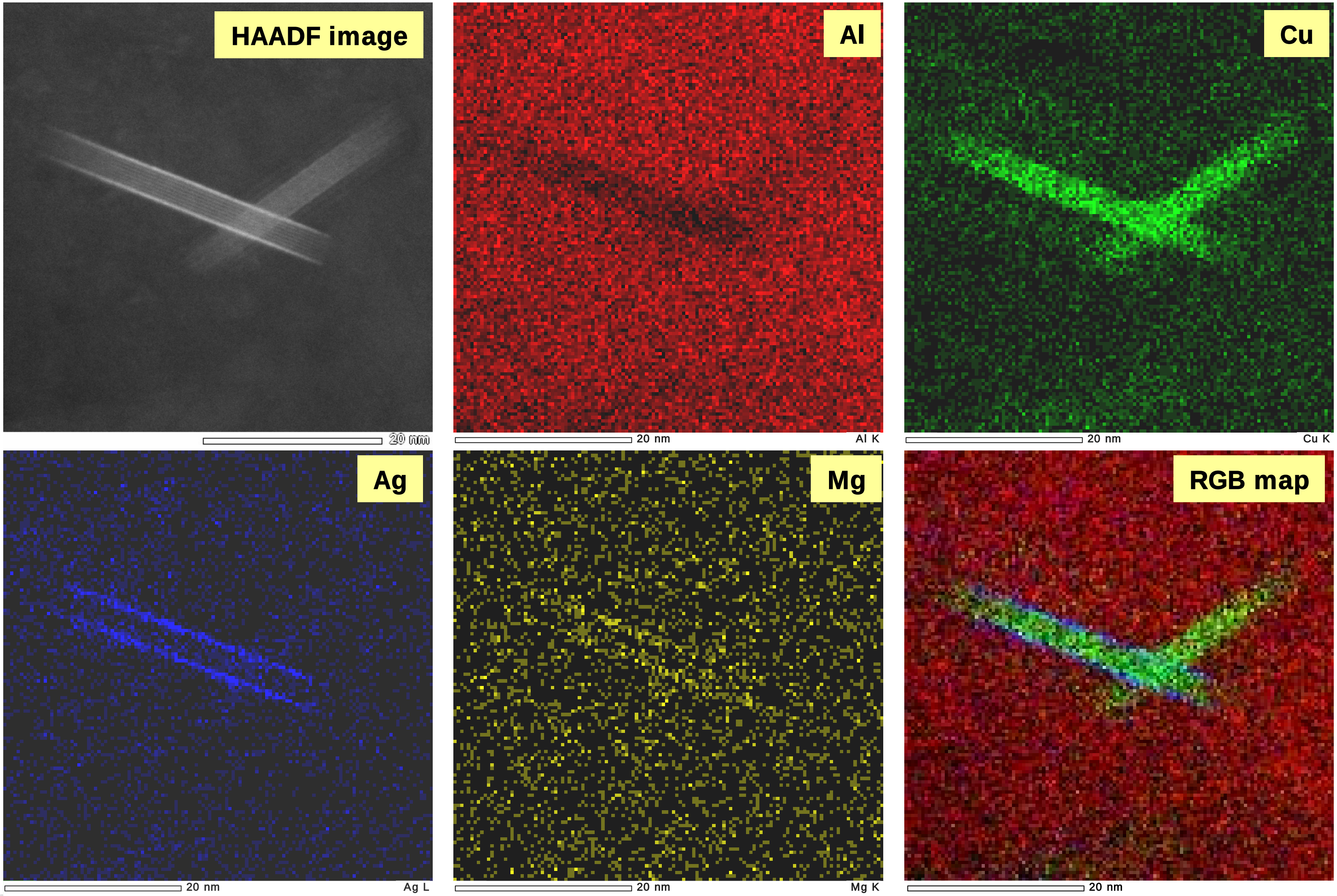
What is the nature of the atomic layers between matrix and omega phase?





