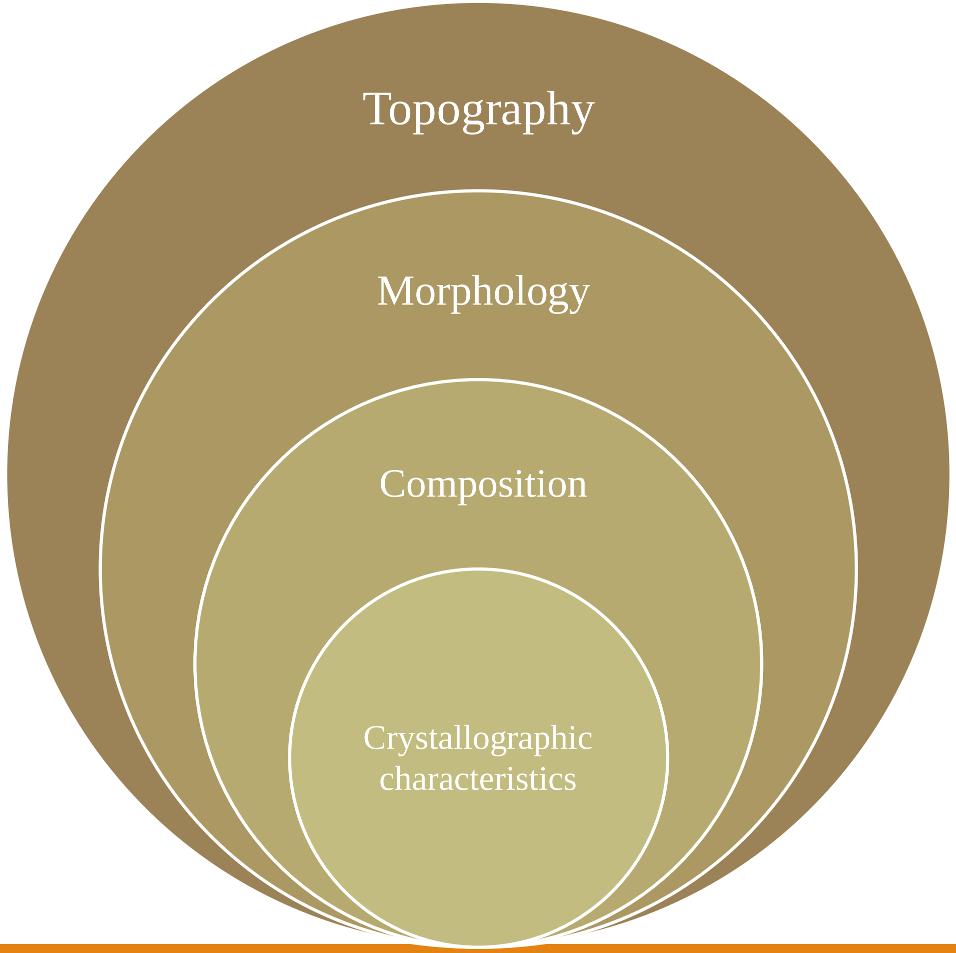
Scanning Electron Microscopy, SEM

ERKKA KOSKENNIEMI, TRANG PHAM, 3.5.2023

Content

- Introduction to Scanning Electron Microscope
  - Purpose
  - How does SEM work
  - Challenges
  - Advantages/Disadvantages
  - Research examples

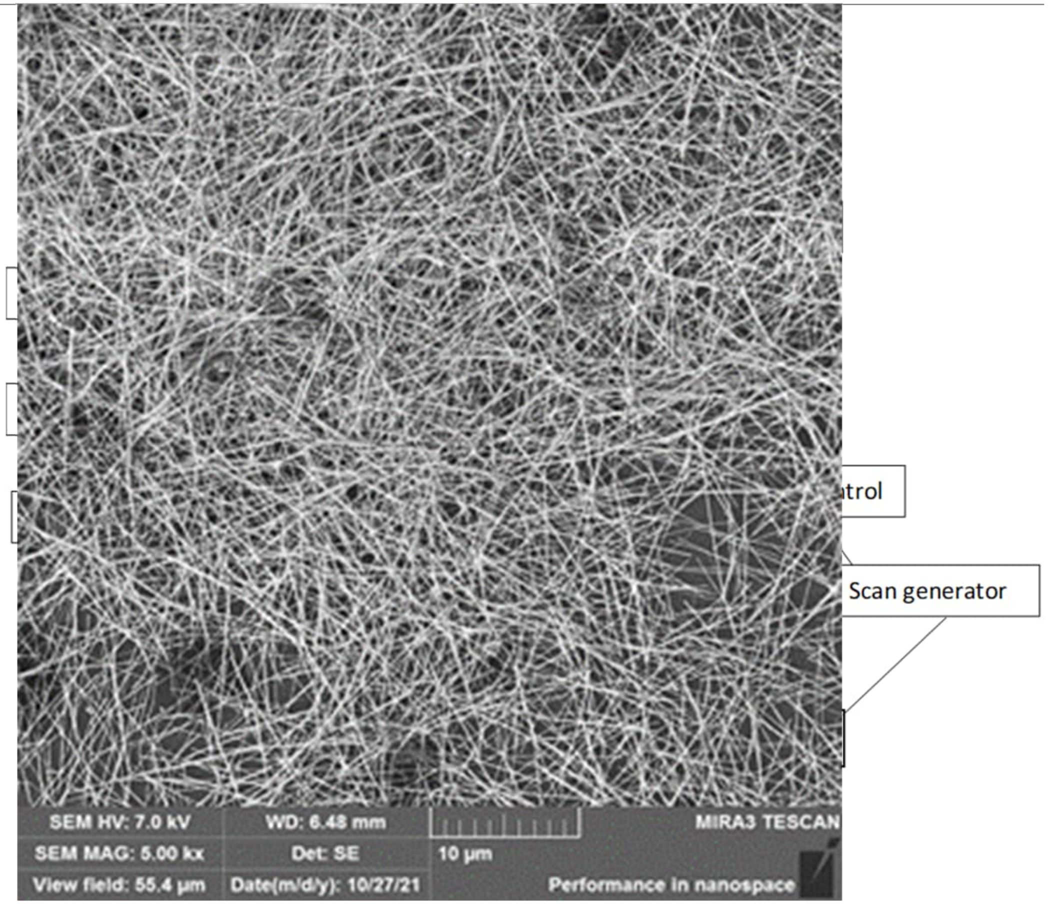
What is SEM used for?



