Novel lab based CT for Hierachical Framework Multiscale Imaging for Geomaterials

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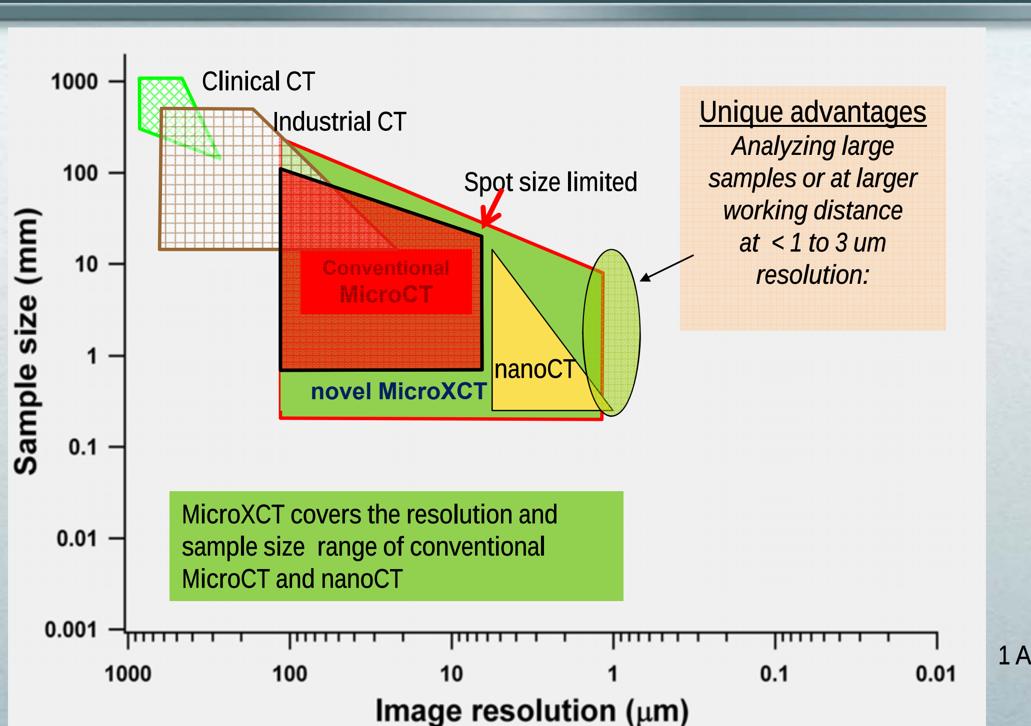
Outline

1. Concept of Hierachical Framework/Multiscale imaging
2. X-ray imaging principles overview
3. Applications : for lab based submicron MicroXCT and nanoXCT
   - Soft materials, composites
   - Geomaterials
   - Multiscale examples

Overview : Different CTs as function sample size & resolution



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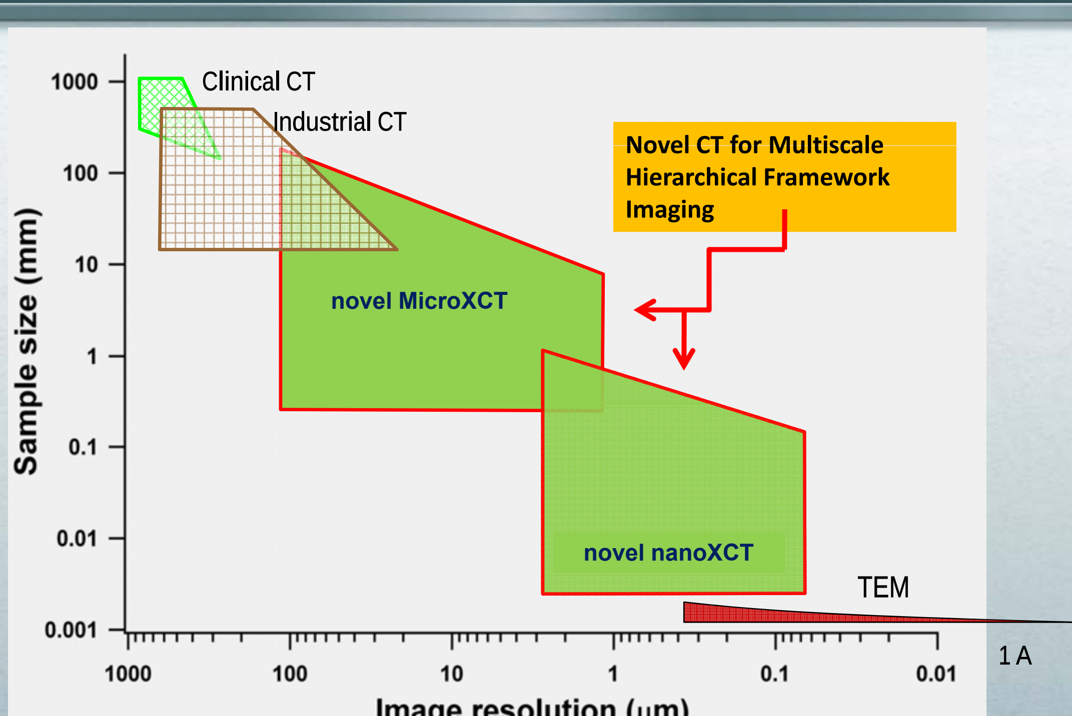
Different CTs as function sample size and resolution



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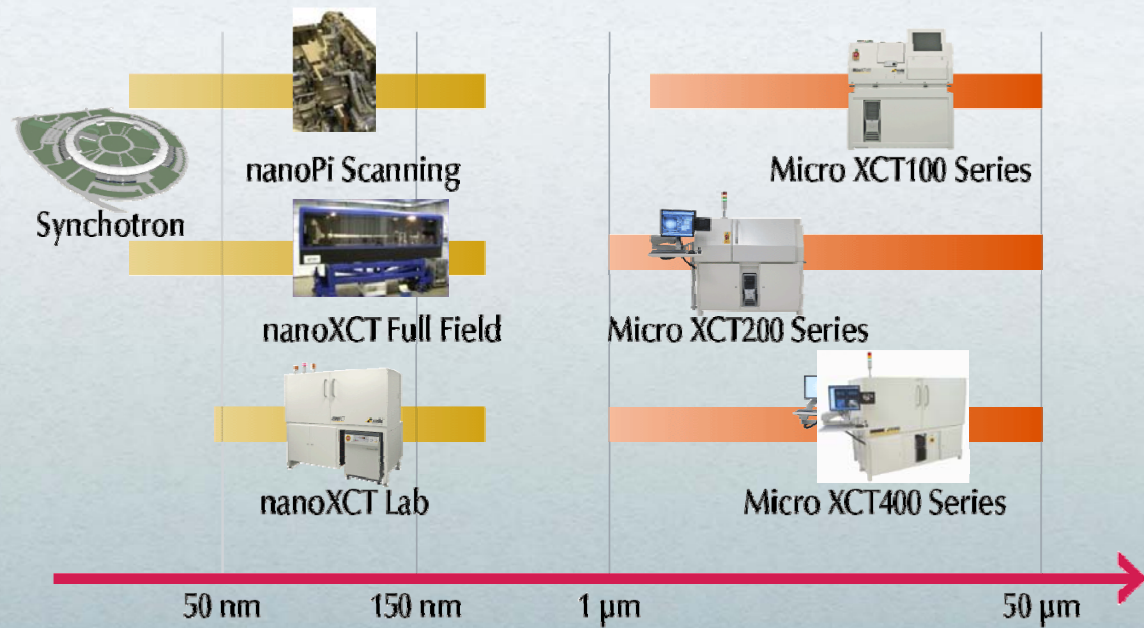
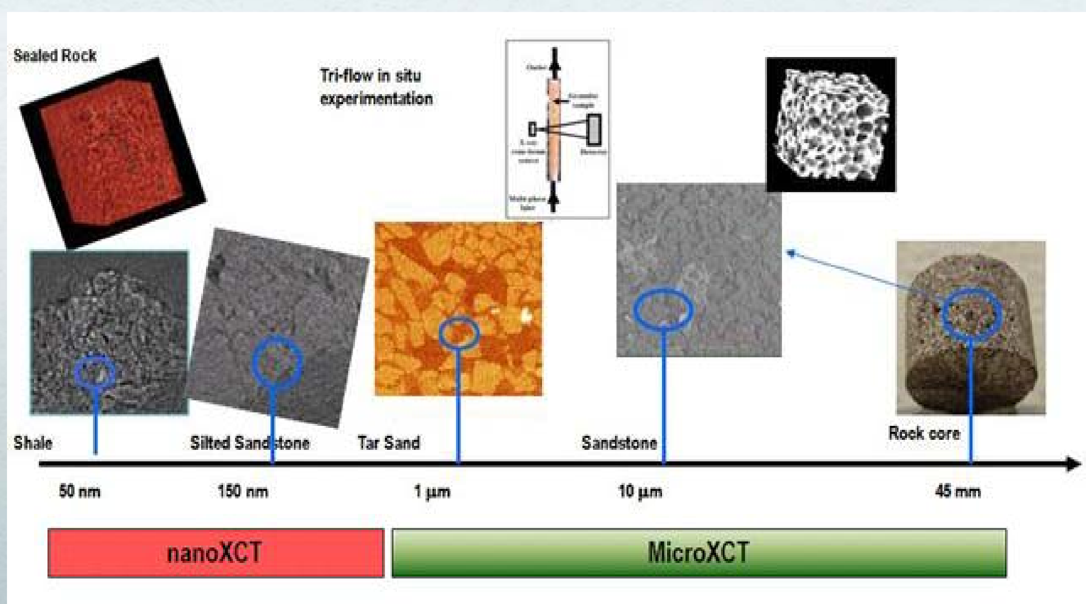
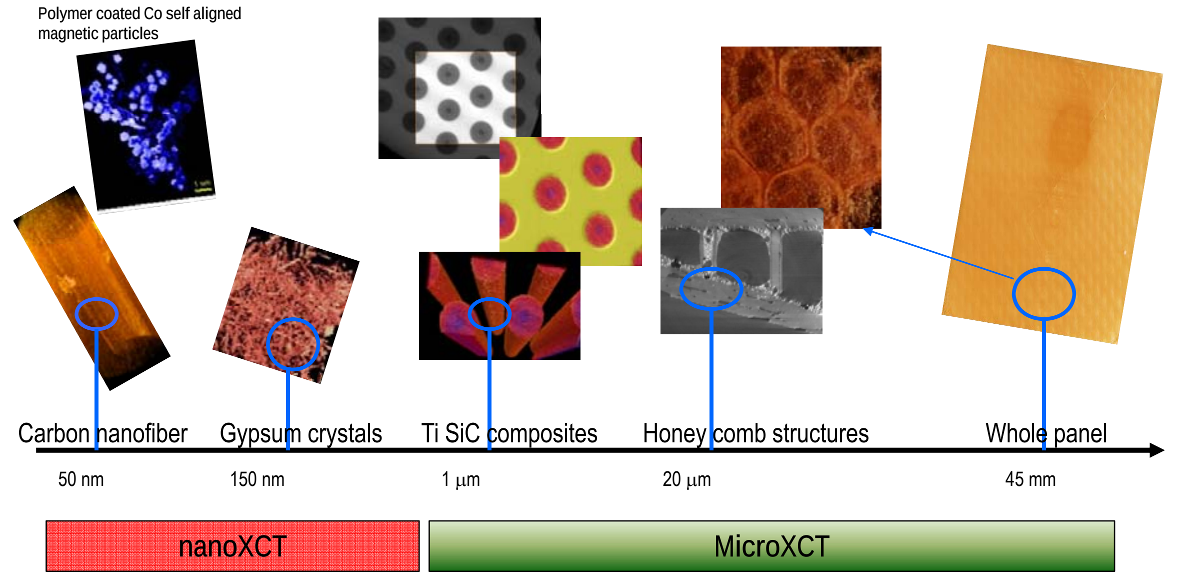
Xradia Solution – from Synchrotron to laboratory



