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Small Scale Testing within a Correlative Multi-scale Framework

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BP International Centre for Advanced Materials,
University of Manchester
& Research Complex at Harwell

NPL-EPSRC Fellow & FEI Research Fellows, University of Manchester

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Materials Design Challenge

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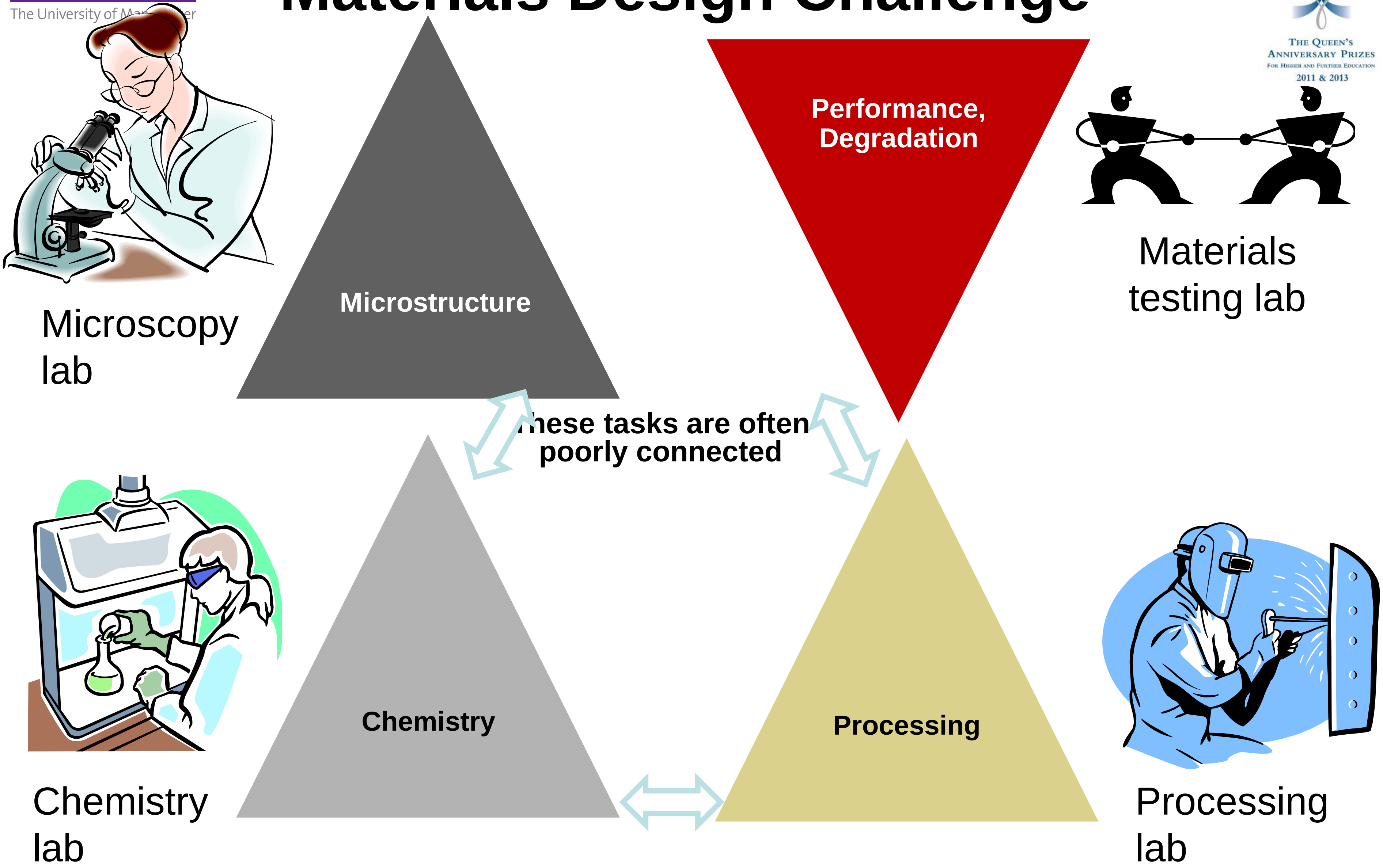

