Material research and analyses

development, testing and characterization of materials for the nuclear energy industry

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Workshop Image Analysis in Nuclear Research, 9.-11. 9. 2024, ÚTIA AV ČR, Prague, Czech Republic

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New materials development

Materials play a key role in the development of mankind

Technical progress comes with the discovery and use of new materials

Prospective materials with excellent properties need to be tested and verified for their functionality in the given application.

Experiments in the Environment

Ageing experiments – accelerated ageing of materials and the study of property degradation

Material stress, thermal loading, irradiation, and corrosion experiments

Verification of the suitability of new manufacturing methods – 3D printing, PVD coating, plasma sintering…

Stone Age Stone tools and pottery	Iron Age Fe-C alloys	Industrial revolution steels	Jet engines Ni superalloys	Information technology Silicon	?Graphene? ?Perovskite? ?Accident tolerant fuels (ATFs)?

Key materials in the nuclear industry

Fuel for fission: Uranium (Natural/Enriched), Plutonuim, Thorium, Ceramics (Fuel pellets)

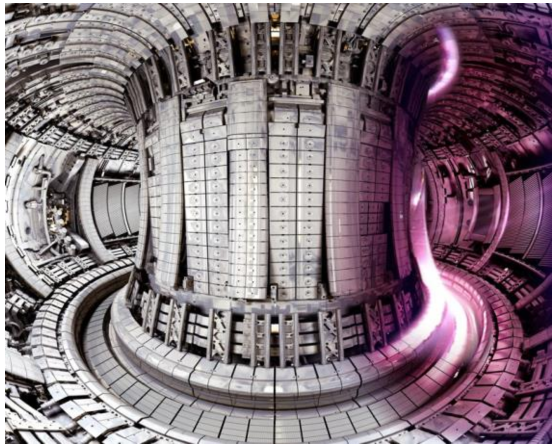
Reactor core: Zirconium (Zircalloy), Graphite, Heavy Water, Beryllium, Cadmium, Boron, Hafnium

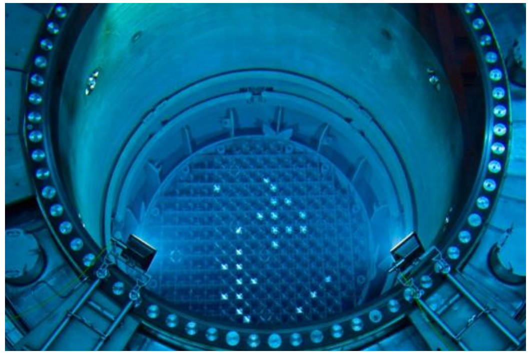
Fusion: Tungsten, Tritium

Structural components: Stainless Steel, Lead, Concrete, Nickel alloys

Coolants: Water, Liquid Metals, Gas (He, CO2)

All nuclear materials undergone a long period of development, testing and qualification and their properties are continuously improved to enhance safe and reliable operation.





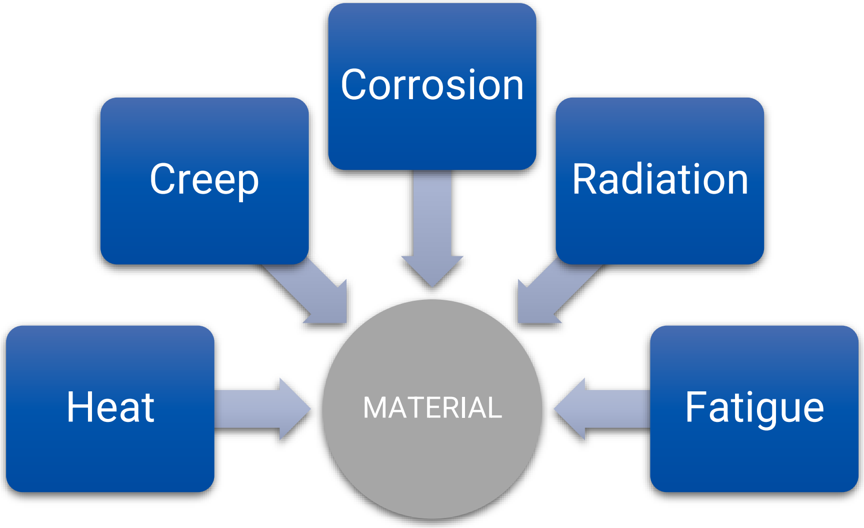
Challenges for materials in operation

nuclear engineering puts high requirements on quality and reliability of all materials due to the extreme conditions inside a reactor core

components are monitored within the framework of surveillance programmes for **degradation assessments** of the NPP’s construction materials

Construction material requirements:

- High creep resistance at operating temperature
- High corrosion resistance
- Advanced fatigue properties
- High ductility and thermal stability
- Resistance to radiation damage
- Longevity over the lifetime of the reactor



Degradation of structural materials during long-term operation

Radiation effects – neutron exposure

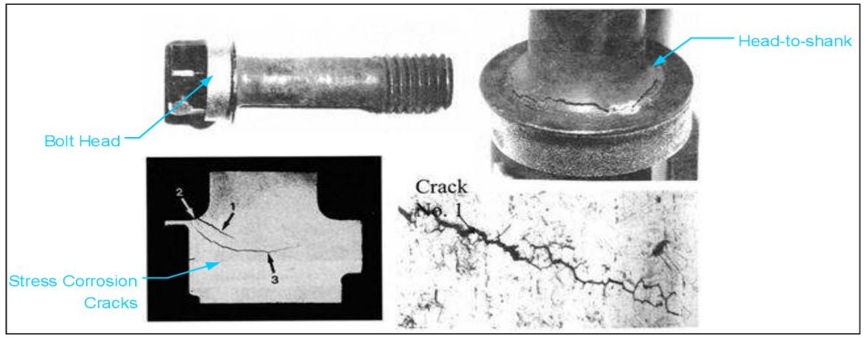
- Microstructural changes
- Radiation hardening and loss of ductility
- Radiation-induced seggregation/solute clustering
- Swelling/Radiation growth/RIVE of aggregates
- Creep

Fatique - Crack initiation and growth

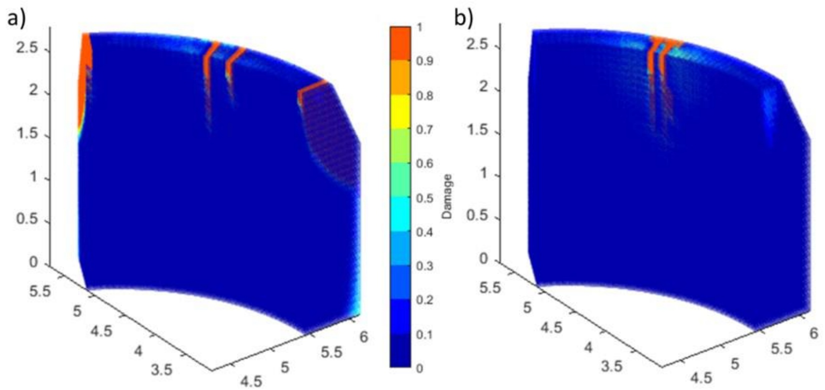
Environmental impacts

- Environmetally assisted cracking - EAC
- Stress-corrosion cracking - SCC
- Iradiation-assisted stress-corrosion cracking - IASCC

Fuel rod damage



IASCC of RPV internals – baffle bolts



Modelling of long-term biological shield damage

Degradation is affecting the component performance and may lead to failure!!

How to prevent component failure?

