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Advances In 3D Tomography And 4DSTEM: Perspectives To Study Semiconductor Devices

Nicolas Gauquelin, Daniel Arenas Esteban, Mikhail Mychinko, Andrey Orekhov, Daen Jannis, Evgenii Vlasov, Sandra van Aert, Johan Verbeeck and Sara Bals

EMAT, University of Antwerpen, Antwerpen, Belgium

Outline

- Introduction
  - Semiconductors metrology and its challenges
- Advances in tomography of nanoparticles
  - Electron tomography introduction
  - Conventional vs. Fast tomography
  - Atomic resolution tomography
  - SEEBIC as an alternative
- 4DSTEM in materials science
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  - Fluctuation microscopy in ZrCu metallic glasses
  - Biasing - Ca2RuO4: Stripes formation after quench of the electric field
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  - Introduction to GAA-FET
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  - EELS-EDX tomography
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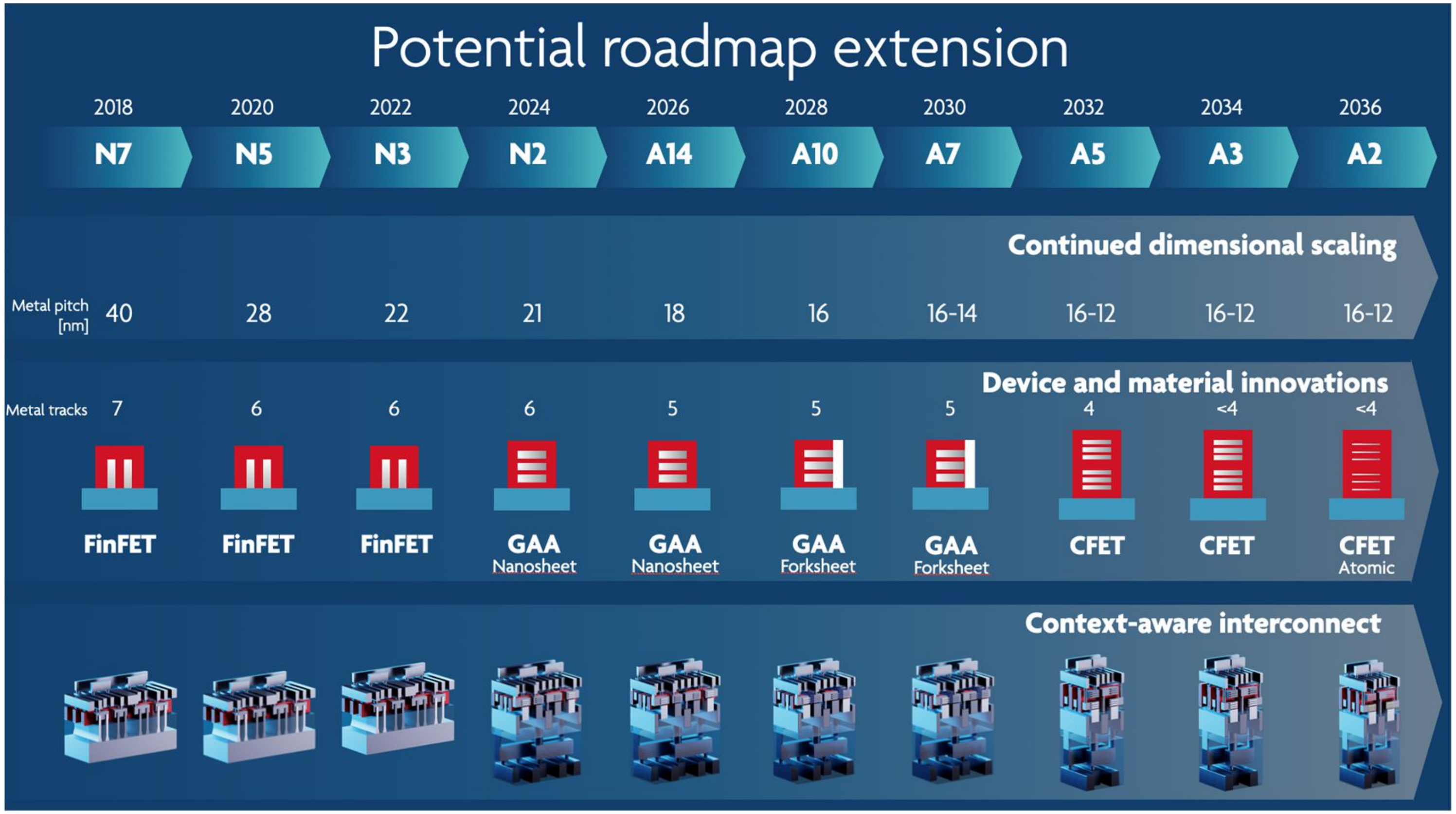
- Perspectives and outlook

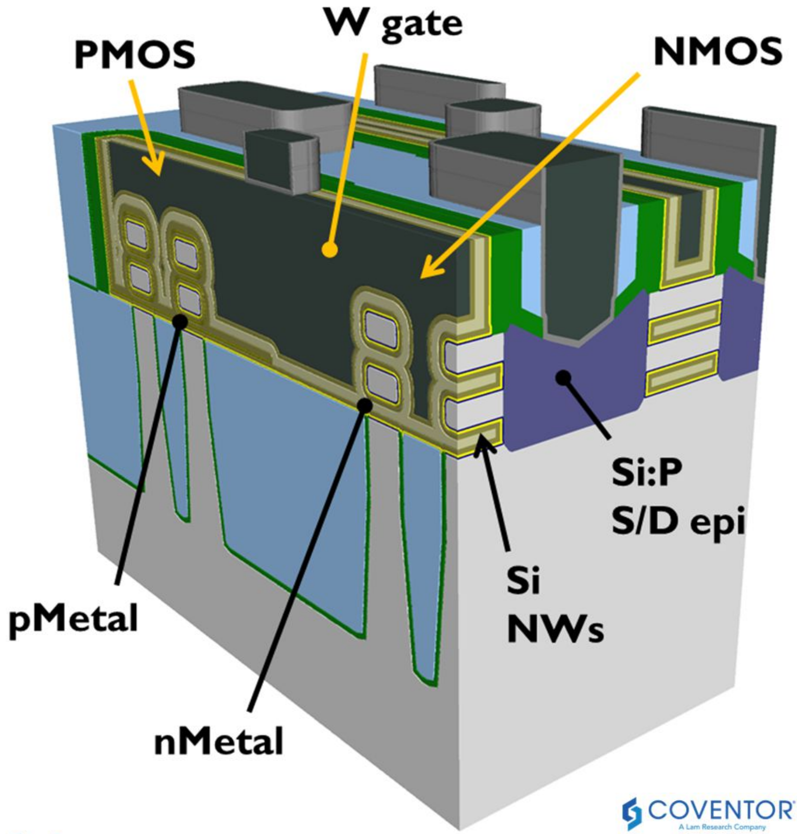
Increasing complexity of semiconductor technology



From IMEC

Challenges for TEM analysis

- Interface sharpness
- Ge diffusion
- Composition
- Layer continuity
- Shape and size
- Strain evolution
- 3D device
- Nanowire dimensions < typical TEM specimen thickness
- Many materials in nm layer thickness



Courtesy of P. Favia - IMEC

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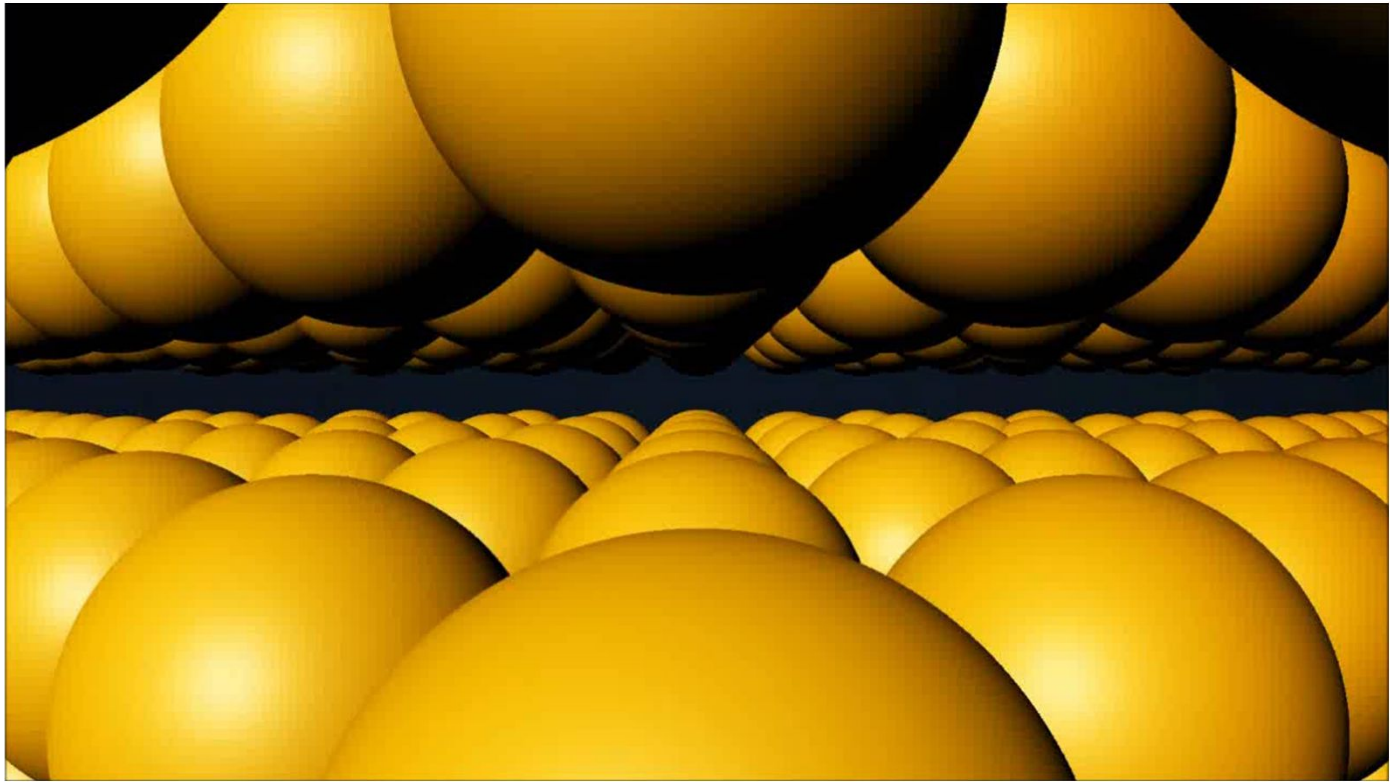
In the electron microscope



From the 2D images to the 3D object

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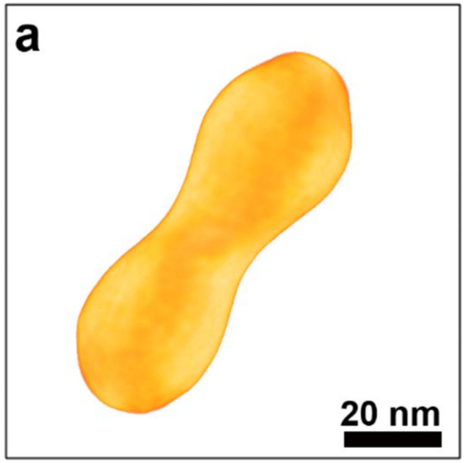
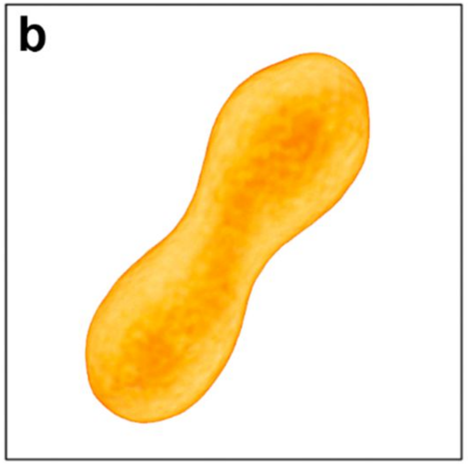
Fast tomography

Normal Tomo



49 images
1 hour

Fast Tomo



700 images
5 minutes

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W. Albrecht and S. Bals, J. Phys. Chem. C 2020, 124, 50, 27276–27286 (2020)

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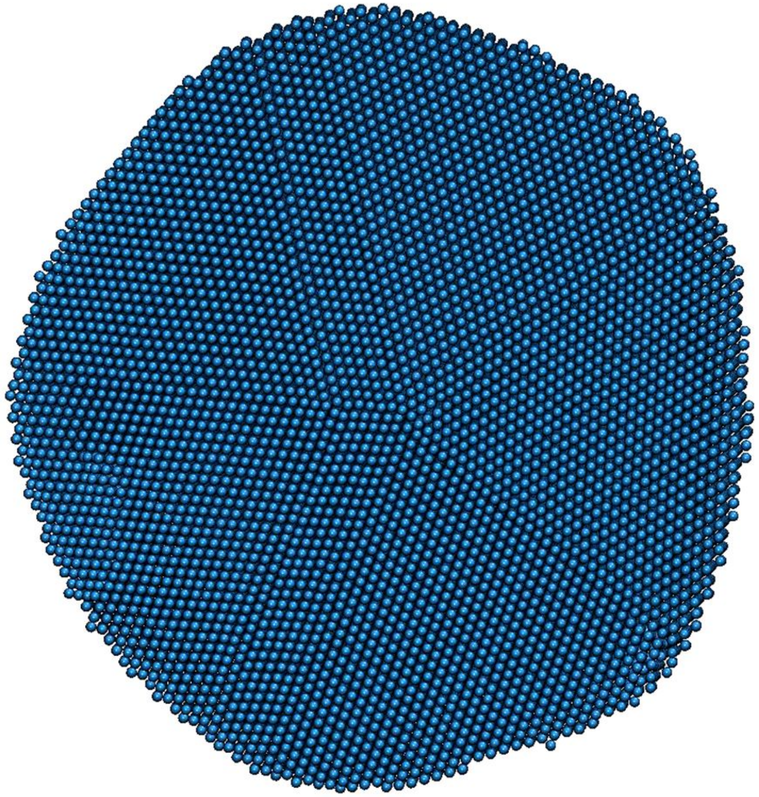
Atomic tomography



Goris, B. et al., Nano Lett. 15, 10, 6996 (2015)

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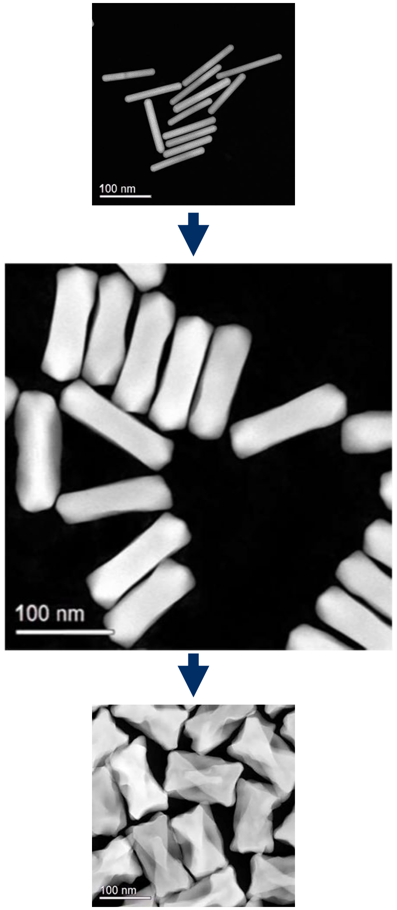
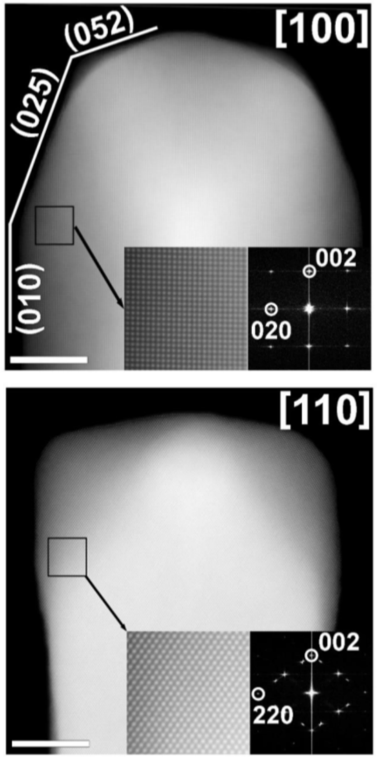
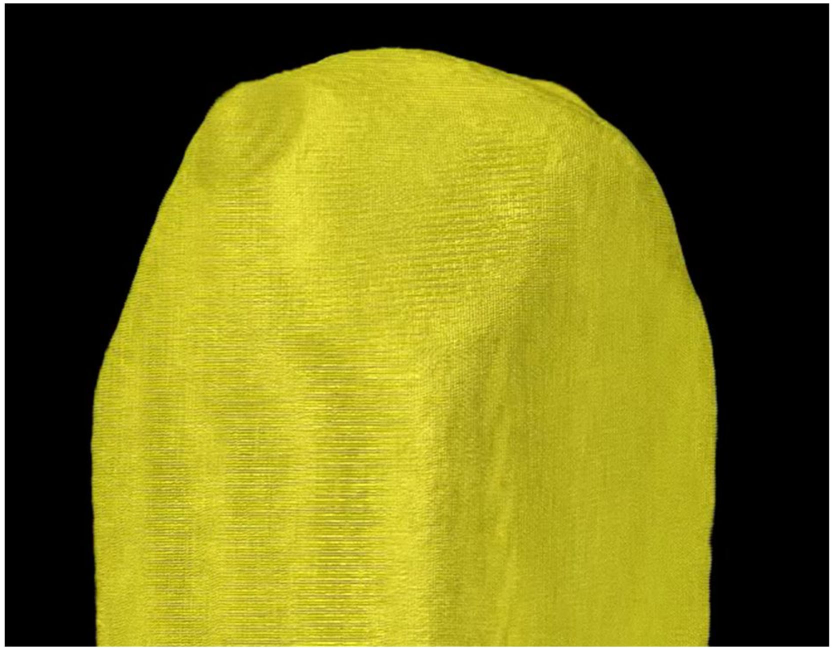
Chiral structure and facets



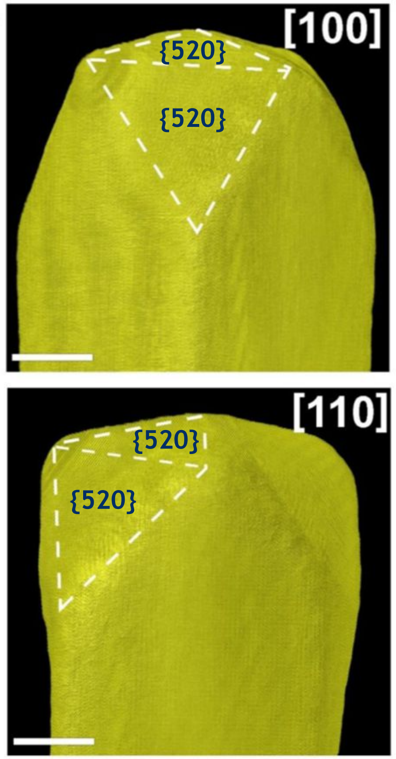
HAADF-STEM projections



High resolution 3D reconstruction



View along [100] and [110]



Scale bars are 10 nm

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Ni B., Mychinko M., Gómez-Graña S., Morales-Vidal J., Obelleiro-Liz M., Heyvaert W., Vila-Liarte D., Zhuo X., Albrecht W., Zheng G., González-Rubio G., Taboada J.M., Obelleiro F., López N., Pérez-Juste J., Pastoriza-Santos I., Cölfen H., Bals S., and Liz-Marzán L.M./ Adv. Mater. 2023, 35, 2208299.

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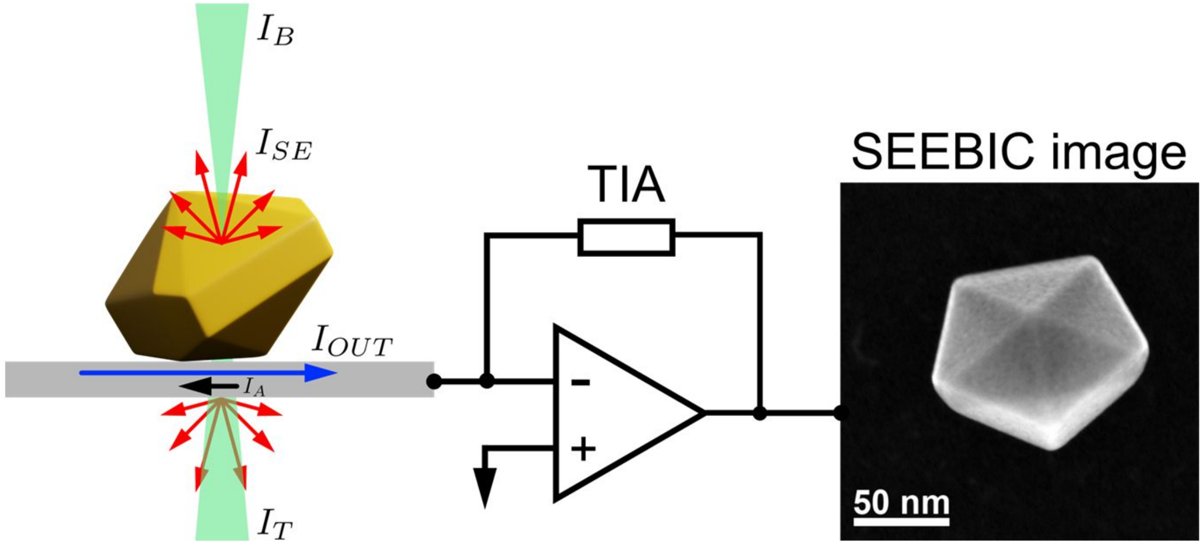
SEEBIC principles

