ANALYTICAL METHODS FOR CHARACTERIZATION OF NANOMATERIALS

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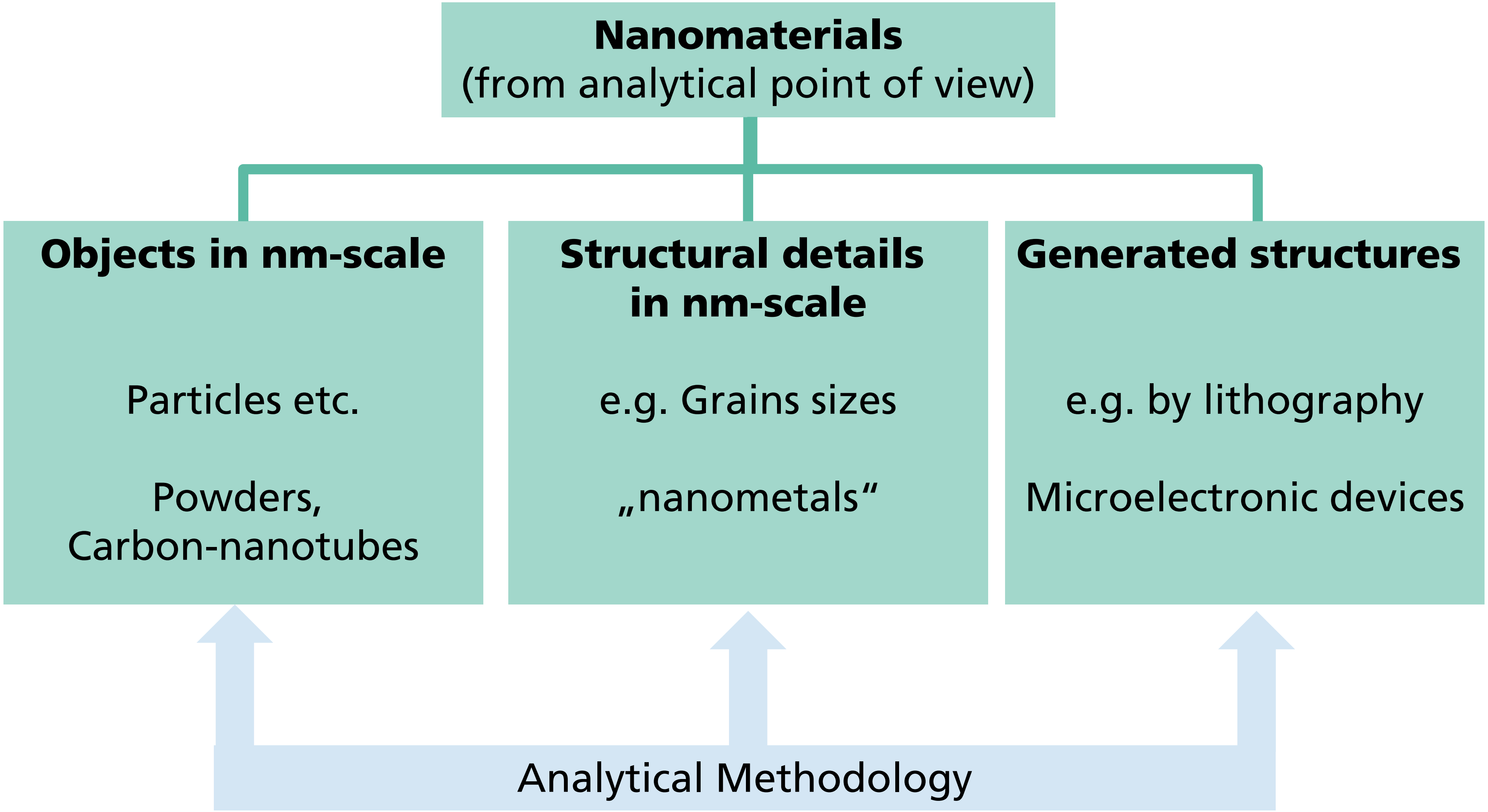
IKTS

Outline

- Introduction, Targets of characterization
    - Size, shape, structure, chemistry, crystallography
- Imaging methods
    - Electron microscopy, Ion microscopy, Atomic force microscopy
- Elemental and structural analysis
    - X-ray spectroscopy, Electron spectroscopy, X-Ray diffraction
- Summary and prospectives

Analytical methods for characterization of nanomaterials



Nanomaterials
(from analytical point of view)

Objects in nm-scale

Particles etc.

Powders,
Carbon-nanotubes

Structural details in nm-scale

e.g. Grains sizes

„nanometals“

Generated structures

e.g. by lithography

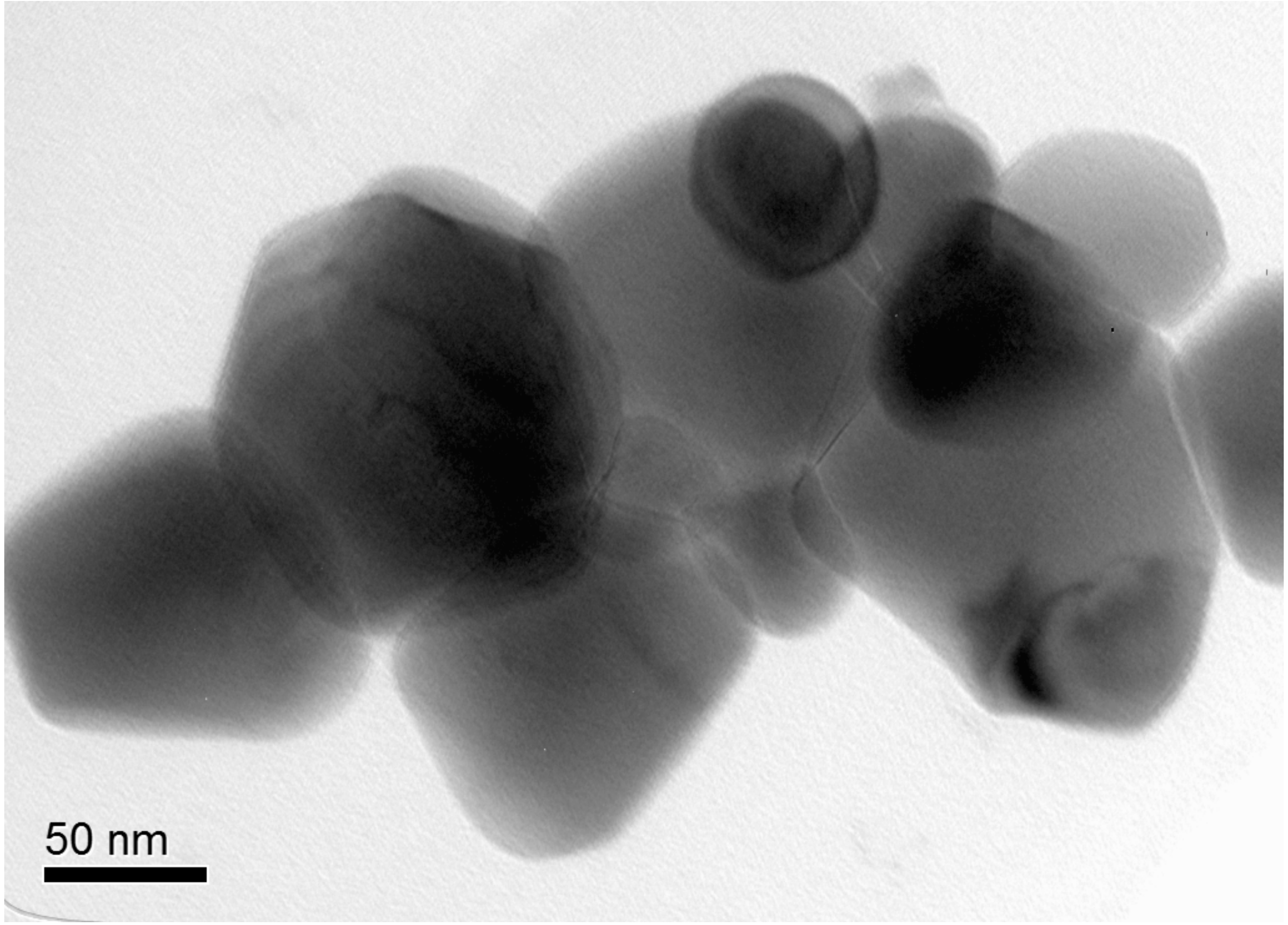
Microelectronic devices

Analytical Methodology

Analytical methods for characterization of nanomaterials

Powders, Particles etc.

TiAlN-
Particles etc.