Multi-Length Scale Capabilities : Oil and Gas



Xradia Multi-Length Scale CT Solution



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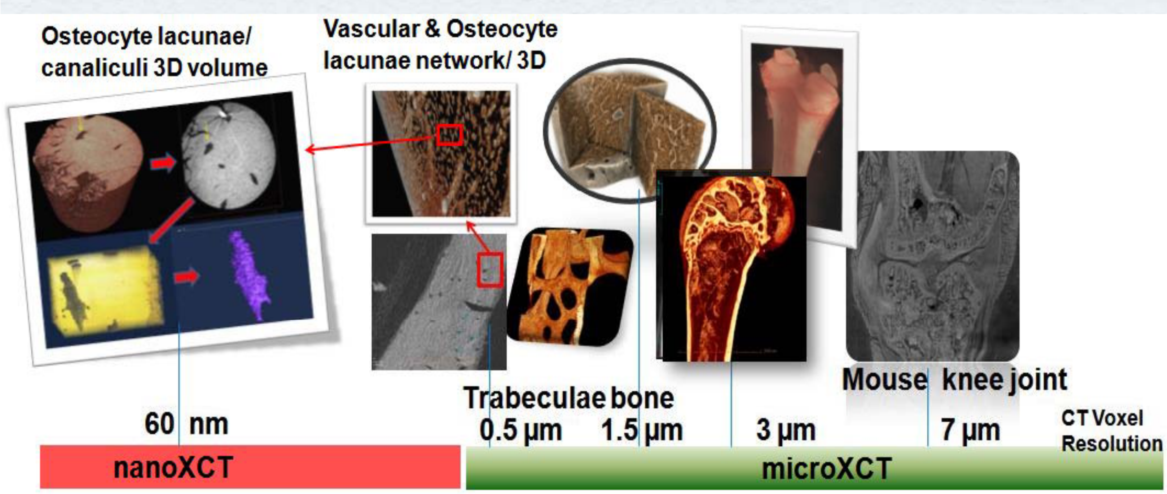
Hierarchical Framework Multiscale Bioimaging: From whole mouse knee to Osteocyte Lacunae/Canaliculi



Lau et al; ESB 2009

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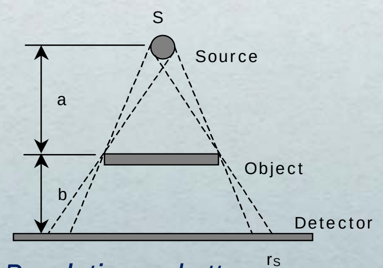
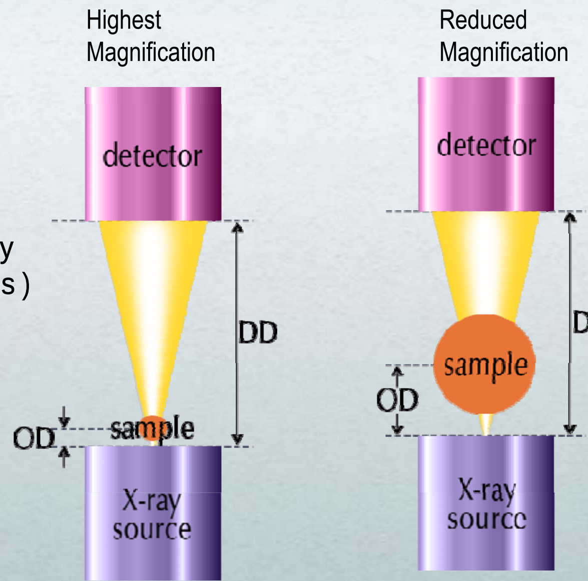
Overview: Conventional X-Ray Imaging (Projection Technique): Resolution/Spot Size/Sample Size Tradeoffs

- Highest Geometric Magnification Requires:
  - Sample must be very close to source and be small
  - X-ray spot size needs to be very small (Microfocus -> nanofocus)





Resolution no better than spot size

$$\text{Magnification, } M = \frac{\text{DD (detector distance)}}{\text{OD (object distance)}}$$

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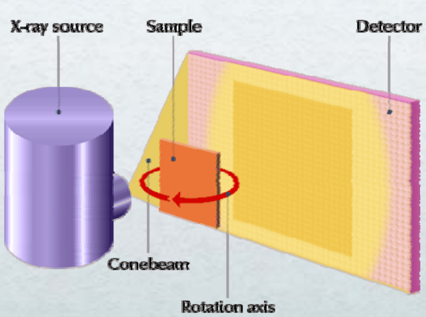
Unique *PhaseEnhanced* Detector Technology: Makes all the differences for range from 50 um to 0.5 um

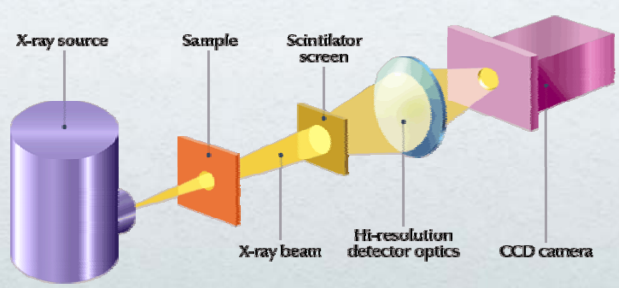
Resolution: Industrial CT (10 µm to mm) MicroCT (5 µm) ; nanoCT (0.5 µm)

Resolution : MicroXCT: Variable to Sub-µm



Projection based Micro CT or nanoCT

- 50 to 127 µm pixels at detector
- Resolution limited to *source spot size, sample size, working distance*
- Contrast is poor for soft materials- polymer, carbon composites



Novel MicroXCT- PhaseEnhanced Detector

- High Resolution detector variable to sub µm pixel
- Resolution not limited to source spot size, sample size or sample working distance
- *PhaseEnhanced* detector : High contrast for soft materials, polymers, hydro carbon

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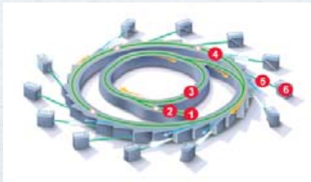
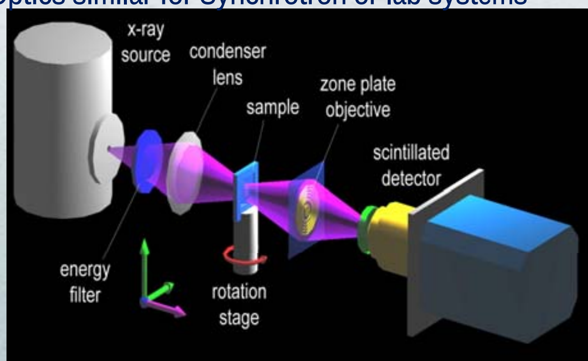
Nanotomography with lab x-ray source: nanoXCT (Resolution to 50 nm)

Nanotomography available in synchrotron



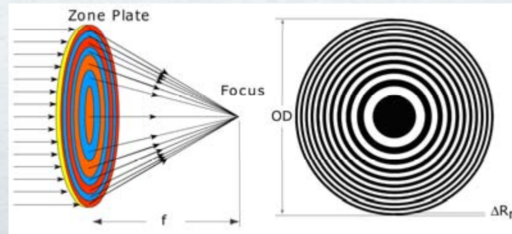
Optics similar for Synchrotron or lab systems



• 3-D resolution: <50nm

nanoXCT

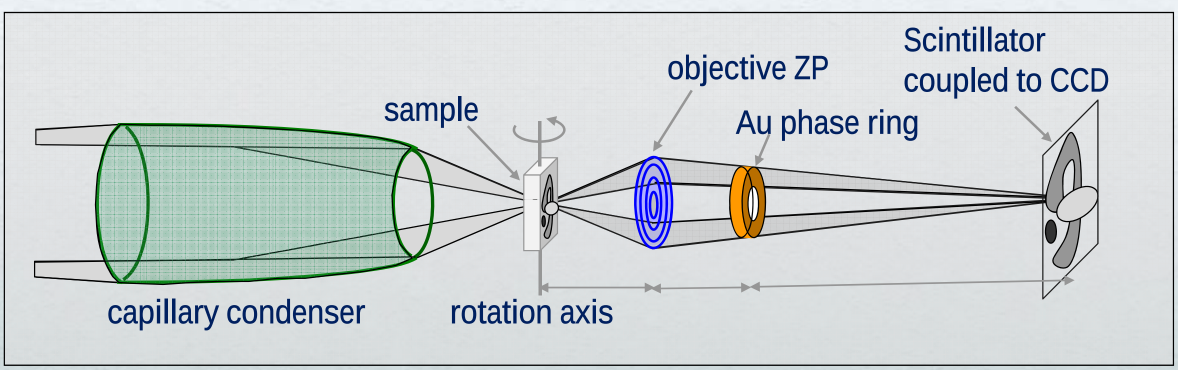
X-ray Optics: Fresnel Lens



$$f = \frac{OD\,\Delta R_N}{\lambda}$$

X-ray Lens Objective

X-ray Imaging with High-resolution Optics and Phase Contrast

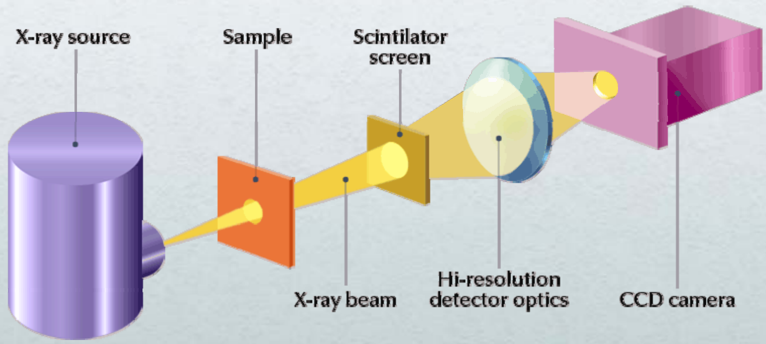


- Key components:
  - High efficiency, matched capillary condenser
  - High-resolution objective zone plate
  - Zernike phase contrast phase plate
  - High-efficiency, high resolution x-ray detector

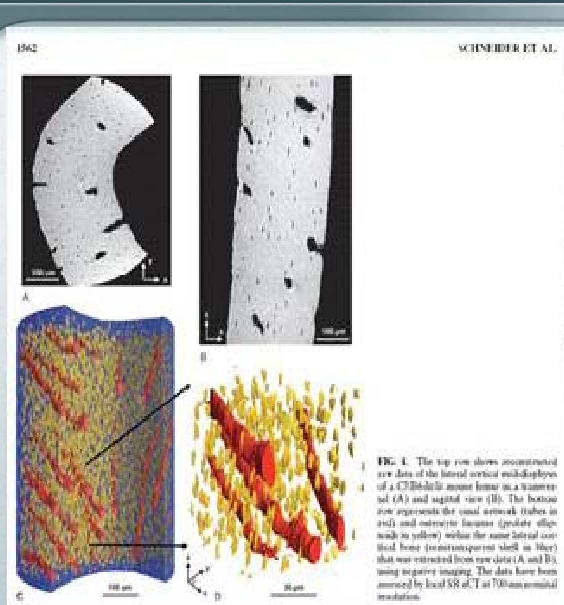
Power of Phase Enhanced Detector:
High Resolution with large samples

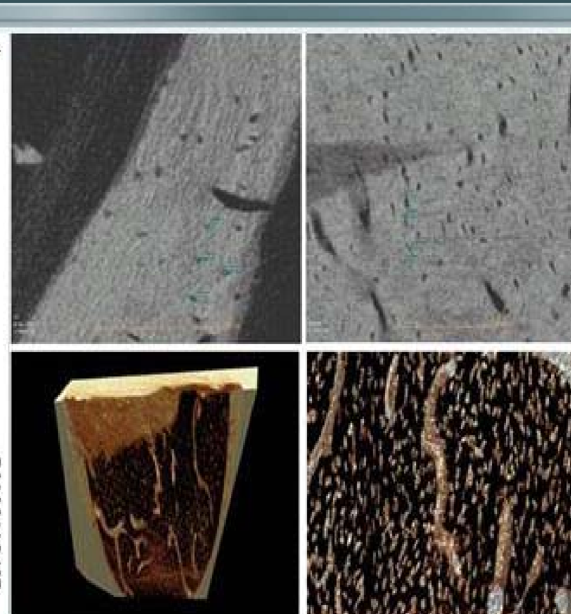
RESOLUTION

Submicron



Synchrotron nanotomography @ 700 nm resolution

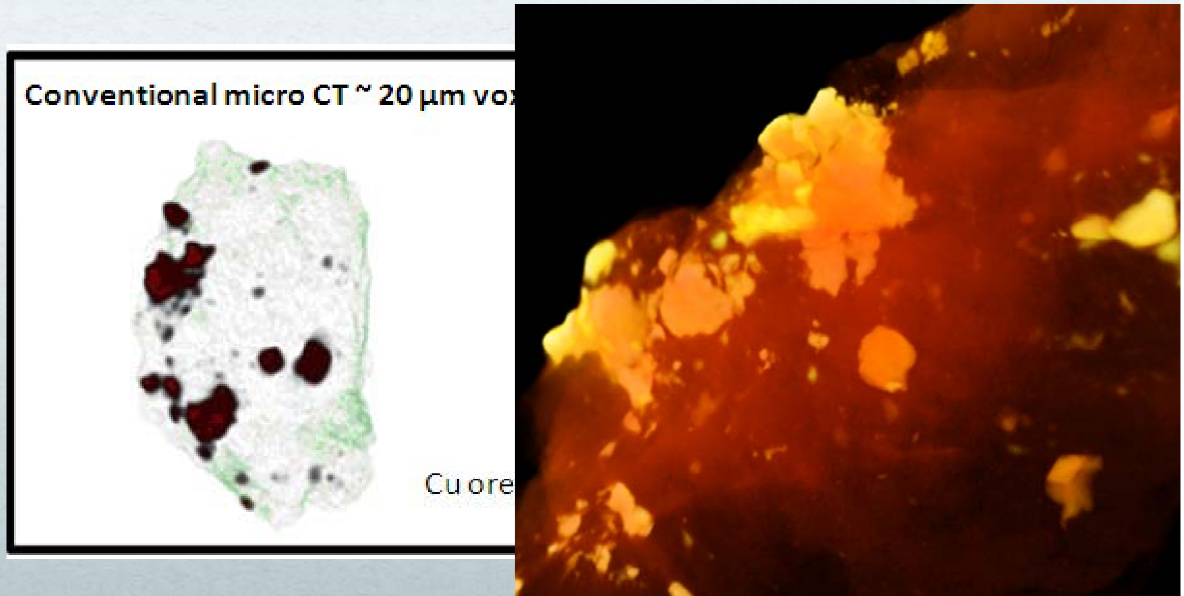




Osteocytes and vascular network in mouse bone
Swiss light source: *Schneider JBMR 2007*