SEM Instrumentantation

Consists of:

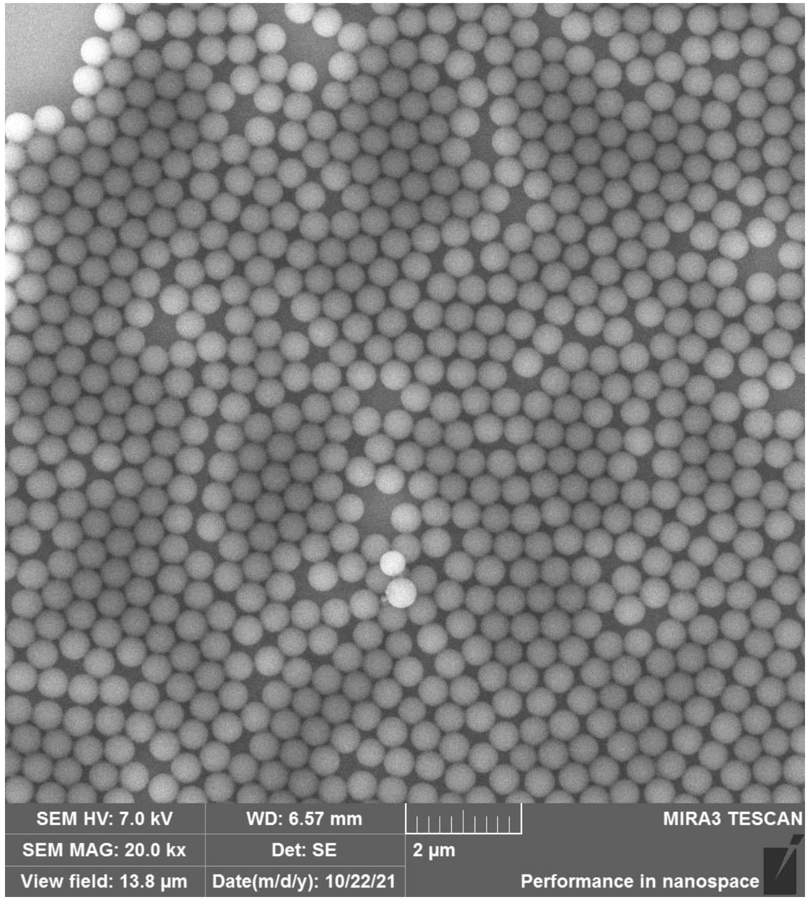
- Electron gun
- Electromagnetic coils (scan coils), objective lenses and condensers.
- Vacuum chamber
- Detector

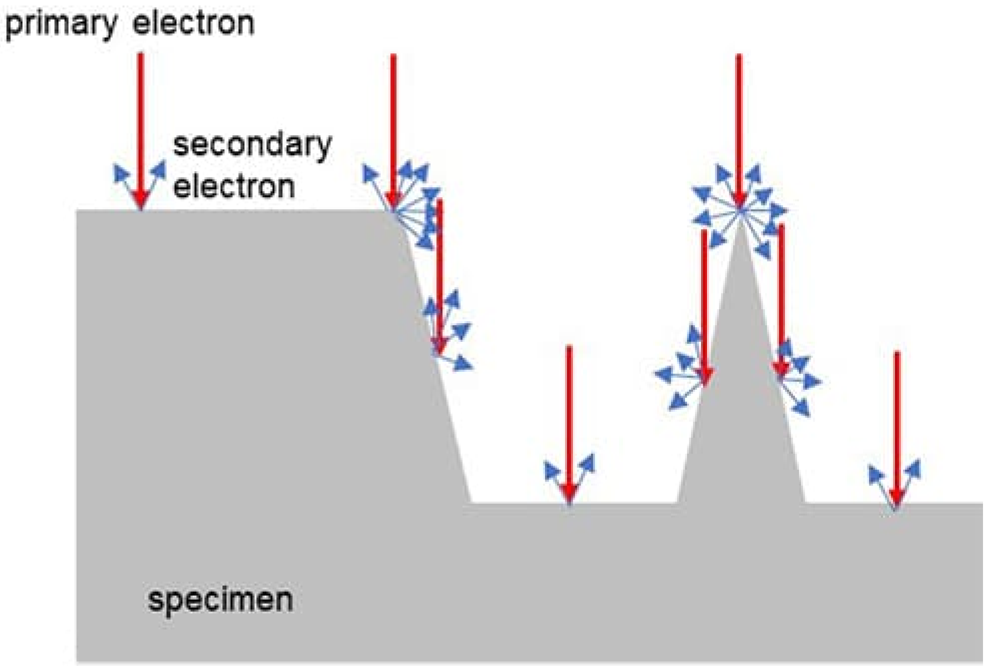


[1]

