New: Windowless high collection angle EDS with 100mm² oval silicon drift detector area

Meiken Falke, Andi Käppel, Igor Nemeth

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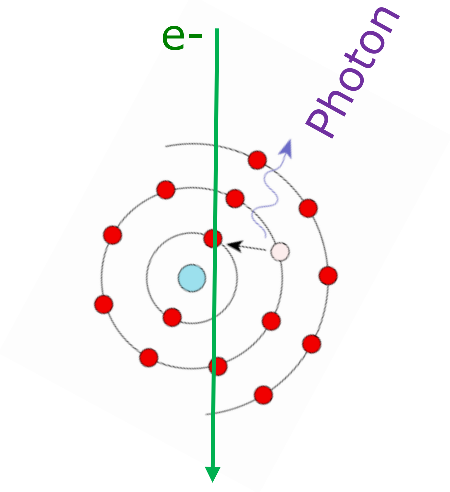
Bruker Nano Analytics

Windowless high collection angle EDS with 100mm² oval silicon drift detector area

Outline

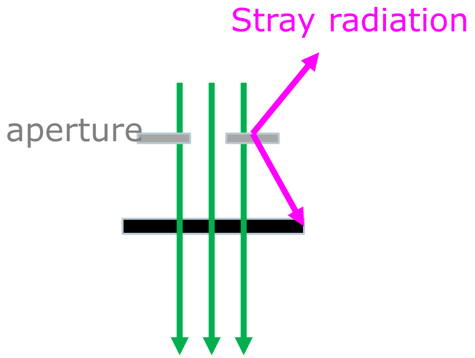
- Intro: EDS for TEM/STEM and SEM/T-SEM
- History, current specifications
- Geometry Considerations
  - Constraints in microscopes
  - Specimen holders and mounting
- Application examples
- Short summary



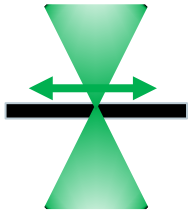
XFlash®6(T)-100 oval

Electron Microscopy

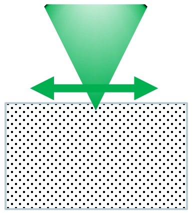
TEM
Parallel illumination



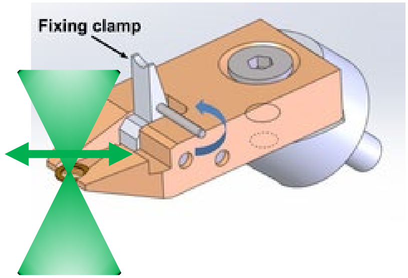
STEM
Scanning TEM



SEM
Scanning EM



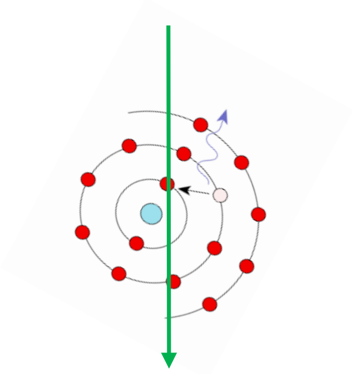
STEM in SEM: „T-SEM“



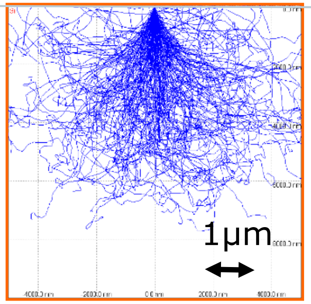
- TKD holder, patented
- Commercial STEM holders and detectors
- Home made versions

Spatial Resolution for Bulk and Electron Transparent Specimens

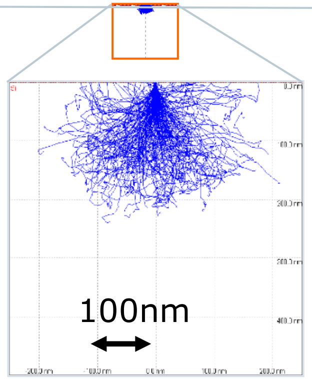
SEM: bulk



High voltage
30kV

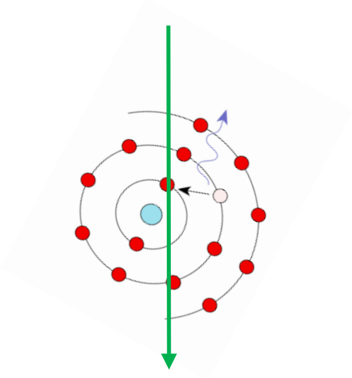


Lower voltage
4kV



Spatial Resolution for Bulk and Electron Transparent Specimens

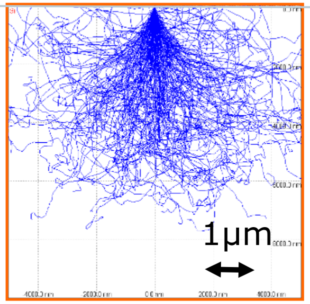
SEM: bulk

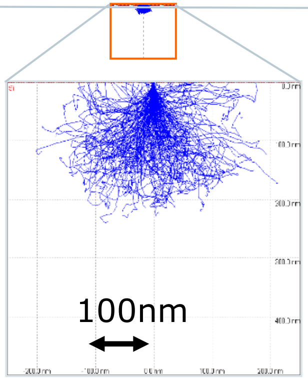


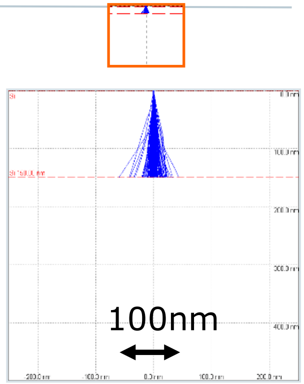
S/TEM, T-SEM (trajectories for 30kV): thin specimen, small probe

High voltage 30kV

Lower voltage 4kV



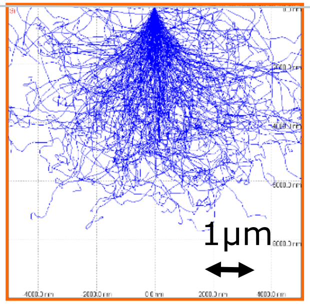




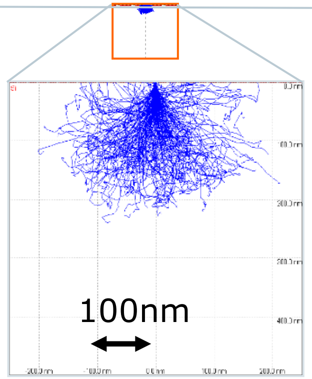
Spatial Resolution for Bulk and Electron Transparent Specimens

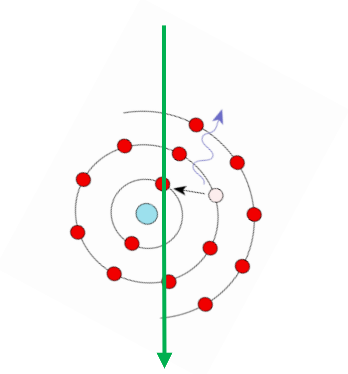
SEM: bulk

High voltage
30kV

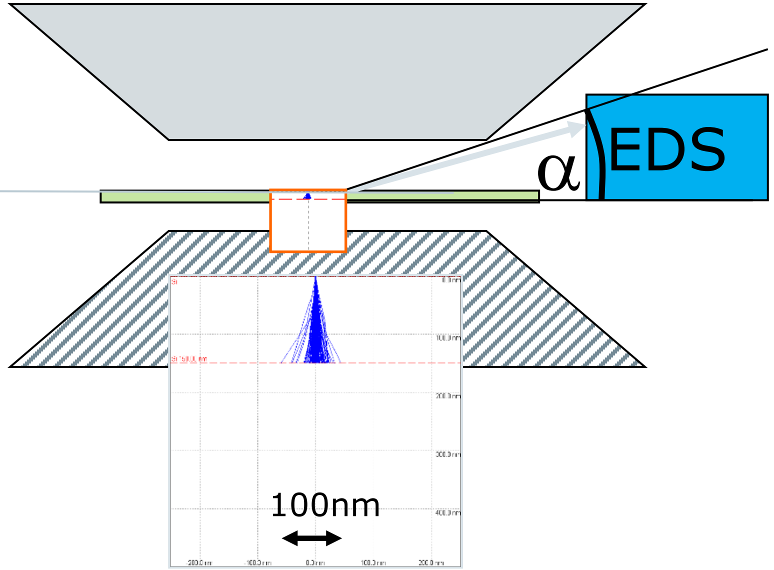


Lower voltage
4kV



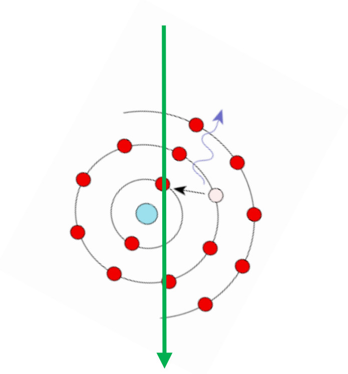


S/TEM, T-SEM (trajectories for 30kV):
thin specimen, small probe



Spatial Resolution for Bulk and Electron Transparent Specimens

SEM: bulk



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High voltage
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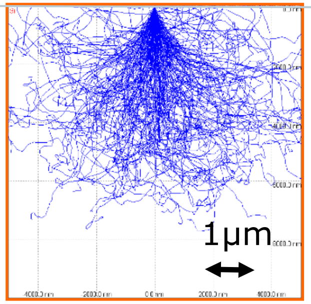
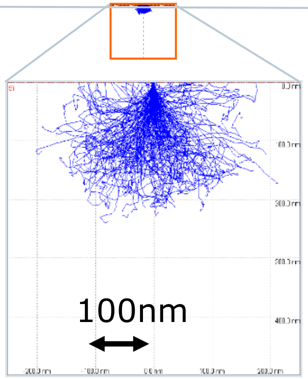
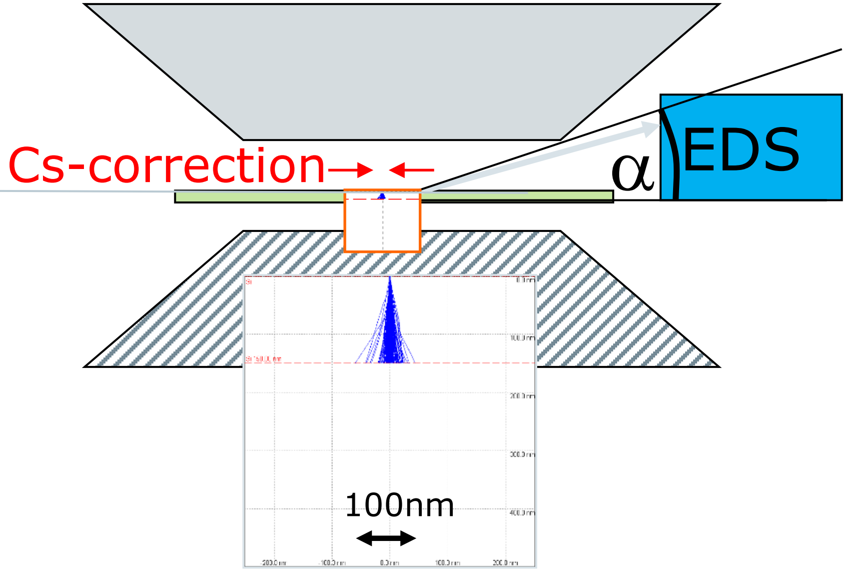


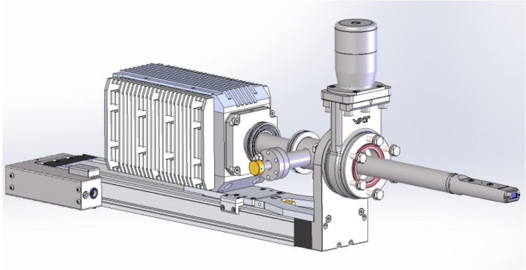
Lower voltage
4kV





XFlash®6-100 / 6T-100 oval; History

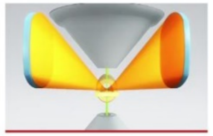
1. Tailored to fit Nion STEM



UHV,
dedicated STEM,
CFEG

2. Special variation for ThermoFisher SCIENTIFIC

DualX



3. Now launching for additional and retrofit adaptions on all suitable TEM/STEM and SEM/T-SEM types e.g.
Jeol (F200),
TFS (Tecnai and Titan),
Hitachi … Tescan … Zeiss

Windowless high collection angle EDS with 100mm² oval silicon drift detector area and All advantages of the ESPRIT analysis software

XFlash®6-100 oval for SEM

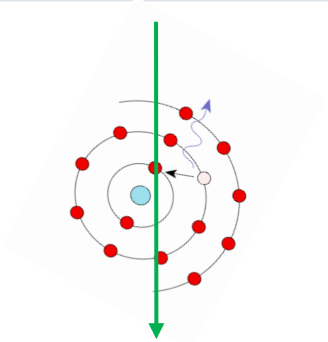
- Compatible to all types of SEM, FIB-SEM incl. STEM in SEM (T-SEM)
- Accelerating voltage up to 30 kV
- Optimized for each microscope geometry
- Solid angle of up to 0.4 sr, compared to approx. 0.08 sr for a round 100mm² detector area on the same SEM

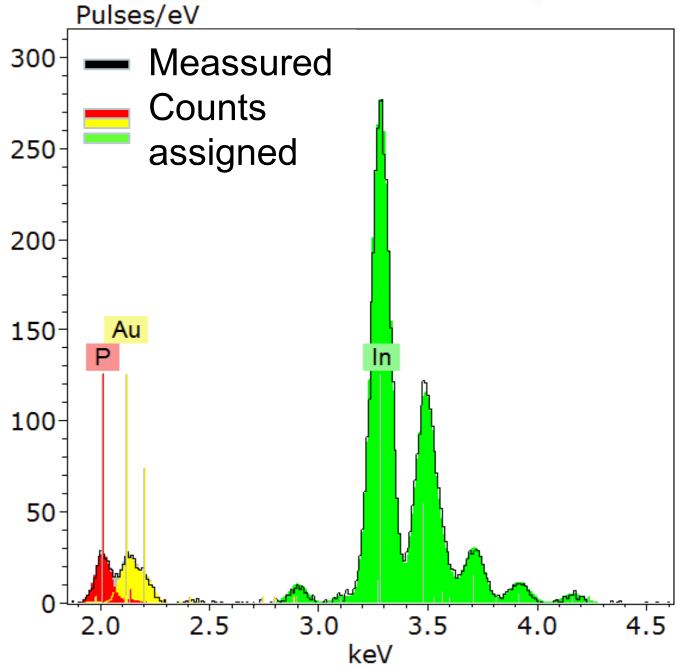
XFlash®6T-100 oval for STEM

- Accelerating voltage up 300 kV, UHV compatible
- Tailored to individual pole piece geometry
- Solid angle up to 0.7 sr or e.g. 0.4 sr, compared to 0.14 sr with round 100mm² area on the same STEM
- Take-off angle up to 13.4° - adaptions vary

Quantitative EDS:

The line intensity for a particular element line / transition:



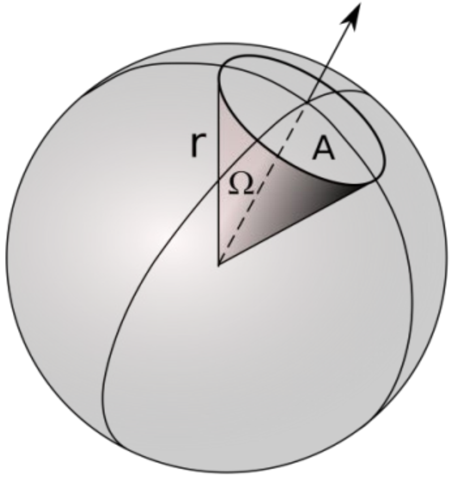


$$\boxed{I_x = N_A \, \sigma_A \, \omega_A \, \Omega \, \varepsilon \, N_e}$$

I_x	number of X-ray photons in a characteristic peak of species A
N	number of atoms per unit volume
σ	ionization cross section (Casnati et al., 1982, Bote et al., 2009)
ω	fluorescence yield (Hubbell et al., 1994, Krause, 1979)
Ω	solid angle / geometrical collection efficiency
ε	detection quantum efficiency (e.g. window: SLEW or no window or other)
N_e	number of incident electrons

+ absorption, fluorescence, other effects…

What is the Solid Angle for X-ray Collection?

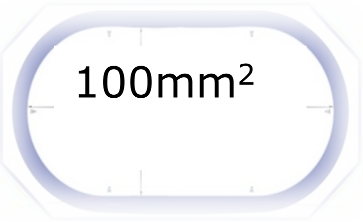


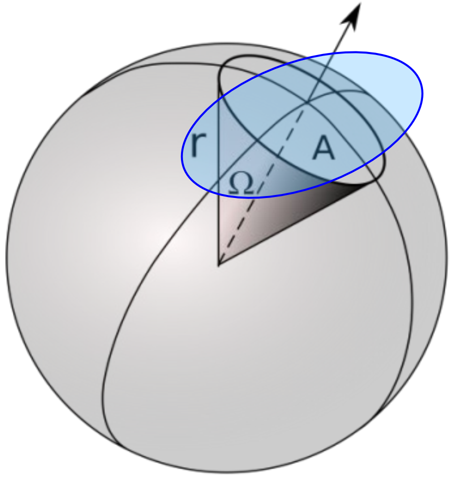
wikipedia

$$\Omega = A / (r)^2$$

Geometrical Concept,
Do not mix with ε, the detection quantum efficiency!

What is the Solid Angle for X-ray Collection?





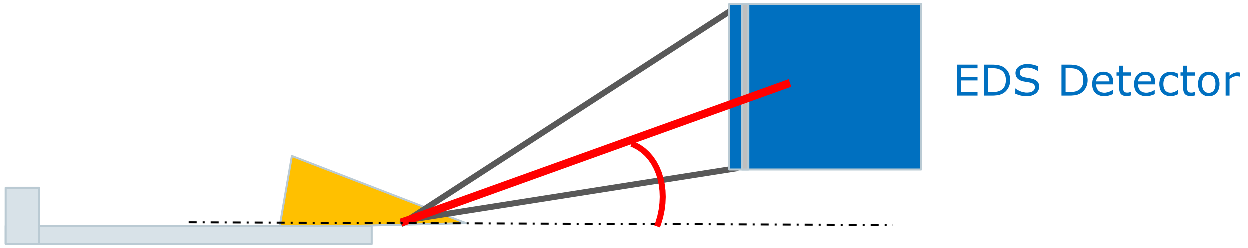
wikipedia

?

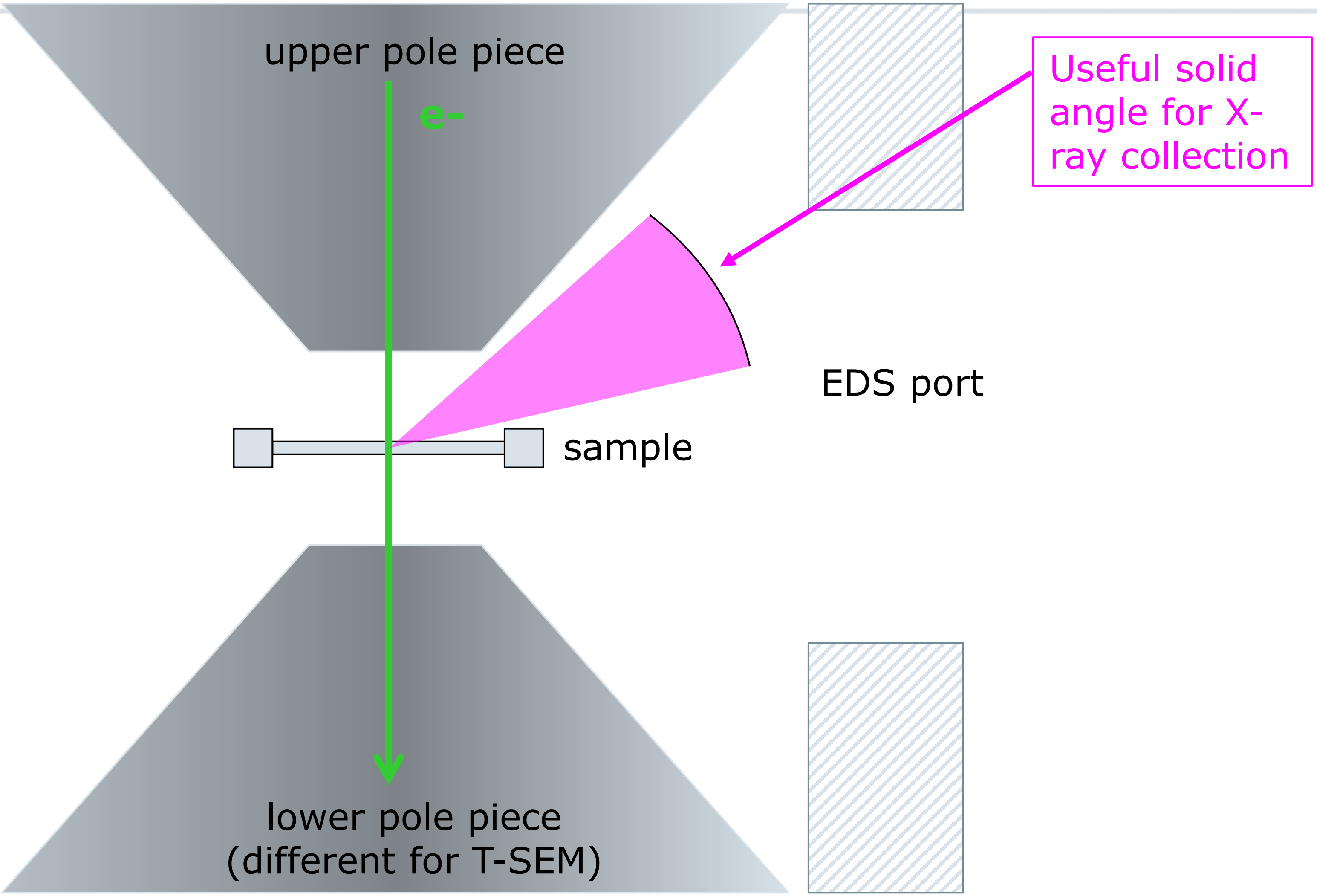
$$\boxed{\Omega = A / (r)^2}$$

Geometrical Concept,
Do not mix with ε, the detection quantum efficiency!