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Materials Science Challenge

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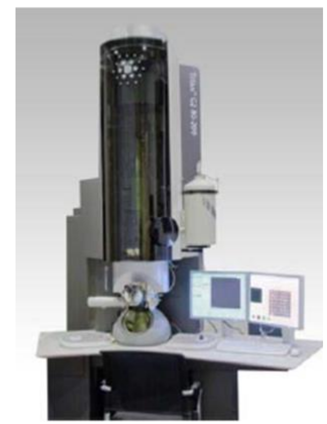
To design better high performance materials we need to:

- Identify the critical length and time scales
- Bring multimodal information to bear on RoI
- Spatially correlate information at different scales

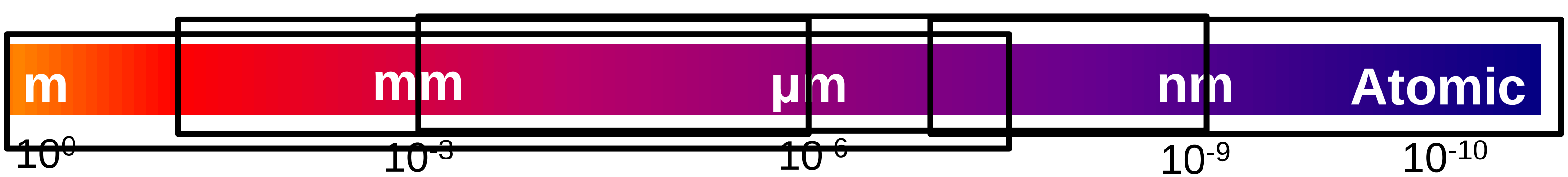
Optical Microscopy

X-ray Tomography

SEM

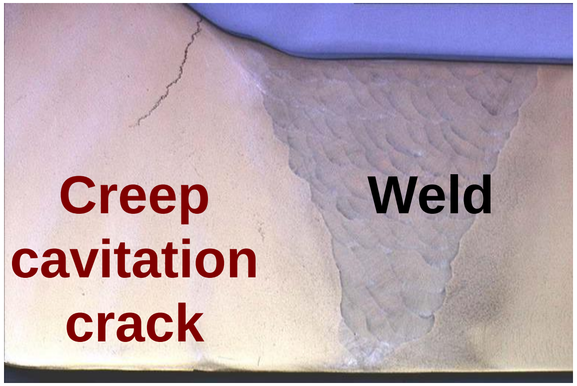


S/TEM



Consider degradation by creep cavitation
e.g. boiler spine in a nuclear reactor

- Stainless steel component
- Combination of high temperature and residual stress local to a weld
- Creep cavitation cracking can be life limiting



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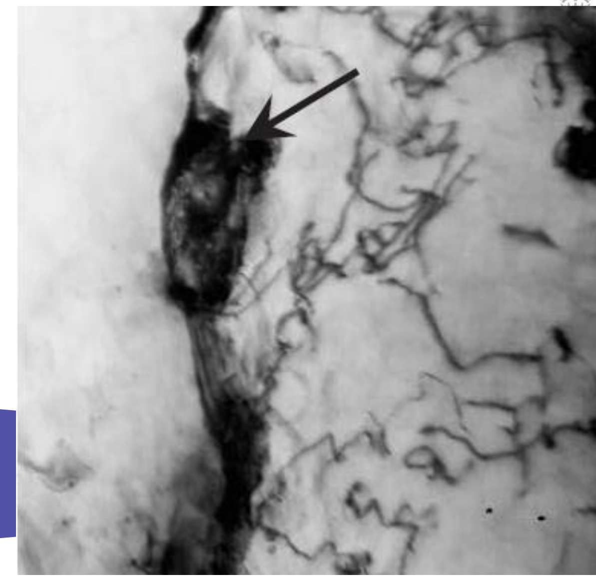
Creep starts at the atomic scale