SEEBIC

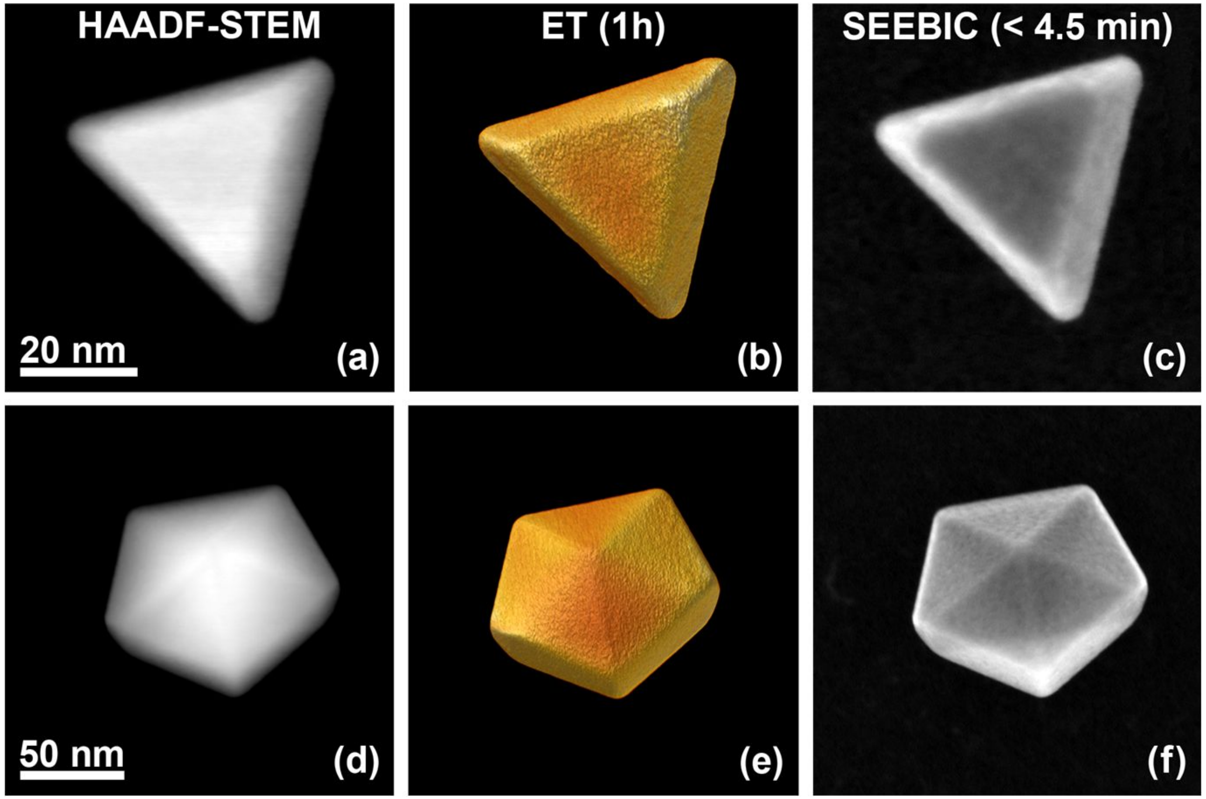
- signal arises from holes induced by the emission of SEs from the electron-transparent sample
- detected signal is equal but opposite to the generated SE current



$$I_B = I_T + I_A + I_{BSE} + I_{SE} + {V_S}/{R_S} + I_{out}$$

$$-I_{OUT} = I_{SE} + {V_S}/{R_S}$$

SEEBIC for visualization of NP morphology



- SEEBIC enables to gain information about surface morphology from a single image in several minutes, whereas ET may take up to 1 hour of acquisition time for tilt series and requires additional processing

E. Vlasov, et al., *ACS Mater. Lett.*, **5** (2023)

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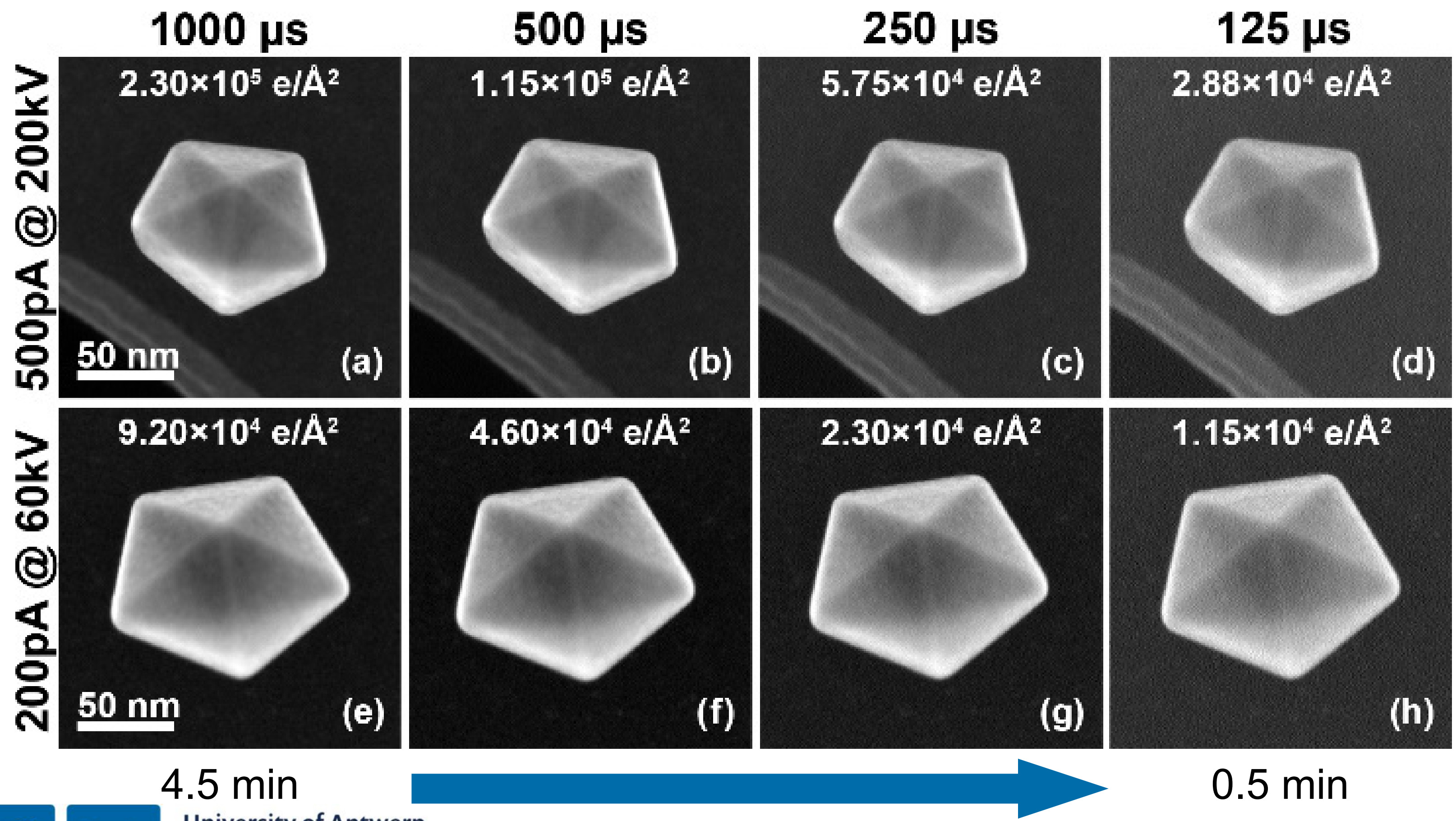
SEEBIC for visualization of NP morphology



>100× faster

2× more dose efficient

E. Vlasov et al., *ACS Mater. Lett.*, **5** (2023)

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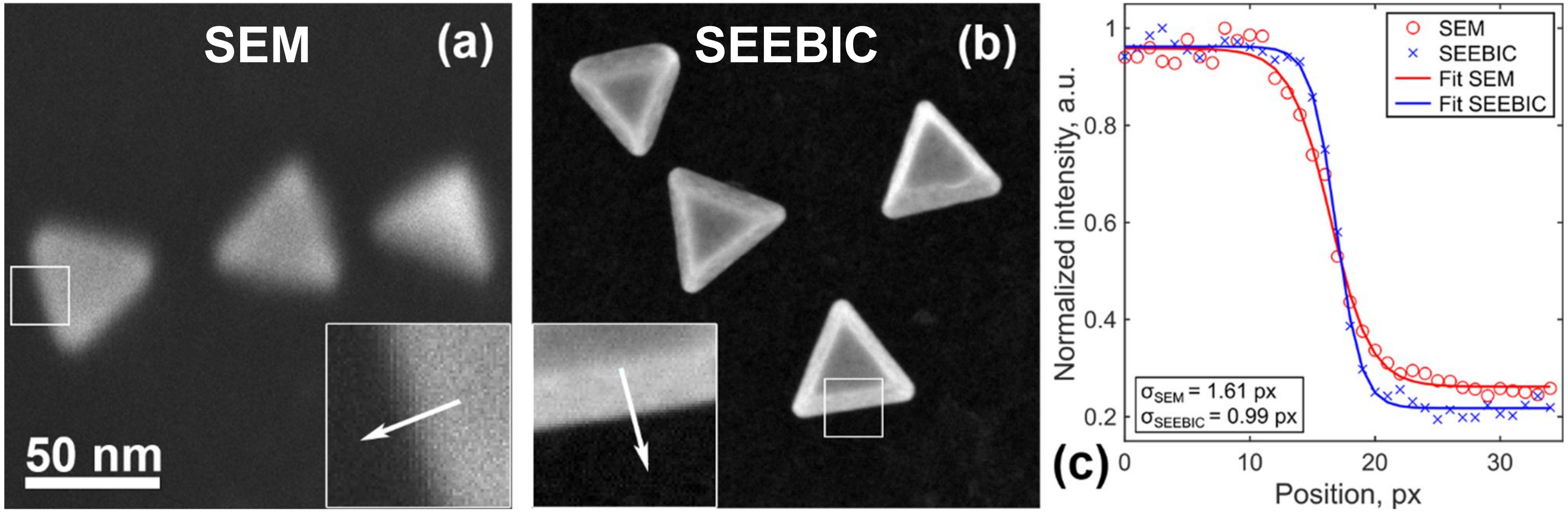
SEEBIC vs SEM



E. Vlasov, A. Skorikov, A. Sánchez-Iglesias, L. M. Liz-Marzán, J. Verbeeck, S. Bals, *ACS Mater. Lett.,* **5** (2023)

- SEEBIC has superior spatial resolution compared to SEM (1.3 nm vs 4.2 nm)
- SEEBIC resolution is limited by the selected sampling and can be pushed to the obtainable probe size (~100 pm)

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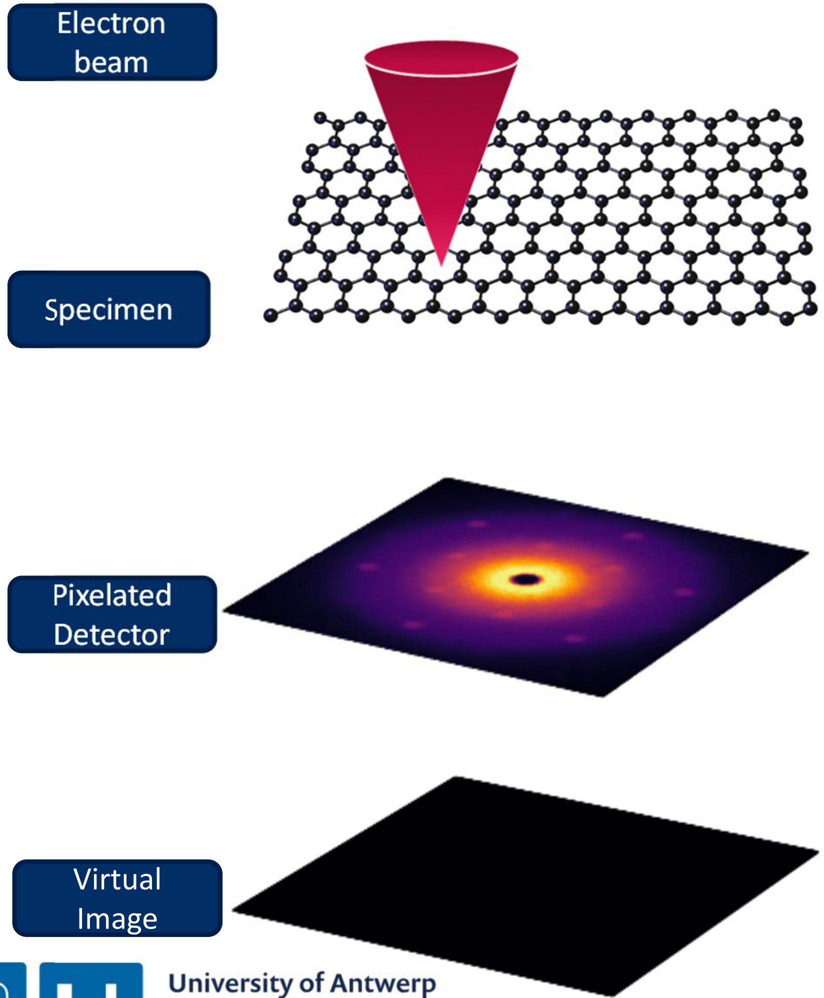
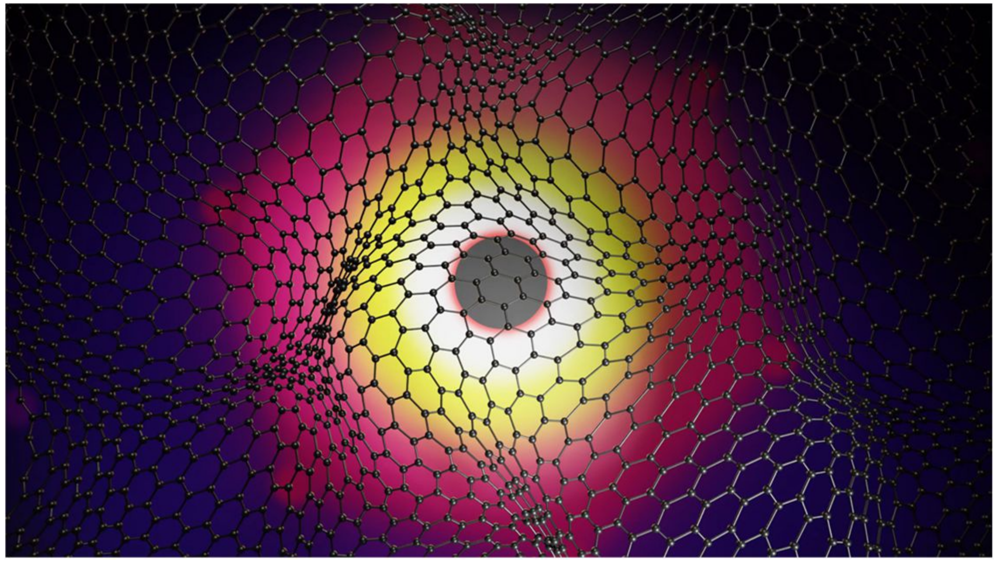
4D STEM





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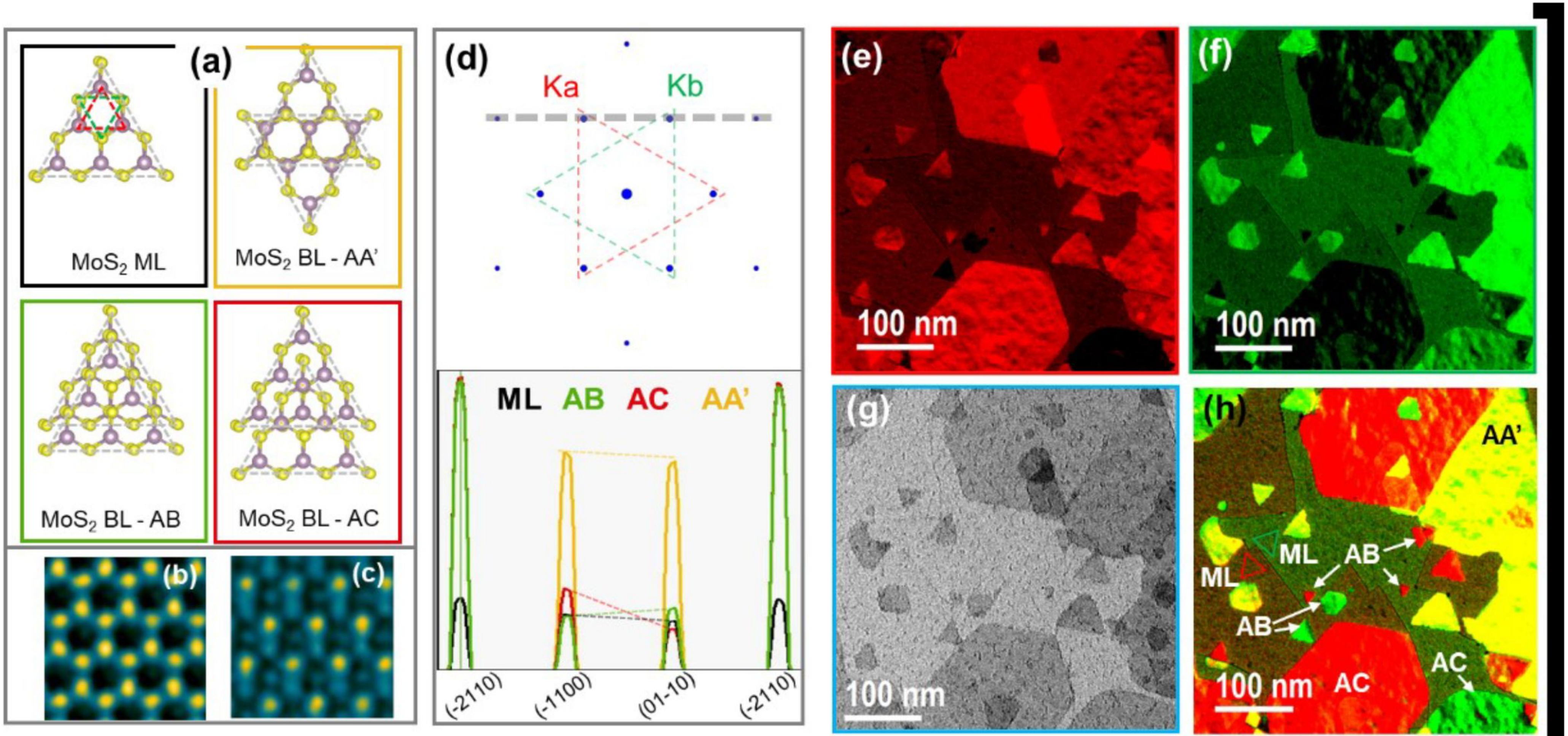
Unraveling stacking in MoS2 bilayer



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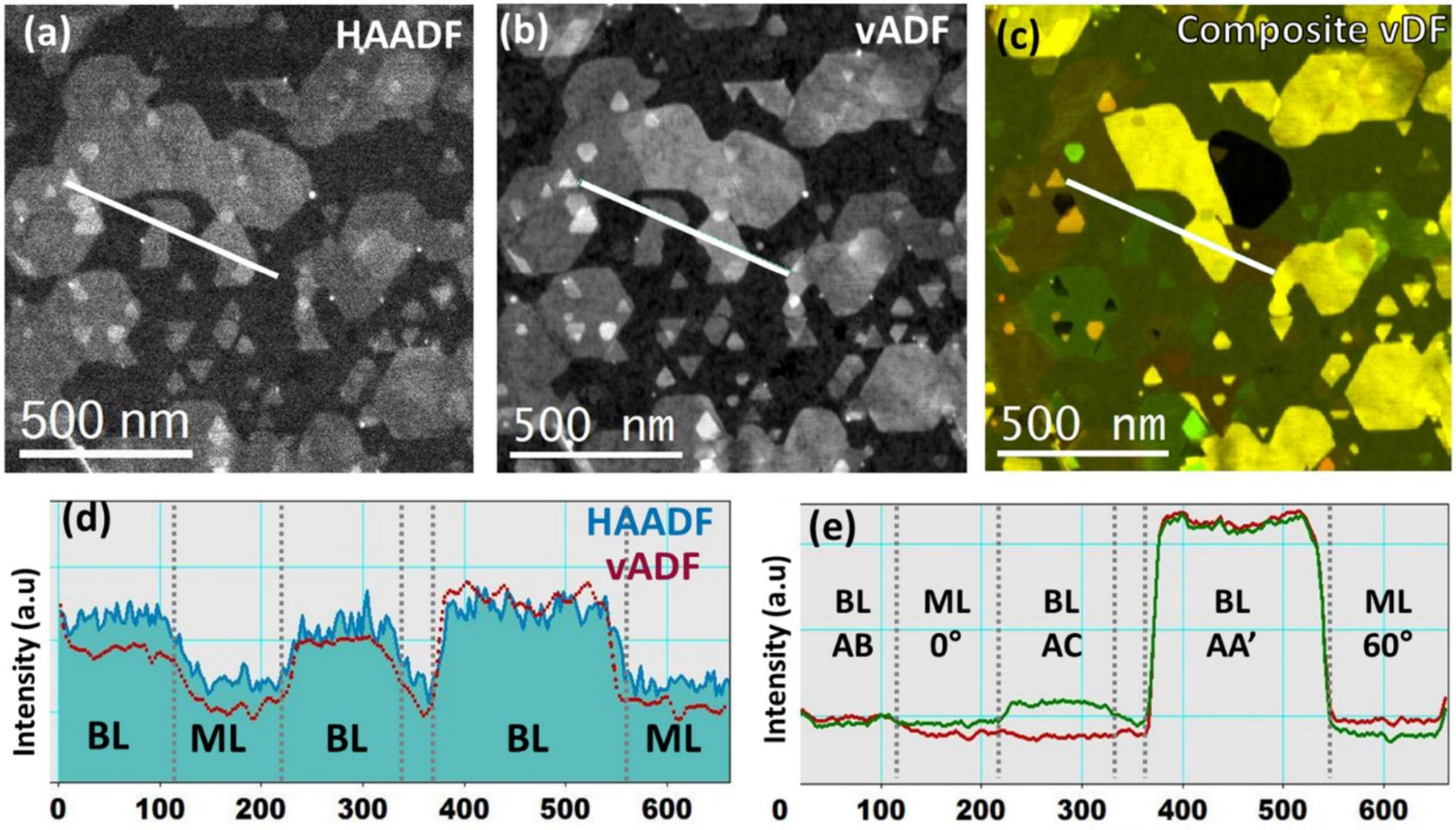
Nanotechnology. 2020 Oct 30;31(44):445702