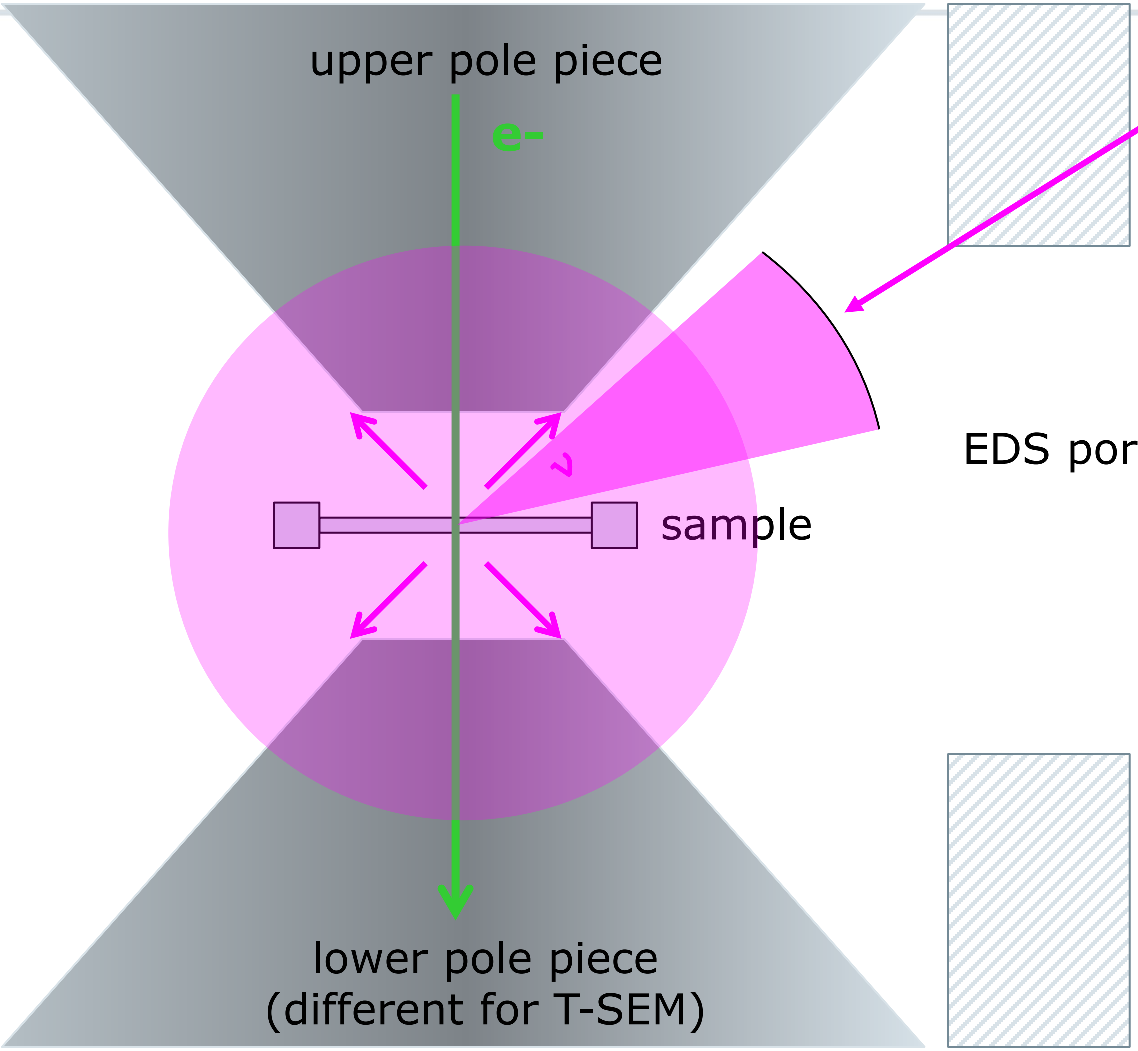
What determines **the solid angle** and … the **take-off angle** ?



Geometric Constraints

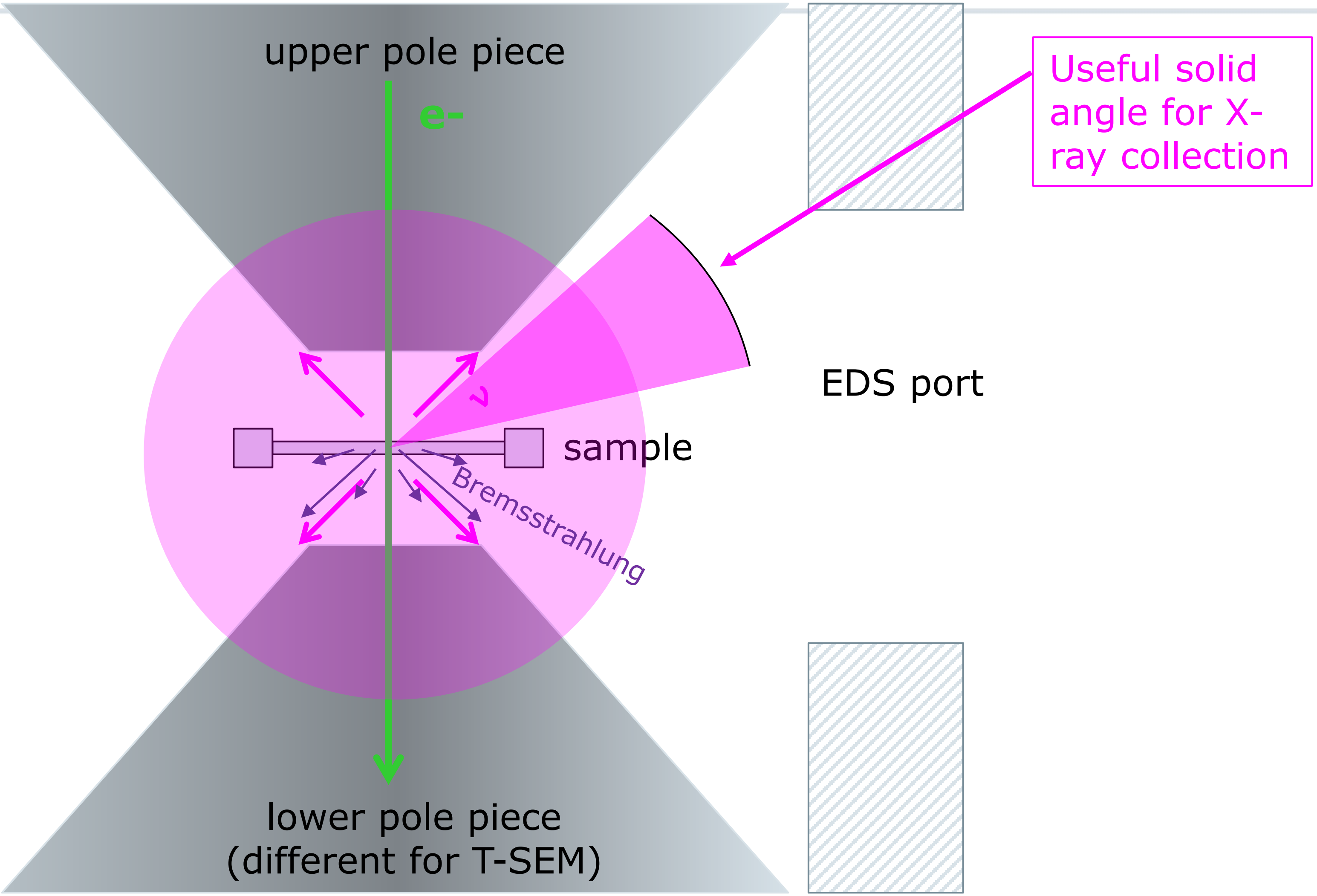


Geometric Constraints

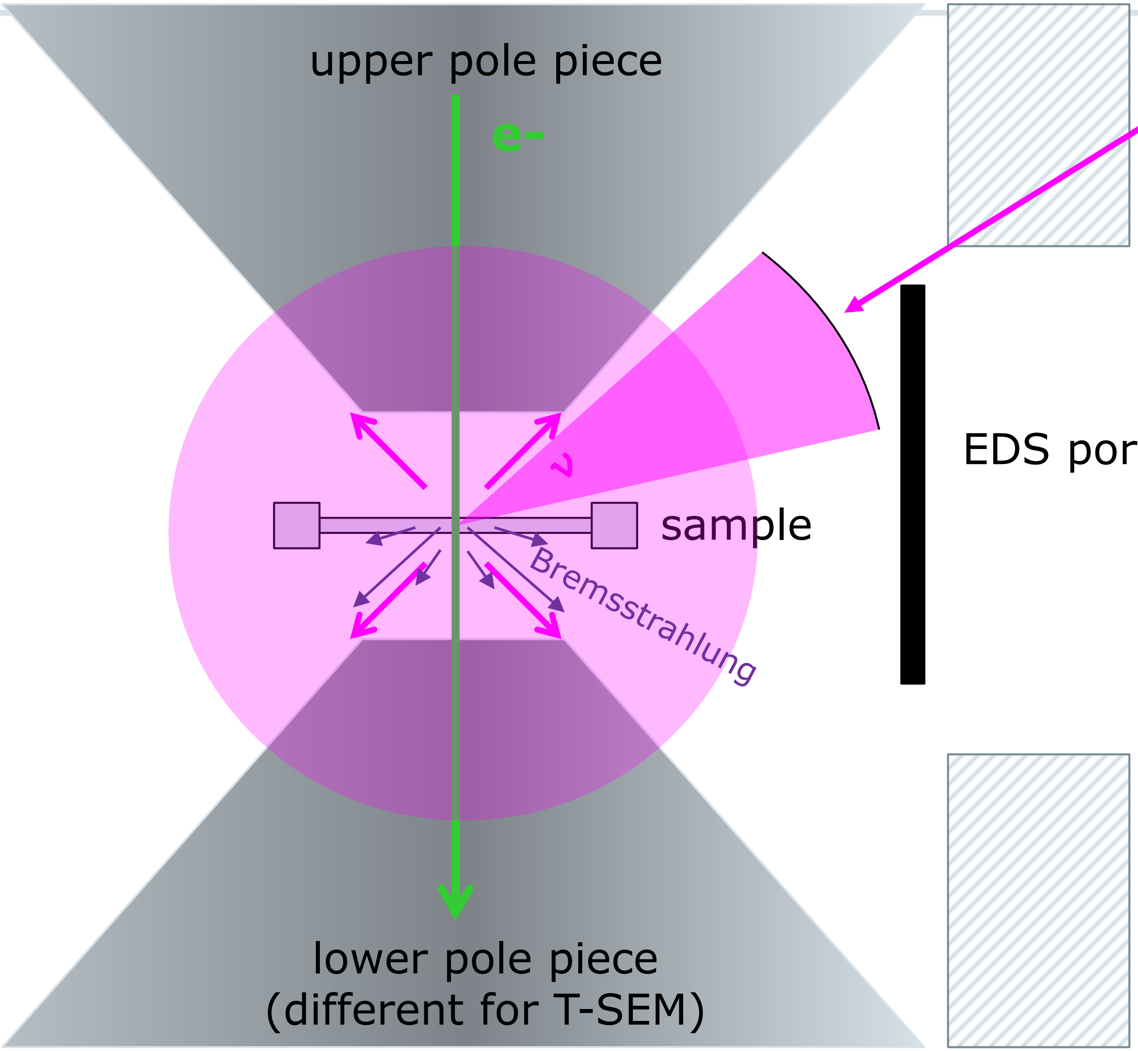


Useful solid angle for X-ray collection

Geometric Constraints



Geometric Constraints



upper pole piece

e-

λ

sample

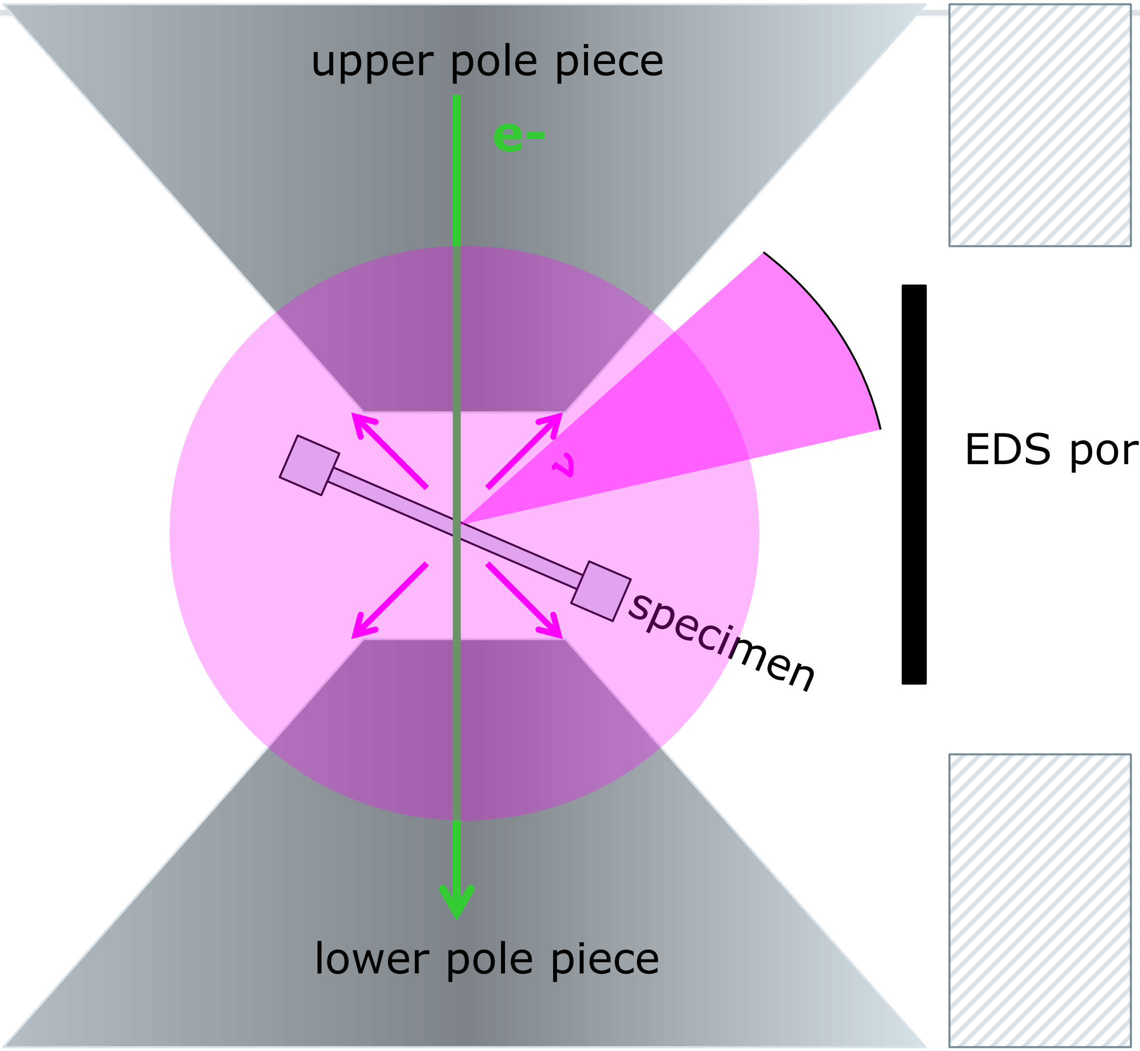
Bremsstrahlung

EDS port

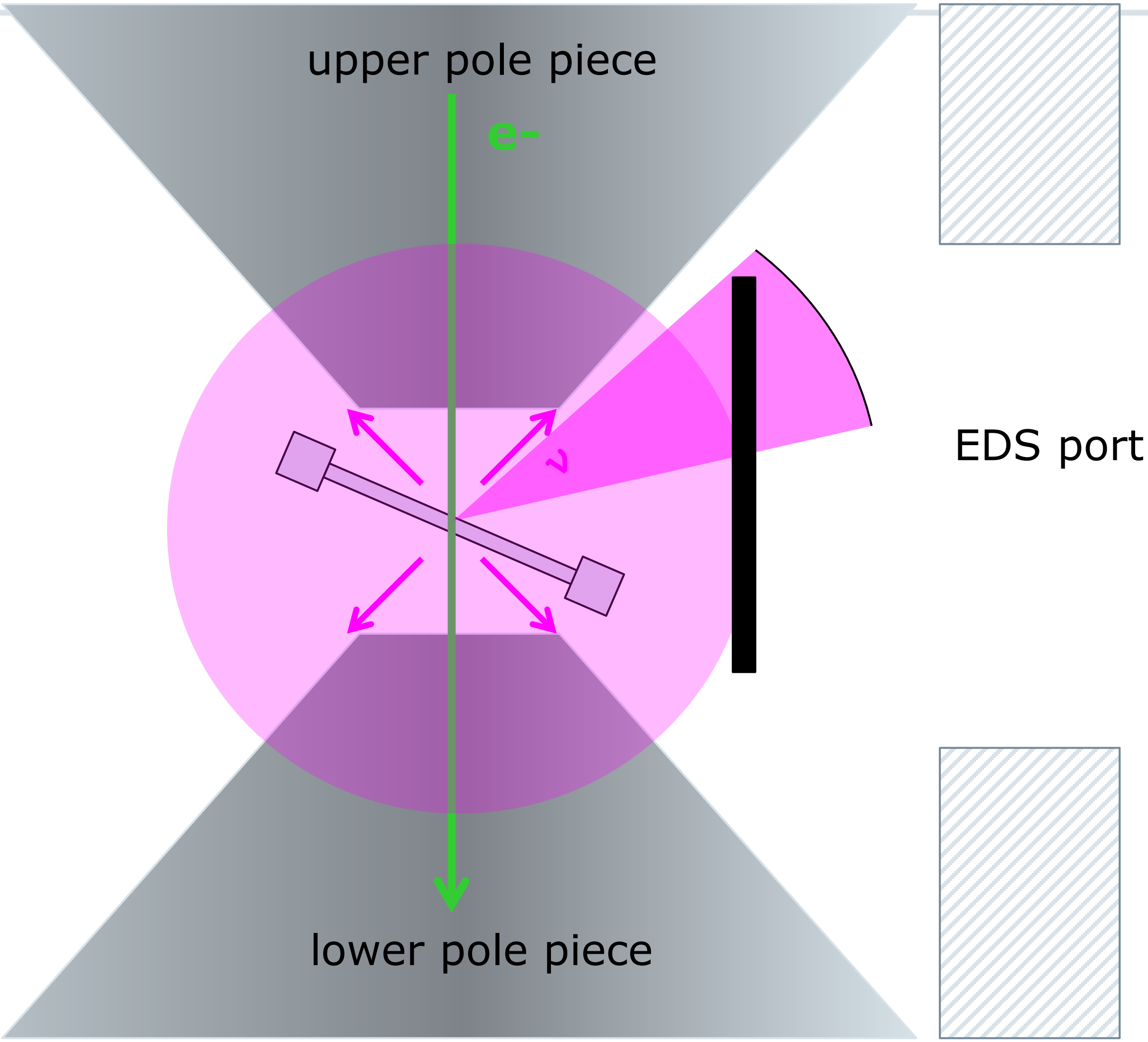
lower pole piece
(different for T-SEM)