How does SEM work? Secondary electrons

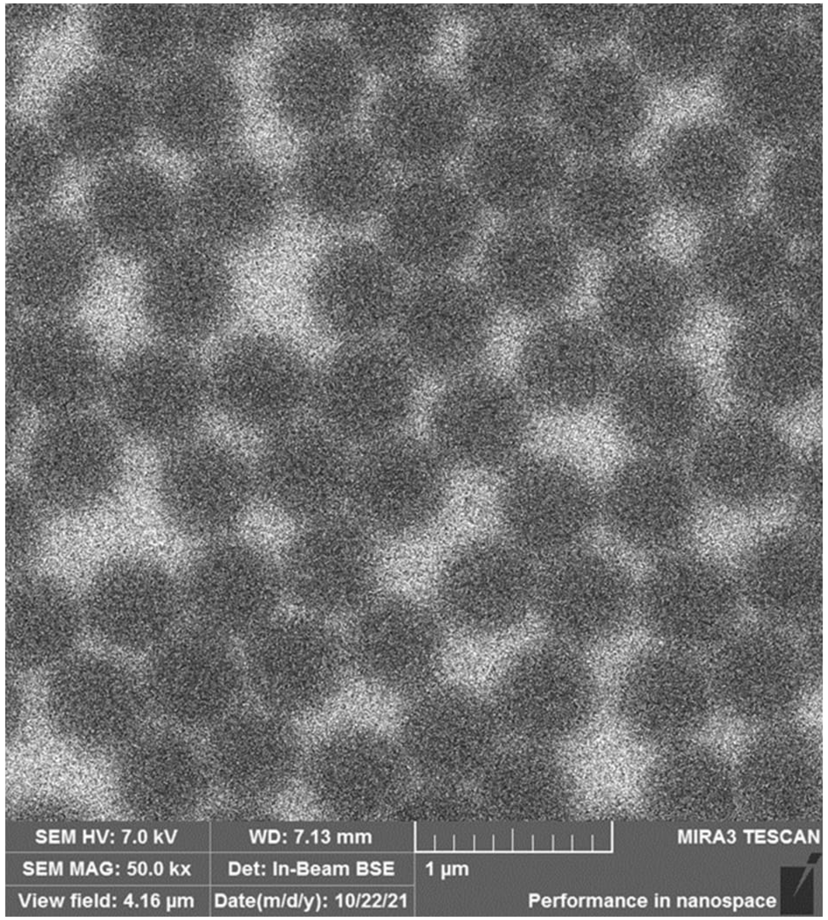
[1]





[2]

How does SEM work? Backscattered electrons



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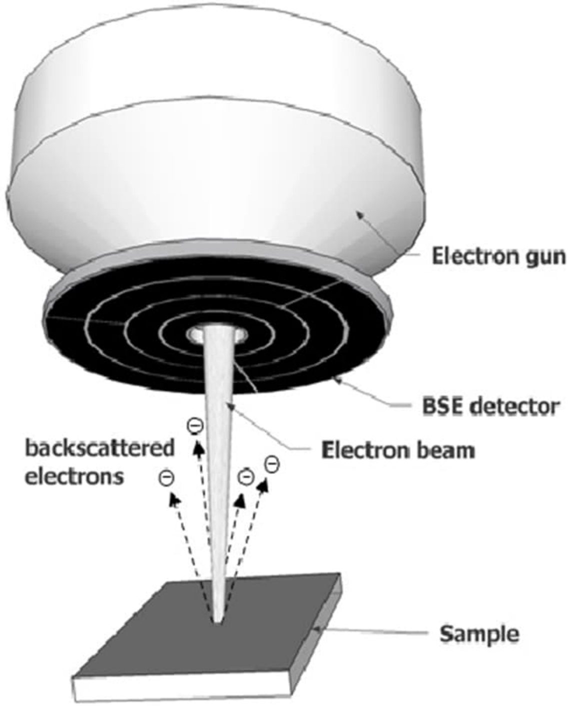
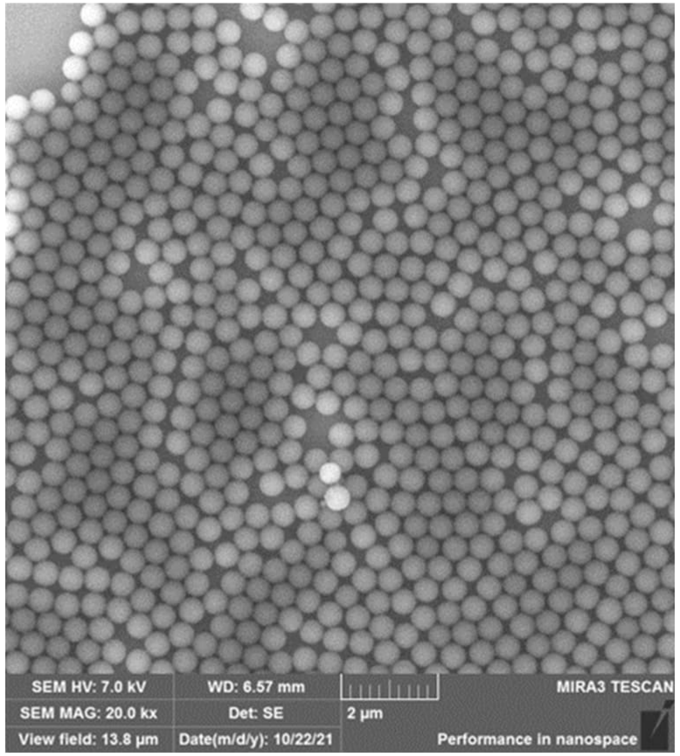


