Advanced Microscopy

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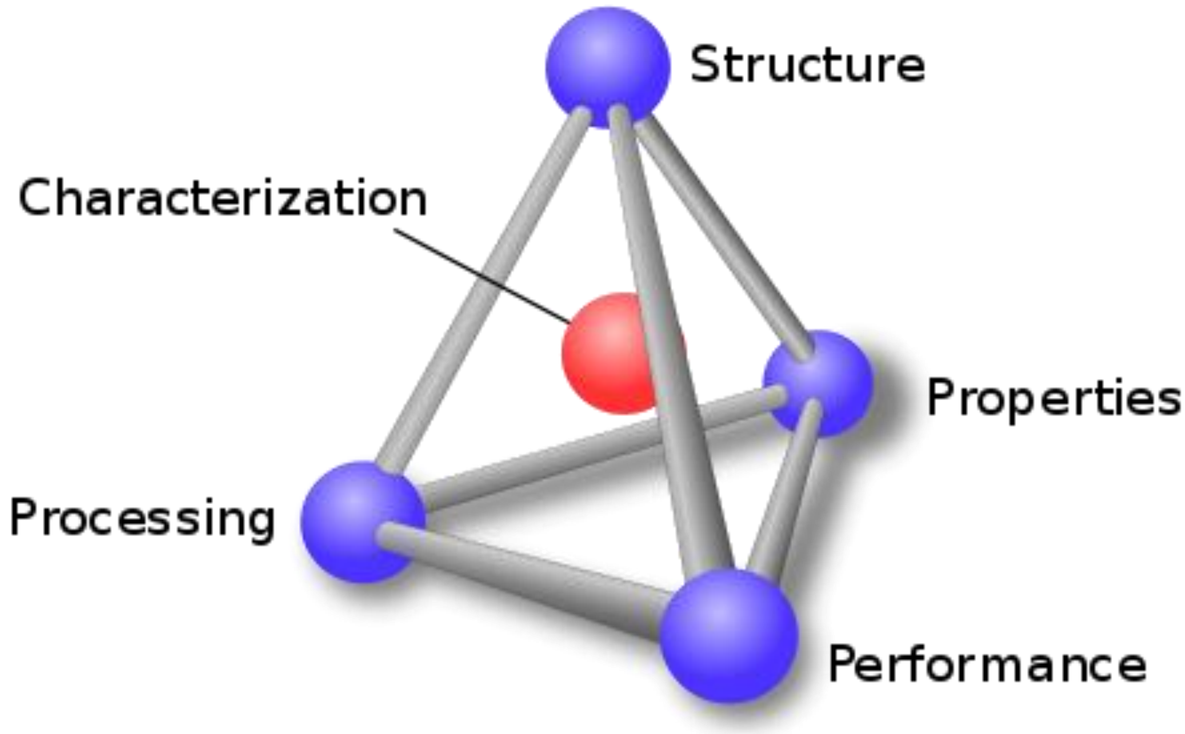
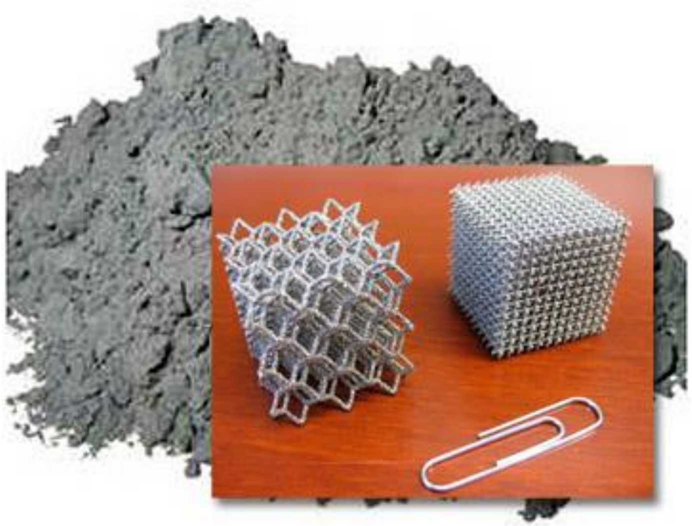
MTEC
a member of NSTDA

Outline

- Overview
  - Impression
  - Related characterization techniques
  - Point of view
- Fundamentals
- Research examples
- Conclusion

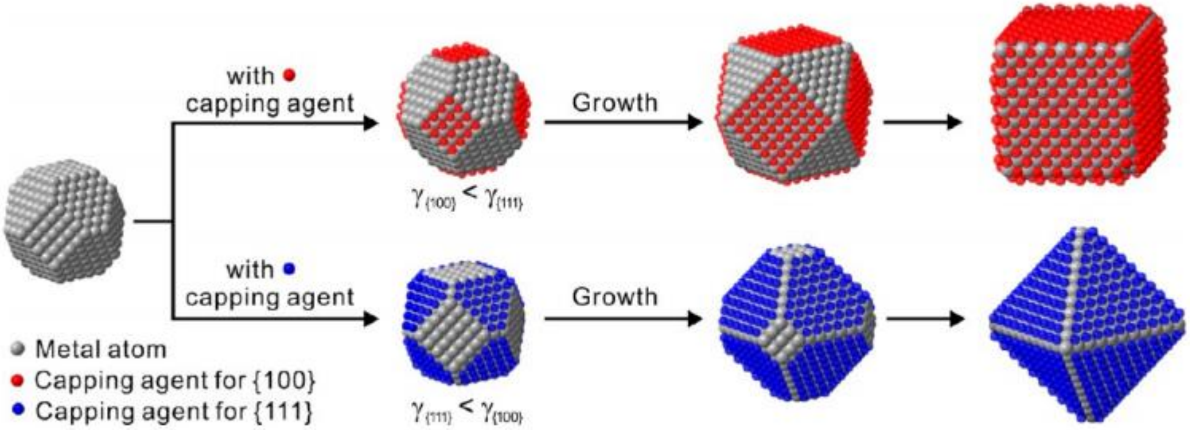


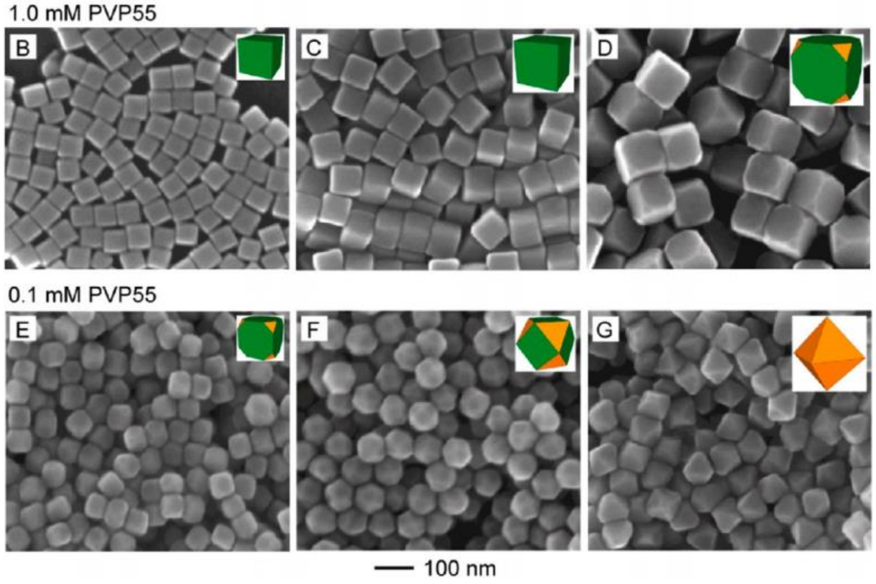
https://en.wikipedia.org/wiki/Materials_science