Useful solid angle for X-ray collection

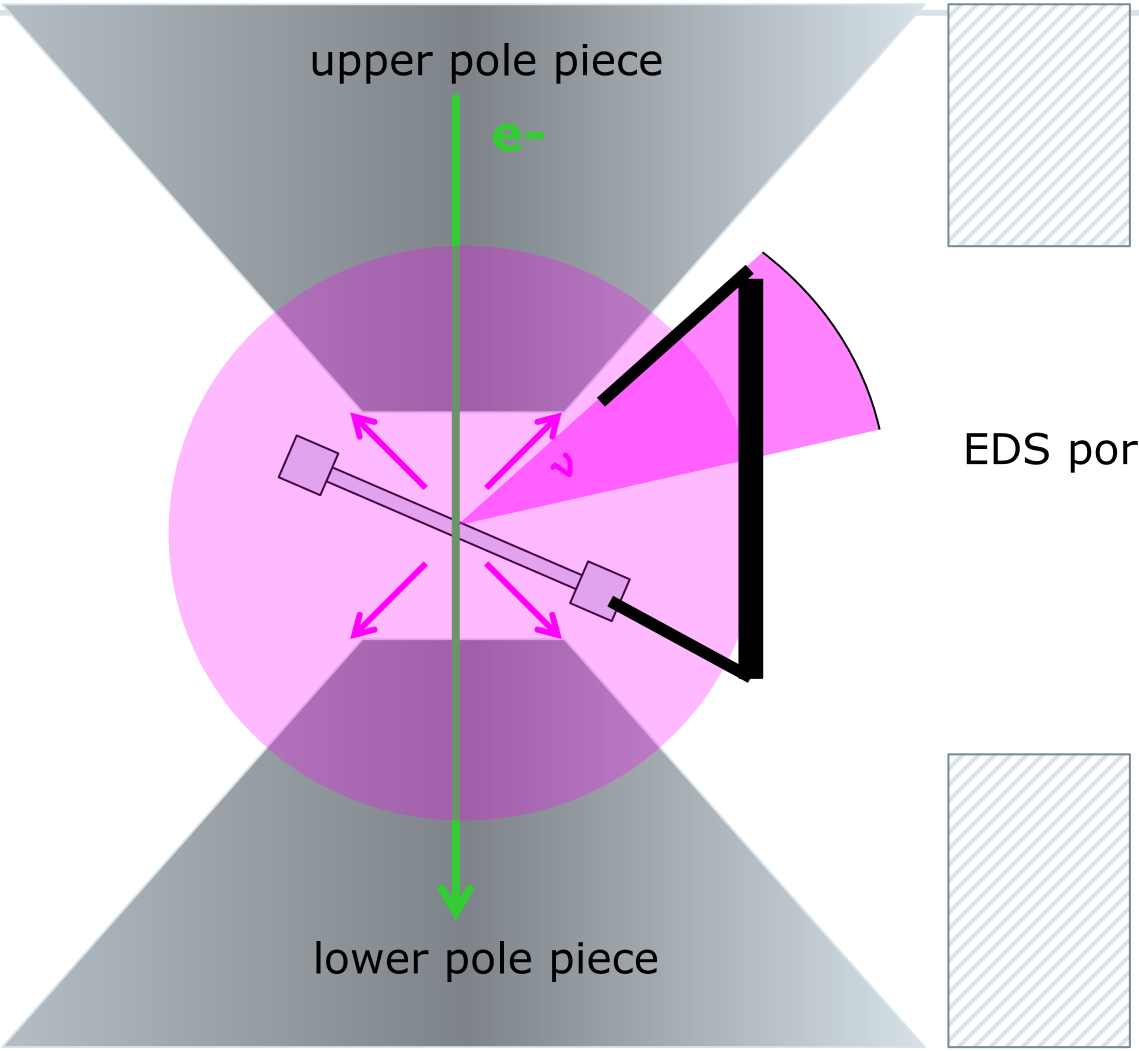
Geometric Constraints



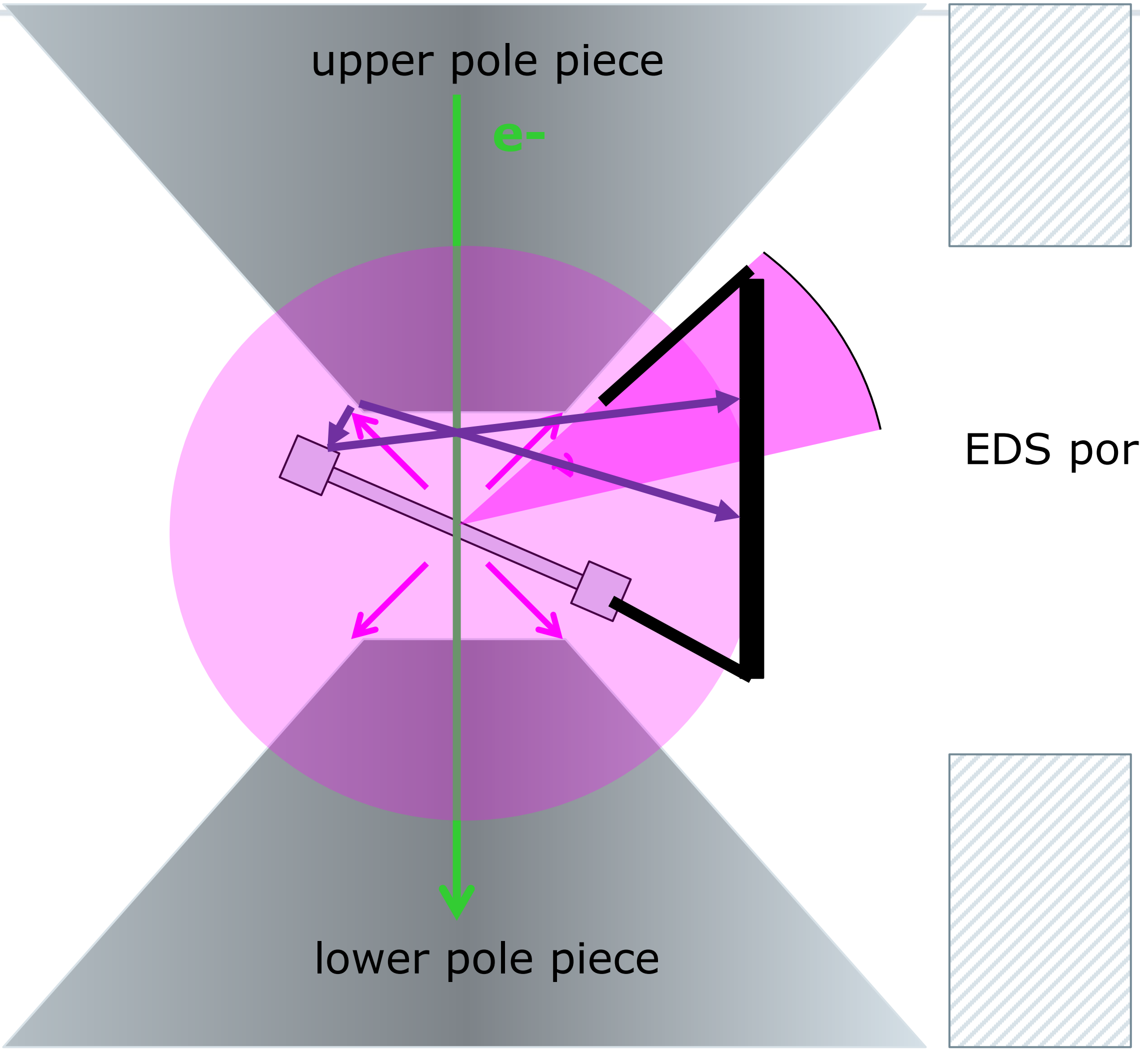
Geometric Constraints



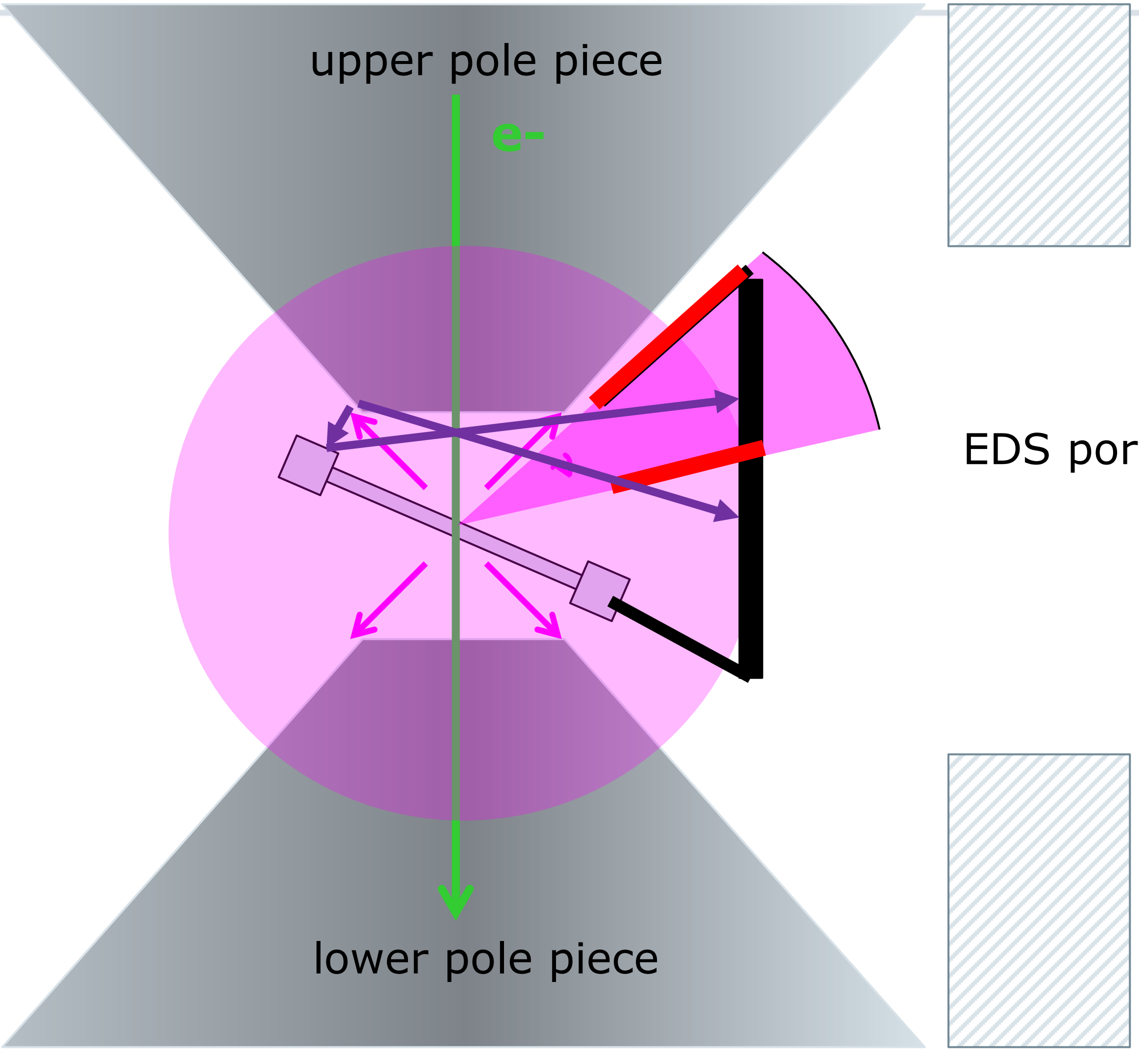
Geometric Constraints



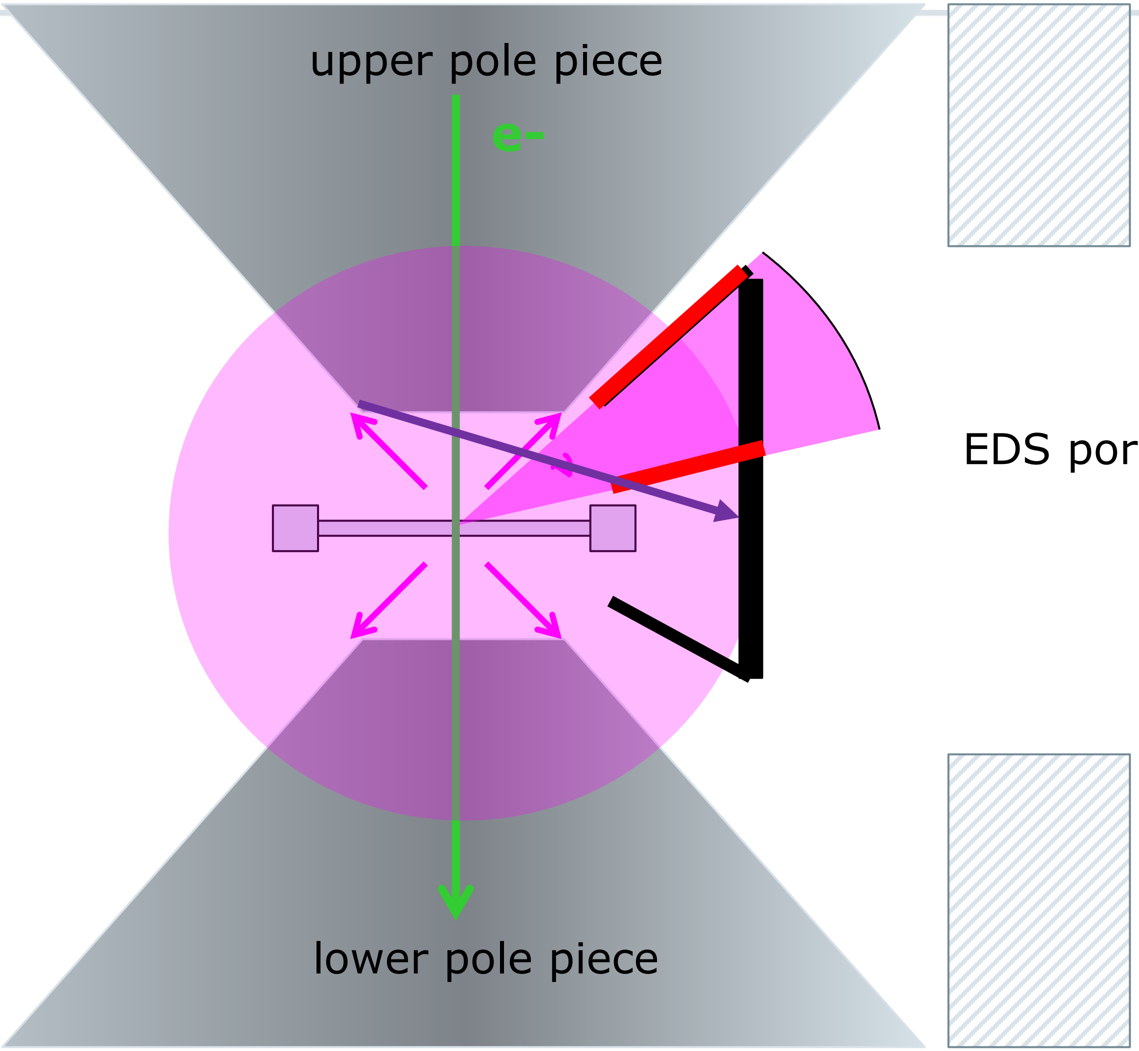
Geometric Constraints



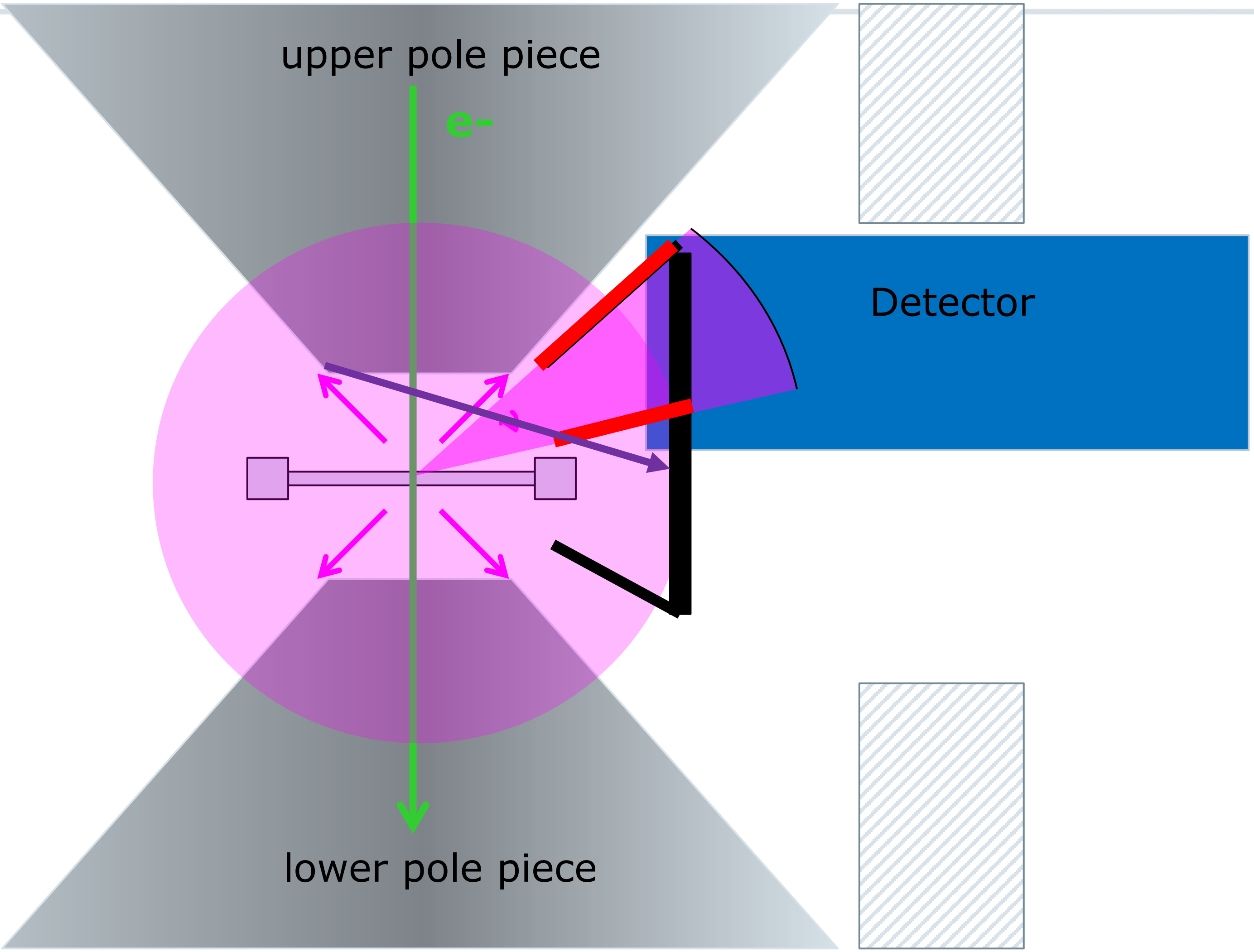
Geometric Constraints



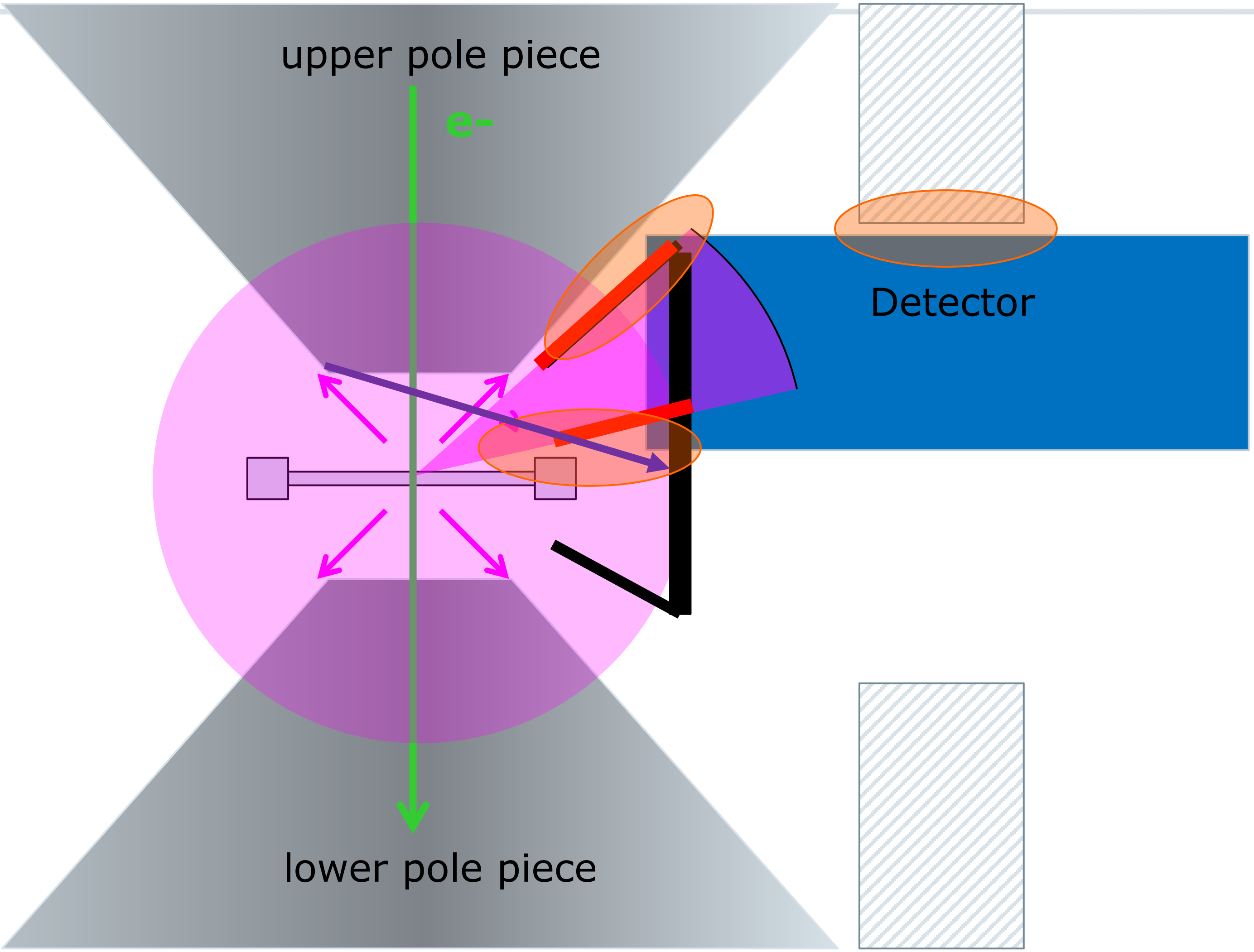
Geometric Constraints



Geometric Constraints

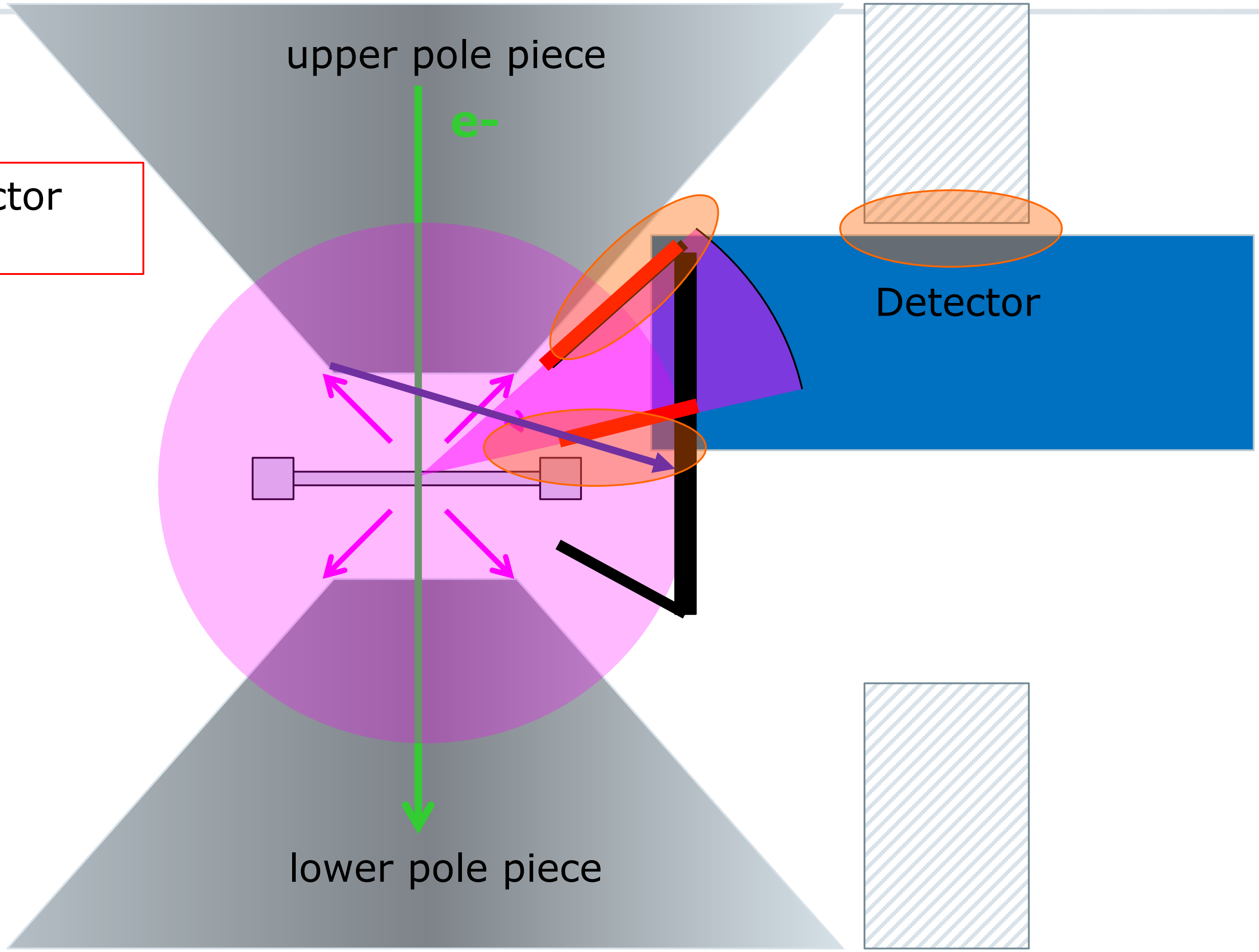


