Material analysis in NTC

New Technologies Research Centre

New Technologies Research Centre ▶ Your partner for research, development and innovation in industrial applications

MAIN FOCUS:

▶ materials for photovoltaics, photonics and microsystems using
  ▶ deposition aparatures PVD, CVD, Pe-CVD
  ▶ electron microscopy
  ▶ X-Ray diffraction
  ▶ optical spectroscopy (atomic and vibration spectroscopy)
  ▶ addition equipment (lithography, profilometer, solar simulator, 4-point probe)
▶ structural analysys and metallography
  ▶ metallographics sample preparation
  ▶ X-Ray diffraction
  ▶ X-Ray spectrometer
  ▶ optical microscope
  ▶ nanoindentation
  ▶ tribometer

APPLICATION:

▶ thin film deposition
  ▶ PVD
  ▶ PE-CVD Si based
▶ material analysis
  ▶ picture analysis
  ▶ hardness measurement and tribological characteristics
  ▶ determining the number and types of phases, structural parameters
  ▶ element composition
  ▶ measuring and determining the optical properties of thin films
  ▶ thickness and roughness measurement of thin films
  ▶ determining the type of cunductivity and resistivity

CENTEM 5

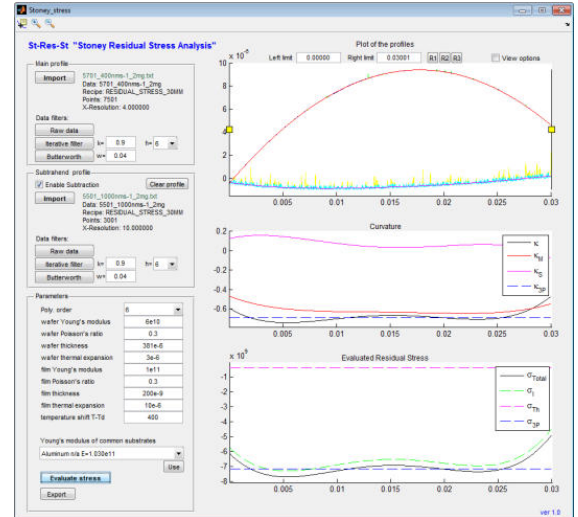## MATERIALS AND TECHNOLOGIES

Surface morphology analysis
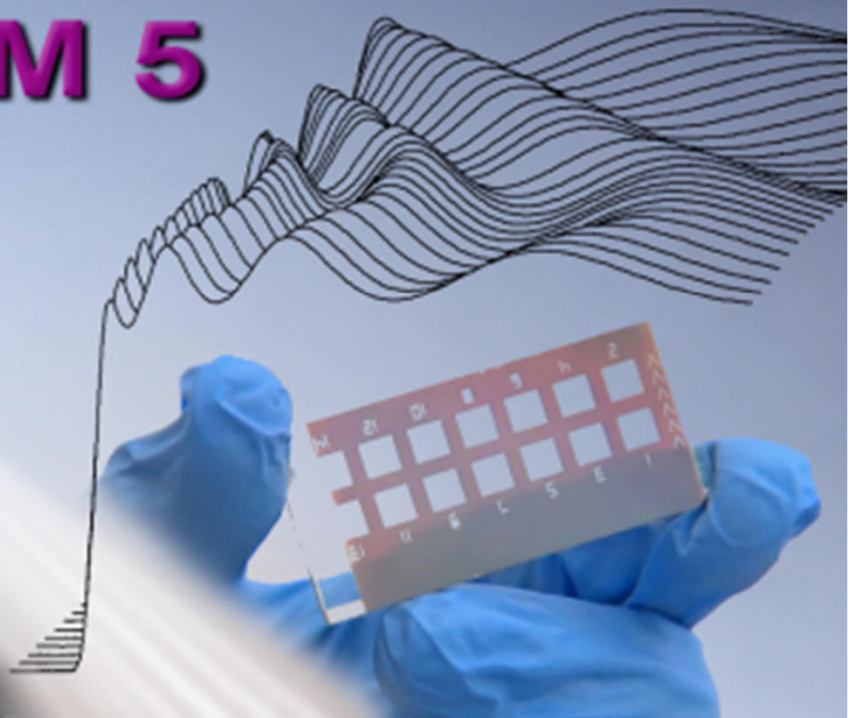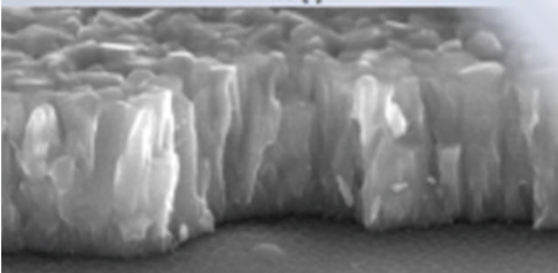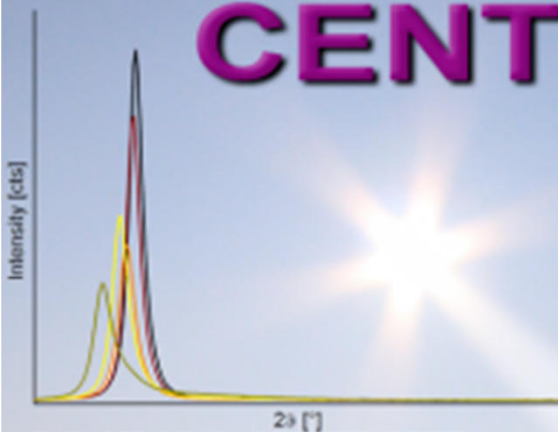
KLA-Tencor P-6 Surface Profiler

Surface analysis system
- vertical range to 1 mm
- scan length 150mm
- applied load 05-50 mg
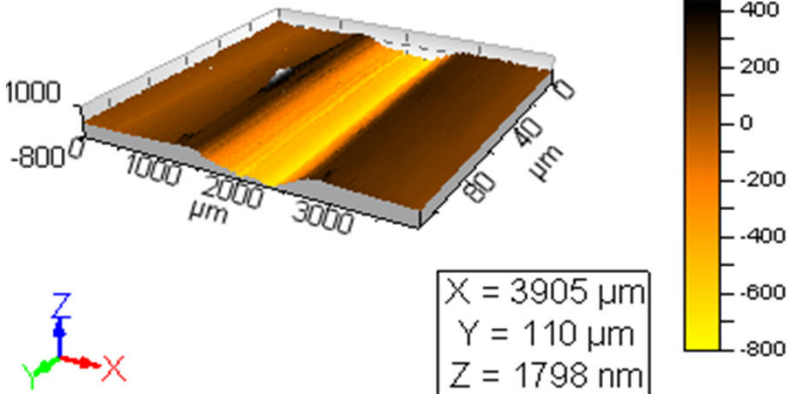- vibration-isolating workstation
- Apex 3D analysis software

2D stress of thin films

Measuring stress of thin films can help optimize processes, prevent cracking and adhesion problems.