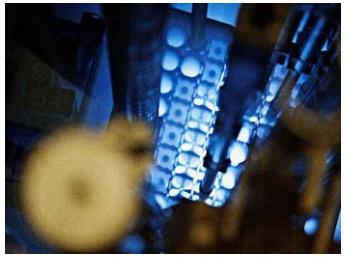
Monitoring and Diagnostics– preventive maintenantce, fuel rod inspections, NDT components check

Testing – surveillance programmes, post-failure analysis and improvements

Experiments – controlled ageing experiments helping assess the margins in operating conditions

Modeling - component lifetime prediction and damage simulation

Irradiation facilities



Research Reactor LVR-15

- **material research, the irradiation of candidate structural materials**
- corrosion testing of materials of reactor primary circuit and internals carried out in experimental loops
- **qualification of components** in radiation fields (neutrons/gamma)
- basic research of material properties
- manufacture of semiconductors by neutron transmutation doping of silicon for the electrical industry
- **production and development of radiopharmaceuticals**, Tc generators

Experimental Reactor LR-0

- the light water zero power reactor
- flexible reactor with flexible core arrangements
- for **determination of neutron-physical characteristics** of various types of reactor lattices, kinetics experiments
- reactor chambers and other I&C equipment testing
- **experiments with various insertion zone types** (graphite, fluorine salts)
- experimental verification of criticality and subcriticality in relation to zone parameters
- verification of codes

Gama irradiation facility

- ^{60}Co emitter
- 180 TBq activity
- irradiation at elevated temperatures,

  cryogenic conditions
- vacuum or inert gas atmosphere
- short and long-term irradiation experiments
- **qualification of components**
- **materials ageing experiments**
- materials for space applications

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DAY 3: Excursion to CVR facility (11th Sept 2024)

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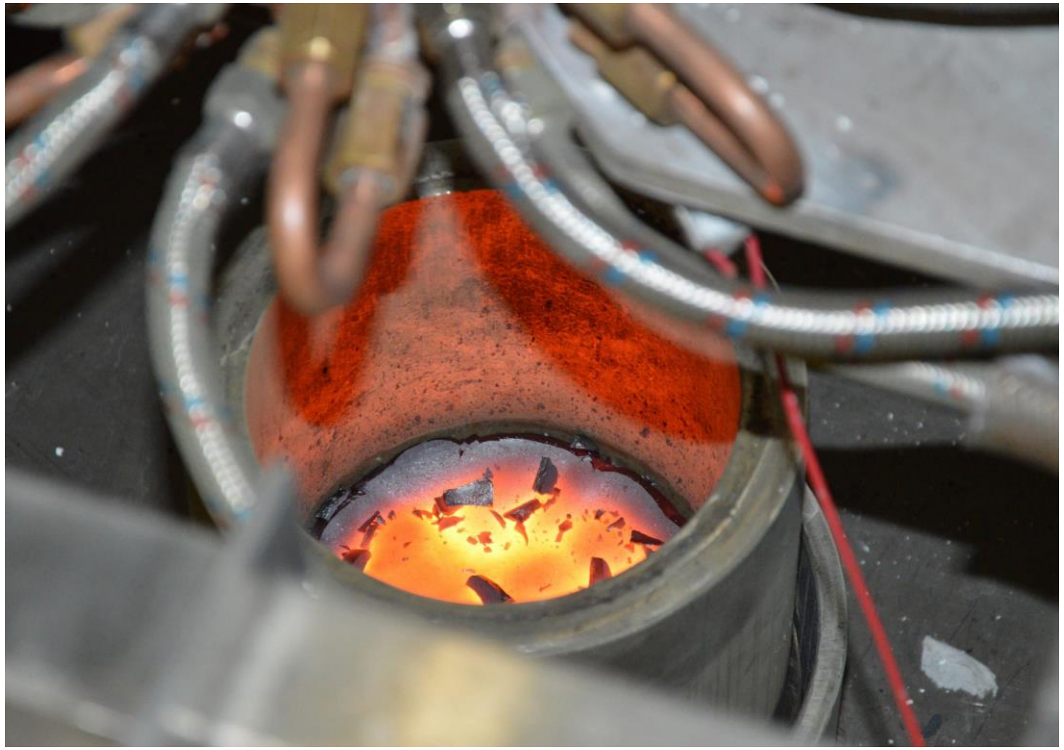
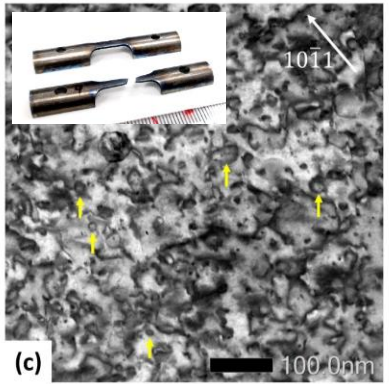
Unique infrastructures for material tests

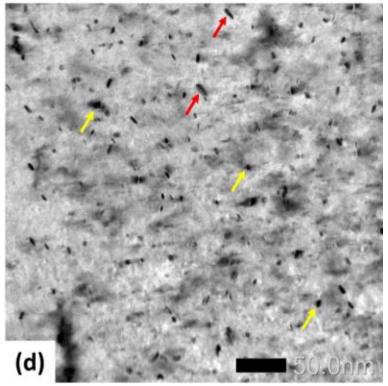
- **Experimental loops** – LWR, SCWR, HTHL, sCO2, metal liquid Pb, PbBi, PbLi, FLiBe loop
- **Hot-cells** – handling of radioactive materials
- Analytical laboratories
- **LOCA** (Loss of Coolant Accident)
- **Cold Crucible** – studies of corium behaviour during severe accidents
- **HELCZA** (High Energy Load Czech Assembly) – ITER primary wall components testing

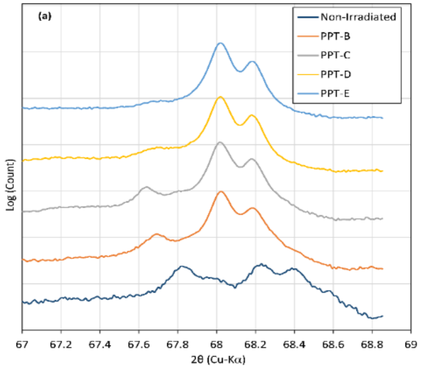


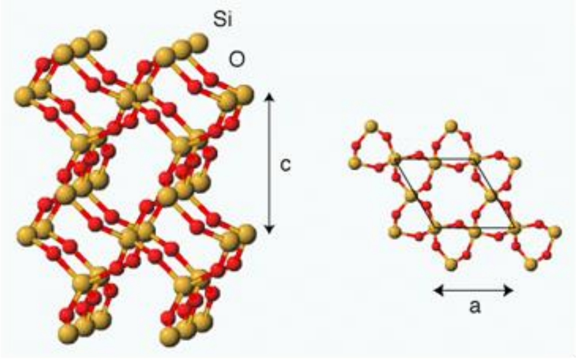
From irradiation to PIE: Accelerated material ageing studies

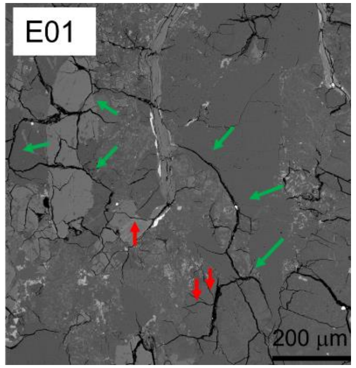
- Irradiation experiments in reasearch reactor LVR-15
- Post-irradiation examination (PIE)
- Degradation of structural components
- Determination of physical and structural changes in materials due to irradiation and simulated environment
- Mechanical testing
- Microstructural characterization
- Quantification of defects











J. Procházka, J. Knotek: Precipitates and cracks analysis

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Importance of the materials microstructure characterization

Understanding Material Properties: The microstructure of a material can strongly influence physical properties such as strength, toughness, ductility, hardness, corrosion resistance, high/low temperature behavior, or wear resistence. *Understanding the microstructure is key to understanding materials behavior in operating conditions.*

Material Design: understanding the role of a structure in a process-structure-property relation. This understanding can help in the design of new materials with desired properties.

Quality and Performance Control: characterization techniques are used to monitor the quality of materials during processing, manufacturing and operation. This helps ensure that the final product meets the required specifications and performance standards and long-term stability.

Development of New Materials: critical for understanding the behavior and developing new materials with specific properties.

Advanced analytical techniques

Microscopy/Micromechanics

Light optical microscopy (LOM)
Scanning electron microscopy (SEM)
Transmission electron microscopy (TEM)
Scanning probe microscopy (SPM)
Atomic force microscopy (AFM)
Nanoindentation (NI)
X-ray microscopy/tomography

These techniques are used to visualise and examine the materials at microscopic level. Applying the spectroscopic methods and image analysis can give us additional information. Combining with in-situ testing can give valuable information.