Geometric Constraints



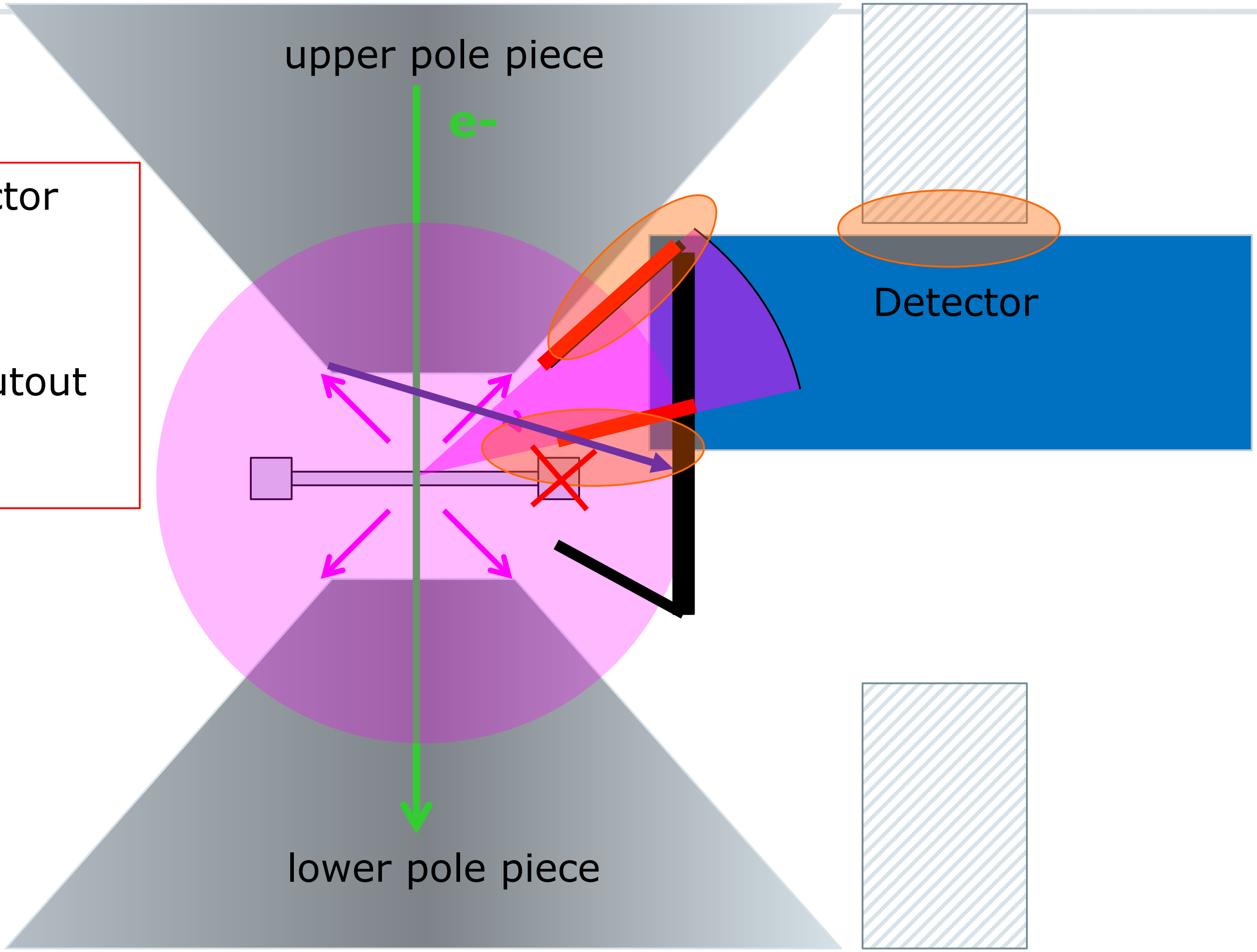
Geometric Constraints

- Optimize detector position

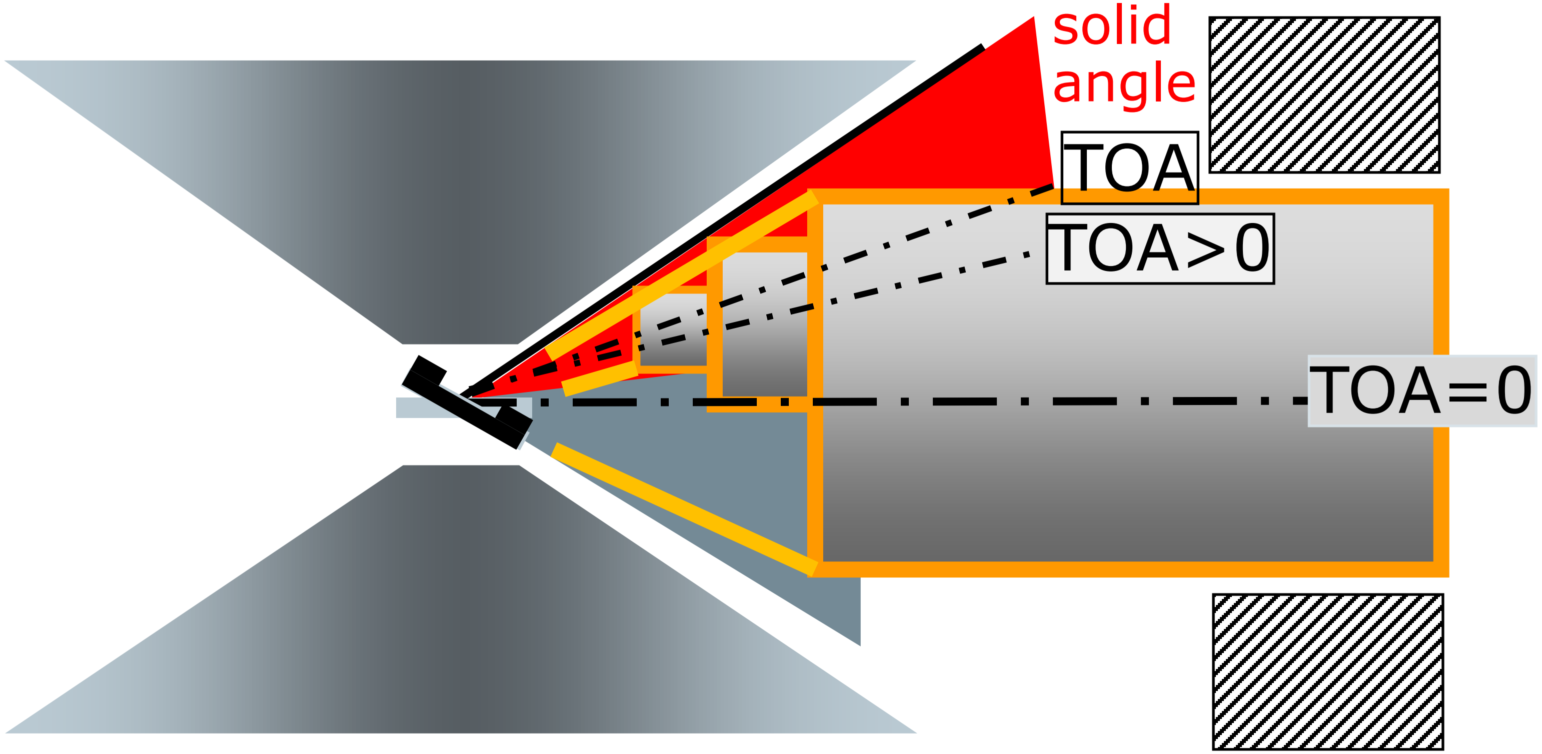


Geometric Constraints

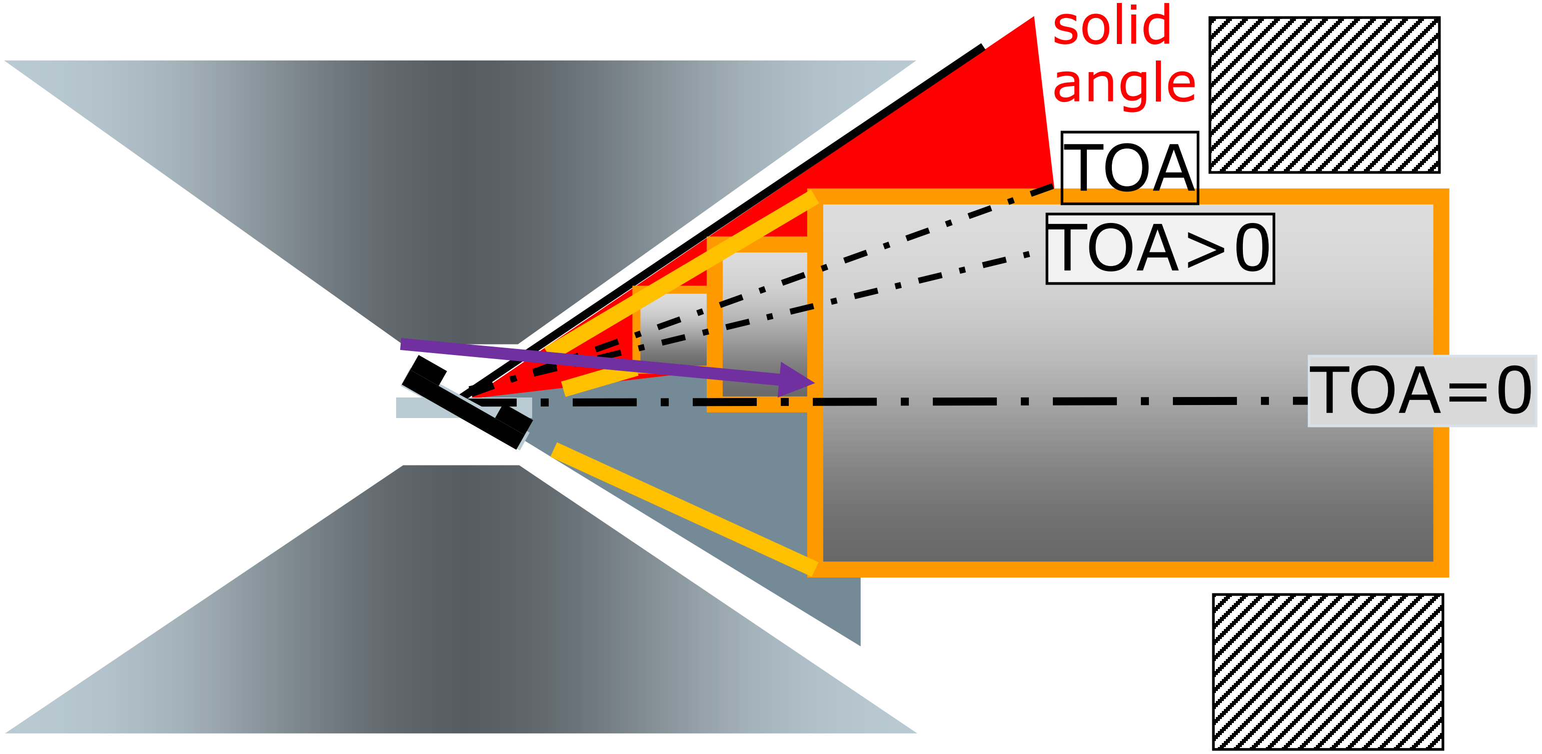
- Optimize detector position
- Use specimen holders with cutout towards EDS detector



Geometric Constraints (TEM);
Solid and take-off angle!!

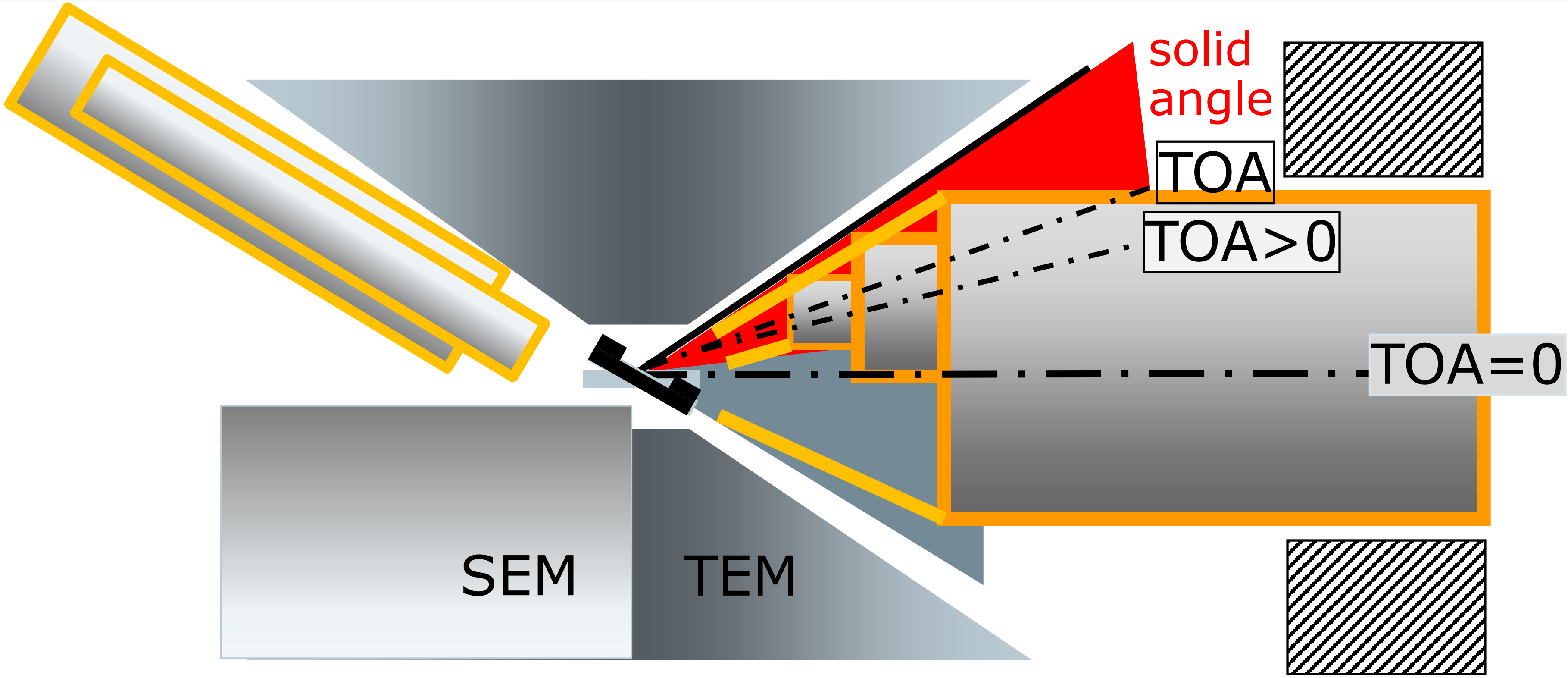


Geometric Constraints (SEM and TEM); Solid and take-off angle!!