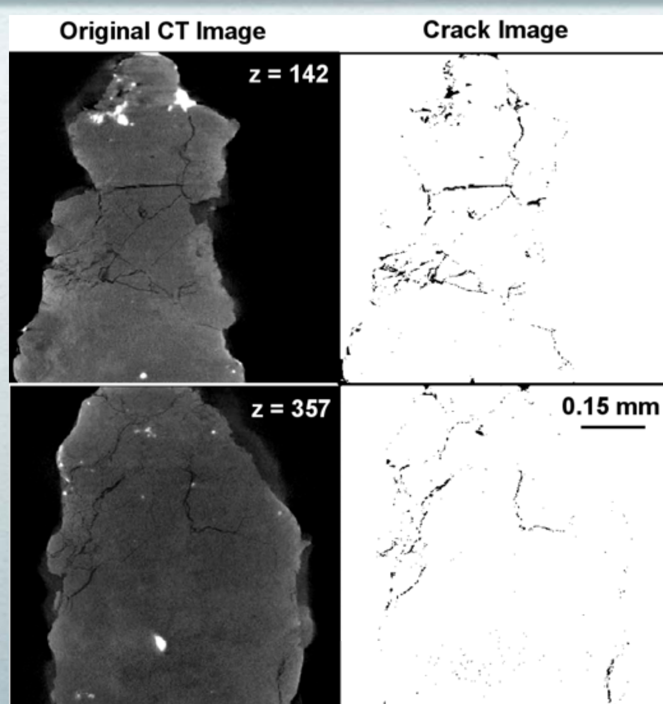
MicroXCT @ 500 nm resolution
Lau et al; ESB 2009

Copper Ore : Higher resolution makes big difference



Copper Ore – (1.70x0.85 mm)

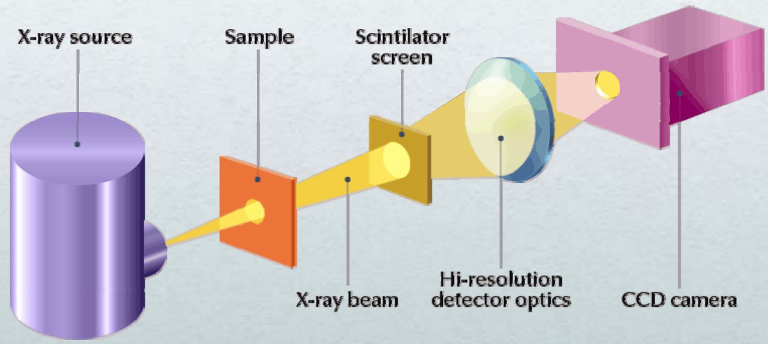
Isolation of Crack Surfaces with MicroXCT (Copper Ore , 1.70x0.85 mm)



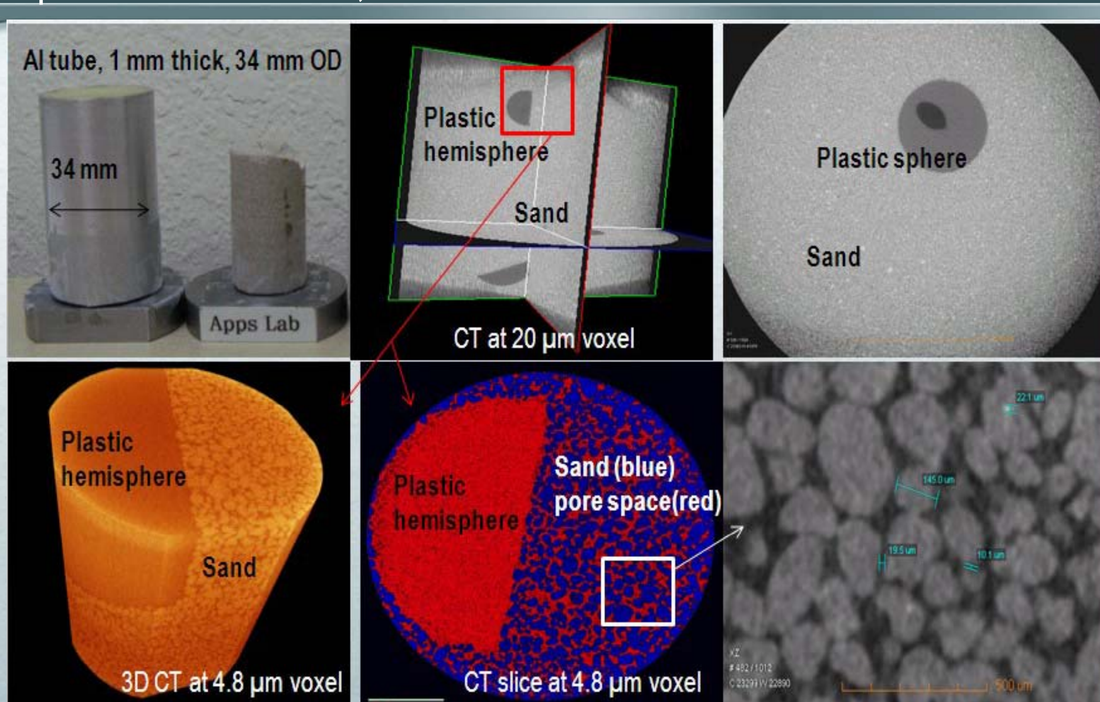
Power of Phase Enhanced Detector:
High Resolution with large samples

SAMPLE SIZE

Multiscale with High resolution

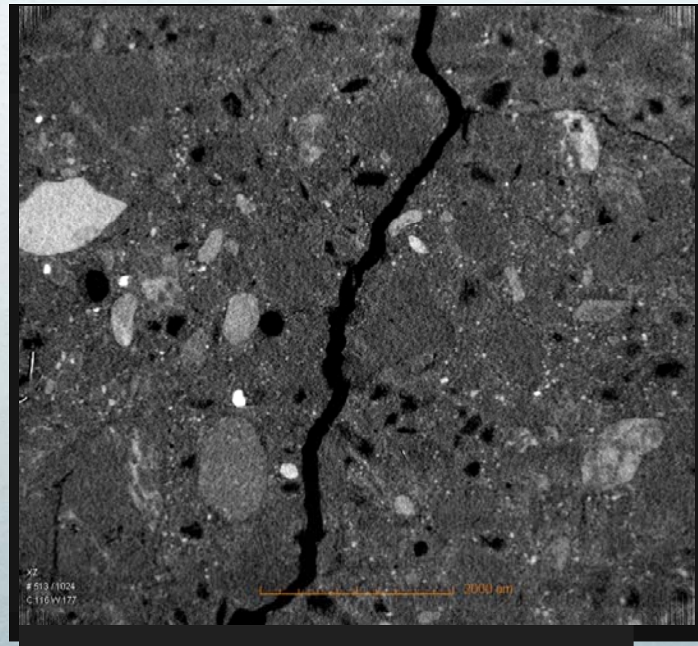


In situ Imaging: Visualization of granular media within pressurized vessel, 34 mm diameter at multiscale resolution

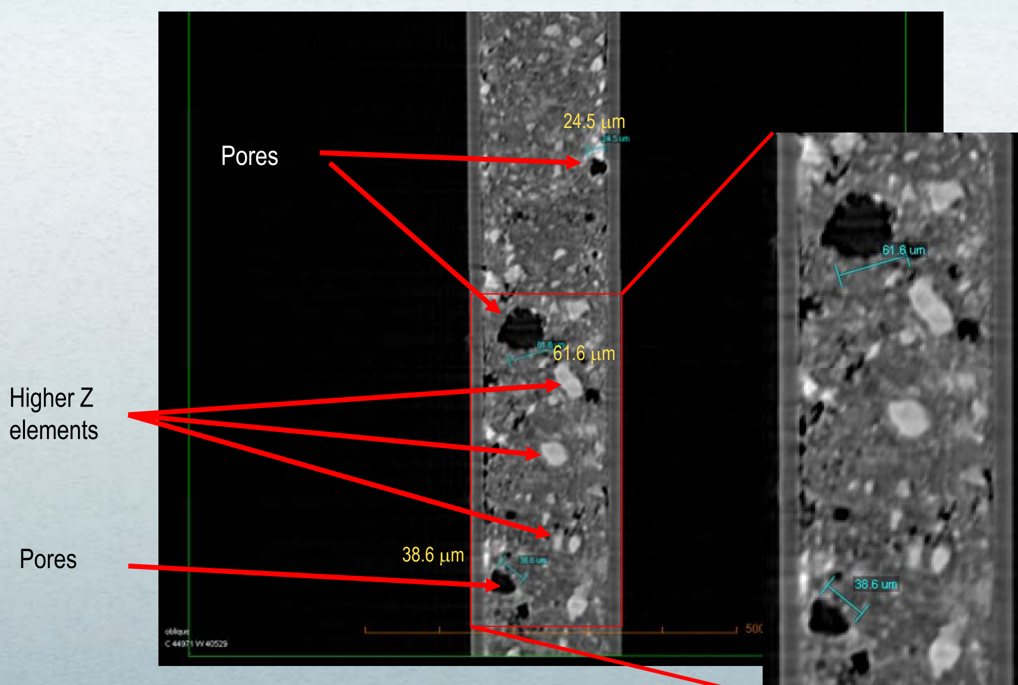


Lau et al, GeoX 2010

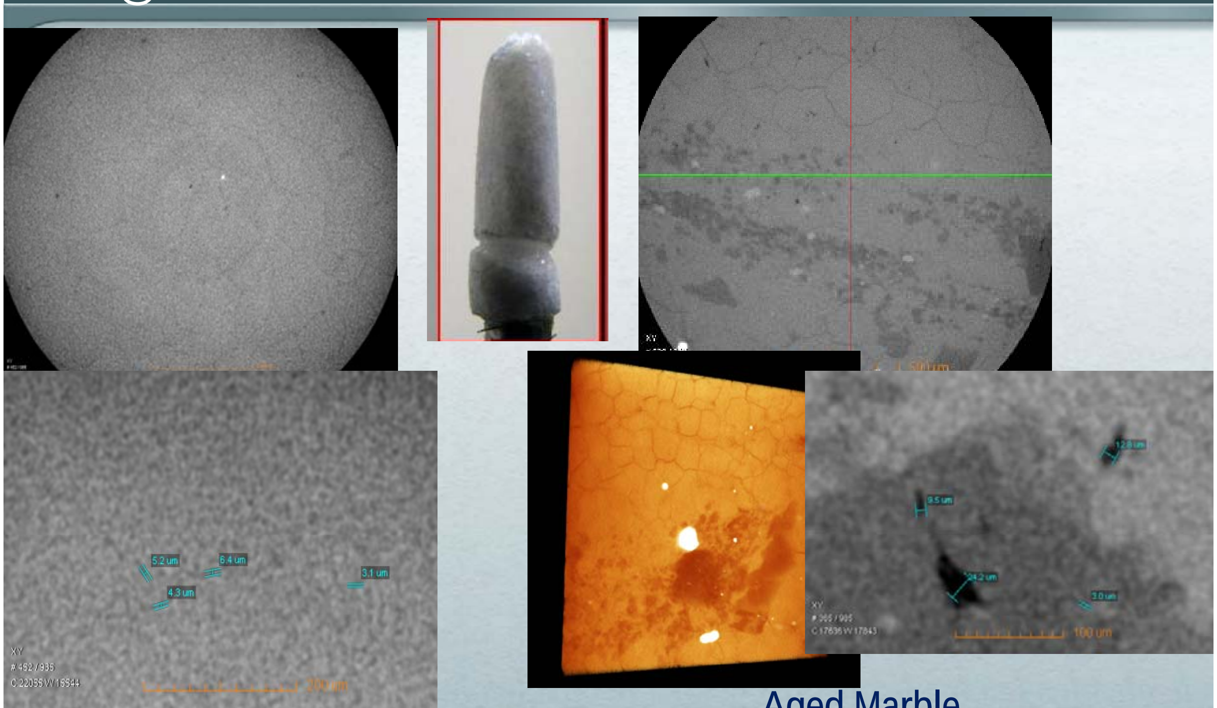
Imaging Cracks in 20 mm concrete post @ 7 um voxel



Cement : Void Characterization @ 0.7 μm pixel resolution



Comparison between fresh marble and aged marble @ 1.5 um voxel



Fresh Marble

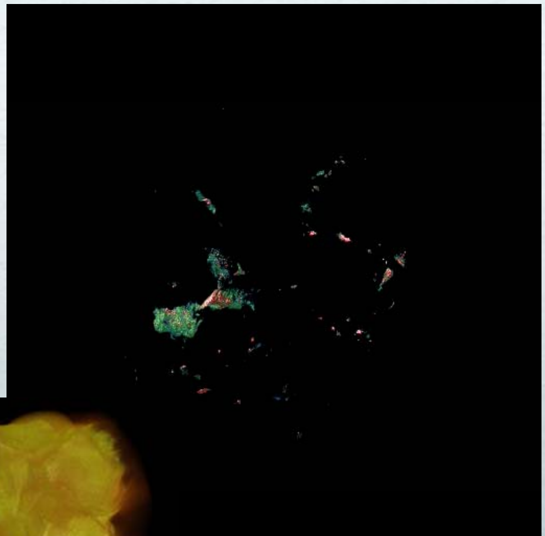
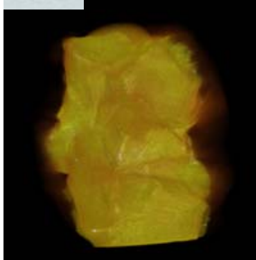
Aged Marble

Diamond – inner beauty or flaws?