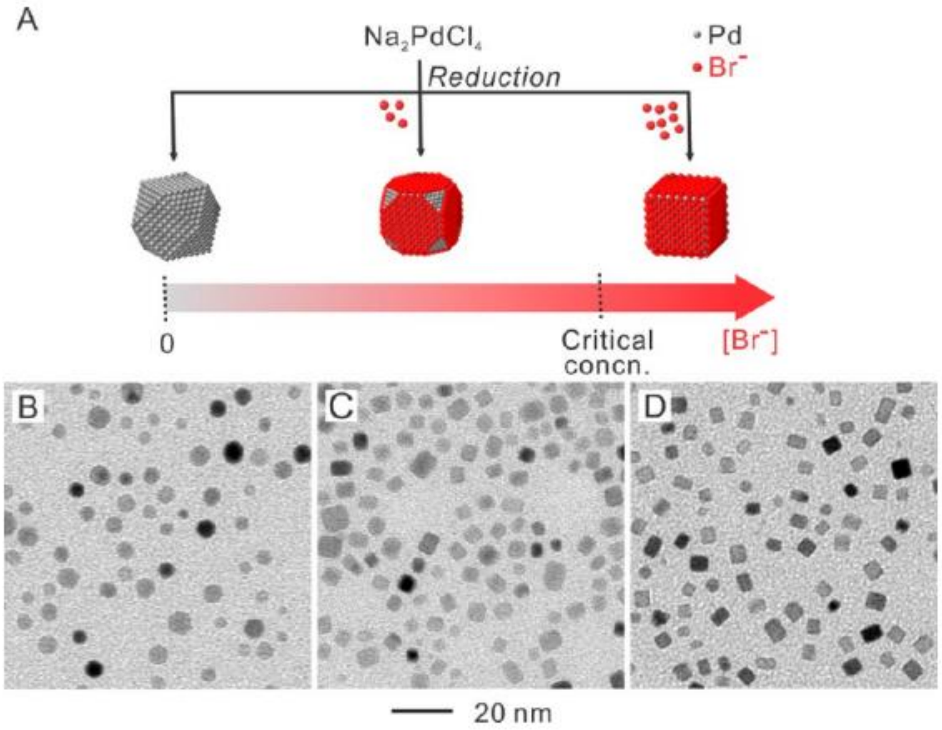


http://www.nist.gov/el/isd/sbm/camm.cfm

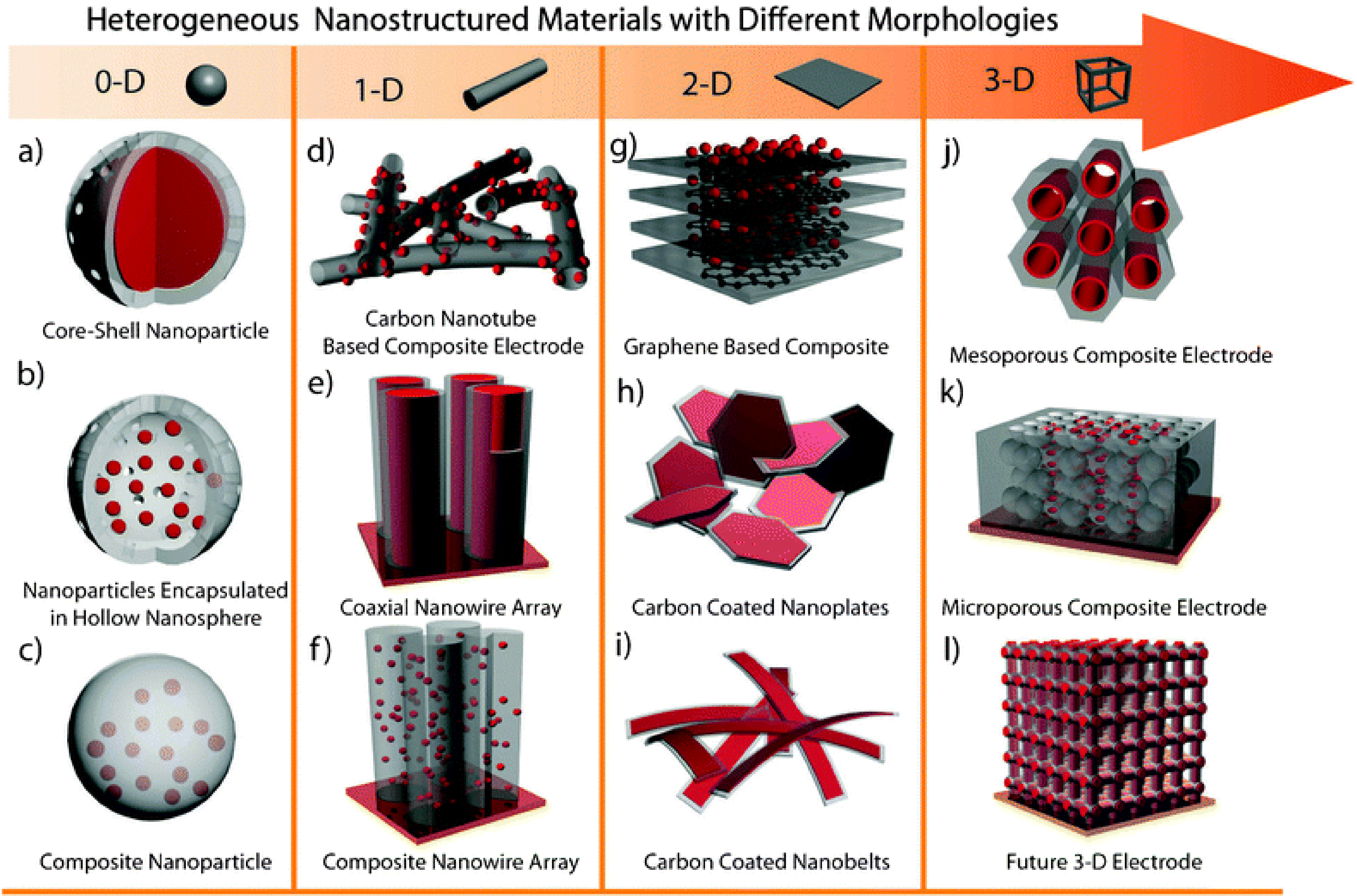
Examples





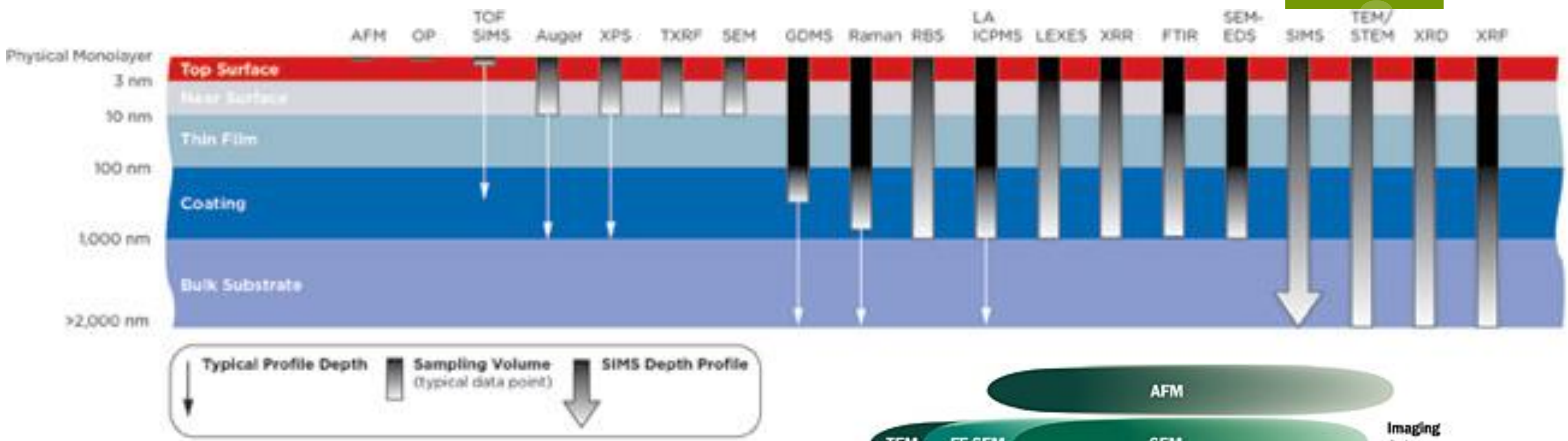


Y. Xia et al. JACS 137(2015)7947.



Liu et al.
Chem. Commun., 2011,**47**, 1384-1404

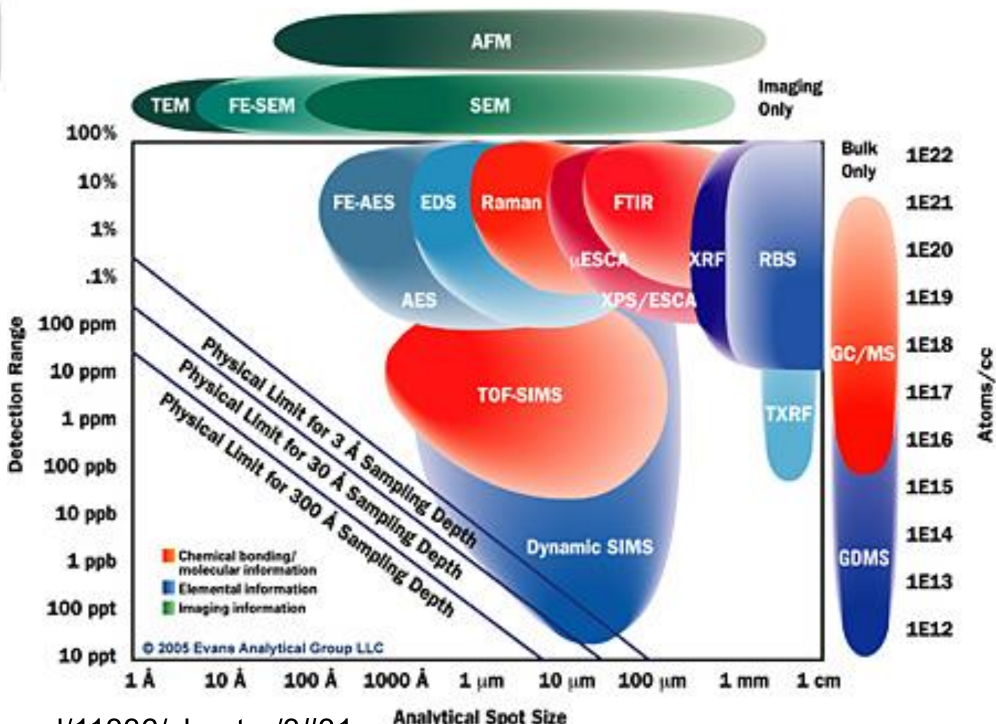
Advanced Materials Characterization



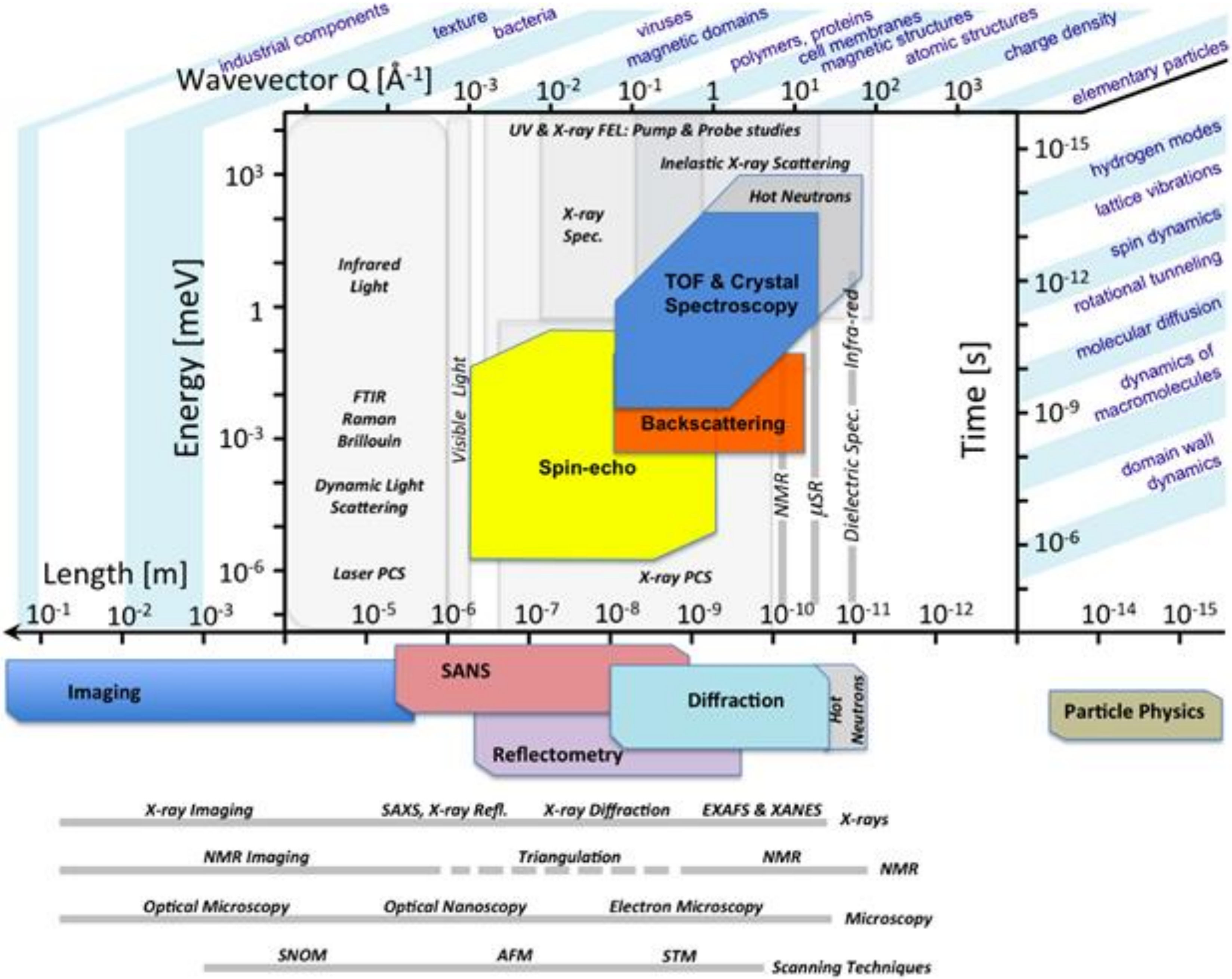
http://www.eag.com/mc/thin-film-analysis.html

Surface	Up to 1 nm	(1-3 atomic layers)
Ultra-thin film	1 to 10 nm	(3-30 atomic layers)
Thin film	10 nm to 2 μm	(30-600 atomic layers)
Bulk	>2 μm	(>600 atomic layers)

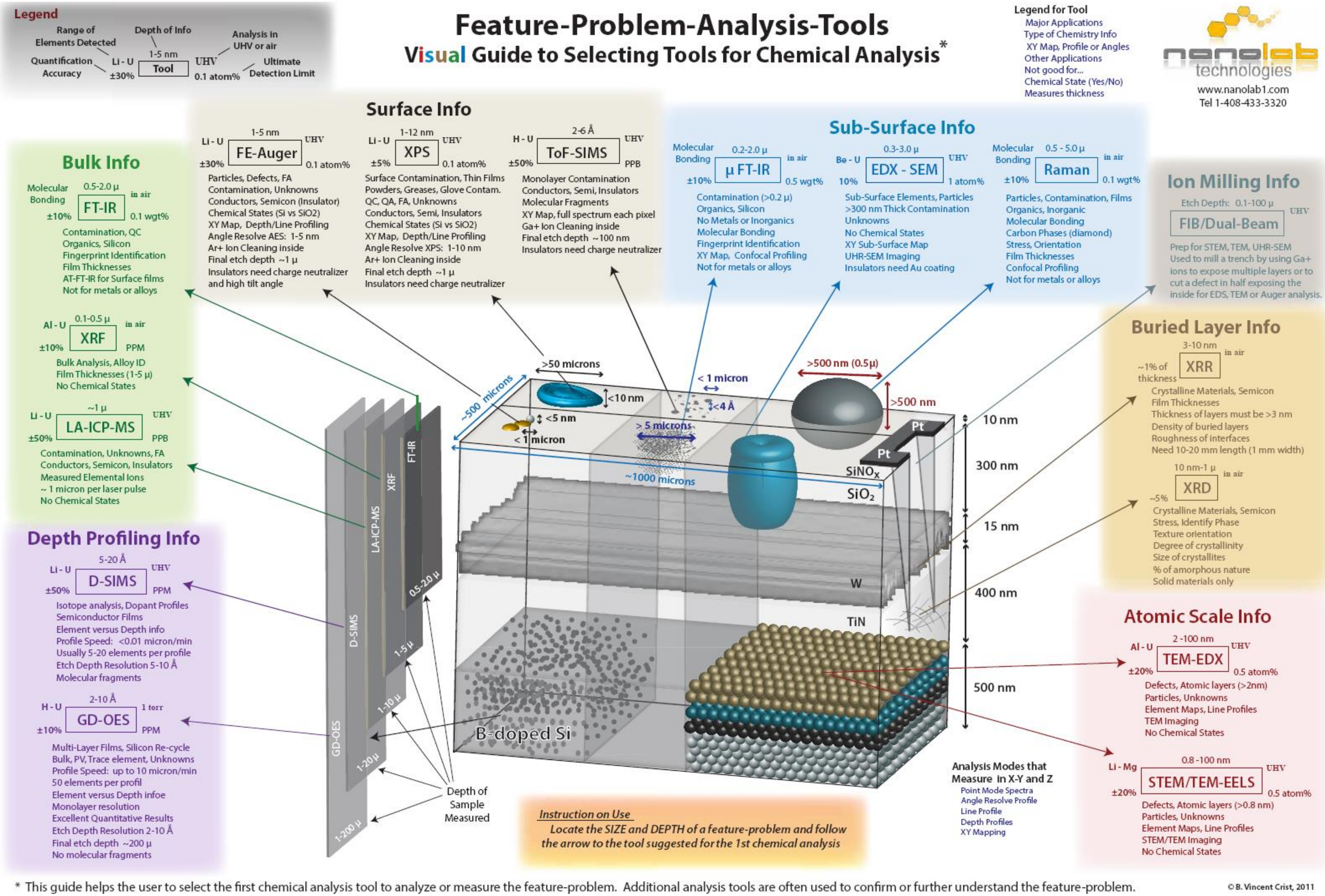
https://www.mccrone.com/mm/surface-analysis/