Spectroscopy/Spectrometry

Energy dispersive spectrometry (EDS)
Wavelength dispersive spectrometry (WDS)
X-ray fluorescence spectroscopy (XRF)
Auger spectroscopy
Raman spectroscopy
Infrared spectroscopy (FTIR)
Electron energy loss spectroscopy (EELS)
Secondary ion mass spectrometry (SIMS)
Atom probe tomography (APT)
Alpha/gama spectroscopy
3D gama tomography

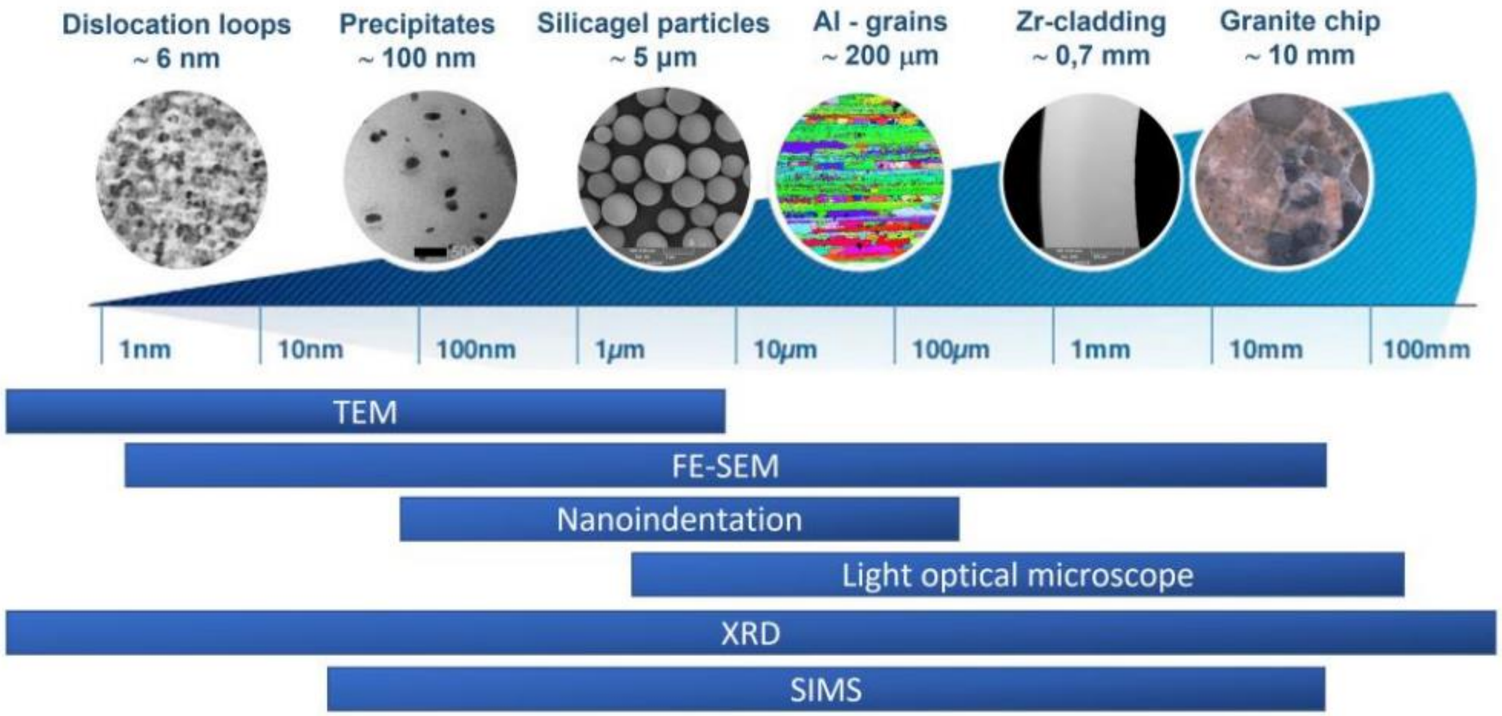
Investigation of the chemical composition, physical structure, and electronic structure of materials at the atomic, molecular, or micro scale.

Diffraction techniques

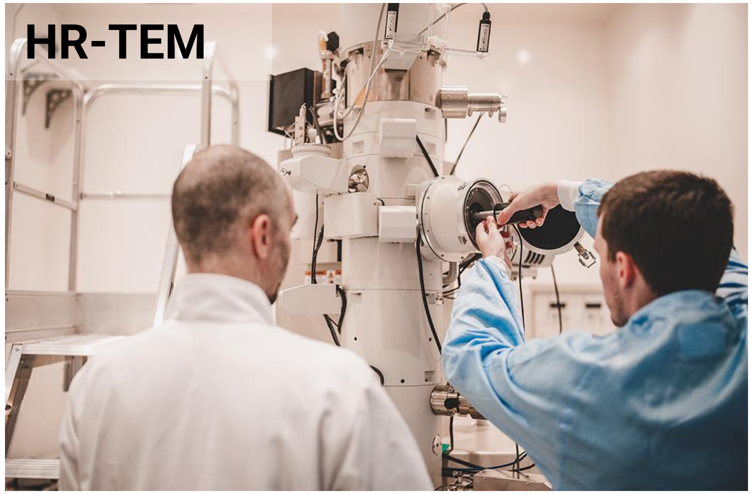
X-ray diffraction (XRD)
Neutron diffraction
Electron backscattered diffraction (EBSD)
Selected-area electron diffraction (SAED)

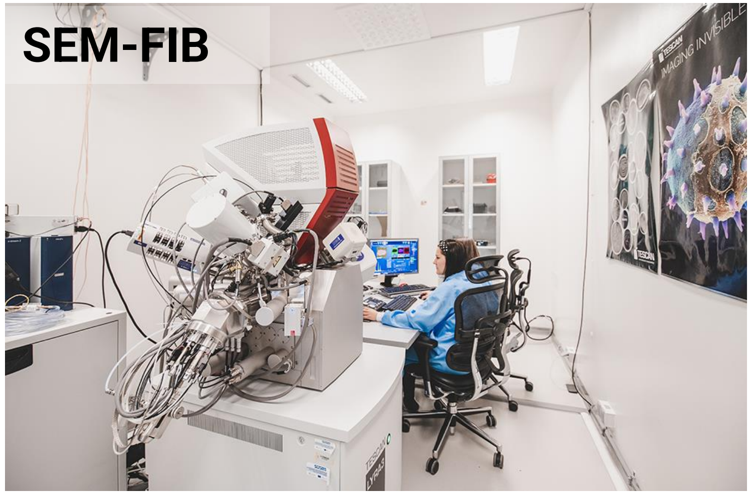
These methods are used to study the crystal structure of materials by observing the pattern produced when incident beam interacts with the periodic structure of the sample.

Complex material characterization on a small scale



Analytical facilities for material research





How can image analysis help us?

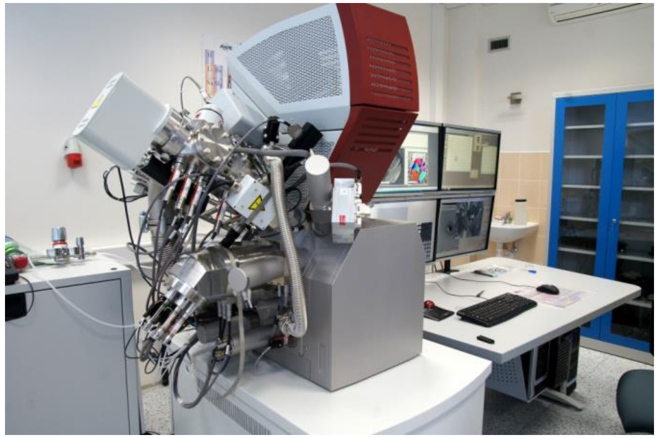
Using imaging and analytical techniques we produce **large datasets**

To effectively handle these image, video and spectral data modern digital image processing techniques are needed:

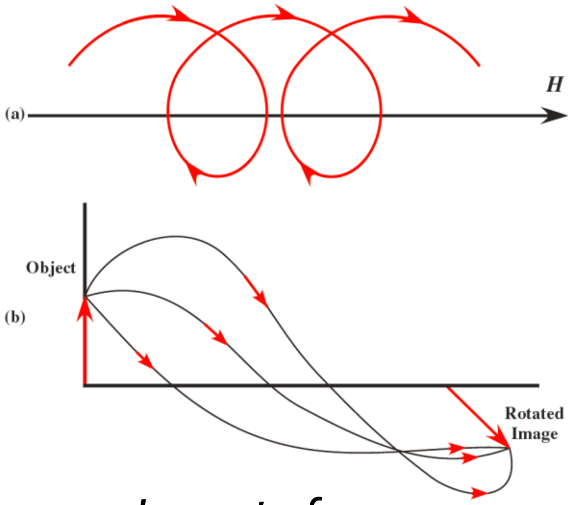
- **automated quantification of features** – enhanced accuracy and efficiency
- **real-time processing** – optimized data aquisition
- **statistical evaluation** and generation of inputs for modeling SW