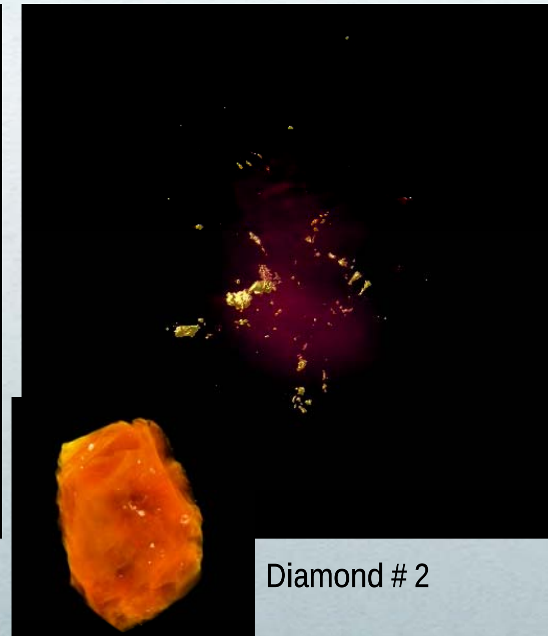
Diamond 1

Diamond 2

Diamond- every one has a fingerprint

Diamond # 1



Diamond # 2

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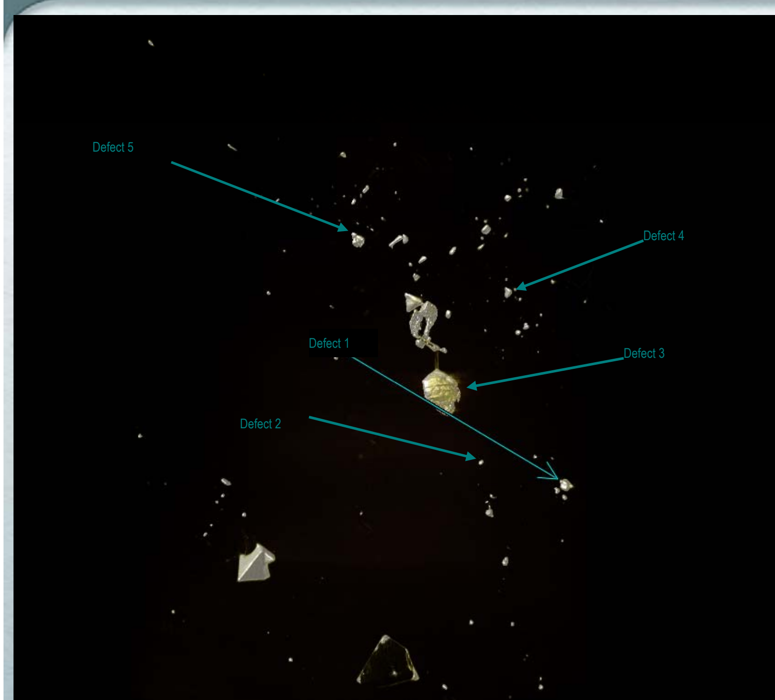
Defect Identification : Diamond 2



Defect	X (um)	Y (um)	Z (um)
1	1141	2267	733
2	1295	1873	802
3	1295	1687	1180
4	957	1950	1653
5	1205	1253	1930

By locating the centers of internal features and recording their relative positions a "fingerprint" of the diamond can be created.

Special filtering is used to threshold the 3D image and highlight the internal structures

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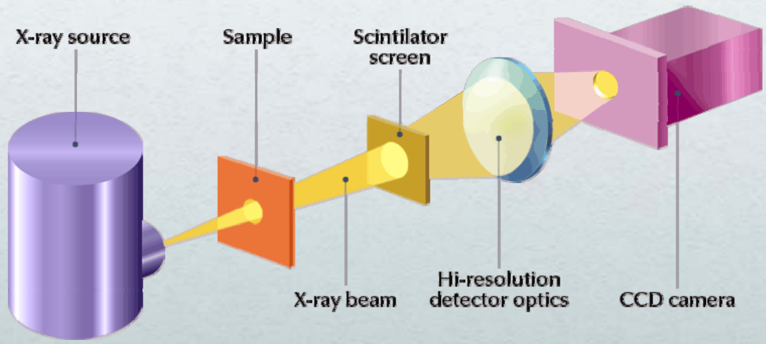
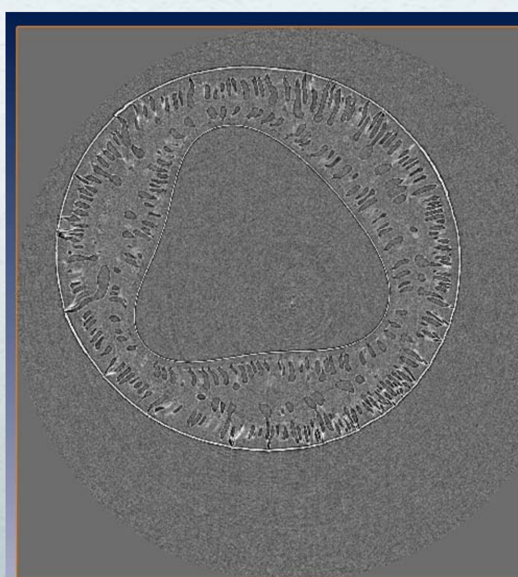
Power of Phase Enhanced Detector: Superior Contrast

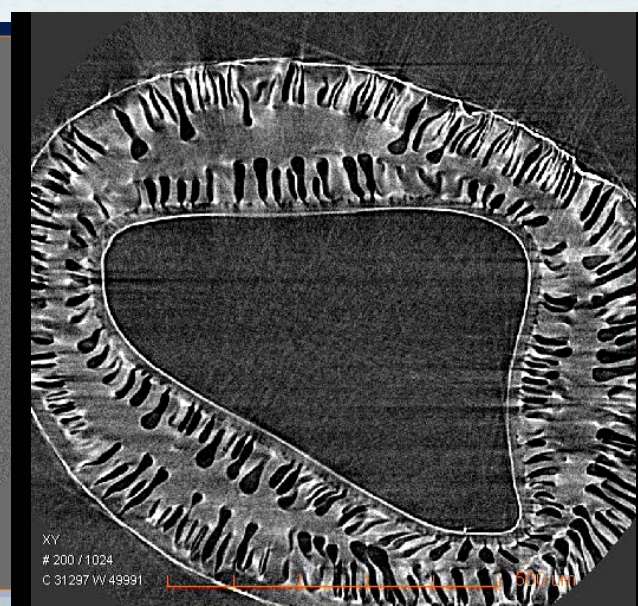
CONTRAST

Superb !



Environmental Filters: Synchrotron vs Lab MicroXCT





Synchrotron @ ESRF @ 0,7 um :

Sample courtesy: Jean-Christophe Rémigy, Laboratoire de Génie Chimique, Toulouse

MicroXCT @ 0.7 um resolution

Paleontology: Synchrotron Phase contrast X-ray tomography (holotomography) @ ESRF

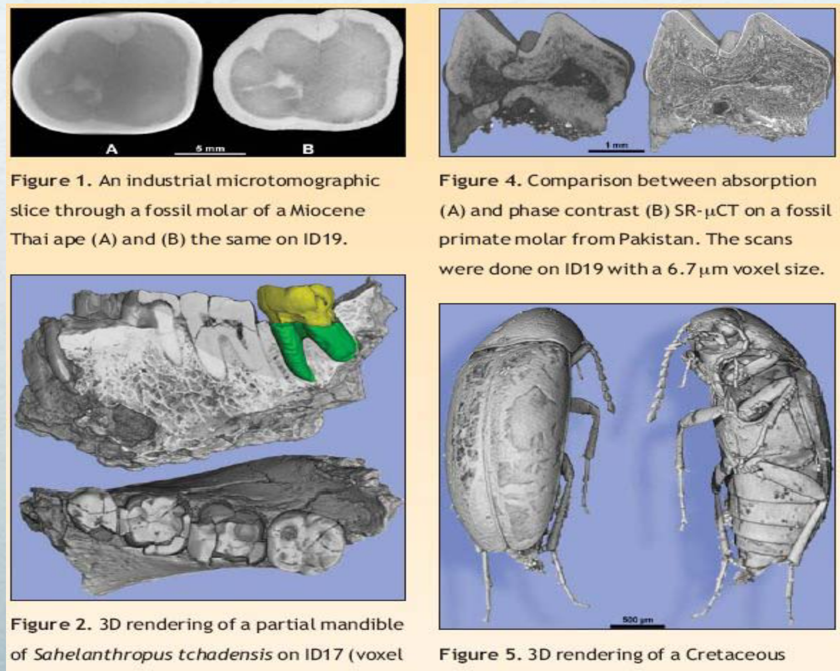


Figure 1. An industrial microtomographic slice through a fossil molar of a Miocene Thai ape (A) and (B) the same on ID19.

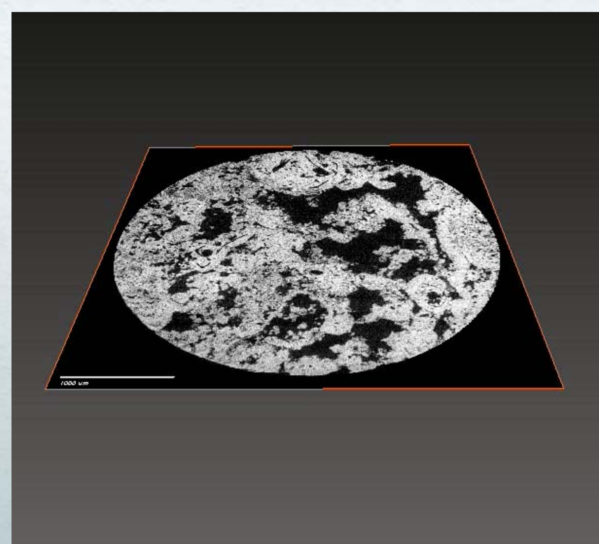
Figure 4. Comparison between absorption (A) and phase contrast (B) SR-μCT on a fossil primate molar from Pakistan. The scans were done on ID19 with a 6.7 μm voxel size.