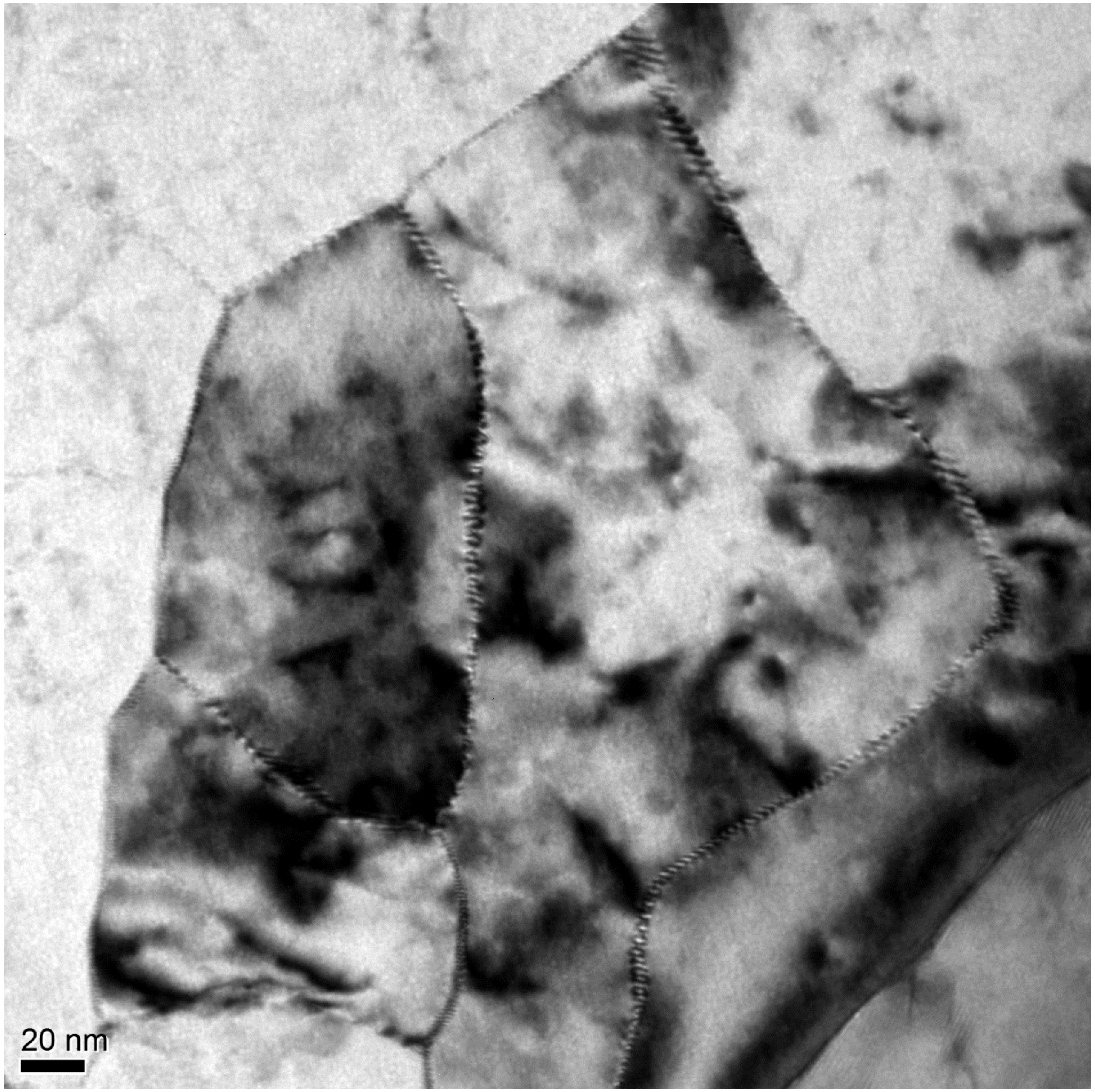


Analytical methods for characterization of nanomaterials

Structural details in nm-scale

e.g. Grains sizes
„nanometals“

Here: Al-alloy after
high pressure
deformation

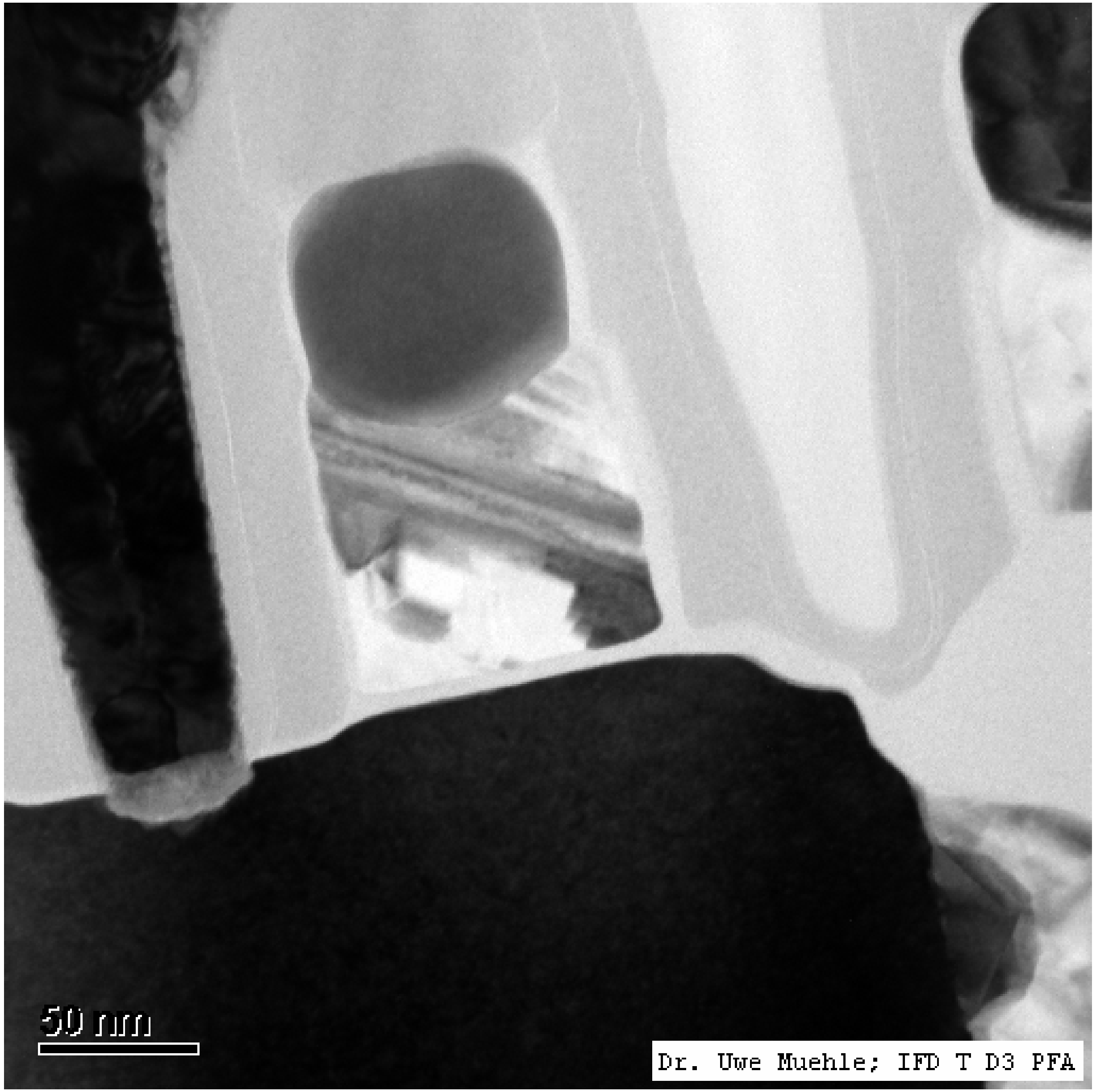


Analytical methods for characterization of nanomaterials

Generated structures

e.g. by lithography

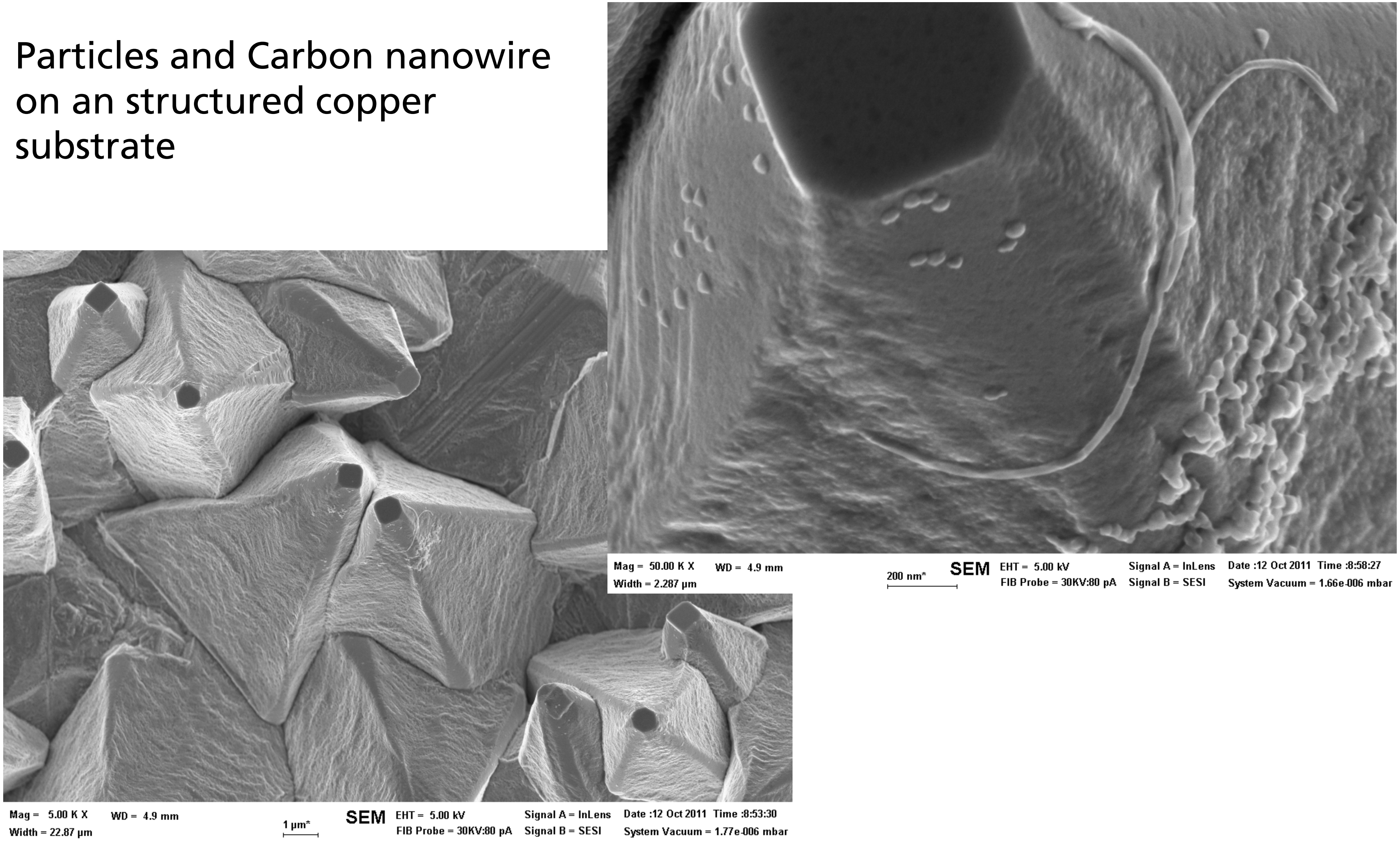
Microelectronic devices:
Transistor of a DRAM
memory cell



Analytical methods for characterization of nanomaterials

Target of analysis **Size**

Particles and Carbon nanowire on an structured copper substrate

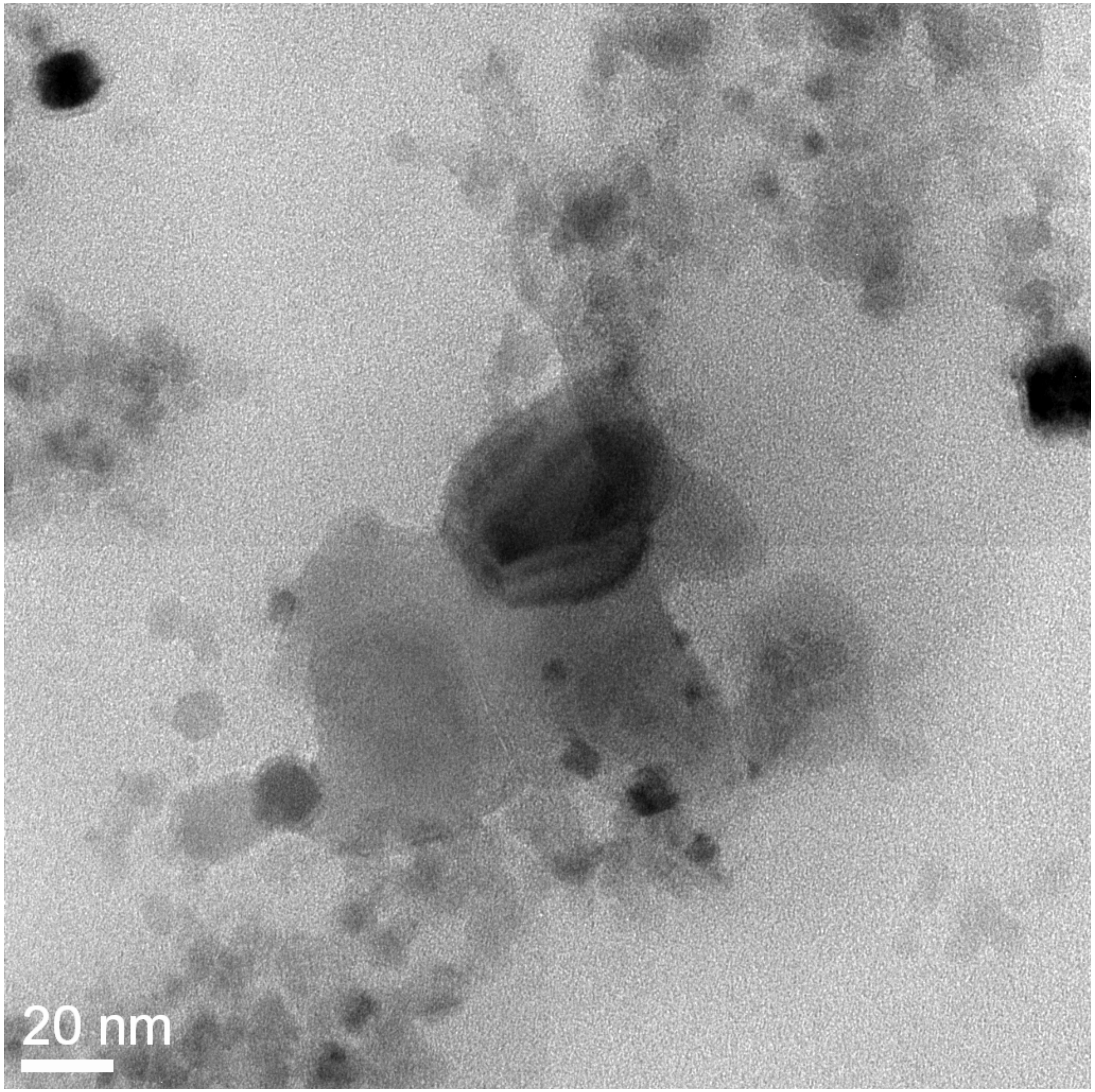


Analytical methods for characterization of nanomaterials

Target of analysis	**Size**

TiCN-particles:

Different fractures of size (+ shape and internal structure)



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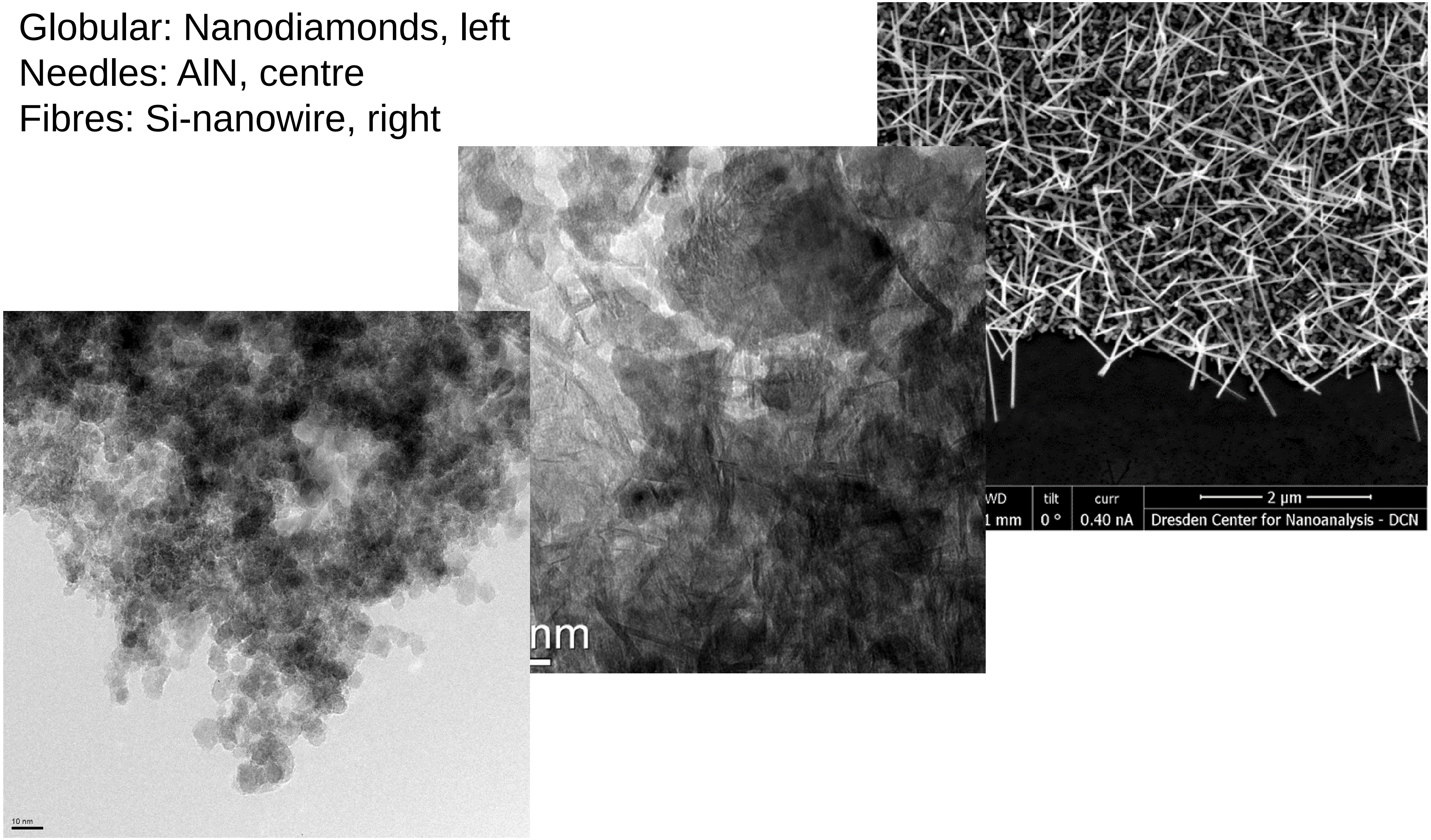
Target of analysis