http://www.nap.edu/read/11336/chapter/3#31



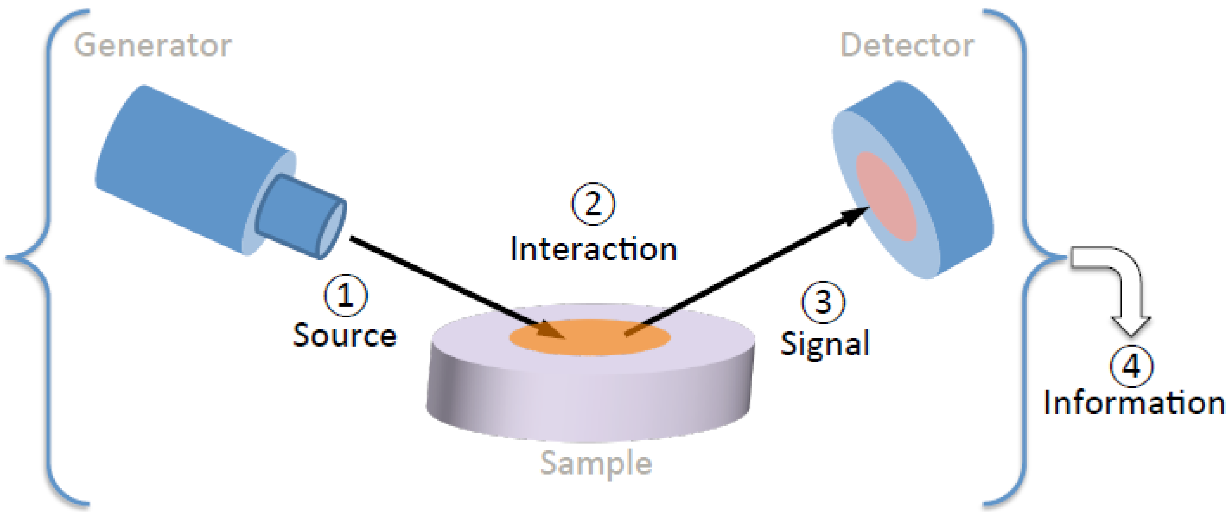
https://europeanspallationsource.se/feature-series-ess-instrument-suite



http://www.nanolabtechnologies.com/images/Feature-Problem-Analysis-Tools.png

Materials Characterization

Basic Principle



① A source radiation is aimed at the sample
② It interacts with the sample in some way
③ As a result of the interaction, a signal is produced
④ Analysis of the signal reveals information about the sample

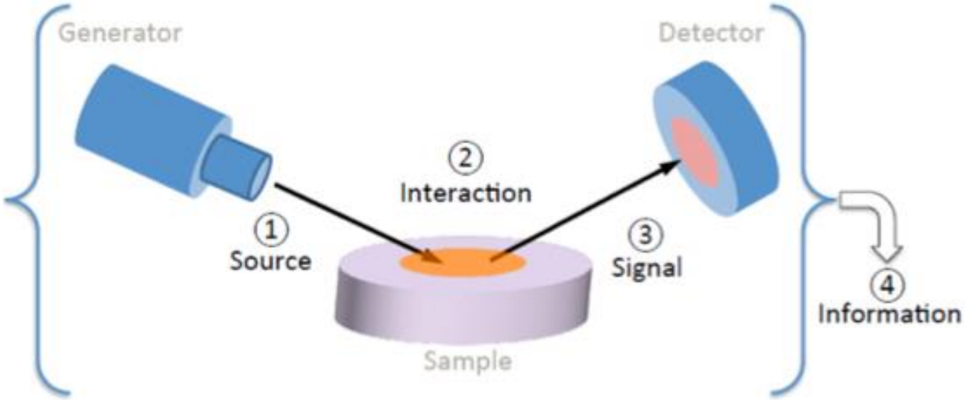
Teaching Materials: Courtesy of Prof. Dr. S. Seraphin, University of Arizona
Visiting professor at MTEC, 2015.

Source

- Photon
- Electron
- Ion
- phonon

Signal

- Photon
- Electron
- Ion
- phonon



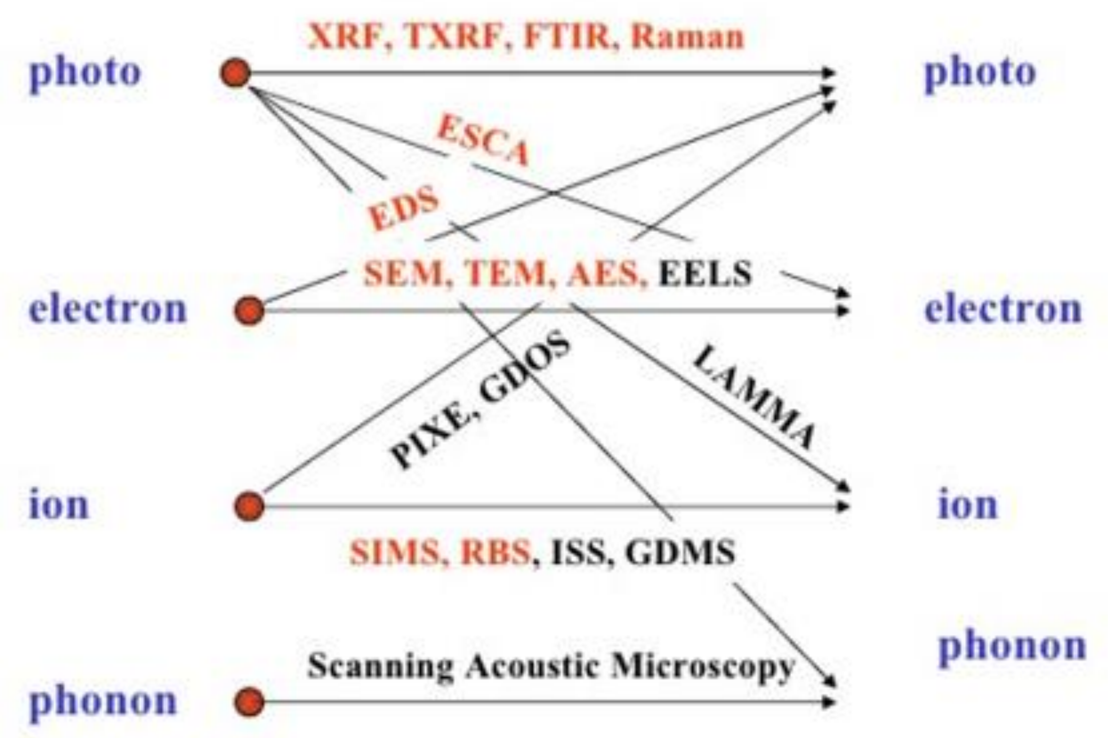


photo
XRF, TXRF, FTIR, Raman
photo
ESCA
EDS
SEM, TEM, AES, EELS
electron
electron
PIXE, GDOS
LAMMA
ion
ion
SIMS, RBS, ISS, GDMS
phonon
Scanning Acoustic Microscopy
phonon

Advanced Microscopy

Super-Resolution Microscopy Tutorial

Overview

Super-resolution microscopy is a collective name for a number of techniques that achieve resolution below the conventional resolution limit, defined as the minimum distance that two point-source objects have to be in order to distinguish the two sources from each other. There are two closely related values for the diffraction limit, the Abbe and Rayleigh criterions. The difference between the two is based on the definition that both Abbe and Rayleigh used in their derivation for what is meant by two objects being resolvable from each other. In practical applications, this difference is small. The Abbe criterion is defined as:

$$r = \frac{0.5\ \lambda}{NA} = \frac{0.5\ \lambda}{n\ sin(\theta)}$$

while the Rayleigh criterion defines the resolution mathematically as:

$$r = \frac{0.61\ \lambda}{NA} = \frac{0.61\ \lambda}{n\ sin(\theta)}$$

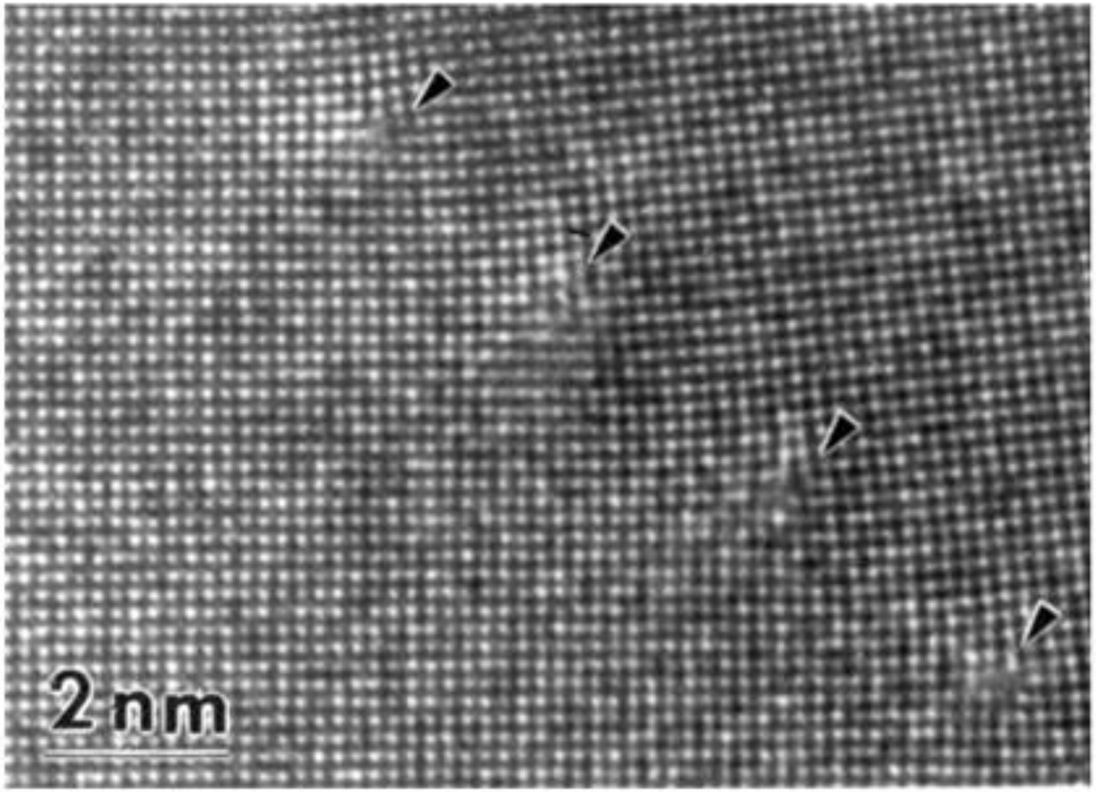


Fig. 1.1 Atomic-resolution electron micrograph of Al 6° [001] symmetric tilt grain boundary with misfit accommodation by [110]/2 edge dislocations (arrowed). Each black spot corresponds to projection of individual Al atomic column.

http://pubs.rsc.org/en/content/chapterhtml/2015/bk9781849738057-00001?isbn=978-1-84973-805-7