GB segregation

Point defects

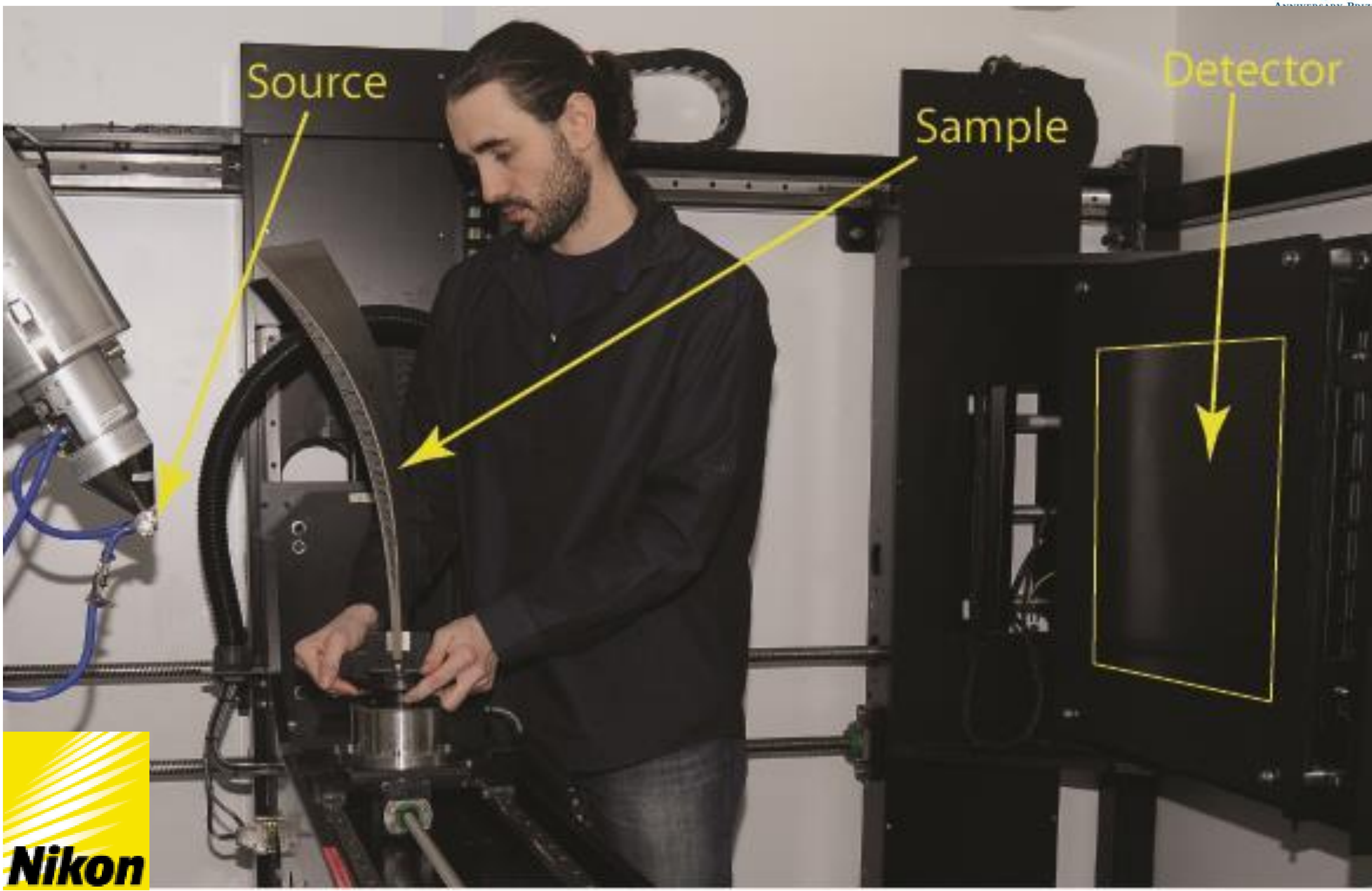
Dislocations

Nanometer **Atomic**

10^{-9} 10^{-10}

TEM

Large scale imaging



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Micron resolution lab. x-ray imaging