Figure 2. 3D rendering of a partial mandible of *Sahelanthropus tchadensis* on ID17 (voxel

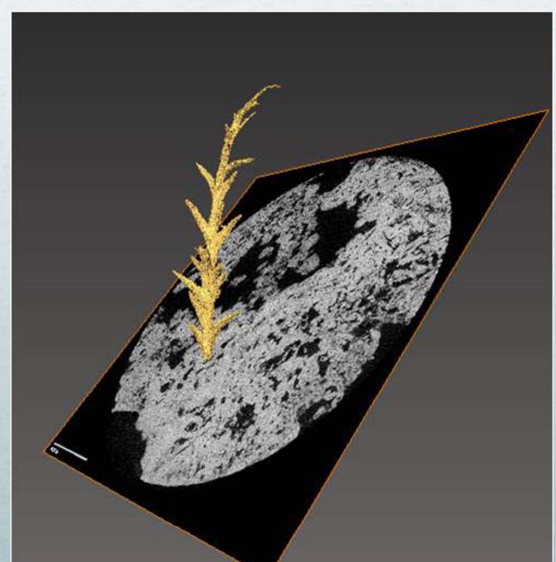
Figure 5. 3D rendering of a Cretaceous

ESRF Newsletter
2005 Vol 42

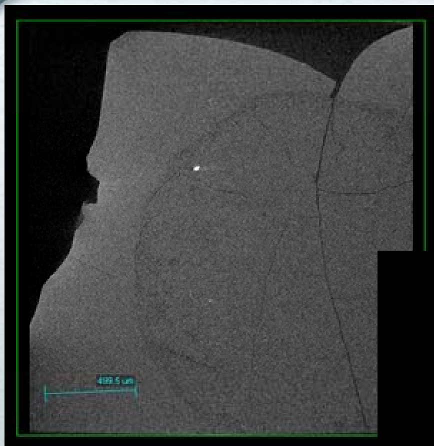
Fossils within Rock: Tribolite and Coral



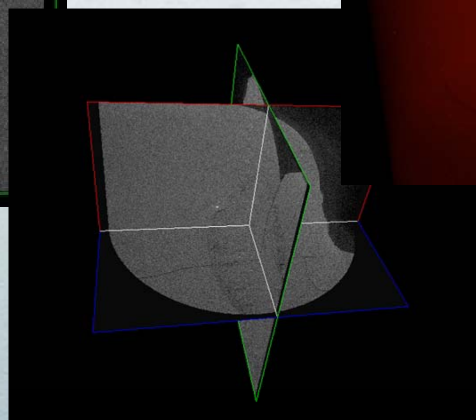
Carbonate Fossils uncovered in limestone; 10X; 90kV 6W, 721 images, 30s/image



Volcanic Rock: High resolution and contrast to detect phases & inclusions



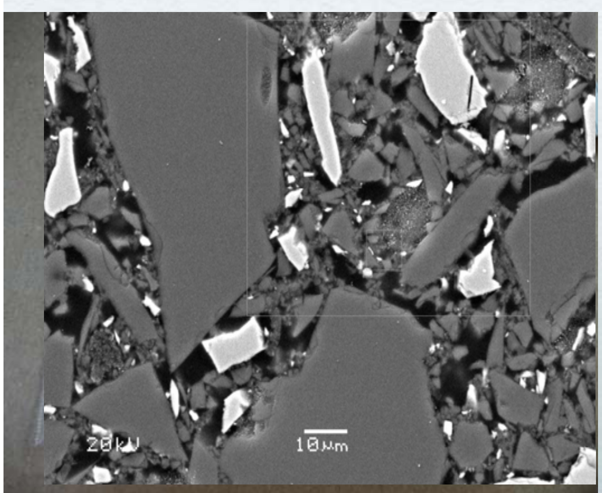
CT slice at 1.5 micro Resolution



3D of inclusion @ 0,75 micro Resolution

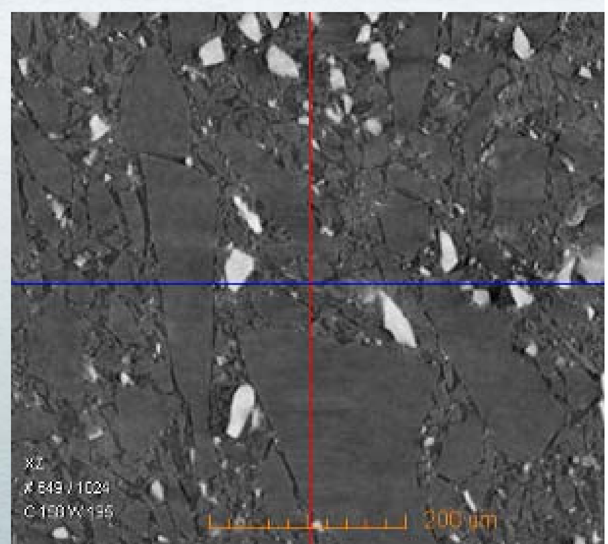
Zirconia doped Alumina: Non invasive Characterization of Multiphases after Sintering or Thermal Cycling

Comparison between SEM & novel MicroXCT



SEM image (2D)

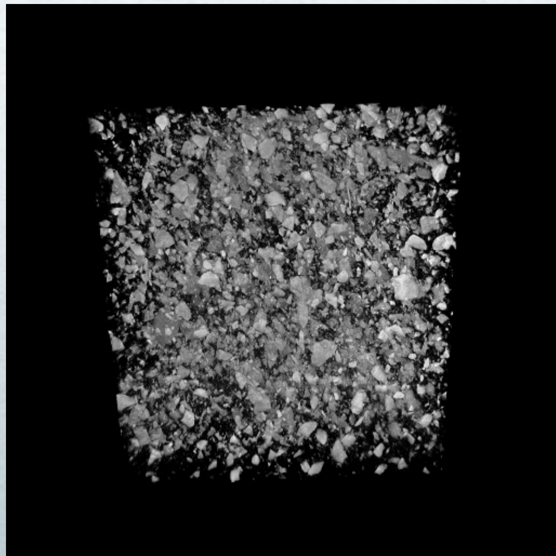
Zirconia doped Alumina refractory turbines casting



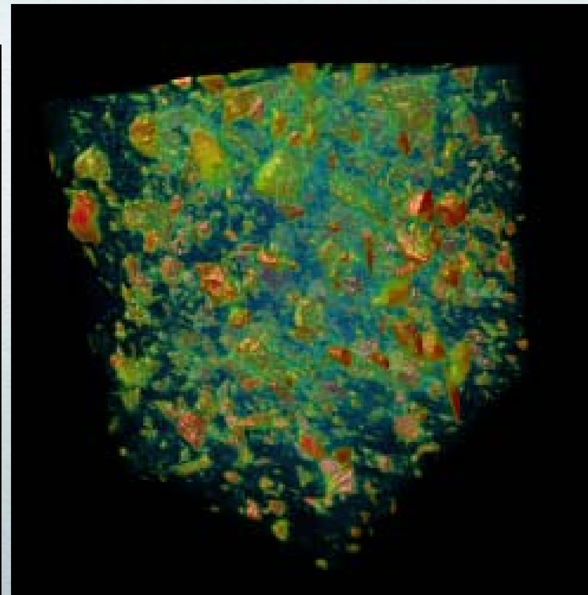
MicroXCT: CT slice @ 0,7 micron pixel resolution

S H Lau et al, Proceedings, CMCEE 2008

Zirconia doped Alumina: Non invasive 3D Characterization of Multiphases after Sintering or Thermal Cycling



MicroCT: Zirconia doped Alumina refractory @ 0,7 micron pixel resolution



S H Lau et al, Proceedings, CMCEE 2008

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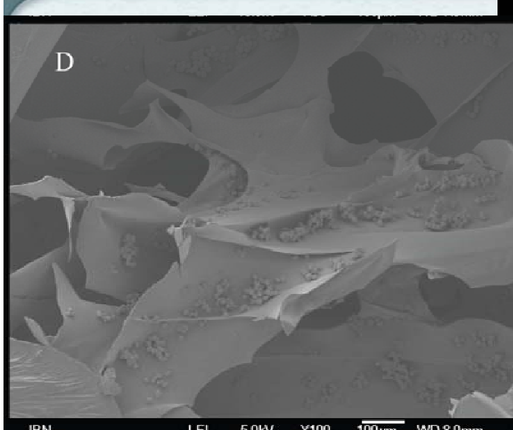
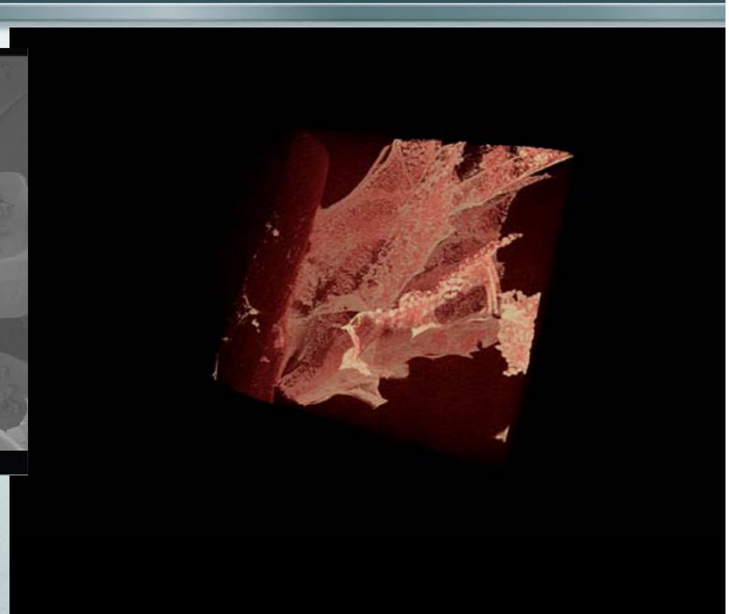
Liver : Polycaprolactone-collagen (PCL-collagen) cultured with rat hepatocytes



SEM: Cellular distribution on collagen not uniformed



CT images (@ 0.7 μm resolution) showed cellular distribution in 3D is highly uniformed

Lau et al., ICMAT 2007

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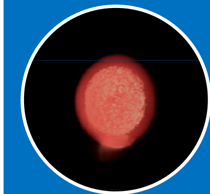
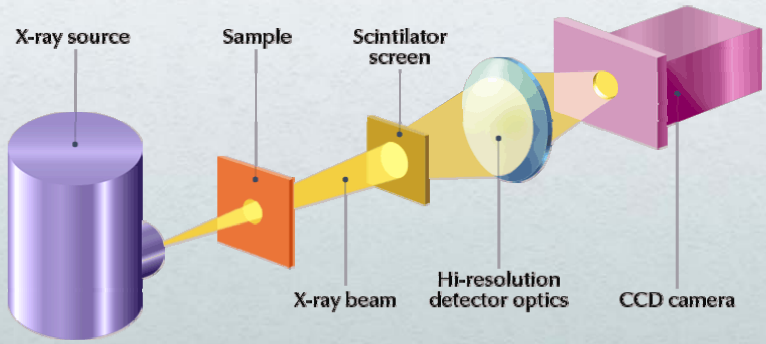
Power of Phase Enhanced Detector: Imaging Soft material in presence of high Z metals



LOW Z TO HIGH Z

Soft materials to metals



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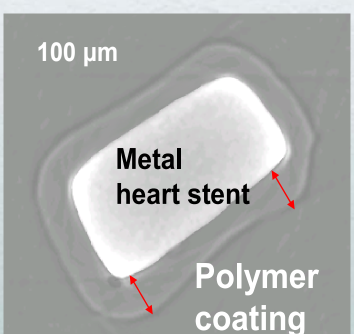
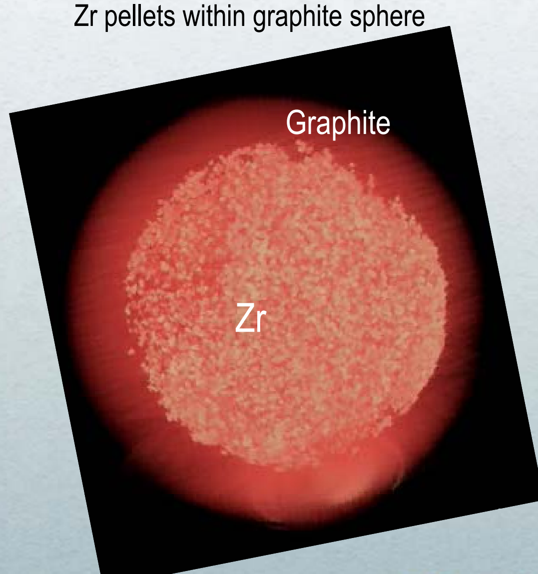
Xradia Contrast advantages: Imaging Low Z materials in matrix of high Z with PhaseEnhanced Technology