How can we do images?
(using scanning electron microscopy)

We have SEM Tescan FERA 3



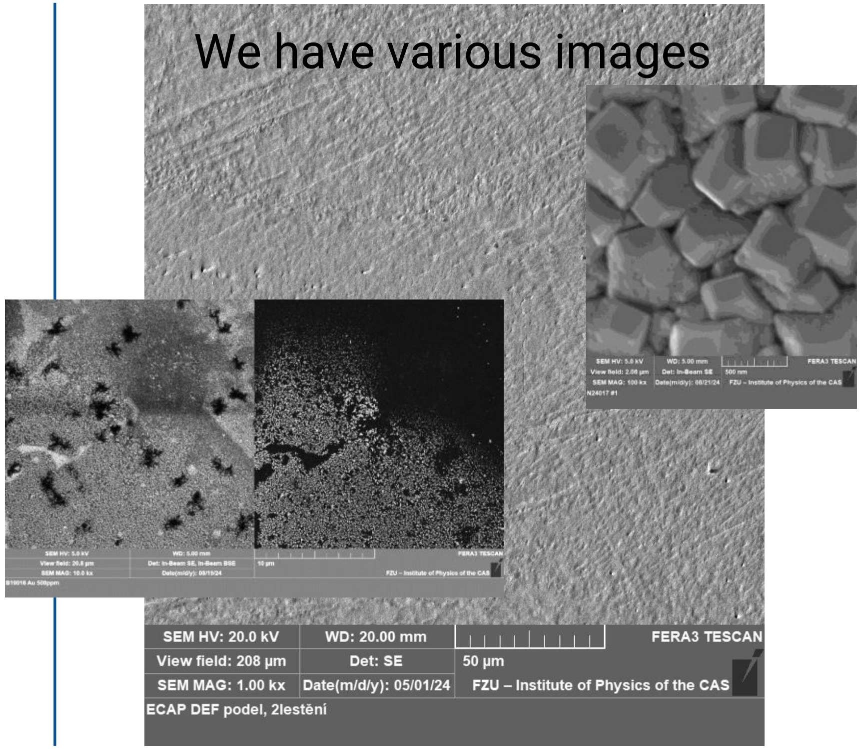
We use electrons



Lorentz force

$$\boldsymbol{F} = -e(\boldsymbol{E} + v \times \boldsymbol{B})$$

We have various images

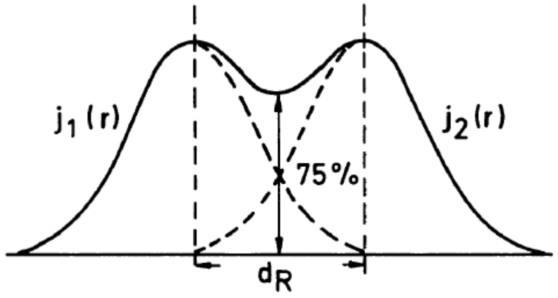


Why we use electron microscopy? (...and not optical microscopy?)

There were found some limitations of optical microscopy

(1873):

$$d = \frac{\lambda}{2n\sin\alpha} \cong \frac{0.6\lambda}{\alpha}$$



Ernst Abbe

Karl Zeiss

When we started with?
Shortly about history

Ernst Ruska

1986 – Nobel price for electron optics

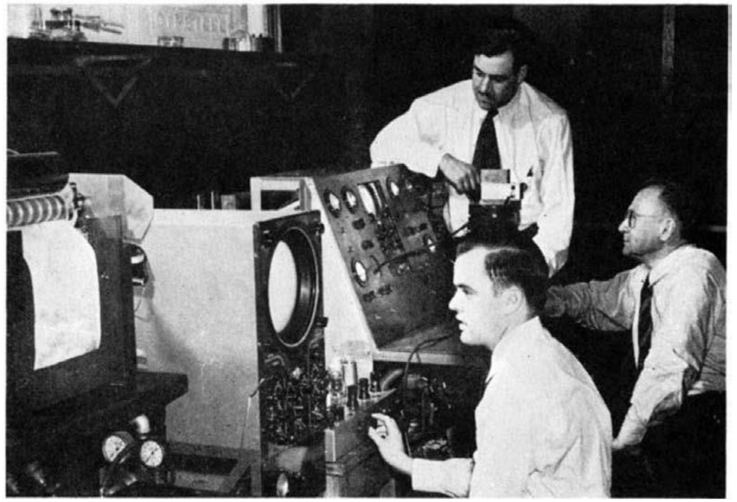
1931 – torroidal coil works as

lens for

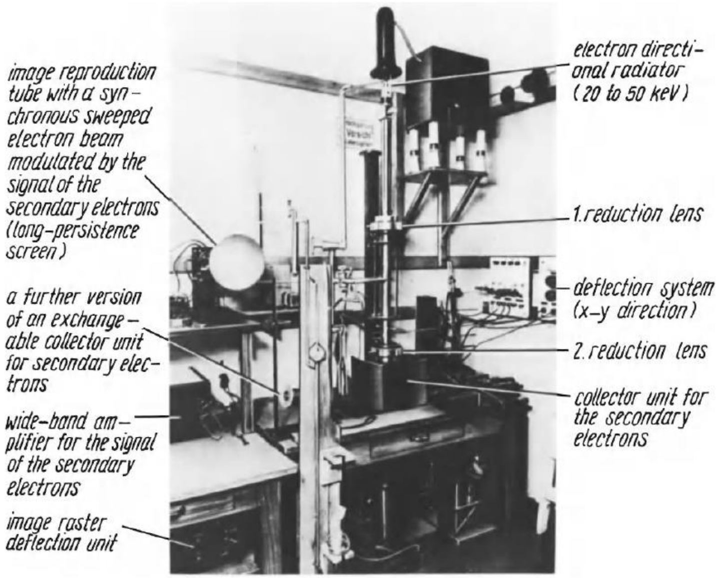
electrons

Max Knoll
Manfred von Ardene

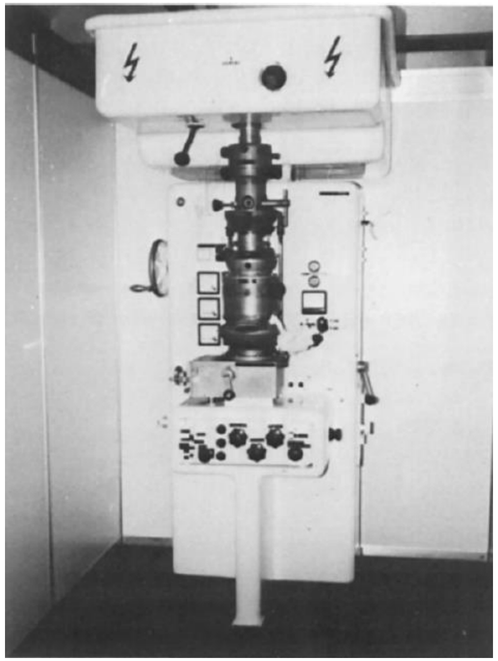
Vladimir Kosma Zworykin



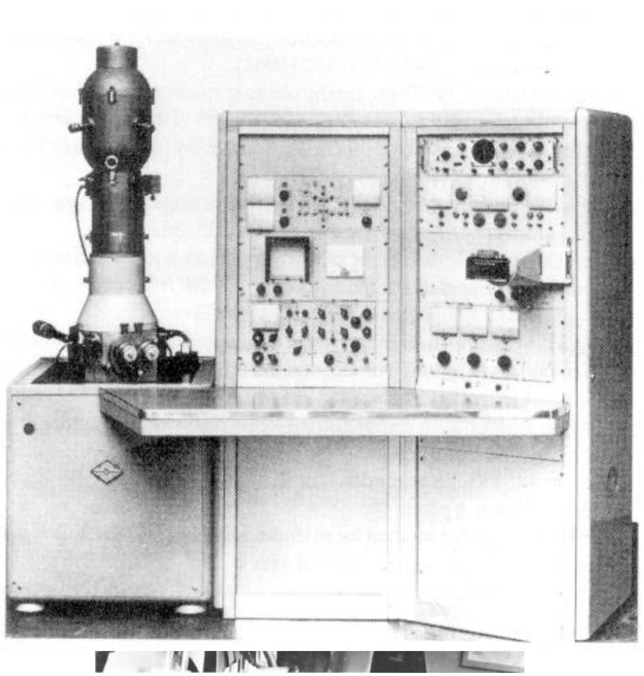
When we started with? Shortly about history