Shape

Globular: Nanodiamonds, left
Needles: AlN, centre
Fibres: Si-nanowire, right



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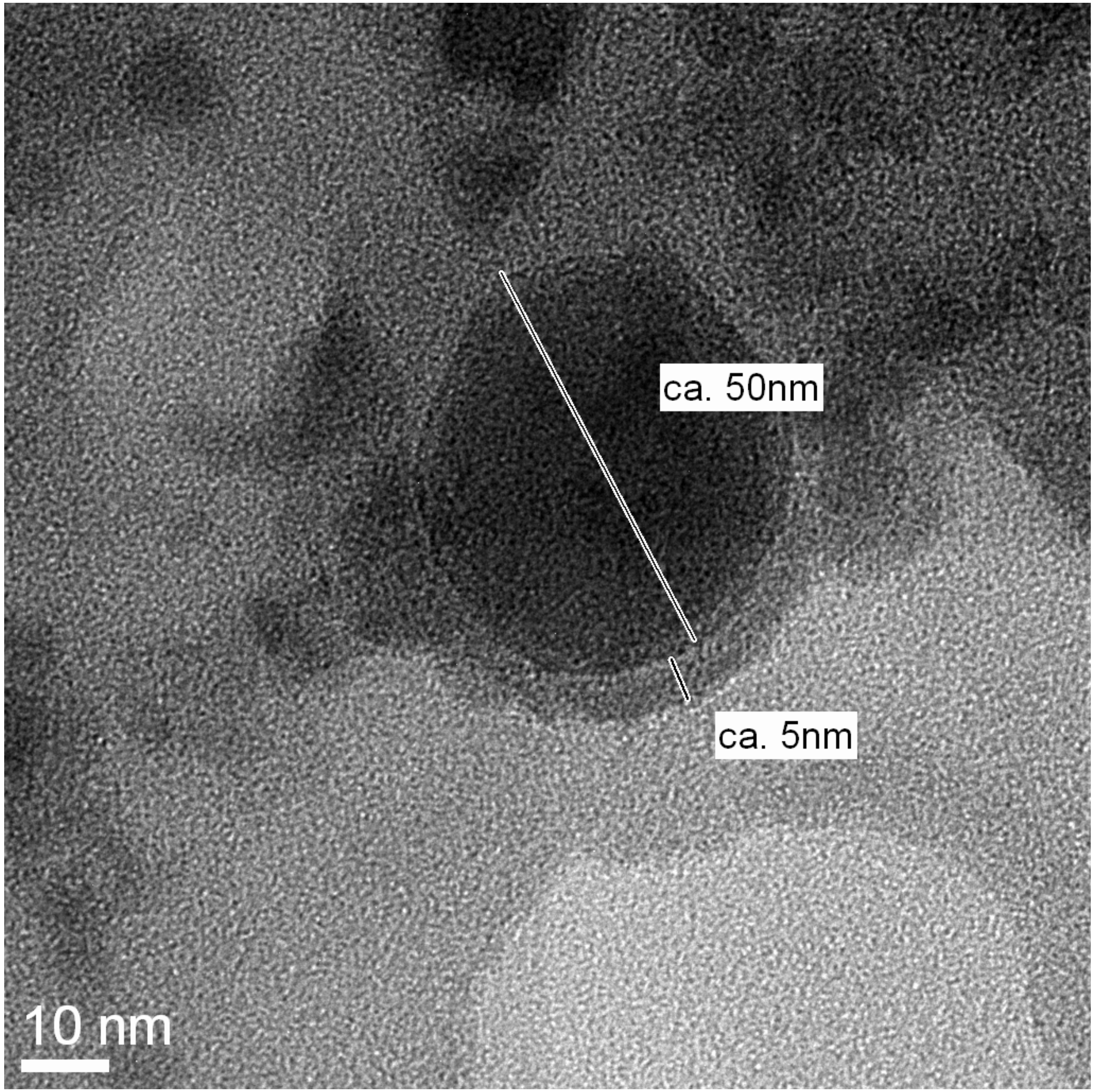
Target of analysis **Structure**

Core-Shell-structure:

TiCN-Particle with oxidic shell (amorphous)



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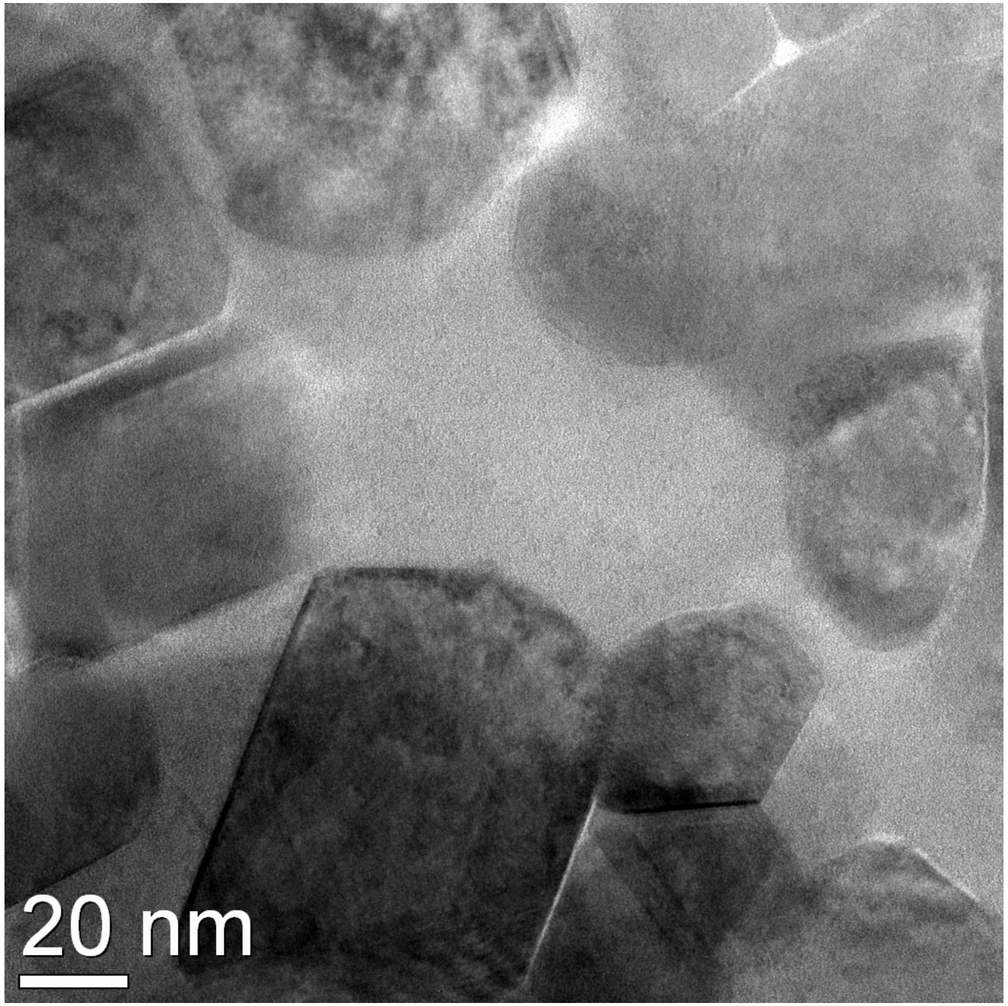
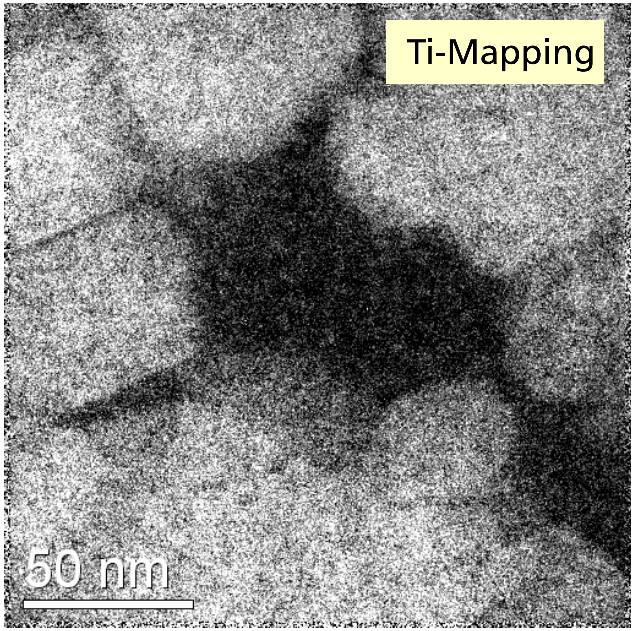
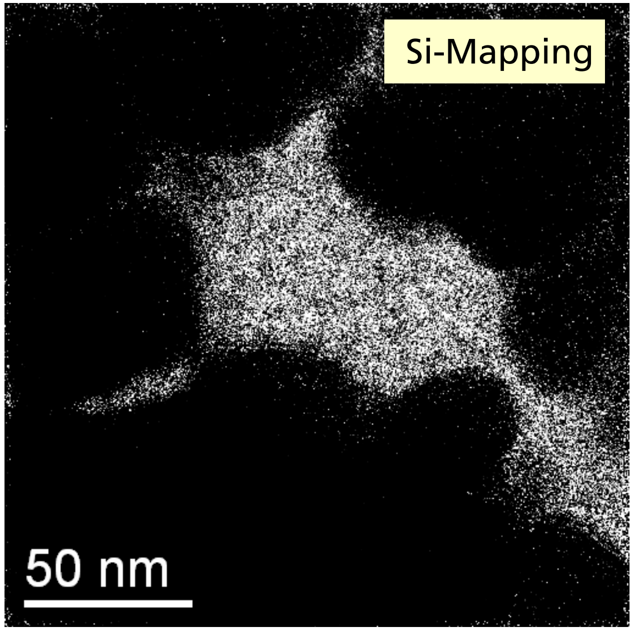
Target of analysis **Chemistry**

Sintered material: TiN/Si_3N_4



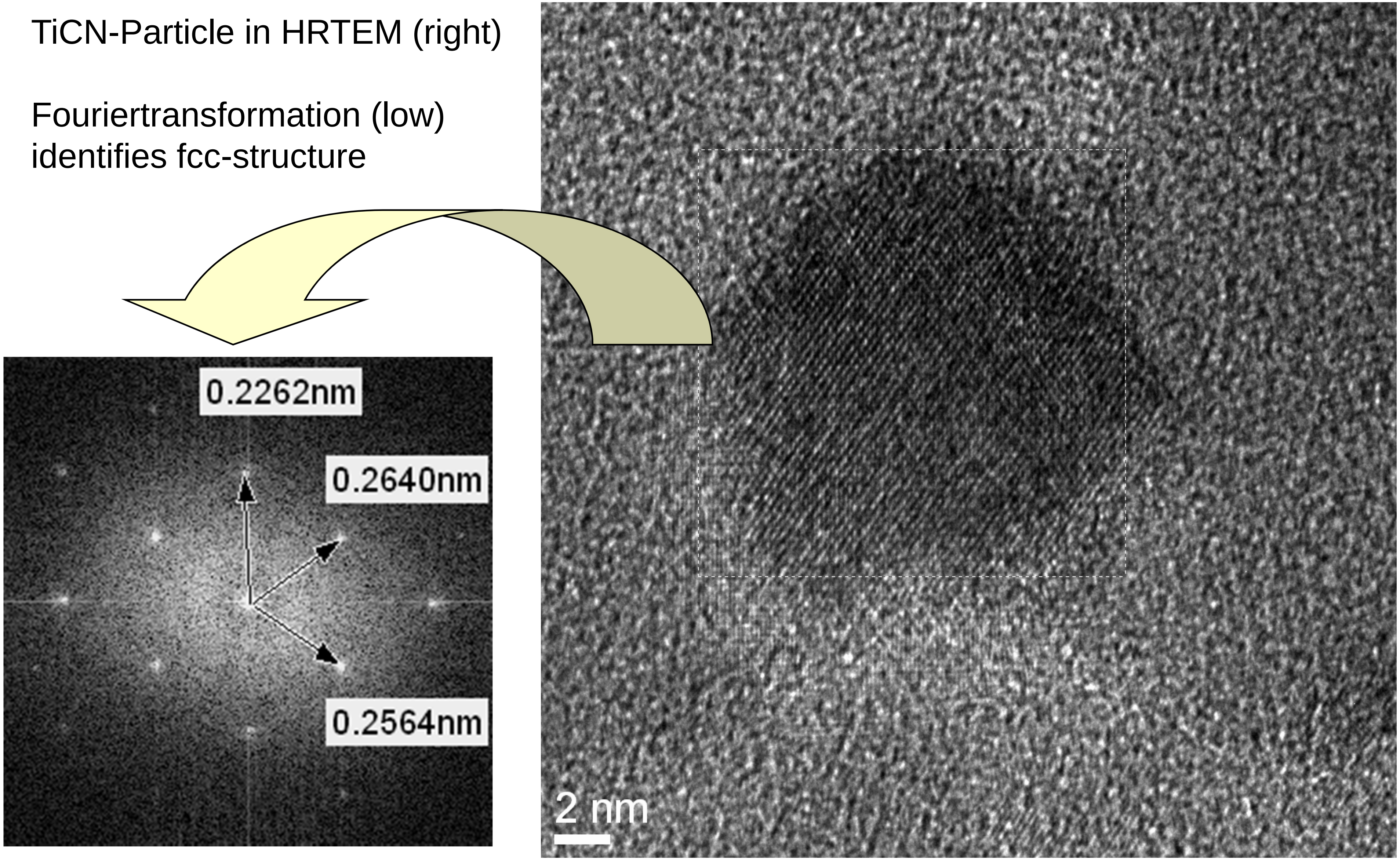




Target of analysis Crystallography

TiCN-Particle in HRTEM (right)