Power of PhaseEnhanced Detector

Polymer coated heart stent



Surrogate Nuclear Fuel
Zr pellets within graphite sphere



Low Z material in presence of high Z matrix

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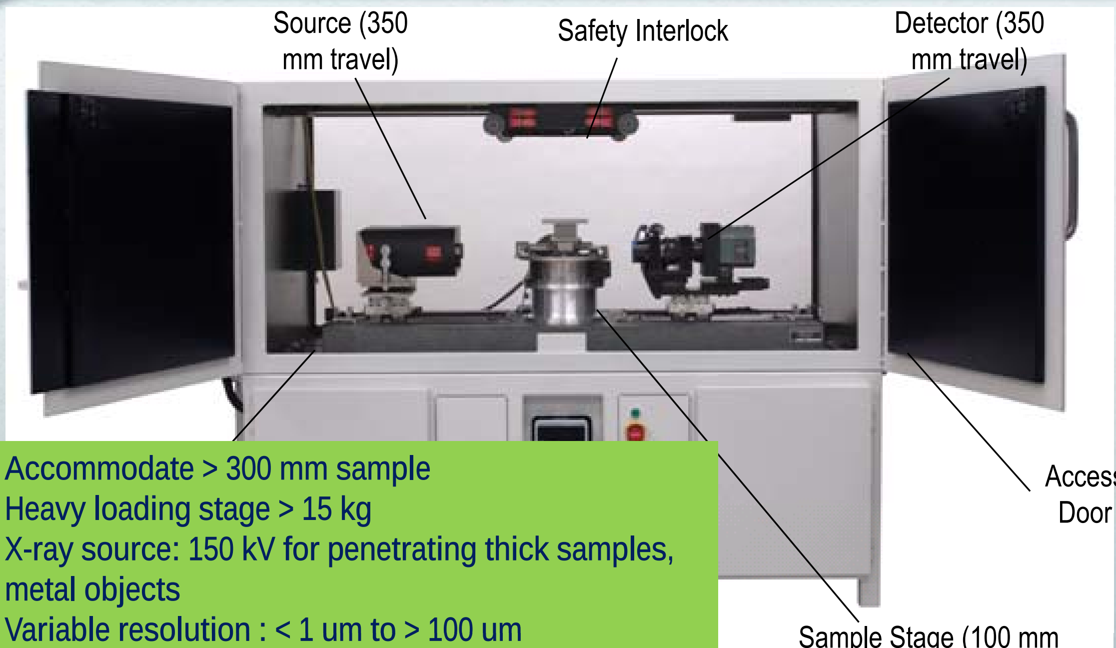
MicroXCT-400: large chamber for R+D



Accommodate > 300 mm sample
Heavy loading stage > 15 kg
X-ray source: 150 kV for penetrating thick samples, metal objects
Variable resolution : < 1 um to > 100 um

Modeling of flow mechanics, permeability, elastic moduli

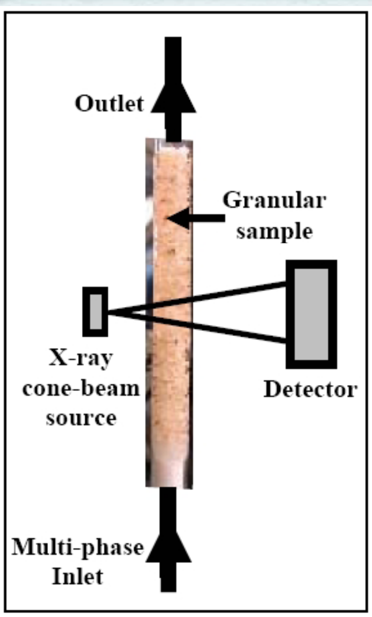
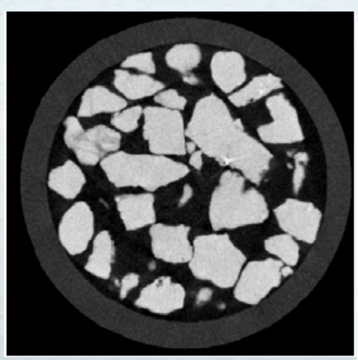


Figure 1: Experimental Schematic.



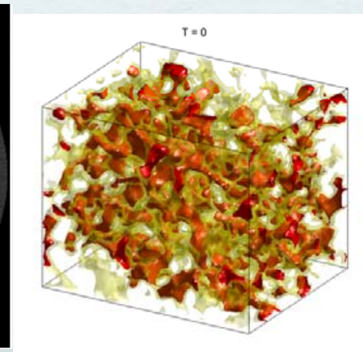
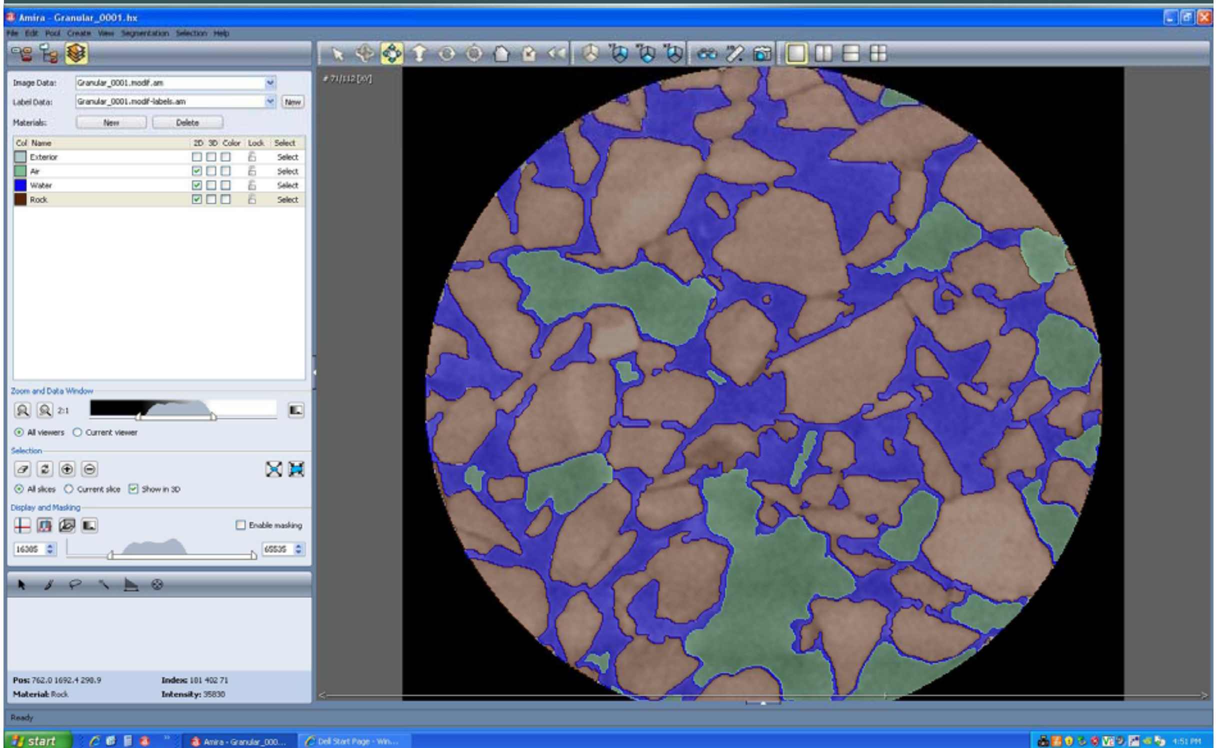


Table 1: Fluids Properties (Al-Wadahi, 1996).

Fluid	Density (g/cc)	Dynamic viscosity (cp)
Water	1.0377	1.1534
BA	1.0265	5.0563
Decane	0.7416	0.9659

Freddy E. Alvarado, Abraham S. Grader, Ozgen Karacan, Phillip M. Halleck.
The Pennsylvania State University-Energy Institute and Department of Energy and Geo-Environmental Engineering

Experiment: Visualizing air- water-composite interface



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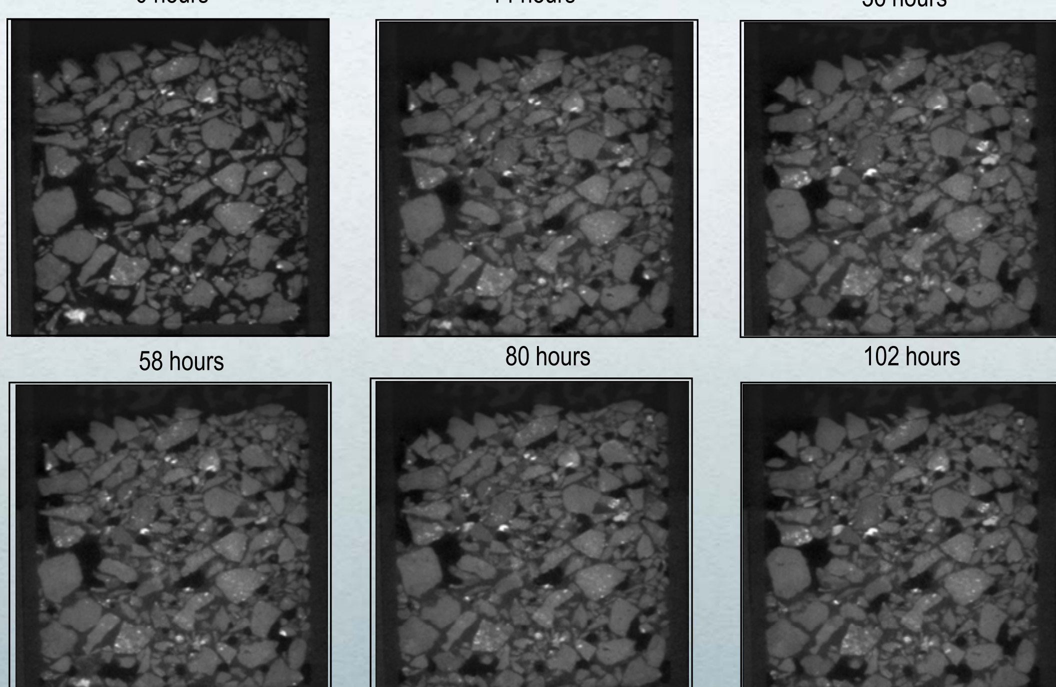
Mining Applications: Mini Column Leaching Testing

0 hours | 14 hours | 36 hours



58 hours | 80 hours | 102 hours

J Miller, C L Lin, Mineral Processing Plant Design 2009

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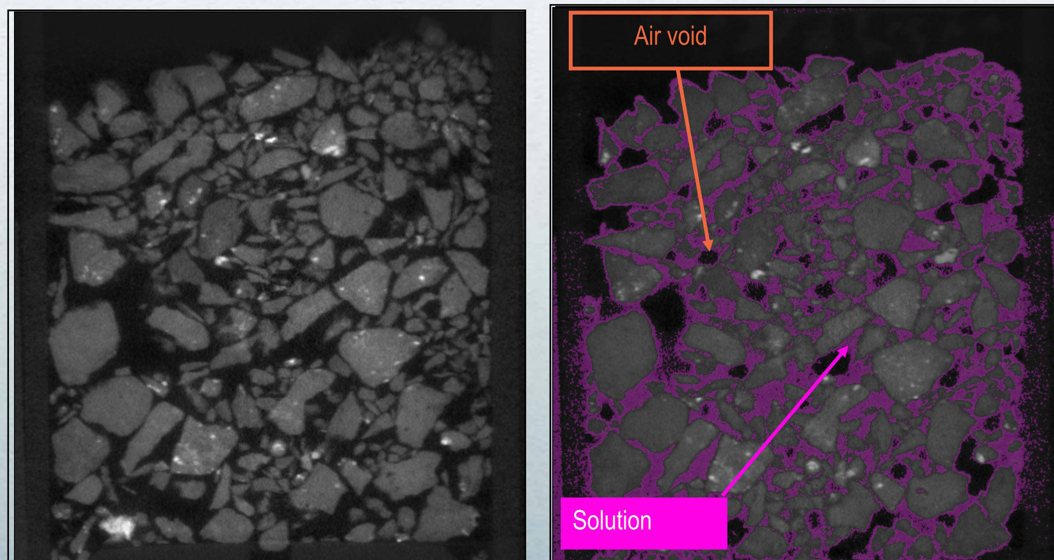
Mini Column Leaching Testing

