EMPA

Materials Science & Technology

Application of advanced and non destructive testing in solid oxide fuel cells

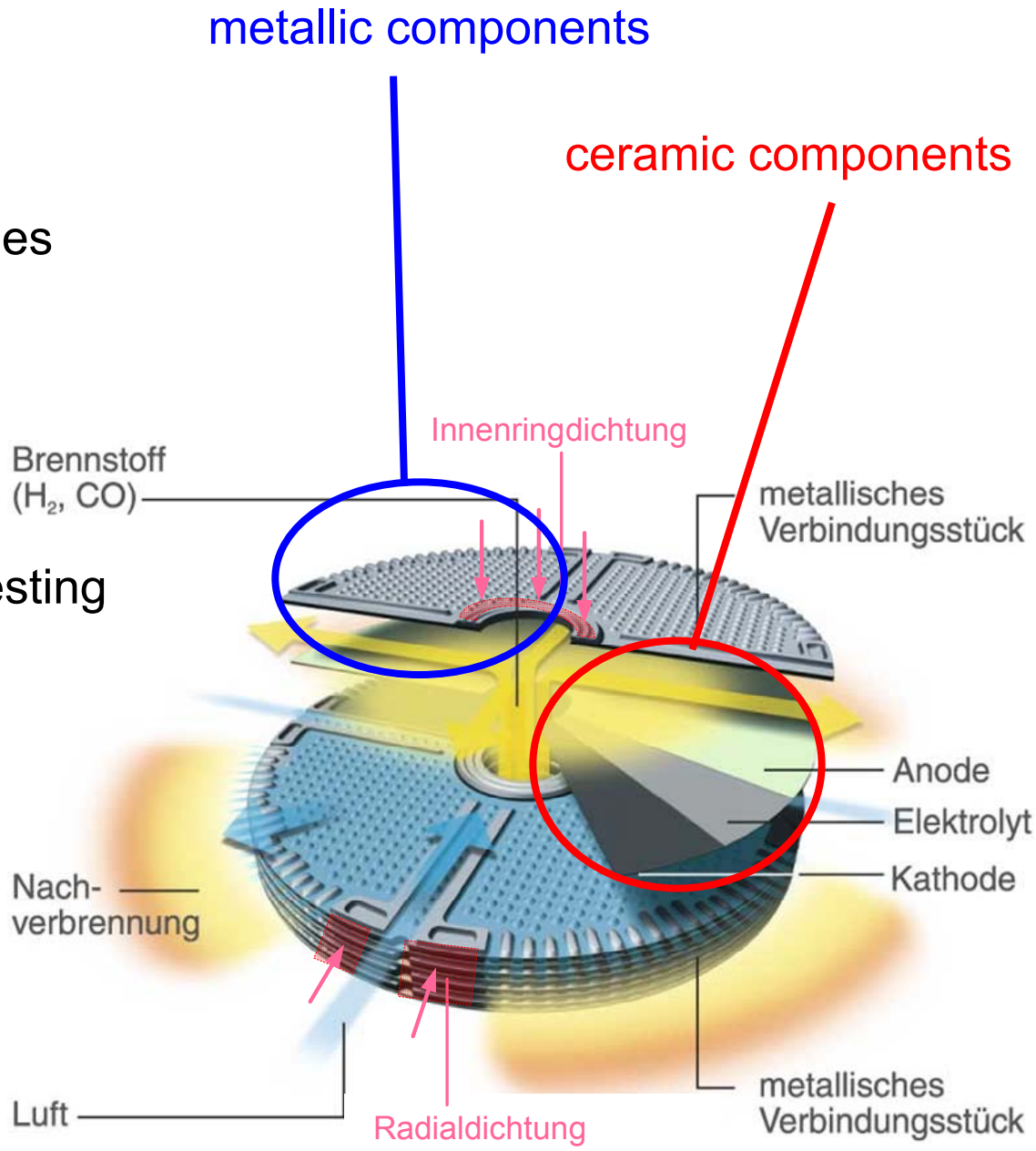
Peter Wyss, Erwin Hack
Laboratory for Electronics/Metrology/Reliability

Artur Braun, Lorenz Holzer, Christian Soltmann, Peter Holtappels Laboratory for High Performance Ceramics

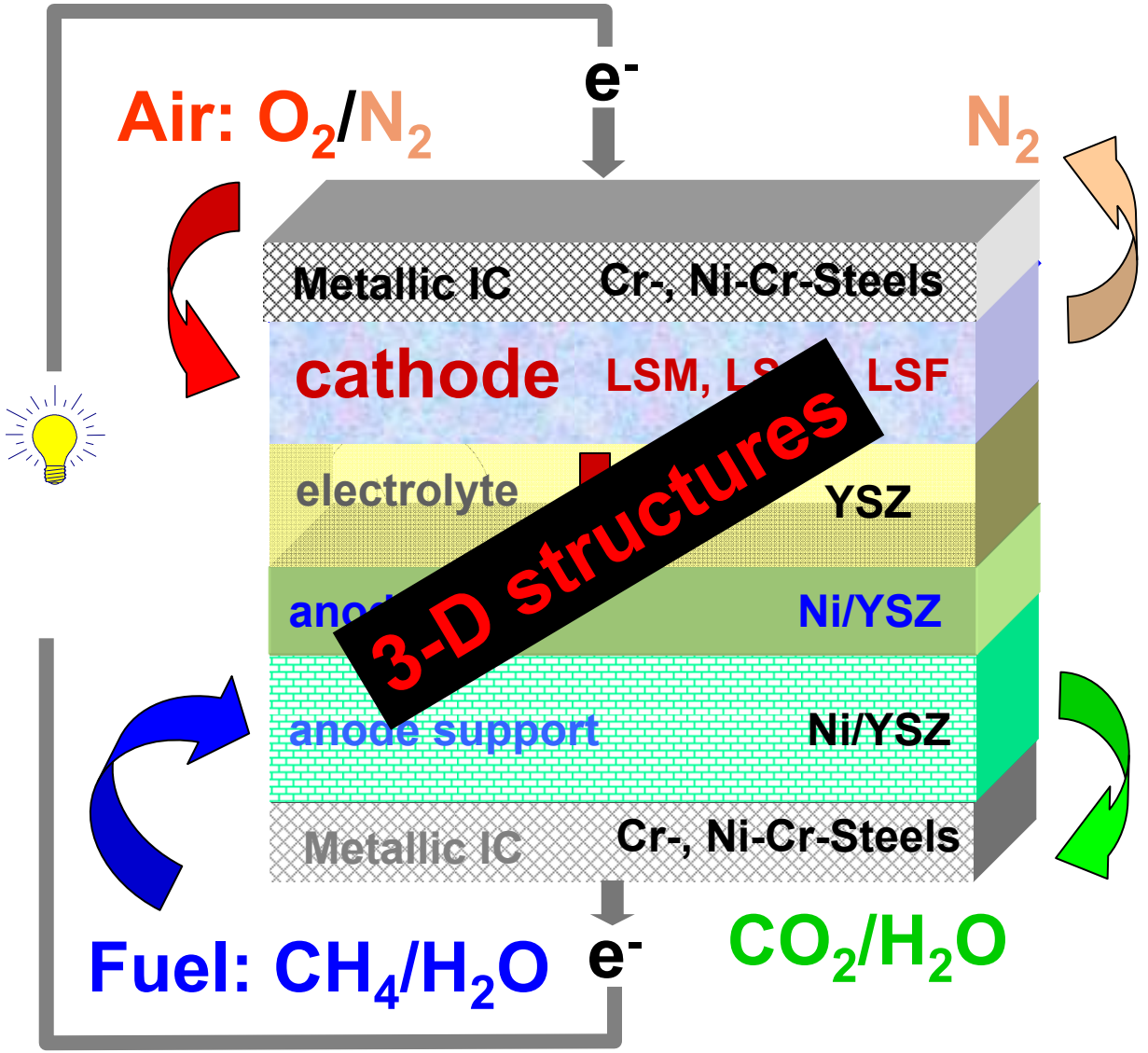
Empa, Swiss Federal Laboratories for Materials Testing and Research
Überlandstr 129 , CH-8600 Switzerland

Outline

- Test items and techniques
- Non-destructive testing
  - neutron tomography
  - x-ray radiography
  - thermography
- Advanced destructive testing
  - FIB tomography
- Outlook



Solid Oxide Fuel Cell - Principles -



SOFC features&scaling

Electrocatalytic activity
Nano/atomic scale

Ionic conductivity
Nano/atomic scale

Electronic conductivity
„Nano/atomic/micro scale“

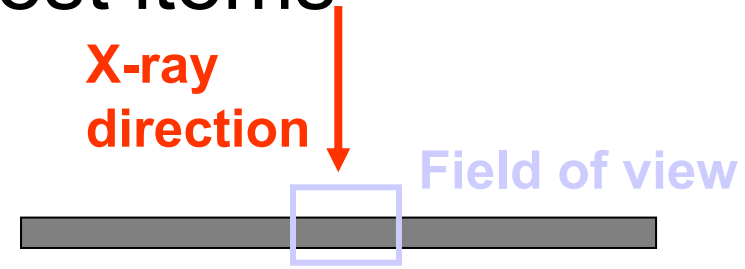
(Open) porosity
Microscale

SEM/OM/EDX
Only 2-D imaging

Non-destructive Testing (NdT)

component	state	potential problem	ndt test method
metallic	machined part	cracks, bad welds	X-ray RT, CT, UT for interconn.
metallic	assembled in stack	corrosion, contamination	RT, CT X-rays, Neutrons ?
ceramic	green machined part	porosity + homogenity, shrinkage cracks	RT, µCT local mode, X-rays, TT
ceramic	assembled and fired to cell	cracks + delaminations (thermal cycling)	RT, µCT local mode, X-rays, TT
ceramic	cells mounted in the stack	fatigue cracks (thermal cycling)	RT, CT, Neutrons + contrast fluid ?