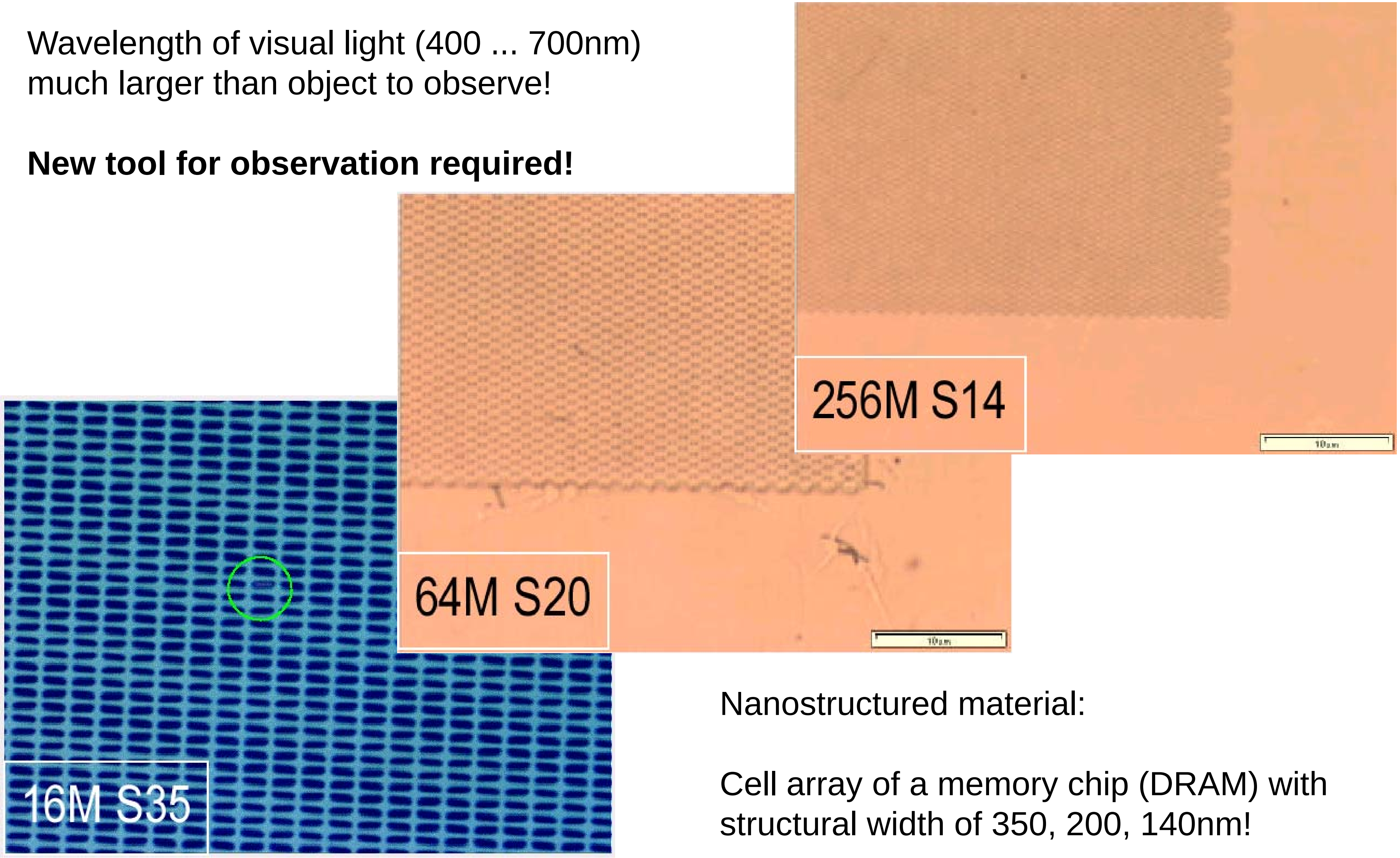
Fouriertransformation (low) identifies fcc-structure



Methods of analysis Imaging: Visual light microscopy

Wavelength of visual light (400 ... 700nm)
much larger than object to observe!

New tool for observation required!



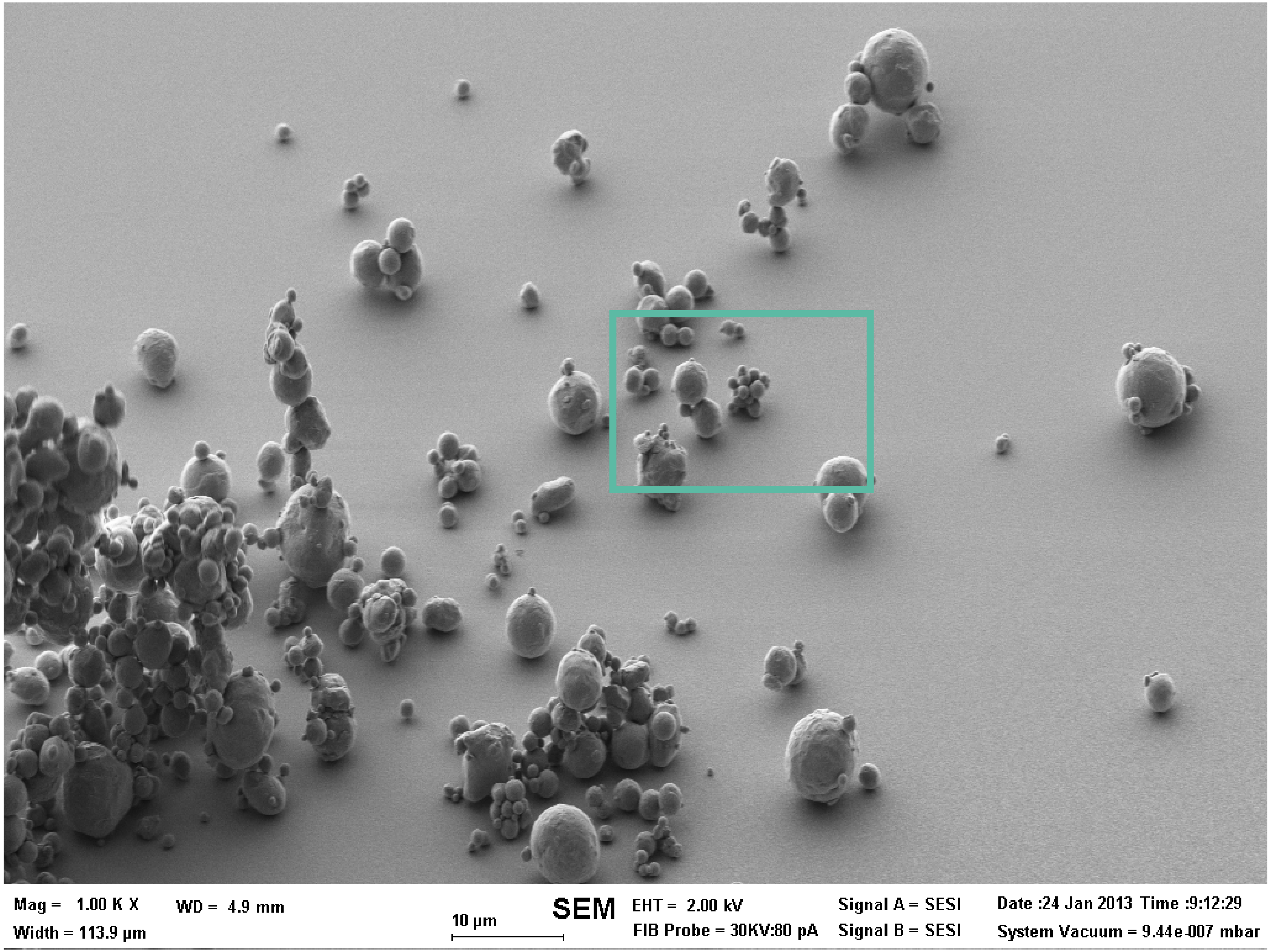
Nanostructured material:

Cell array of a memory chip (DRAM) with structural width of 350, 200, 140nm!

Methods of analysis Imaging: Scanning electron microcopy

Cu-powder:

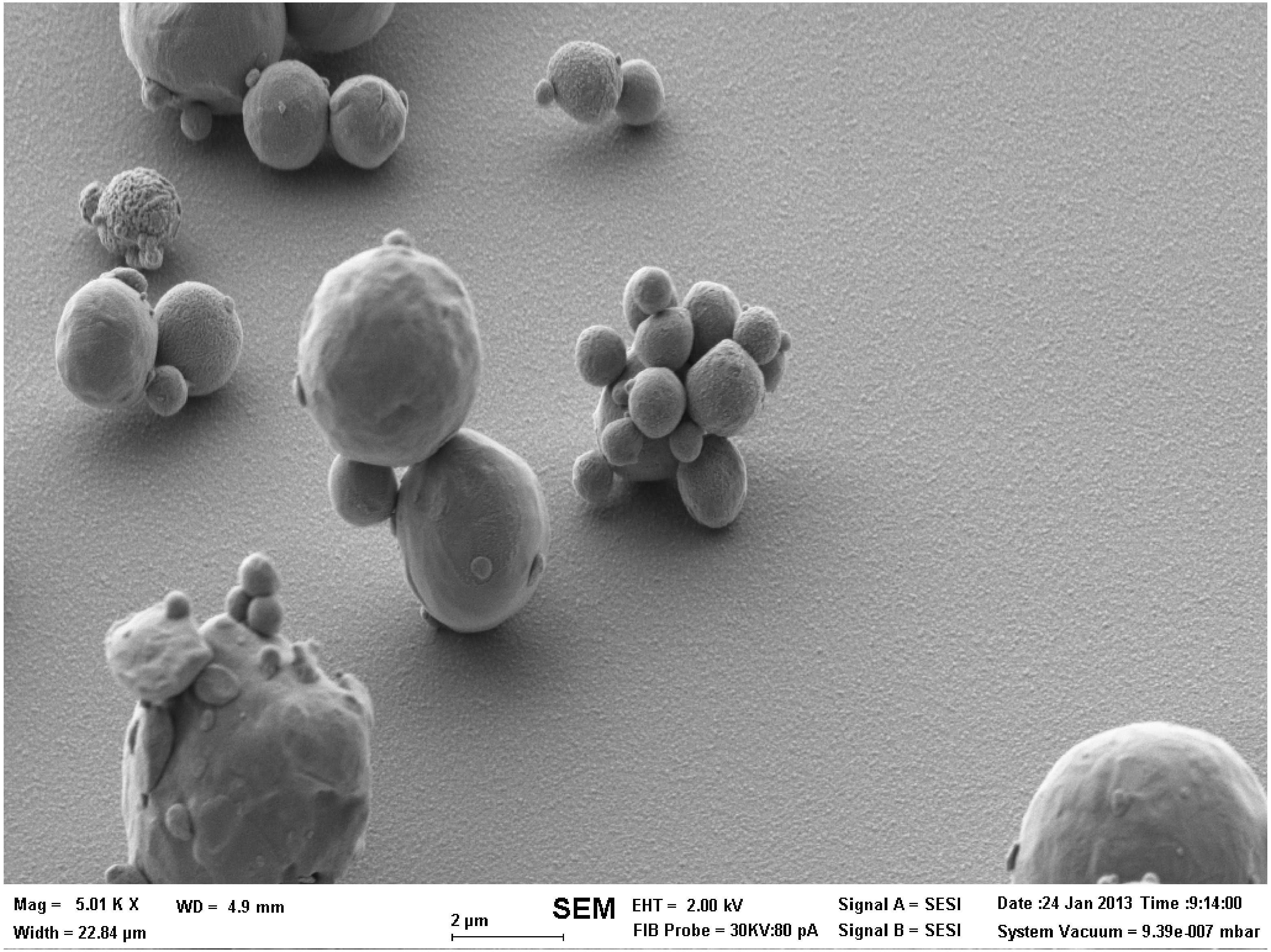
Particle size from several µm down to...



Methods of analysis Imaging: Scanning electron microcopy

Cu-powder:

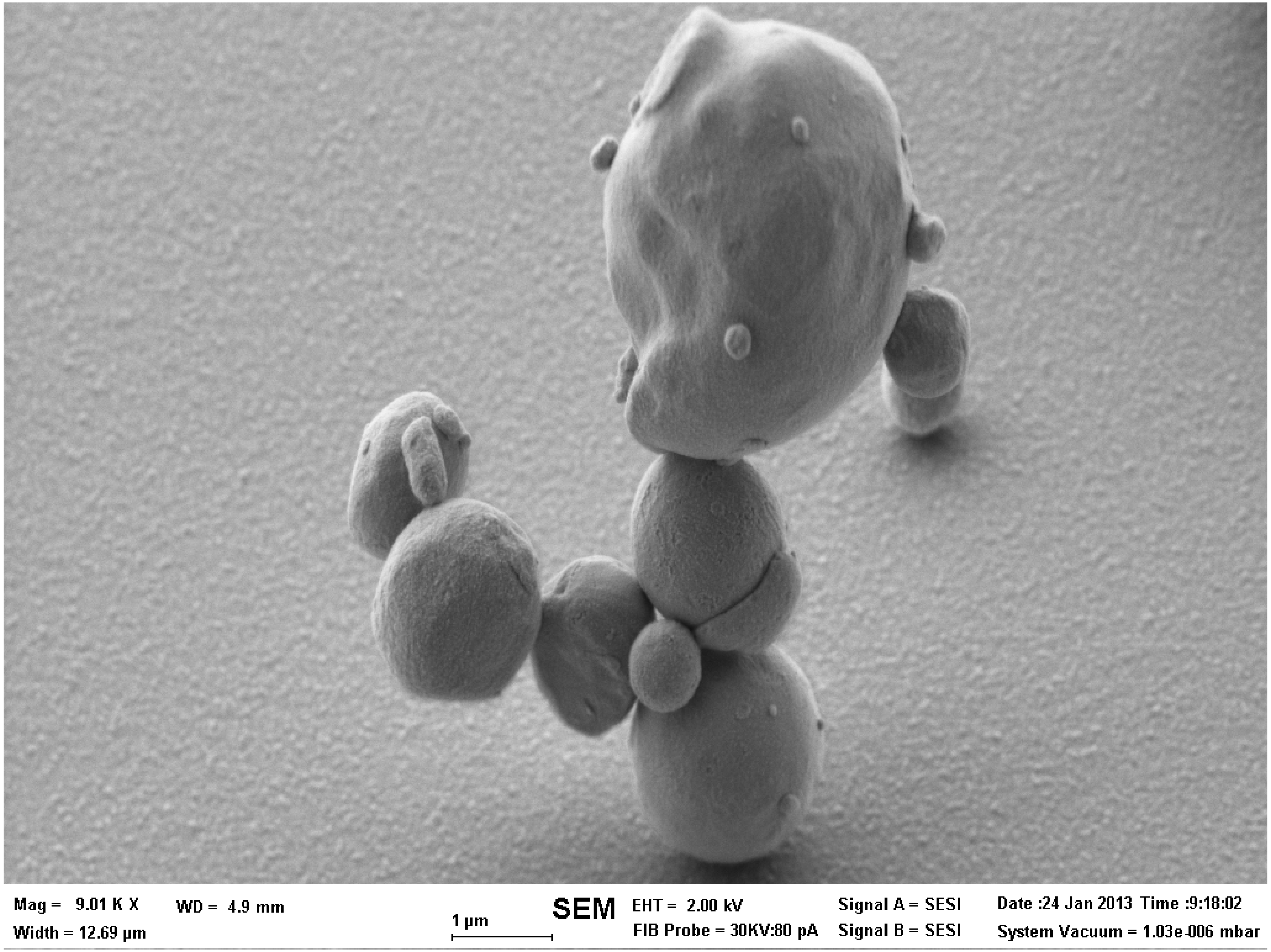
Particle size
in single µm
down to ...