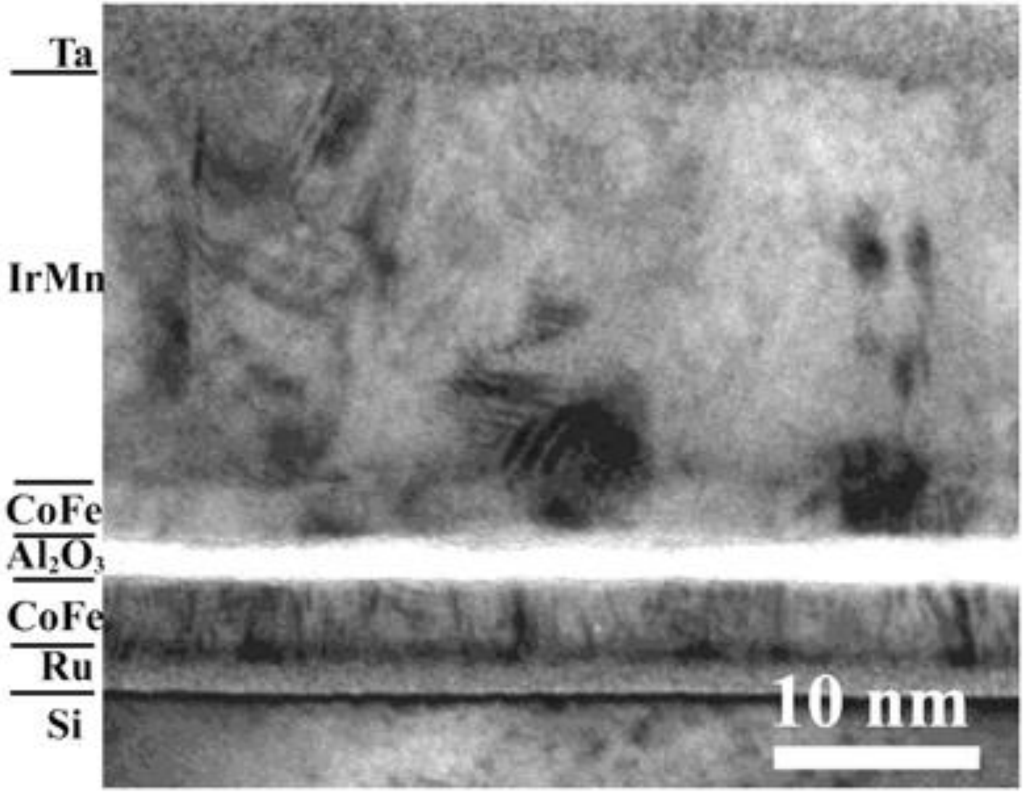


Fig. 1.2 Bright-field electron micrograph showing the cross-section of a typical magnetic tunneling transistor device structure with a Ru seed layer between the Si(001) collector substrate and the CoFe base layer (Reproduced from ref. 11).

http://pubs.rsc.org/en/content/chapterhtml/2015/bk9781849738057-00001?isbn=978-1-84973-805-7

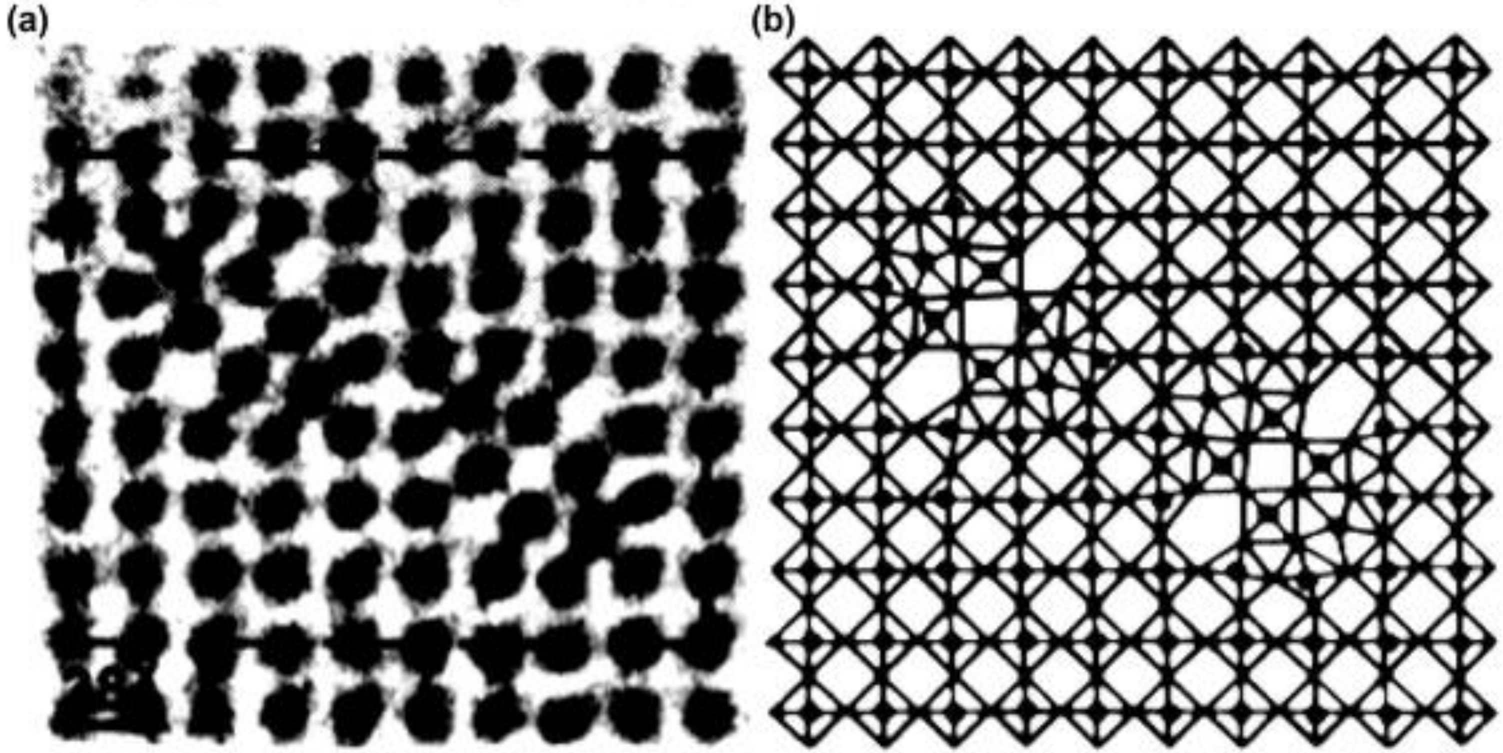
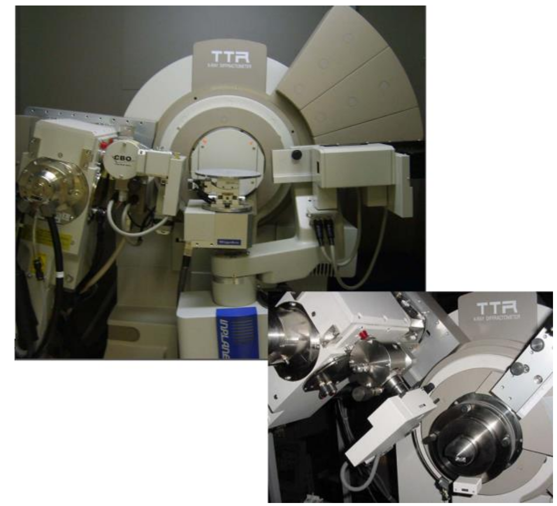
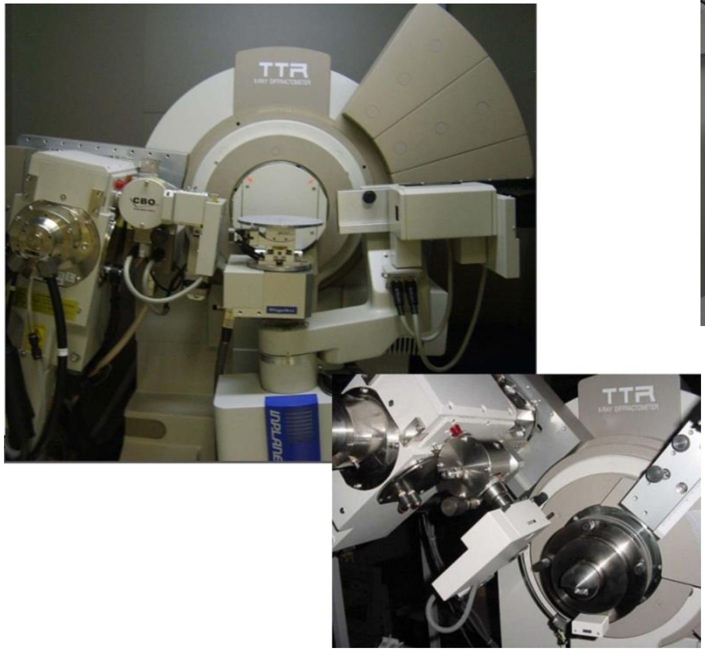


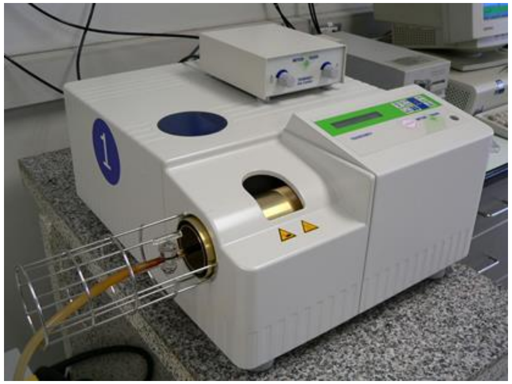
Fig. 1.6 (a) Atomic-resolution electron micrograph of non-stoichiometric (W, Nb)$O_{2.93}$ showing pairs of pentagonal bipyramidal columnar defects. (b) Corresponding structural model. Occupied tunnel sites are located by direct visual inspection (Reproduced from ref. 42).