0 hours

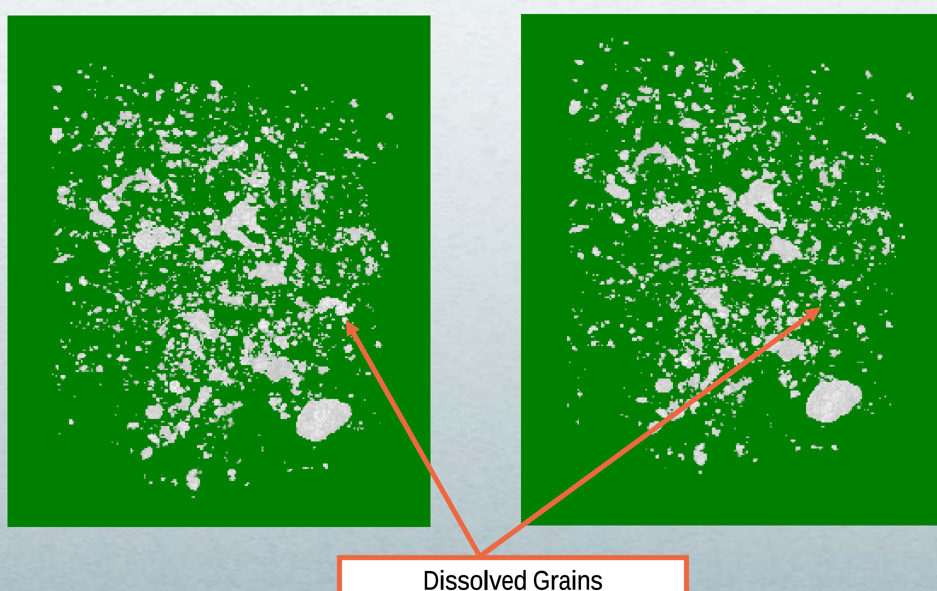
14 hours



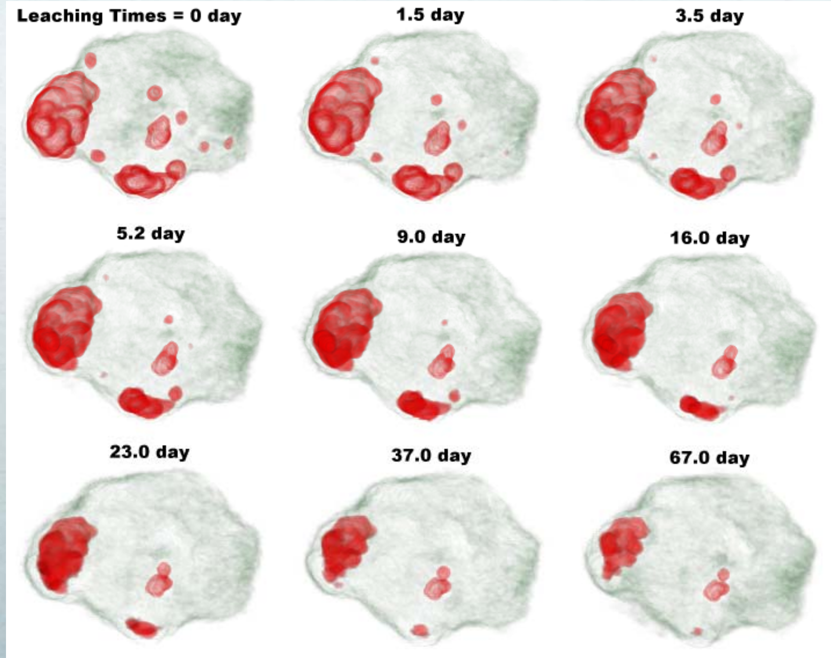
Mini Column Leaching Testing

1 Day

37 Days



Changes in High Density Mineral Grains



J Miller, C L Lin, Mineral Processing Plant Design 2009

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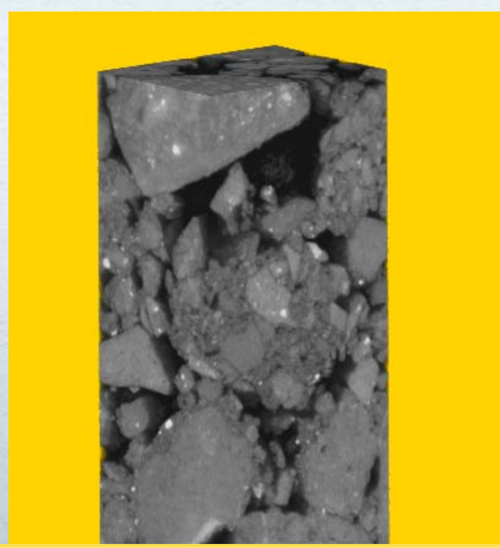
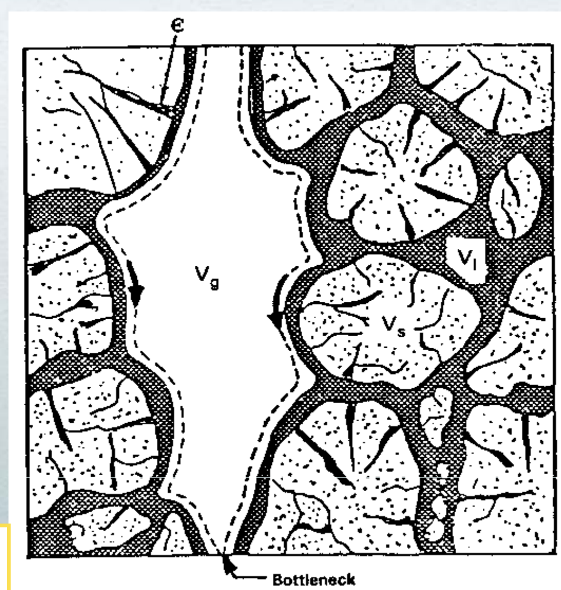
Pore Network Structure for Flow in Packed Particle Beds



- Rock space, V_s (~59%)
- Open porosity within rocks, ε (~3%)
- Solution space, V_l (~20%)
- Air void, V_g (~18%)



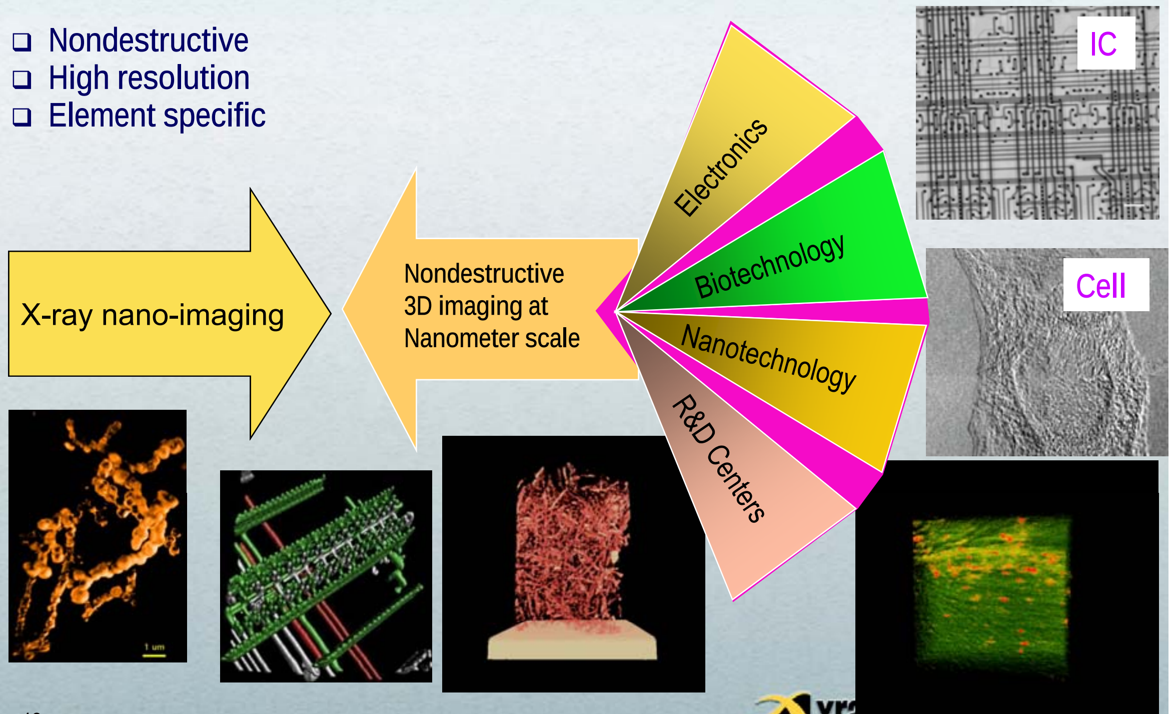
W.J. Schlitt (1984) and R. W. Barlett (1998)

J Miller, C L Lin, Mineral Processing Plant Design 2009

xradia insight in 3D

Nanoscience needs 3D nanoimaging (Seeing is believing): The X-ray imaging advantages

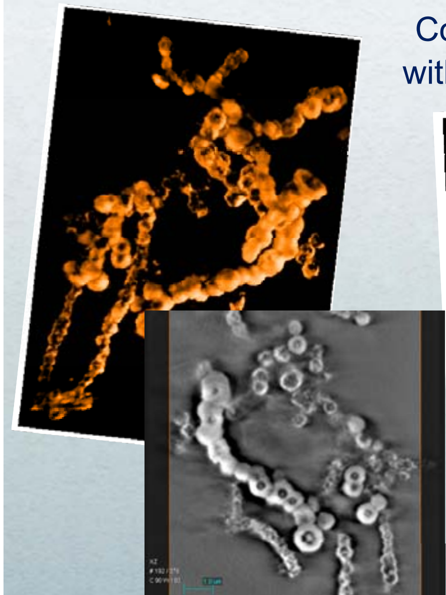
- Nondestructive
- High resolution
- Element specific



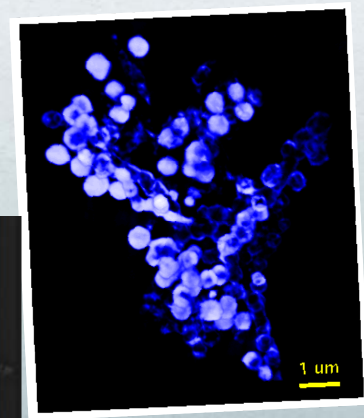
Nanoscale 3D Imaging

Nanotechnology and nano materials with nanoXCT

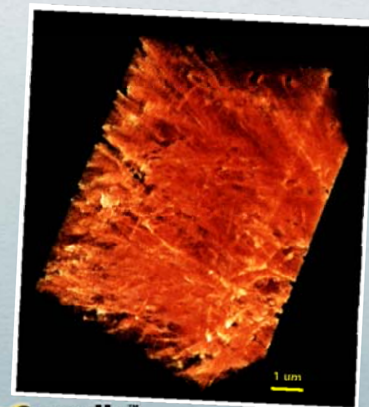
self assembled 0.2-0.8 µm magnetic particles



Cobalt spheres covered with polymer

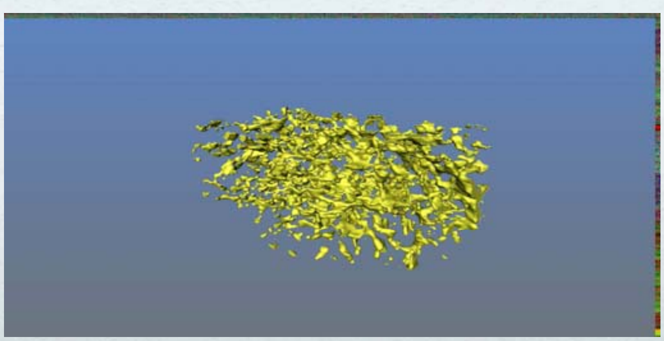


polycarbonate



Sample courtesy Ziyu Wu, BSRF Beijing, China

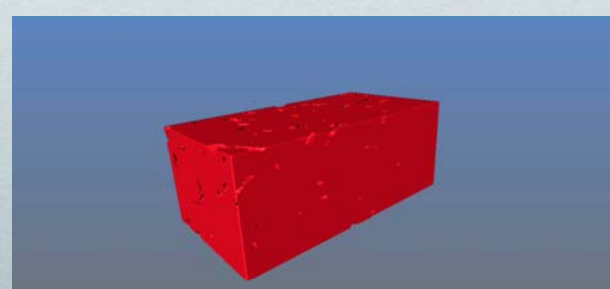
SOFC. Porosity and Material Phase Characterization with nanoXCT at 60 nm voxel



Solid Phase structure
of Ni-YSZ anode

Dimensions ~ 11 x 5 x 5 µm

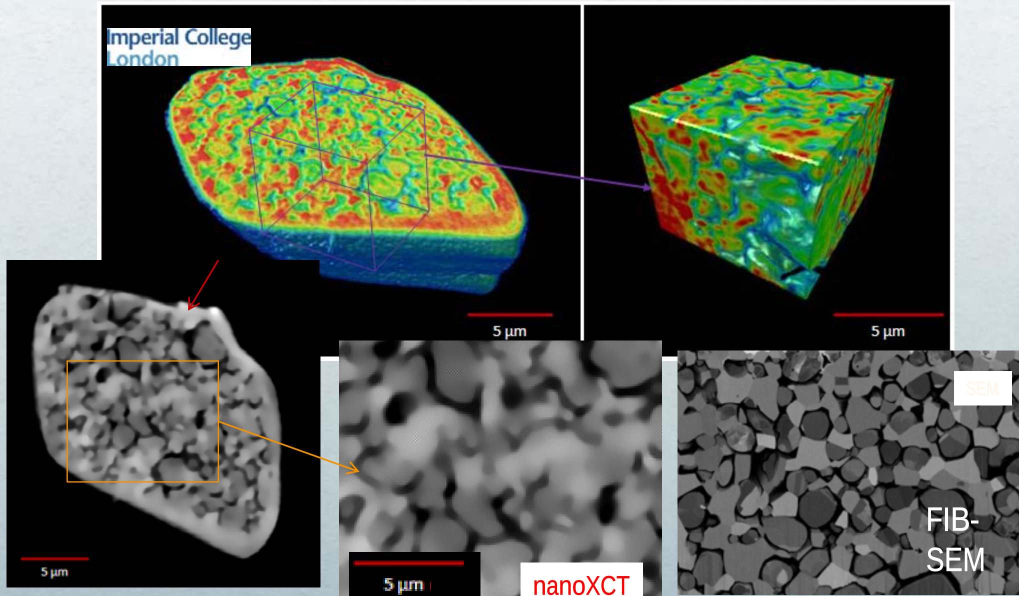
Pore Structure
of Ni-YSZ Anode



Paul Shering et al; ECS May 2009

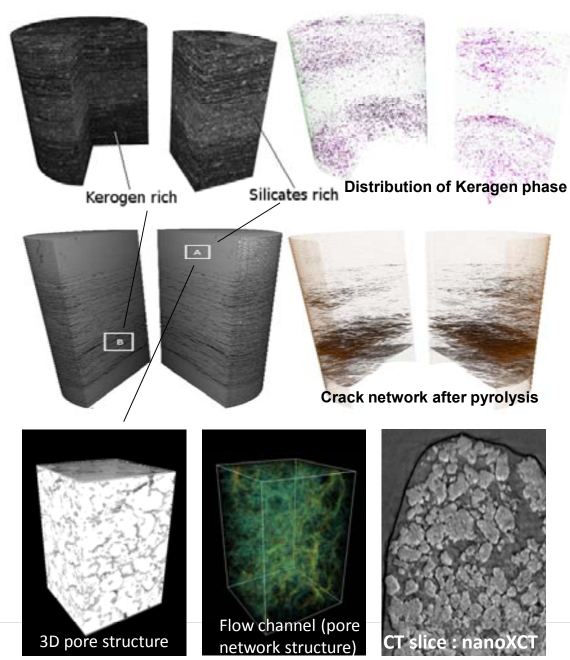
Material Phase segmentation: nanoXCT vs FIB SEM