Methods of analysis Imaging: Scanning electron microcopy

Cu-powder:

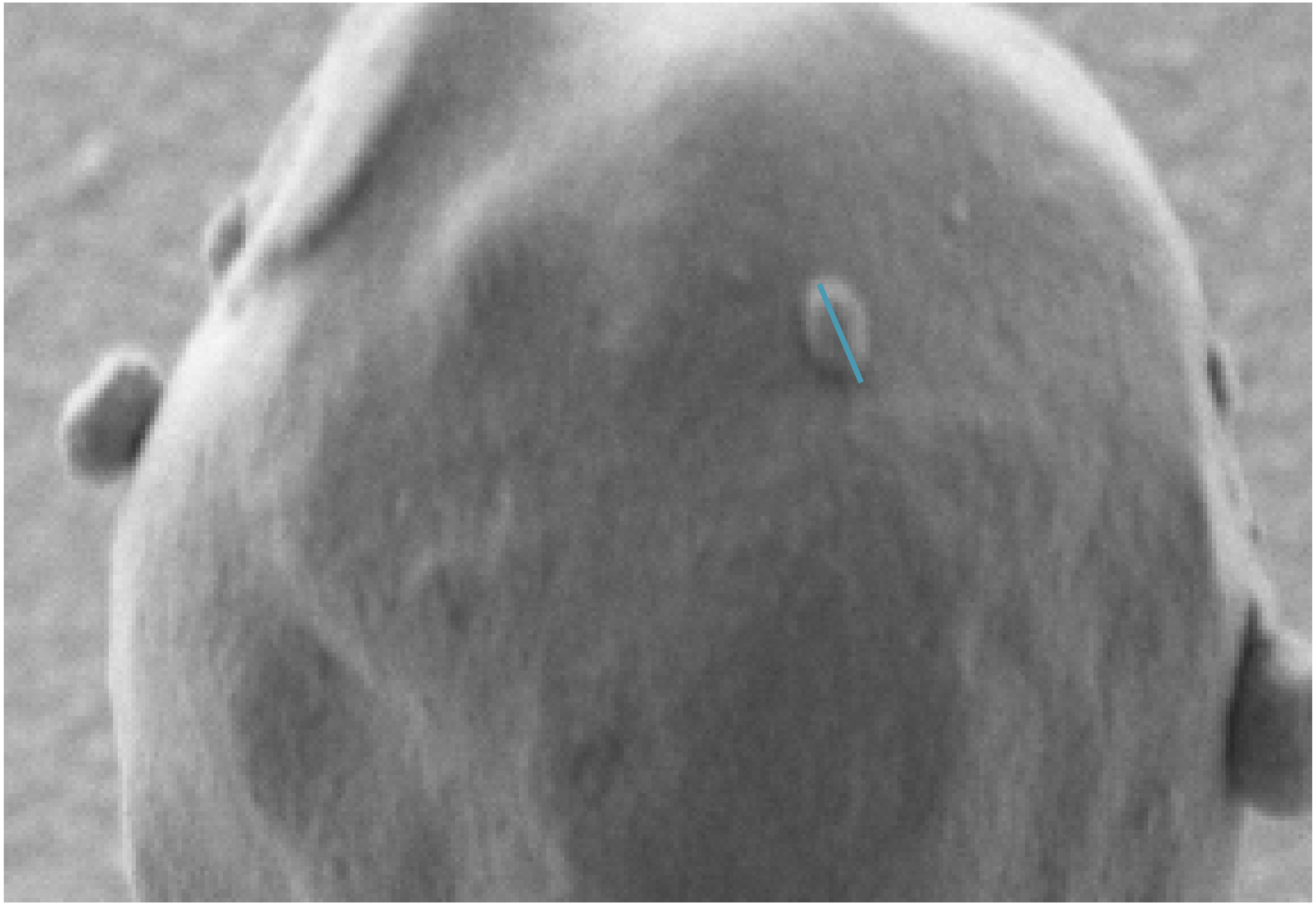
Particle size
in single µm
down to ...



Methods of analysis Imaging: Scanning electron microcopy

Cu-powder:

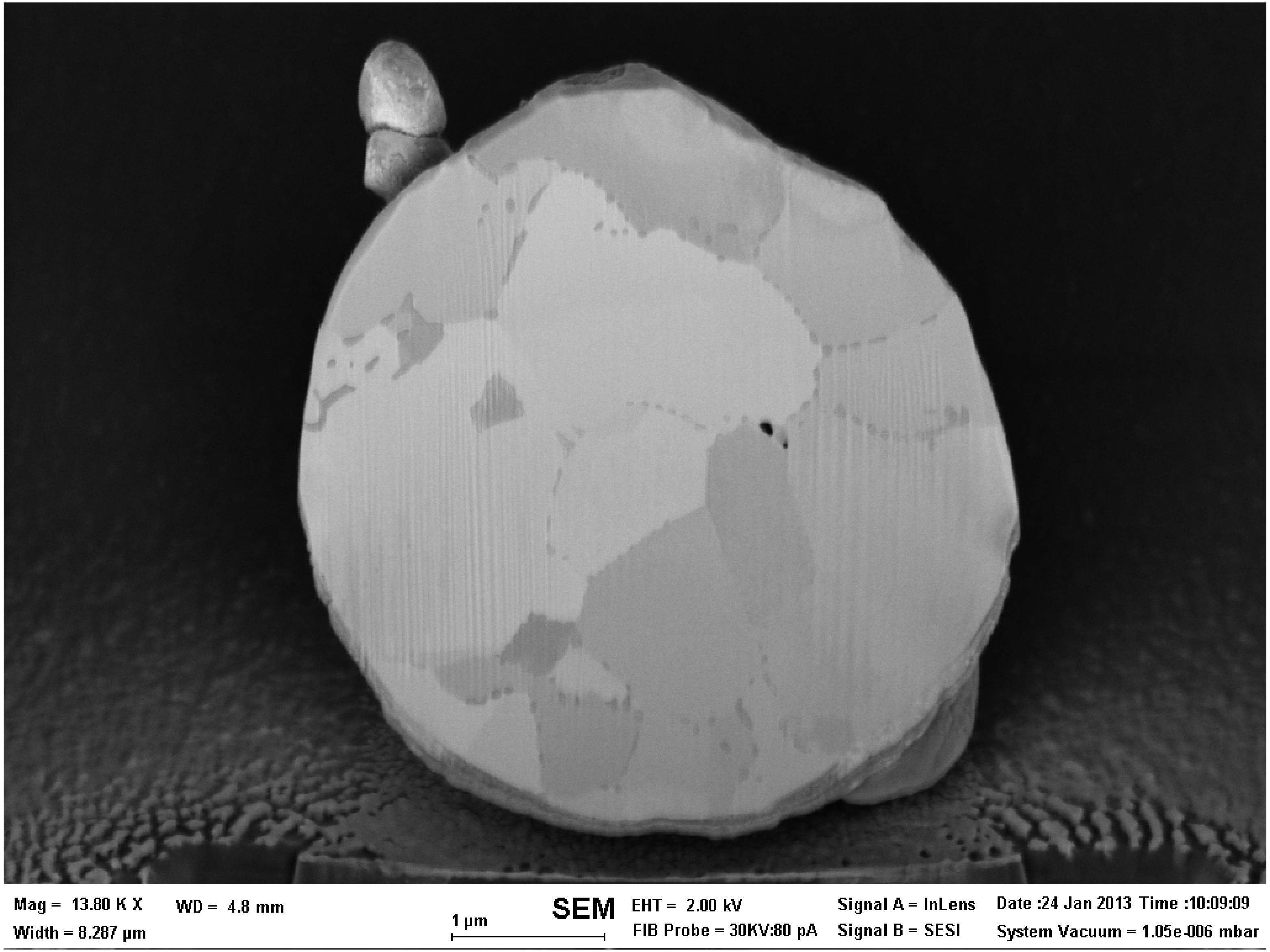
Particle size from single µm down to 63nm



Methods of analysis — Imaging: Focused Ion Beam Technique

Cu-powder:

Internal structure of particles

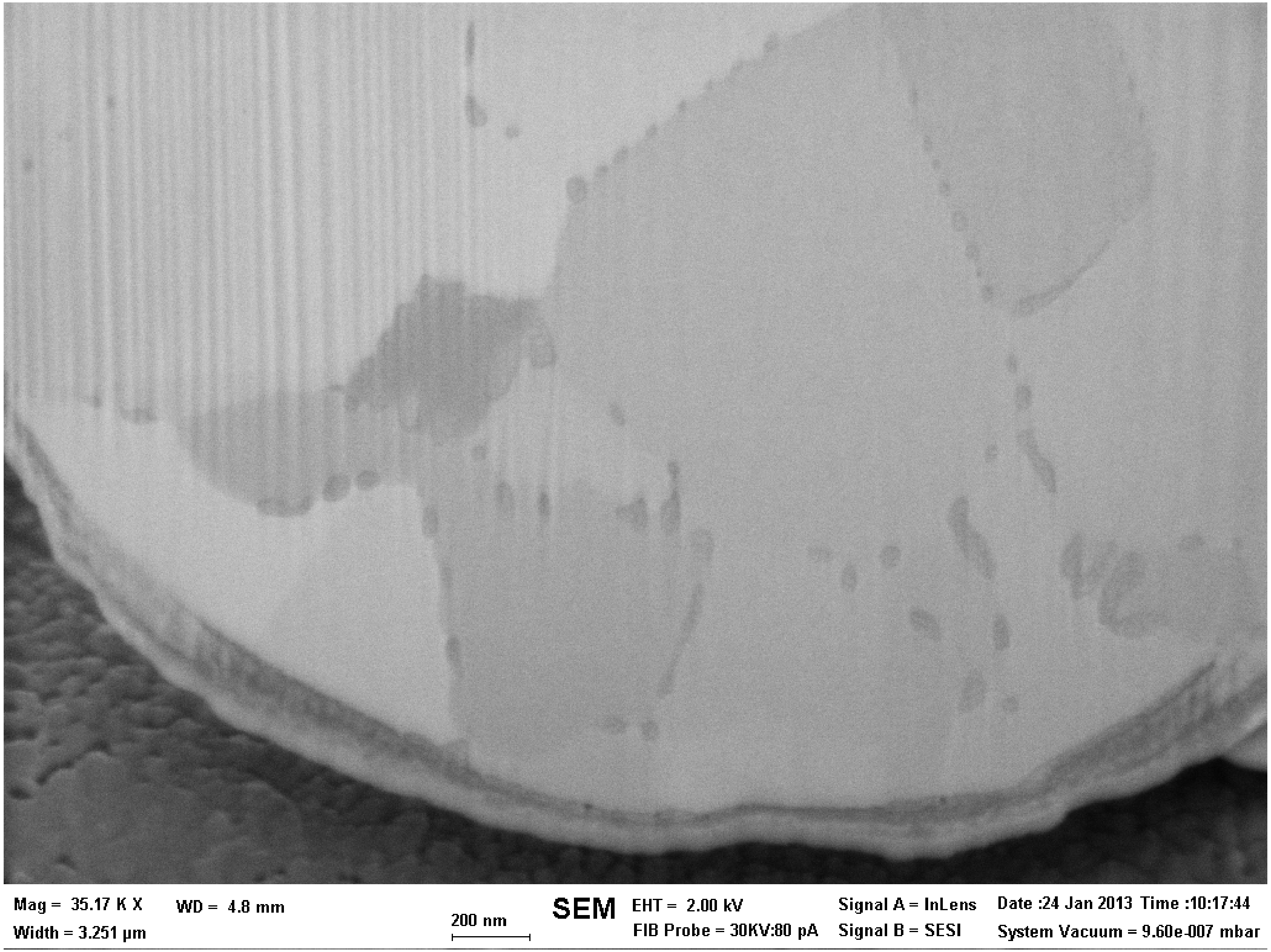


Methods of analysis — Imaging: Focused Ion Beam Technique

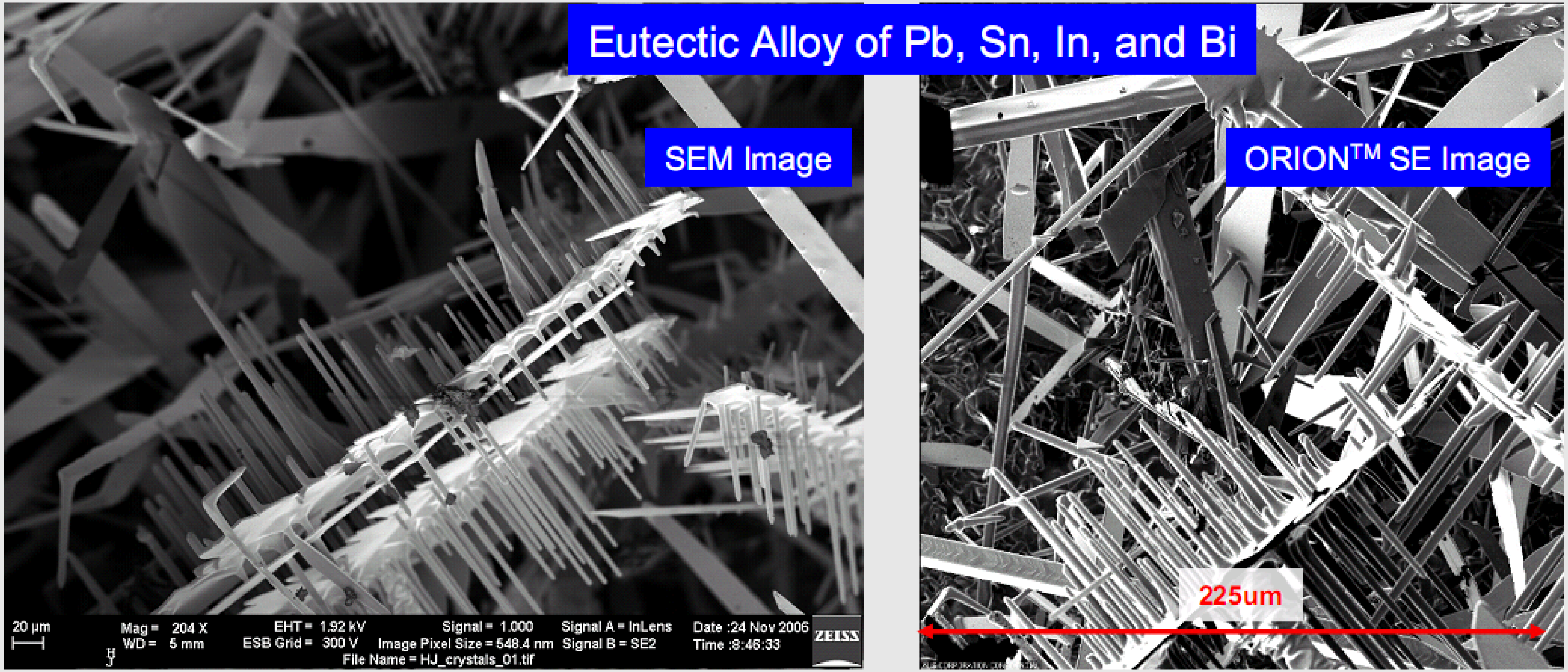
Cu-powder:

Internal structure of particles:

a) Core-shell-structure
b) Precipitations at grain boundaries



Methods of analysis — Imaging: Helium Ion Microscopy



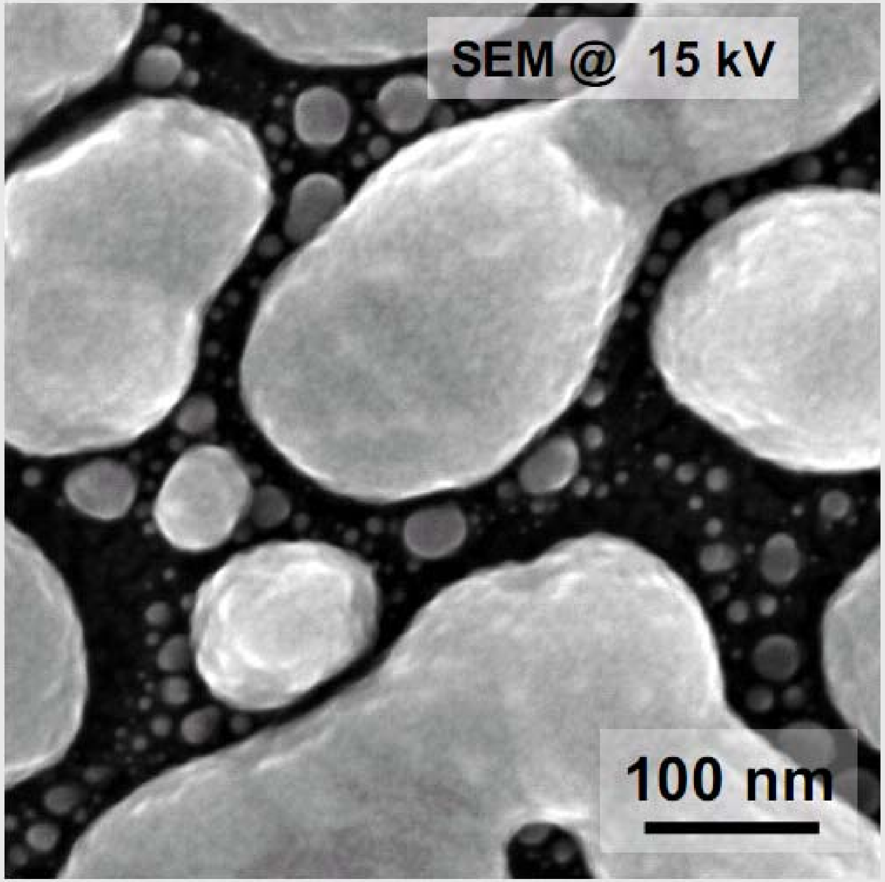
Depth of Field inversely proportional to Half Angle (α_i) of incident beam

Half Angle (α_i) for Orion typically 20 times smaller than for a SEM

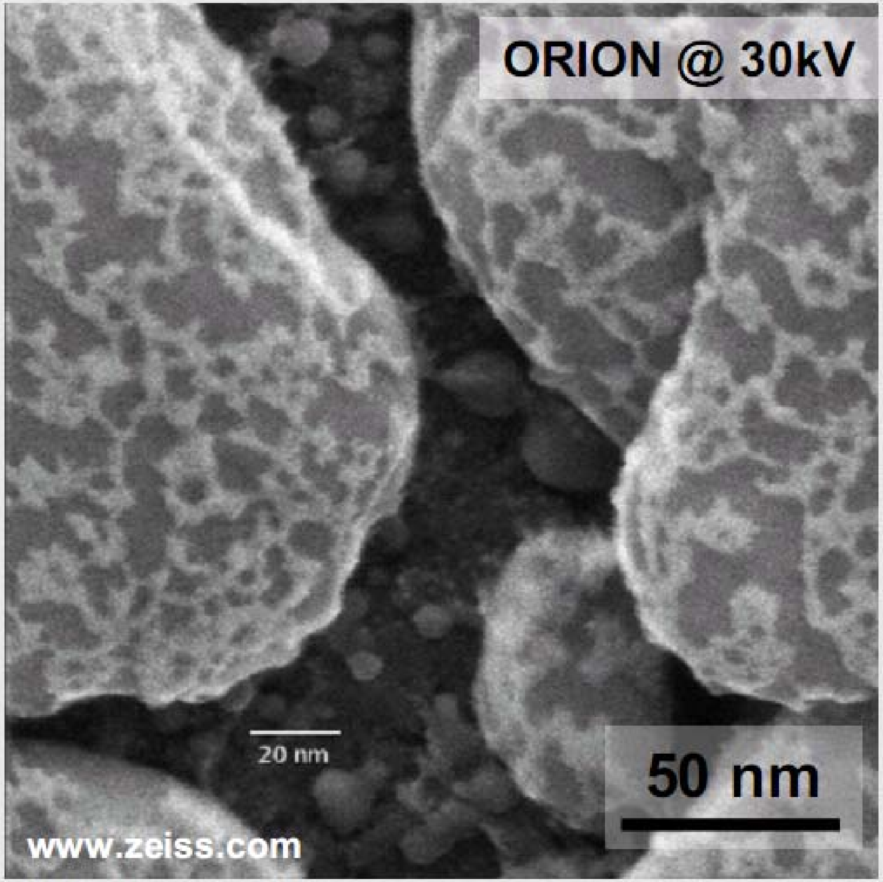
Courtesy: Carl Zeiss NTS, P. Gnauck

Methods of analysis

Imaging: Helium Ion Microscopy



- good SNR
- high contrast between Au and C



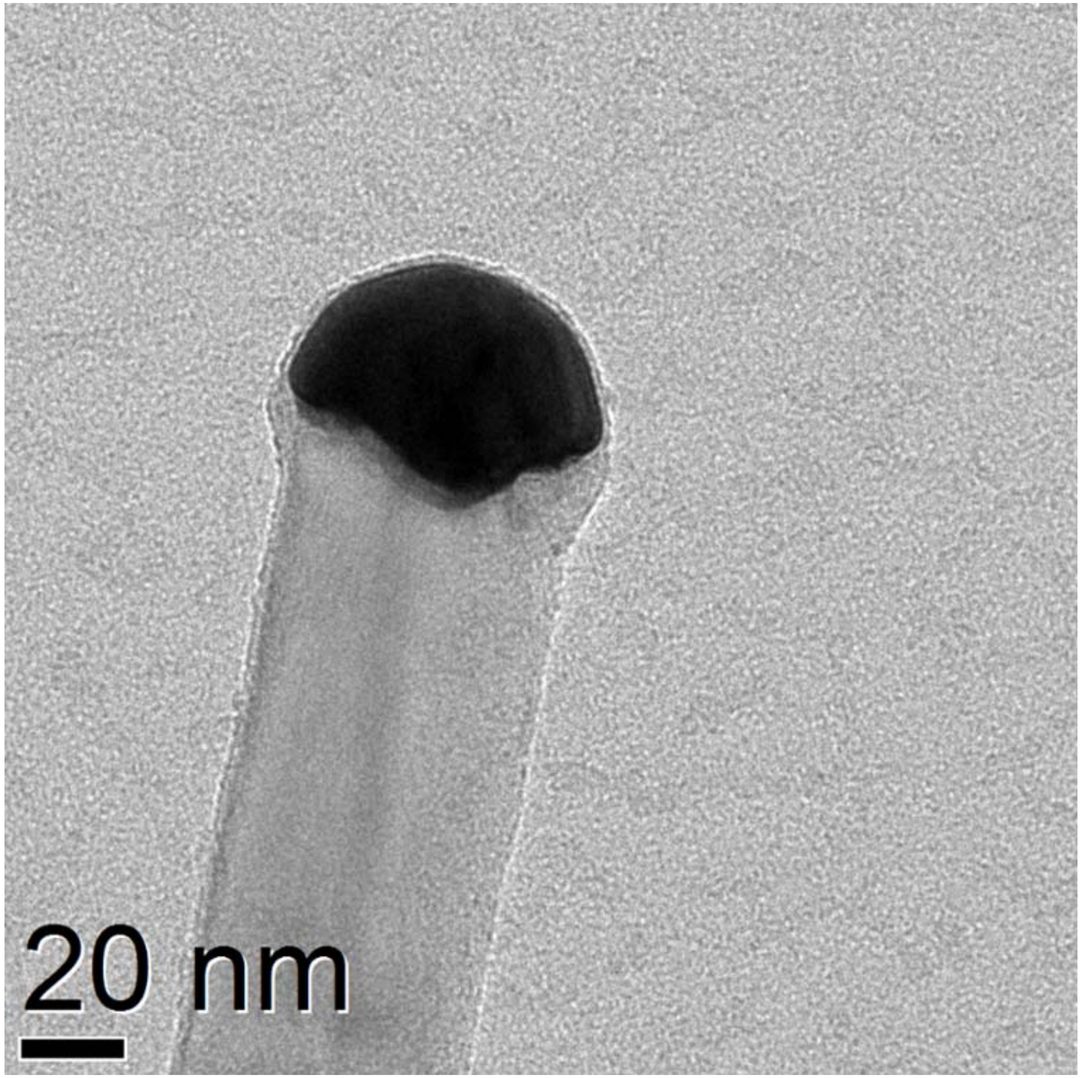
- high surface sensitivity

…surface details that could not be seen become visible

Courtesy: Carl Zeiss NTS, P. Gnauck

Methods of analysis **Imaging: Transmission electron microcopy**

Upper end of a silicon-nanowire (futural electronic structures) with a gold particle as seed



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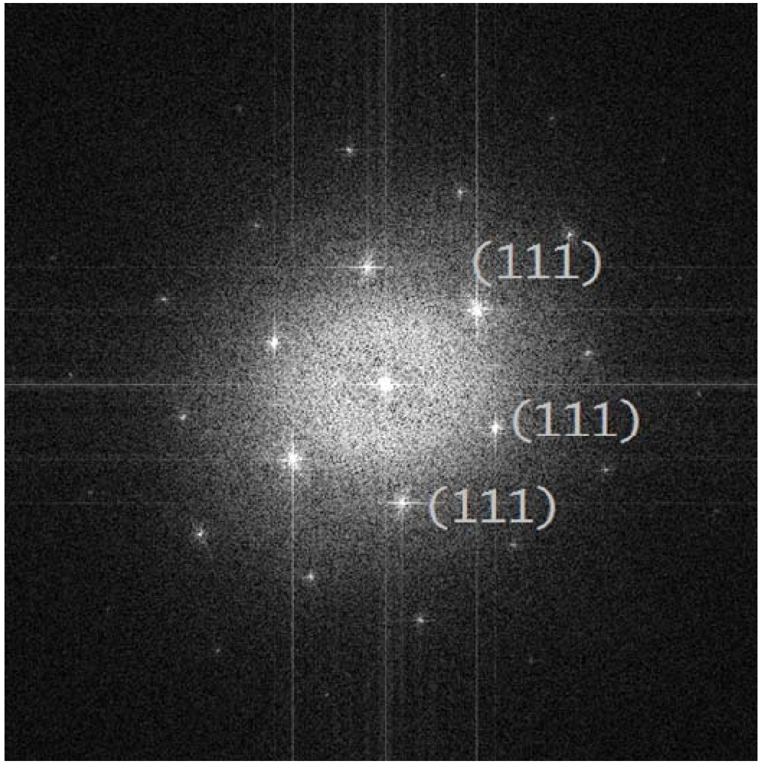
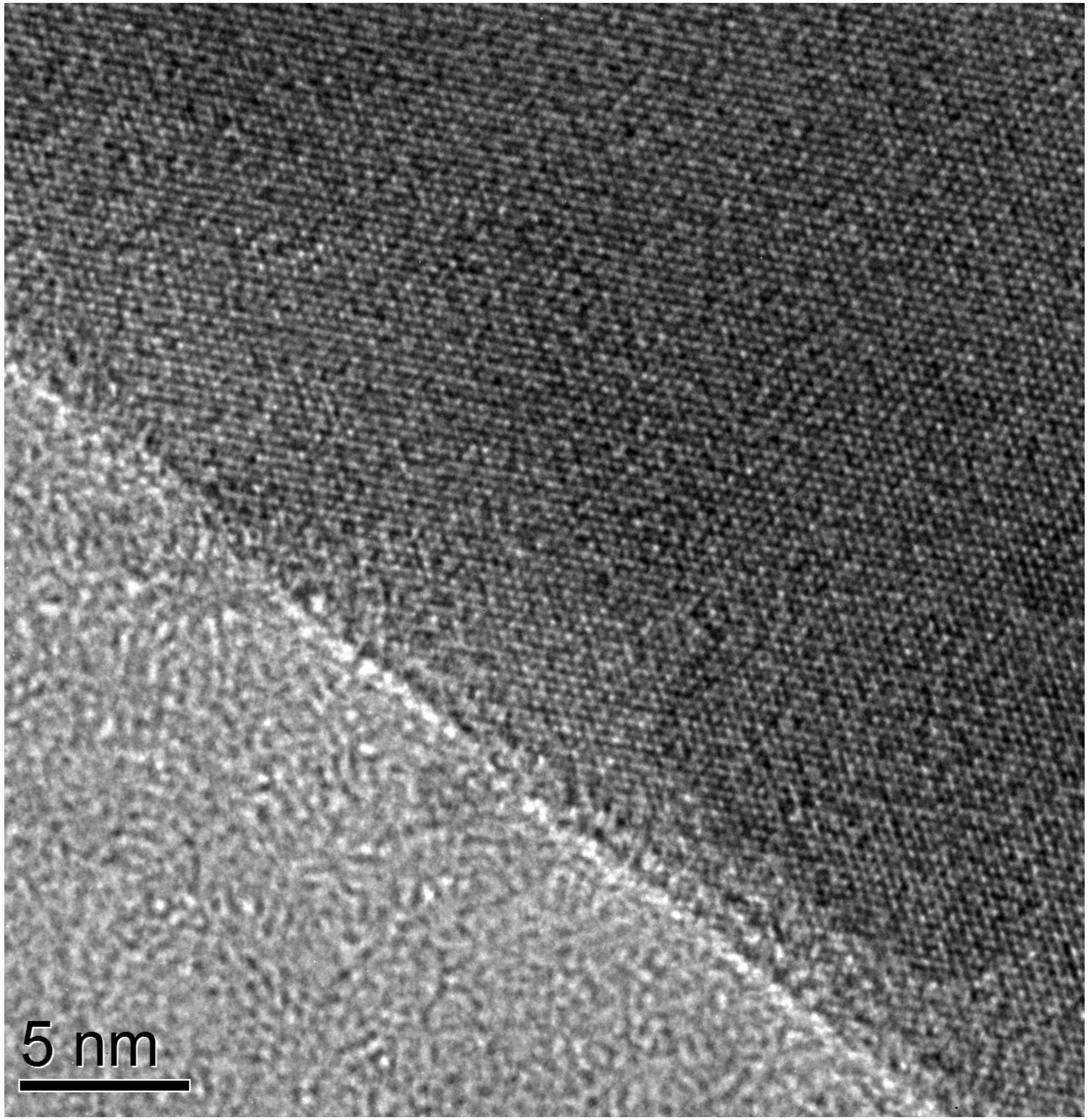
Methods of analysis Imaging: HR Transmission electron microcopy