[4]

Also some X-rays

Difficulties: Edge effect



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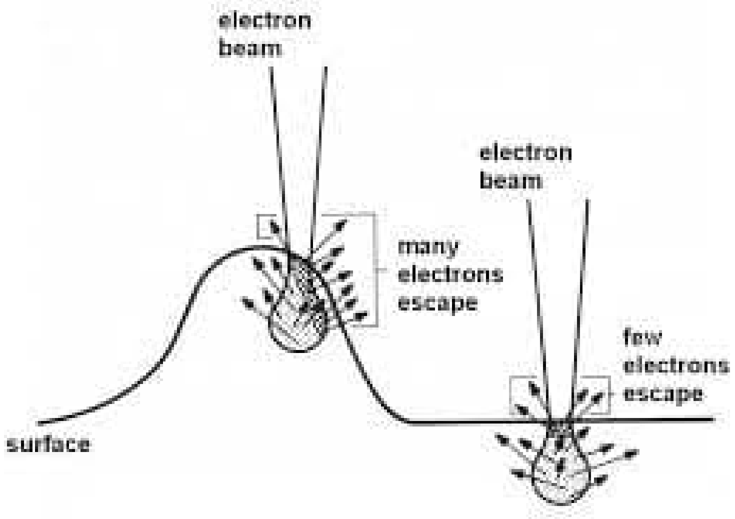


Edge effect [3]

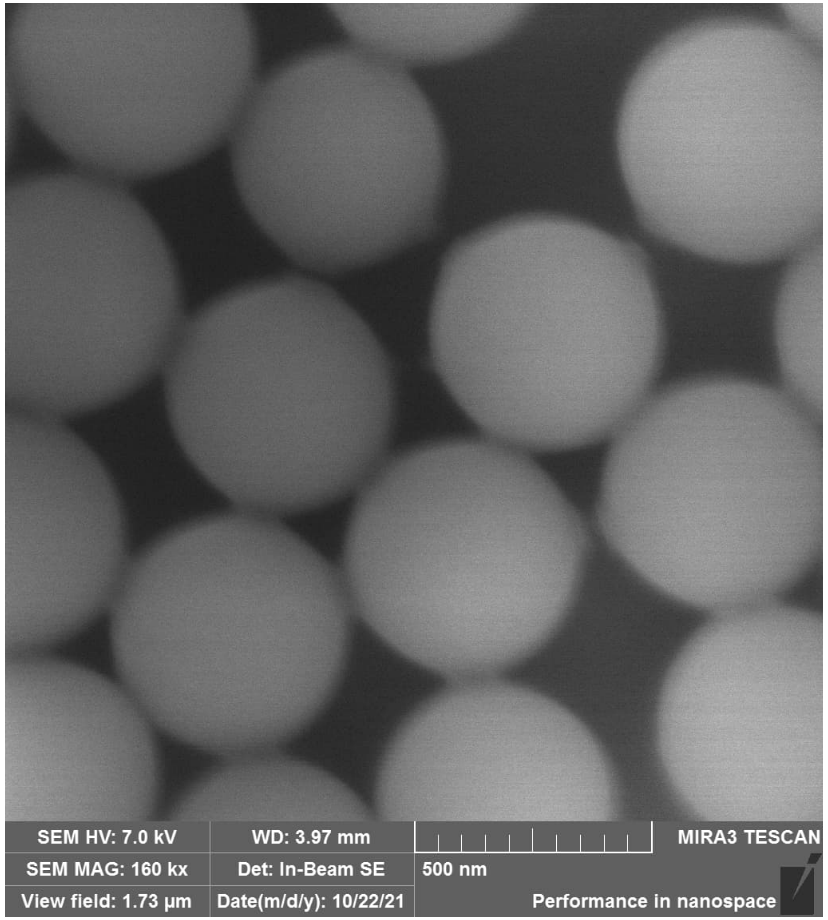
Difficulties: Holes or hills?