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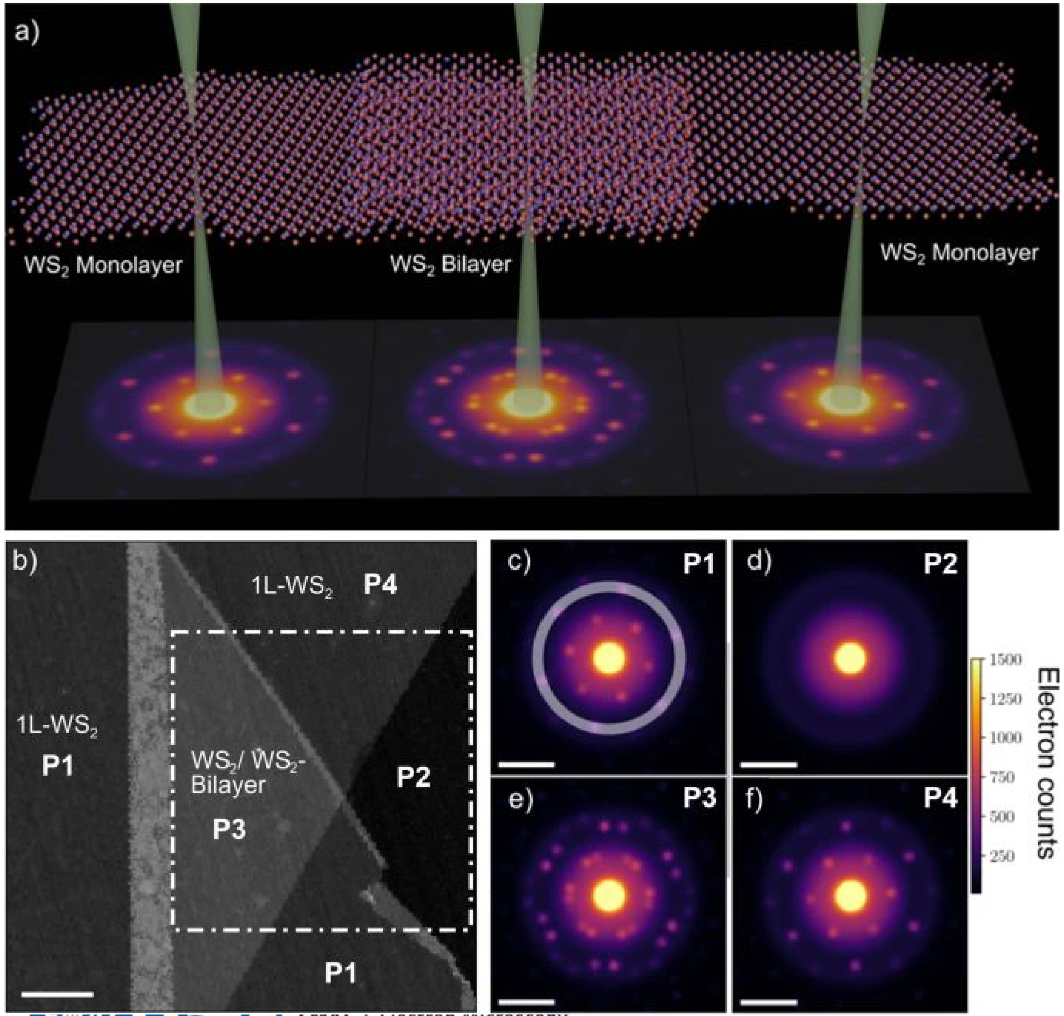
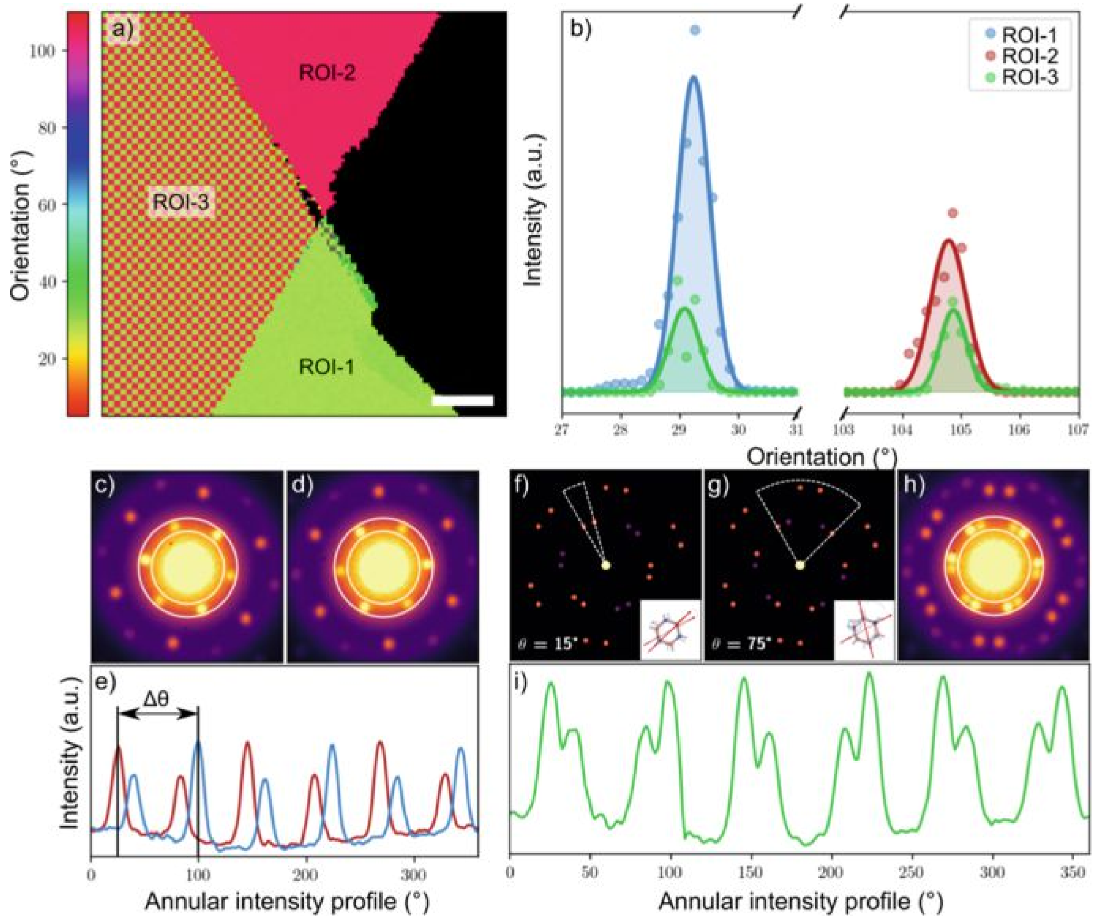
Nanotechnology. 2020 Oct 30;31(44):445702

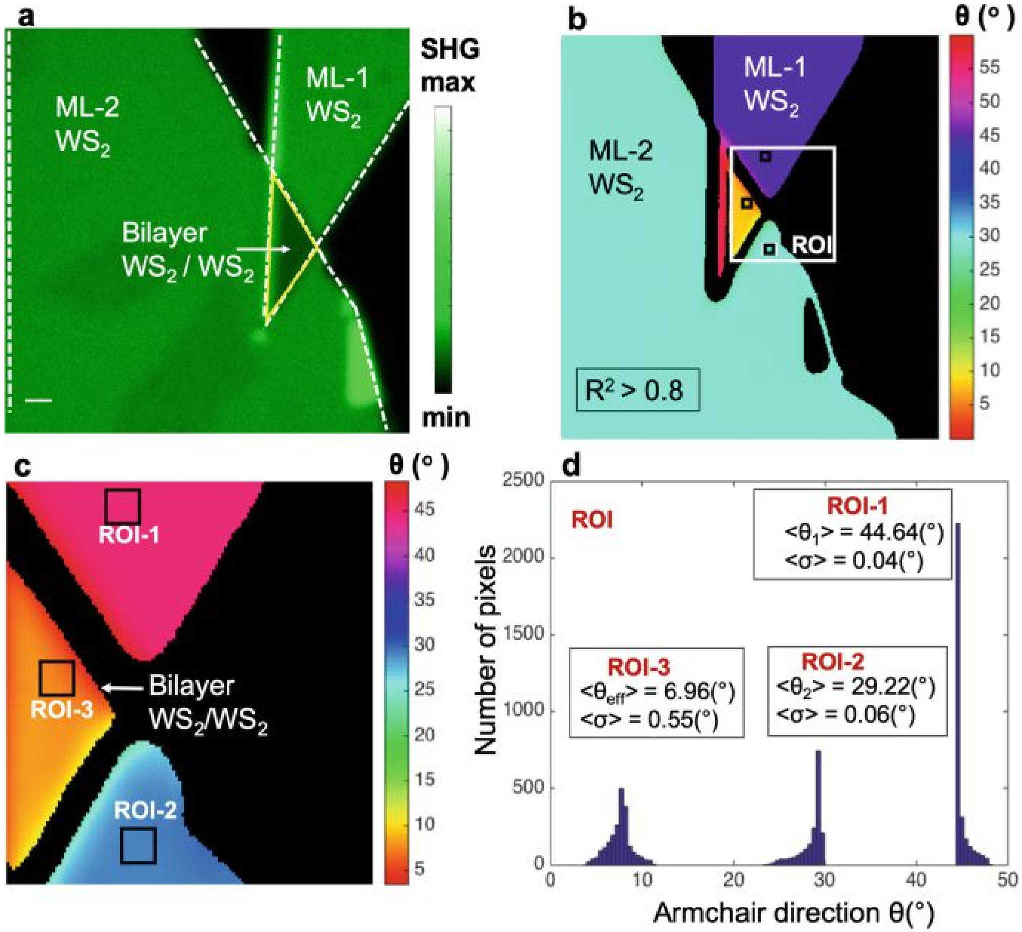
Twist angle measurement in WS2

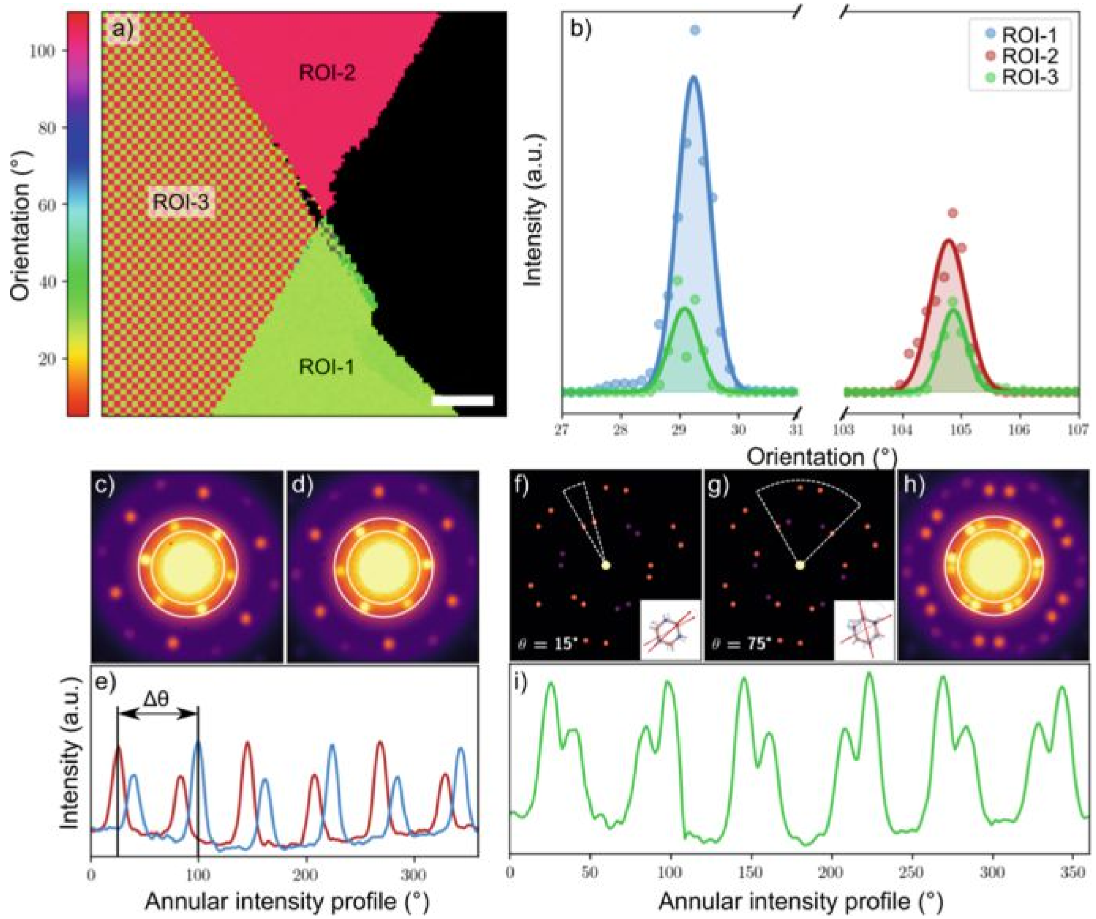




S. Psilodimitrakopoulos et al., npj2D Materials and Applications, 5, 2021; 77

Twist angle measurement in WS2





S. Psilodimitrakopoulos et al., npj2D Materials and Applications, 5, 2021; 77

Correlation with SHG

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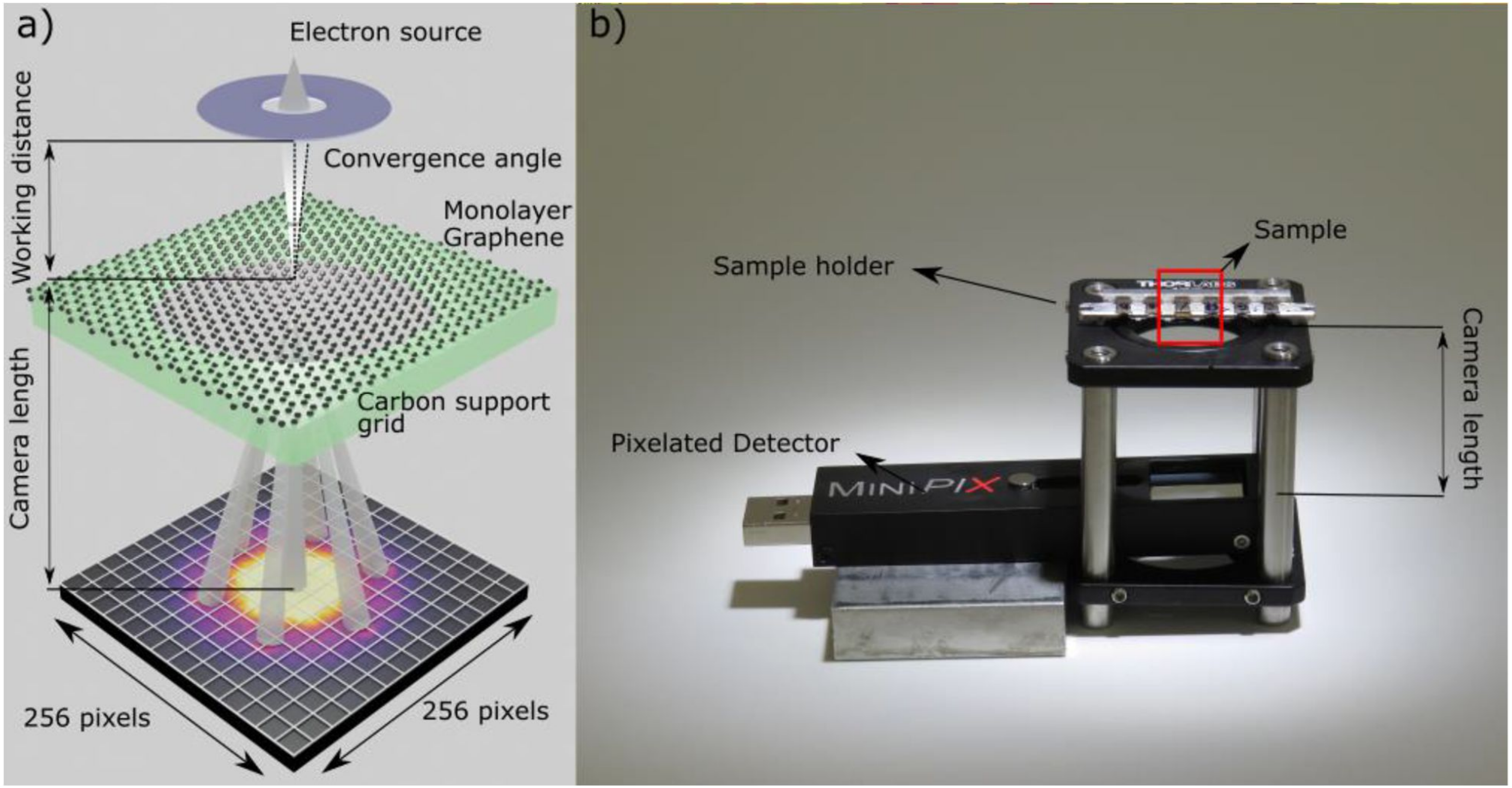
TEM has limited field of view (a couple of $\mu m2$)→Statistics at microscale can be rather low
•Complicated operation mode→Skilled operator with much experience
•Limited space for sample→Heating/Gas/Bias experiments in dedicated holders
•Low interaction due too high velocity electrons (for very thin materials)
→ **SEM**

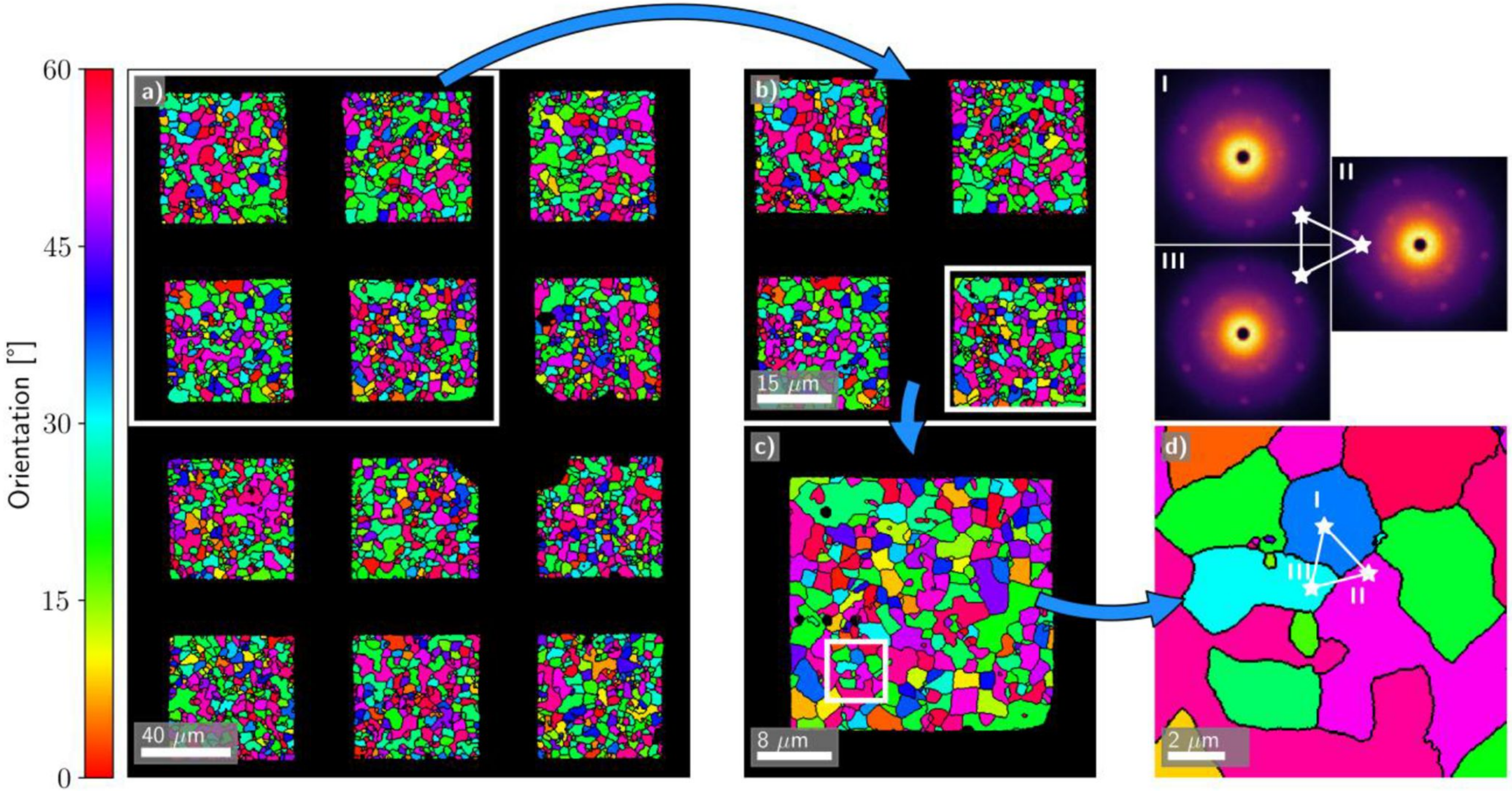
Disadvantage of SEM:
Lower spatial resolution (still better than optical)
Thickness due too lower acceleration voltage
More damage at lower acceleration voltage??? →Ideal for 2D materials

Setup



https://doi.org/10.48550/arXiv.2011.01875

In the SEM large field of view



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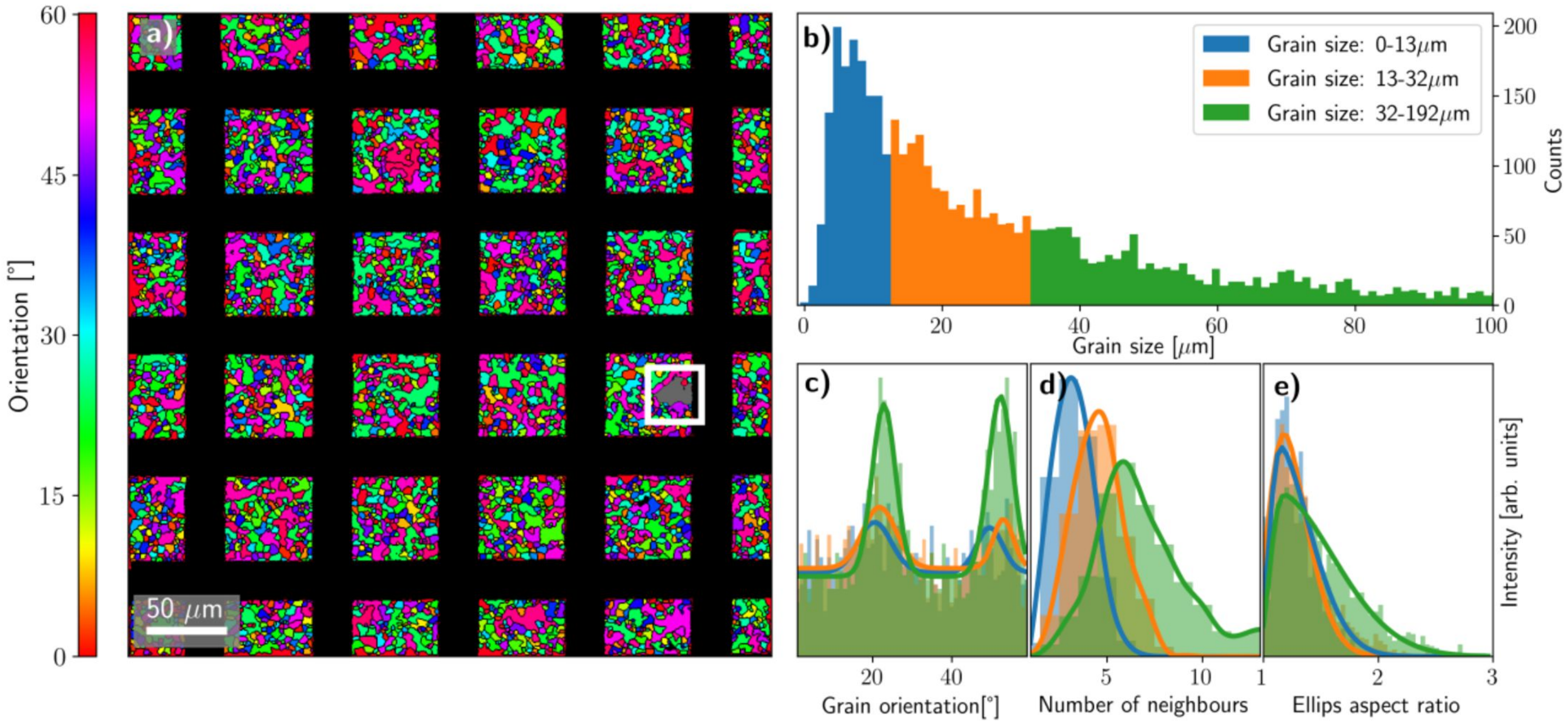
https://doi.org/10.48550/arXiv.2011.01875