Sidewall of a silicon-nanowire in HRTEM for observation of growing direction and interface behaviour





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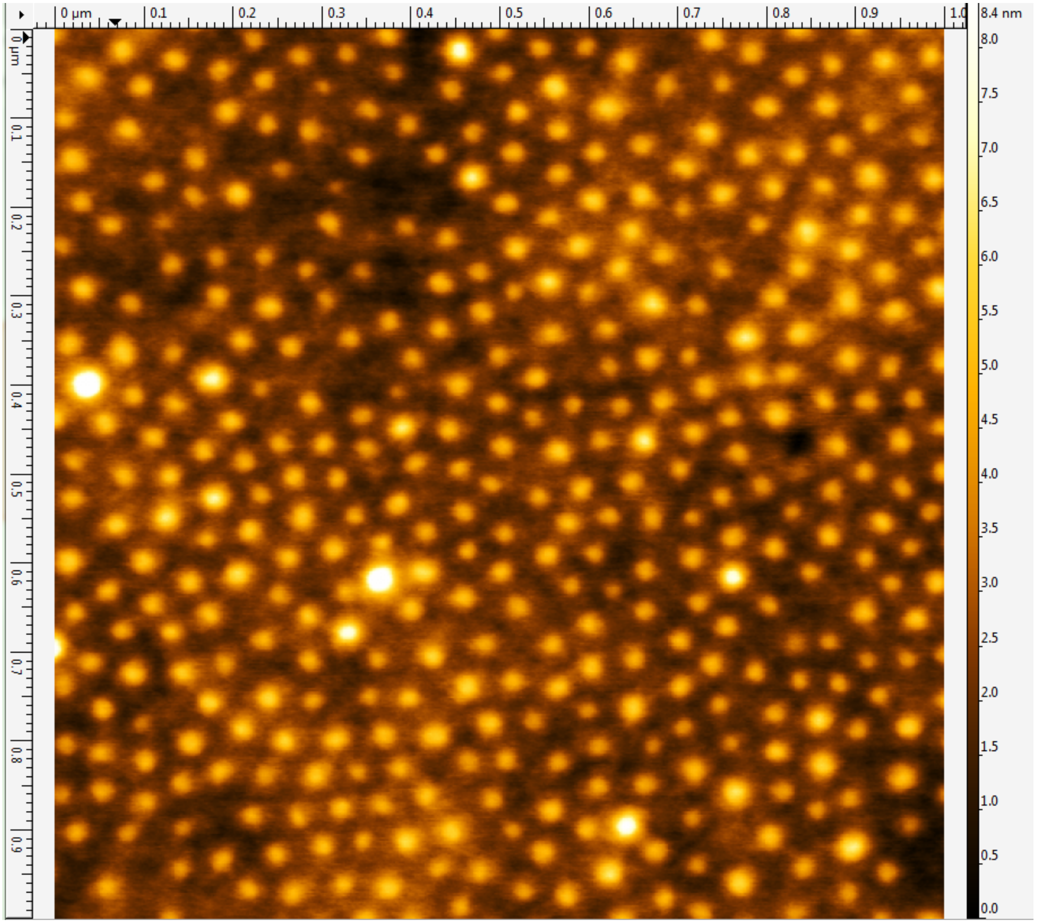
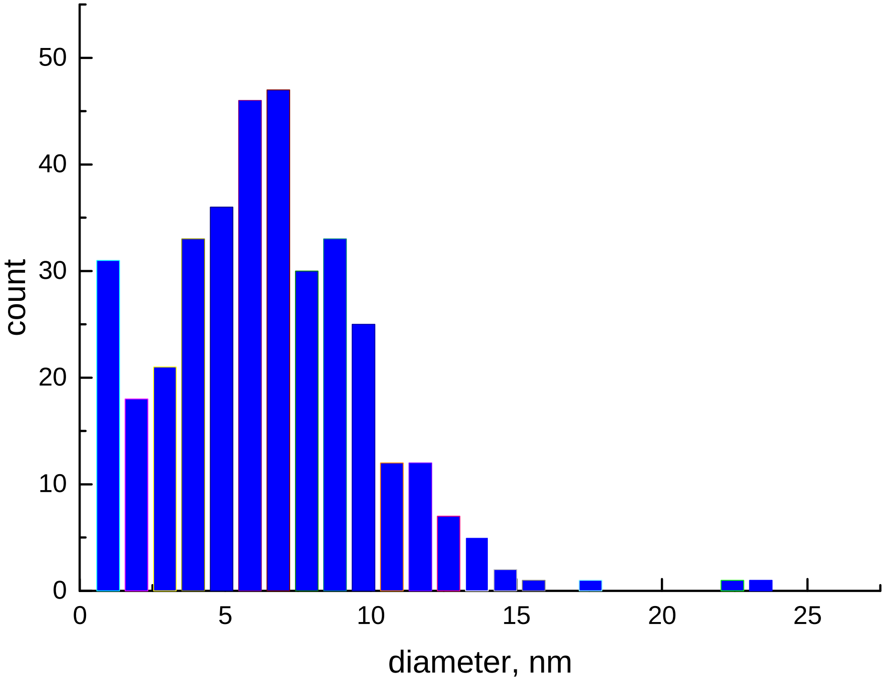
Methods of analysis **Scanning Probe Microscopy**

Topography image (left) and size distribution (right) of a polymer material (foil)



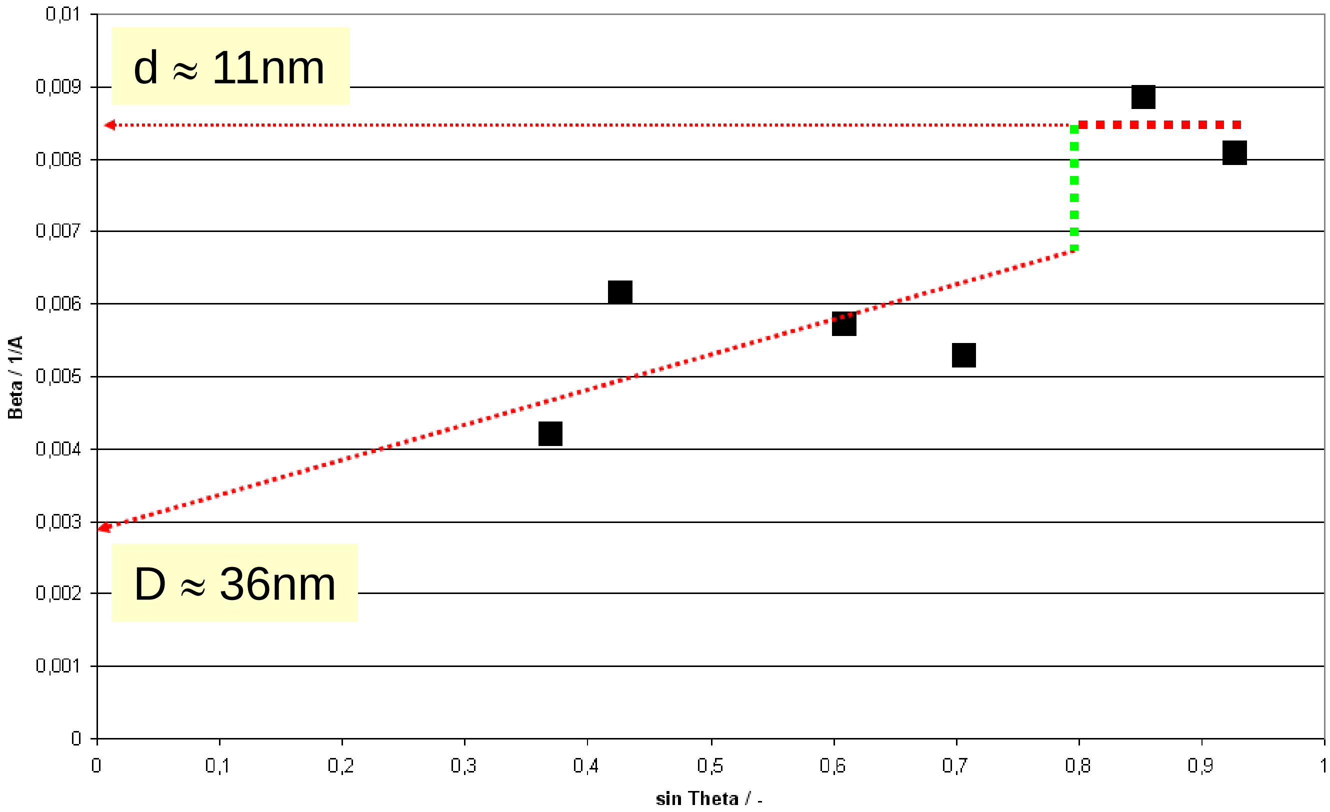
Sample: PS-b-P4VP
(*polystyrene-block-poly(4-vinylpyridine)*)

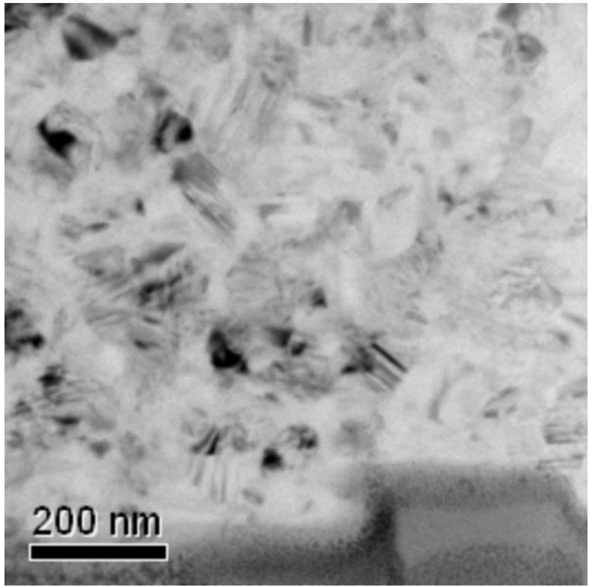


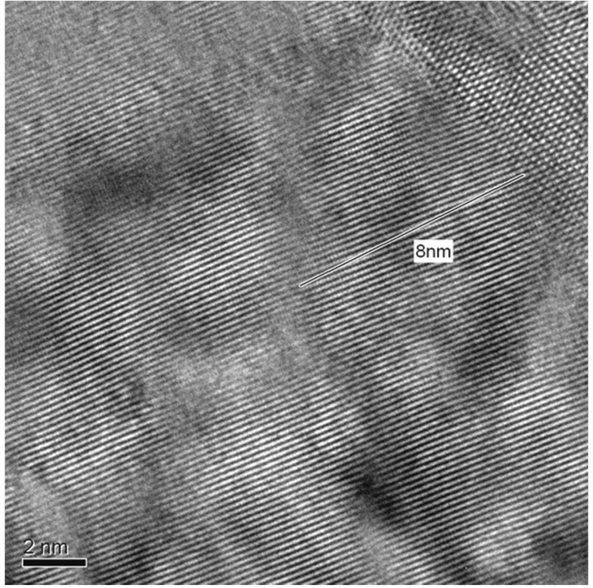
Courtesy: Leibniz-Institut für Polymerforschung Dresden e.V. + Dr. M. Kopyczunska-Mueller (IKTS-MD)

Methods of analysis Diffraction: X-ray Diffraction

X-Ray-Diffraction, Williamson-Hall-Plot:
Linebroadening over sin of diffraction angle (lower image)
characterizes mosaic structure