1937, resolution 100 nm



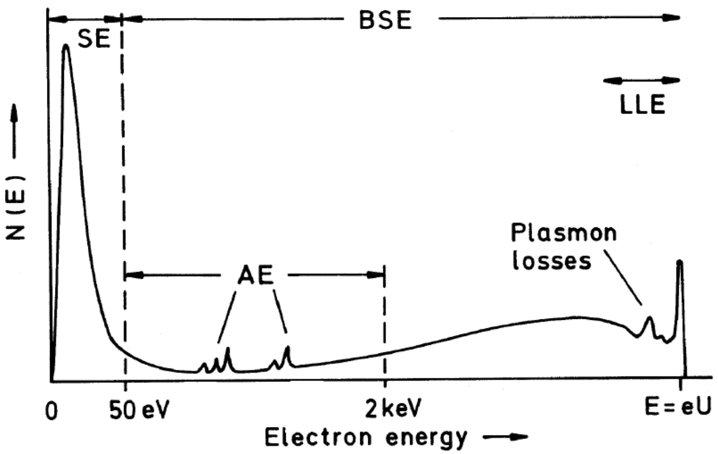
1939, Siemens ÜM 100
The first commercially produced EM (TEM)



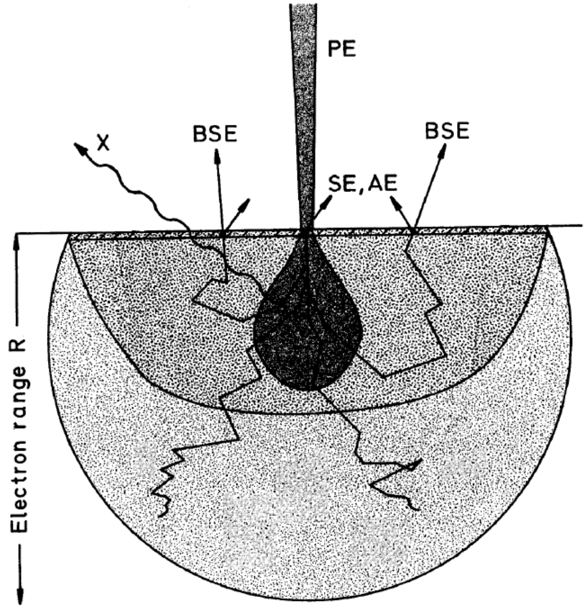
1941, 300 keV TEM
1956, Stereoscan MK1
The first commercially produced SEM

What we get from?

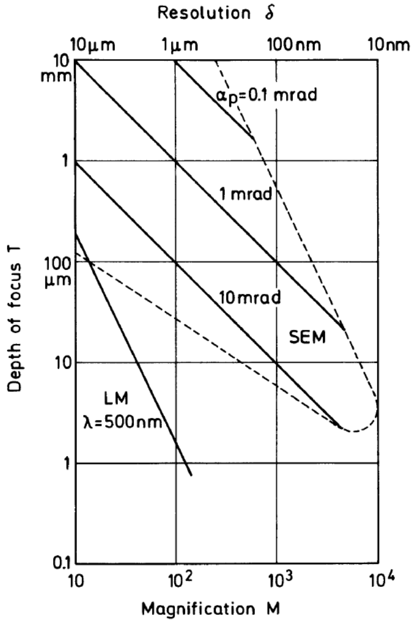
Spectrum of scattered electrons



Interaction volume – different for different types of signal

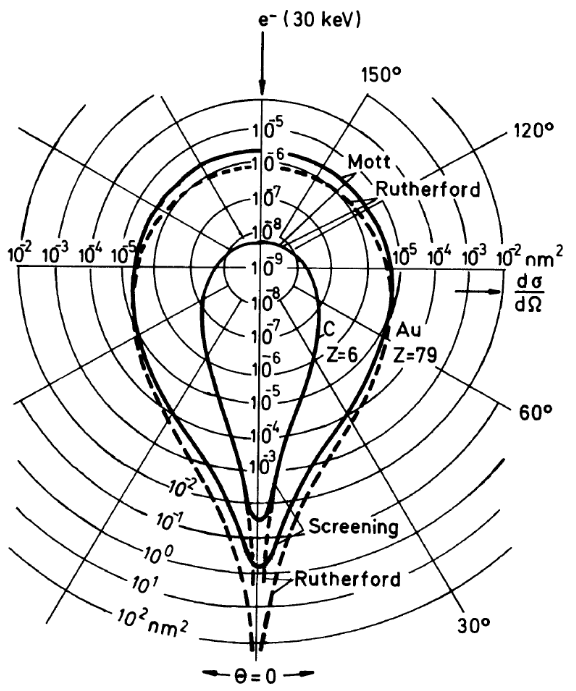


Depth of focus



Scanning Electron Microscopy Physics of Image Formation and Microanalysis, Ludwig Reimer, ISBN: 978-3-642-08372-3 Springer-Verlag Berlin Heidelberg 1985, 1998

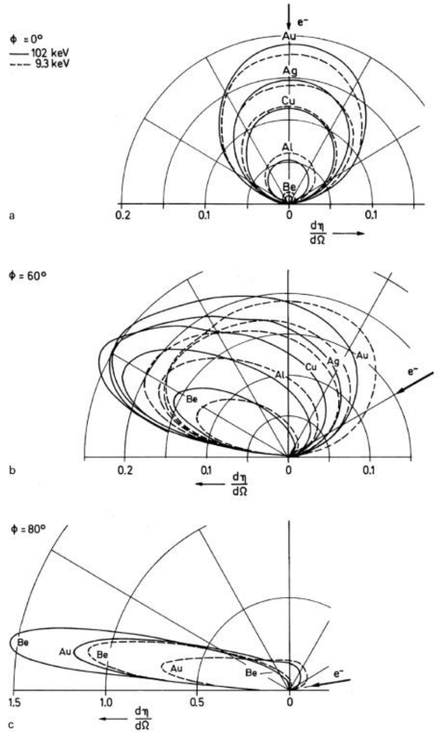
Back scattered electrons



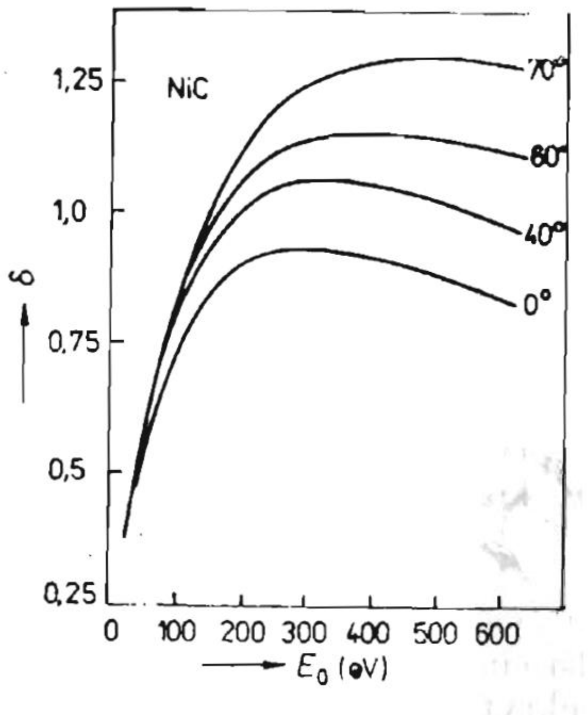
Scattering on cores, i.e. on Coulombic potencial of atomic cores.