Orientation mapping in large field of view 0.168 $mm2$



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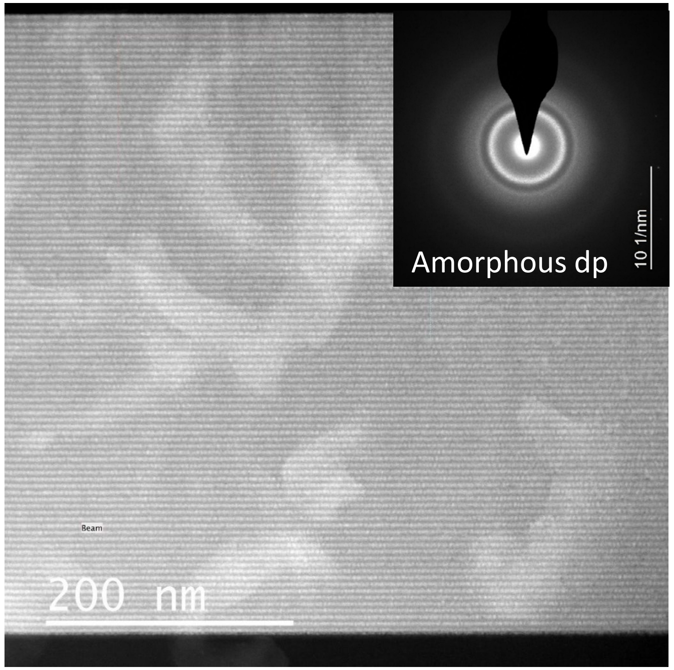
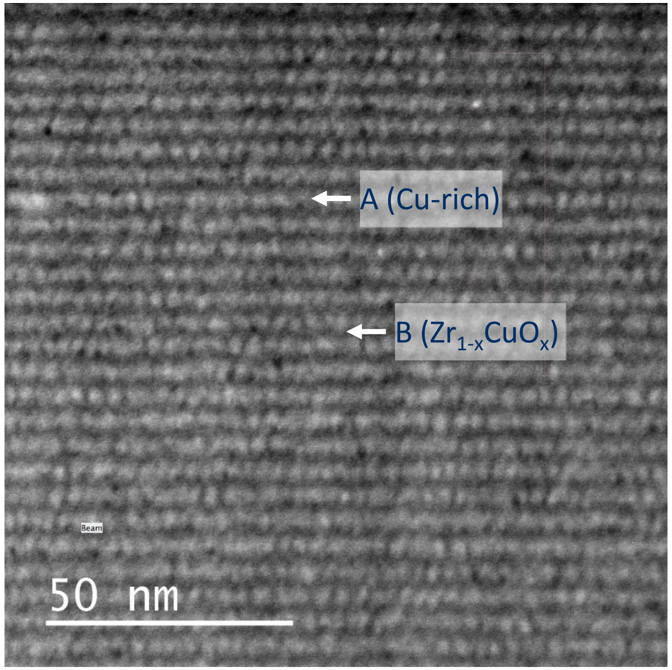
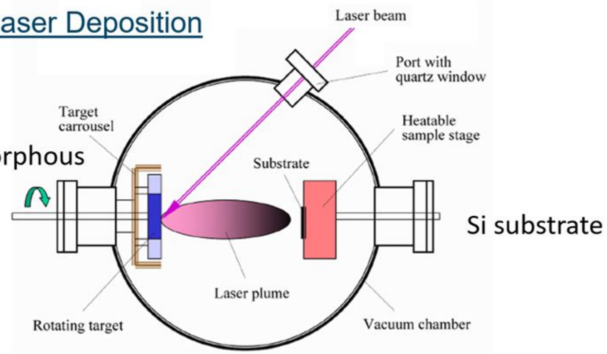
UCLouvain

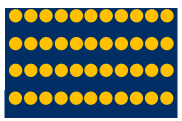
HAADF STEM analysis of ZrCu 020 (He, P = 10 Pa)

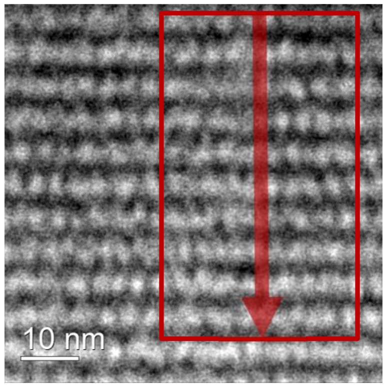


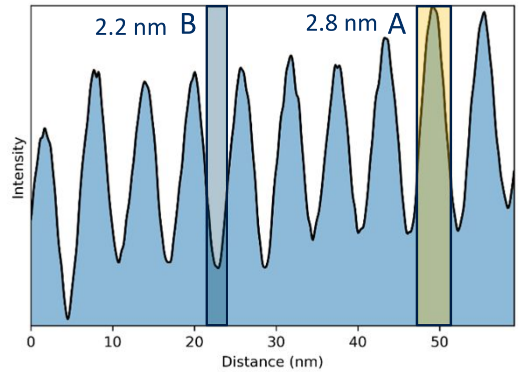




Nanostructured layered structure







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Acta Materialia 213 (2021) 116955

Contents lists available at ScienceDirect

Acta Materialia

journal homepage: www.elsevier.com/locate/actamat

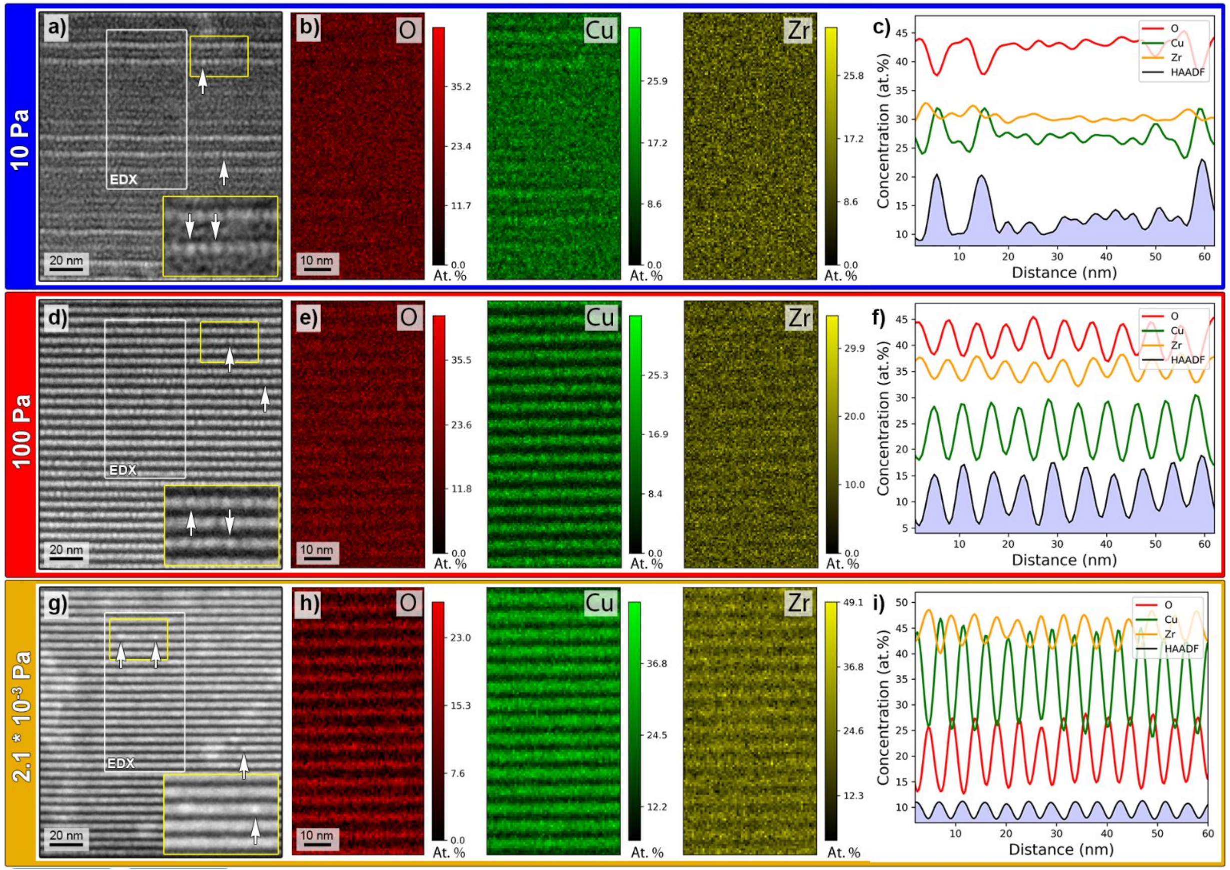
Full length article

Novel class of nanostructured metallic glass films with superior and tunable mechanical properties

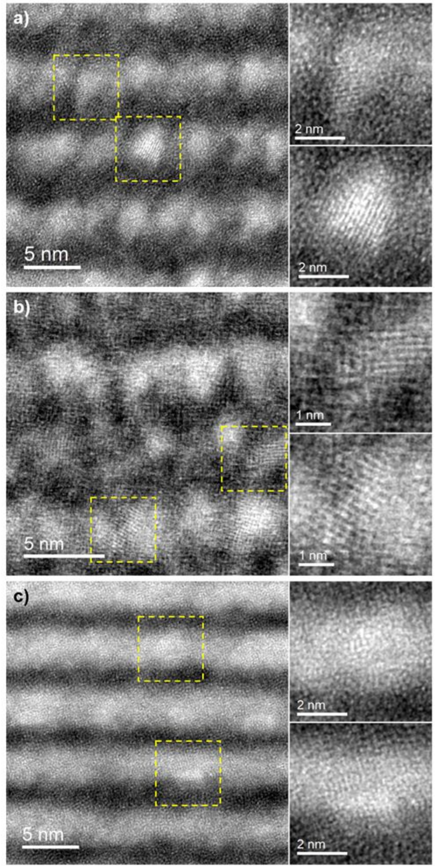
M. Ghidelli[a,b,1,*], A. Orekhov[c,d,e,1], A. Li Bassi[a], G. Terraneo[f], P. Djemia[b], G. Abadias[g], M. Nord[d,e,h], A. Béché[d,e], N. Gauquelin[d,e], J. Verbeeck[d,e], J.-P. Raskin[i], D. Schryvers[d,e], T. Pardoen[c], H. Idrissi[c,d,*]

M. Ghidelli, A. Orekhov, A.L. Bassi, G. Terraneo, P. Djemia, G. Abadias, M. Nord, A. Beche, N. Gauquelin, J. Verbeeck, J.P. Raskin, D. Schryvers, T. Pardoen, H. Idrissi, Novel class of nanostructured metallic glass films with superior and tunable mechanical properties, Acta Mater. 213 (2021), 116955.

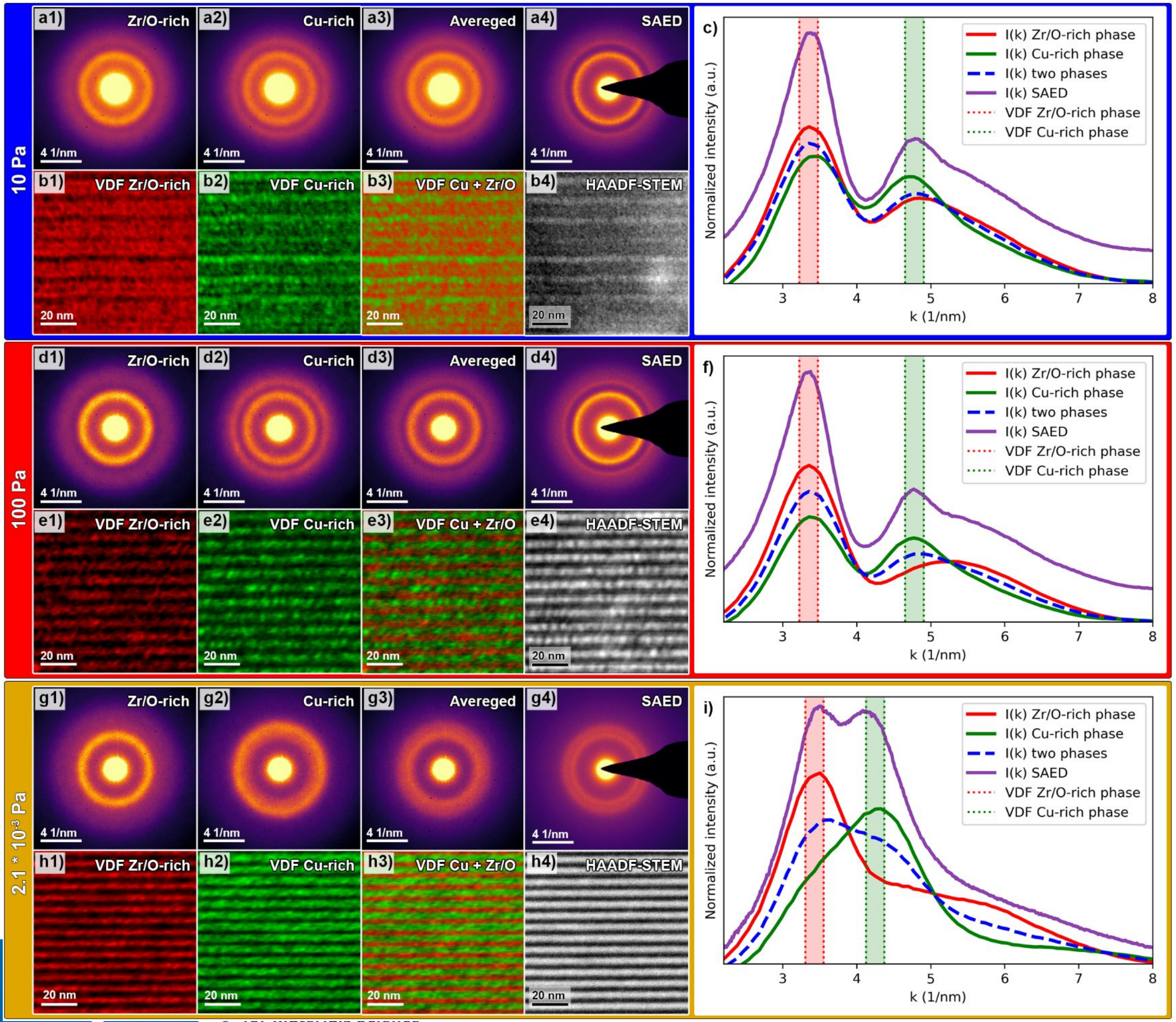
EDX



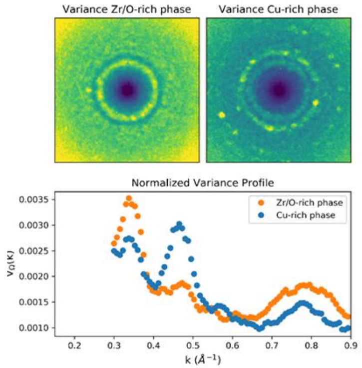
HRSTEM



4D STEM data

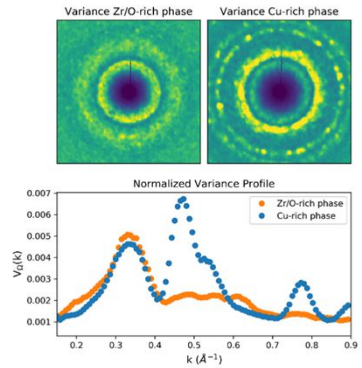


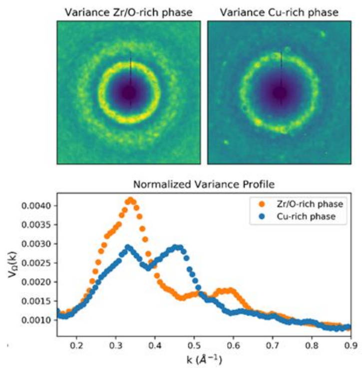
Variance diffraction



crystalline clusters (1-2 nm) in the Cu-rich phase

Zr/O-rich phase exhibits a more ‘disordered’ structure





Cu-rich phase single peak shifted to higher *k*→ changes of the nature of local order (no clusters)

Zr/O-rich e phase similar “disordered”

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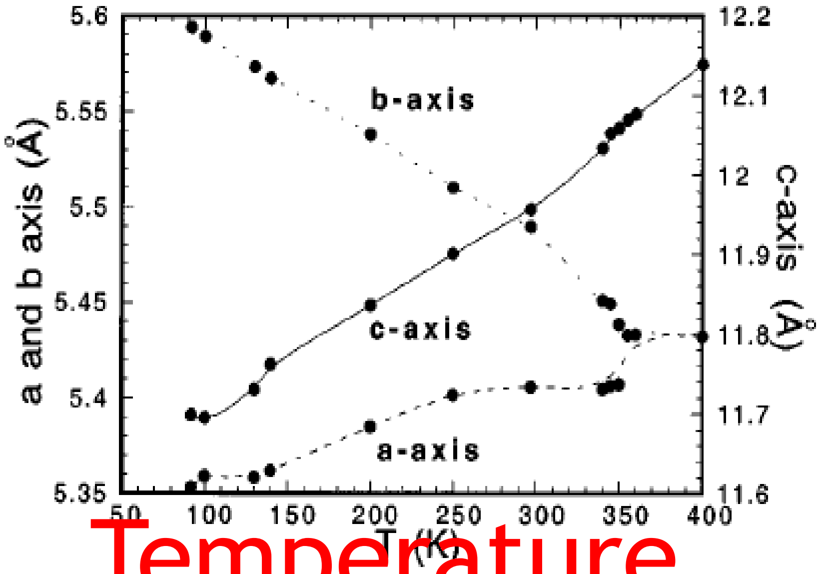
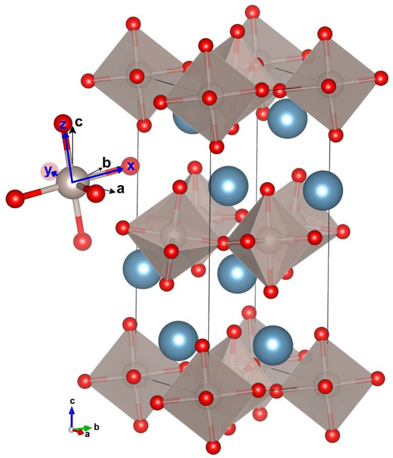
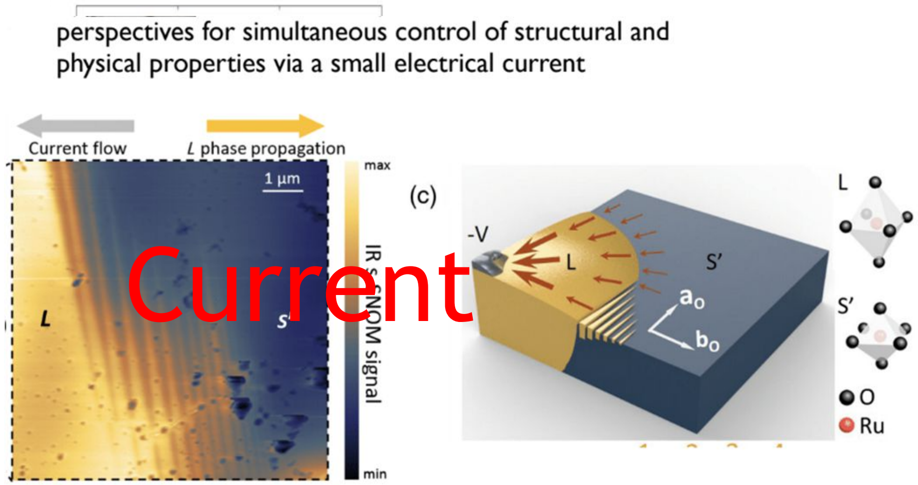


Temperature

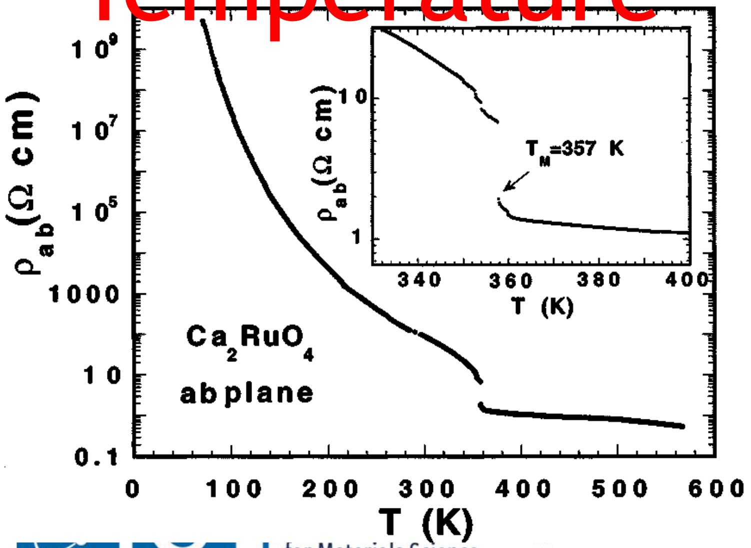




Current

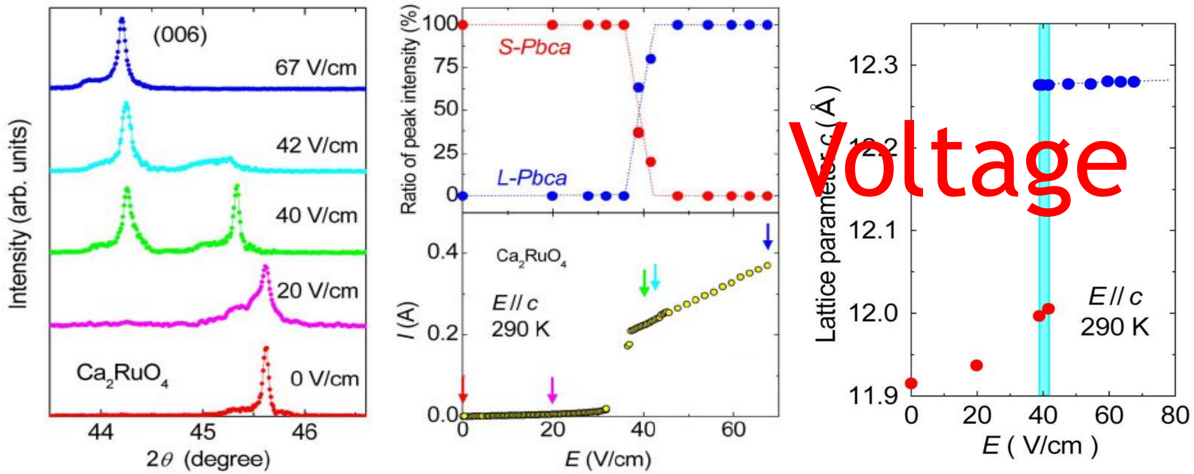
Phys. Rev. X 9, 011032 (2019)

Insulator to metal phase transition happening with T, I or V



for Materials Science

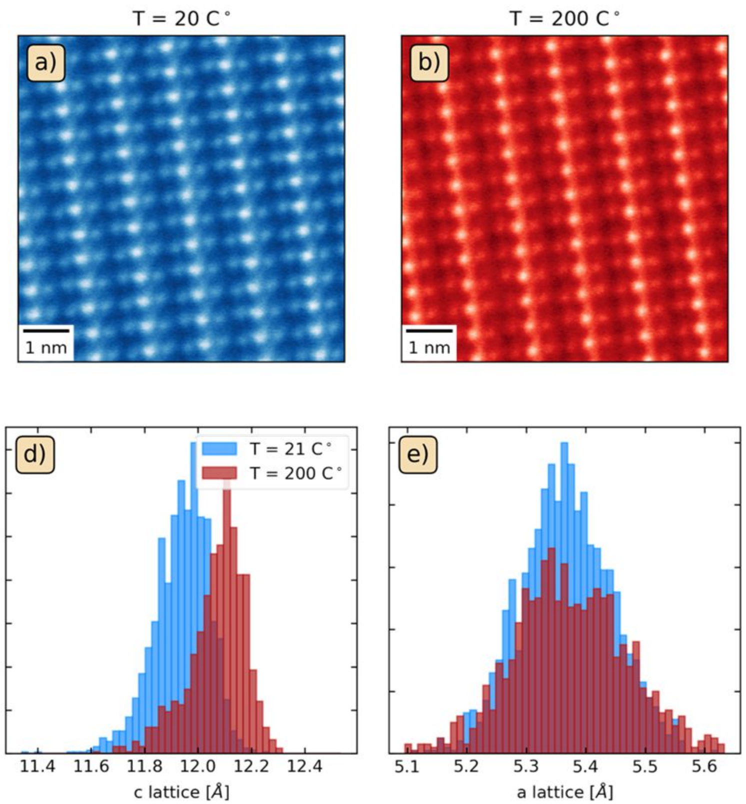
Phys. Rev. B 60, R8422 (1999)