3D imaging of various surfaces

Apex software provides advanced 3D imaging of any area measured.
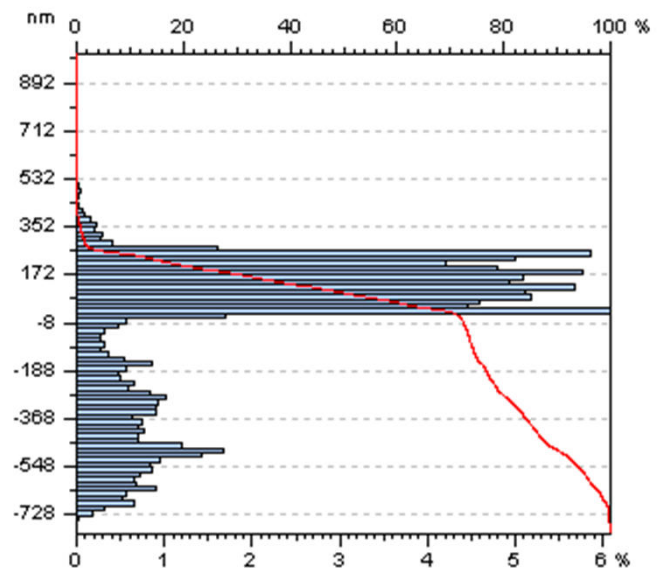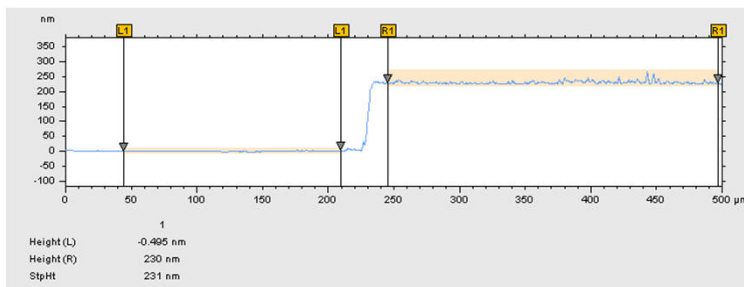

X = 3905 µm
Y = 110 µm
Z = 1798 nm

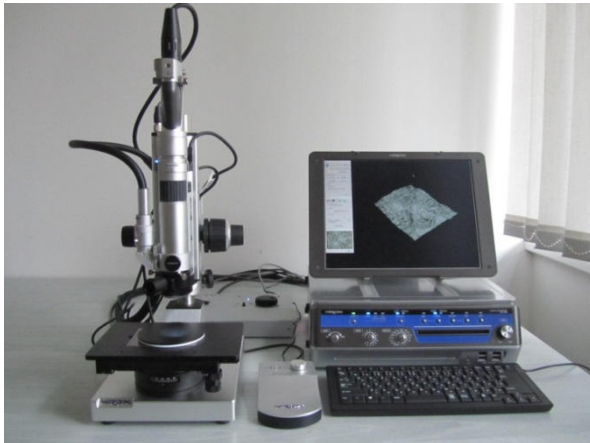## Materials characterization for surface roughness and waviness step heights

Apex analysis software easily calculates more than 40 surface parameters, including surface roughness, waviness, peak count distribution and other parameters. Calculations can be for 2D scans or 3D area scans.
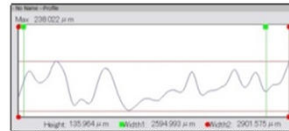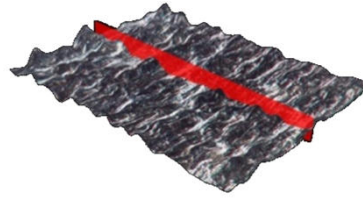
Thin film

Measure step heights and surface roughness after thin film deposition.

Height (L)   -0.495 nm
Height (R)   230 nm
StpHt   231 nm

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Optical microscopy
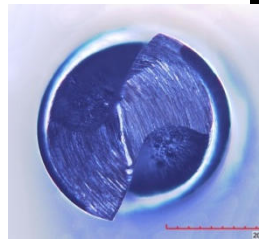
3D digital optical microscope HIROX KH 7700

- Magnification range 5 - 3 500 x
- High depth of field
- Dimensional and shape measurement
- Evaluation of 3D profiles
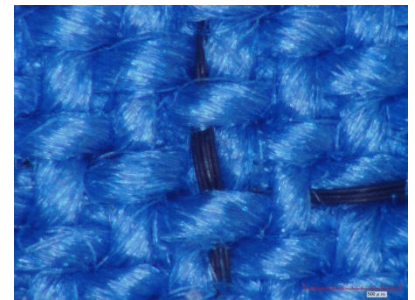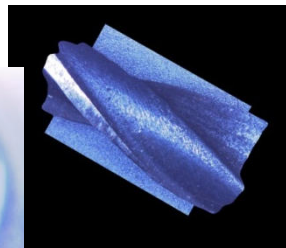- Possibility of big parts evaluation

Profile of WC-17%Co coating after the wear test

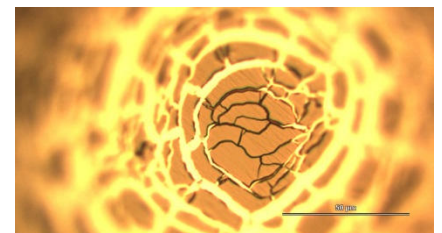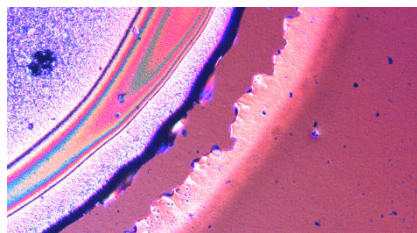Thread from four different angles of observation

3D imaging of drill

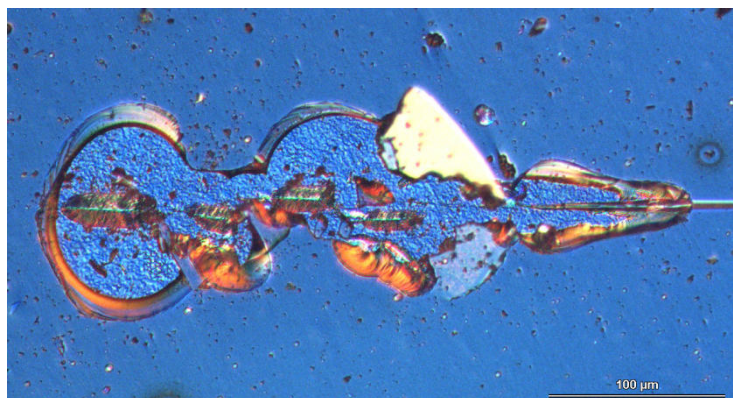Fabric with weaven carbide fibres

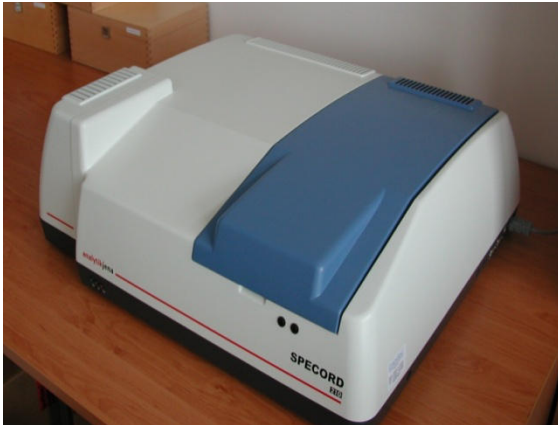## Metallographic microscope Epiphot 200

Indention test of DLC film

Metallographic microscope:
- Brightfield observation
- Darkfield observation
- Simple polarization observation
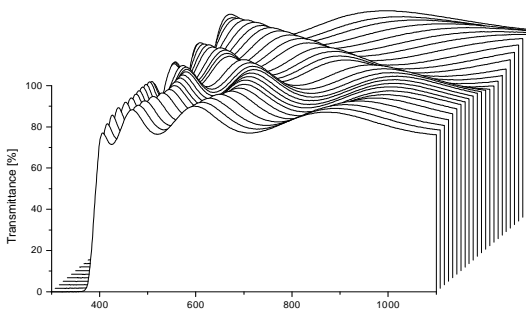- Nomarski DIC
- Magnification 50x – 1000x