หน่วยวิเคราะห์ลักษณะเฉพาะของวัสดุ



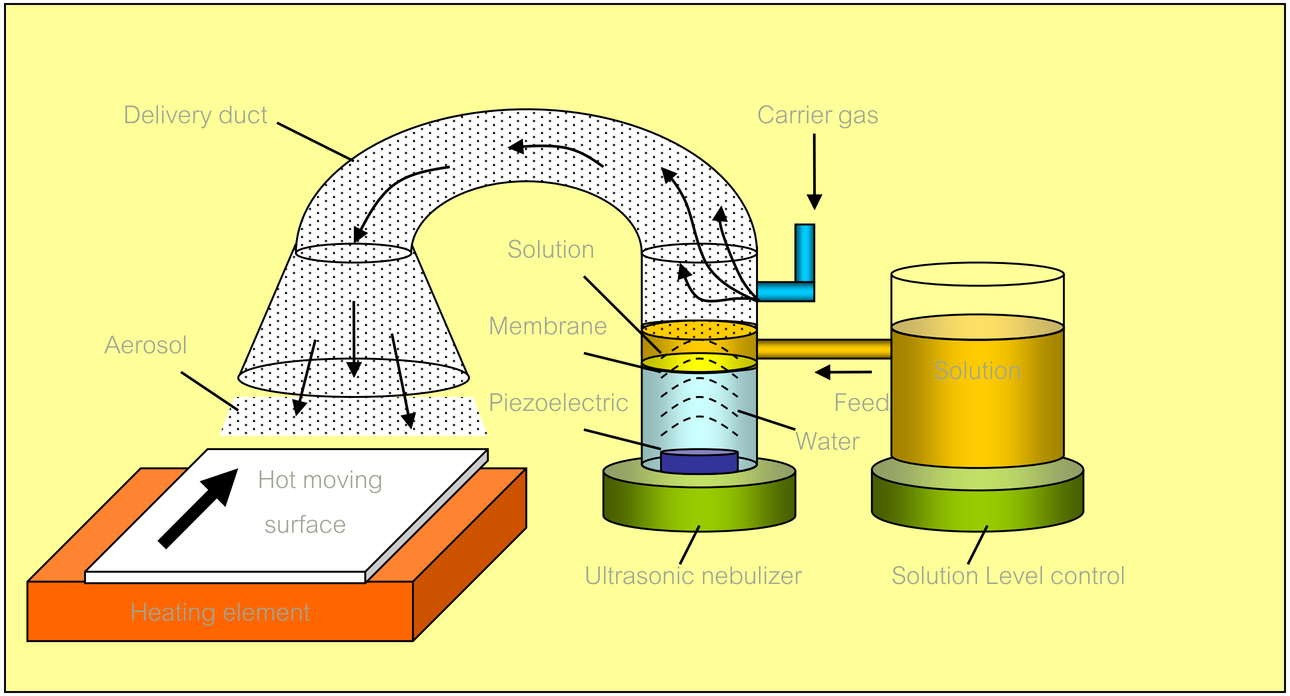
Physical Characterization Lab: PhCL





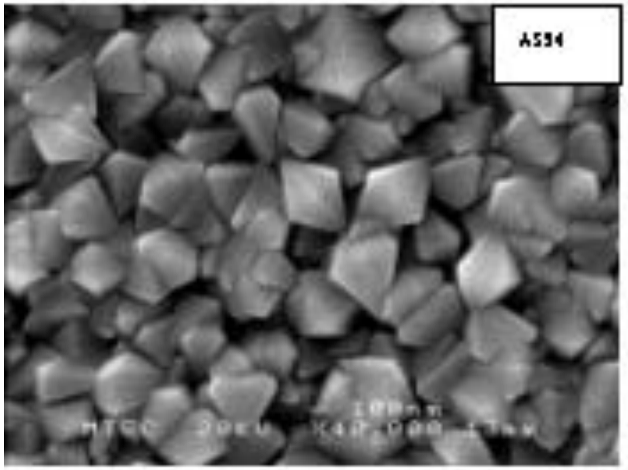
Research Examples

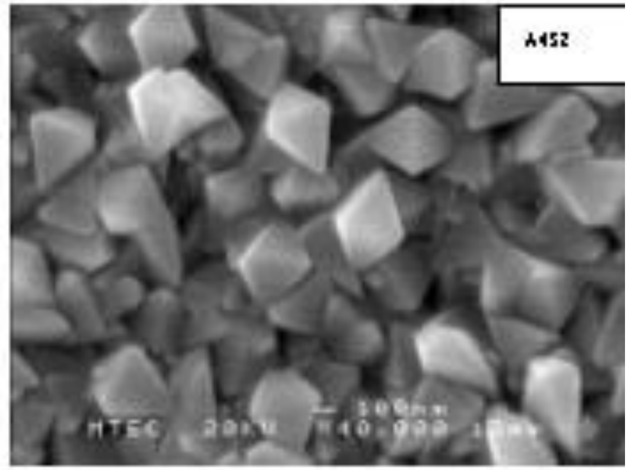
SnO_2:F deposition



Schematic diagram of pyrosol deposition
("pyrosol" = pyrolysis of aerosol)