Holes can look like hills

Hard to differentiate with naked eyes

Solution: Tilting

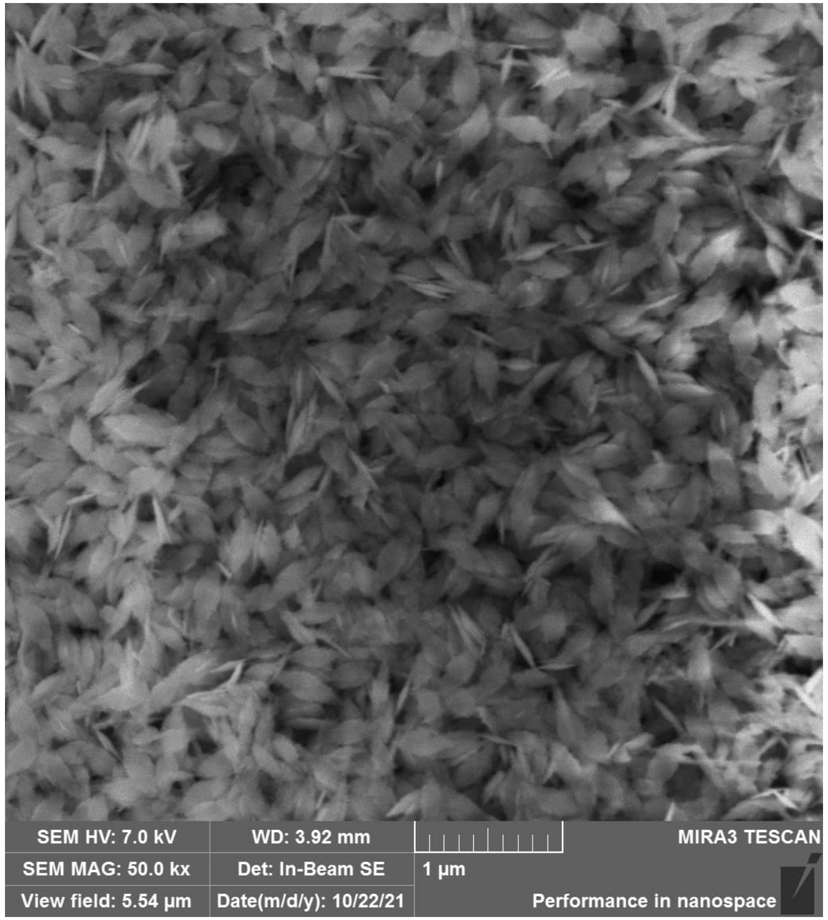
[1]



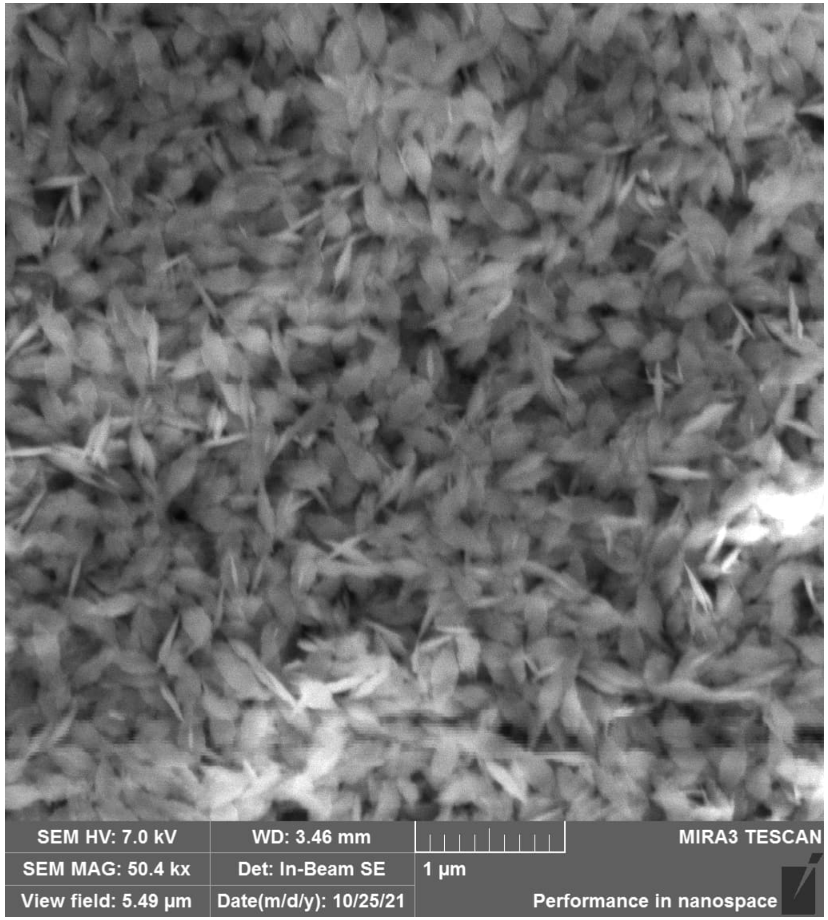
Difficulties:Settings

- Electron gun voltage
  - Nonconductive material is between 1-7 kV,
  - Semiconductive 7-12 kV
  - Metals as much as one wants to.
- Intensity and distance
- Non conductive (electron trap)

[1]



Copper oxide 1 [1]