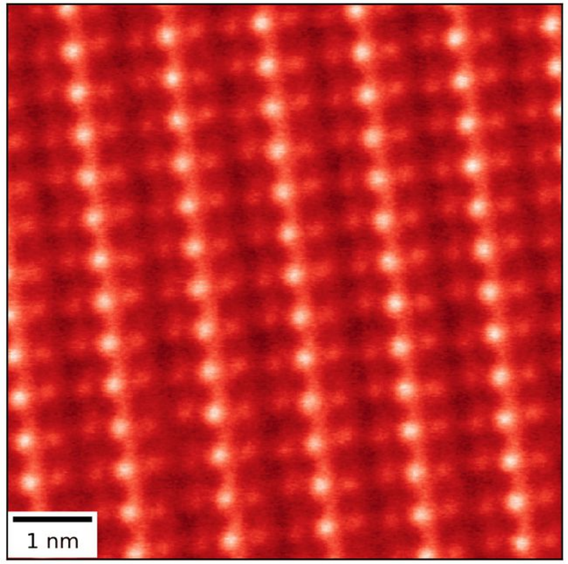


Voltage

F. Nakamura et al., Sci. Rep. 3, 2536 (2013)

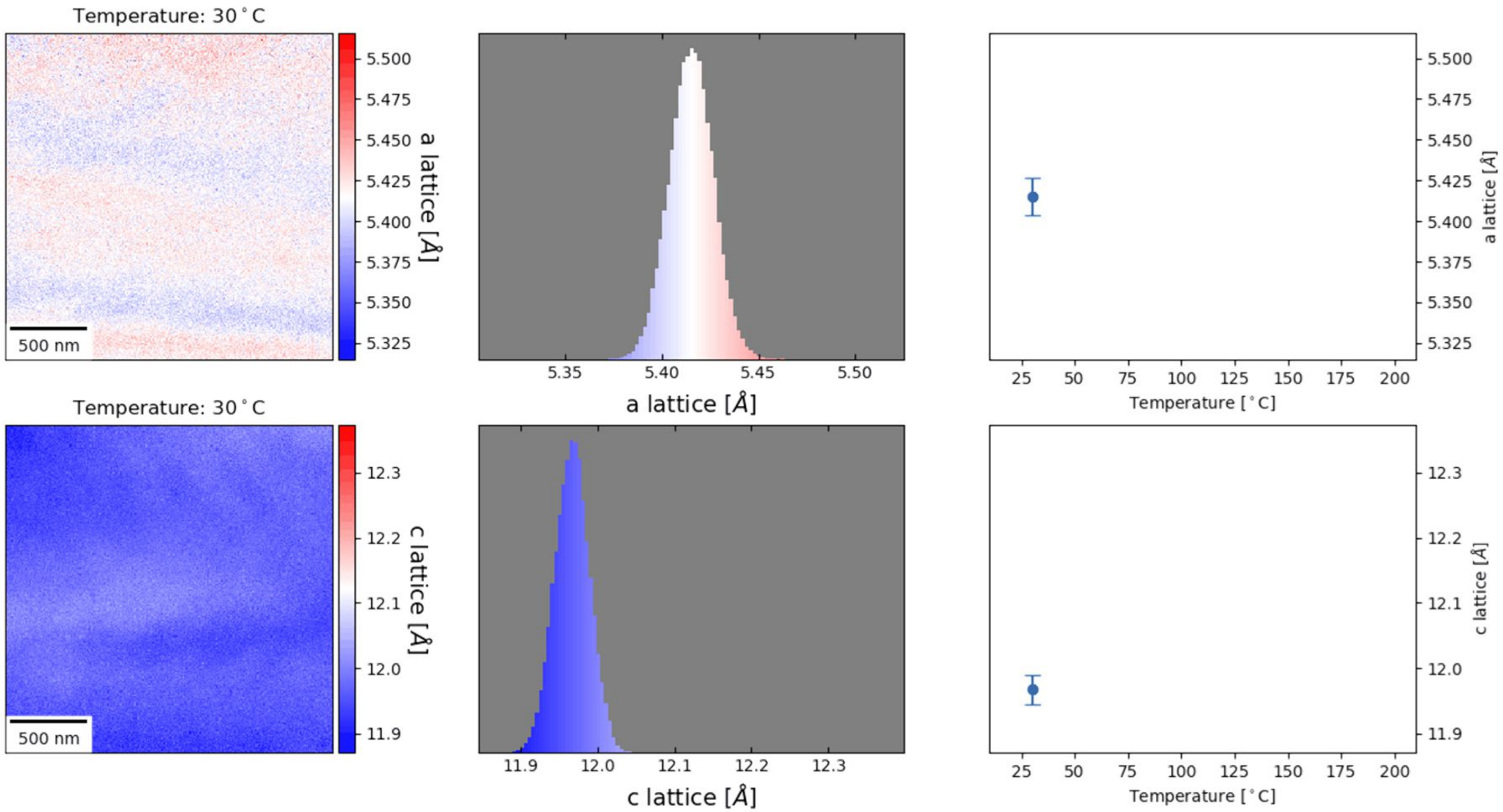
Temperature induced phase transition





N. Gauquelin, F.Forte et al., Nano Letters (2023) https://doi.org/10.1021/acs.nanolett.3c00574

Experiment: Temperature experiment



N. Gauquelin, F.Forte et al., Nano Letters (2023) https://doi.org/10.1021/acs.nanolett.3c00574

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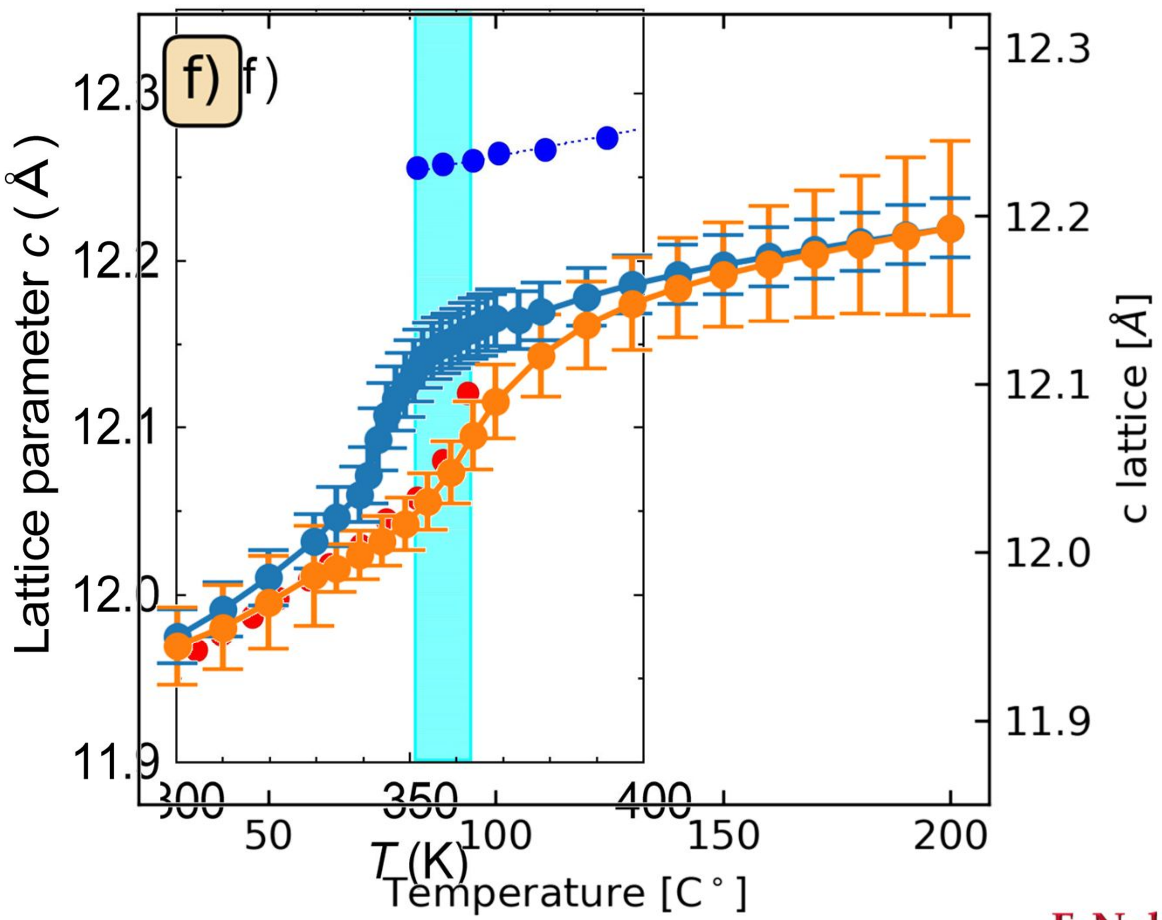
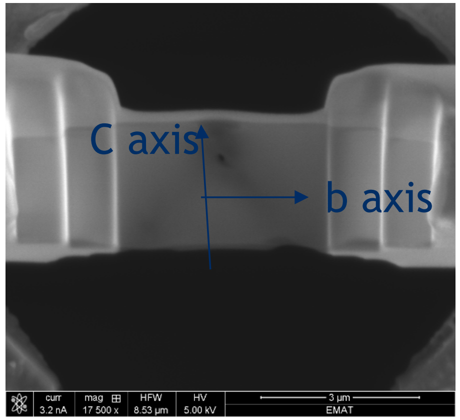
Experiment: Temperature experiment



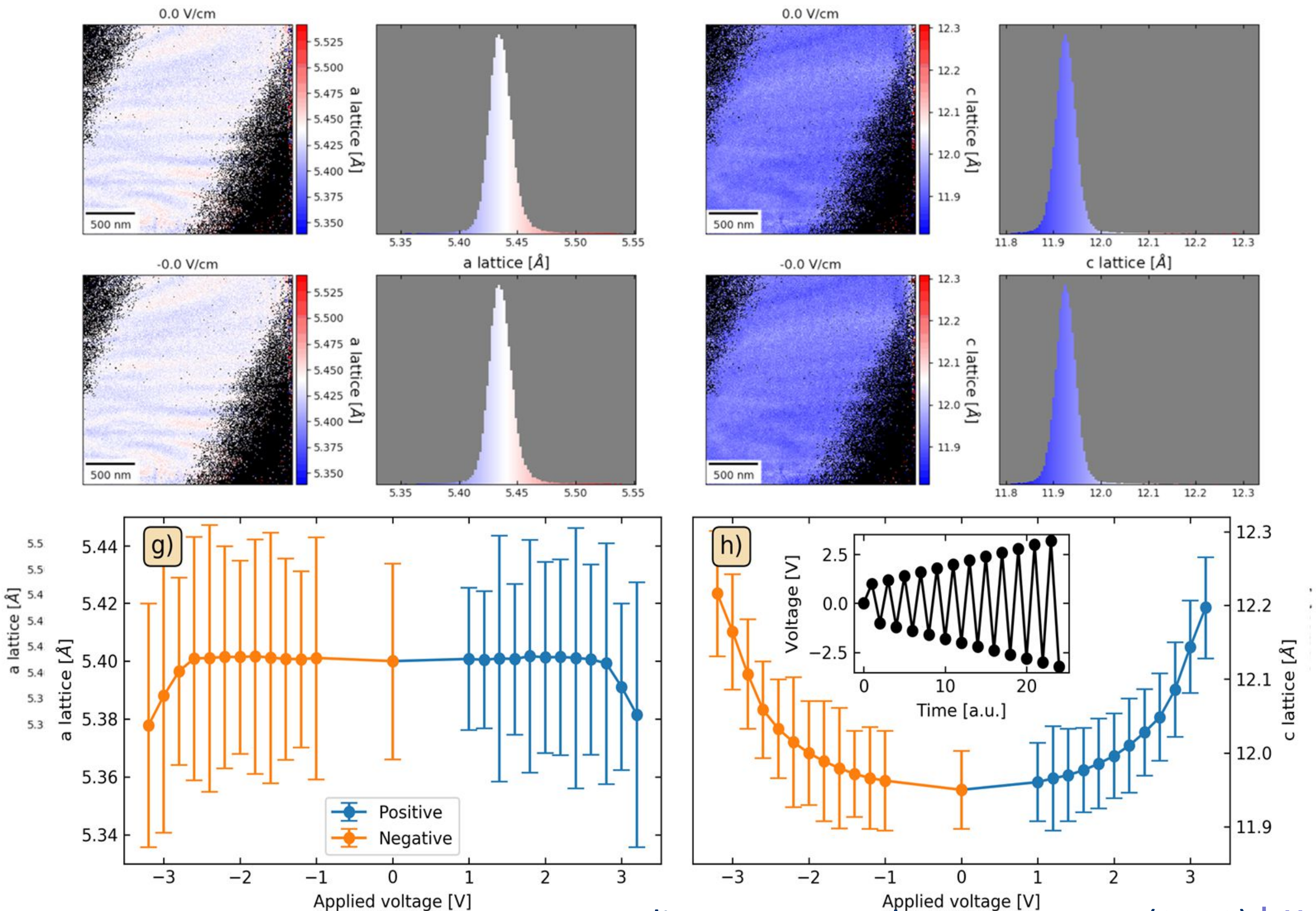
We do not reach the same value of c-lattice parameter than Nakamura et al.

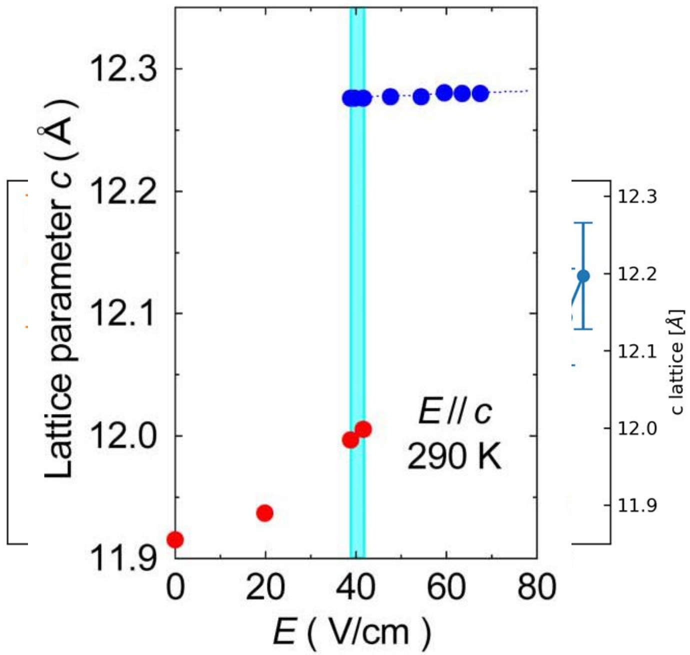
Strain induced by the geometry



F. Nakamura et al., Sci. Rep. 3, 2536 (2013)

Biasing experiment





Voltage sequence:
0 → 1000mV → -1000mV → 1200mV →… →-3200mV

Conclusion: No stripe pattern is observe
using this voltage sequence

N. Gauquelin, F.Forte et al., Nano Letters (2023) https://doi.org/10.1021/acs.nanolett.3c00574

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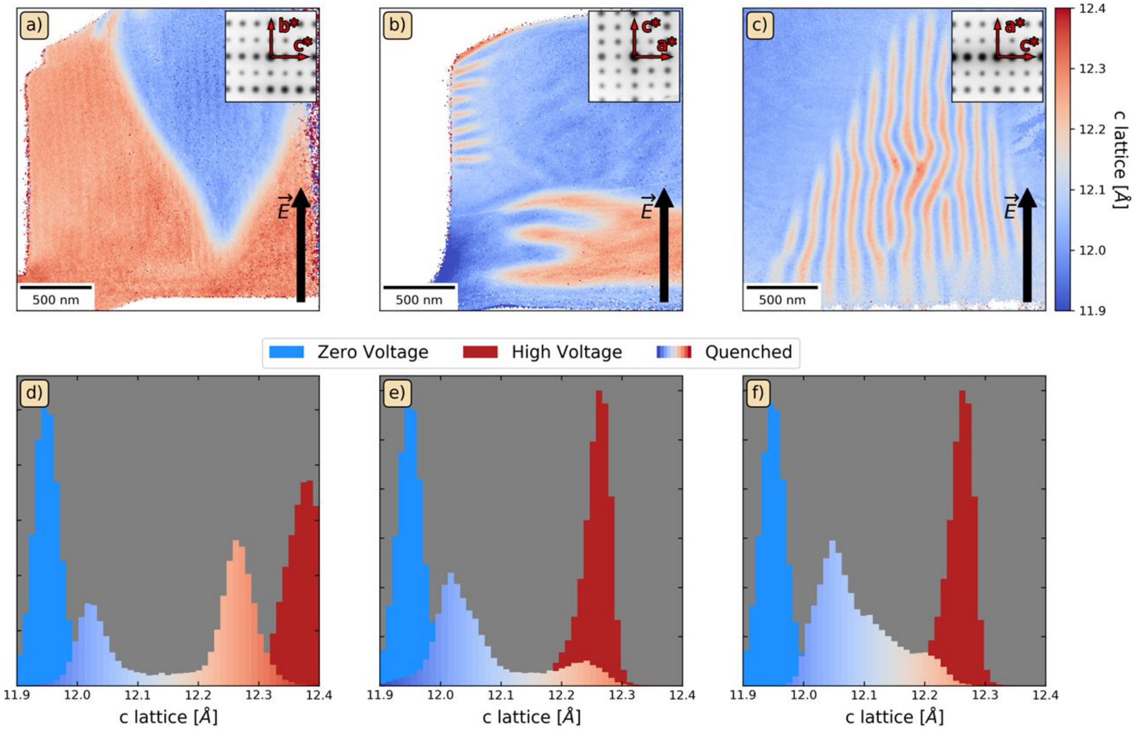
After electric field quench different orientations

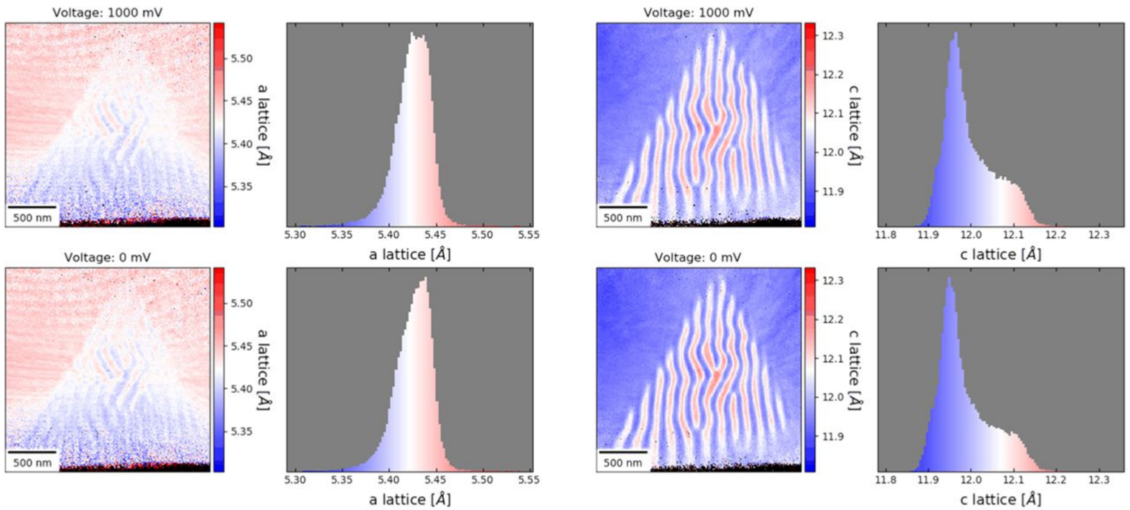
- **After quenching electric field, different patterns arise when changing crystal orientation with respect to electric field**

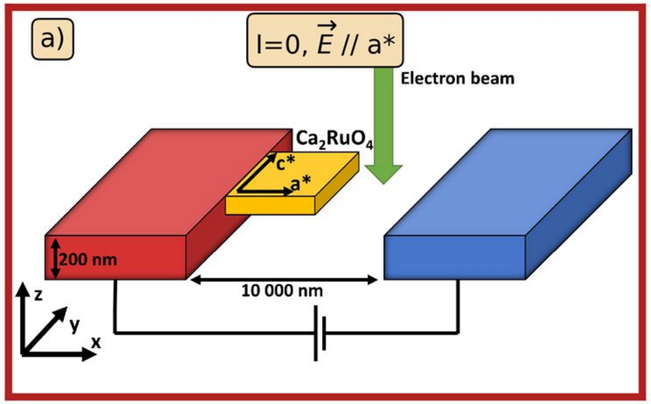


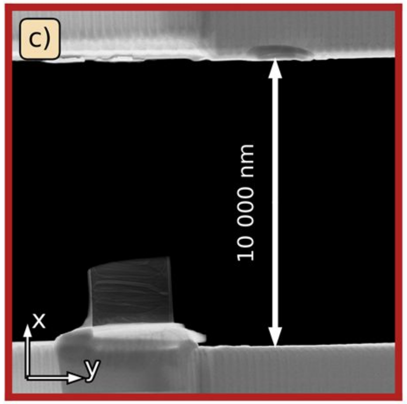
N. Gauquelin, F.Forte et al., Nano Letters (2023) https://doi.org/10.1021/acs.nanolett.3c00574

Evolution lattice parameters

- **4D STEM when applying voltage and quenching after**
  1. row: By applying voltage domains can be modified
  2. row: Quench after brings back to stripe phase







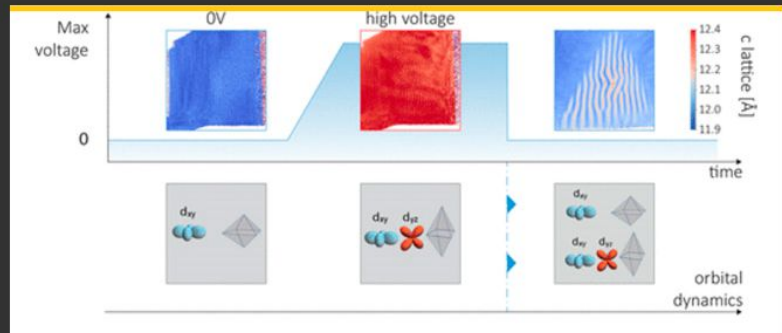
ON THE COVER

Cover by: Gert De Bont

NANO LETTERS

Pattern Formation by Electric-Field Quench in a Mott Crystal

Nicolas Gauquelin*, Filomena Forte, **Daen Jannis**, Rosalba Fittipaldi, Carmine Autieri, Giuseppe Cuono, Veronica Granata, Mariateresa Lettieri, Canio Noce, Fabio Miletto-Granozio, Antonio Vecchione*, **Johan Verbeeck**, and Mario Cuoco