Scratch test of DLC film

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Optical spectroscopy

UV/VIS Spectrophotometer Specord 210 BU

- Spectral range 190 – 1100 nm
- Measurement of transmittance, absorbance and reflectance in dependence on the wavelength
- Possibility of optical parameters determination:
    - Thin film thickness measurement
    - Spectral refractive index and extinction coefficient
    - Dielectric functions
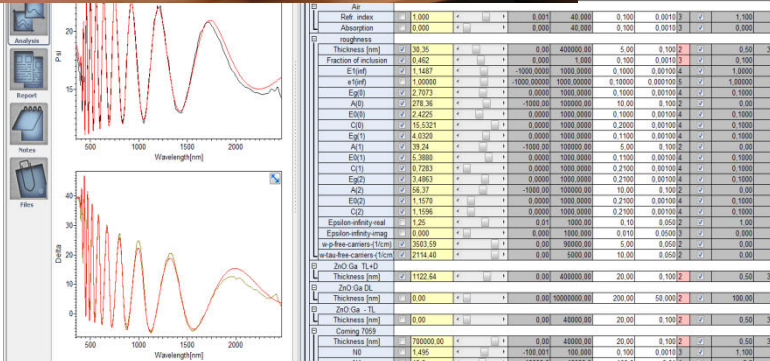   - Optical band gap in transparent semiconductors

Accessories

- Transmittance Attachment (of solid samples)
- Absolute Reflectance Attachment (determine the absolute reflectance of reflecting films)
- Variable Angle Reflectance Attachment (range 10 – 60°)
- Integrating Sphere (for the measurement of diffuse transmittance and diffuse reflectance)

Transmittance spectrums measured on variable thickness thin films

Spectroscopic ellipsometer SENTECH SE850

- Spectral range 240 – 2500 nm
- Ellipsometric parameters ψ, Δ, transmittance and reflectance measurement in dependence on the wavelength
- Possibility of determination parameters of individual thin layers:
    - Thin film thickness and surface roughness
    - Optical properties
        - Spectral refractive index and extinction coefficient
        - Dielectric functions
        - Optical thickness of band gap in transparent semiconductors
    - Electrical properties
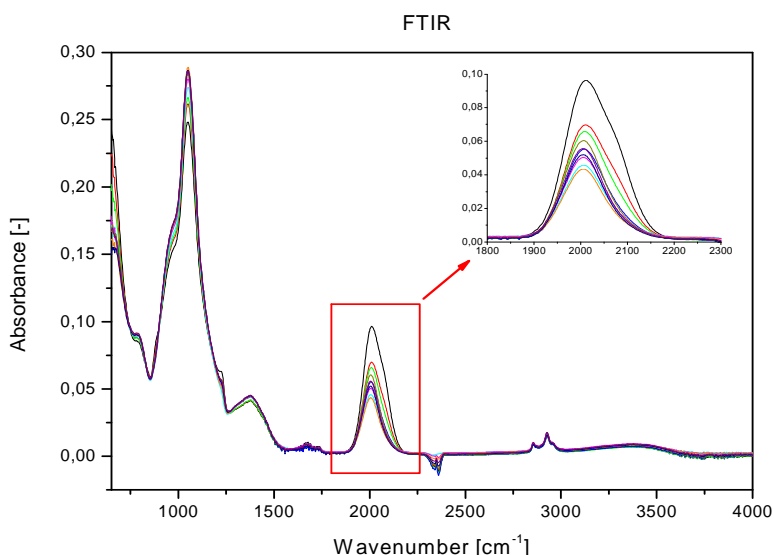   - Resistivity, concentration and mobility of charge carriers

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FTIR Spectrometer NICOLET 380

- Max / min range limit 7800 – 350 cm^{-1}
- Measurement of transmittance, absorbance and reflectance in dependence on the wavenumber

Accessories

- Specular Reflection Accessory (for surface analysis)
- Smart SAGA (for the analysis of thin films on reflective substrates, the 80 degree angle of incidence, sensitive measurement of films as thin as 0.1 nm)
- iTR Ge attachment with reflectance amplification effect in range 4000 – 400 cm^{-1}
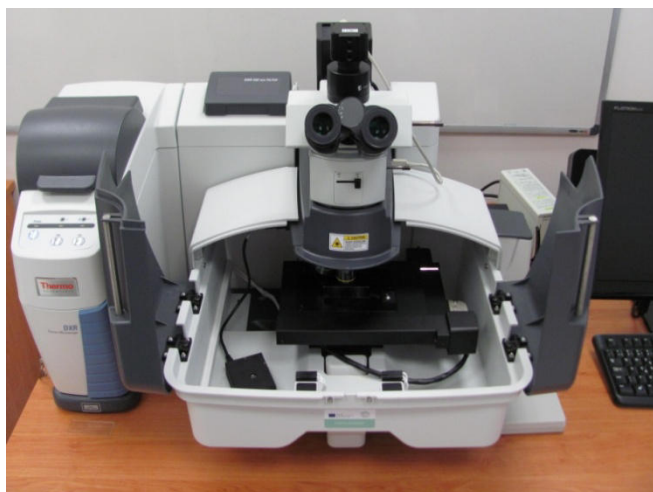
DXR Raman Microscopy

- Spectral range 3000 – 50 cm^{-1}
- 4 excitation lasers (532nm, 633nm, 780nm high-performance, 780nm high-luminance), motorized table with 3-axis movements
- Microscope have trinocular for visual view and video-screen simultaneously
- Head can carry 5 objectives – 4x, 10x, 20x, 50x, 100x with long working distance
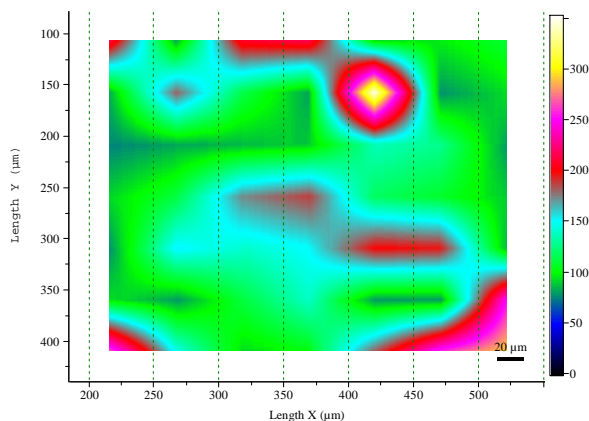