Electrons with high kinetic energy

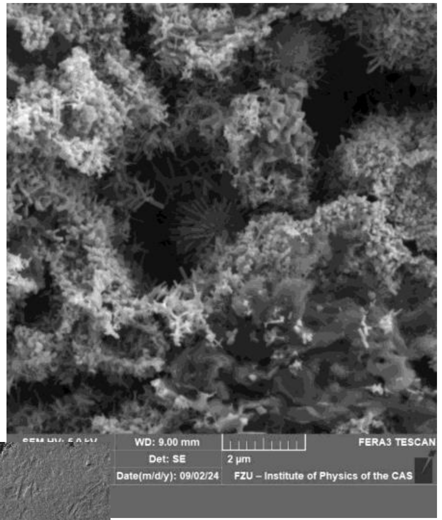
Gives mainly information about composition

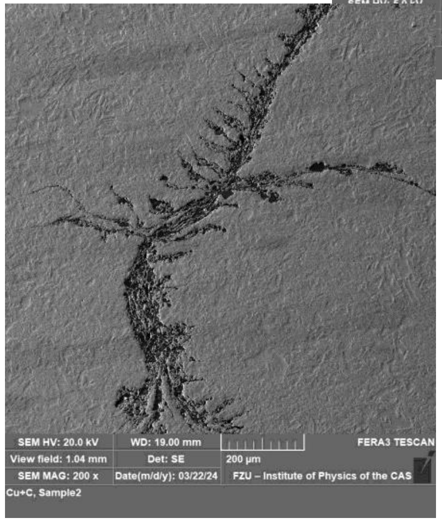


Secondary electrons

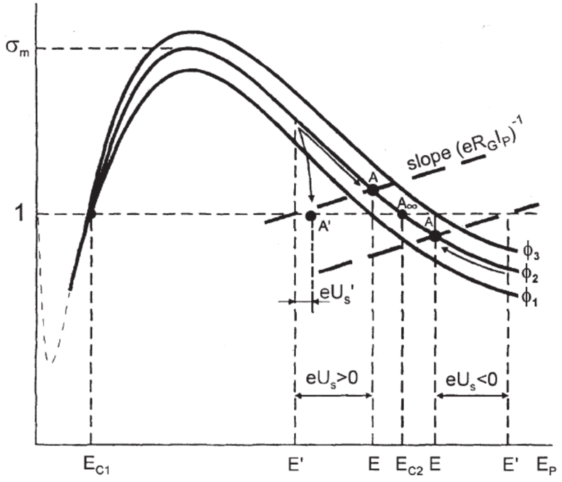


- Scattering on electron shells, i.e. ionization
- Small kinetic energy (idealy up to 50 eV)
- Easy spoil
- Gives information mainly about surface topology

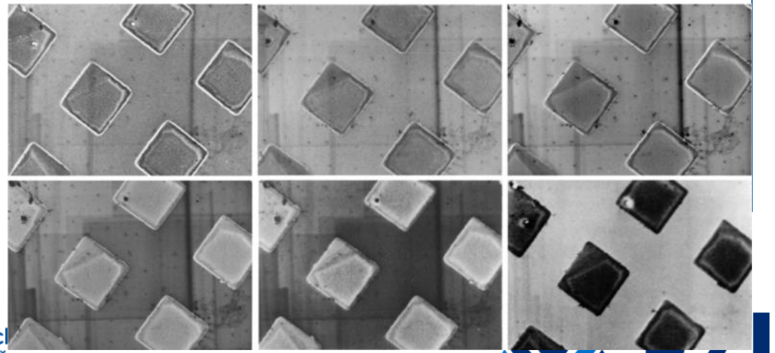




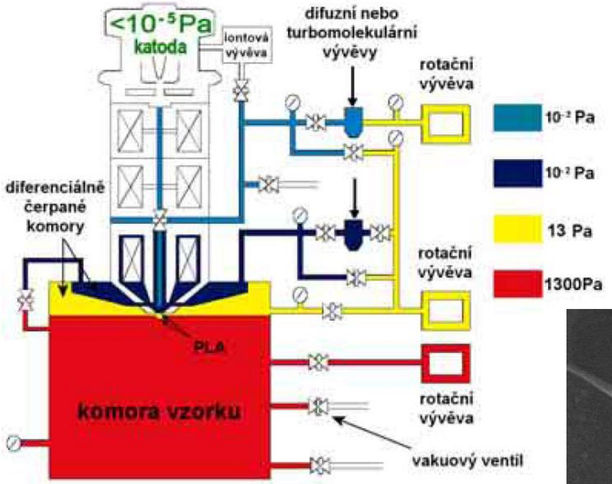
Low-voltage electron microscopy
Low-vacuum electron microscopy