Copper oxide 2

Data on the SEM image

SEM HV: Voltage used by Electron gun

SEM MAG: Current Magnification

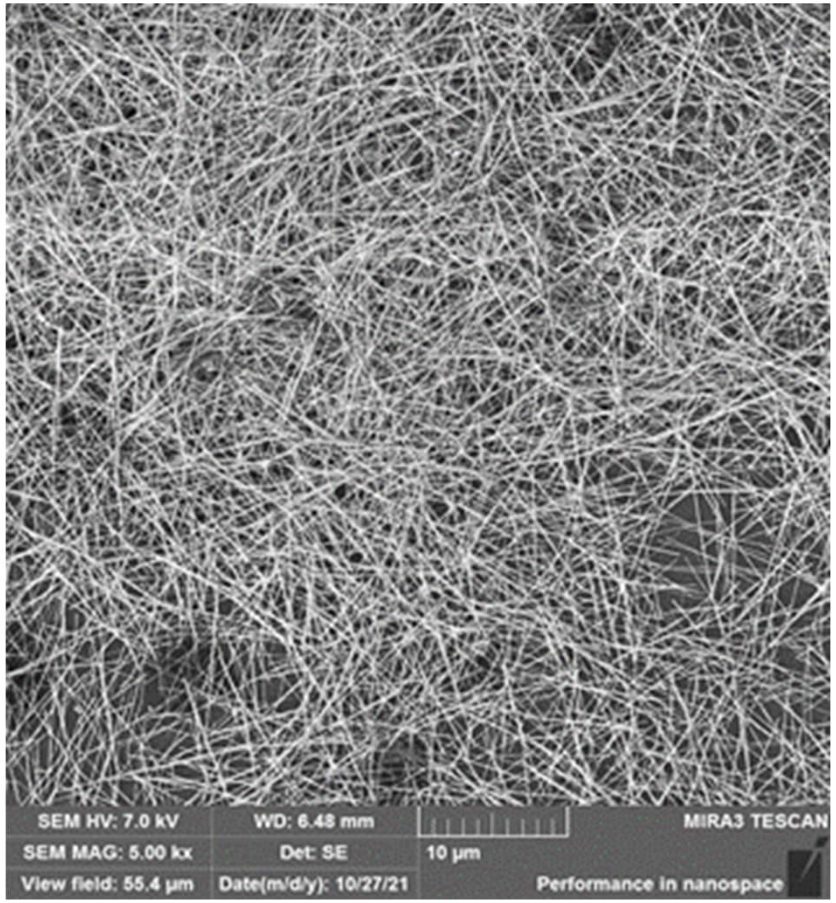
WD: Working distance, Distance between sample and electron gun

Det: Detector type. Either SE, BSE or In-beam SE/BSE

View field: Scale of the Sem picture shown. Length of the side of the image

Date (m/d/y): Date of the experiment

[1]



Advantages and disadvantages

Advantages

- Creates highly accurate 3D feed! (SE)
- Precision 10-1 micrometers!
- Elemental distinction (BSE)
- Elemental analysis also possible (X-Rays, EDS)
- Sample is not harmed*

Disadvantages

- Too much intensity can harm the sample
- Samples of poor electric conductivity require covering (electric tap)
- Biological samples require dehydration
- limited information under the surface

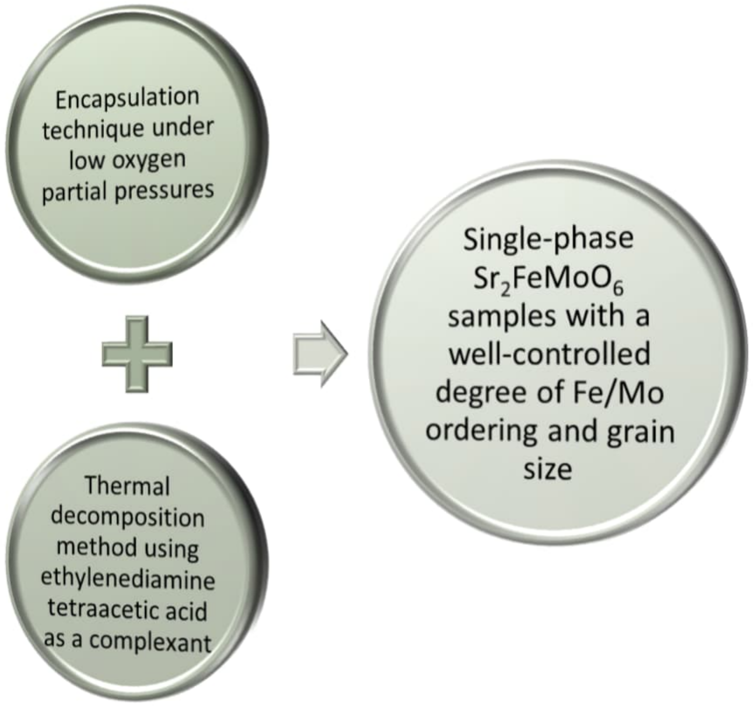
Research Examples

Simple and Efficient Route to Prepare Homogeneous Samples of Sr_2FeMoO_6 with a High Degree of Fe/Mo Order

Y. H. Huang, J. Lindén, H. Yamauchi, and M. Karppinen

View Author Information

Cite this: *Chem. Mater.* 2004, 16, 22, 4337–4342
Publication Date: September 24, 2004
https://doi.org/10.1021/cm0493288



Characteristic methods used in the study:

- ❑ XRD: Crystal structure and composition → confirm the formation of the Sr_2FeMoO_6 phase, and analyze the degree of Fe/Mo order.
- ❑ SEM: analyze the morphology → particle size and shape of the samples.
- ❑ EDS: elemental analysis → confirm the presence of strontium, iron, and molybdenum.
- ❑ Vibrating sample magnetometry (VSM): magnetic properties → confirm the ferromagnetic properties.