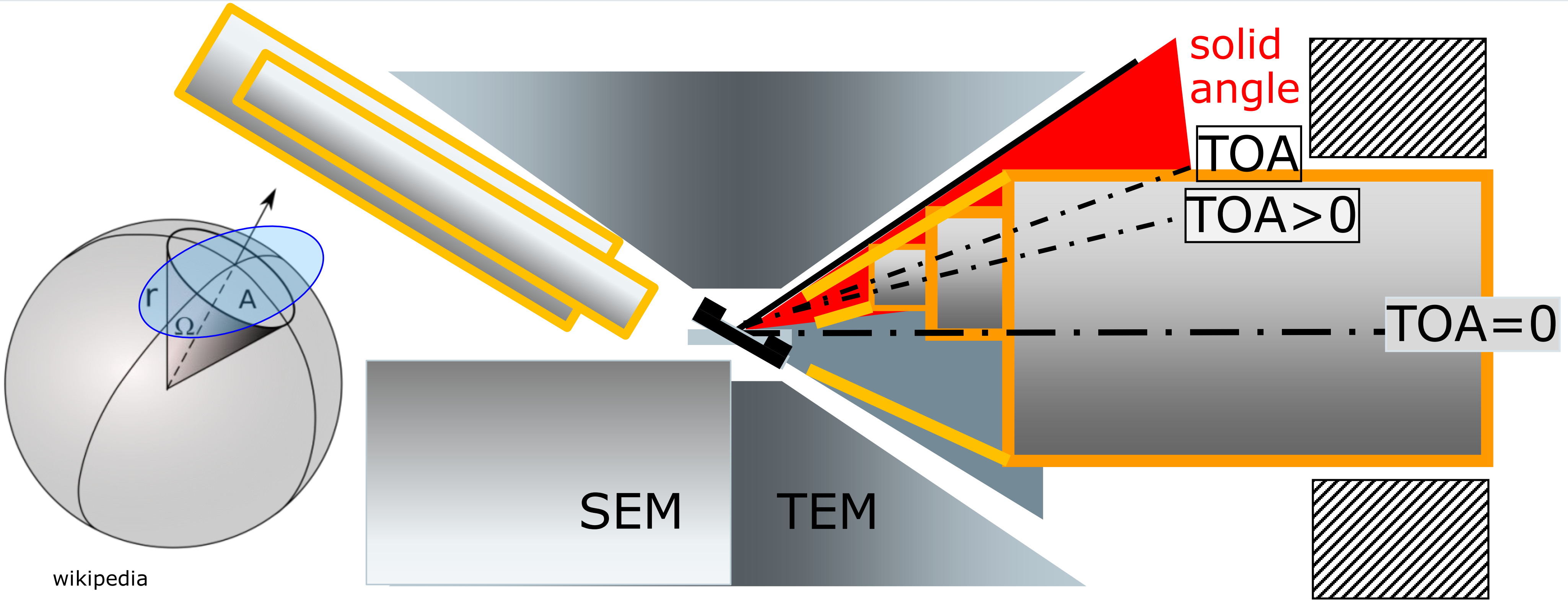


Inverse solid angle! … how much of the surroundings do we see?
A small collimator opening is better to avoid system peaks.

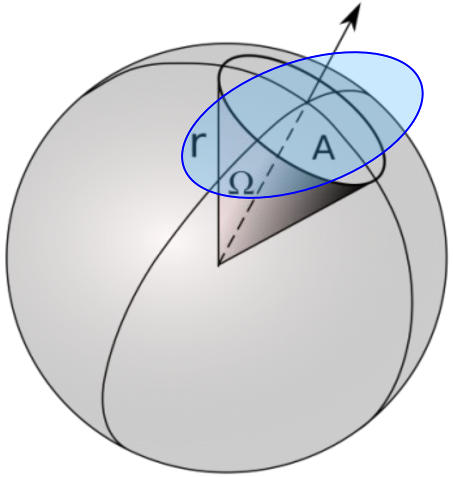
Geometric Constraints (SEM and TEM); Solid and take-off angle!!



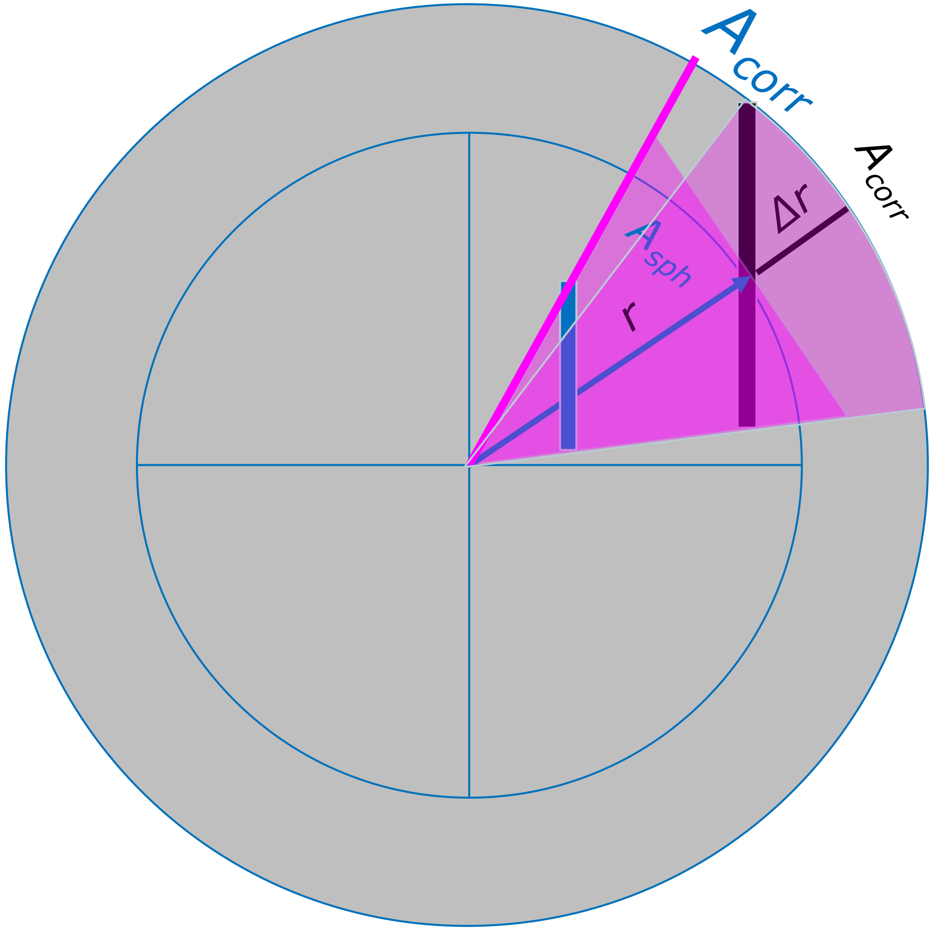
Geometric Constraints (SEM and TEM); Solid and take-off angle!!



Solid Angle Correction for Flat SDD in 2D



wikipedia



The solid angle of $A_{D\_Flat\_vertical}$

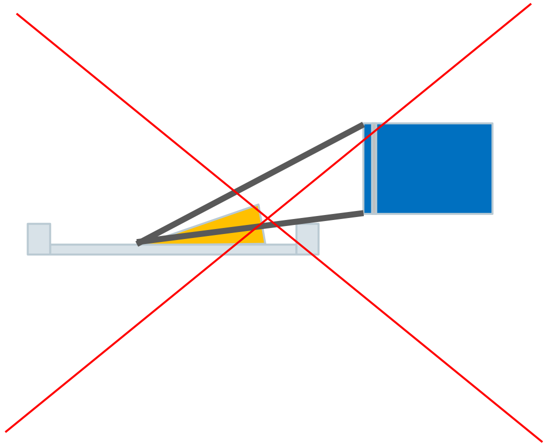
$\boldsymbol{\Omega = A_{corr}}$ / $(r+\Delta r)^2$

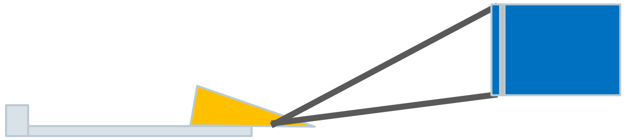
Smaller or oval detector closer to specimen can enable

- Larger A_{corr}
- ➢ Larger solid angle

A large take-off angle above the sample is needed to avoid shadowing and stray radiation > use small or oval area! …
If it fits.

Specimen Mounting: Side View





- Thinnest part of specimen must point towards EDS detector
- The specimen holder must have a cutout towards EDS detector
- Make sure that the path for X-rays is not obstructed by specimens itself or grid parts or holder
- Beware: most holders are turned 180° during insertion

Specimen Mounting: Top View along Column

Detector

Image source: www

Specimen Mounting: Top View along Column

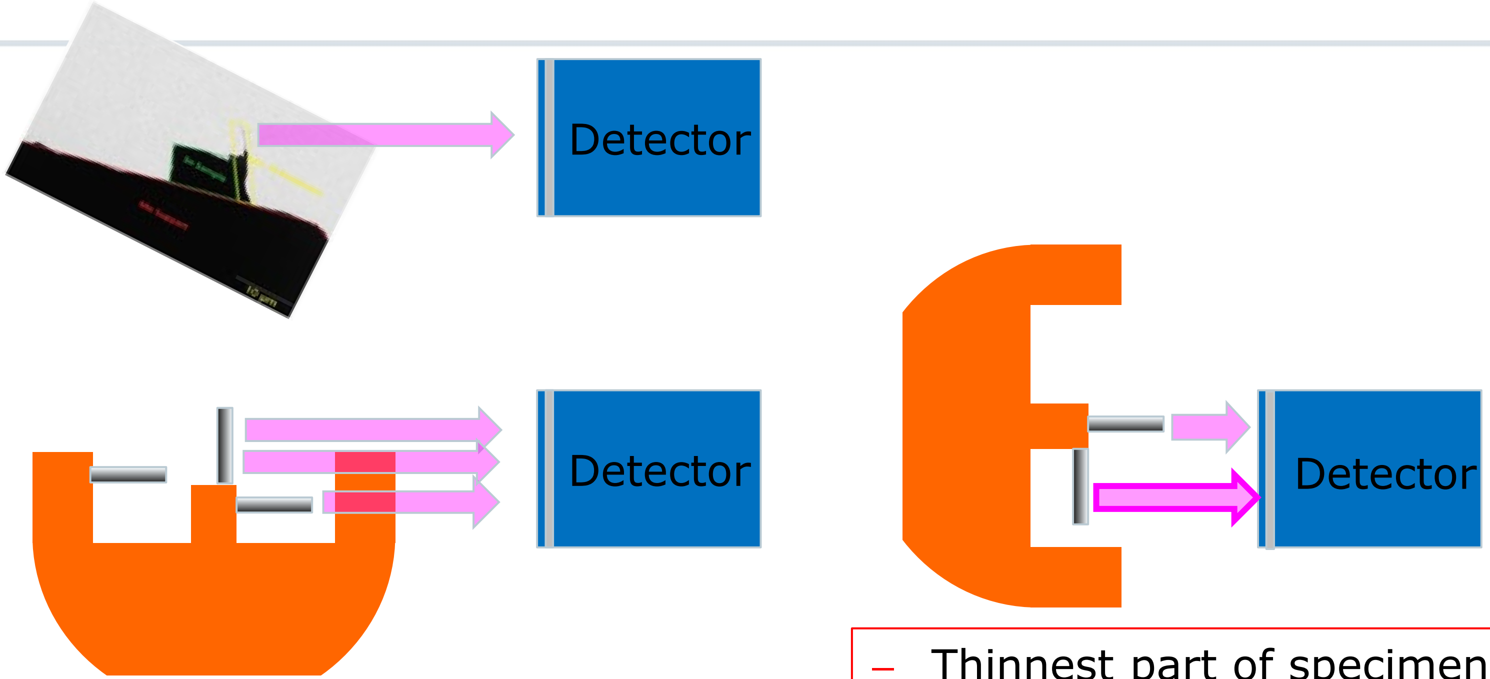
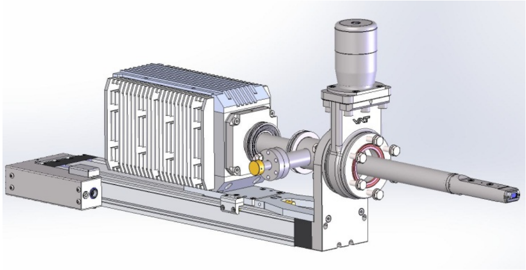



Image source: www

- Thinnest part of specimen must point towards EDS detector
- Make sure that the path for X-rays is not obstructed
- Consider tilt effects

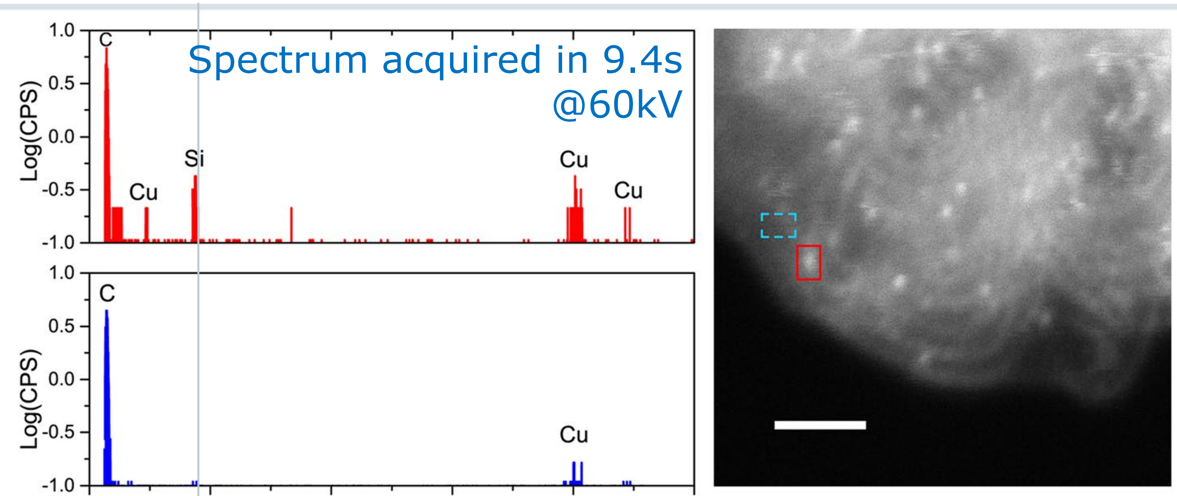
XFlash®6-100 / 6T-100 oval; History

1. Tailored to fit Nion STEM



UHV,
dedicated STEM,
CFEG

Single Atom ID in Nano-Diamonds from Space; Using XFlash®6T-100 oval

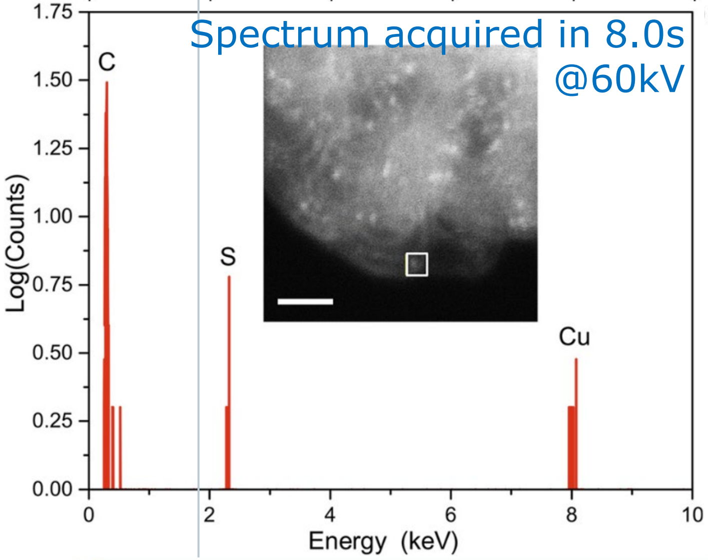


Spectrum acquired in 9.4s @60kV

FIG. 1. High-angle ADF image and EDX spectra on (red solid line box) and off (blue-dashed line box) a Si atom on amorphous carbon.

The scalebar is 1 nm.

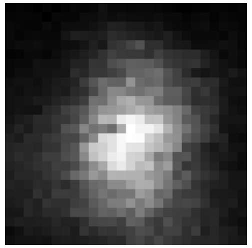
NRL (Naval Research Lab)
Nion UltraSTEM used @ 60 kV, dedicated STEM, CFEG;
$\Omega \sim 0.7$sr, TOA > 13°



Spectrum acquired in 8.0s @60kV

FIG. 2. High-angle ADF image and EDX spectrum of a S atom on amorphous carbon.

The scale bar is 1 nm.



Typical tracking window for EDS of a single atom.

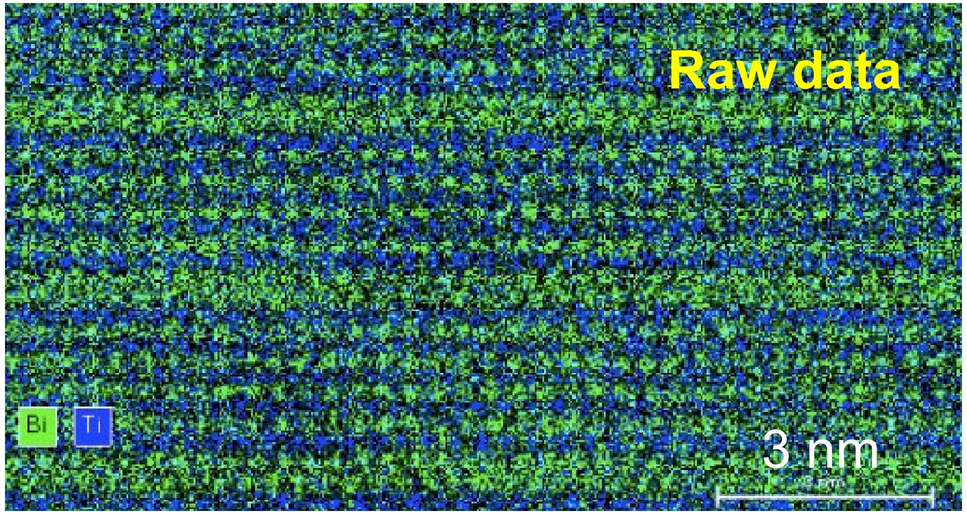
http://creativecommons.org/licenses/by/4.0/;
*R. M. Stroud et al., APL **108**, 163101 (2016) open access*

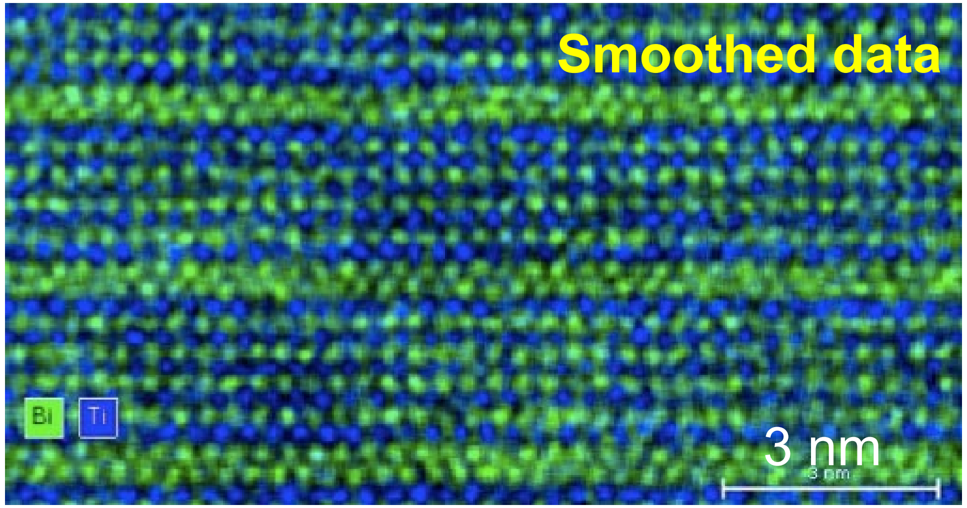
*T. C. Lovejoy et al., APL **100**, 154101 (2012): 30mm^2, 0.1sr*

R. M. Stroud: et al., APL 108, 163101 (2016), http://dx.doi.org/10.1063/1.4947002 open access

Element Mapping of Multiferroic $Bi_6Ti_xFe_yMn_zO_{18}$ Using XFlash®6T-100 oval

TCD (Trinity College Dublin)
Nion UltraSTEM200XE 200 kV,
Dedicated STEM, CFEG;
$\Omega \sim 0.7$sr, TOA > 13°





432x225 pixels,
4.1 msec/pix => 400 sec for map.
No drift correction.
Bi = green, Ti = blue.

courtesy Lynette Keeney, Clive Downing and Valeria Nicolosi. TCD, Ireland.