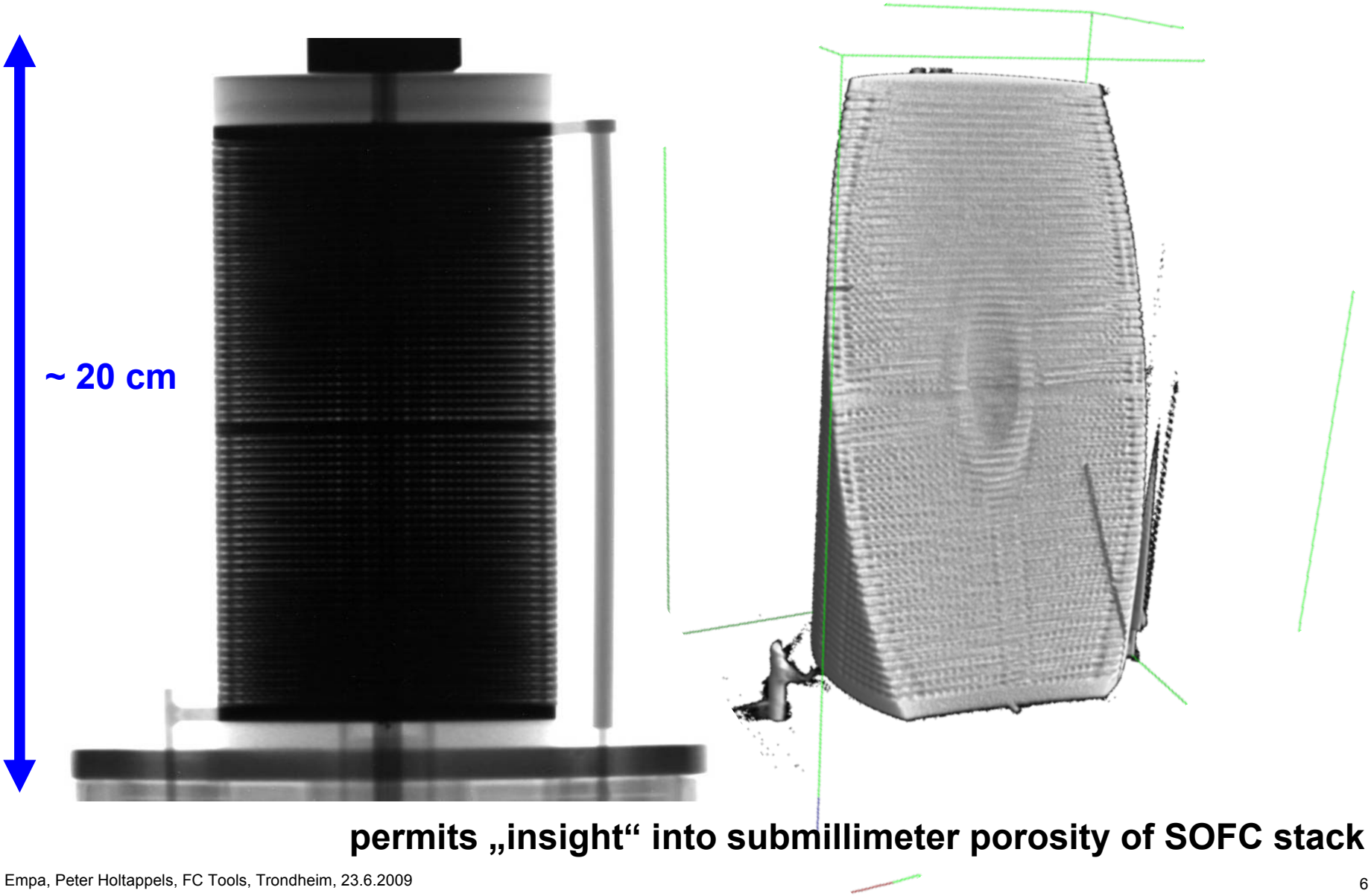
1. NdT methods and test items



RT system data (typical)

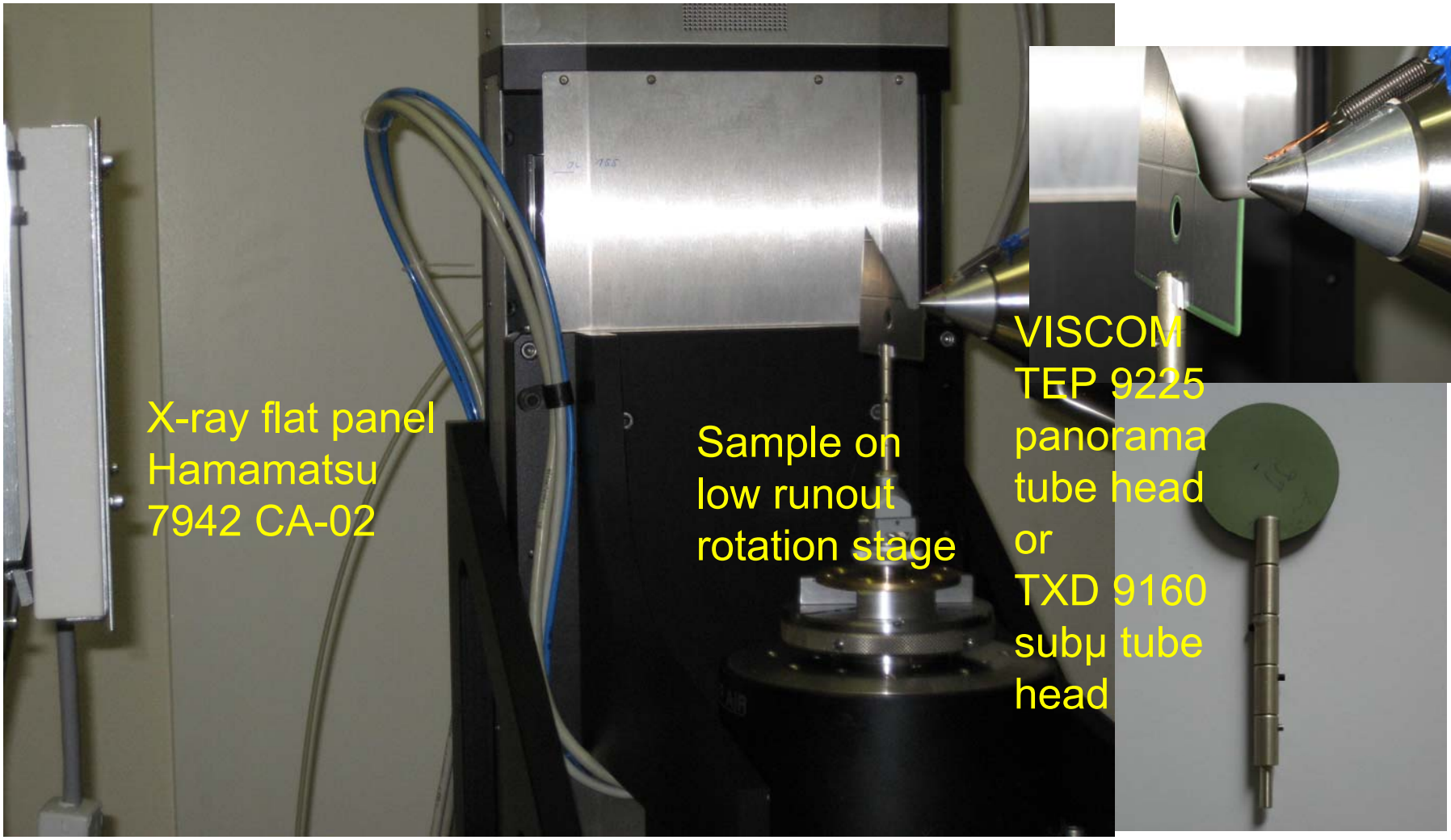
source	beam geometry	resolution in µm	field of view (FOV) in mm	typ. penetrable ZrO_2 in mm
Neutrons	almost parallel	100	300	> 200
X-ray Mini / Micro focus	conical	10	400	20
X-ray Synchrotron	parallel	1	10	2