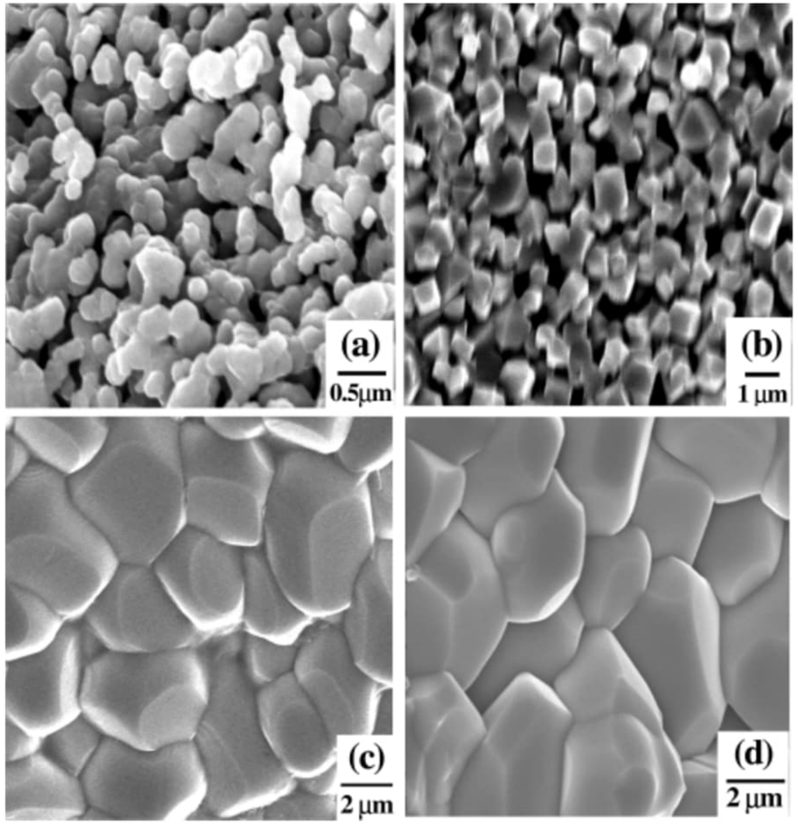
SEM images for Sr_2FeMoO_6 samples obtained at different sintering temperatures.

- T = 900°C (24h) - round grains (~ 0.1 μm), aggregation.
- T = 1000°C (50 h) – cubic shape (~0.5 μm).
- T = 1150°C - polyhedral grains (~ 3 μm).

→ morphology looks highly homogeneous

→ shape of the grains depends mainly on Ts

→ the sintering of the grains/crystallization becomes enhanced as Ts increases.



SEM images for Sr_2FeMoO_6 samples under different conditions: (a) 900 °C for 24 h, (b) 1000 °C for 50 h, (c) 1150 °C for 50 h, and (d) 1150 °C for 100 h. [5]

RESEARCH ARTICLE | FEBRUARY 10 2005

Large low-field magnetoresistance effect in Sr_2FeMoO_6 homocomposites

Y. H. Huang; J. Lindén; H. Yamauchi; ... et. al

Check for updates

Appl. Phys. Lett. 86, 072510 (2005)
https://doi.org/10.1063/1.1864241

homo-composites exhibited a large low-field magnetoresistance effect.

Characteristic methods:

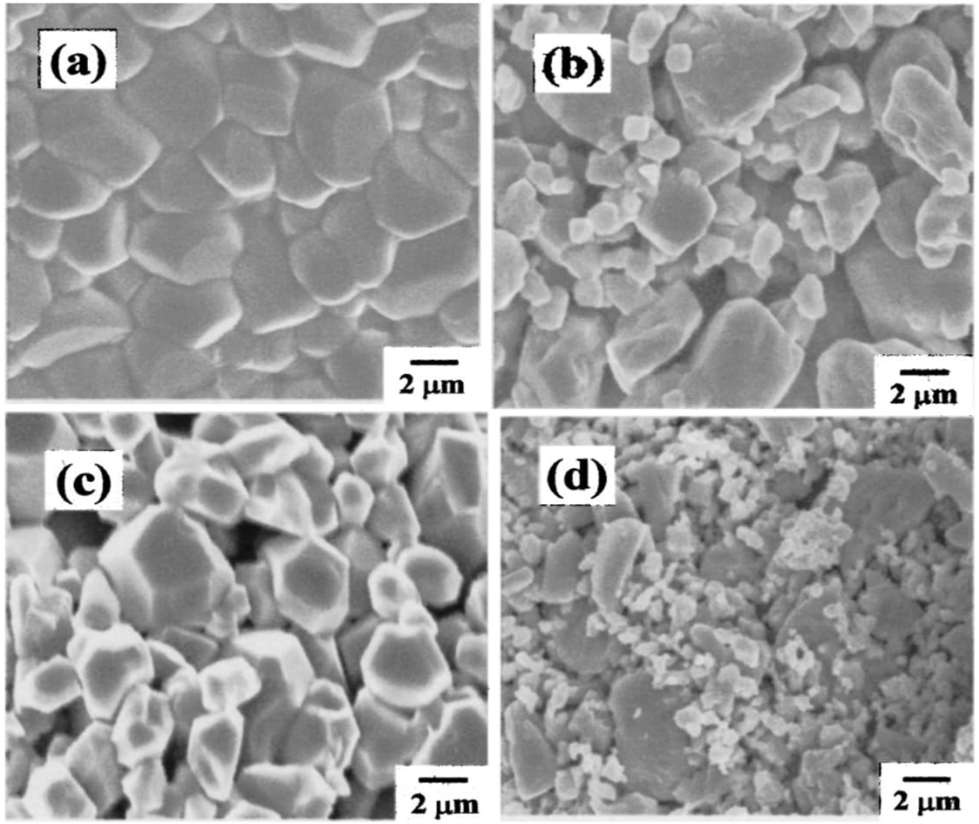
- ❑ XRD → crystal structure and phase purity.
- ❑ SEM → morphology and microstructure of the samples.
- ❑ EDS → elemental composition
- ❑ Magnetization measurements → magnetic properties.
- ❑ Electrical transport measurements → magnetoresistance effect.