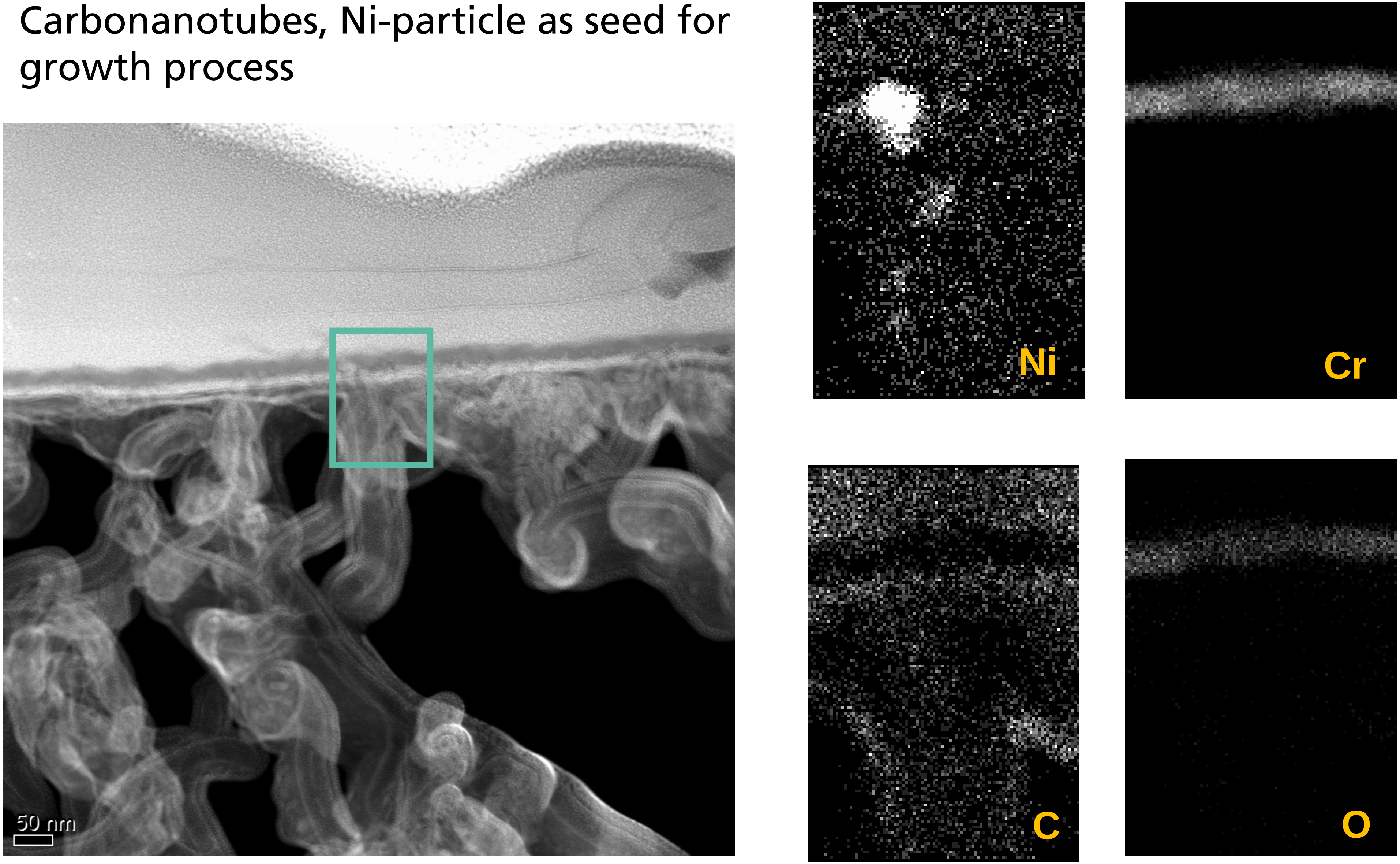






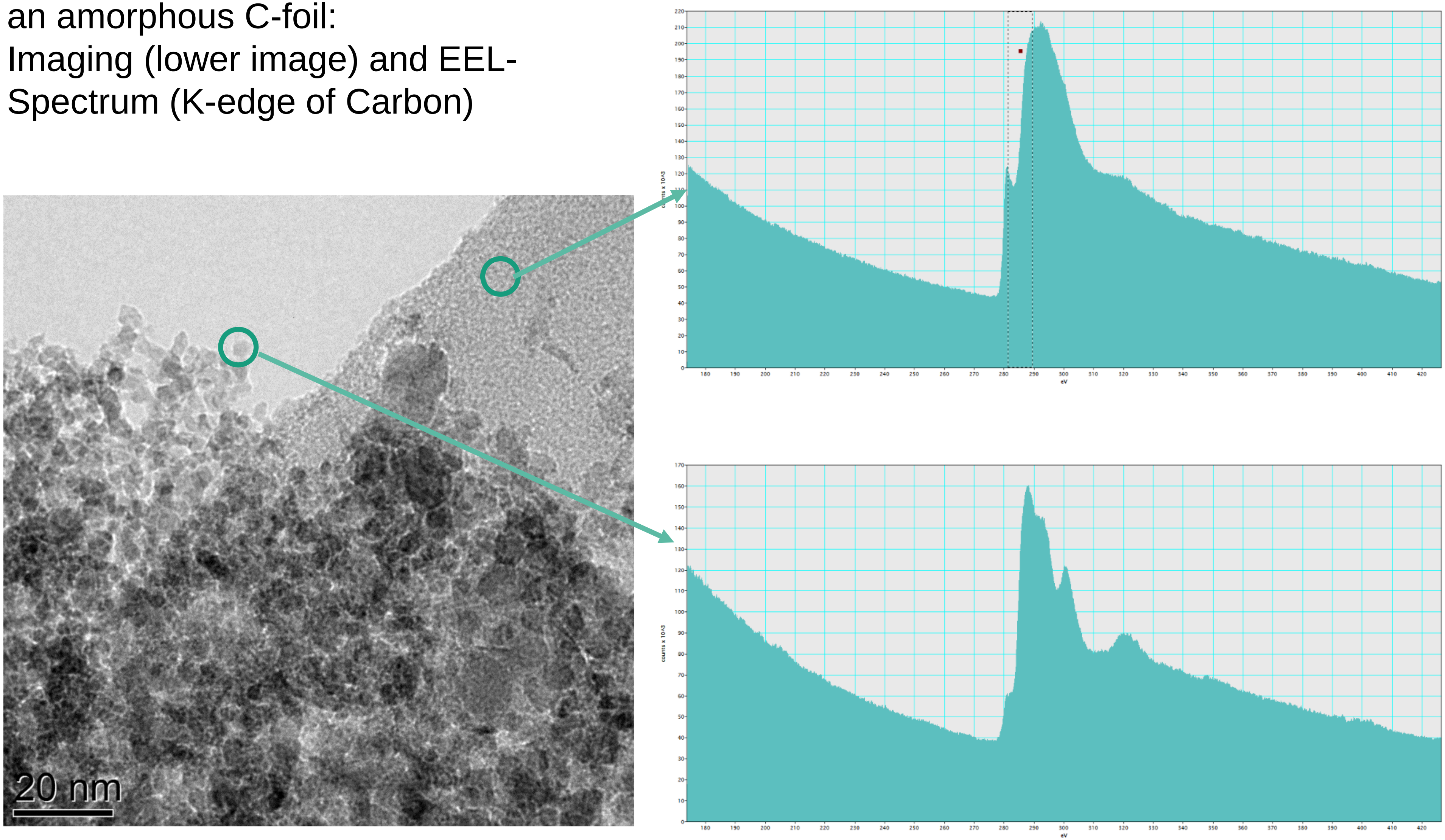
Methods of analysis **Energy dispersive X-ray Spectroscopy (TEM)**

Carbonanotubes, Ni-particle as seed for growth process



Methods of analysis Electron Energy Loss Spectroscopy

Nanodiamonds on
an amorphous C-foil:
Imaging (lower image) and EEL-Spectrum (K-edge of Carbon)



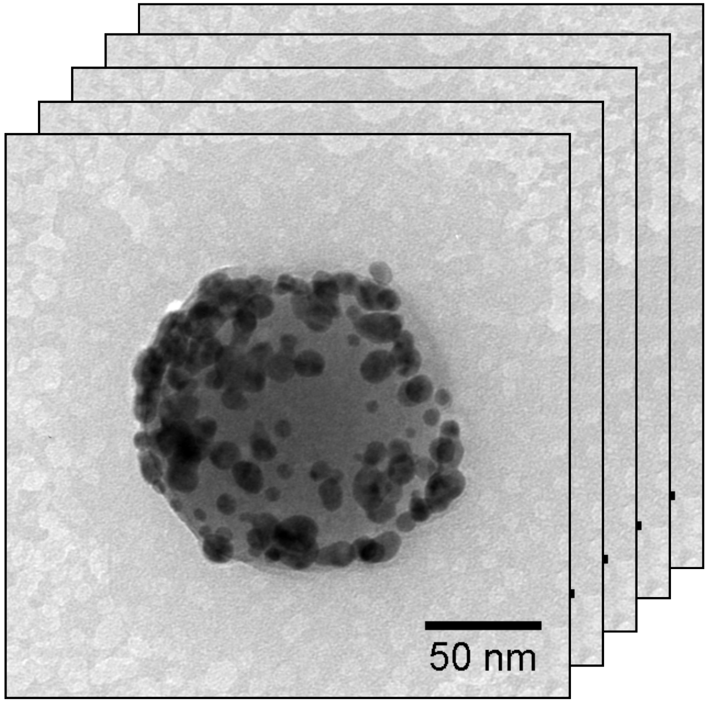
Methods of analysis — Electron Tomography

A 3D-world on the nanoscale: Example: Au nanoparticles on silica sphere

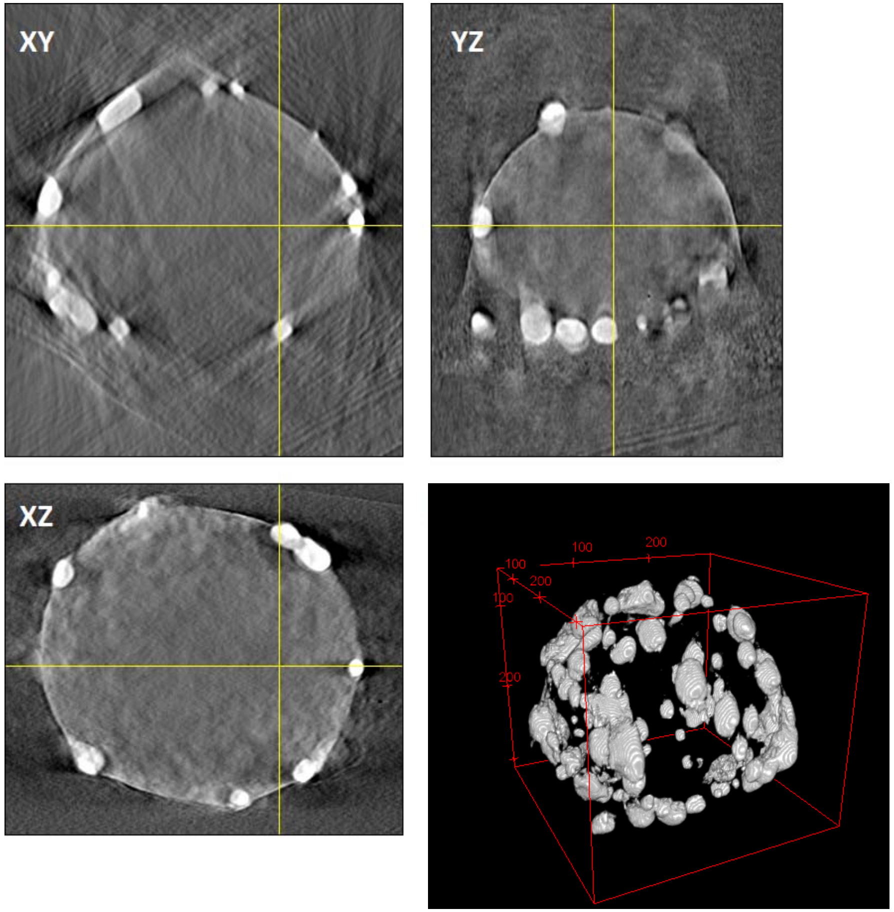
Recorded tilt series

range: -60° to +60°

step: 1°



Reconstructed volume (WBP)



Summary

- For characterization of nanomaterials a large number of aspects might be of interest: Size, shape, structure, chemistry, crystallography, ...
- Due to the wavelength‘ electron microscopy in its different variations is most employed method for characterization of nanomaterials.
- Ion microscopy (Focused Ion Beam, Helium Ion Microscopy) are interesting and new add-ons.
- Spectroscopic methods and 3D-related methods (tomography) complete the results.

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Analytical methods for characterization of nanomaterials

European Advanced Training Course

Nano-scale Materials

Characterization-Techniques and Applications

9 - 11 June 2015, Dresden, Germany

Dresden Fraunhofer Cluster Nanoanalysis (DFC

For further information please contact:

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Analytical methods for characterization of nanomaterials

E. Zschech
Survey of analysis techniques for multiscale materials characterization

E. Langer and S. Mucke
Imaging and element analysis of materials: Scanning electron microscopy and focused ion beam technique
- Introduction to SEM and FIB - Application in industry: Si-based and organic micro- electronics - Challenges and limits of the techniques

U. Muehle and M. Loeffler
Atomic resolution studies of materials and interfaces: Transmission electron microscopy - Imaging: Setup and contrast mechanisms - Structure and strain analysis: Diffraction techniques - Elemental analysis: EDX and EELS/EFTEM - Electron tomography - In-situ studies

P. Konda Gokuldoss
3D atomic structures in nanoscale materials: Atom probe tomography - Experimental and analysis techniques - Sample preparation with focused ion beam - Application in materials science and nanoelectronics

J.-U. Schmidt and J. Heber
Thin film analysis: Optical analysis and metrology, X-ray reflectometry
- Ellipsometry
- Interferometry
- Application to photonic microsystems

L. M. Eng
High-resolution studies of surface topography and near-surface properties: Scanning probe microscopy
- High-resolution structure analysis in semiconductors: Dopand profiles
- Mechanical strain fields in semiconductors
- Magnetic nanofields in magnetic thin films and nano-particles
Structures and fields at atomic dimensions

A. Clausner
Mechanical properties of nano-scale materials and thin films: Nanoindentation and related techniques
- Hardness, Young`s modulus and yield stress of nano-structures
- Nano-scale behavior of metals, ceramics, and glasses
- Properties and structure of nano-porous materials

J. Gluch and M. Loeffler
3D imaging of materials: Micro- and nano X-ray tomography
- X-ray tomography: from micro to nano
- Resolution and field of view
- Lab-based systems vs. synchrotron research
- Applications in materials science, electronics and biology