Possibilities of Raman spectroscopy

- Identification of chemical compounds
- Homogeneity of structure
- Polymers and biopolymers application
- Pharmaceutics application
- Mineralogy, authenticity and quality of rare minerals
- Control of raw materials
- Art and archeology

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Resistivity of thin films and solar simulator

4-point probe

- Measurement of low and high resistivity of thin films and bulk materials
- Determination of the type of conductivity of semiconductors
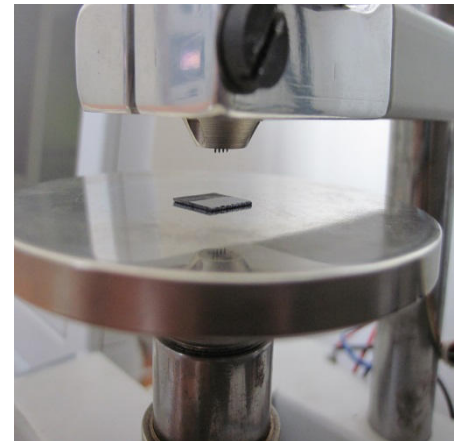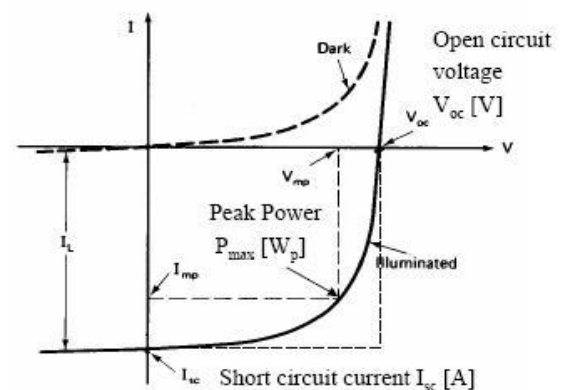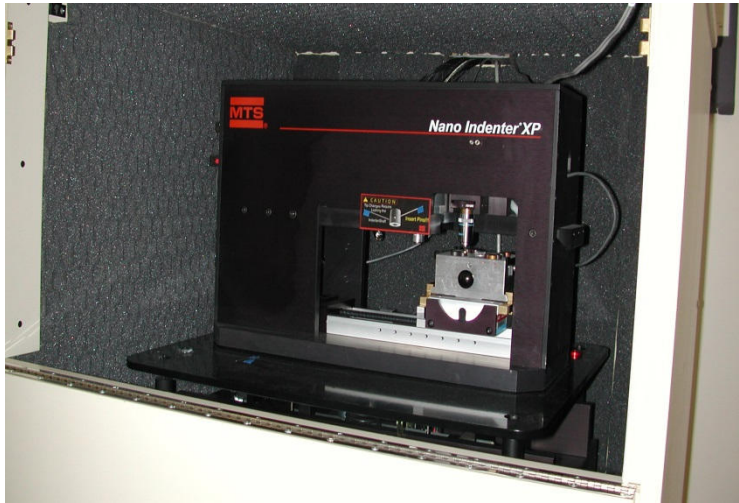

Newport Oriel class AAA

Solar simulator Oriel class AAA

- Spectral range 400 – 1100nm
- Uniform and collimated light on an area 100x100mm
- Xenon arc lamp 450W
- 100mW/cm
- Filter AM 1,5 Global
- Meets the standards EIC 60904-9:2007 JIS C 8912, ASTM E927-05

- Measurement of current-voltage characteristics of solar cells
- Determination of solar cell parameters:
  - Open circuit voltage (Voc)
  - Short-circuit current (Isc)
  - Current density (Jsc)
  - Fill factor (FF)
  - Solar cell efficiency (μ)
  - Maximum output power (Pmax)

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NanoIndenter XP with system CSM
(Continuous Stiffness Measurement)

SPECIFICATIONS OF NI XP:

Displacement resolution	<0.01 nm
Total Indenter travel	2 mm
Maximum indentation depth	>500 µm
Max. load with high-load option	10 N (1 kg)
Load resolution	50 nN (5.1 µgm)
Contact force	<1.0 µN
Positioning accuracy	1.5 µm
Video screen	25X
Objective	10X & 40X & 100X

POSSIBILITIES OF NI XP:

Superior means of nanomechanical characterization
Full characterization of film – substrate interactions
Hardness and modulus as a function of depth
Creep measurement
Microscratch test, Profilometer
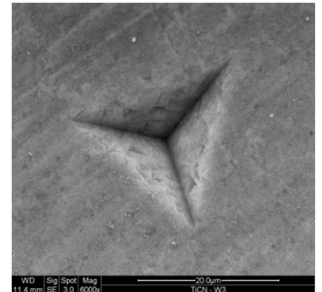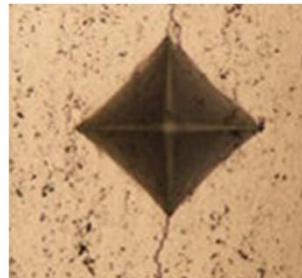
Tribometer CSEM

SPECIFICATIONS OF TRIBOMETER:

- Pin-on-disc method
- Precisely calibrated friction and wear measurements at elevated temperatures up to 800°C
- Efficient heating/cooling system to accurately maintain desired test temperature
- High thermal stability
- Automatic shut-off at selected track length or friction coefficient threshold
- Powerful software package for PC-controlled data acquisition and instrument control

POSSIBILITIES OF TRIBOMETER:

- Simulate in-service conditions up to 800°C

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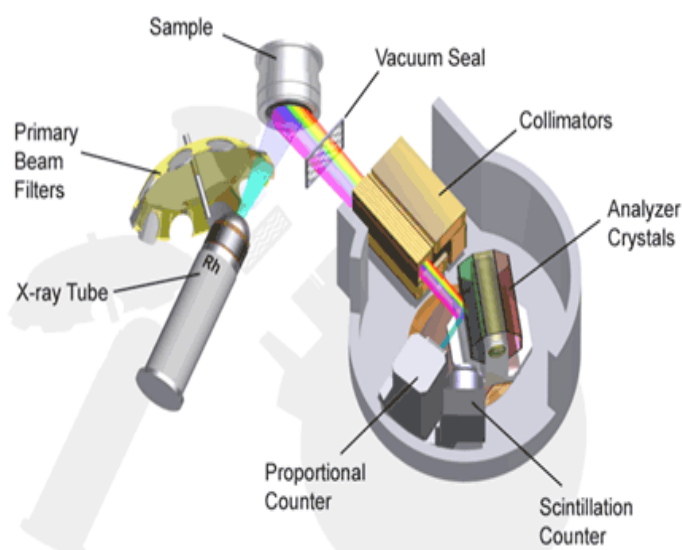NEW TECHNOLOGIES
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Bruker AXS S4 EXPLORER

Wavelength dispersive X-ray fluorescence spectrometer (WDXRF)