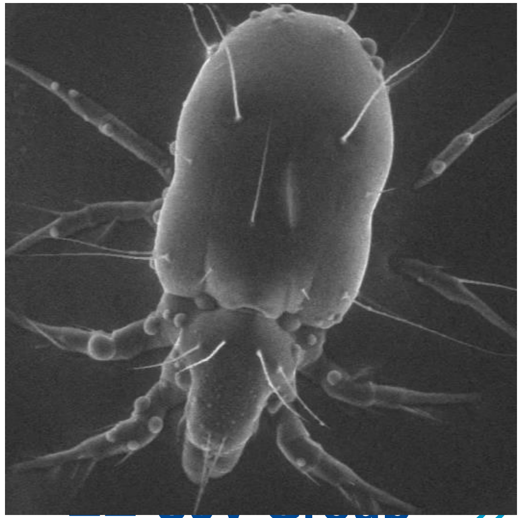


Luděk Frank

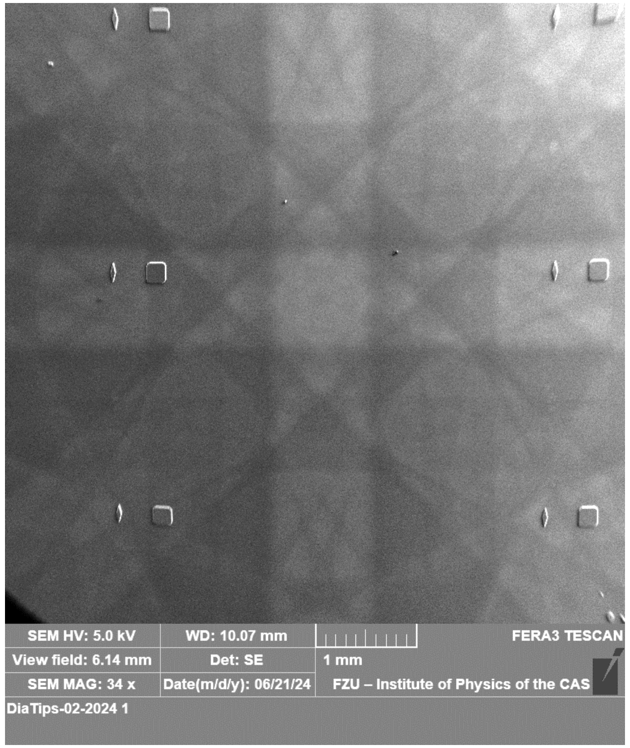


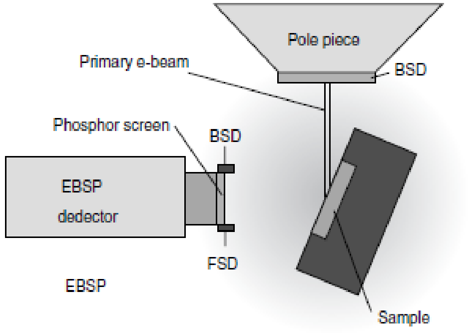
Vilém Neděla

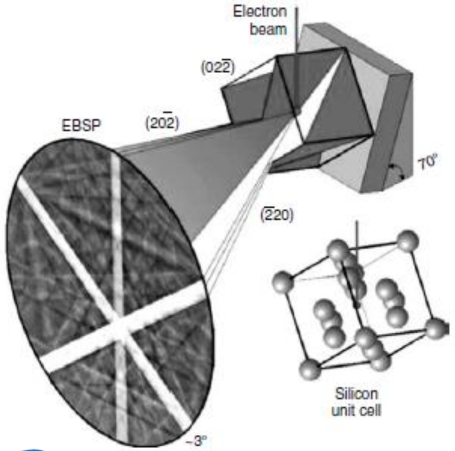


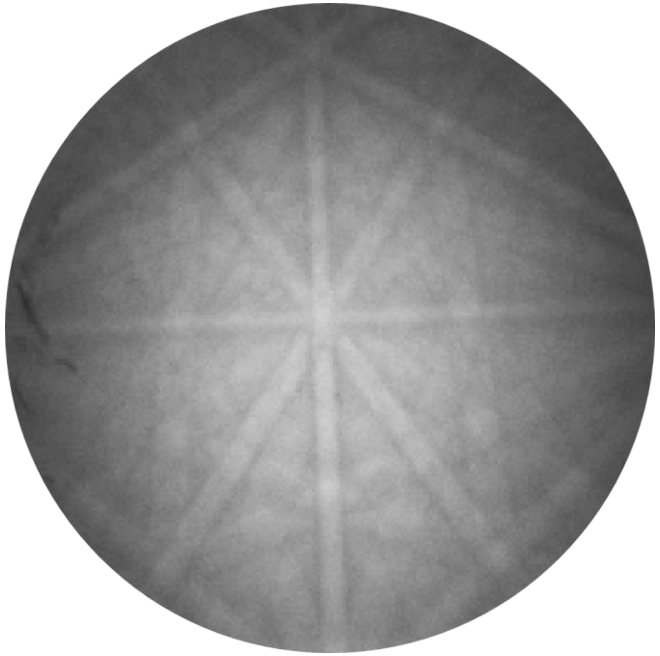


Enhanced (strange) back-scattered electrons – channeling and EBSD



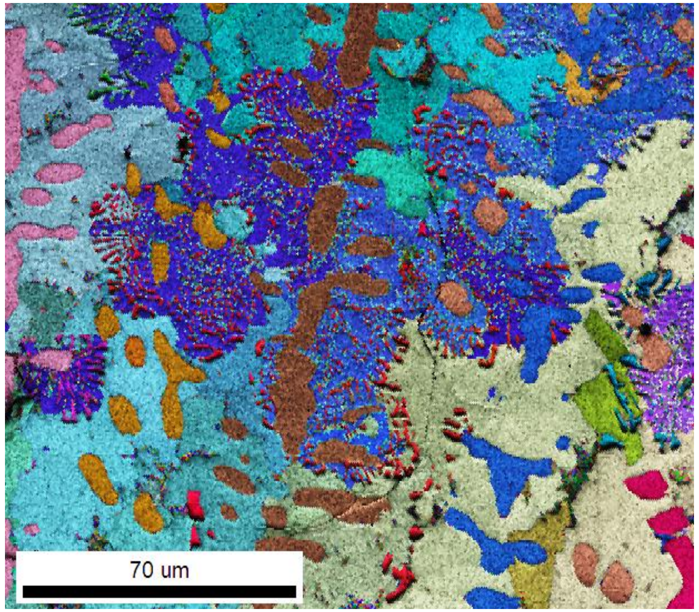


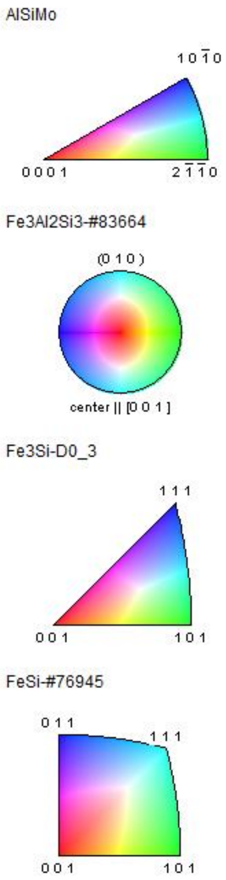


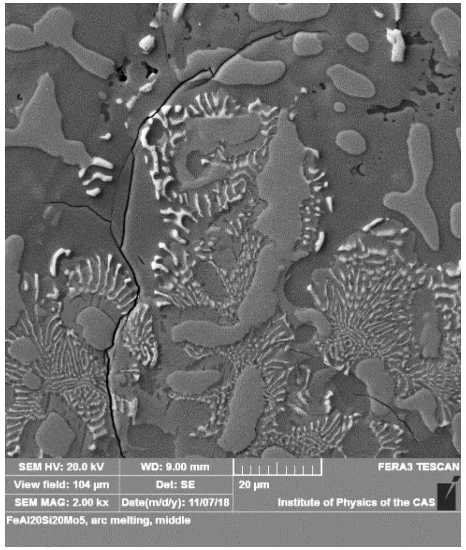


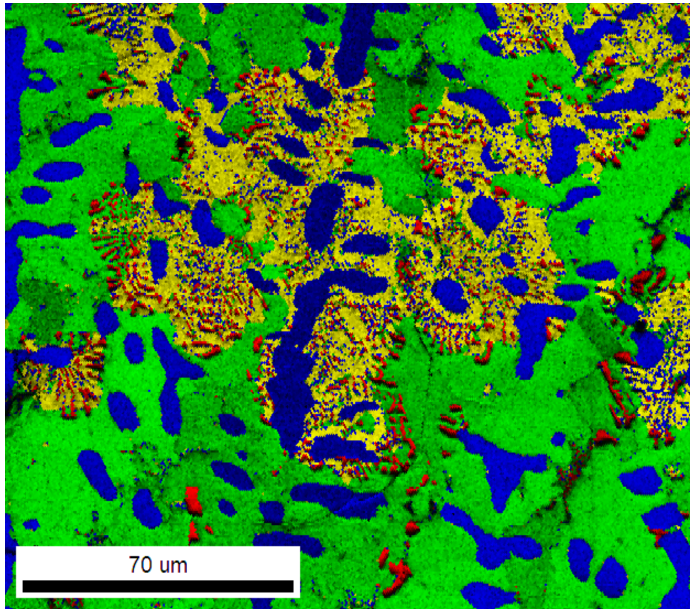
Scanning Microscopy for Nanotechnology Techniques and Applications - Weilie Zhou, Zhong Lin Wang (Eds), Springer Science+Business Media, LLC, 2006

Electron back-scatter diffraction
What it gives?







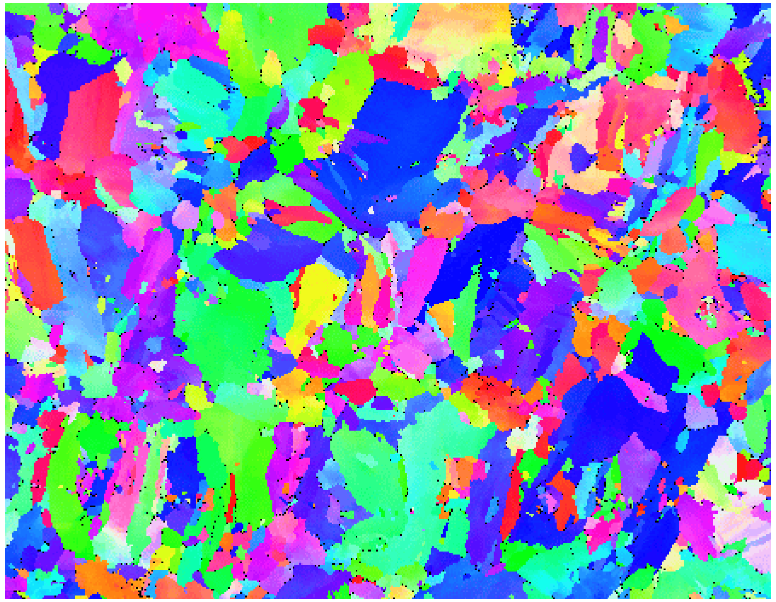


Color Coded Map Type: Phase

	Phase	Total Fraction	Partition Fraction
(red)	AlSiMo	0.055	0.055
(green)	Fe3Al2Si3-#83664	0.532	0.532
(yellow)	Fe3Si-D0_3	0.185	0.184
(blue)	FeSi-#76945	0.229	0.229