Specimen:
$Bi_6Ti_xFe_yMn_zO_{18}$
See:
"Direct atomic scale determination of magnetic ion partition in a room temperature multiferroic material" Scientific Reports ***7****, Article number: 1737 (2017) open access*

L. Keeney at al.: Scientific Reports **7**, Article number: 1737 (2017) open access

XFlash®6-100 / 6T-100 oval; History

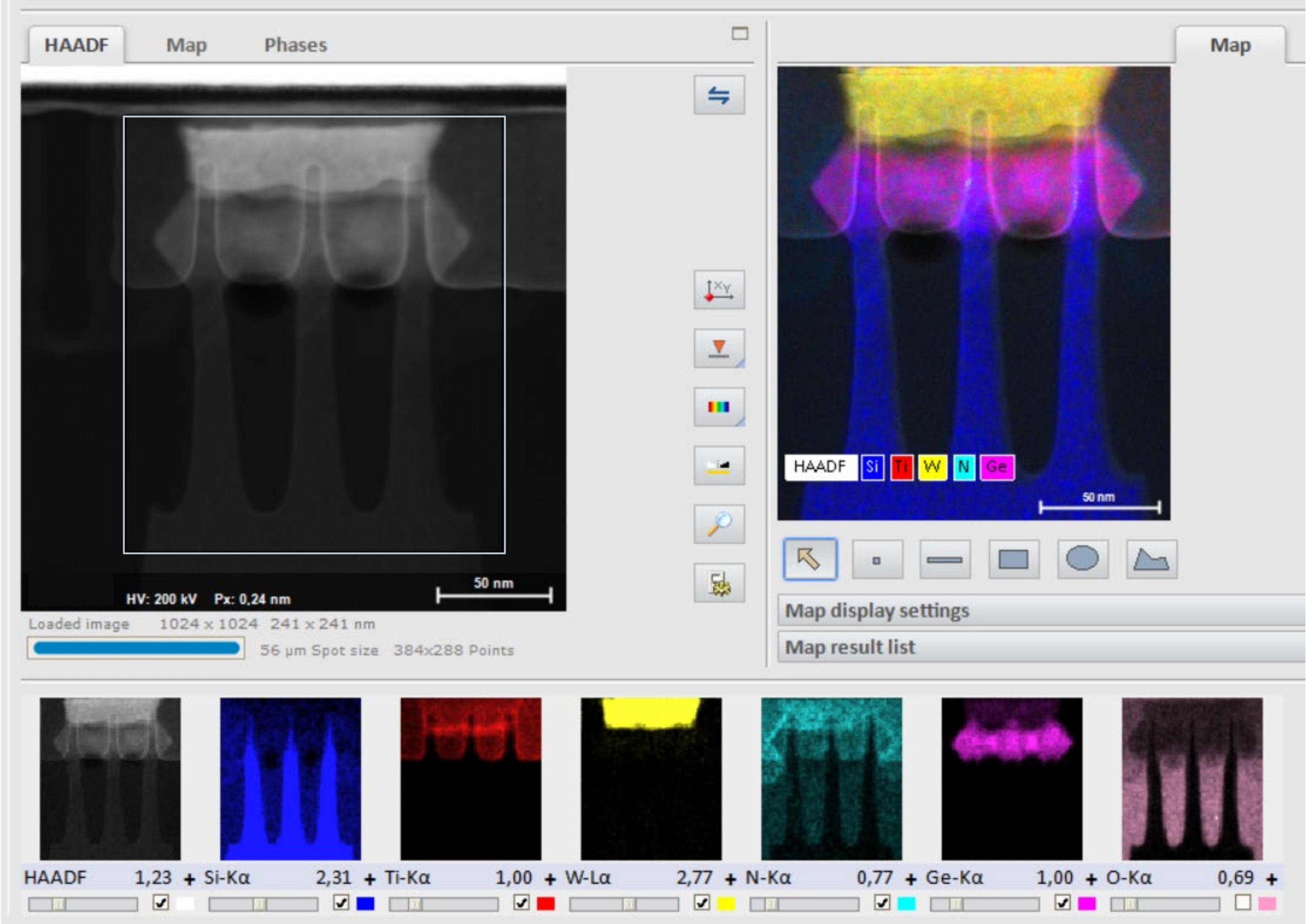
3. Now launching for additional and retrofit adaptions on all suitable TEM/STEM and SEM/T-SEM types e.g.
Jeol (F200),
TFS (Tecnai and Titan),
Hitachi … Tescan … Zeiss

XFlash®6T-100 oval on conventional aberration corrected STEM with FEG

Quantitative Element Mapping of Semiconductor Nanostructures

TFS Titan 80-300 with
Bruker XFlash®6T-100-oval
EDS detector;
Ω > 0.4sr, TOA ~ 12°

Raw data;
Acquisition Time: 20 min.



HV: 200 kV Px: 0,24 nm

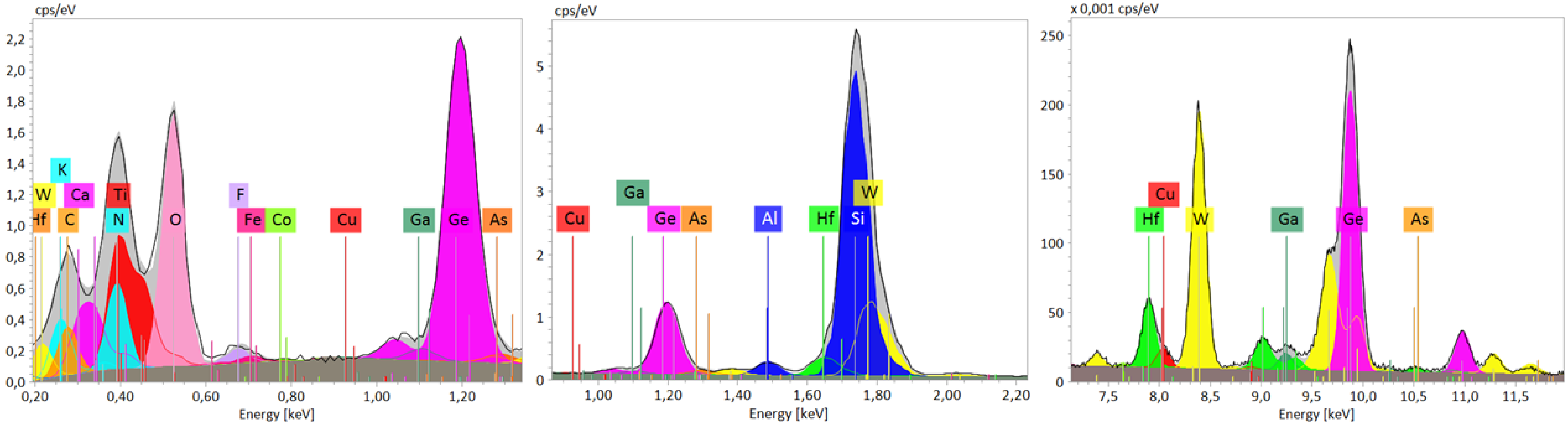
Loaded image 1024 x 1024 241 x 241 nm

56 µm Spot size 384x288 Points

HAADF 1,23 ☑ Si-Kα 2,31 ☑ Ti-Kα 1,00 ☑ W-Lα 2,77 ☑ N-Kα 0,77 ☑ Ge-Kα 1,00 ☑ O-Kα 0,69 ☐

Data Courtesy: ACE

Quantitative Element Mapping of Semiconductor Nanostructures



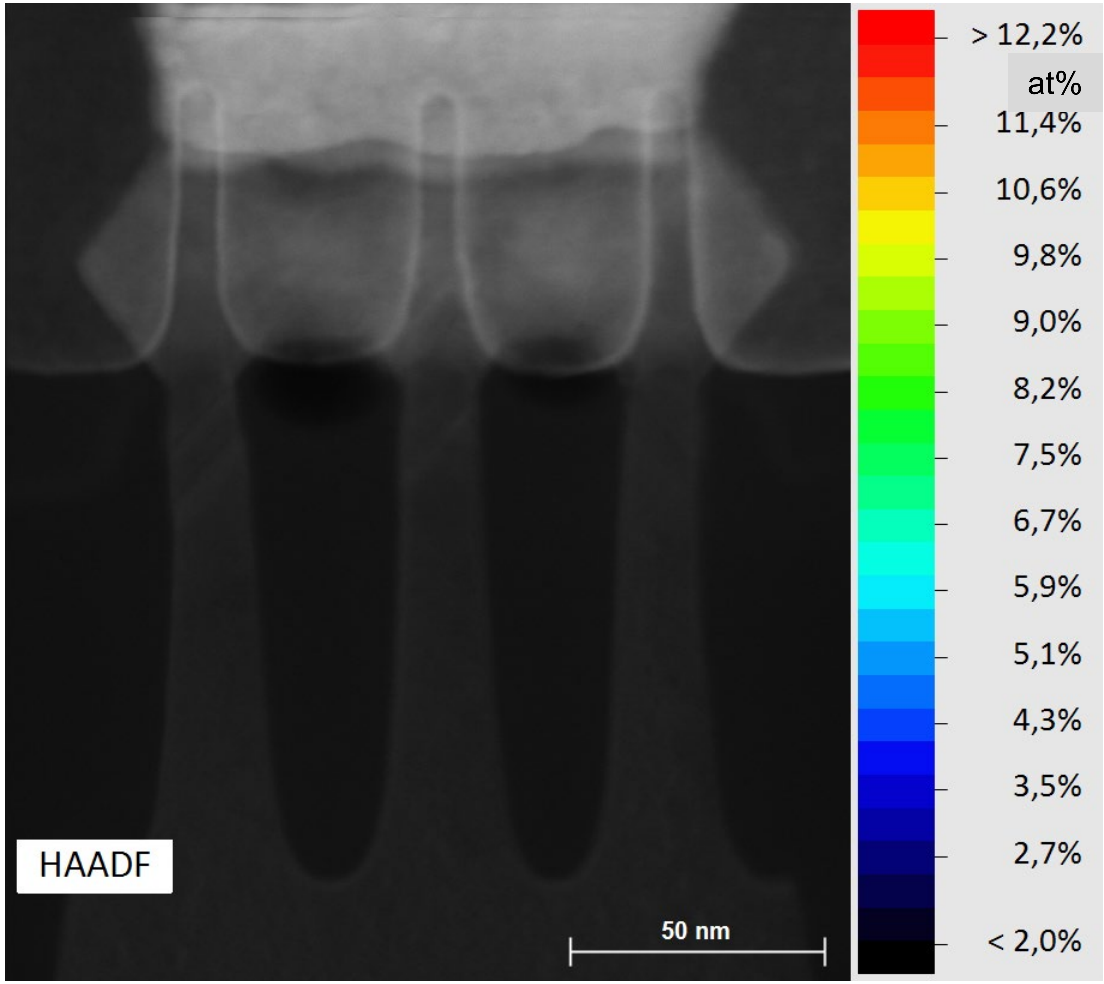
Deconvolution Result

Data Courtesy: ACE

Quantitative Element Mapping of Semiconductor Nanostructures

TFS Titan 80-300 with
Bruker XFlash®6T-100-oval
EDS detector;
Ω > 0.4sr, TOA ~ 12°

Quantified using the
Cliff-Lorimer Method: at%.

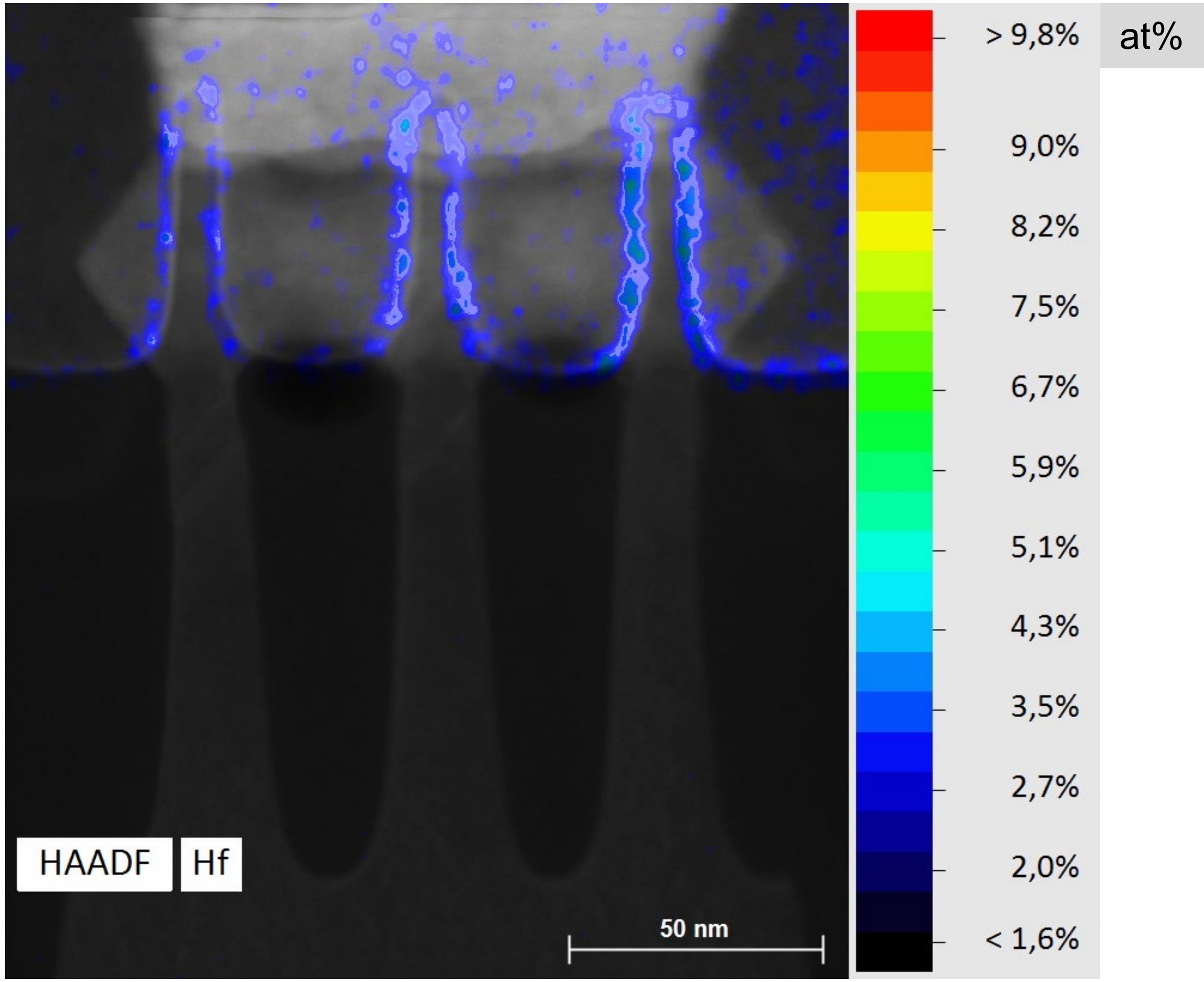


Data Courtesy: ACE

Quantitative Element Mapping of Semiconductor Nanostructures

TFS Titan 80-300 with
Bruker XFlash®6T-100-oval
EDS detector;
Ω > 0.4sr, TOA ~ 12

Quantified using the
Cliff-Lorimer Method: at%.

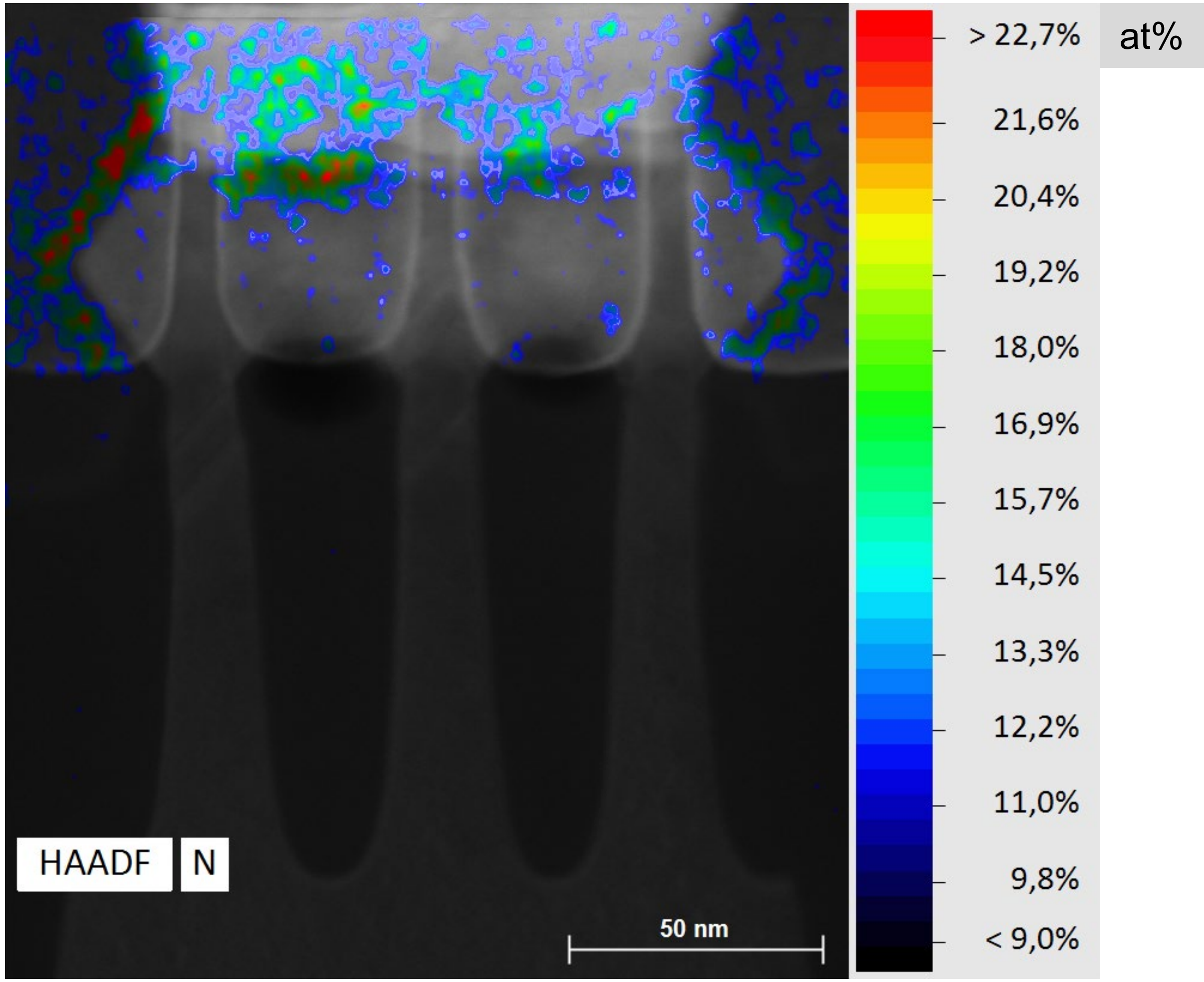


Data Courtesy: ACE

Quantitative Element Mapping of Semiconductor Nanostructures

TFS Titan 80-300 with
Bruker XFlash®6T-100-oval
EDS detector;
Ω > 0.4sr, TOA ~ 12

Quantified using the
Cliff-Lorimer Method: at%.