Neutron tomography



permits „insight“ into submillimeter porosity of SOFC stack

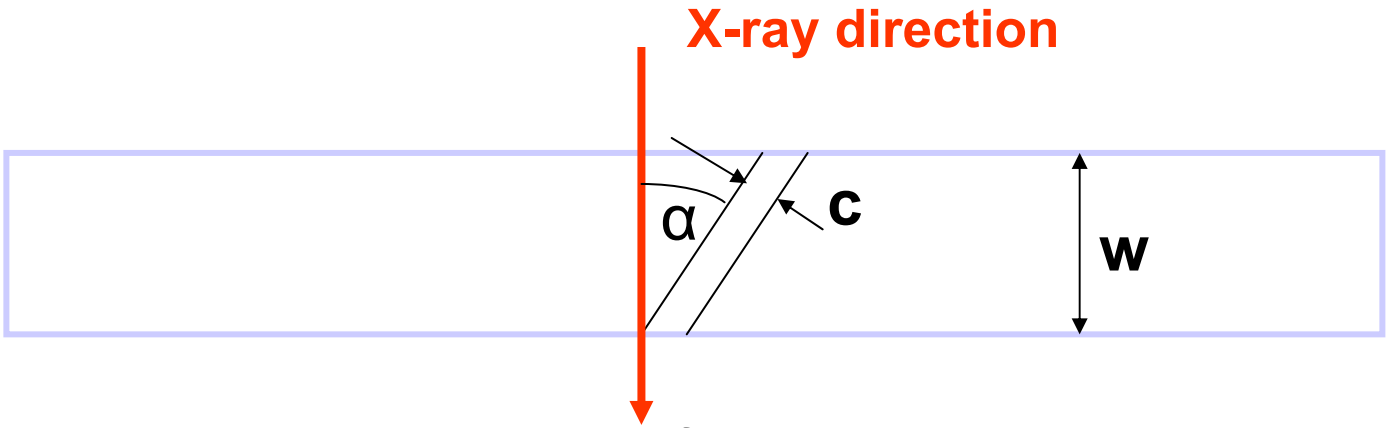
2. RT and local µCT to Empa YSZ pellets



The RT / CT microscopy (macroscopy) system

RT and local µCT to Empa YSZ pellets

The crack detection limit in radiography

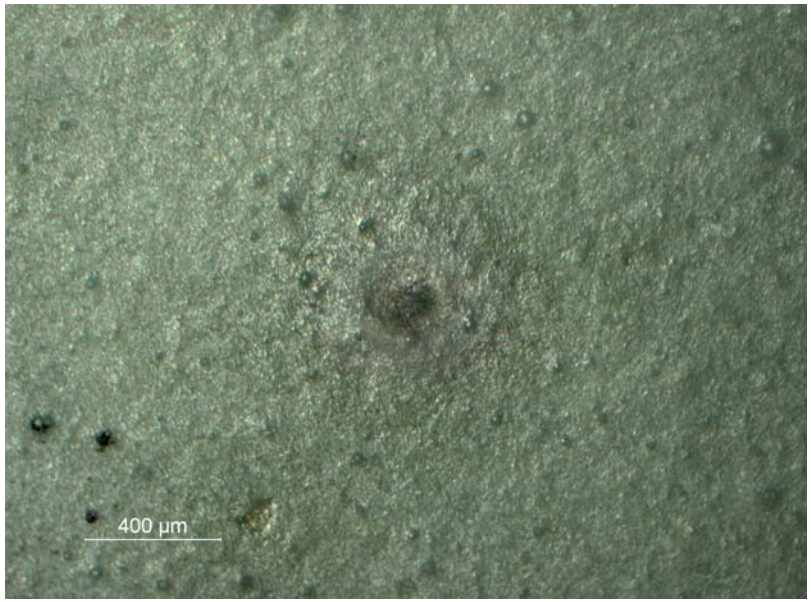


crack detection is possible if:

$$c > \begin{cases} 0.01\ w * \sin\alpha \\ \text{tube spot size} * 0.5 \end{cases}$$