- nanoXCT: non-destructive characterization of SOFC material



Paul Shering et al;
ECS May 2009

Multiscale example : Oil shale



Oil shale before pyrolysis
MicroXCT @ 1 um voxel resolution

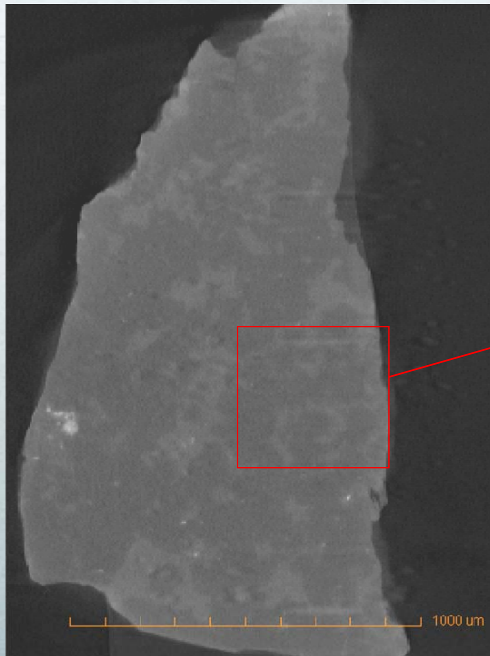
MicroXCT

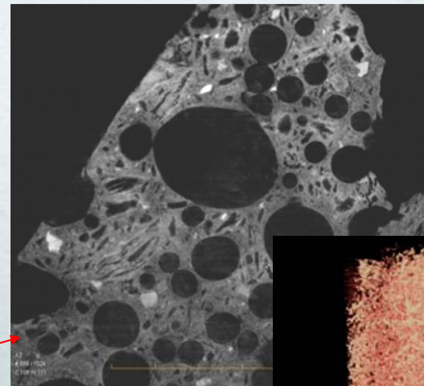
Oil shale after pyrolysis
MicroXCT @ 1 um voxel resolution

nanoXCT

Oil shale after pyrolysis
nanoXCT @ 60 nm voxel resolution
Silicates rich region (A)

Gypsum : Resolution to 0.7 um and 60 nm



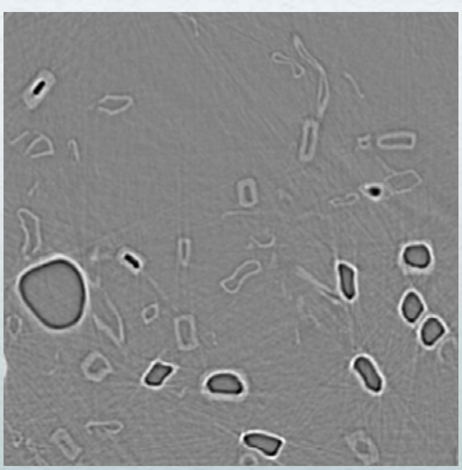


CT Slice at 0.7 um resolution

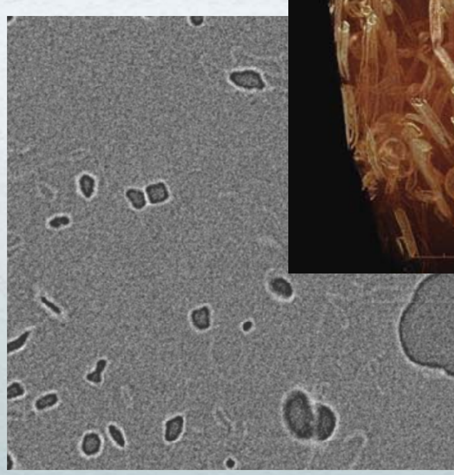
3D at 60 nm resolution voxel

Paper fiber: Synchroton 0.7 um vs Lab MicroXCT

Non-processed non-enhanced raw data



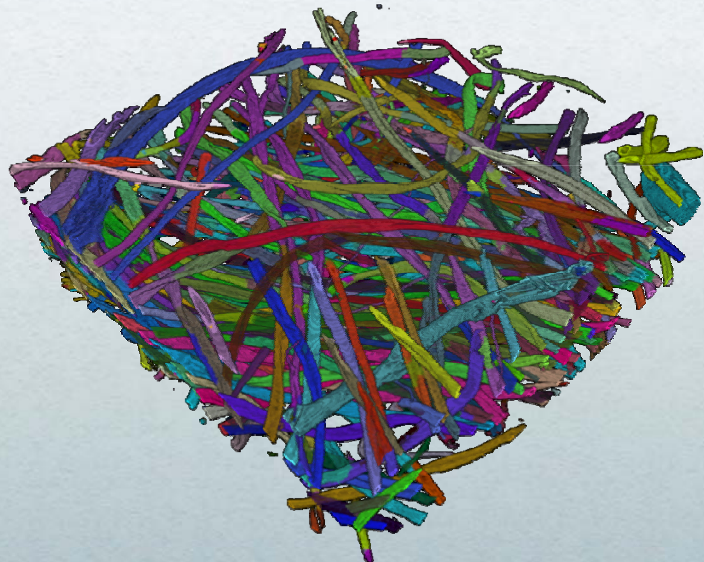
ESRF Grenoble 0.7 µm



MicroXCT 0.7 µm

Sample courtesy: Prof J Timonen, Univ Jyvaskyla

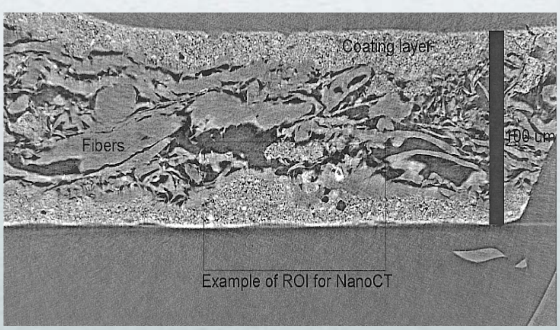
Segmentation of individual fibers



The individual fibers can be segmented from materials that have well-defined fibers. This will also allow an analysis of the bonding properties of the network.

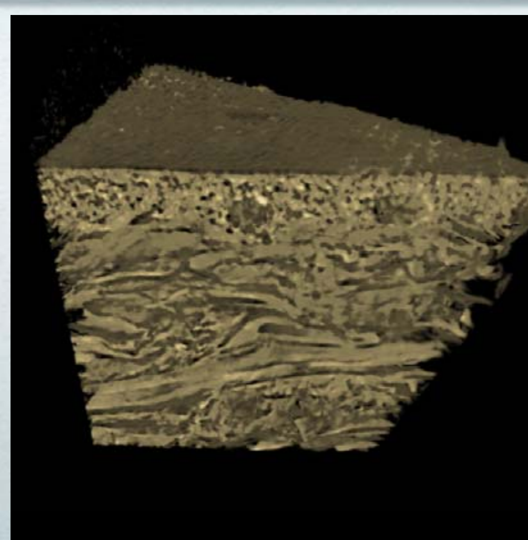
Sample courtesy: Prof J Timonen, Univ Jyvaskyla

Coated paper with Calcium Carbonate; Synchrotron nanotomography vs lab nanoXCT



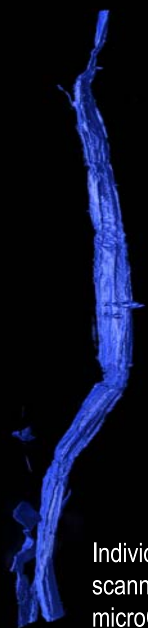
Coated Paper Sample; ESRF

Courtesy of U. of Jyvaskyla



60 x 60 µm FOV,150 nm resolution, 200X
Zernike phase contrast
70 um thick sample, Zernike phase contrast

Individual fiber imaging



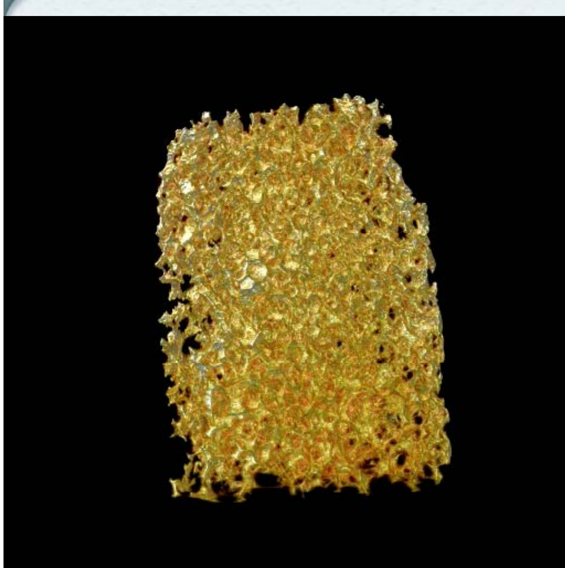
Individual fiber scanned with Xradia microCT with a spatial resolution of ~1 µm

Individual fiber scanned with Xradia nanoCT with a spatial resolution of 150 nm.

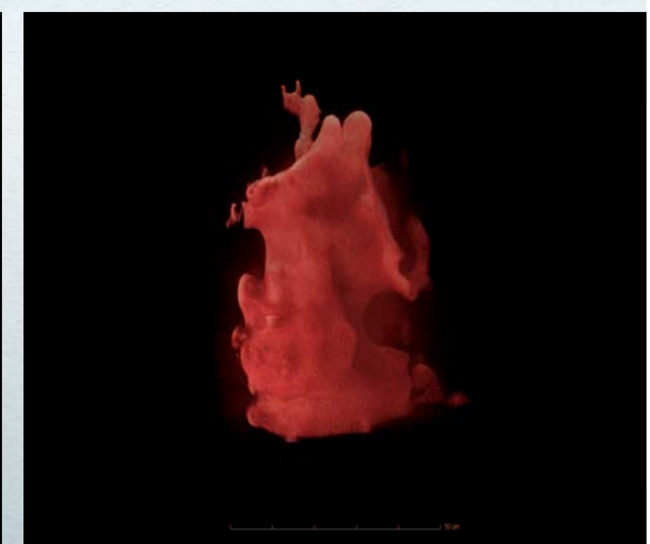
Gold coated.

Sample courtesy: Prof J Timonen, Univ Jyvaskyla

TiO2 Foam: Multiscale characterization



MicroCT 3D Rendered image @ 0.7 microns



nanoCT: 3D Rendered image @ 60 nm

Sample courtesy, Prof Withers, Univ Manchester

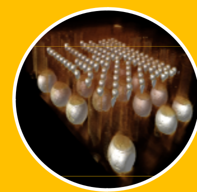
Advantages of novel Xradia MicroXCT

RESOLUTION

Submicron

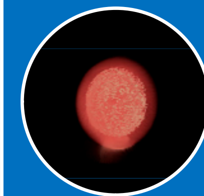
CONTRAST

Superb !



SAMPLE SIZE

Multiscale with High resolution



LOW Z TO HIGH Z

Soft materials to metals

Image Quality ⟷ Versatility

Summary

- Described non invasive 3D characterization of materials
- Technique requires little or no sample preparation
- Highly advantageous for pore characterization, failure evaluation, 3D buried structures
- Multiscale characterization solution from mm to 50 nm