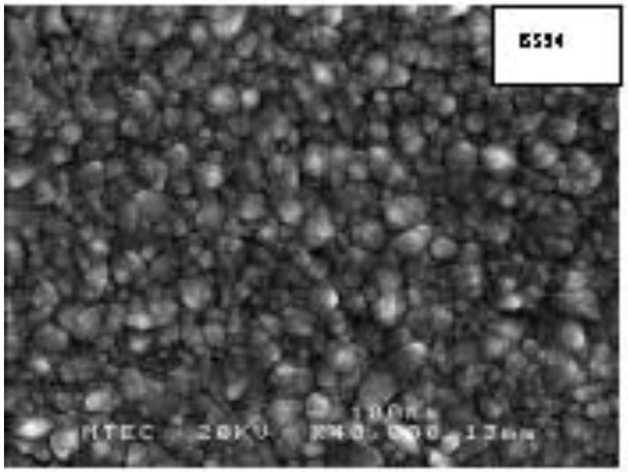
394 ^{o}C, condition A

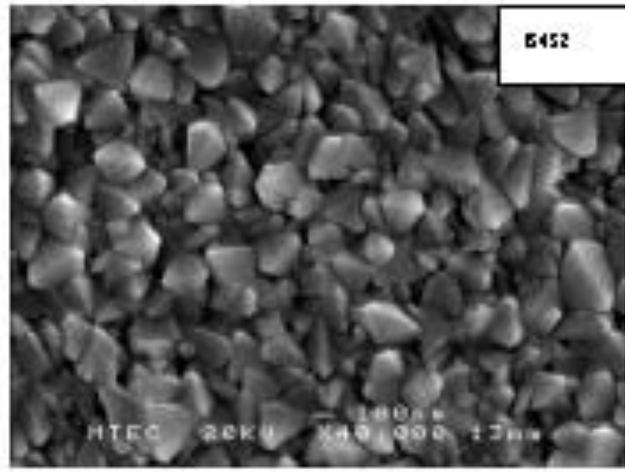




432 ^{o}C, condition A

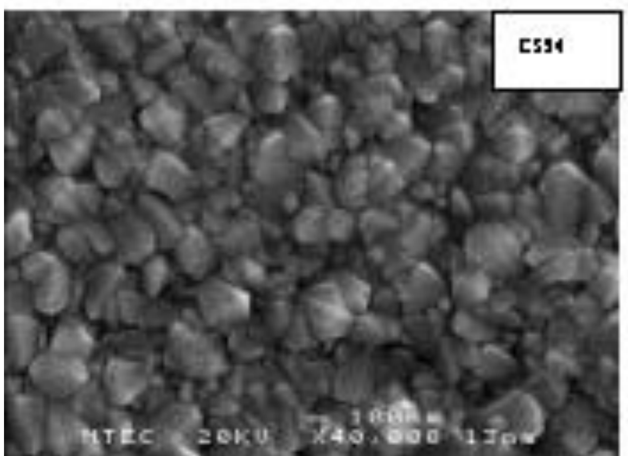
394 ^{o}C, condition B

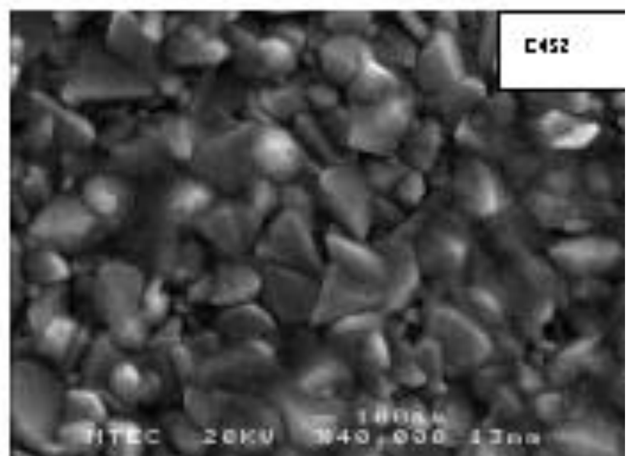




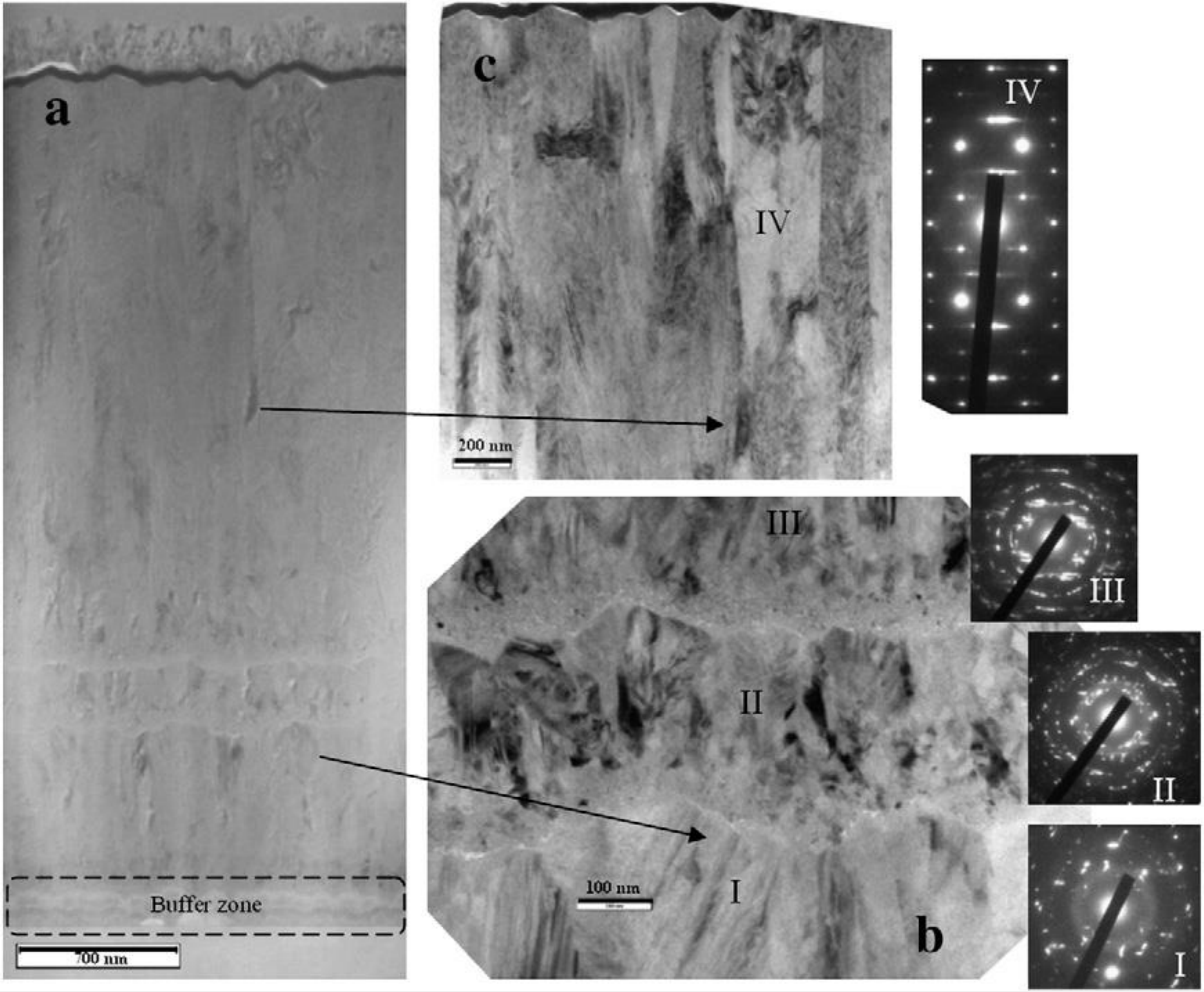
432 ^{o}C, condition B

394 ^{o}C, condition C

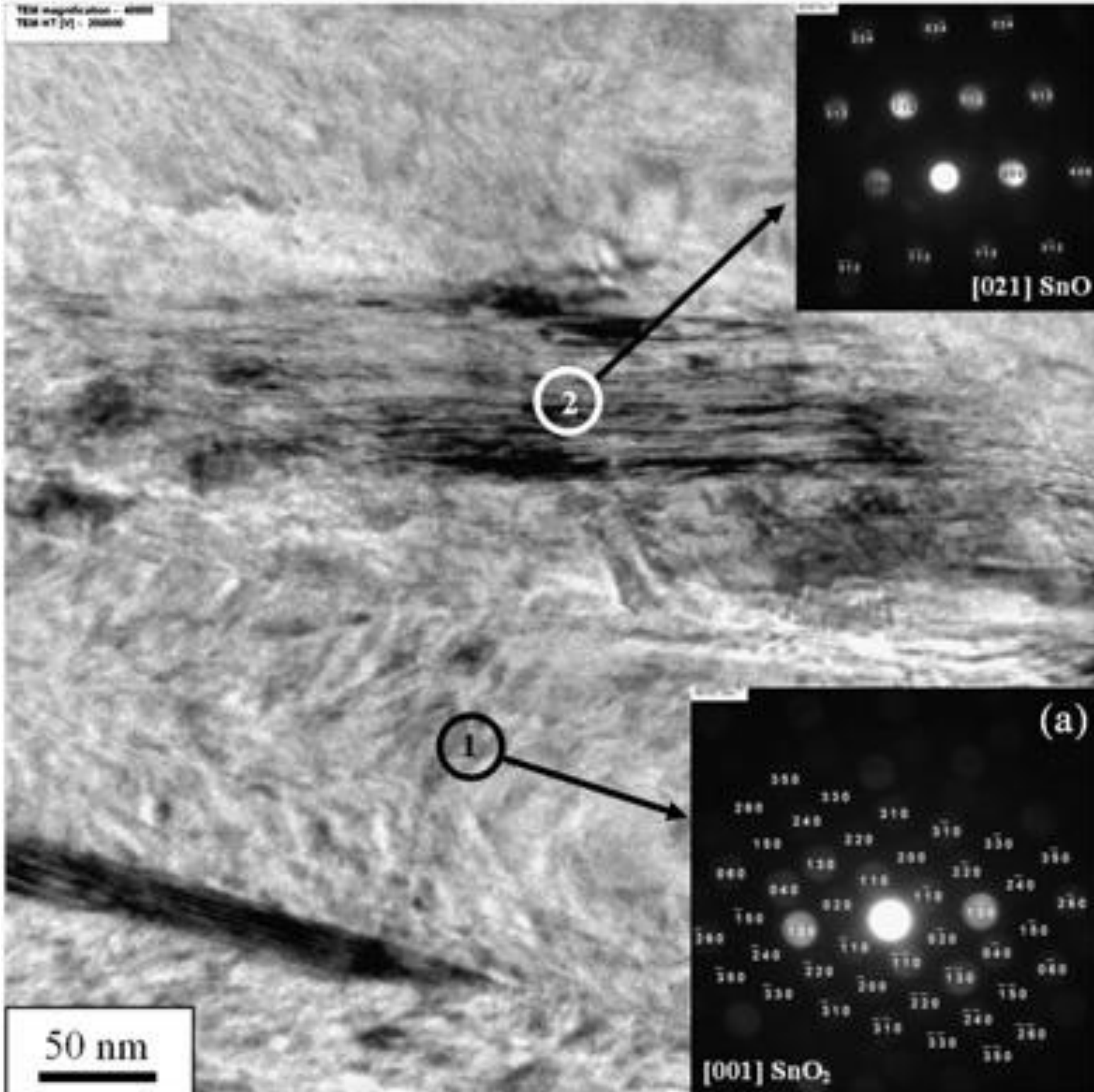




432 ^{o}C, condition C

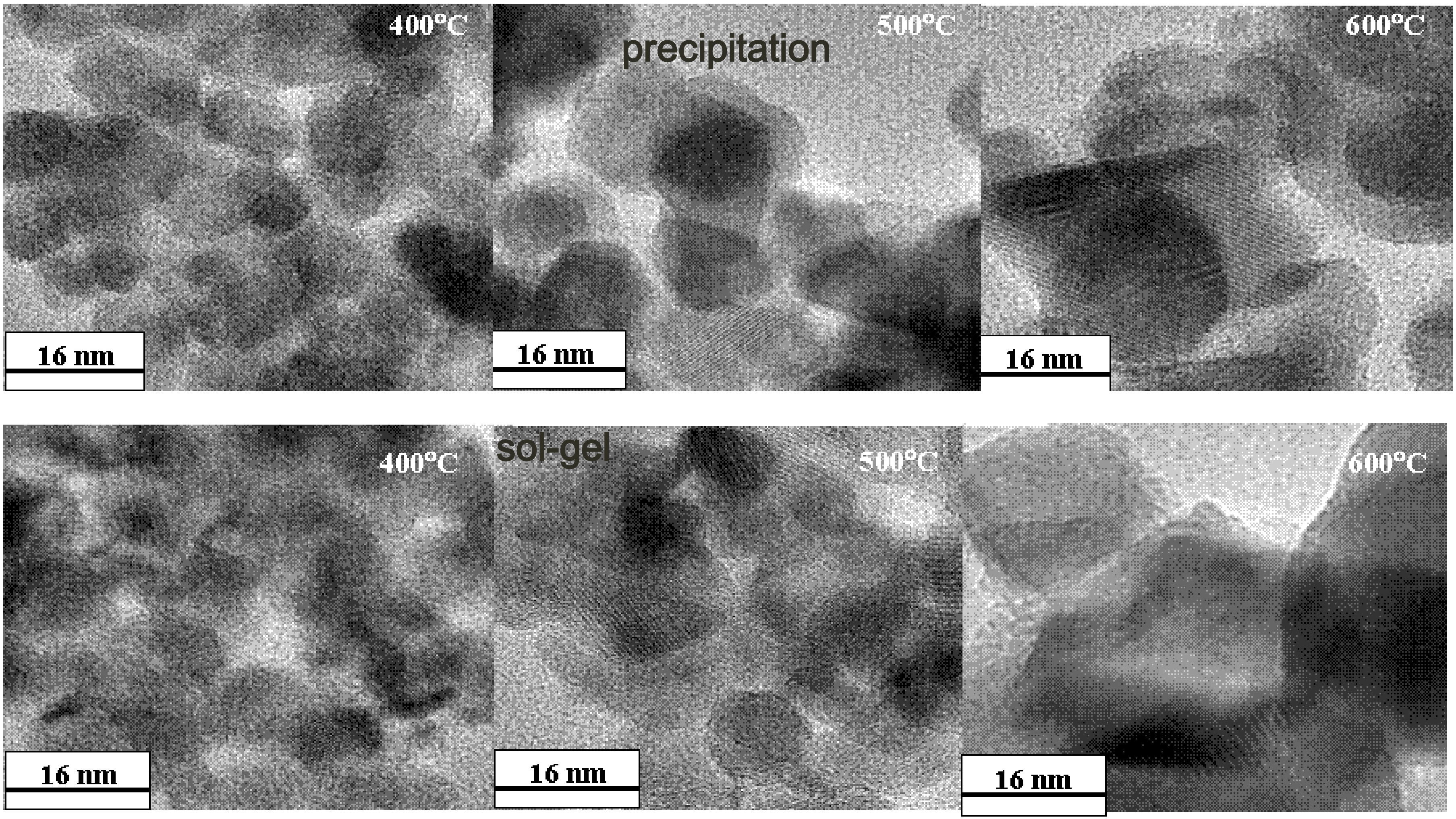


a
Buffer zone
700 nm
c
IV
200 nm
IV
III
II
100 nm
I
b
III
II
I



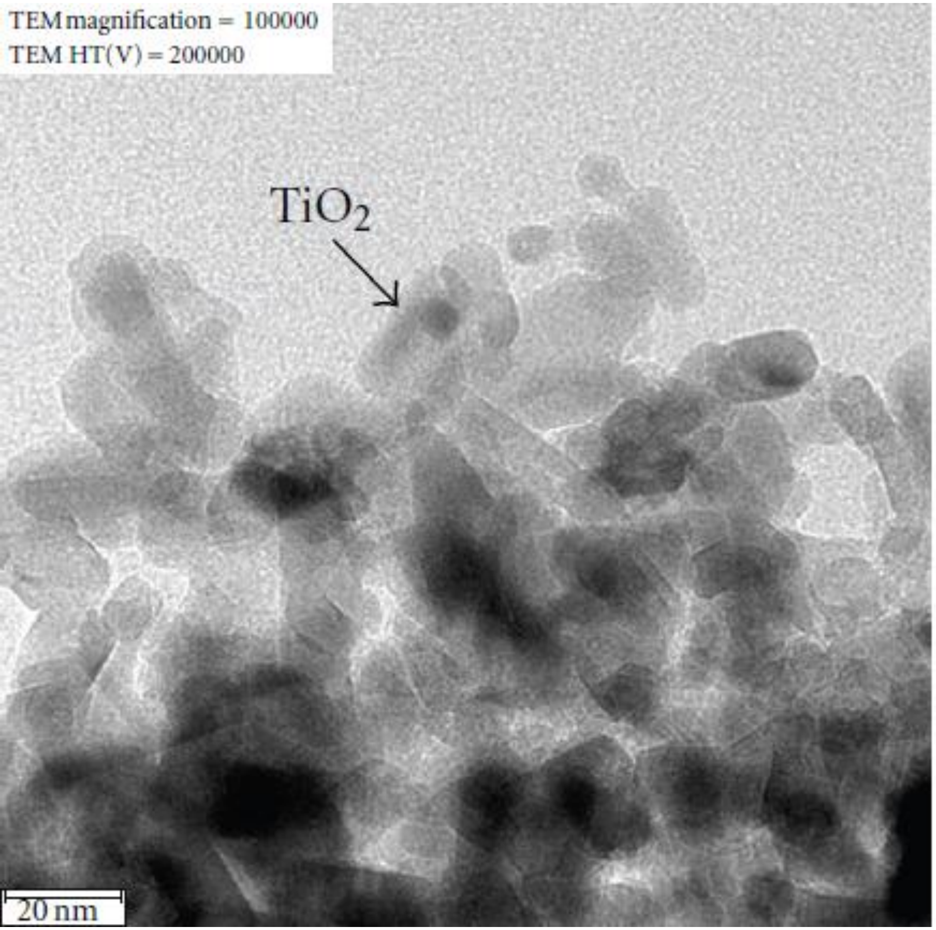
Materials Letters 65 (2011) 2610–2613

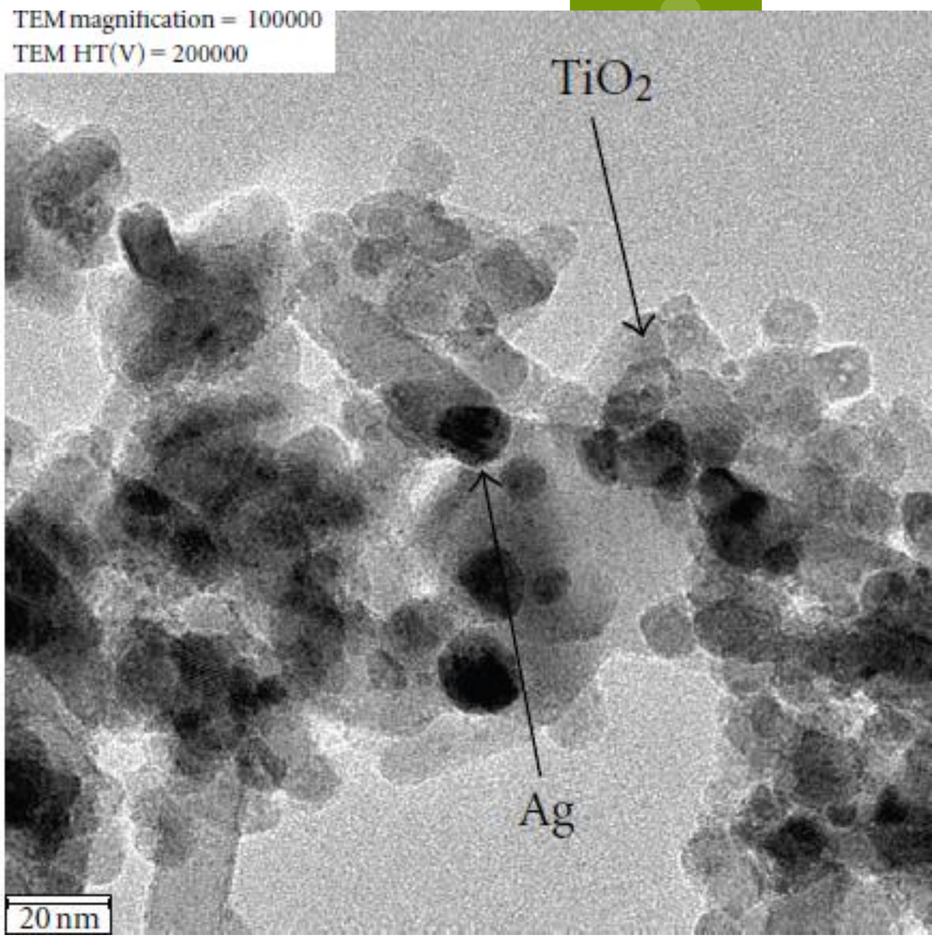
TiO_2 nanoparticles (precipitation vs sol-gel)



Eur. J. Inorg. Chem. ***2008, 974–9***

TiO_2 & Ag nanoparticles



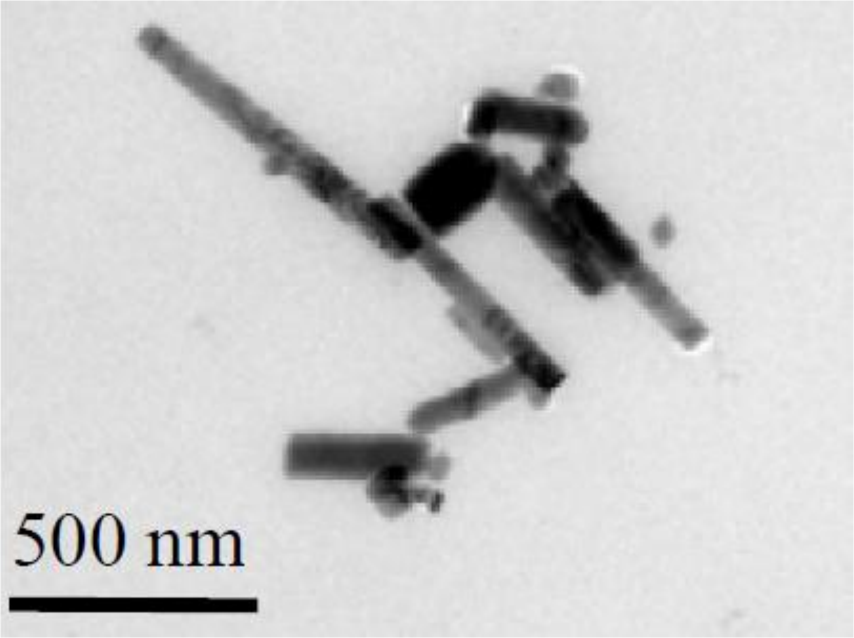


TEM micrographs of the TiO2 nanotubes (a) before and (b) after immersion in the $AgNO_3$ solution with UV exposure for 120minutes.