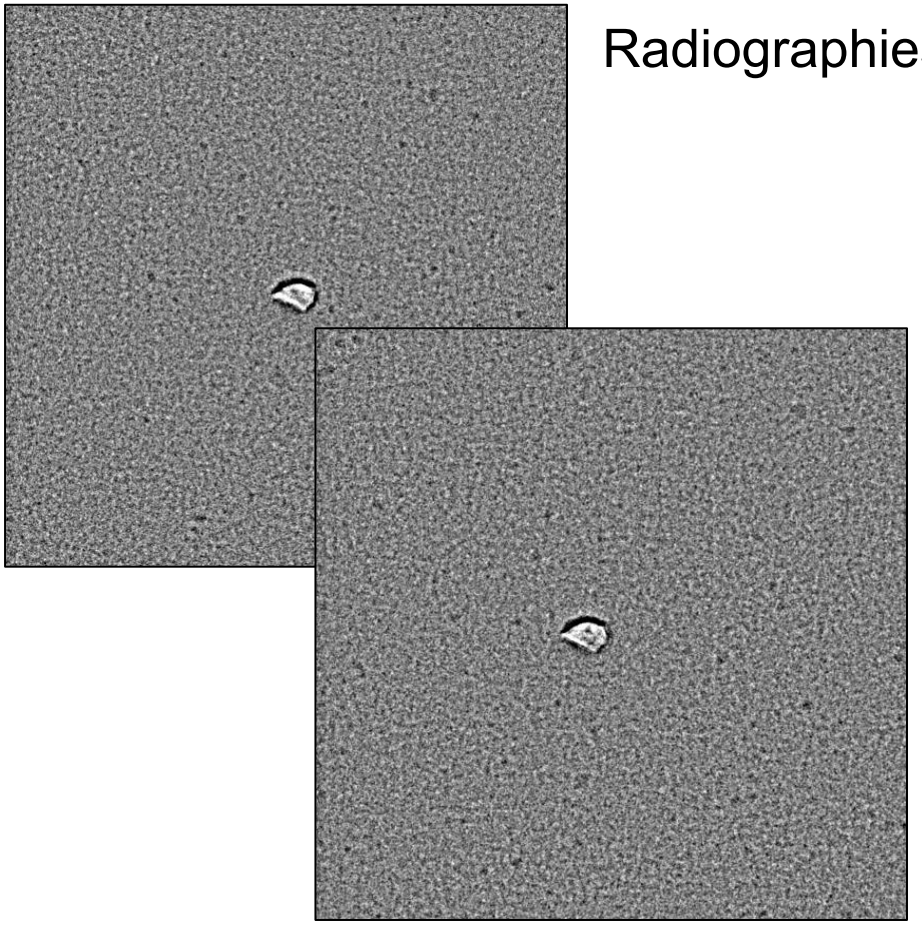
RT and local µCT to Empa YSZ pellets

Visible light microscopy



of a hidden coarse grain

Radiographies

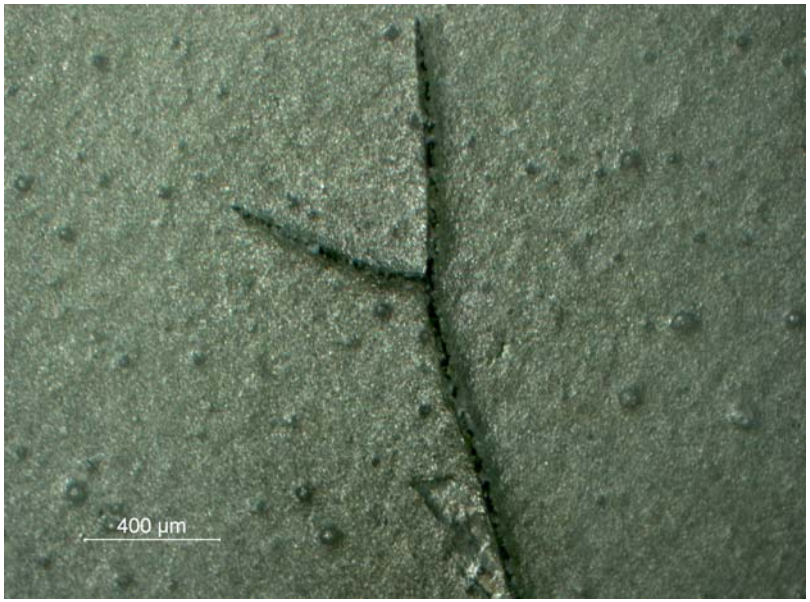


FOV 5 x 5 mm, pixelsize 2.5 µm

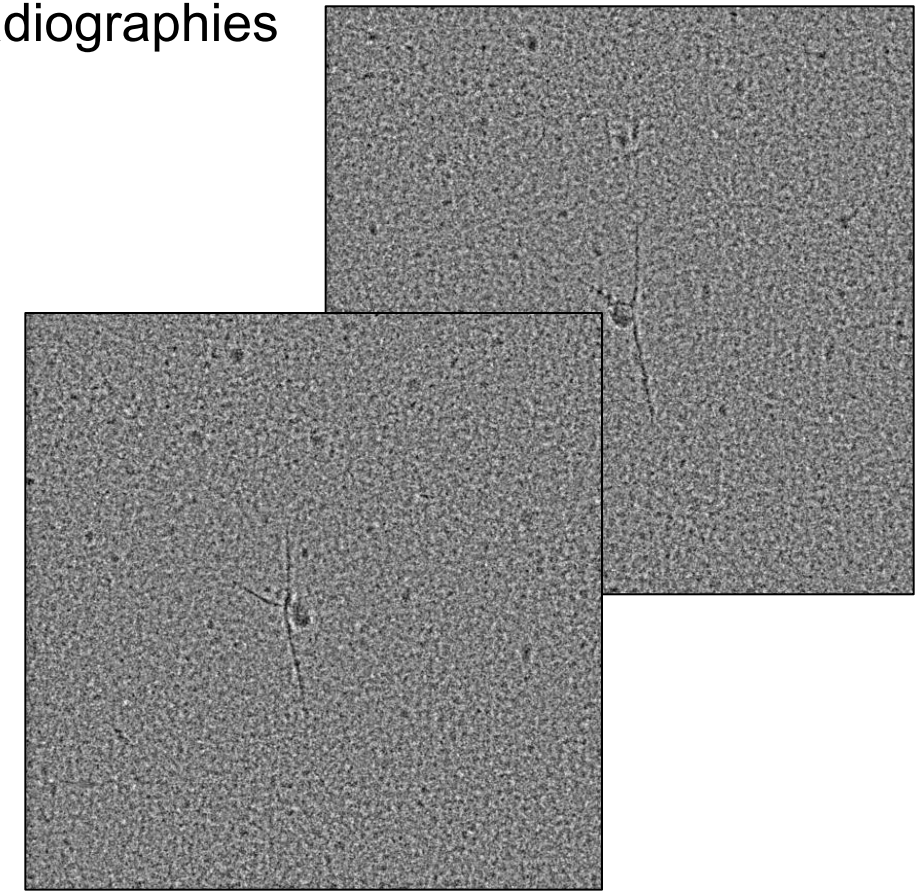
RT and local µCT to Empa YSZ pellets

Radiographies

Visible light microscopy



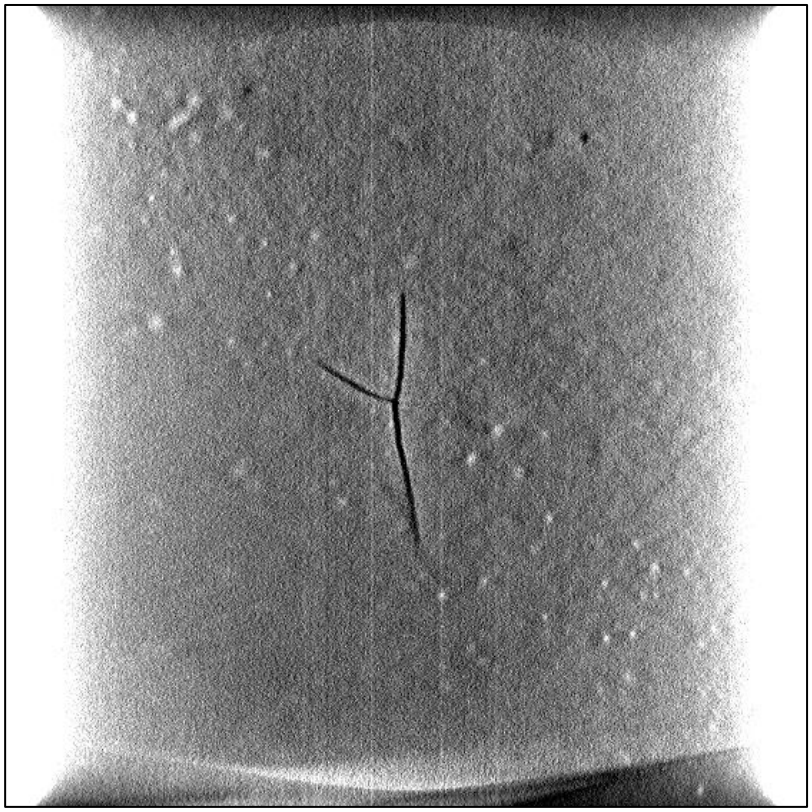
of a shrinkage crack



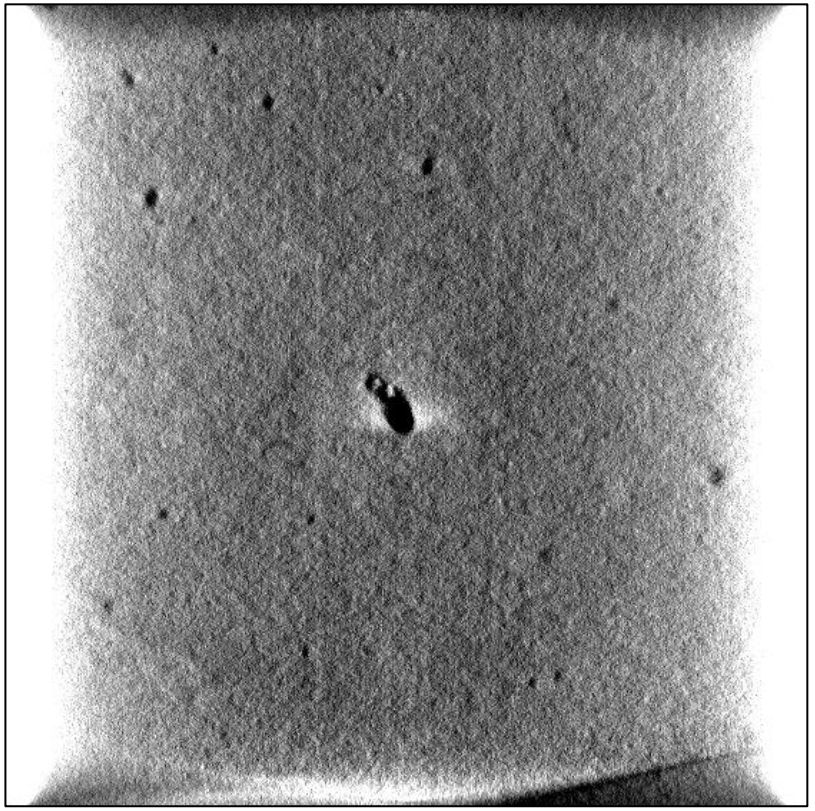
FOV 5 x 5 mm, pixelsize 2.5 µm

RT and local µCT to Empa YSZ pellets

Local X-ray µCT of crack in pellet Ø 35 x 1 mm

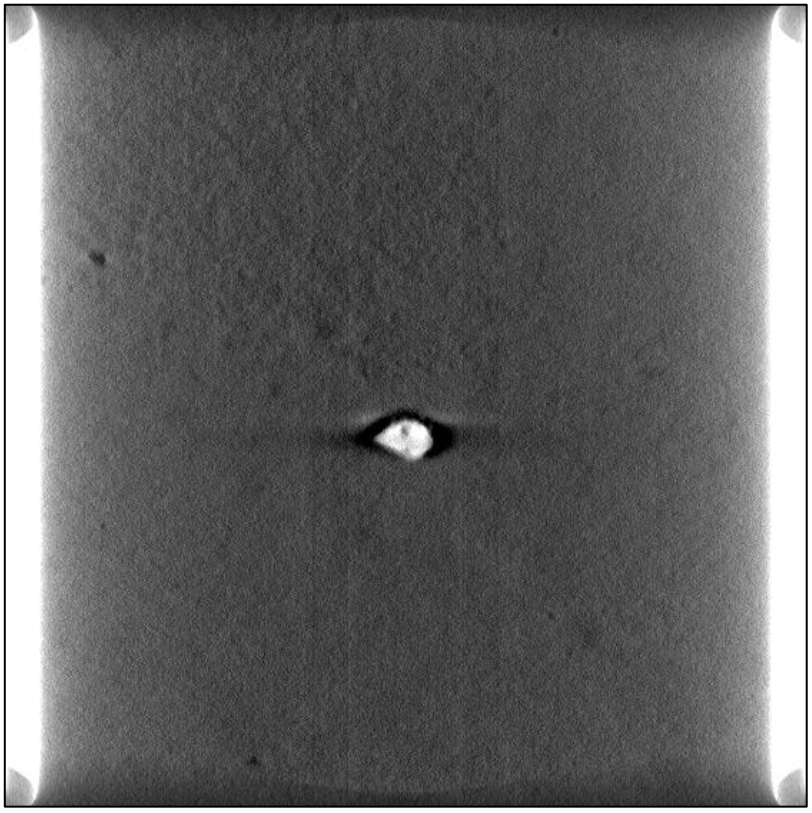


Field of view
5 x 5 mm,
voxelsize 5 µm

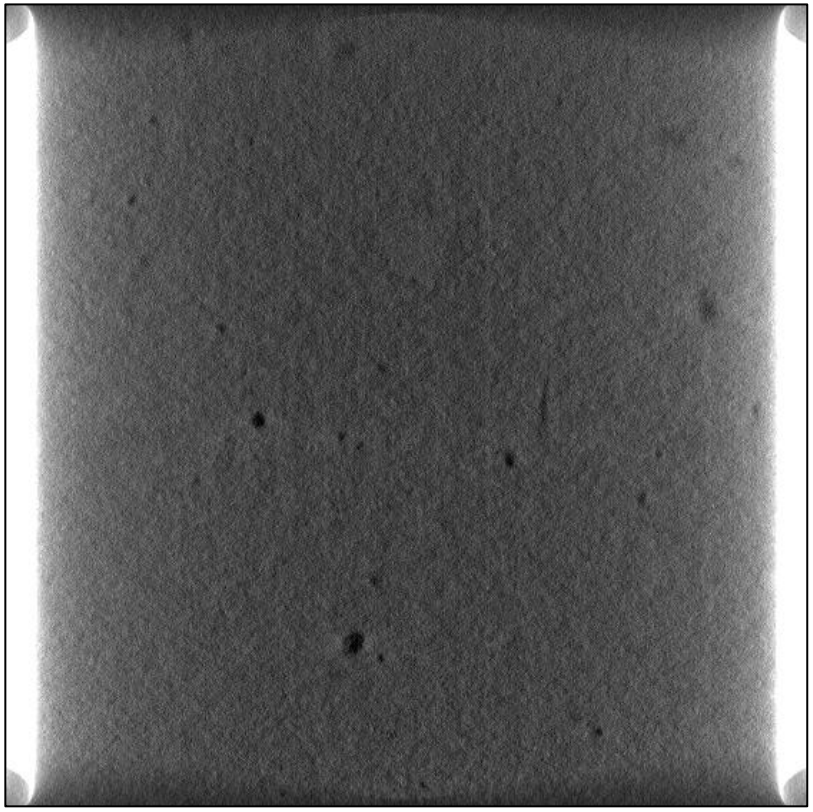


RT and local µCT to Empa YSZ pellets

Local X-ray µCT of hidden coarse grain



Field of view
5 x 5 mm,
voxelsize 5 µm