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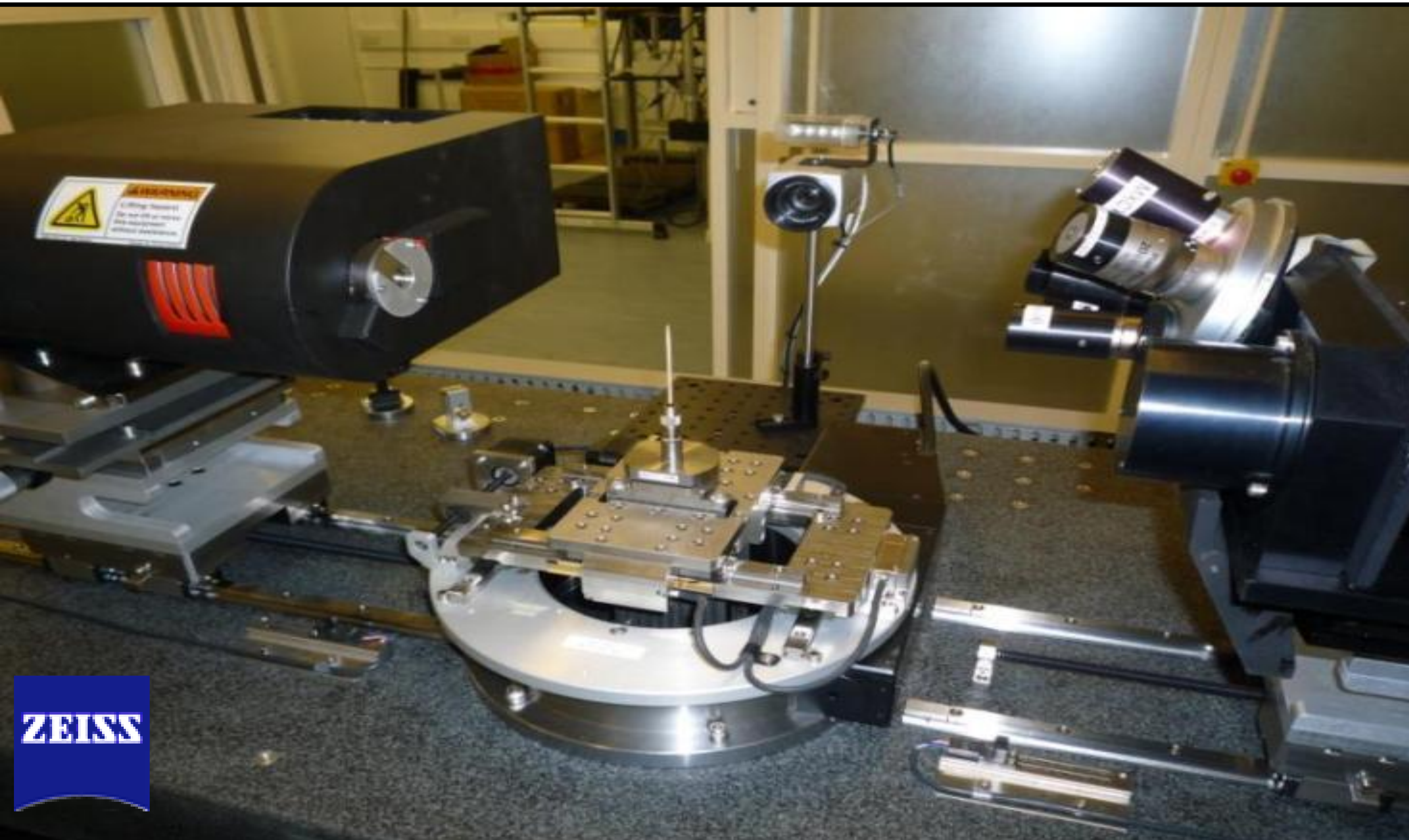

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Nanoscale lab. x-ray imaging