Article | Published: 13 September 2023

Outline

- Introduction
  - Semiconductors metrology and its challenges
- Advances in tomography of nanoparticles
  - Electron tomography introduction
  - Conventional vs. Fast tomography
  - Atomic resolution tomography
  - SEEBIC as an alternative
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  - Fluctuation microscopy in ZrCu metallic glasses
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- Perspectives and outlook

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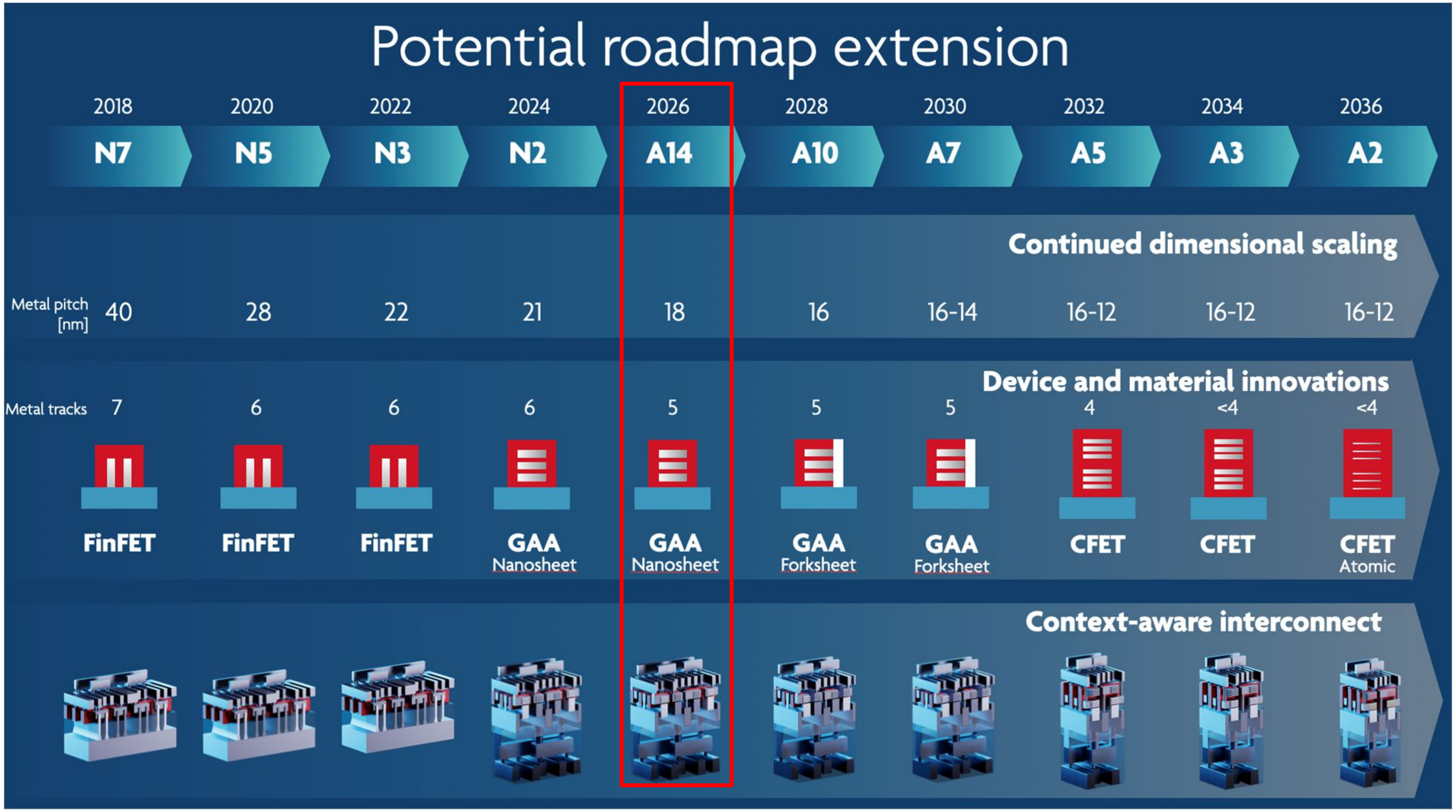
Increasing complexity of semiconductor technology



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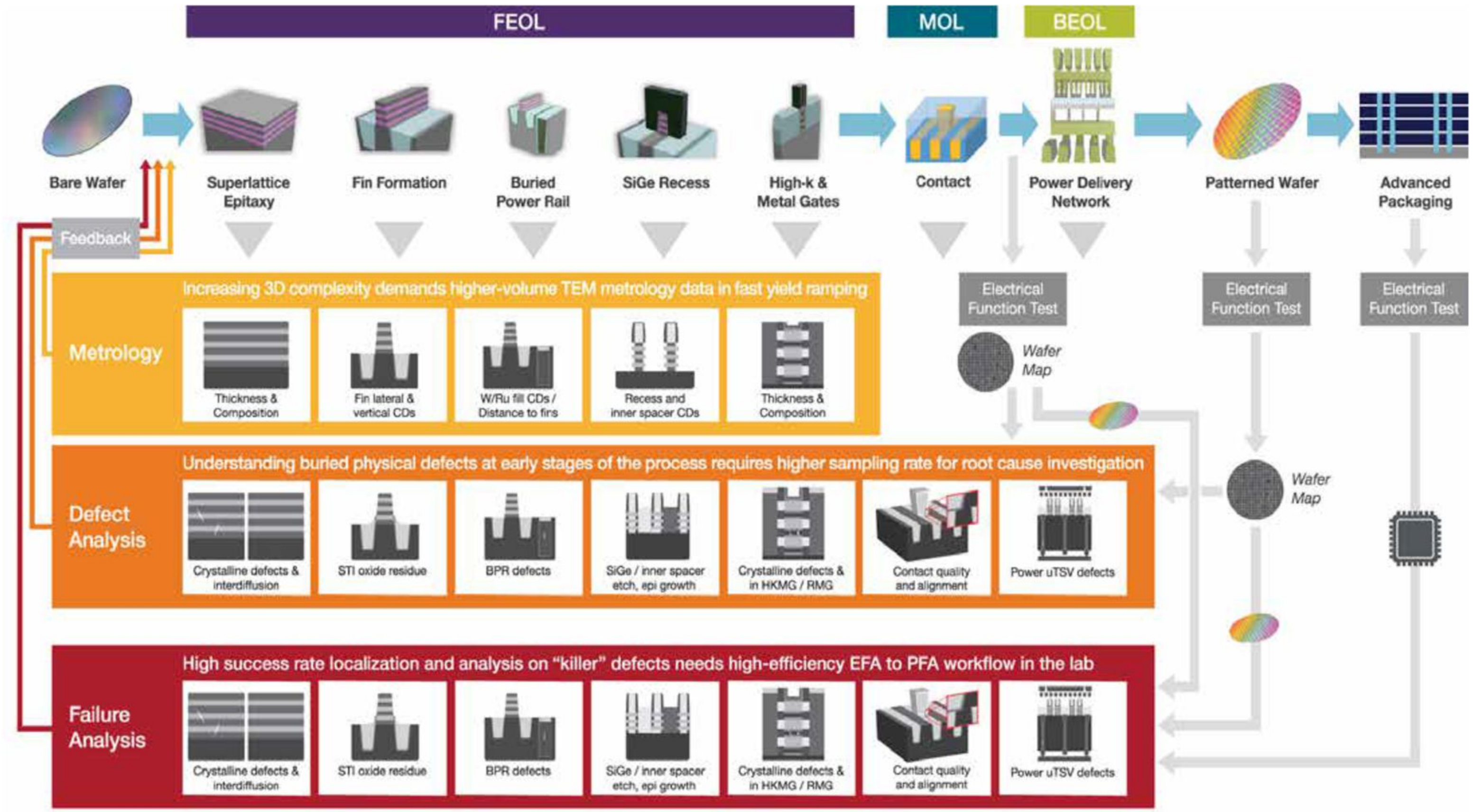
From IMEC

Challenges in metrology of GAA-FET



From Thermofisher Scientific White Paper (2024)

pMOS GAA Si NW-FET sample

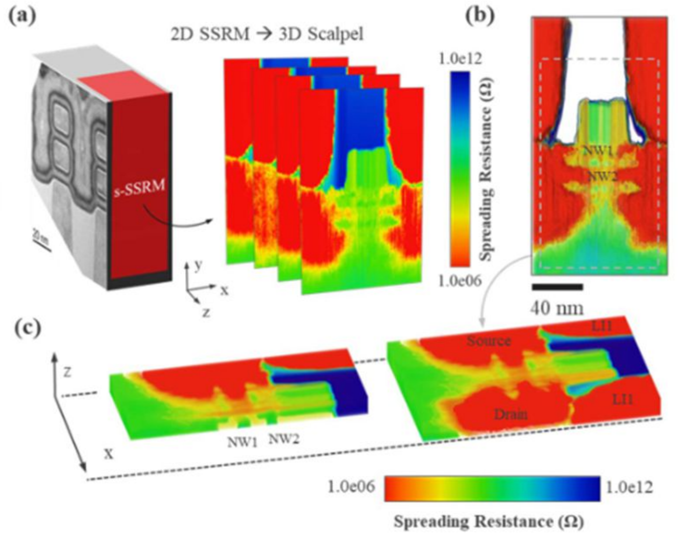


Fig. 5. 3D resistance map obtained by scalpel SSRM technique stacking SSRM 2D images for the pMOS GAA Si NW-FET. Highly conductive volumes are set in red, non-conductive in blue. Local interconnect, source and drain, as well as nanowires surrounded by the metal gates can be identified. Carrier distribution into NWs can be analyzed.

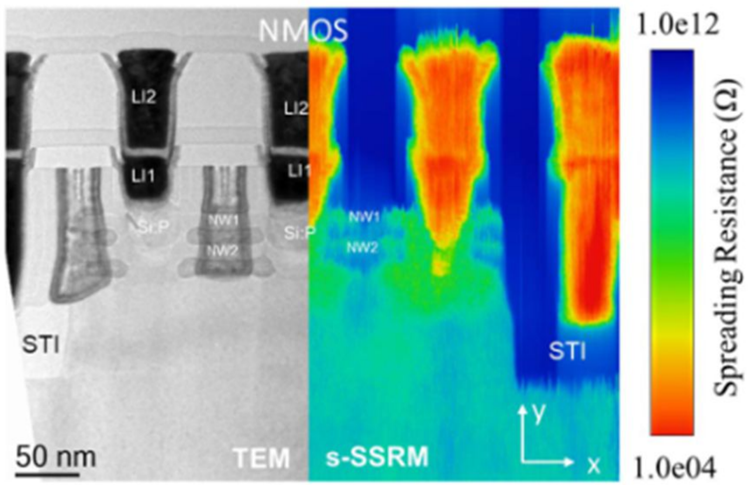
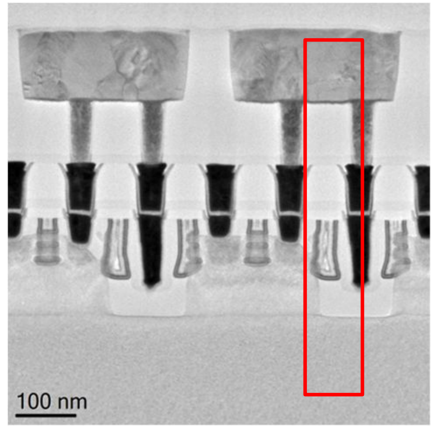
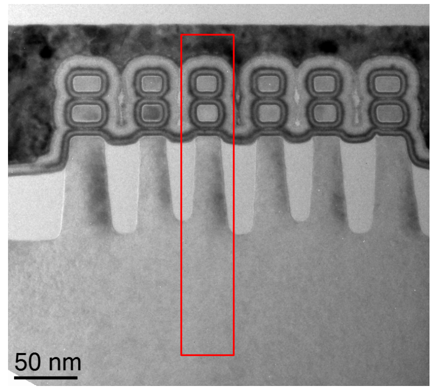


Fig. 6. (left) TEM cross-section along the NWs of the nMOS GAA Si NW-FET. (right) Scalpel SSRM 2D spreading resistance map at low contact force for the same device showing the diffusion of dopants into the NWs. Metals are set in red, highly conductive Si is in light green.





3D-carrier Profiling and Parasitic Resistance Analysis in Vertically Stacked Gate-All-Around Si Nanowire CMOS Transistors -
Conference: 2019 IEEE International Electron Devices Meeting (IEDM)

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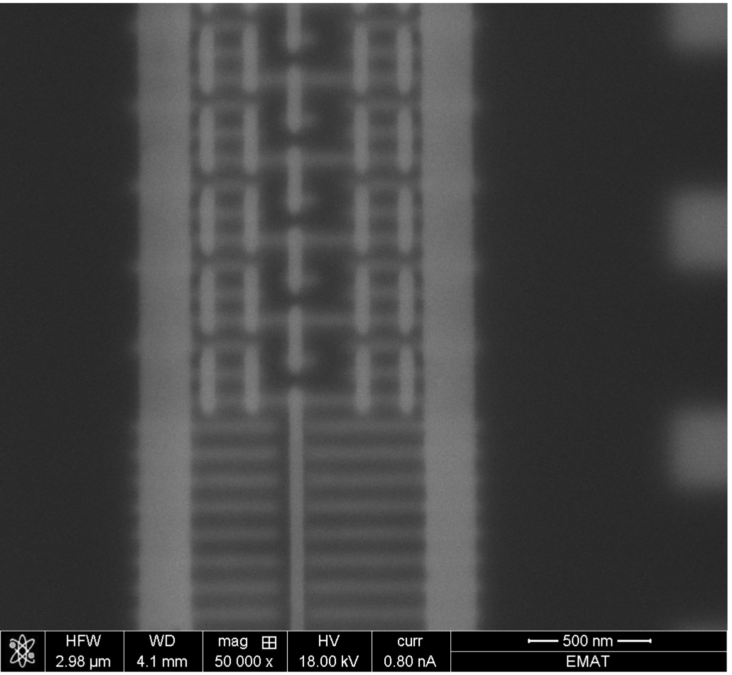
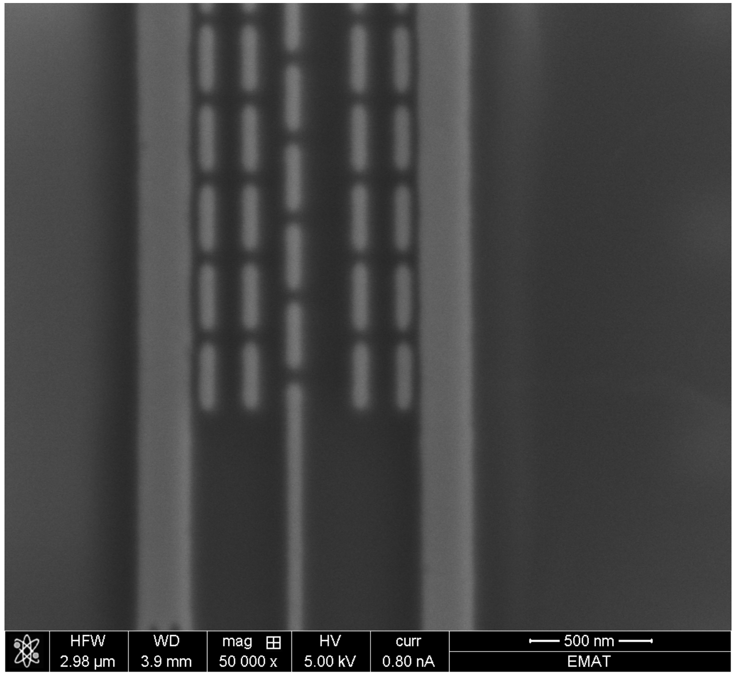
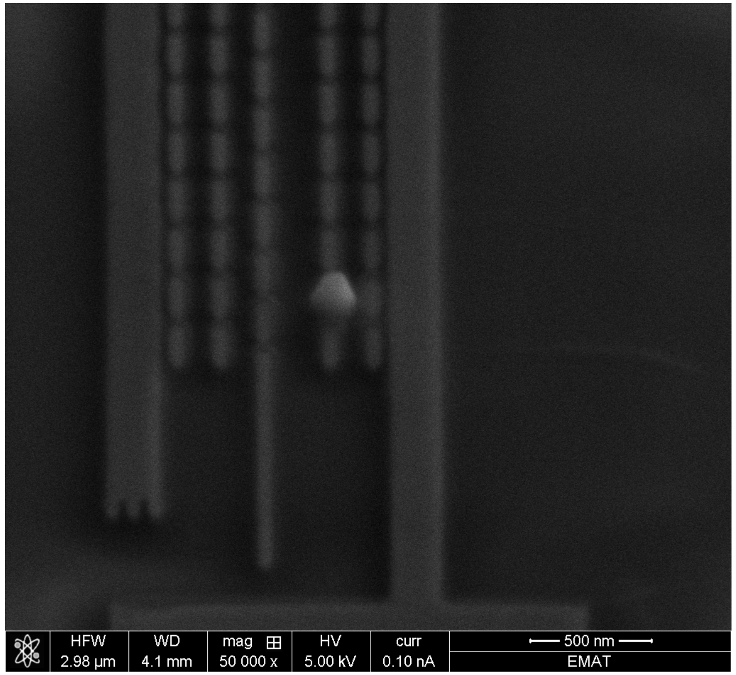
Preparation of the needle



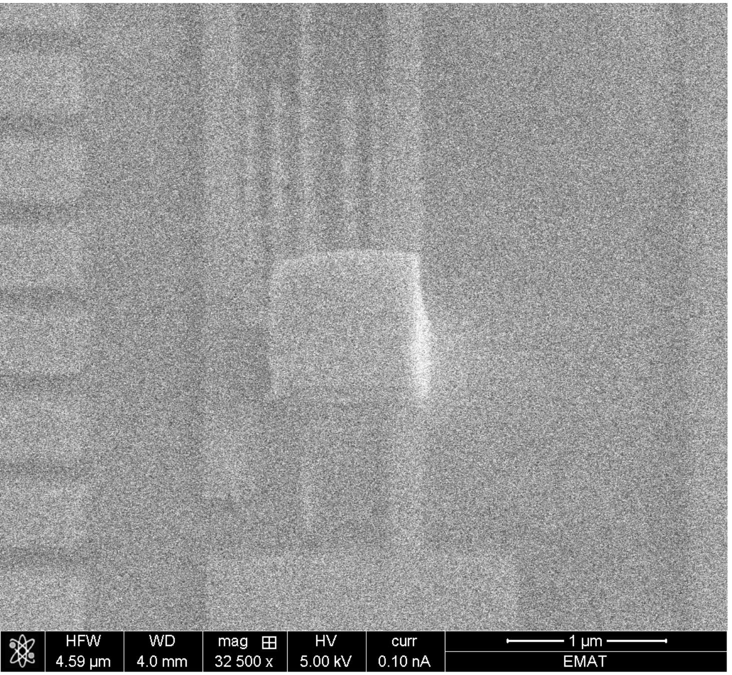


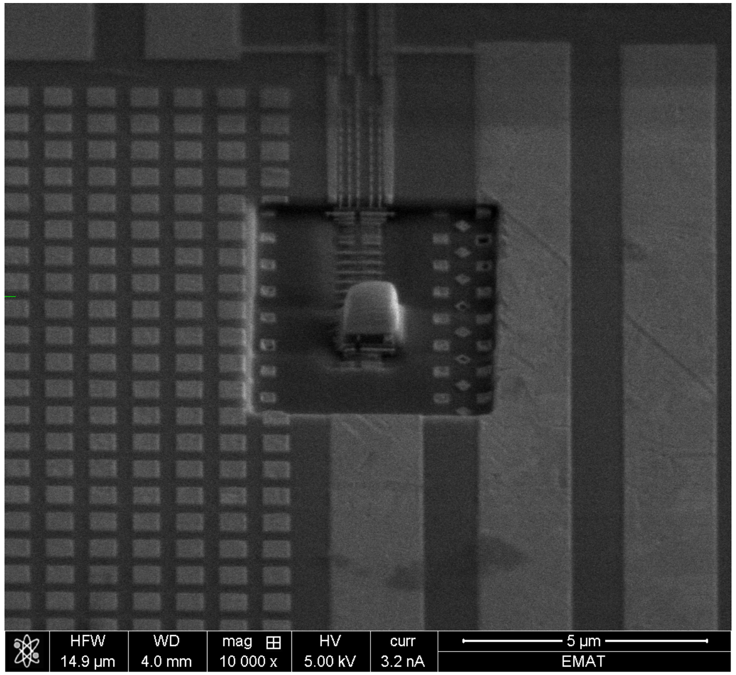


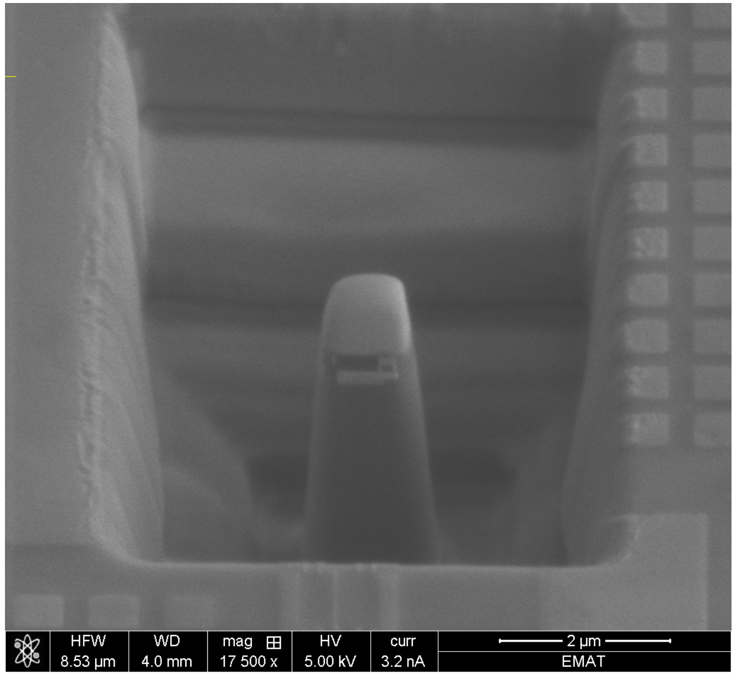
Imaging with different voltages → sensitivity to depth