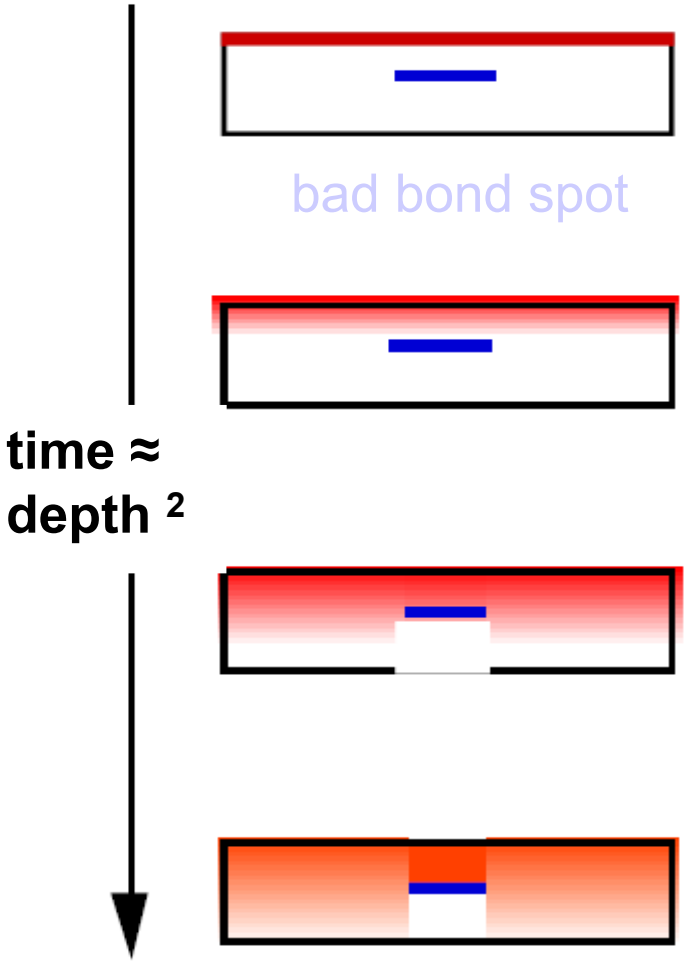


TT, RT and local µCT to HTceramics cells

Camera		Cedip JADE
Camera type		Array
Resolution		240 x 320 pix
Wavelength range		3-5 µm
Frame rate		Full: 170 Hz ROI: 9 kHz
Lateral resolution		15 x 15 μm^2
NETD		20 mK
Temp. range		-20 – 1300 °C
Lock-In frequency		< 5 kHz

The thermography cam, heart of the TT system

TT, RT and local µCT to HTceramics cells
Thermography testing (TT), impulse method



flash hits surface

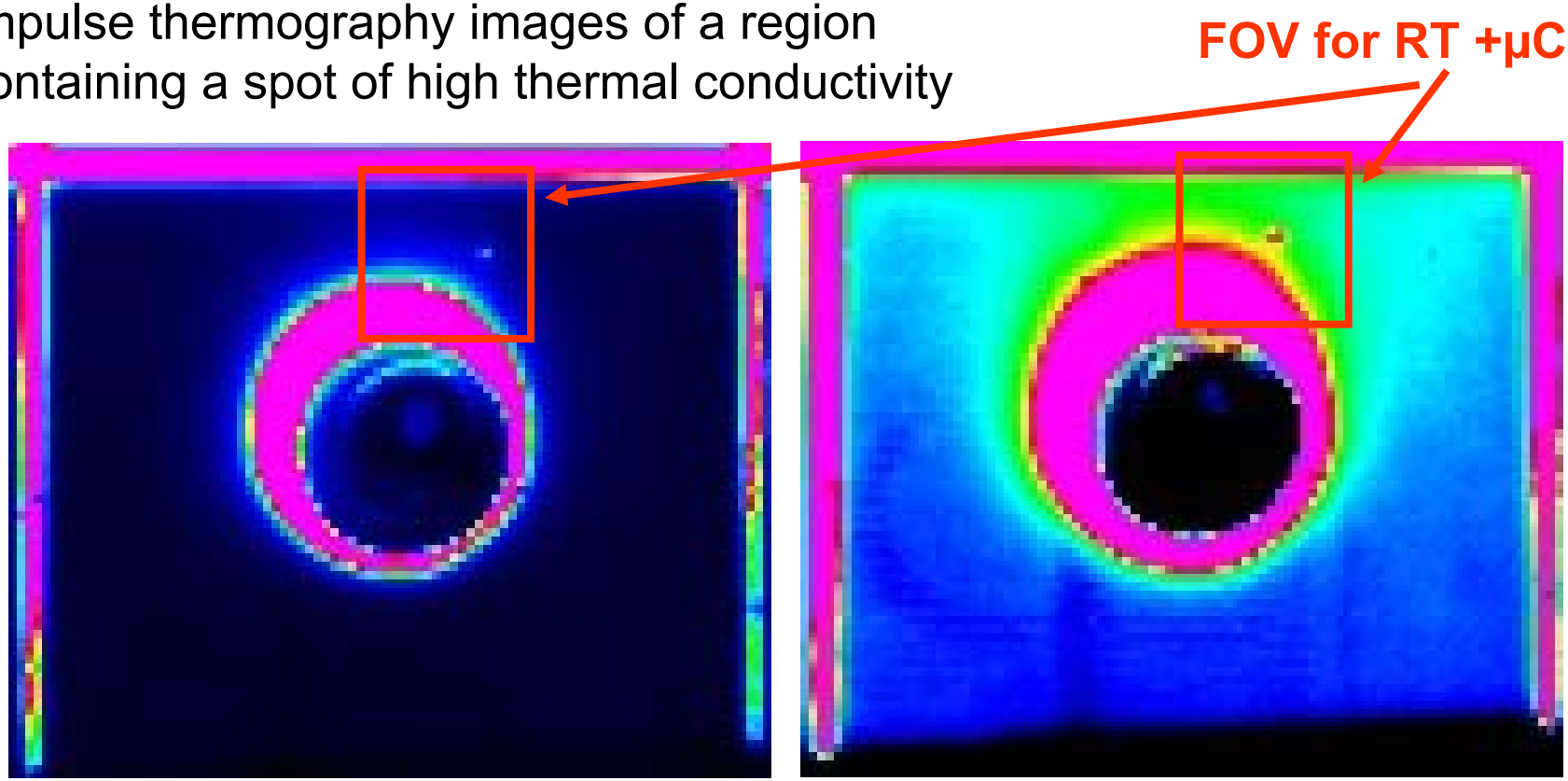
lateral resolution ≈ 2 x depth

diffusion wave propagates

bad bond spot stops the heat diffusion wave and after some time a thermic contrast appears: positive at the impulse side, negative at the rear side

TT, RT and local µCT to HTceramics cells

Impulse thermography images of a region containing a spot of high thermal conductivity