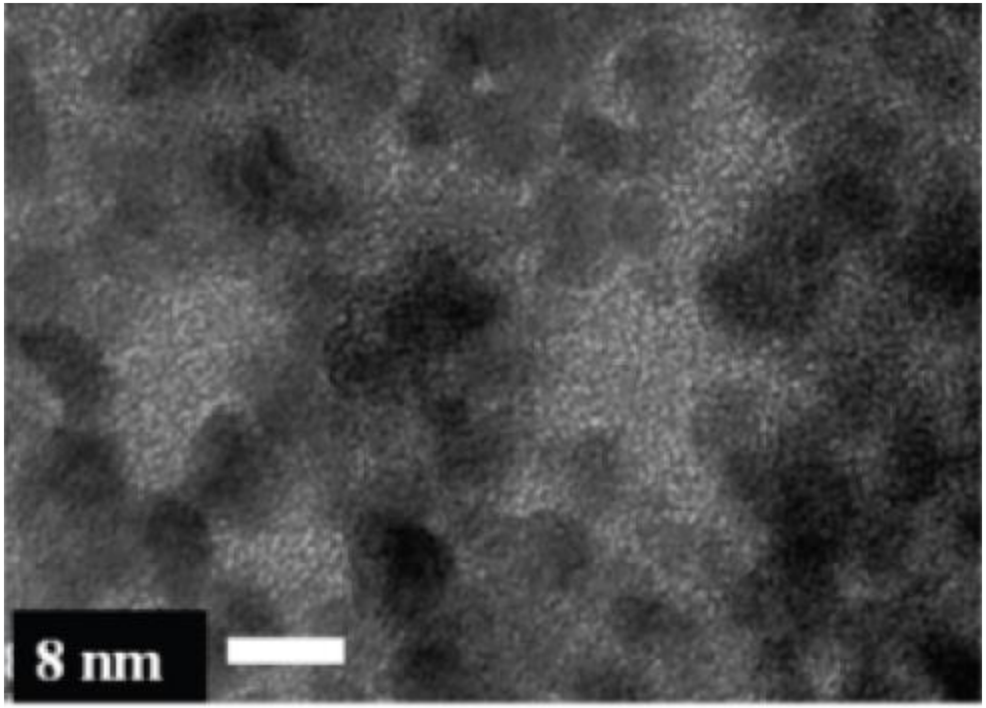
International Journal of Photoenergy, volume 2011, DOI: 10.1155/2011/258635

ZnO nanoparticles (solvothermal vs precipitation)

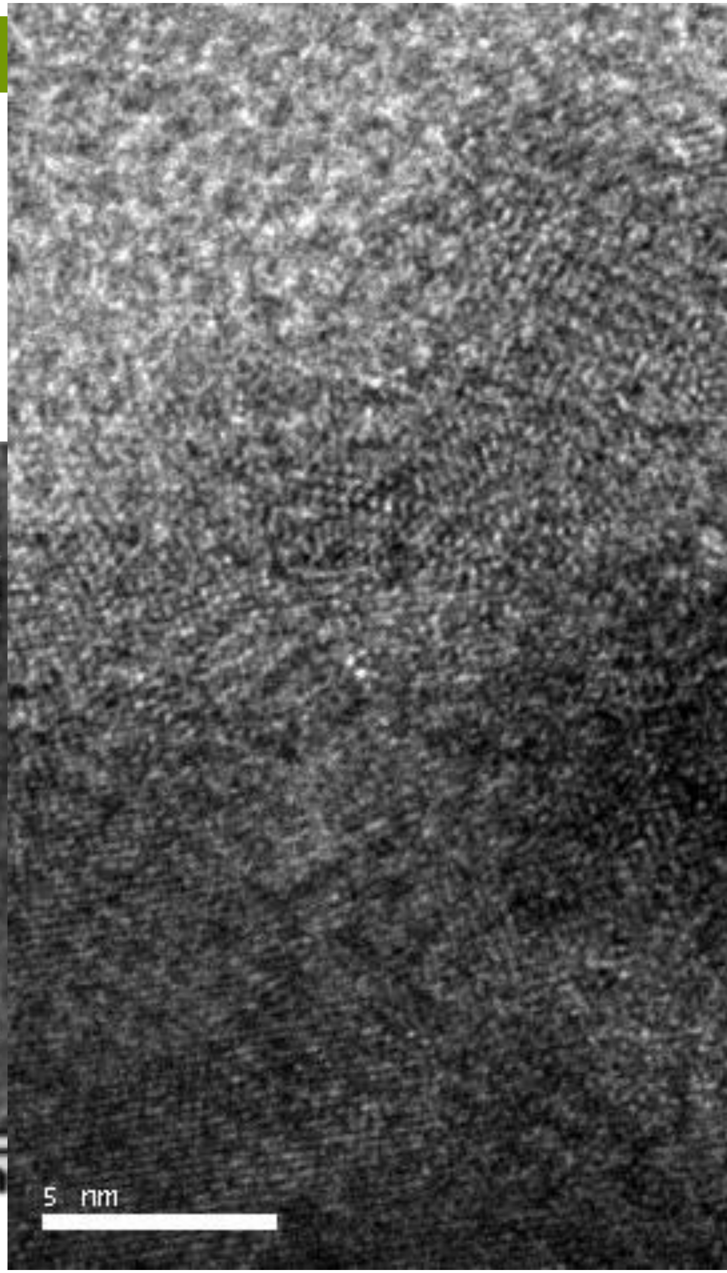
- solvothermal
- nanorods

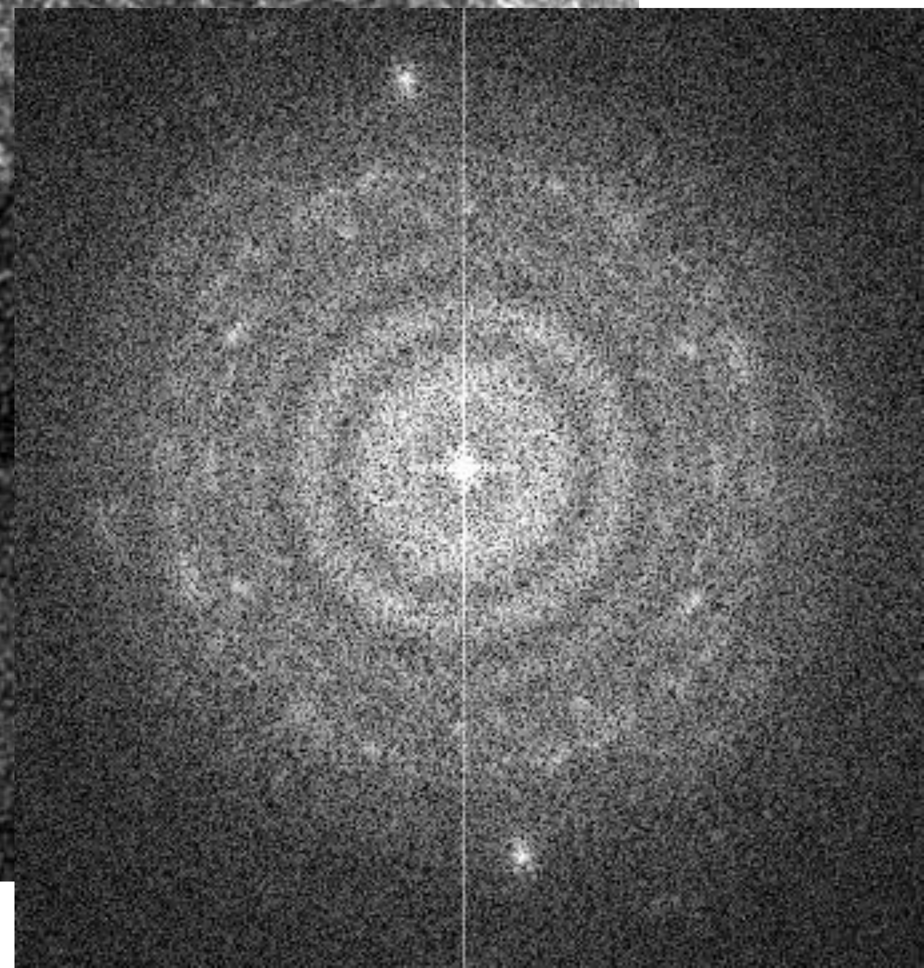


- Precipitation
- spheres

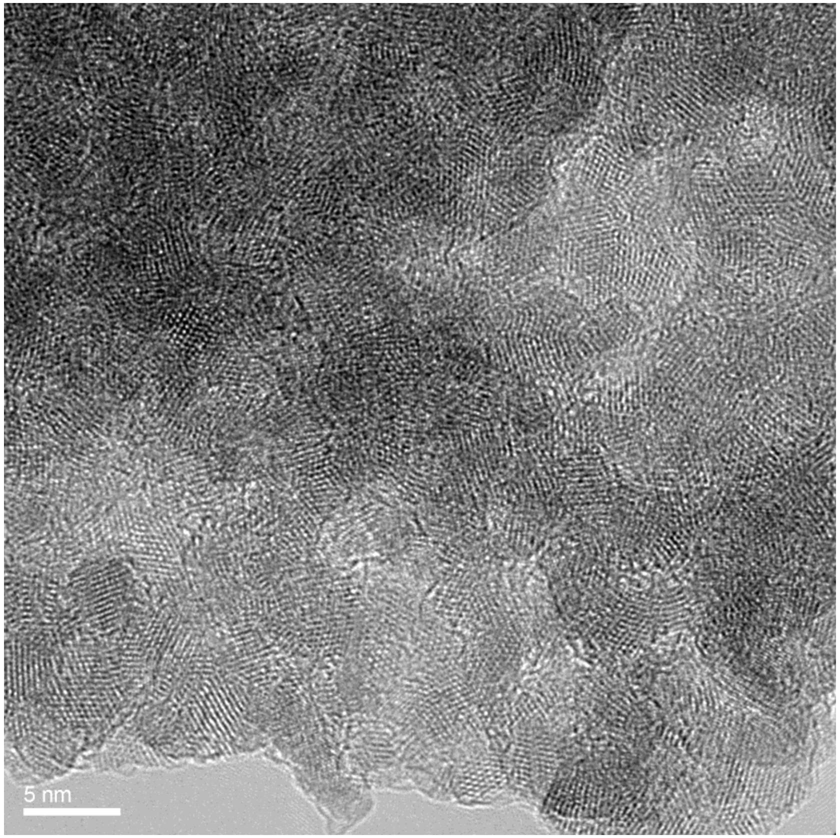


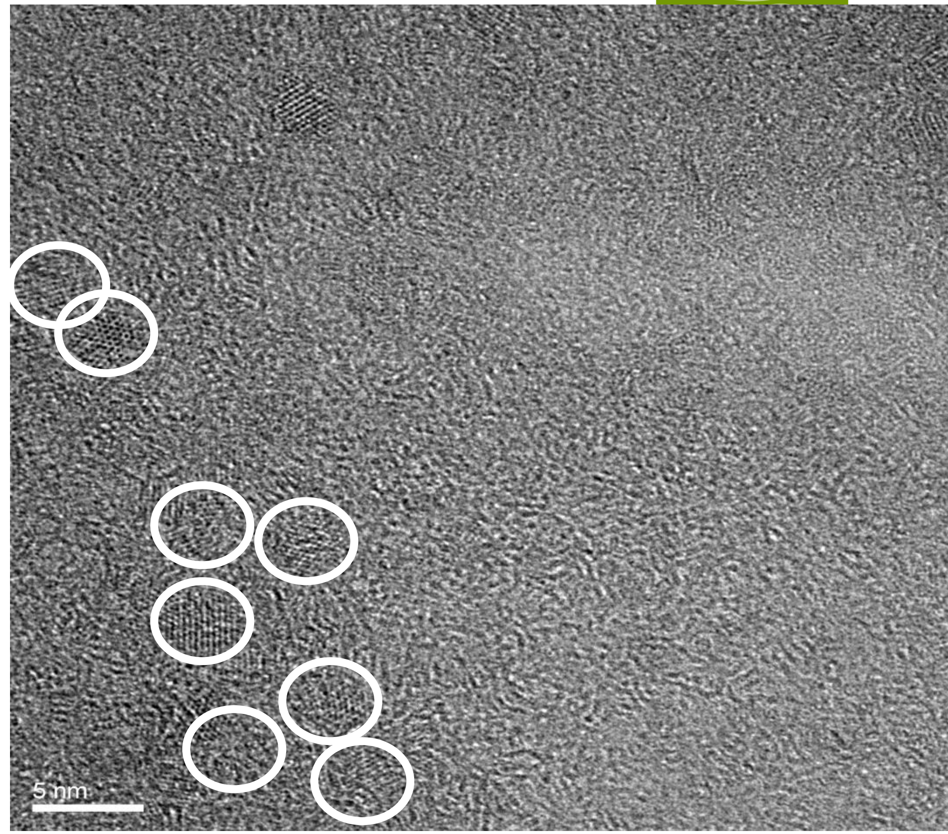
HR





HREM images of $ZnS:Mn^{2+}$





Chitosan capped $ZnS:Mn^{2+}$

High concentration of Polycrystalline agglomerates of 3-5 nm crystallites

Average crystallite size = 2.54 nm

Crystallite density = 80,971 μm^2.