Mark targeted location with e-beam Pt

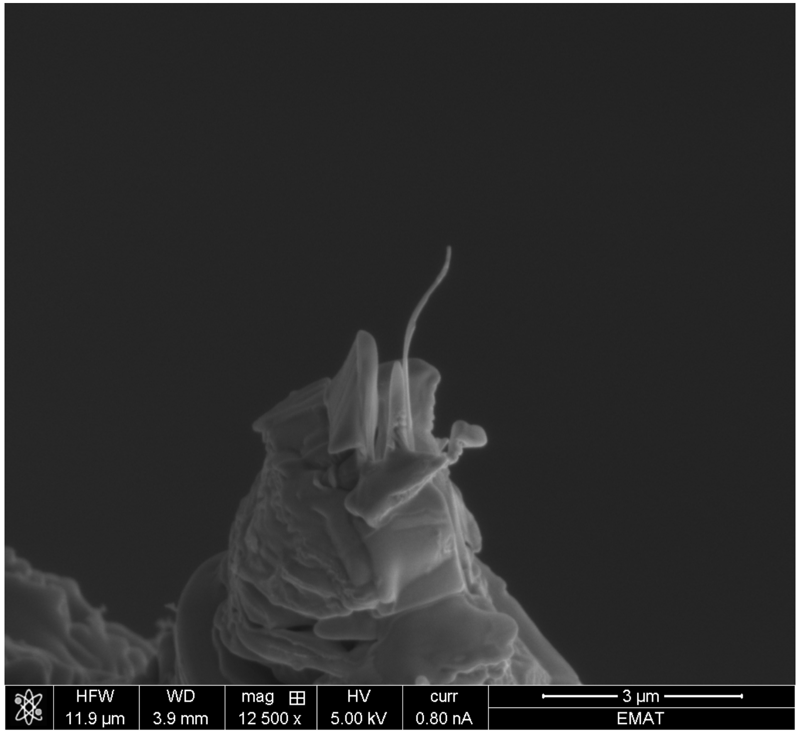
Preparation of the needle







Final prepared needle



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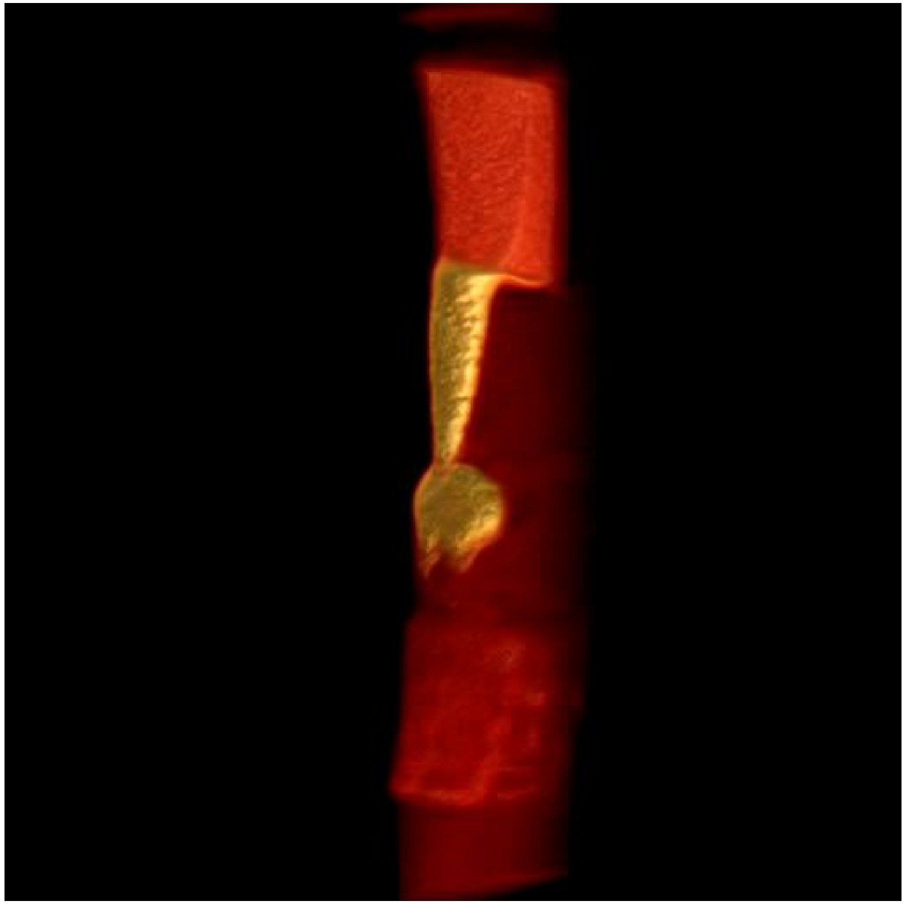
HAADF-tomography of the dummy gate



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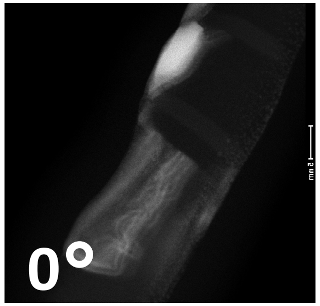
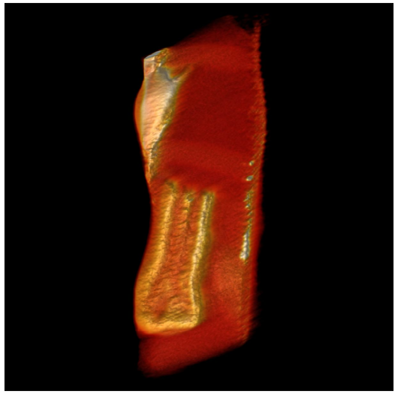
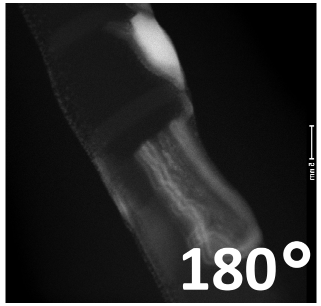
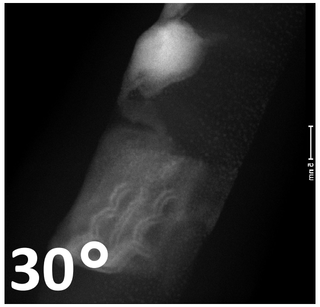
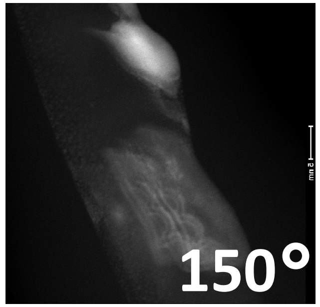
HAADF → 5 Gb

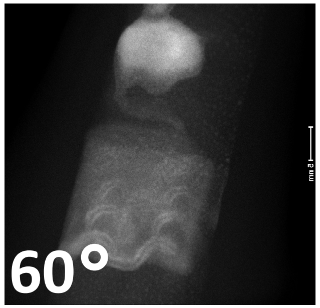


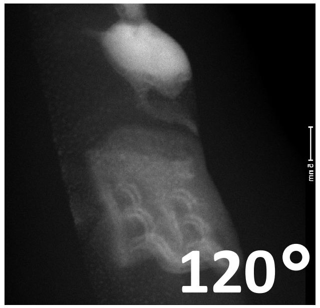


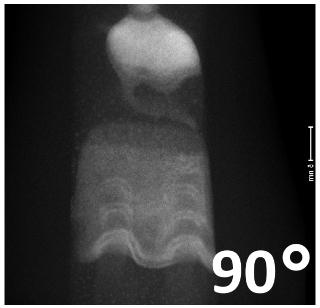












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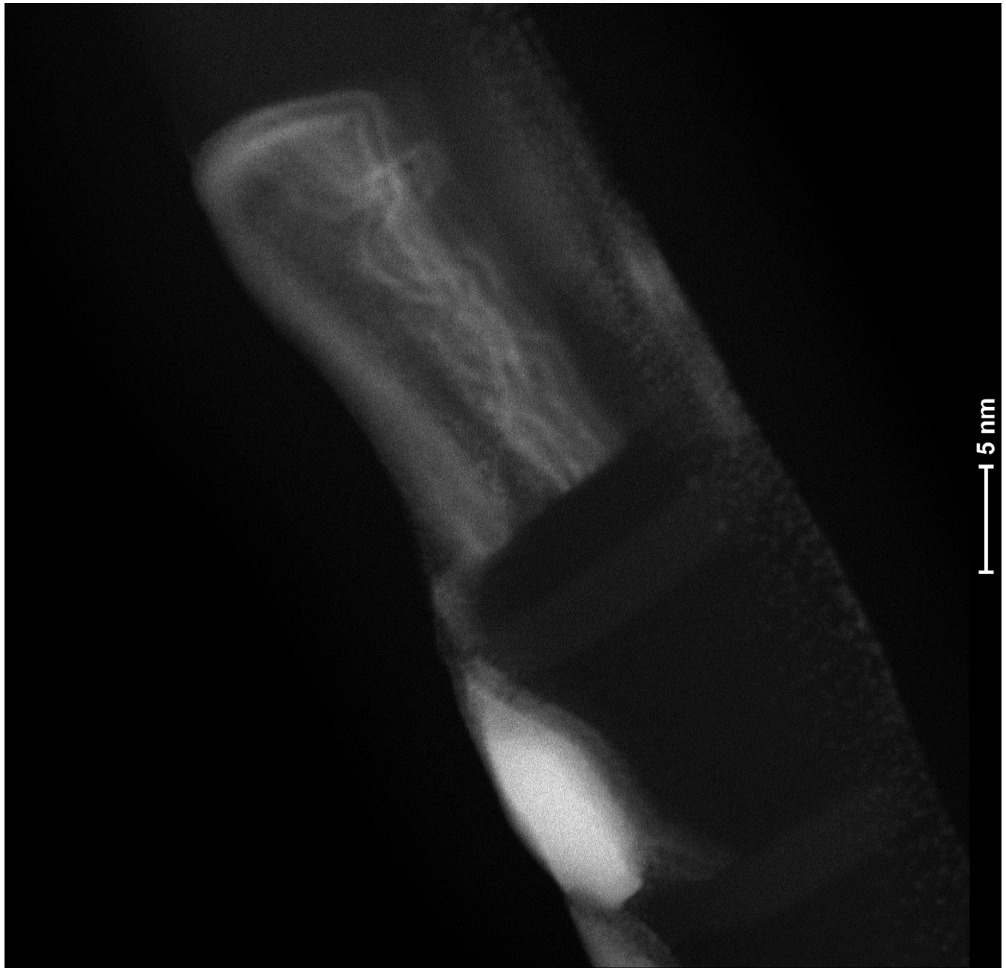

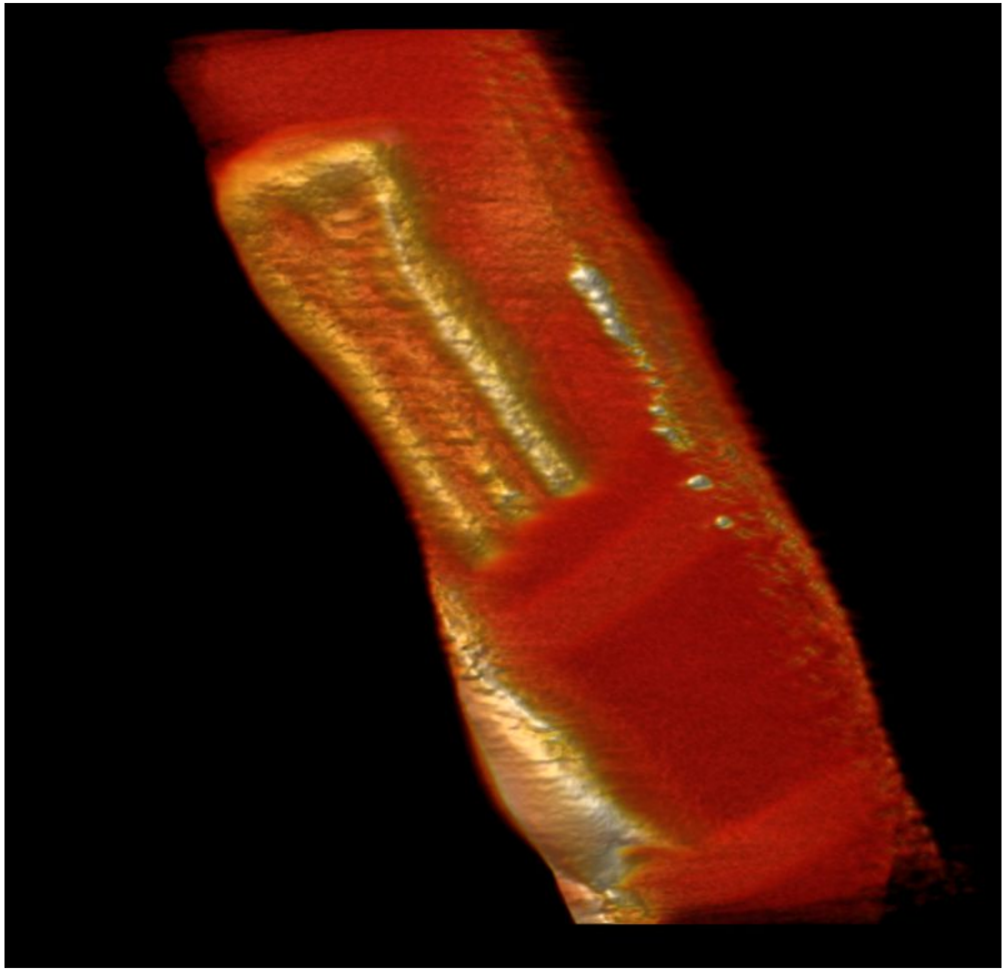

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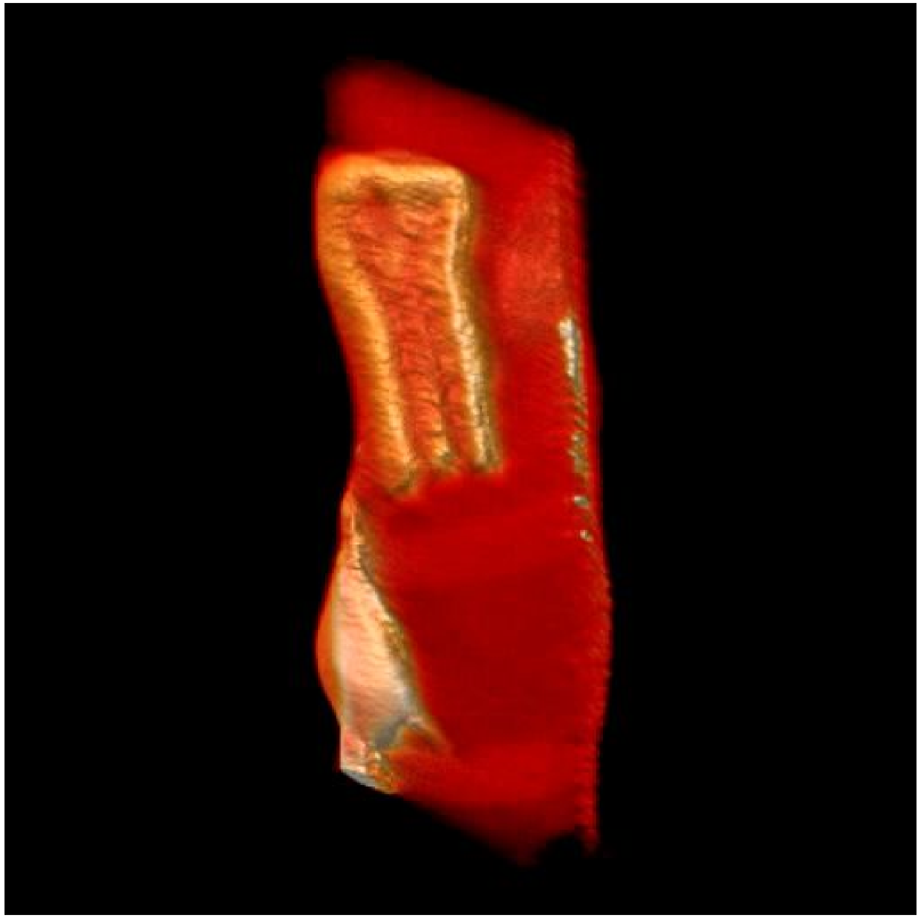

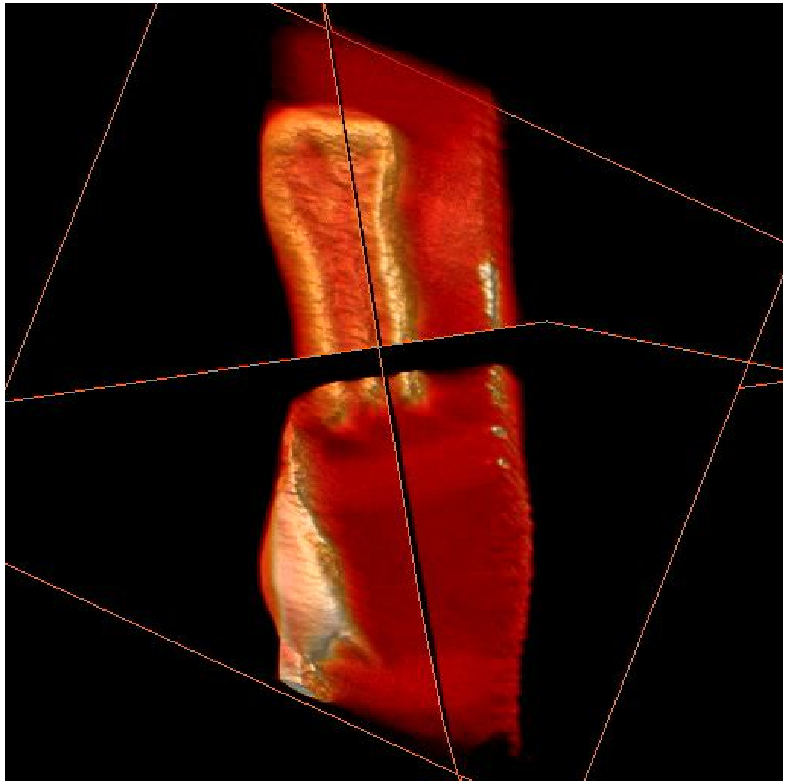

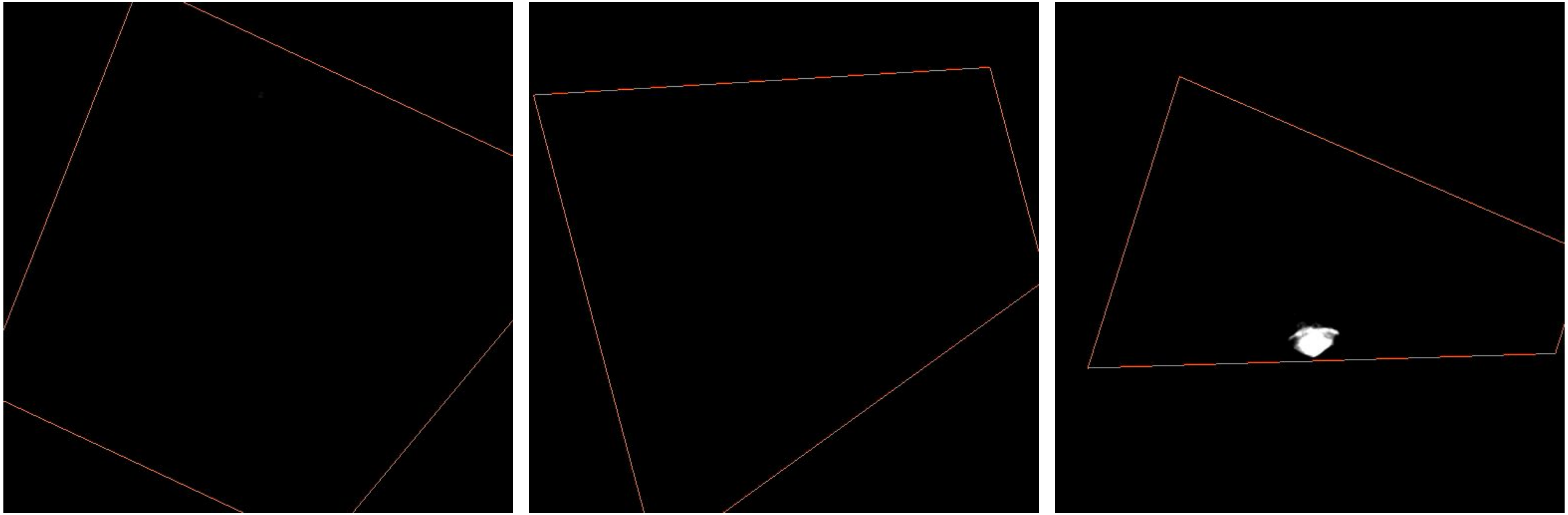

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Lamella EDX

D05 PMOS - EDS01

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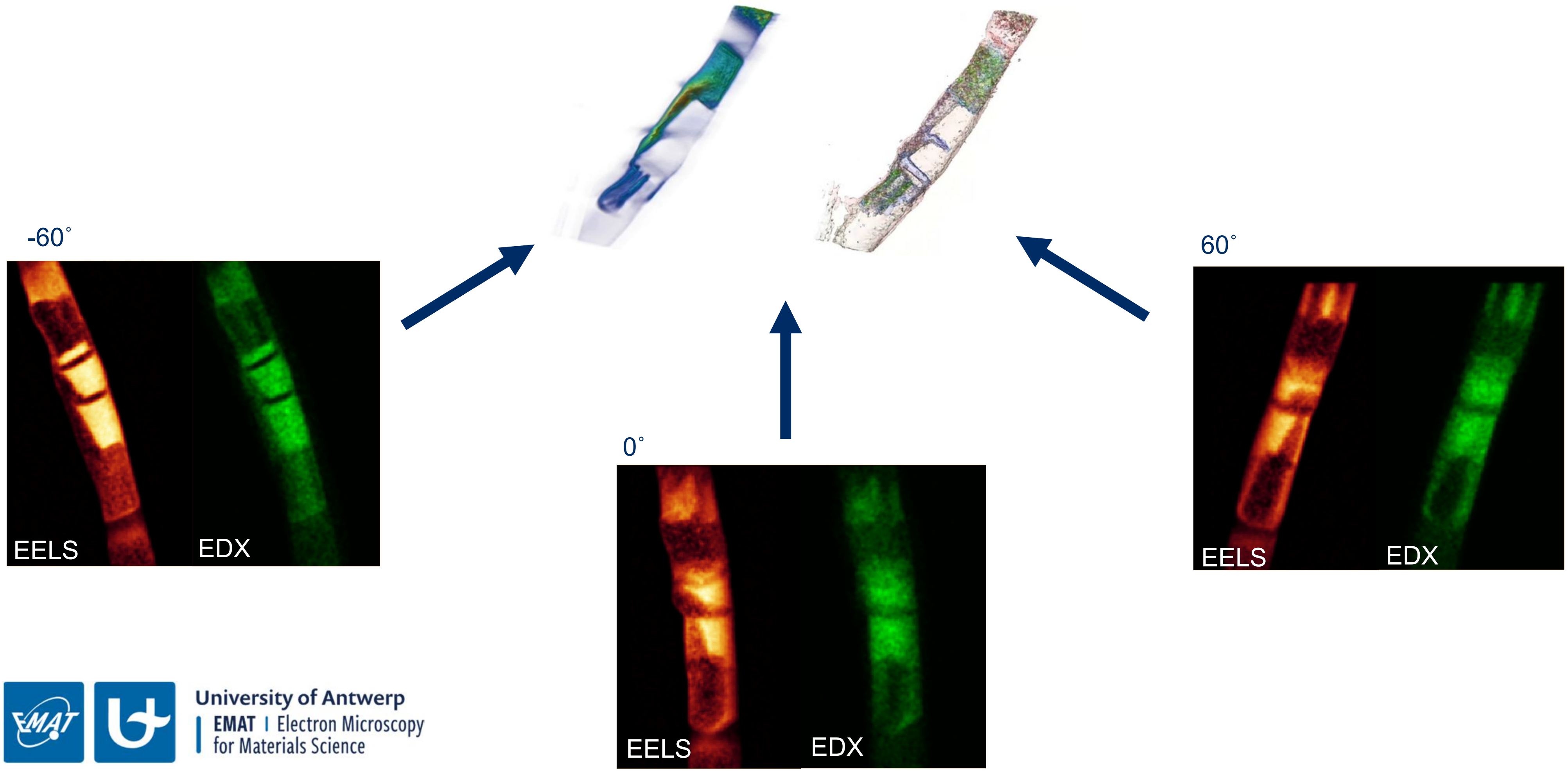
Courtesy of P. Favia - IMEC