- Qualitative and quantitative analysis
- Analyse of solid and powder materials (minerals, ceramics, metals, glass, polymers…)
- Fast and easy sample preparation
- Elements from Be to U in all kind of samples
- Typical detection limits (LLD): ~ 1 to 10 ppm
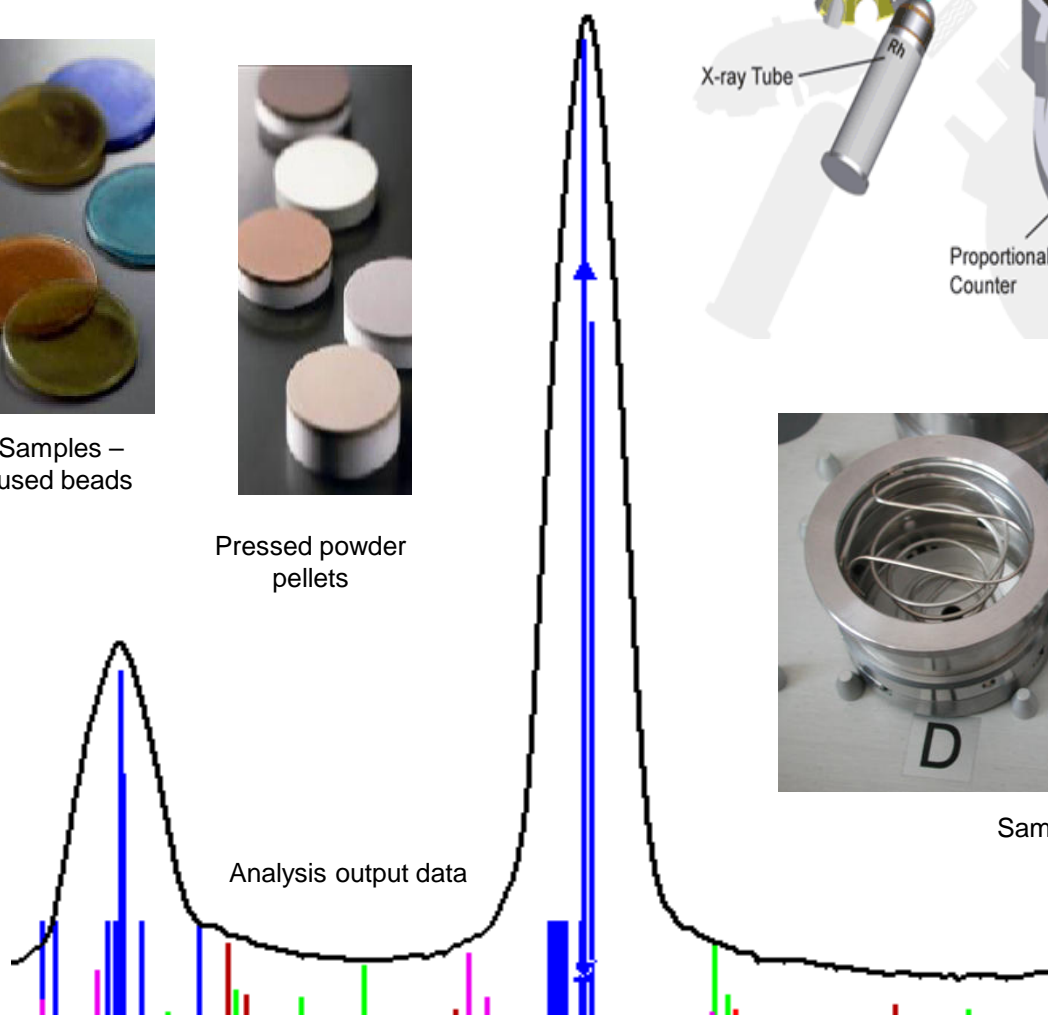- Analyse metals, pressed powder pellets or fused beads

Samples – fused beads

Pressed powder pellets

Analysis output data

Samples holders

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X-Ray diffraction

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Automatic diffractometer Panalytical X'Pert Pro

Applications:

- Qualitative and quantitative phase analysis
- Residual stress analysis
- Texture analysis
- Analysis of changes in the crystal structure
- High temperature phase transformations
- Ultra fast data collection with X'Celerator
- Thin film analysis – signal measured from thin film only without influence of signal from substrate – Xe detector (primary monochromator possible)
- Can be used for bulk materials, powders and thin films

Configuration for measure of high-temperature phase transformations
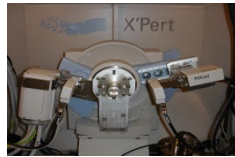
Theta-theta goniometer

Diffraction record of fine-grained material - ZnO powder
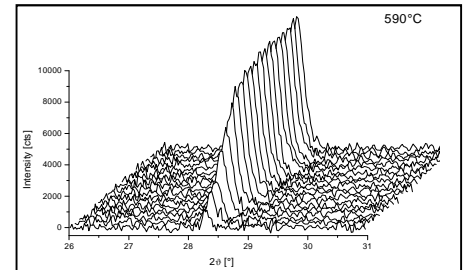
Development of recrystallization of amorphous silicon

Automatic diffractometer Bruker AXS D8 Discover with 2D detector

Measurement possibilities :

- Qualitative and quantitative phase analysis
- Qualitative and quantitative texture analysis
- Residual stress analysis (lattice stress)
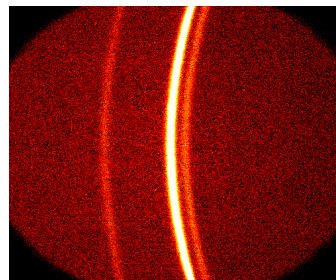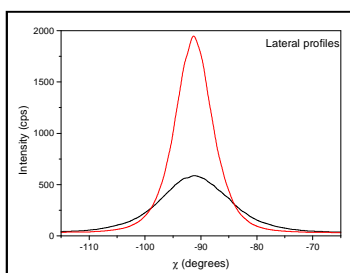- Microstructure (microstrain, crystallite size)
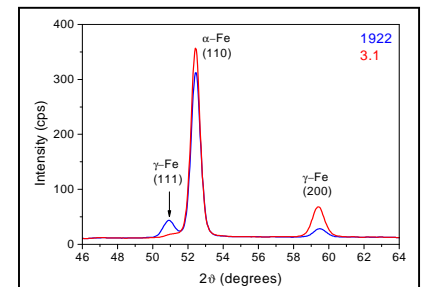
Eulerian cradle and 2D-detector
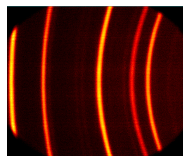
Typical record of a fine-grained material – Al_2O_3 powder
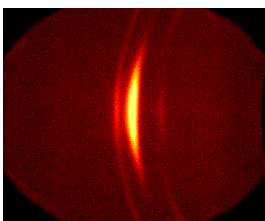
ZnO thin film with preferred orientation

Investigation of austenite content in „TRIP" steels by means of X-ray diffraction technique

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Thin film deposition

## BOC Edwards TF600 electron beam evaporation and sputtering system

- Deposition of thin films using PVD technology
- RF Sputtering – up to 600 W
- DC Sputtering – up to 1,5 kW
- Electron beam evaporation processing
- Automatic process control
- Turbomolecular and dry rotary pumping system
- Ultimate pressure: $4,35 \times 10^{-7}$ mbar
- Process gas : Argon
- Working gas : Nitrogen, Oxygen