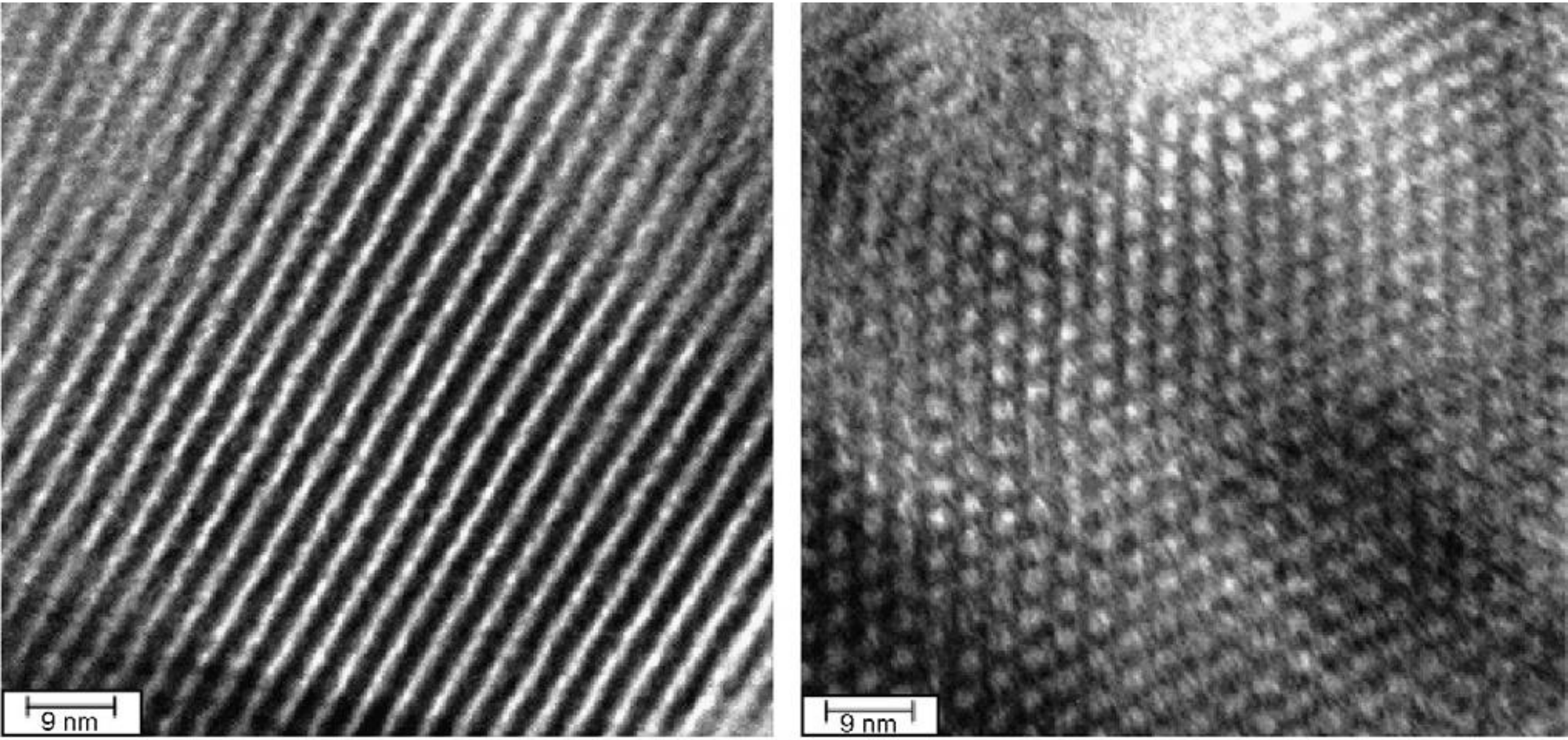
PVA capped $ZnS:Mn^{2+}$

Rare Polycrystalline agglomerates of 3-5 nm crystallites (encircled for visual effects)

Average crystallite size = 2.56 nm

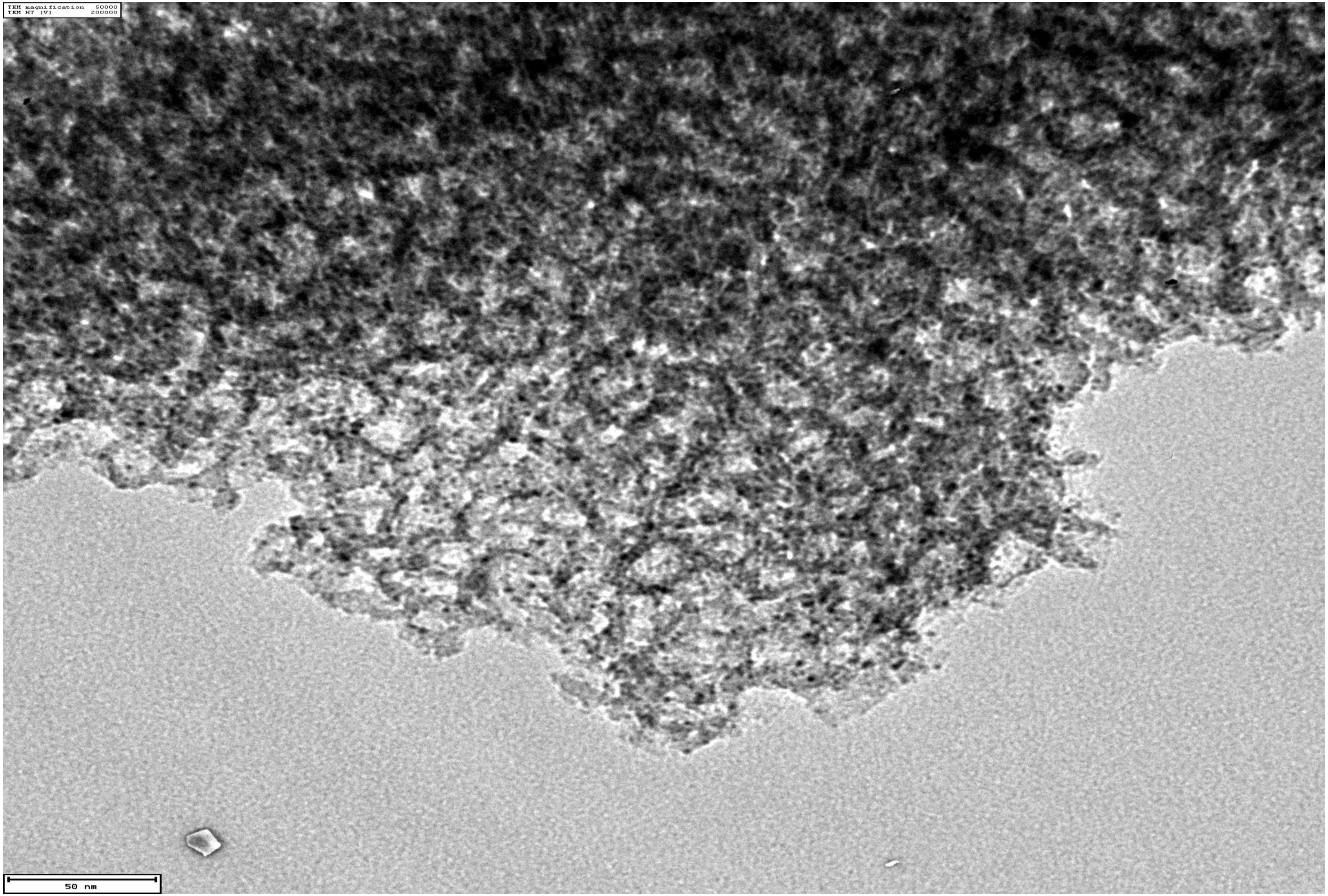
Crystal density = 19,856 μm^2

TEM images of mesoporous silicas



perpendicular (left) and parallel (right) to the pores

Journal of Molecular Catalysis B: Enzymatic 56 (2009) 246–252


TEM magnification 50000
TEM HT [V] 200000
50 nm

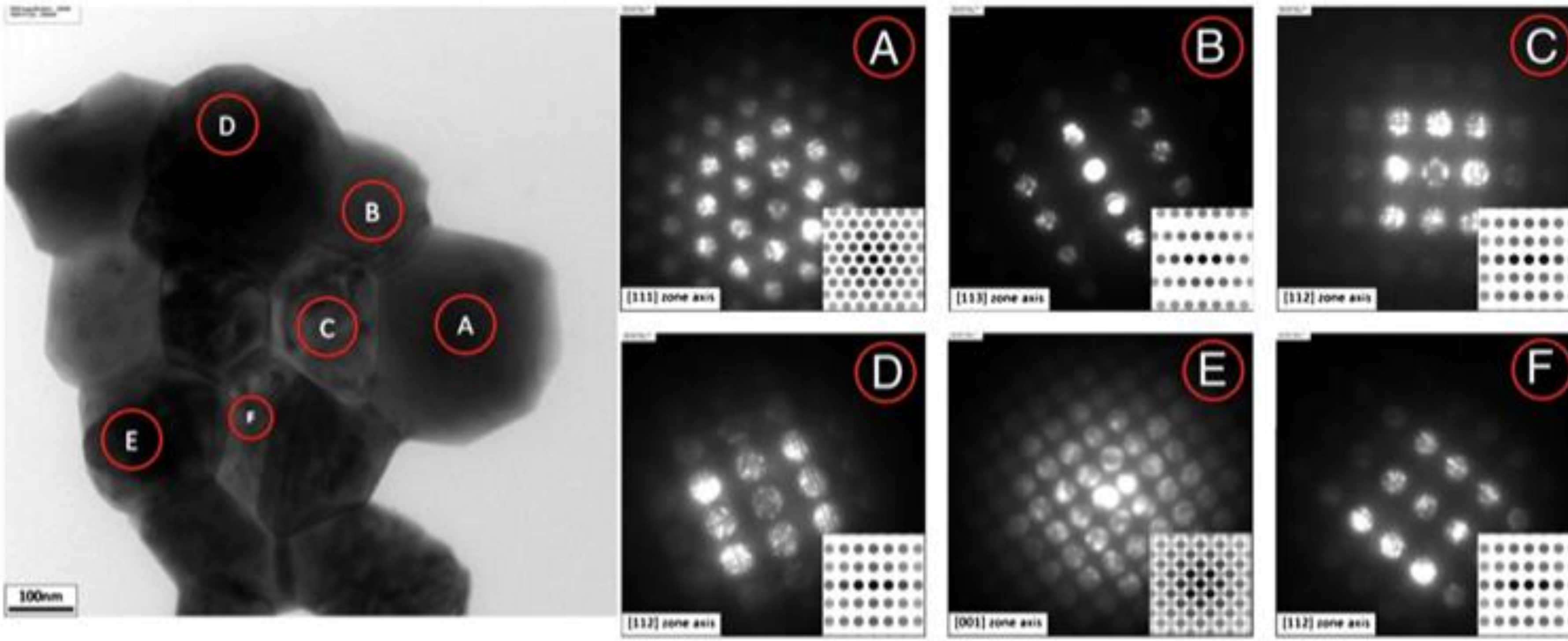


Fig. 4. CBED patterns of $Ba(Zr_{0.5}Ti_{0.5})O_3$ obtained from 6 grains (A–F).

Materials Letters 82 (2012) 205-207

Conclusion

- Advanced Microscopy is not difficult but
  - Equipment expensive
  - Maintainance expensive
  - Equipment operators must be skillful
- But it's conventional
- In order to characterize advanced nanomaterials
  - Advanced microscopy
  - Advanced spectroscopy
  - More expensive equipment
  - More skillful operators

Thank you

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