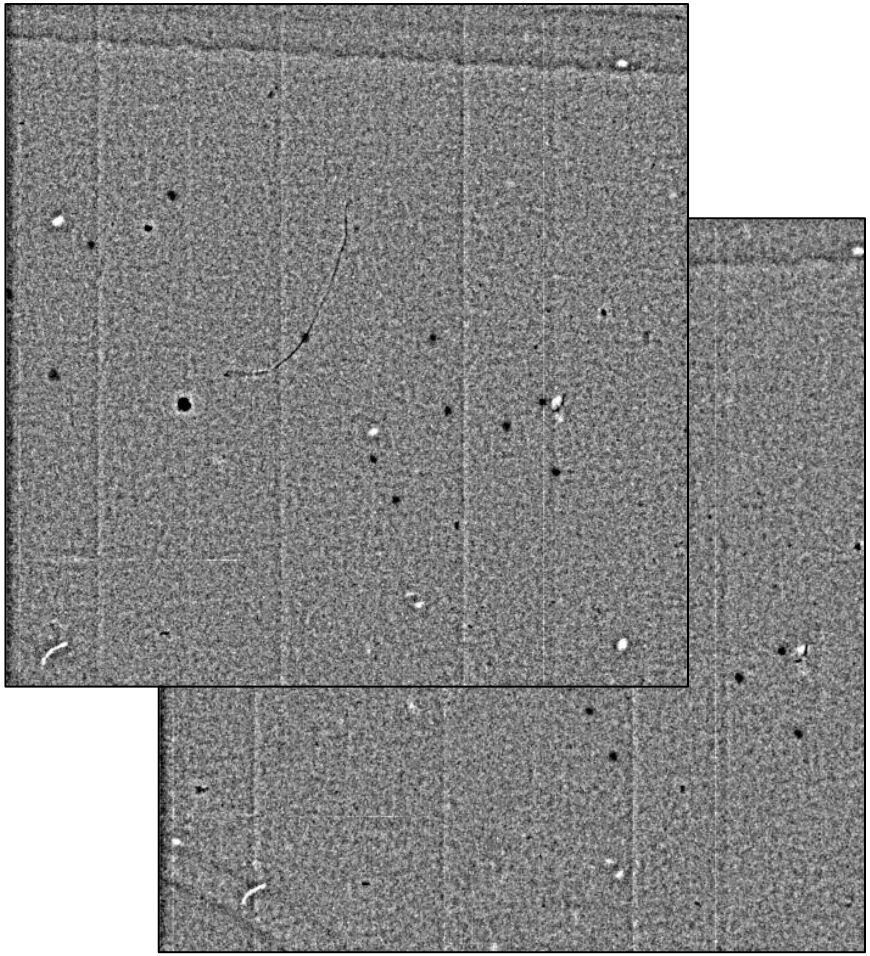
Istantaneous after flash

20 msec later

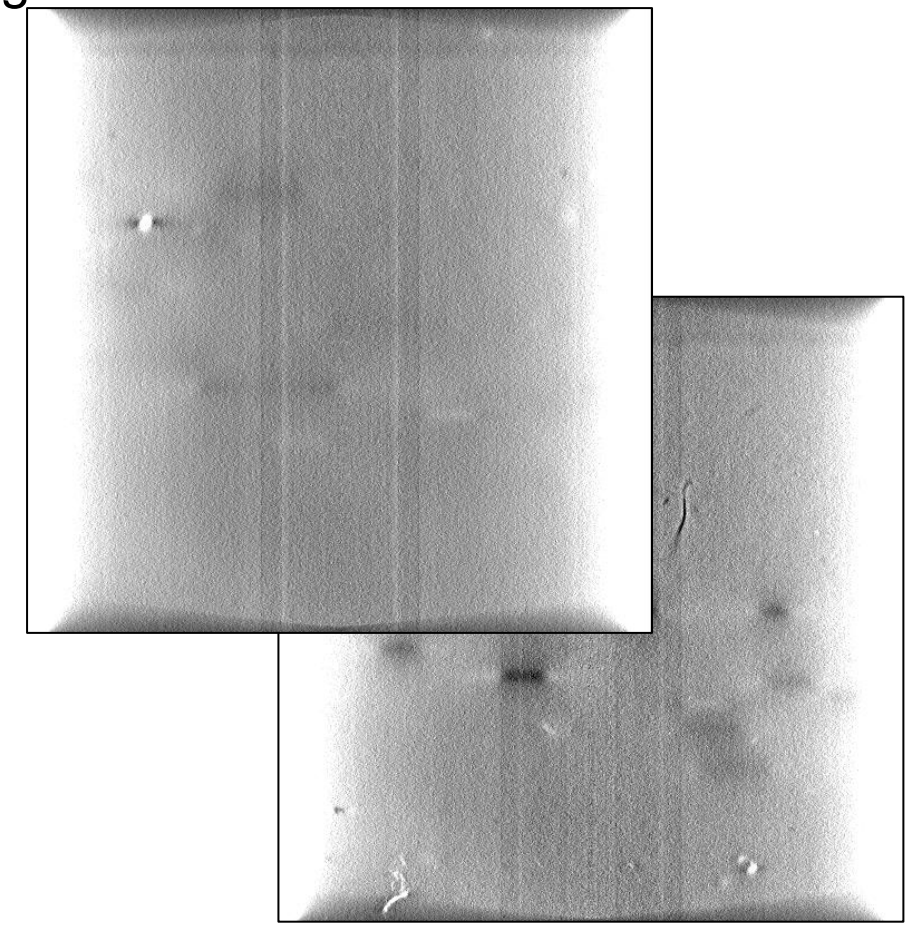
FOV 180 x 150 pixel or 28 x 25 mm, Pixelsize 167 µm

TT, RT and local µCT to HTceramics cells

The same region (FOV 5 x 5 mm) imaged with:



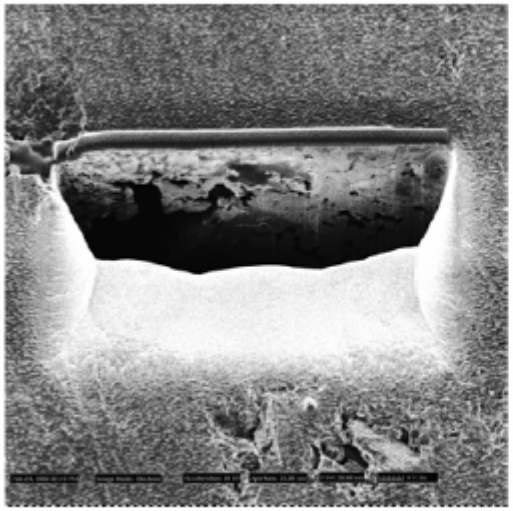
Radiographies, pixel size 2.5 µm

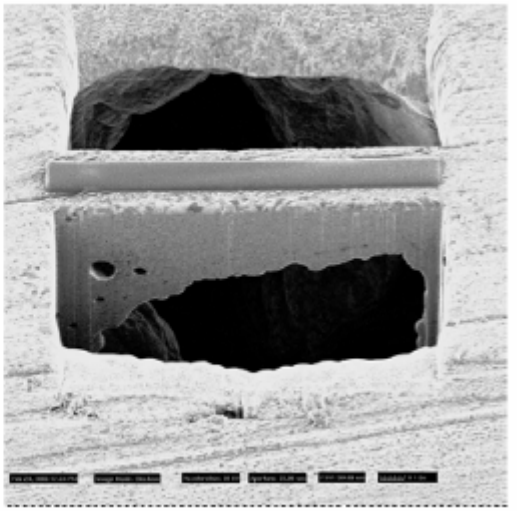


Local tomography, voxel size 5 µm

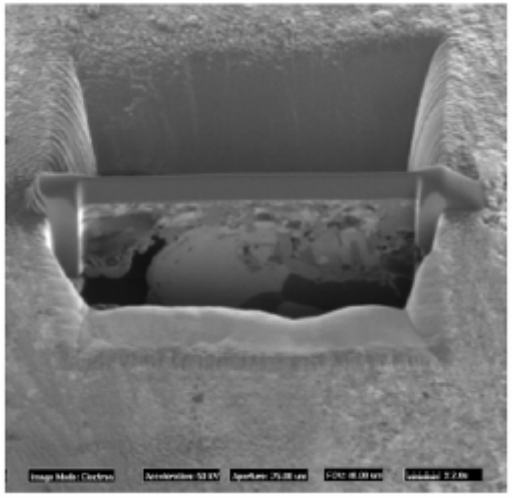
Focussed Ion Beam (FIB) technique

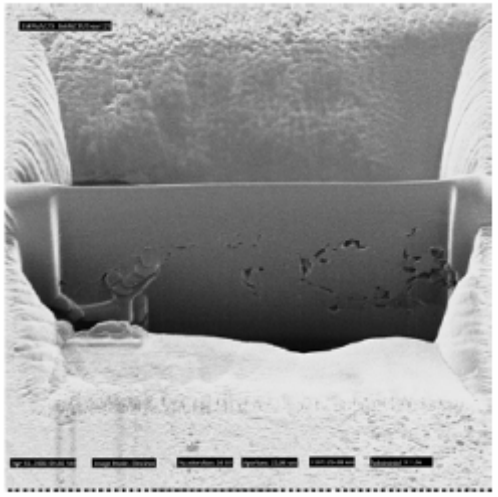
Conventional preparation procedure

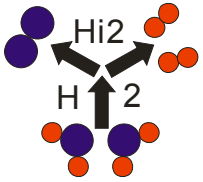




Advanced preparation procedure

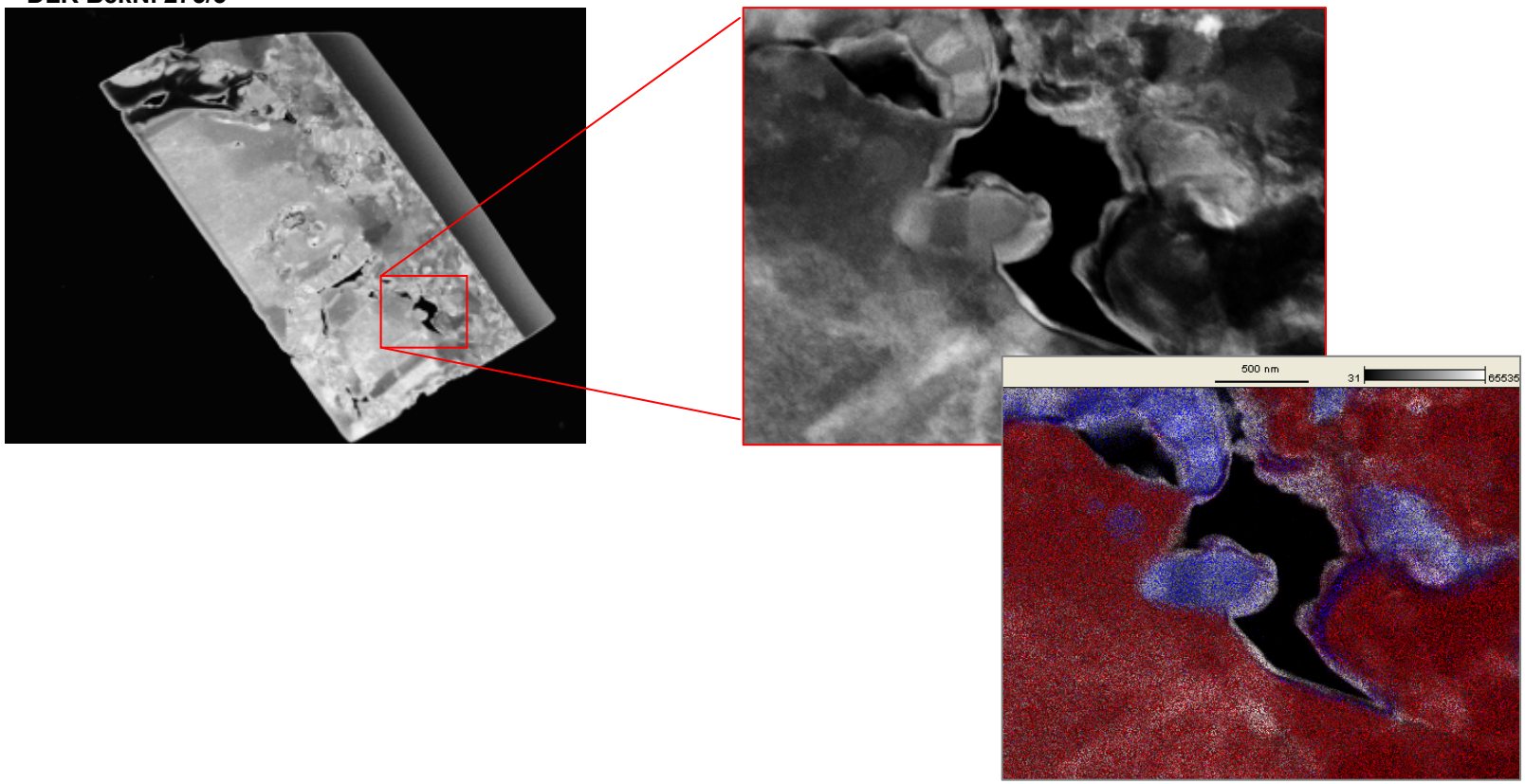






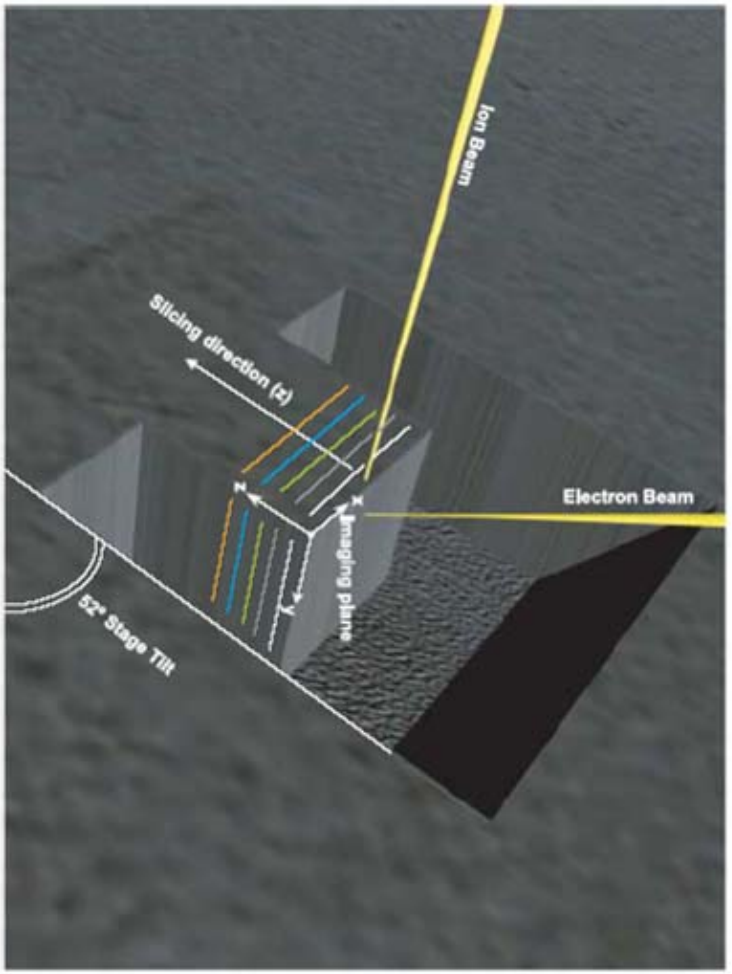
TEM: Imaging & elemental analysis

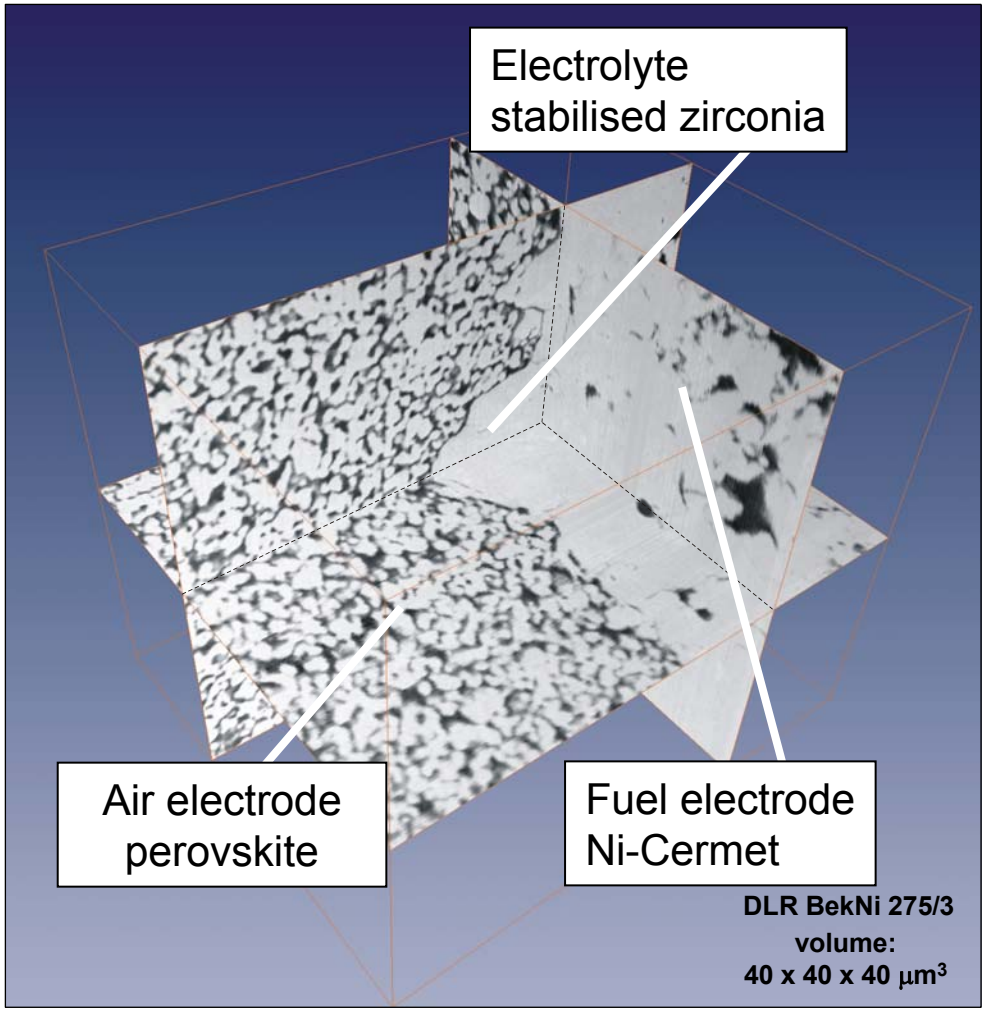
DLR BekNi 275/3



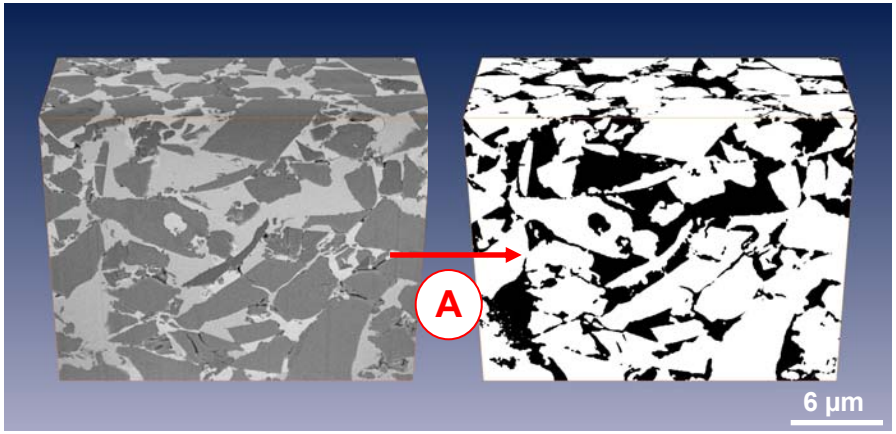
chemical map: Ni Zr

FIB-Nanotomography: 3-D structure of a FC



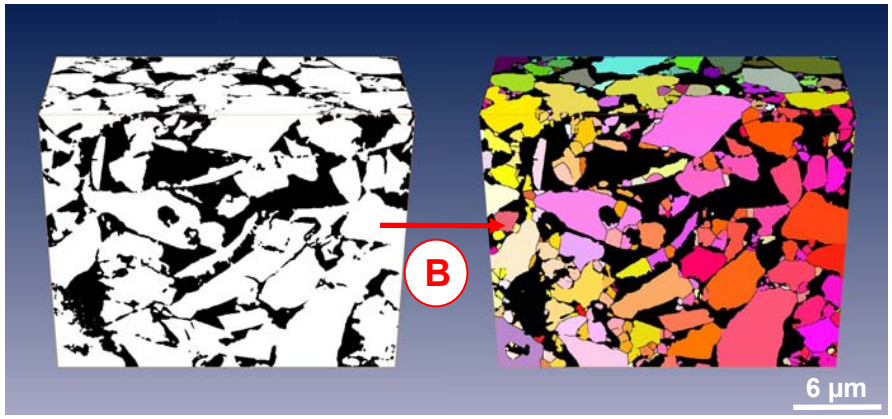


Nanotomography: Informationsgewinn



3D Imaging

Particulate und micro structure



Distinction

Crystallite-Particulate aggregates

Use of Information:
Modeling
Understanding degradation
→e.g. sulphur poisoning

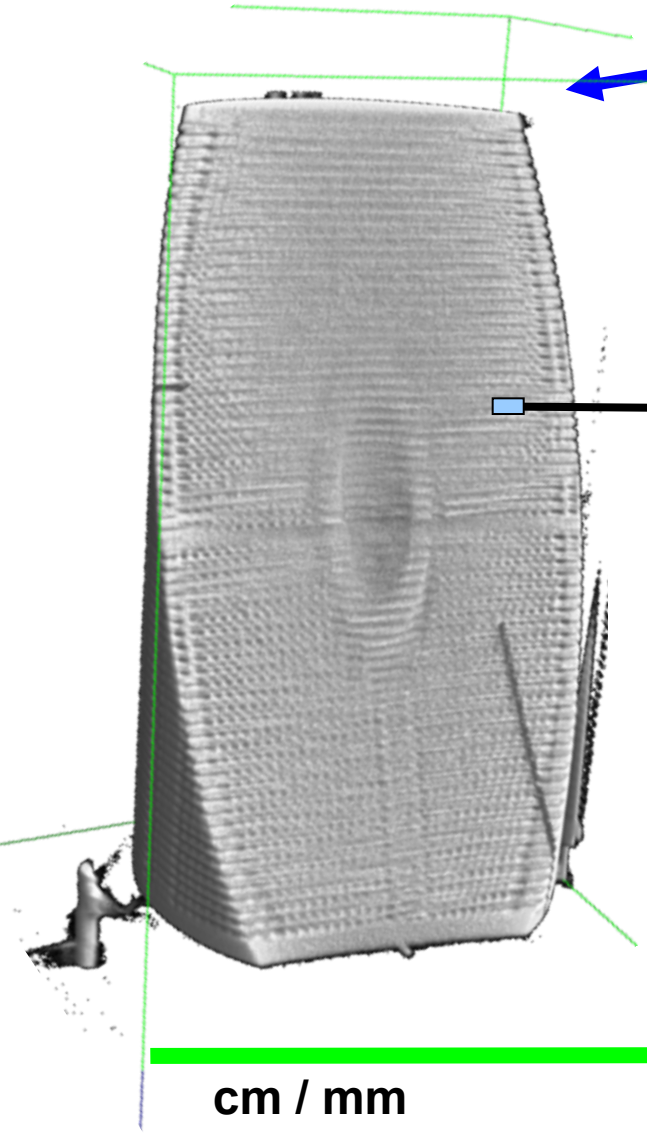
Advanced characterisation

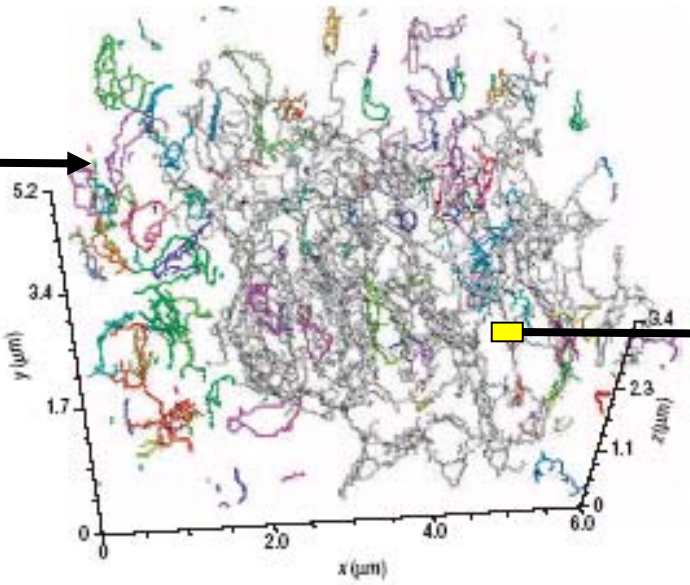
EMPA
Materials Science & Technology

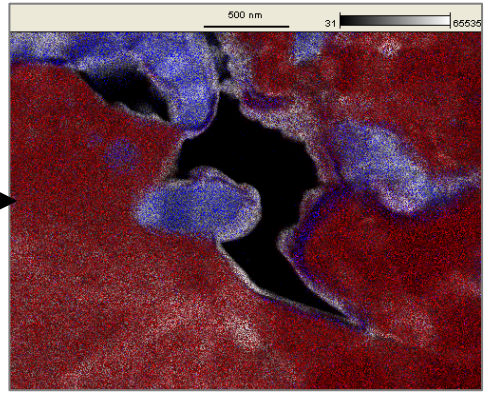
access to
large scale facilities

software

sample preparation







Source: J.R. Wilson et al.,
Nature Materials 5 541-544, 2006

cm / mm → **micro meter** → **nano meter**

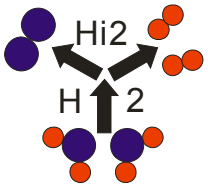
NdT methods and diagnostics strategy

- Visual testing (VT), including visible light microscopy
  - will be done in any case
- Ultrasonic testing (UT)