Glow discharge

Deposition chamber
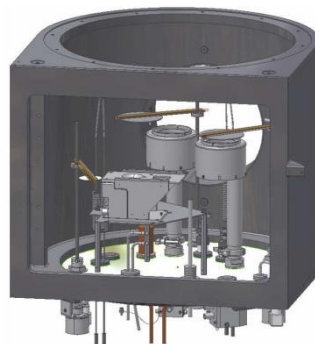
SAMCO PD 220NA PE-CVD system

- Deposition of silicon oxide (SiOx)
- Deposition of silicon nitride (SixNy)
- Deposition of silicon oxy-nitride (SiOxNy)
- Deposition of hydrogenated amorphous silicon (a-Si:H)
- Fully automatic "one-button" operation time with full manual override
- Homogeneous deposition area up to 300mm
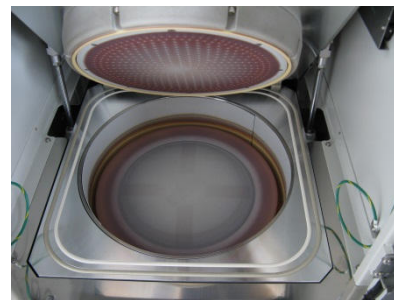
Elettrorava PE-CVD 5-chamber deposition system
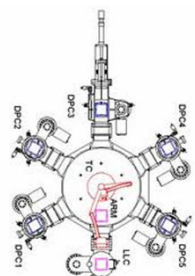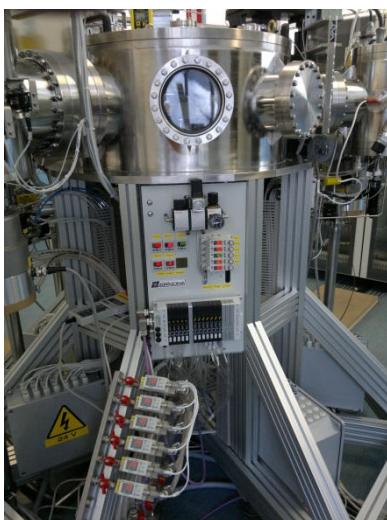
- Grow of thin film solar cell based on silicon in p-i-n / n-i-p configuration
- Deposition of silicon oxide (SiO_x), silicon nitride (Si_xN_y) and silicon oxo-nitrides (SiO_xN_y) with refractive index and semiconductor band-gap control
- Silicon doping to p- and n-conductivity type
- Simple and tandems structures, substrate / superstrate, micromorph

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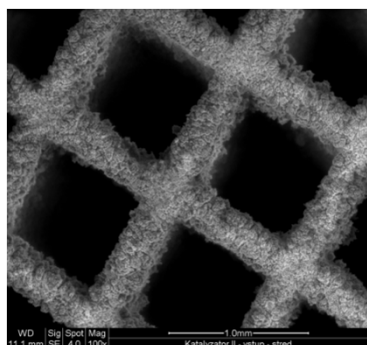# Scanning electron microscopy
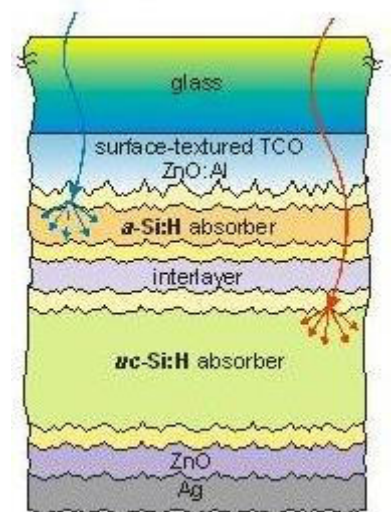
FEI Quanta 200

High-performance thermal emission SEM with EDS (microprobe) and WDS from EDAX
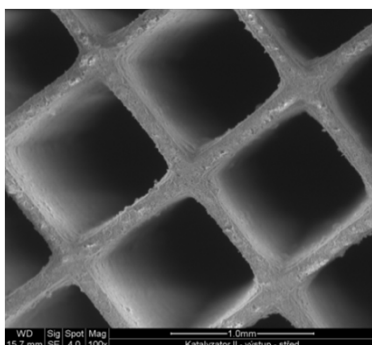
- Resolution
  - High-vacuum
    - 3.0 nm at 30 kV (SE)
    - 4.0 nm at 30 kV (BSE)
    - 10 nm at 3 kV (SE)
  - Environmental mode (ESEM) for nonconductive samples without coatings
    - 3.0 nm at 30 kV (SE)
- Accelerating voltage: 200 V – 30 kV
- Probe current: up to 2 µA – continuously adjustable

Solving the problems with solid sediments in the car catalyst created during the car operation

Input - 100x

Output - 100x

## ZnO thin film prepared by magnetron sputtering
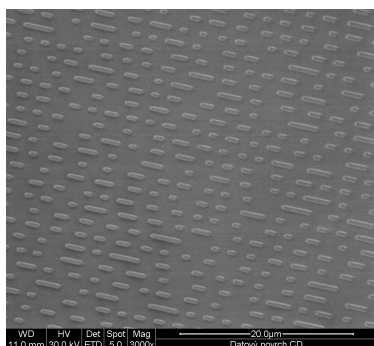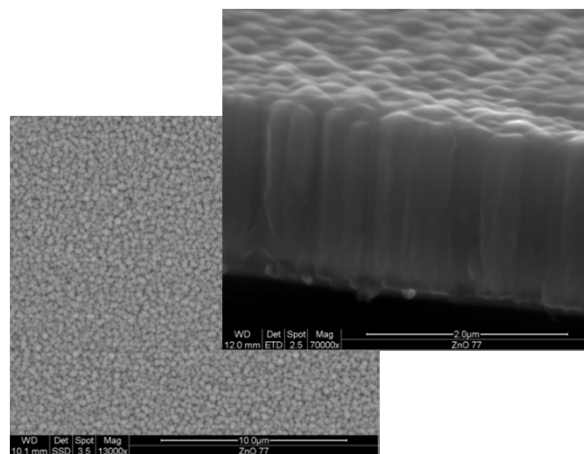
CD surface
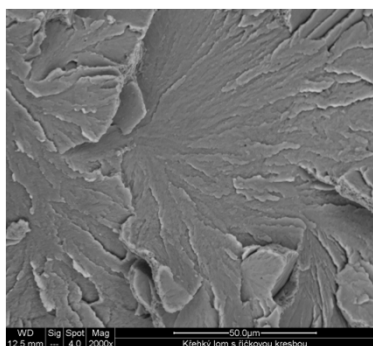
Brittle fracture
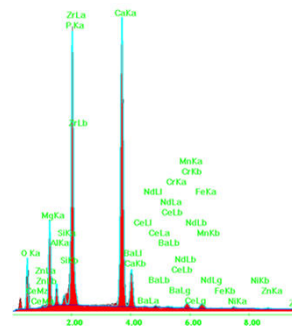
Element analysis