SEM images for Sr_2FeMoO_6 homo-composites

- Homogeneous morphology with two different grain sizes.
- The larger grains ~ 3.8 μm, while the smaller grains ~ 1μm.
- Dense and uniform microstructure.

→ high magnetic and transport properties.

LFMR strongly depends on the relative amounts of the two components and their grain sizes.



SEM images for typical Sr_2FeMoO_6 homo-composites: a) *x* = 0, b) *x*=0.2, *Ts*=950 °C, c) *x*=0.2, *Ts*=1100 °C, d) *x*=0.5, *Ts*=950 °C. [6]

CHEMISTRY OF MATERIALS
Article
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Atomic Layer Deposition of Lithium Phosphorus Oxynitride

Mikko Nisula,[†] Yohei Shindo,[‡] Hideyuki Koga,[‡] and Maarit Karppinen*,[†]

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[‡]Higashifuji Technical Center, Toyota Motor Co., 1200 Mishuku, Susono, Japan

- ❑ Development of ALD process for producing high-quality LiPON thin films with a high N-to-P ratio → high ionic conductivity → coatings in 3D Li-ion micro-batteries.
- ❑ Use a novel N-containing P precursor + a lithium precursor → simultaneous incorporation of P and N in the films.

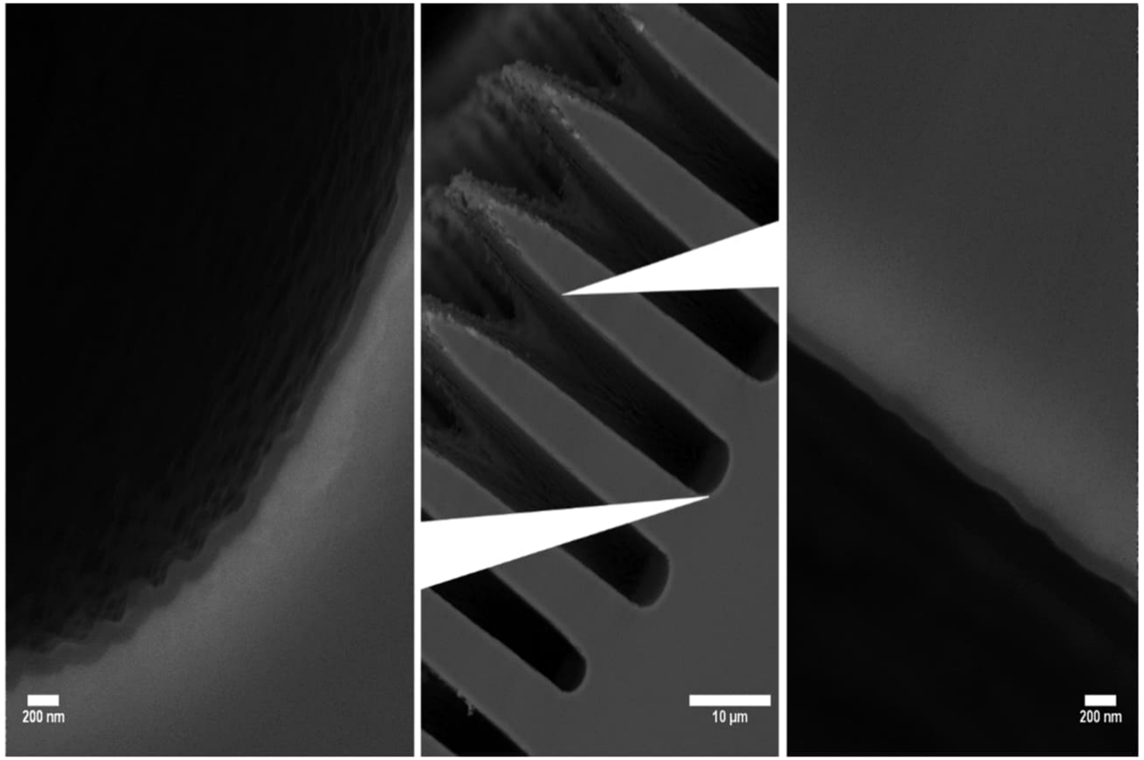
Characteristic methods :

- ❑ XPS → chemical composition of the thin films.
- ❑ Rutherford backscattering spectroscopy (RBS) and nuclear reaction analysis (NRA) → elemental composition/thickness of the thin films.
- ❑ FTIR → chemical bonding in the thin films.
- ❑ SEM → morphology and thickness of the thin films.
- ❑ Electrochemical impedance spectroscopy (EIS) → ionic conductivity of the thin films.

Cross-section SEM images of LiPON deposited of 3D-microstructured silicon.

The conformality of the LiPON ALD process was tested on a micron scale trench pattern ~40 μm deep and ~5 μm wide:

- Thin film is very uniform in thickness at the top and the bottom of the trench.
- Nanoscale roughness of the substrate is perfectly replicated.
- Excellent thickness obtained ~80 nm



SEM images of LiPON deposited of 3D-microstructured silicon. [7]

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Thank you