Data Courtesy: ACE

Quantitative Element Mapping of Semiconductor Nanostructures

TFS Titan 80-300 with
Bruker XFlash®6T-100-oval
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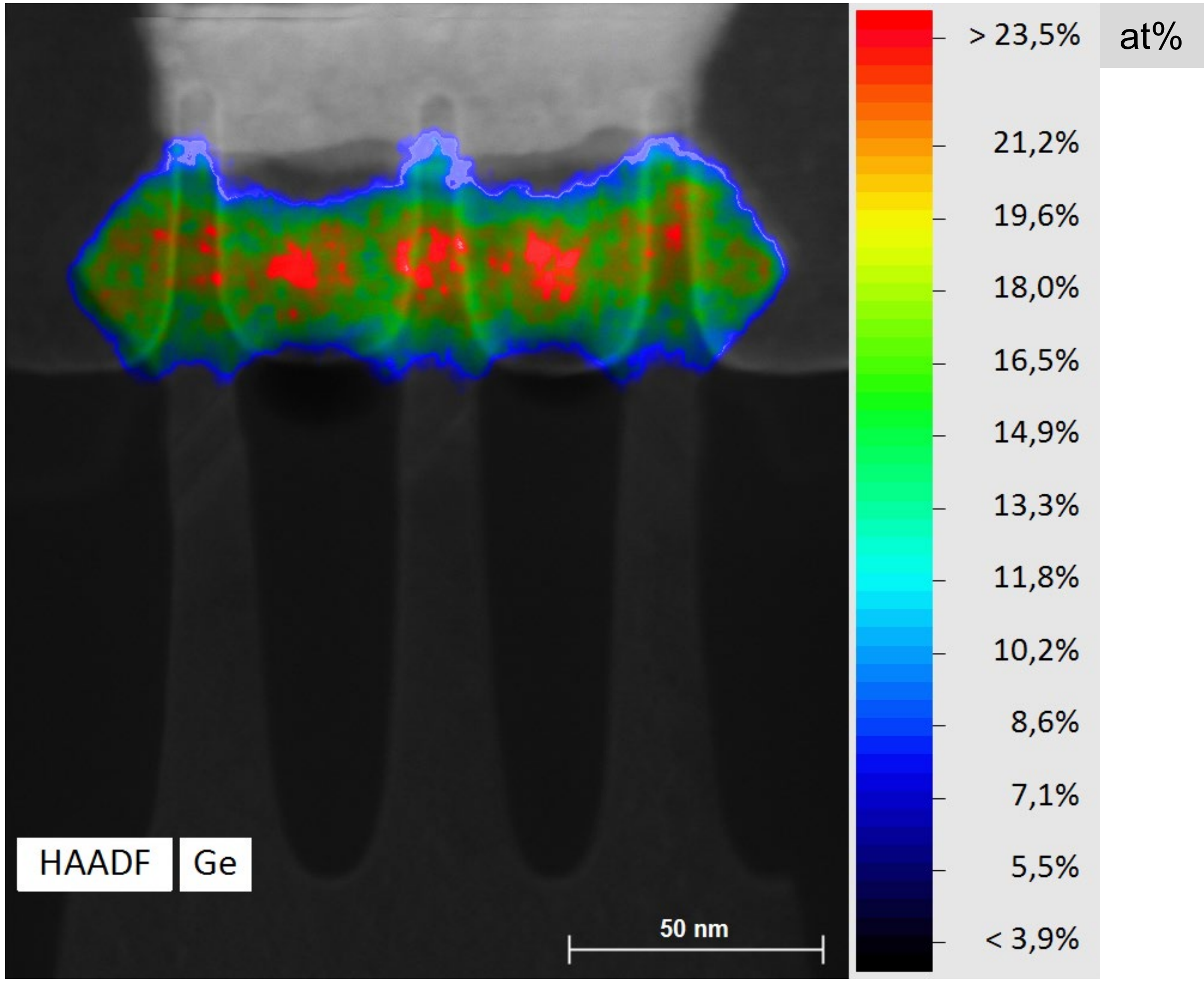
Ω > 0.4sr, TOA ~ 12

Quantified using the
Cliff-Lorimer Method: at%.

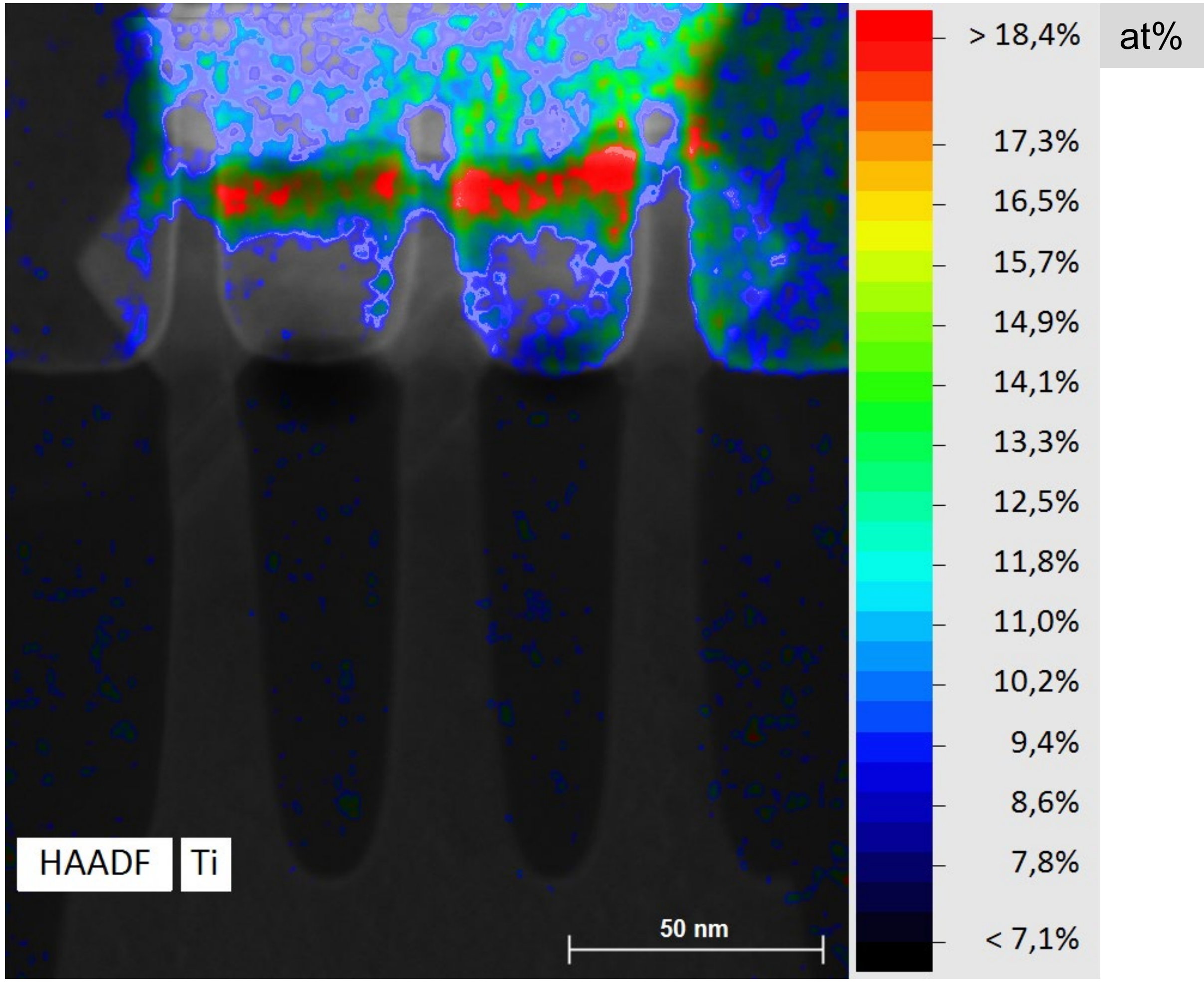


Data Courtesy: ACE

Quantitative Element Mapping of Semiconductor Nanostructures

TFS Titan 80-300 with
Bruker XFlash®6T-100-oval
EDS detector;
Ω > 0.4sr, TOA ~ 12

Quantified using the
Cliff-Lorimer Method: at%.

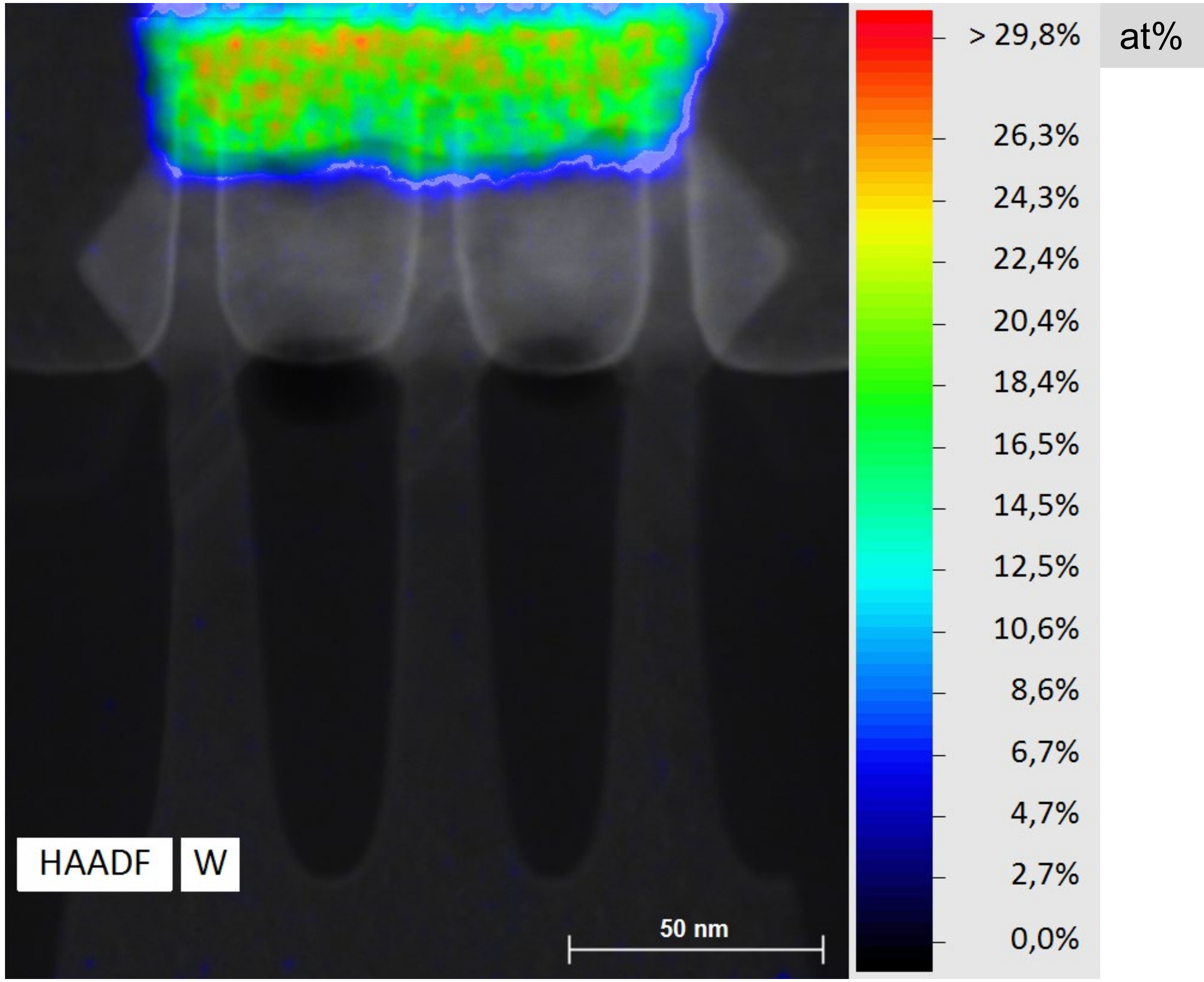


Data Courtesy: ACE

Quantitative Element Mapping of Semiconductor Nanostructures

TFS Titan 80-300 with
Bruker XFlash®6T-100-oval
EDS detector;
Ω > 0.4sr, TOA ~ 12

Quantified using the
Cliff-Lorimer Method: at%.

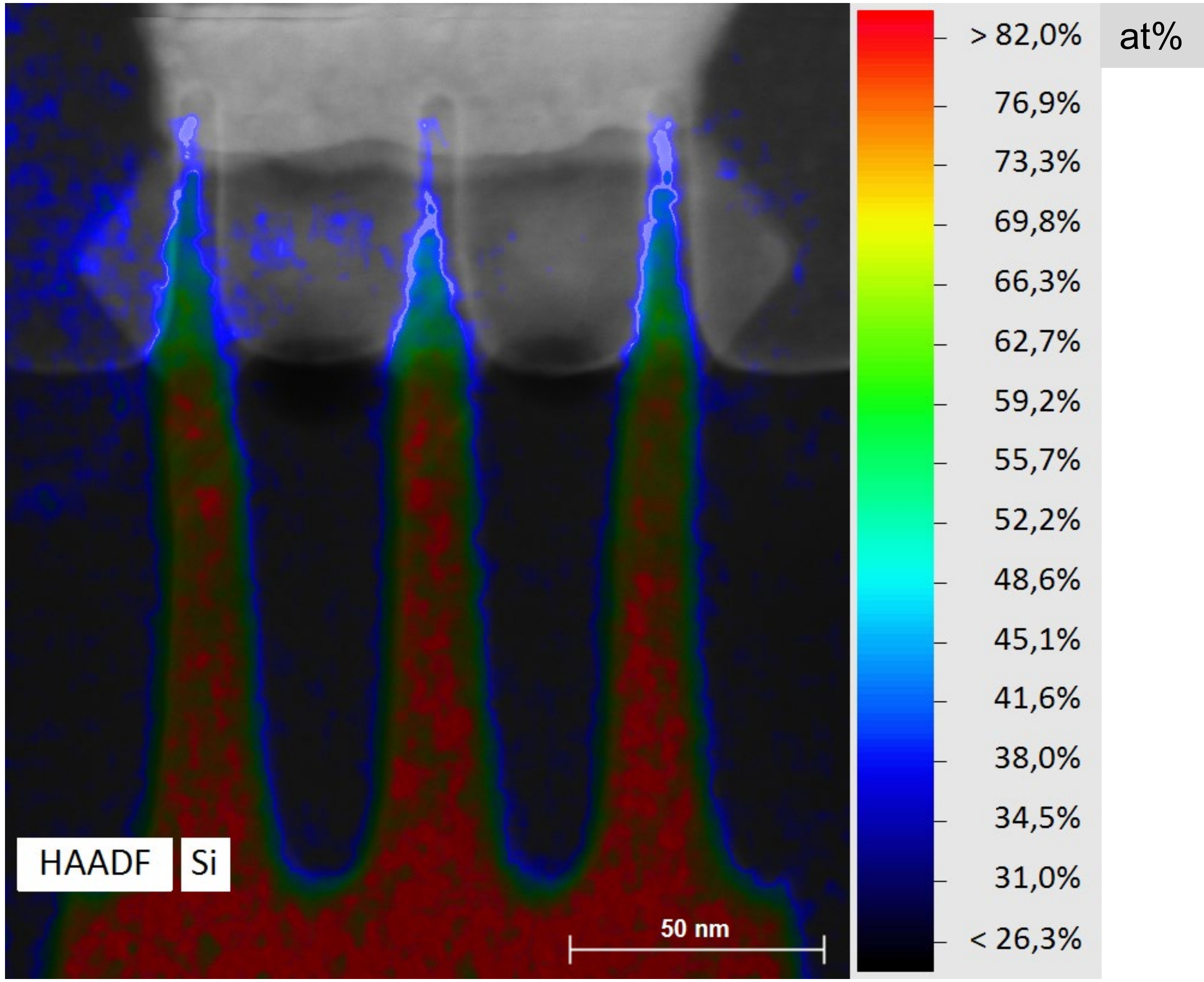


Data Courtesy: ACE

Quantitative Element Mapping of Semiconductor Nanostructures

TFS Titan 80-300 with
Bruker XFlash®6T-100-oval
EDS detector;
Ω > 0.4sr, TOA ~ 12

Quantified using the
Cliff-Lorimer Method: at%.

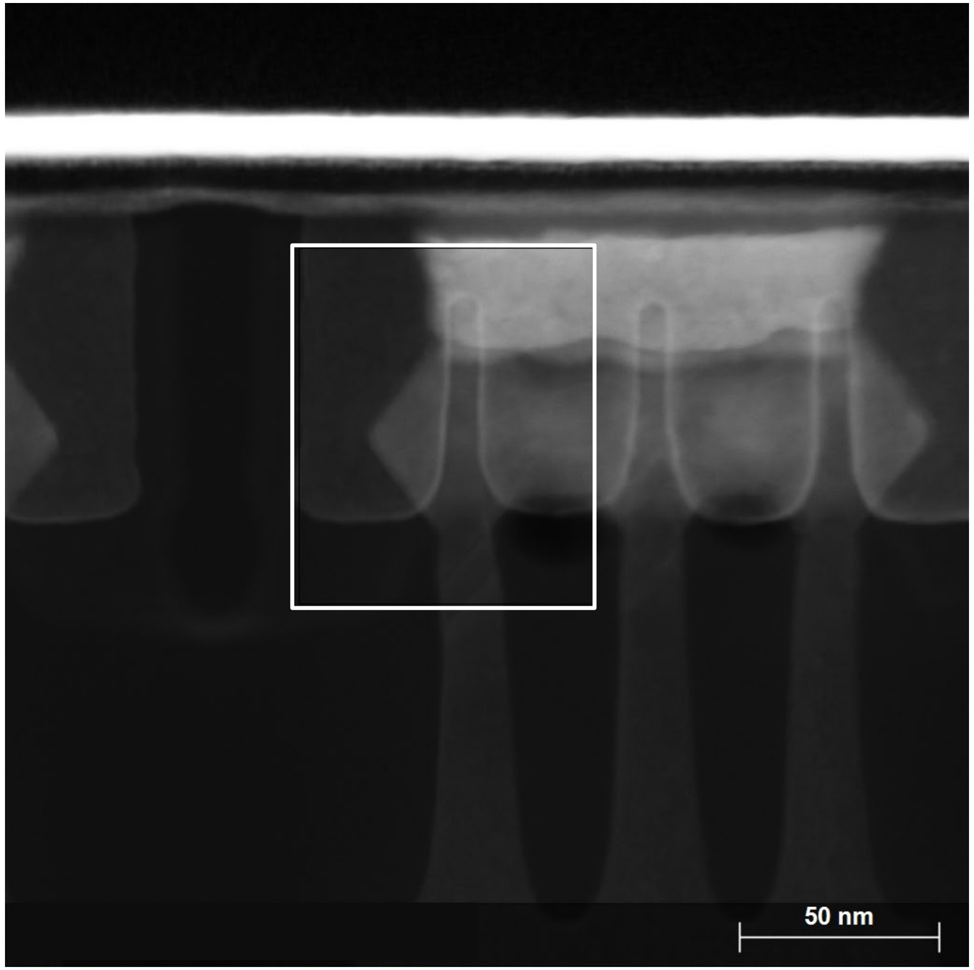


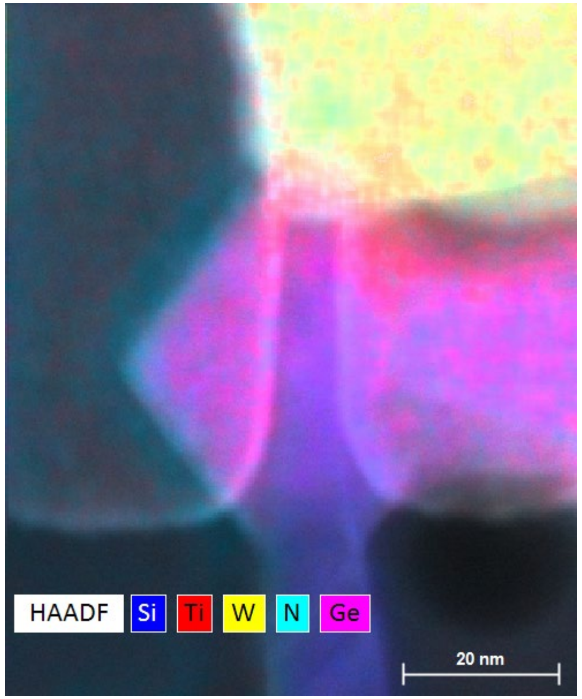
Data Courtesy: ACE

Quantitative Element Mapping of Semiconductor Nanostructures

TFS Titan 80-300 with
Bruker XFlash®6T-100-oval
EDS detector;
$\Omega > 0.4$sr, TOA ~ 12

Raw data;
Acquisition time: 24min.