Source: Projects of MaT NTC

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JEOL JSM-7600F

- Ultra-high resolution Field Emission SEM
- Resolution 1nm at 15kV, 1.5nm at 1kV in GB mode
- Element detectors EDS, WDS
- Electron microdiffraction detector (EBSD)
- Build-in energy filter (r-filter) of measured electrons
- Gentle Beam to reduce damage of sensitive samples and reduce charging of non-conductive samples
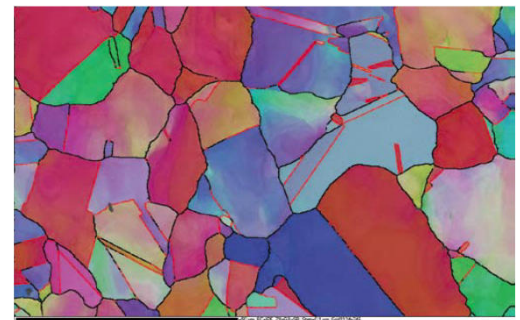- Magnification 25 – 1 000 000x
- Accelerating voltage: 100 V – 30 kV
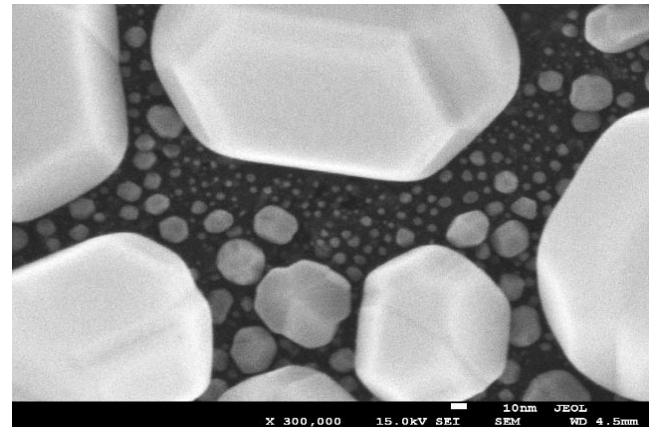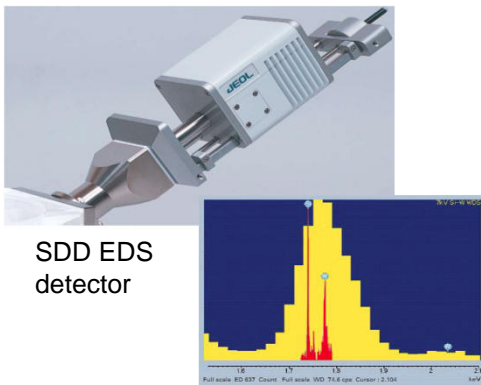
Gold particles, 15 kV, mag. 300 000x
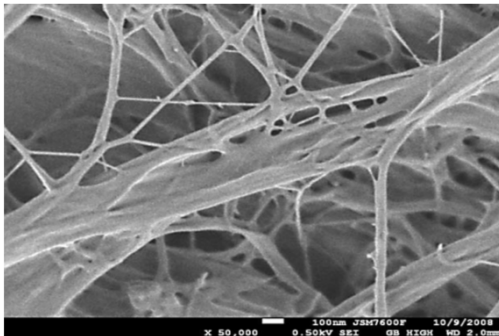
Paper filter, 2.0 kV, mag. 25 000x

O

Ni

Ti
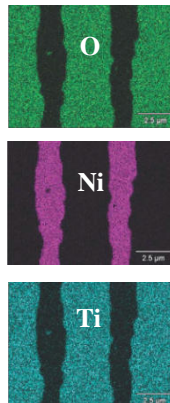
EDS maps of elements on multilayer capacitor cross-section

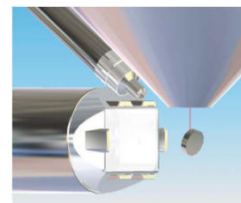EBSD orientation map of austenitic steel crystallites
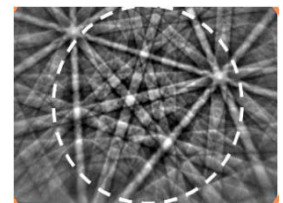
SDD EDS detector

WDS detector

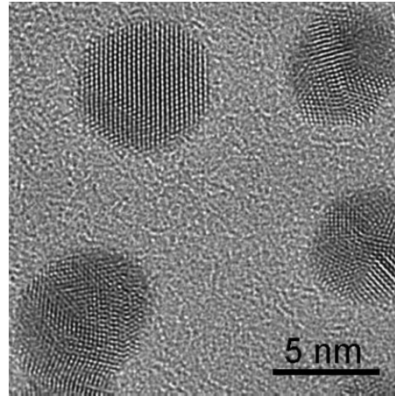Combined spectrum

HLK Nordlys II S EBSD system

Kikuchi bands

Source: JEOL

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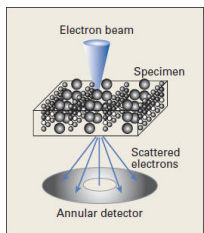
# Transmission electron microscopy

TEM mode, Au nanoparticles

5 nm

TEM Jeol JEM-2200FS

- Accelerating voltage: 80 – 200 kV
- In-column energy filter (Ω-filter)
- High-contrast imaging
- 3D reconstruction of samples
- Resolution 0.19 nm
- Magnification
  - TEM mode 50 to 1 500 000x
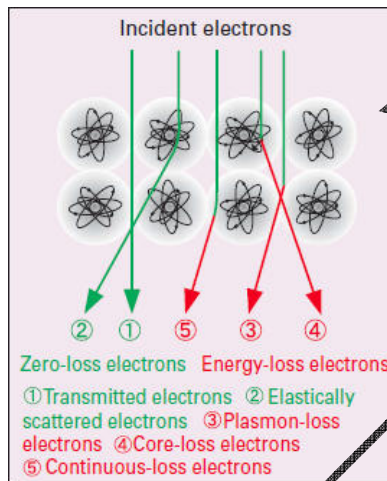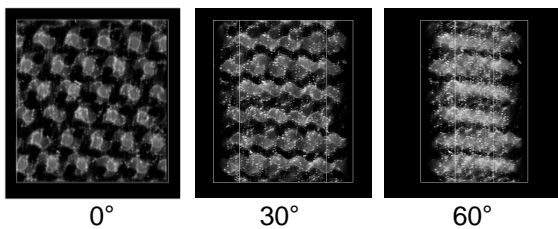 - STEM mode 100x to 150 000 000x

In-column omega filter
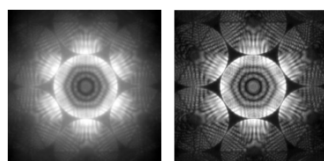
High-Angle Annular Dark Field (HAADF) detector

0.136 nm
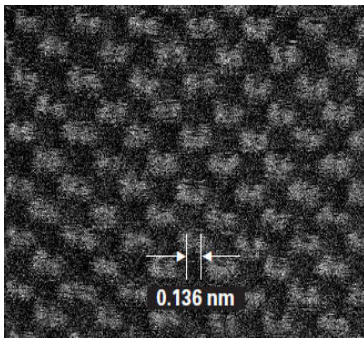
Si plane [110], STEM mode, HAADF detector