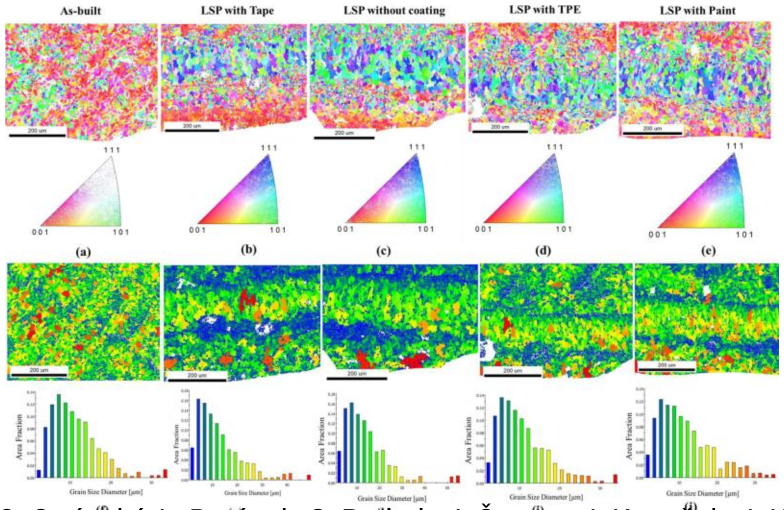
Boundaries: <none>

Electron back-scatter diffraction Applications

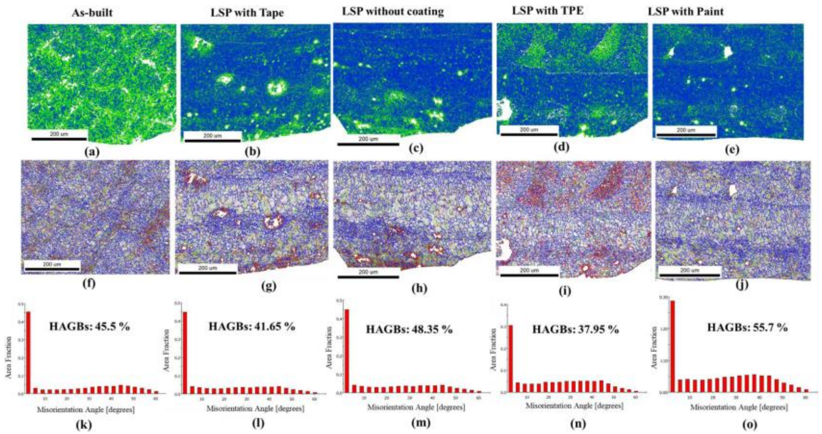


https://www.linkedin.com/posts/jack-donoghue-a613389a_ebsd-3debsd-additivemanufacture-activity-7236684161011363842-RGEb?utm_source=share&utm_medium=member_desktop

Orientation maps
Inverse pole figure, i.e. texture
Grain size spatial distribution
Grain size histogram



Kernal average misorientation
Grain boundaries – position and type
Misorientations histogram



O. Stránský, L. Beránek, S. Pathak, J. Šmaus, J. Kopeček, J. Kaufman, M. Böhm, J. Brajer, T. Mocek, F. Holešovský, *Effects of sacrificial coating material in laser shock peening of L-PBF printed AlSi10Mg*, Virtual and Physical Prototyping, 19:(1), e2340656-1 – e2340656-15, (2024)

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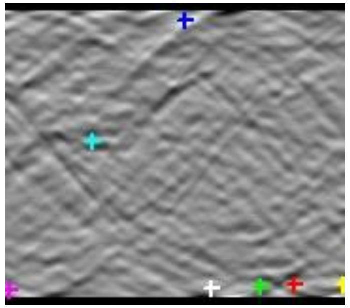
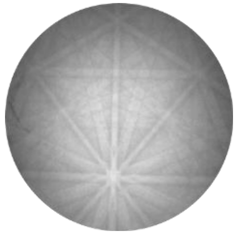
EBSD data processing integral transformations

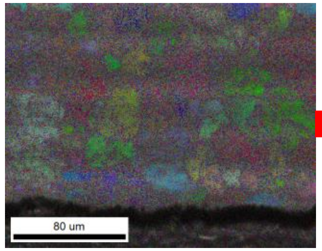
Hough transform - 1962

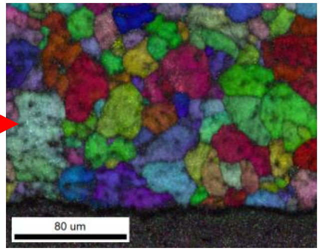




$$r = x \cdot \cos\theta + y \cdot \sin\theta$$

$$R(\rho, \theta) = \int_{-\infty}^{\infty} \int_{-\infty}^{\infty} f(x, y) \cdot \delta(\rho - x \cdot \cos\theta - y \cdot \sin\theta)\, dx\, dy$$



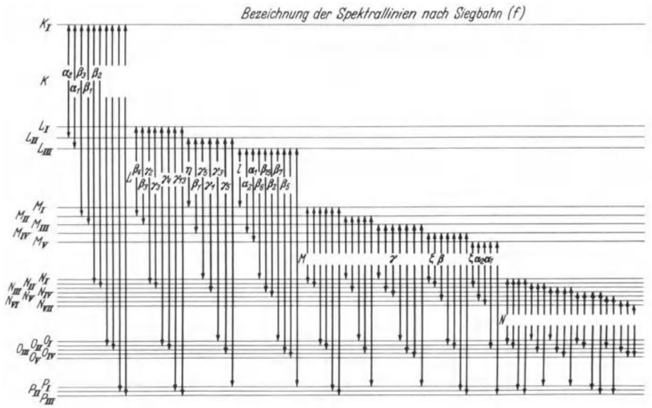


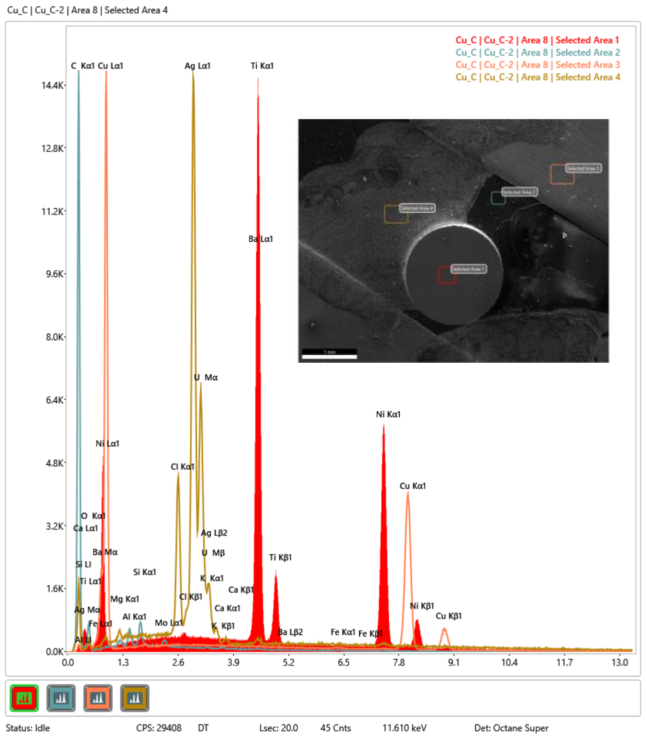
Radon transform - 1917

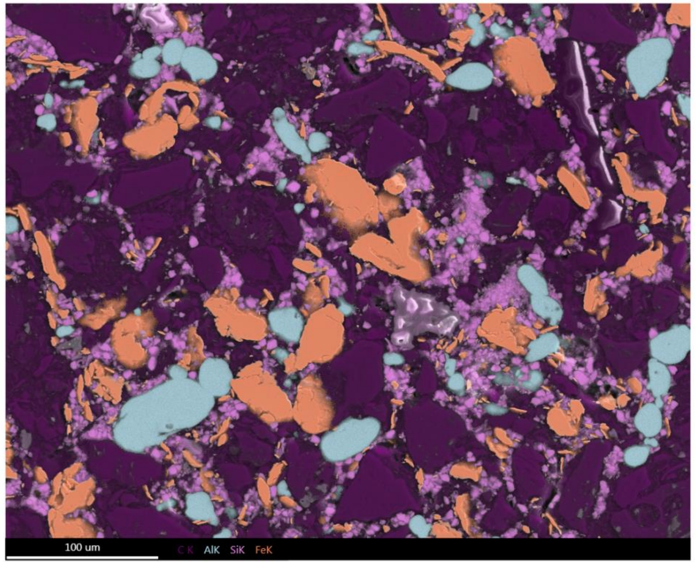
$$(x(t), y(t)) = t(\sin\alpha, -\cos\alpha) + s(\cos\alpha, \sin\alpha)$$

$$\mathcal{R}[f](\alpha, s) = \int_{-\infty}^{\infty} f(x(t), y(t))\, \mathrm{d}t$$

Energy Dispersive Spectroscopy
Looking for elemental composition







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