Methodology

EELS+EDX → 60 Gb

EELS-EDX Tomography



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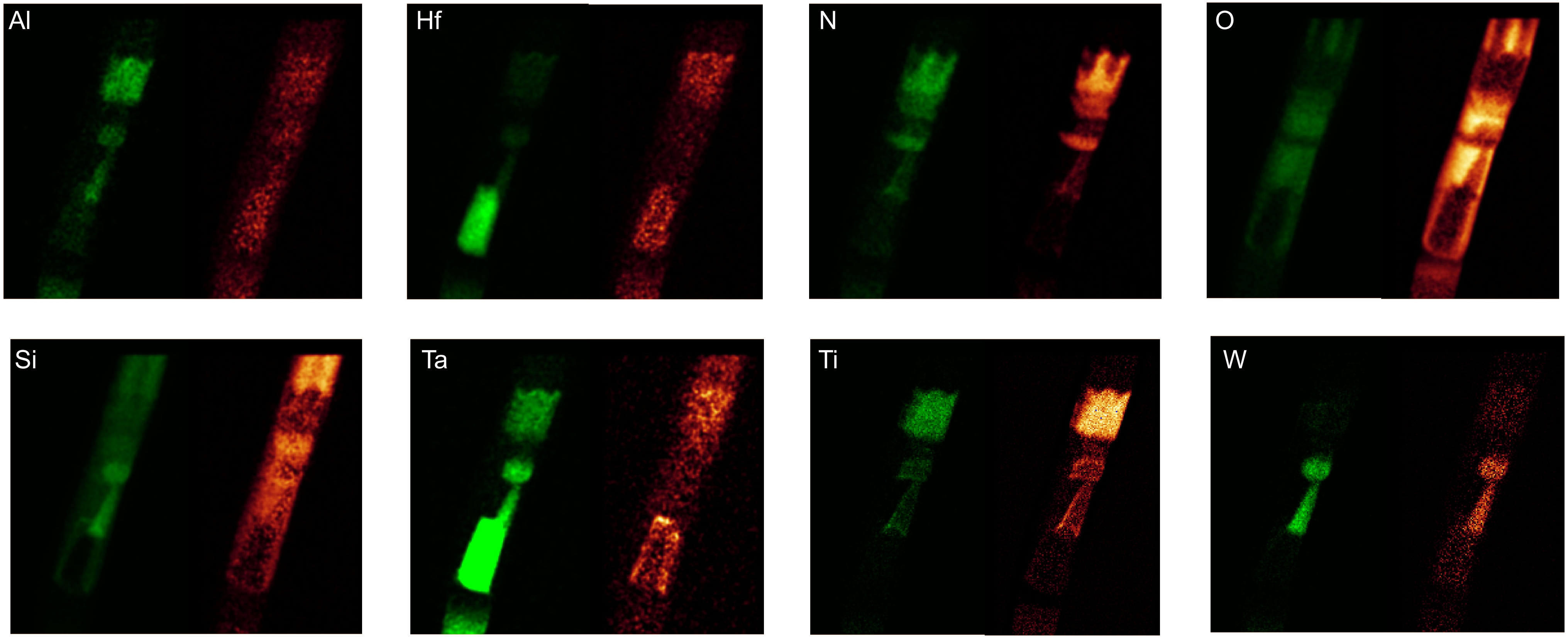
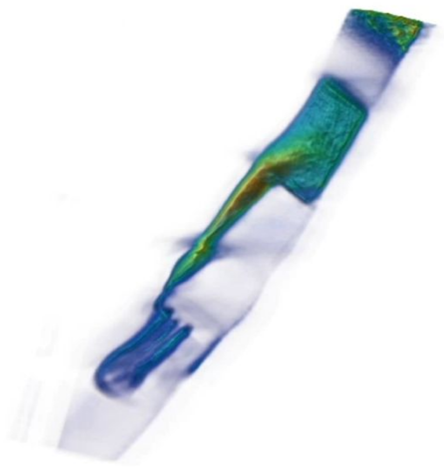
Signals

EDX EELS

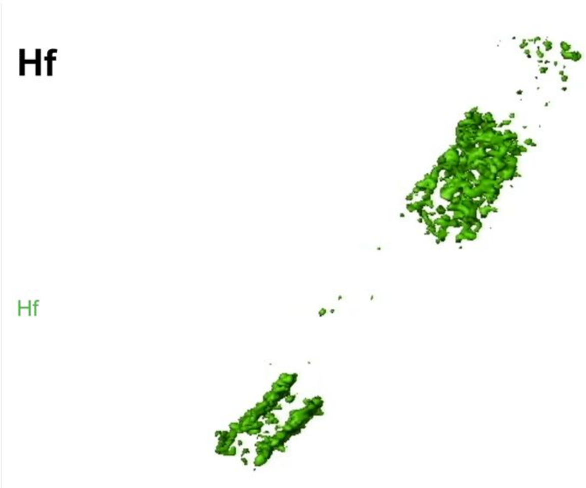


EELS-EDX Tomography

HAADF



EELS

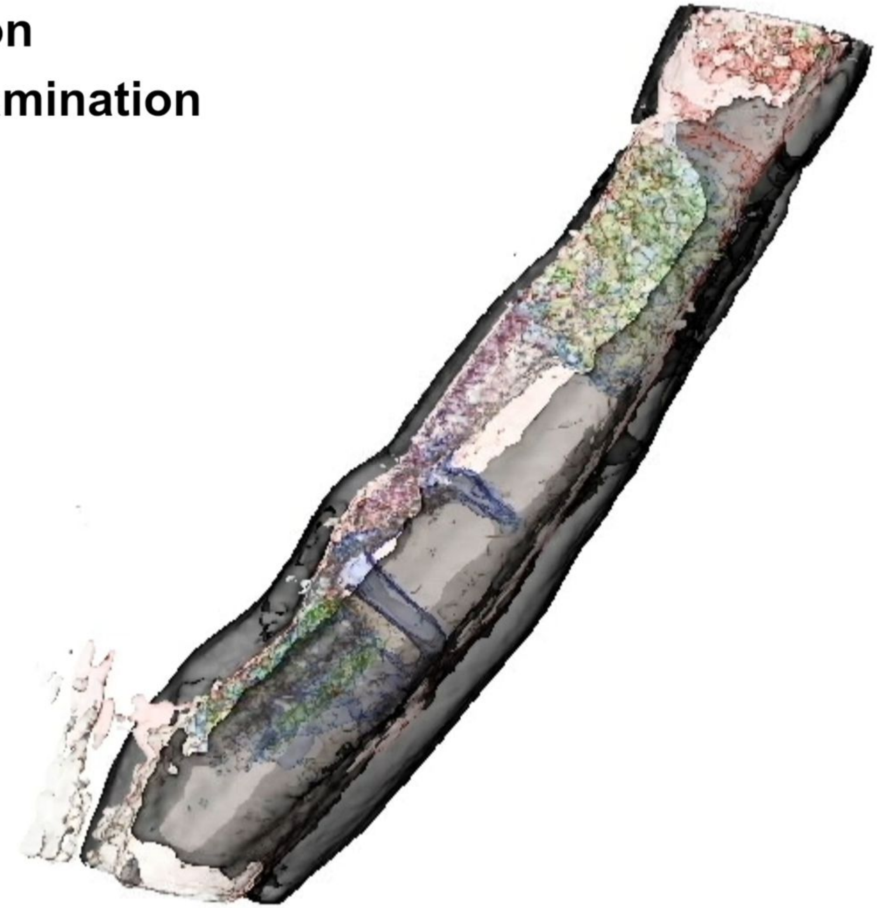


EDX

Carbon Contamination

Carbon Contamination

C
Hf
Ta
Ti
Al
W
N
Si
O



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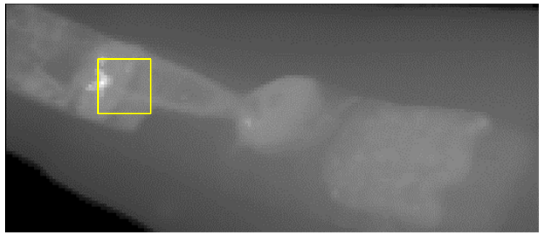
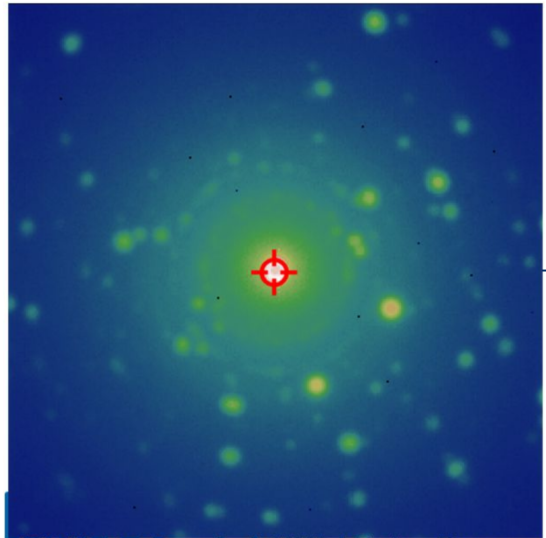
4DSTEM→ 1 Tb

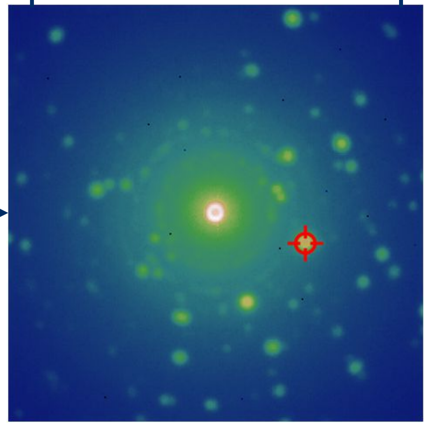
4DSTEM projection



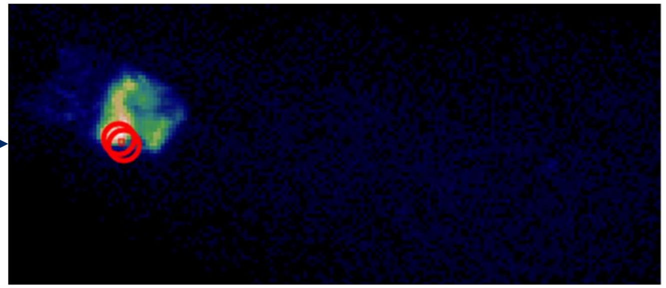
Sum of diffraction patterns datasets

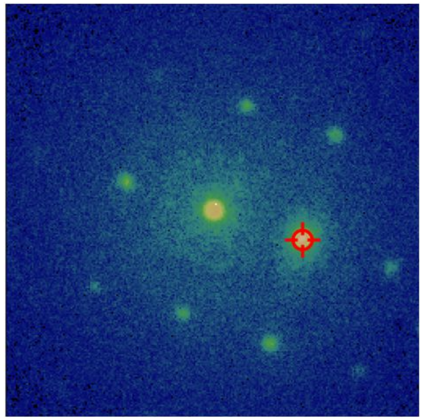


Map of a specific diffraction spot to identify the crystal phase on the sample



Study the crystal phase on individual diffraction patterns





Cu <100>

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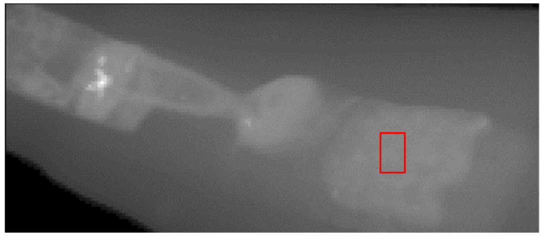
Map of a specific diffraction spot to identify the crystal phase on the sample

4DSTEM projection



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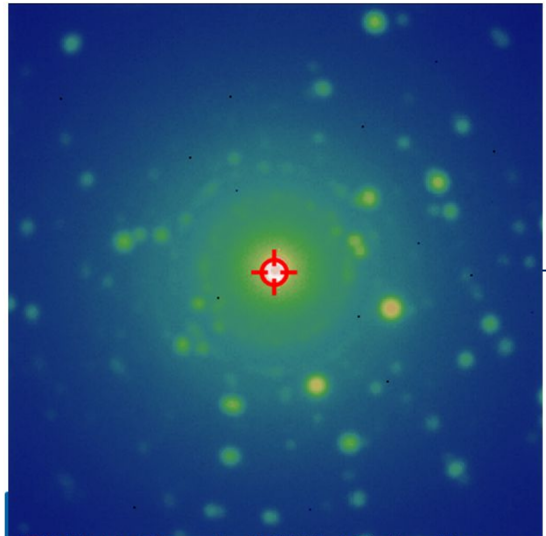
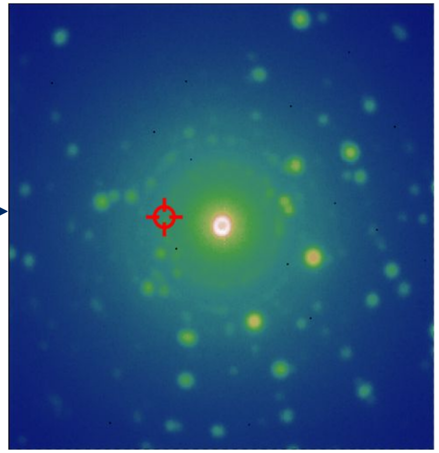
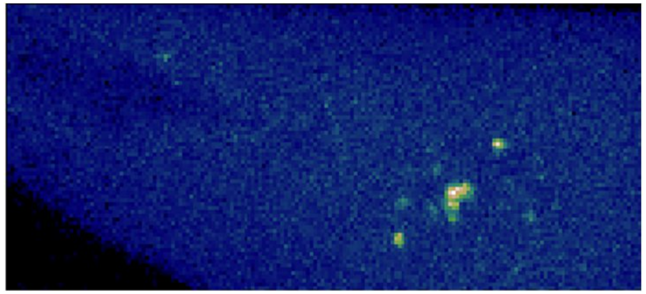
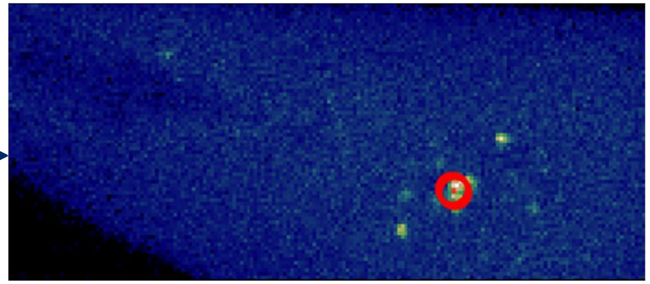
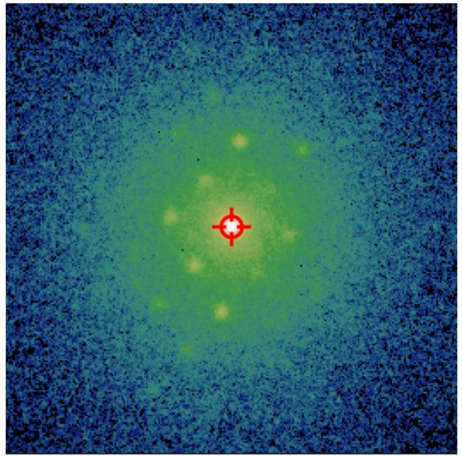






Study the cristal phase on individual diffraction patterns





Si <110>

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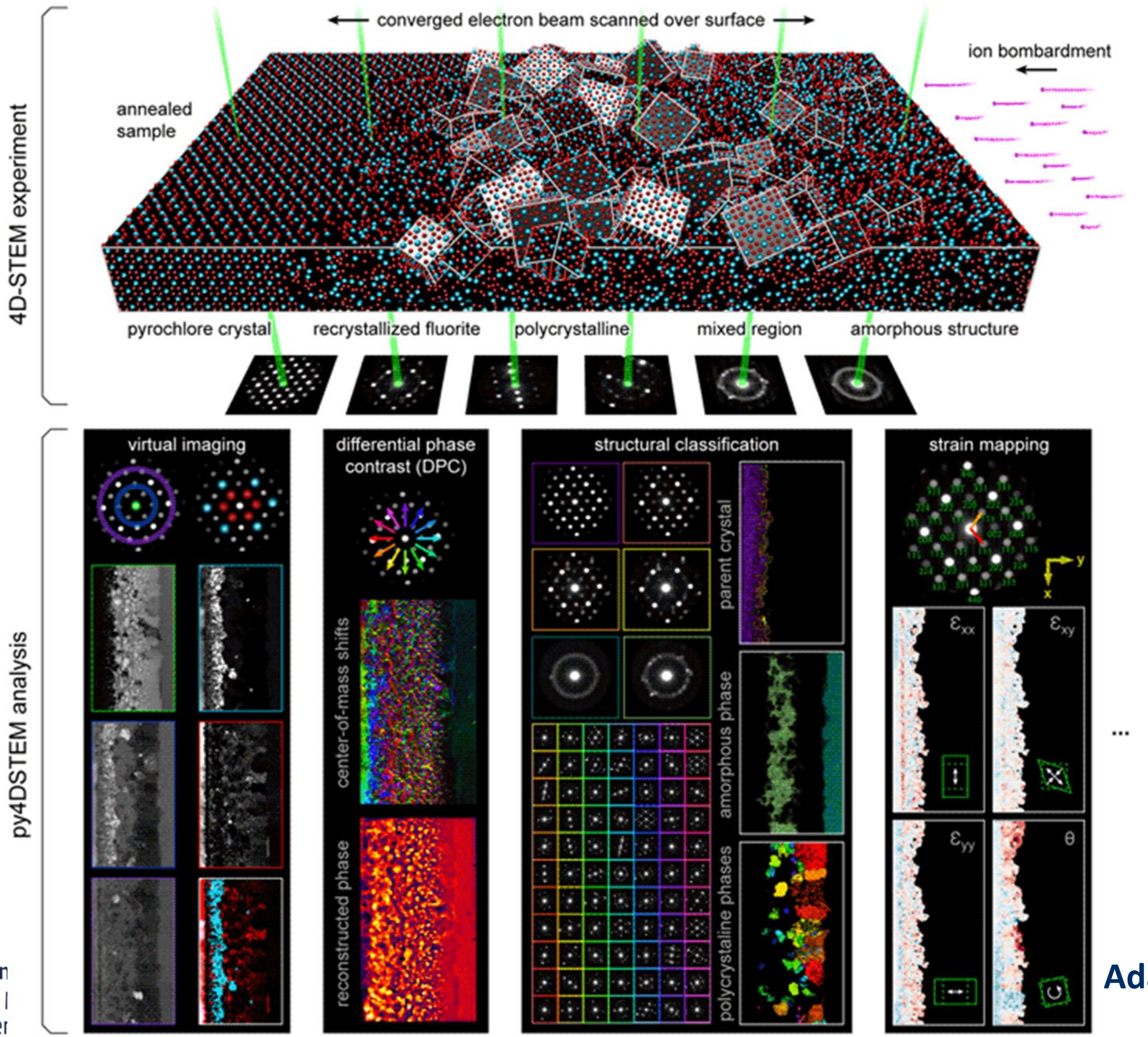
Possibilities in 4DSTEM



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Adapted from C. Ophus

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Outlook

- Tomography
  - became more accessible through fast tomo (faster) and developments in algorithms
  - Alternative SEEBIC available for shape determination
  - Multimodal
- 4DSTEM
  - Possible to determine stacking, defects, orientation of 2D materials
  - Possible to do in SEM
  - Can get information on crystalline and amorphous materials
- Tomography of complex semiconductor
  - Combination of different techniques possible and necessary
  - Sample preparation is the most critical step
  - Necessity to have 3D information

Acknowledgements

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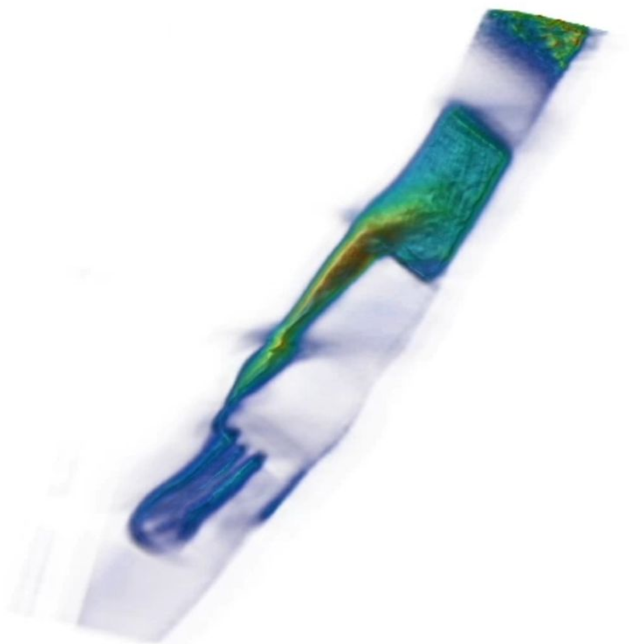
Thank you for your attention

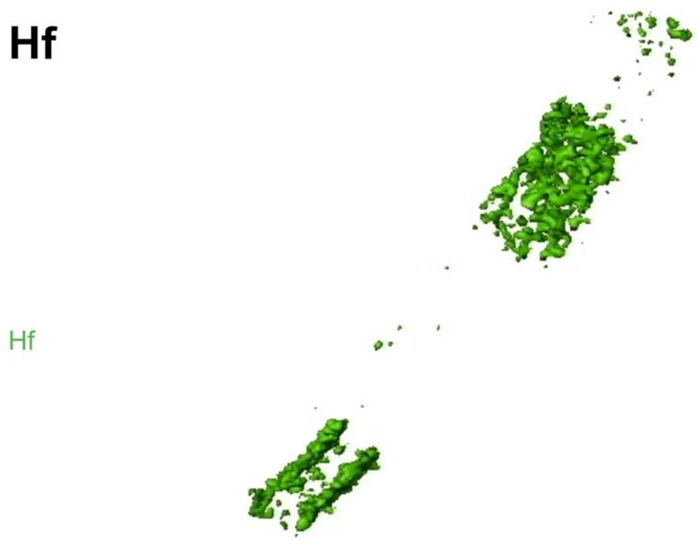
QUESTIONS ???

EELS Tomography

HAADF

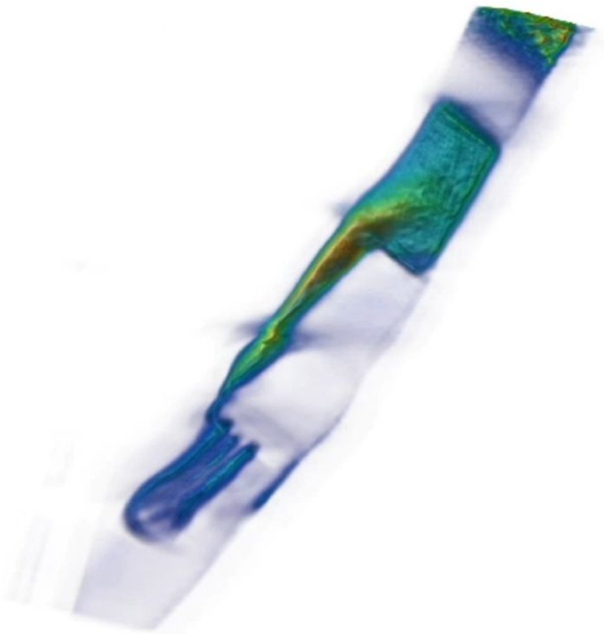


EELS



EDX Tomography

HAADF



EDX

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