3D reconstruction of sample structure from series of tilted TEM images

0°   30°   60°

Automatic tilting procedure

Result of 3D morphology Pd particles on polymer surface

Incident electrons

② ① ⑤ ③ ④
Zero-loss electrons   Energy-loss electrons
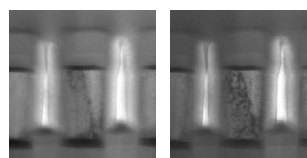①Transmitted electrons ②Elastically scattered electrons ③Plasmon-loss electrons ④Core-loss electrons ⑤Continuous-loss electrons
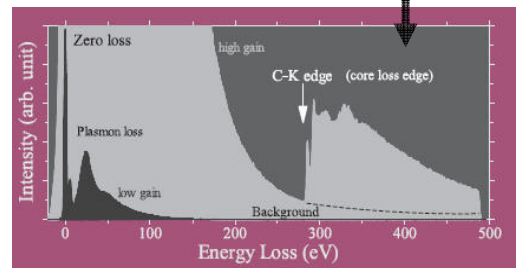
Without Ω-filter   Filtered

Electron diffraction of SI [111] at 200kV

Without Ω-filter   Filtered

DRAM memory at 200kV

Specimen   Electron beam
Omega-type energy filter
Zero-loss electrons   Energy-loss electrons
Energy dispersion plane
Energy selectig slit
Energy-filtered image Observation plane

Zero loss   high gain
C-K edge (core loss edge)
Plasmon loss
low gain
Background
Intensity (arb. unit)
Energy Loss (eV)   0 100 200 300 400 500
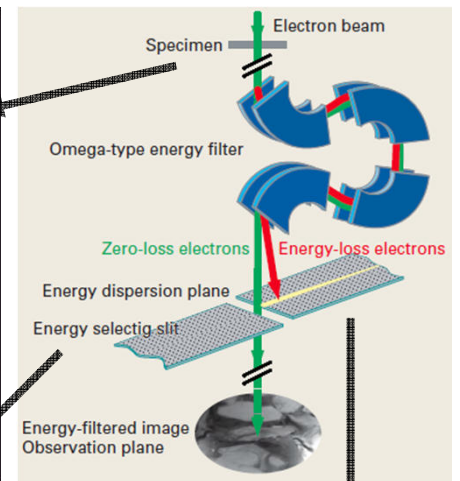
Electron Energy Loss Spectrum (EELS)

N   O   Ti
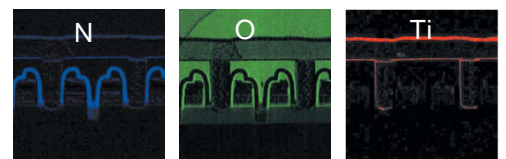
EELS TEM mapping of element distribution in FLASH memory

Source: JEOL

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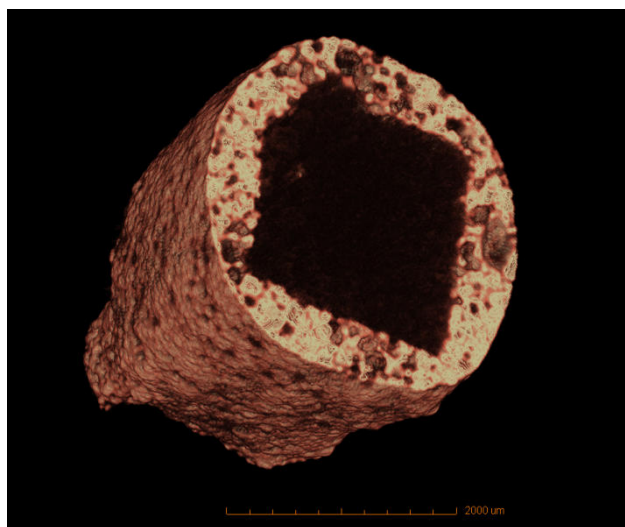**NEW TECHNOLOGIES RESEARCH CENTRE UNIVERSITY OF WEST BOHEMIA**
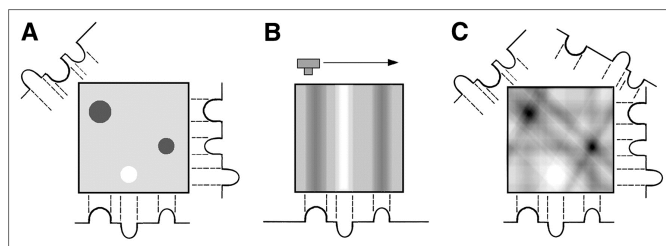
X-ray Xradia MicroXCT-400

- High-resolution Computer Tomography
- Non destructive 3D measurement of the sample
- Without special sample treatment
- Maximal dimension of sample 50 cm
- Maximal weight of sample 15 kg
- Field of view up to 7 cm
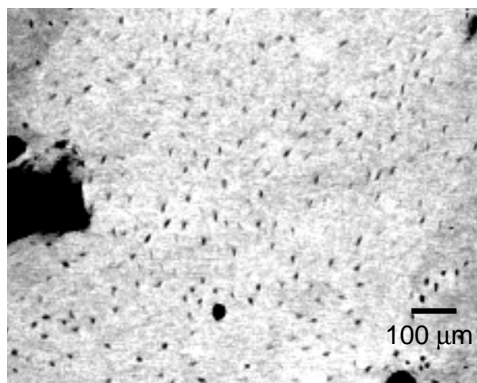- Available resolution 0.2 – 0.5 µm
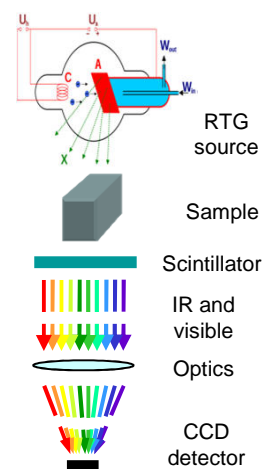- Accelerating voltage 20 – 90 kV

Principle of 3D reconstruction of internal sample structure

3D reconstruction of blood vessels

Virtual slice through the human bone

100 µm

RTG source

Sample

Scintillator

IR and visible

Optics

CCD detector

X-ray picture collection

Size and distribution of pores inside the safety match
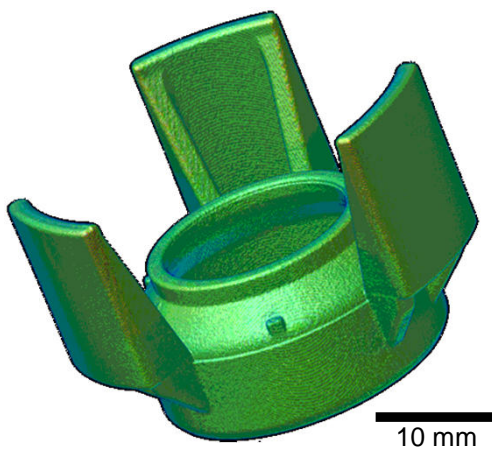
3D model of plastic molding to compare the product with the CAD template

10 mm

Tobacco in cigarettes with filter

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