  - requires flat surfaces and low damping, 3D possibility
- Eddy current testing (ET)
  - for electrical conductors only
- Magnetic testing (MT)
  - for ferritic materials only
- Thermography (TT)
  - best for close to surface items, 3D possibility
- Radiographic testing (RT, CT, XTM)
  - No requirements to surfaces and damping, 3D possibility

- Quality assurance
  - cell production
  - stacking
- Life time / Durability
  - comparison of pre and post test state by NdT possible
  - failures affecting mechanical properties visible by NdT
  - Combination of NdT advantageous
  - identification of points for destructive analysis
- 3D imaging → real structures
  → validation of 2D analysis
  (e.g. SEM, OM)

Acknowledgement

- Defne Bayraktar, EMPA
- Ulrich Vogt, EMPA
- Thomas Graule, EMPA
- Günter Schiller, DLR
- SINQ, PSI
- Josef Sfeir, Hexis
- HTceramix



4. A possible NdT procedure for cells

Impulse thermography overview (FOV = 80 x 80 mm)
Pixel size 0.33 mm, meas. time per cell ≈ 2 min

Impulse thermography close up (FOV = 8 x 8 mm)
Pixel size 33 µm , meas. time per cell ≈ 10 min (automated)

Radiography overview (FOV = 80 x 80 mm)
Pixel size 20 µm , meas. time per cell ≈ 5 min

Radiography close up (FOV = 8 x 8 mm)
Pixel size 4 µm (OVHM - Movie) , meas. time per item ≈ 10 min (auto)

Local tomography (FOV = 8 x 8 mm) , meas. time per item ≈ 60 min
voxel size 4 µm or calculation of items depth by
evaluating the trajectories from OVHM -Movie