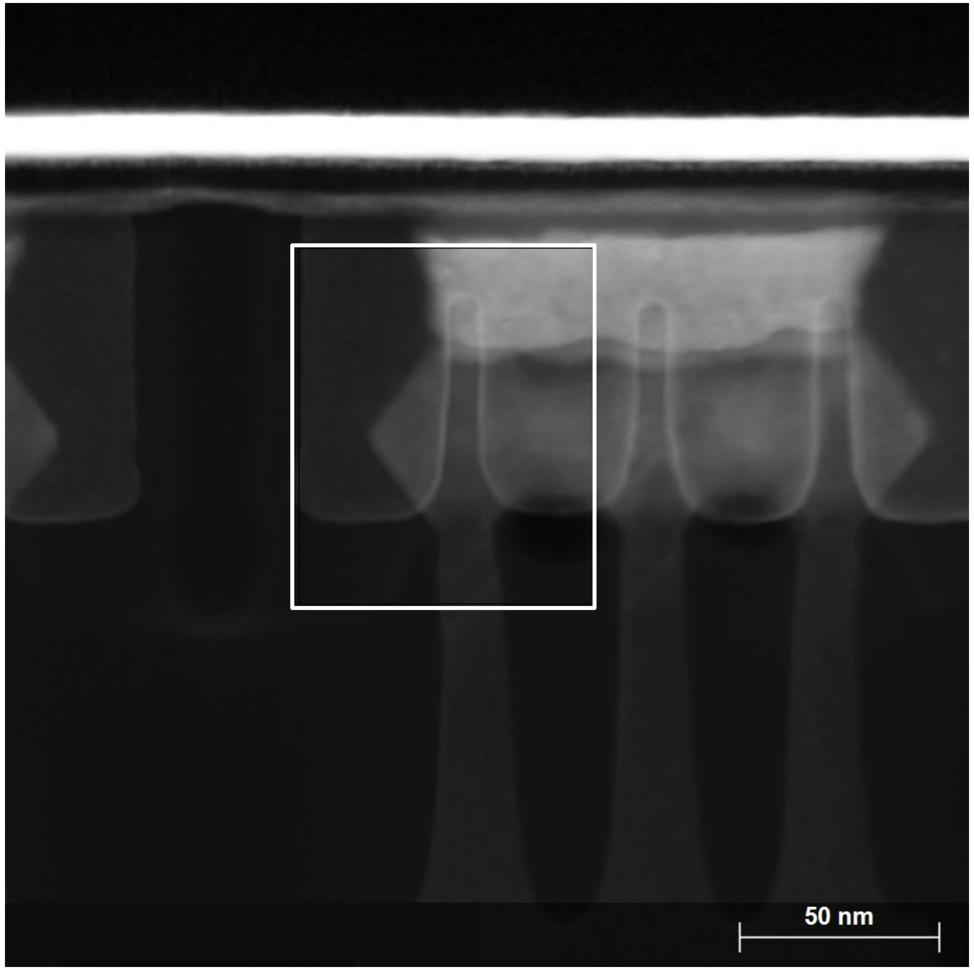


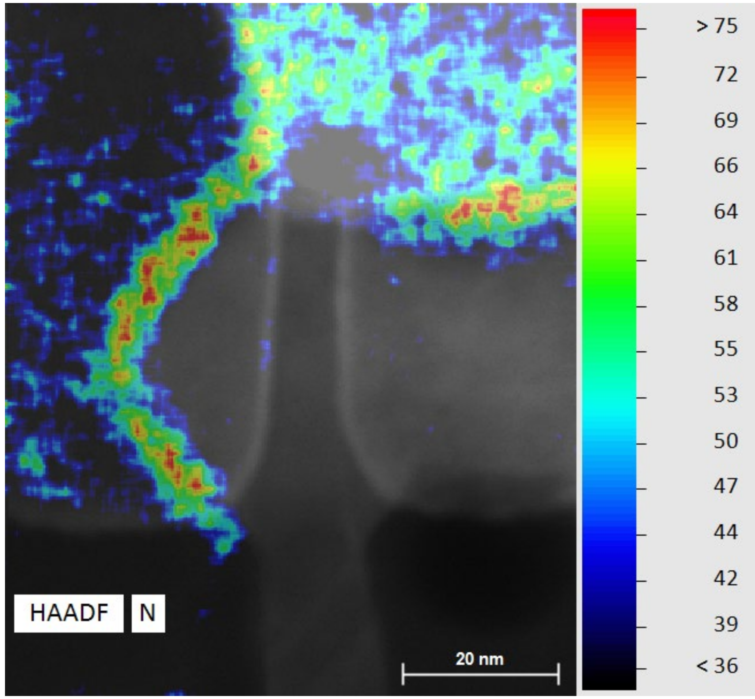
Data Courtesy: ACE

Quantitative Element Mapping of Semiconductor Nanostructures

TFS Titan 80-300 with Bruker XFlash®6T-100-oval EDS detector.

Raw data;
Acquisition time: 24min.



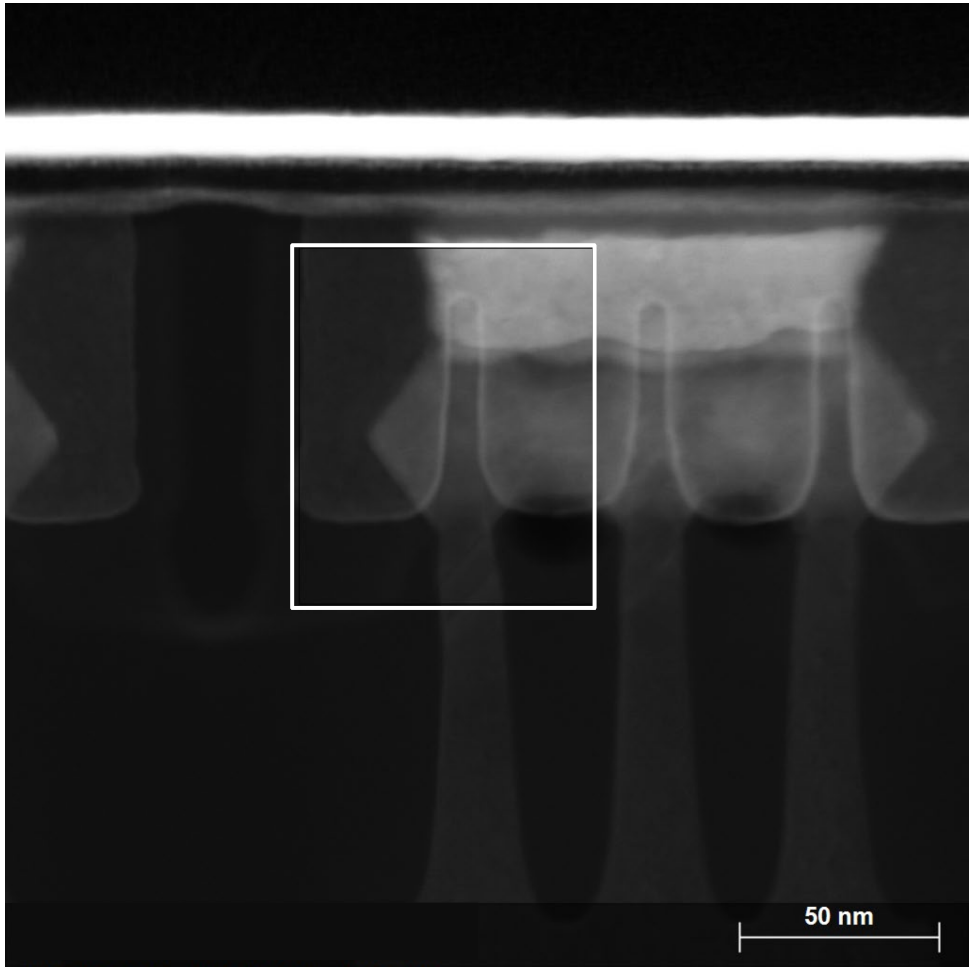


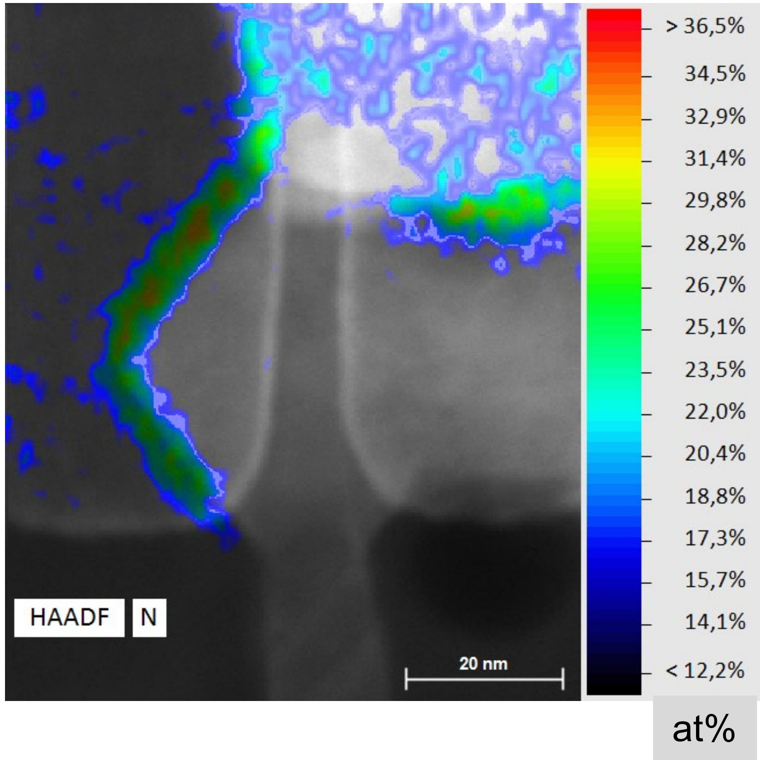
Data Courtesy: ACE

Quantitative Element Mapping of Semiconductor Nanostructures

TFS Titan 80-300 with
Bruker XFlash®6T-100-oval
EDS detector.

Quantified using the
Cliff-Lorimer Method: at%.



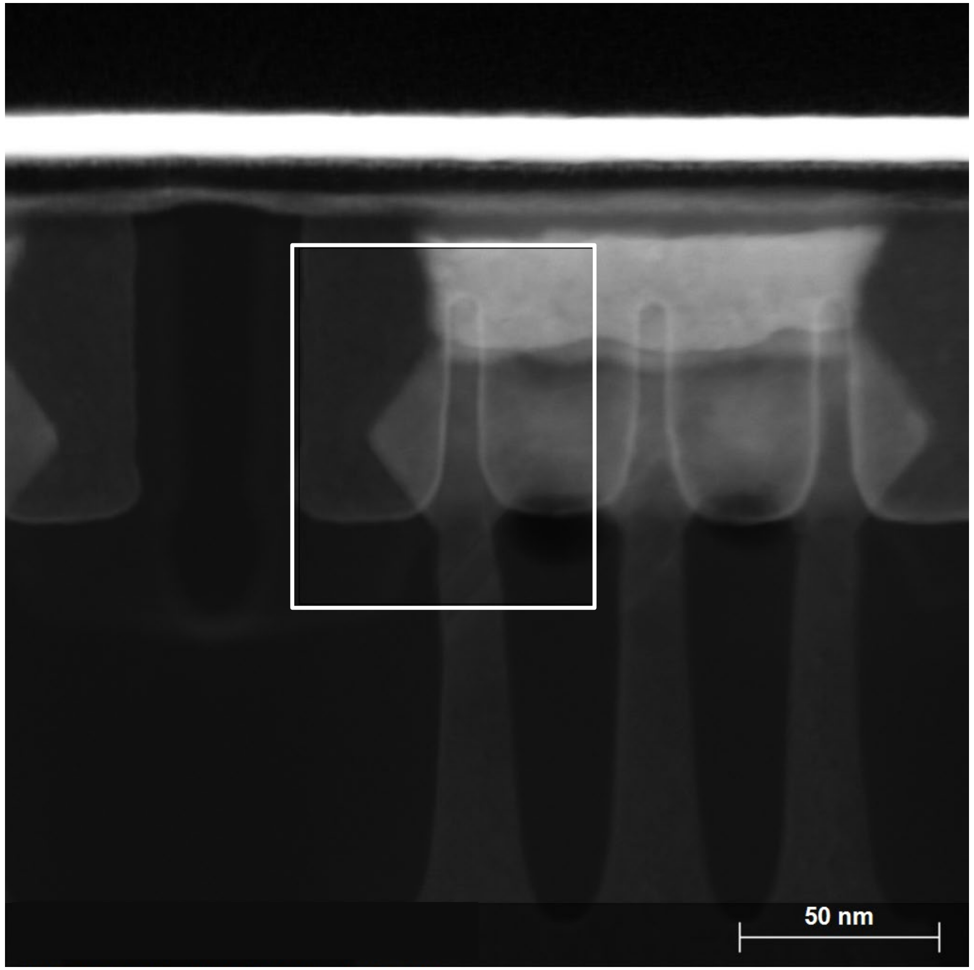


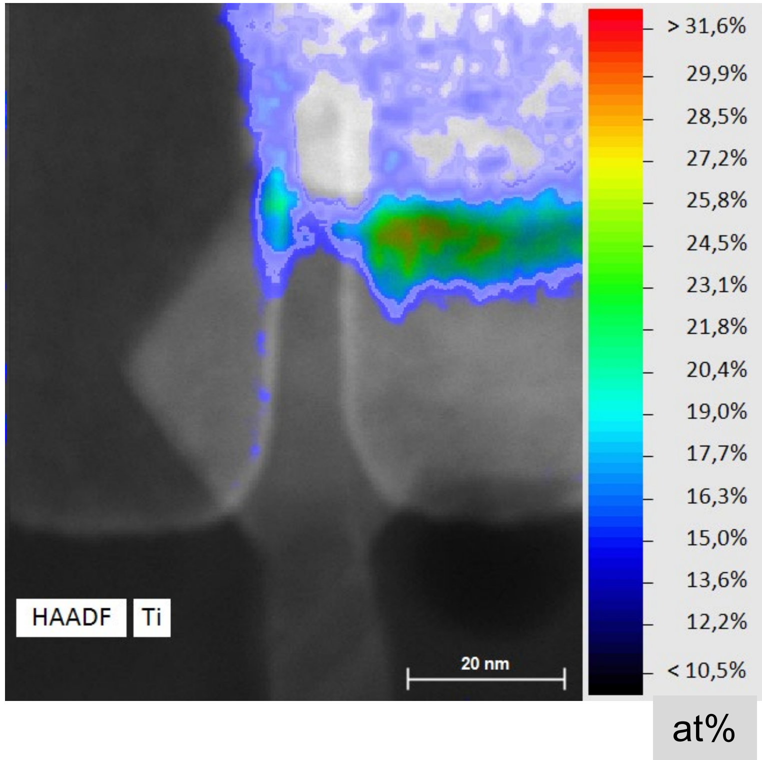
Data Courtesy: ACE

Quantitative Element Mapping of Semiconductor Nanostructures

TFS Titan 80-300 with Bruker XFlash®6T-100-oval EDS detector.

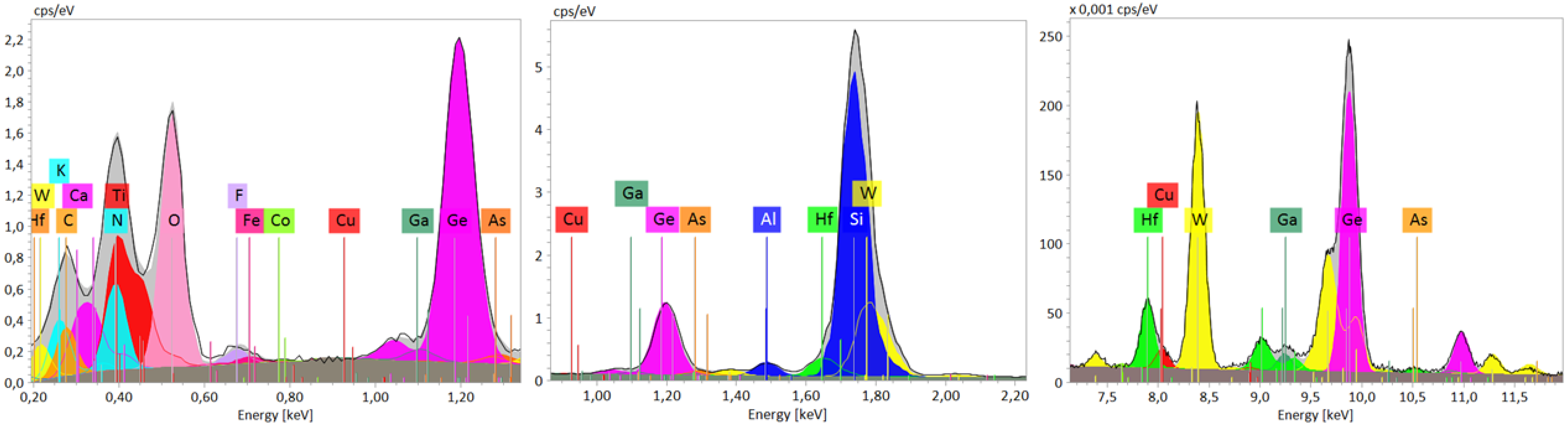
Quantified using the Cliff-Lorimer Method: at%.

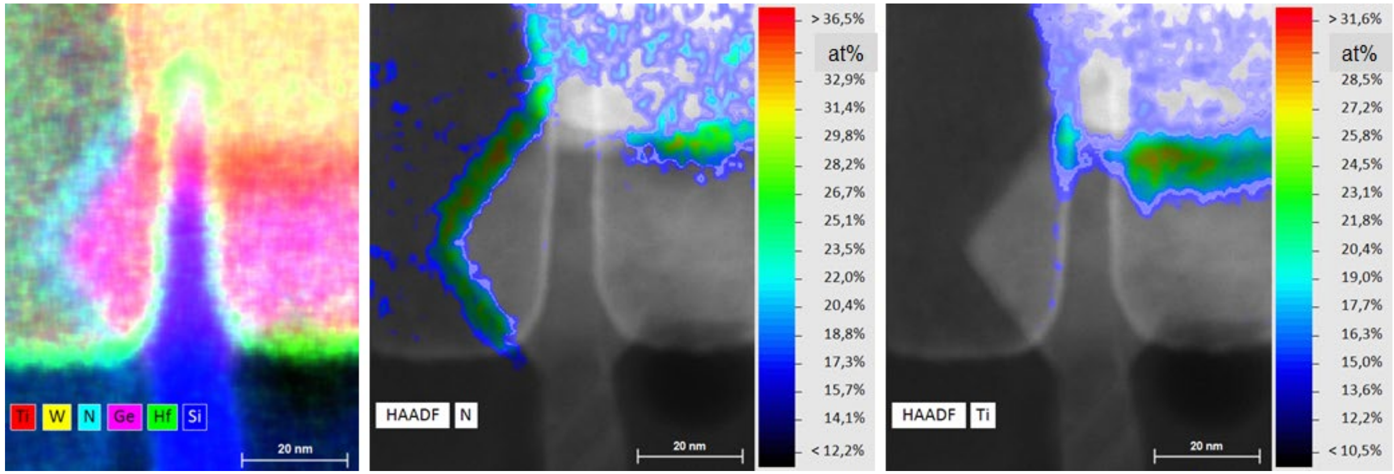




Data Courtesy: ACE

Quantitative Element Mapping of Semiconductor Nanostructures:
Deconvolution and Quantification Result





Data Courtesy: ACE

Windowless high collection angle EDS with 100mm² oval silicon drift detector area.

Summary

So far realized

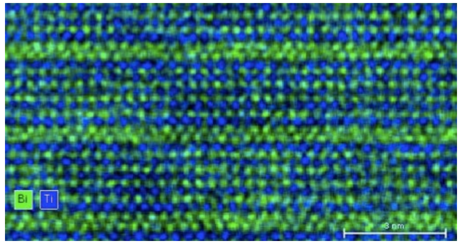
TEM:
- Nion (CFEG)
- TFS: Tecnai, Titan
- Jeol: F200
- … more to come

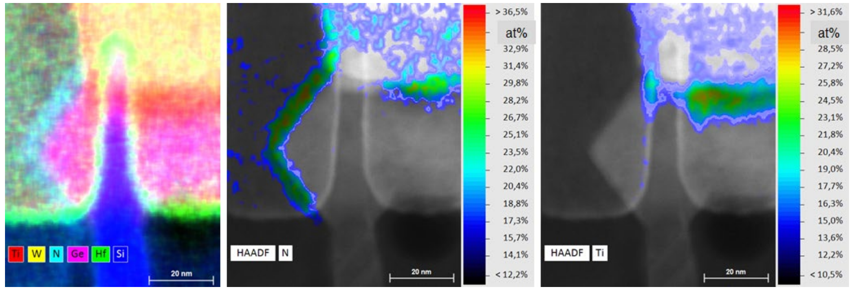
SEM: available as well, EDS up to 30kV

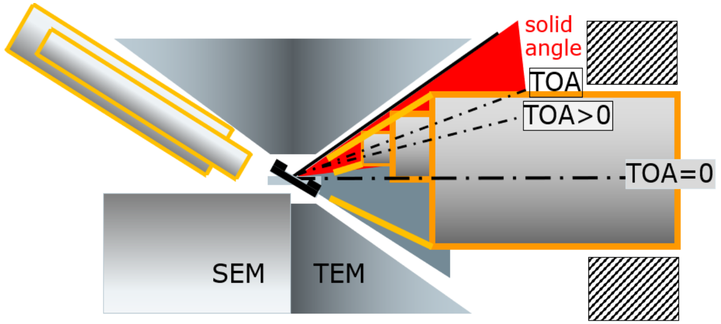
We work and collaborate with all manufacturers!

Carefully discuss with us the best option for your setup!

For more examples check out the Bruker EDS Applications web page:
https://www.bruker.com/products/x-ray-diffraction-and-elemental-analysis/eds-wds-ebsd-sem-micro-xrf-and-sem-micro-ct/quantax-eds-for-tem/applications.html







Q&A

Are there any questions?

Please type in the questions you might have
in the Q&A box and press *Send*.

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