EDS mapping

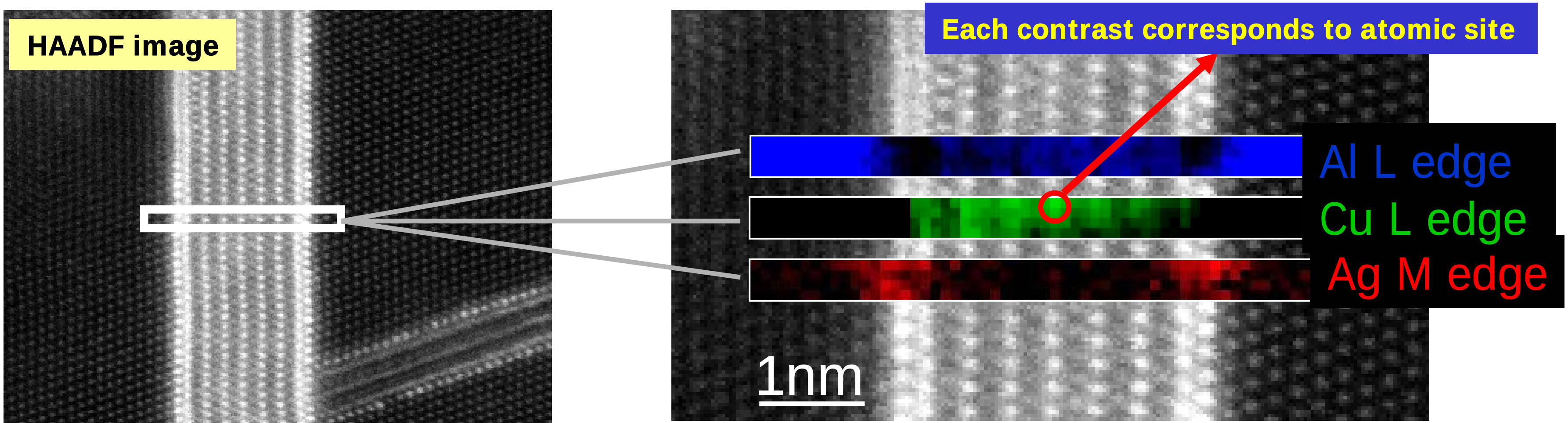
Specimen: courtesy of Prof. Hono, NIMS

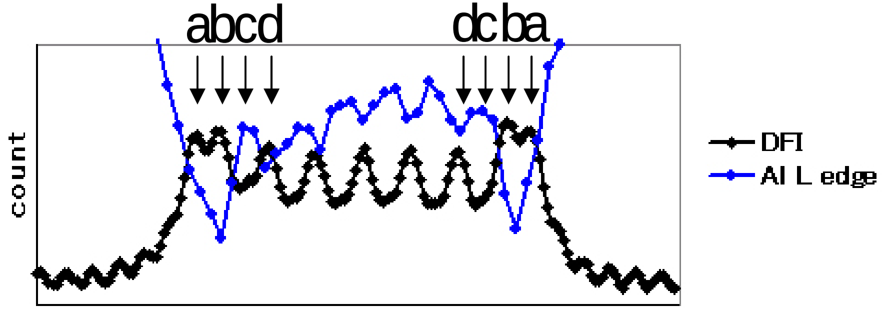


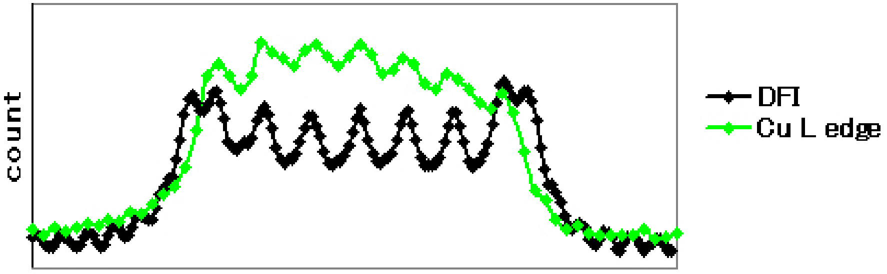
Measurement condition
Pixel number 128x128pixel
Frame number 0.5msec x 150 times
Total time : 20 min
Probe size 0.13 nm
Magnification M

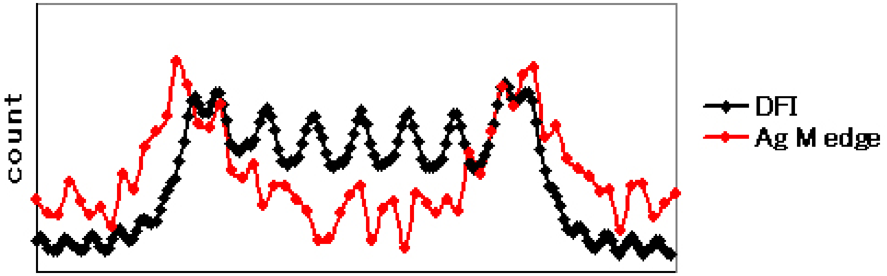
Ag and Mg layer can be visualized in only 20 min!

Atomic resolution EELS mapping









JEM-2100F with CESCOR

Spherical Aberration corrector
for
Probe forming Lens

CEOS
Corrected Electron Optical Systems GmbH

JEOL
Serving Advanced Technology

To conclude

Cs corrected STEM :

- **higher resolution imaging in BF and HAADF modes**
- **high throughput elemental/chemical analysis (EDS, EELS)**
- **atomic scale elemental/chemical analysis (EDS, EELS)**
- **accessible and robust tool for materials R&D (alloys, ceramics, interfaces, …)**