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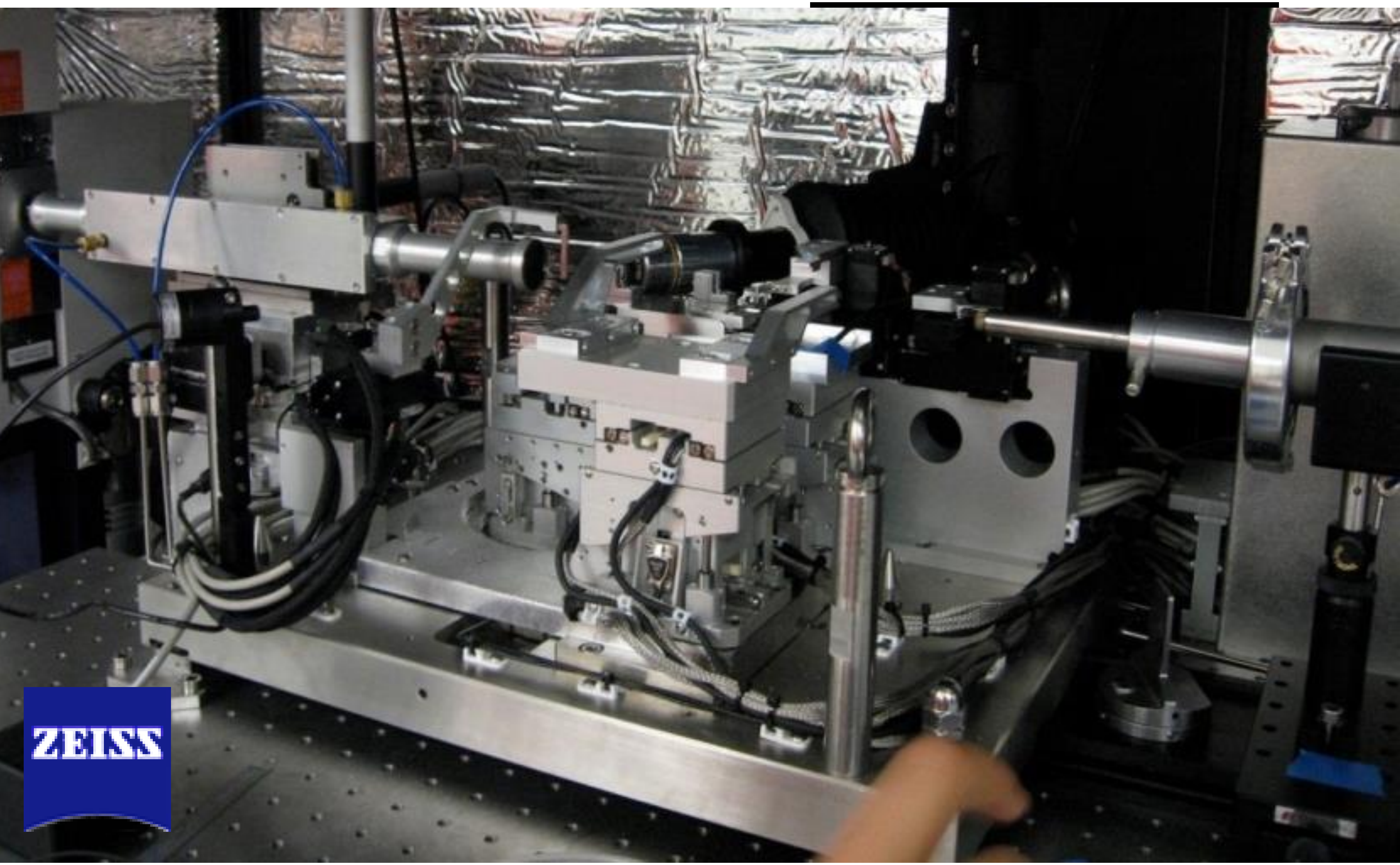

Ultra high resolution imaging and chemistry

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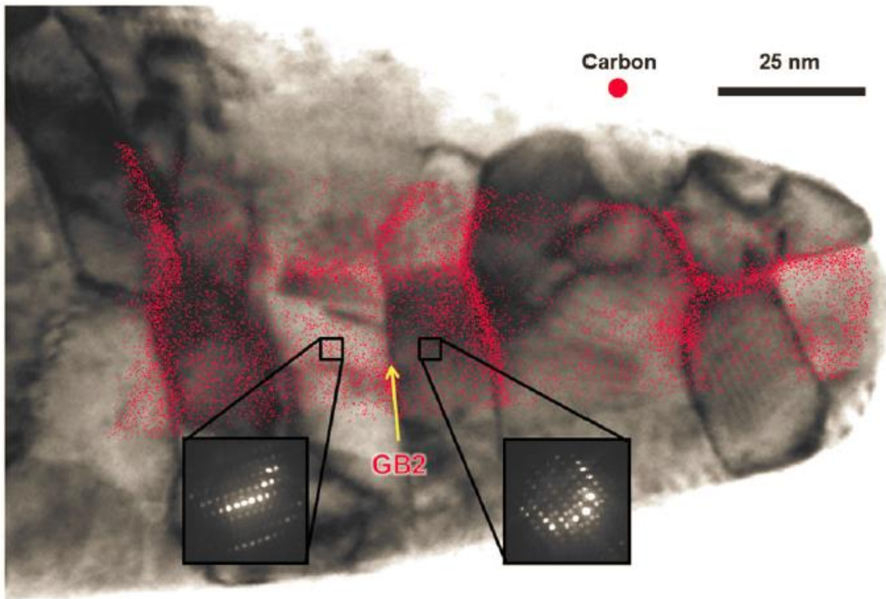
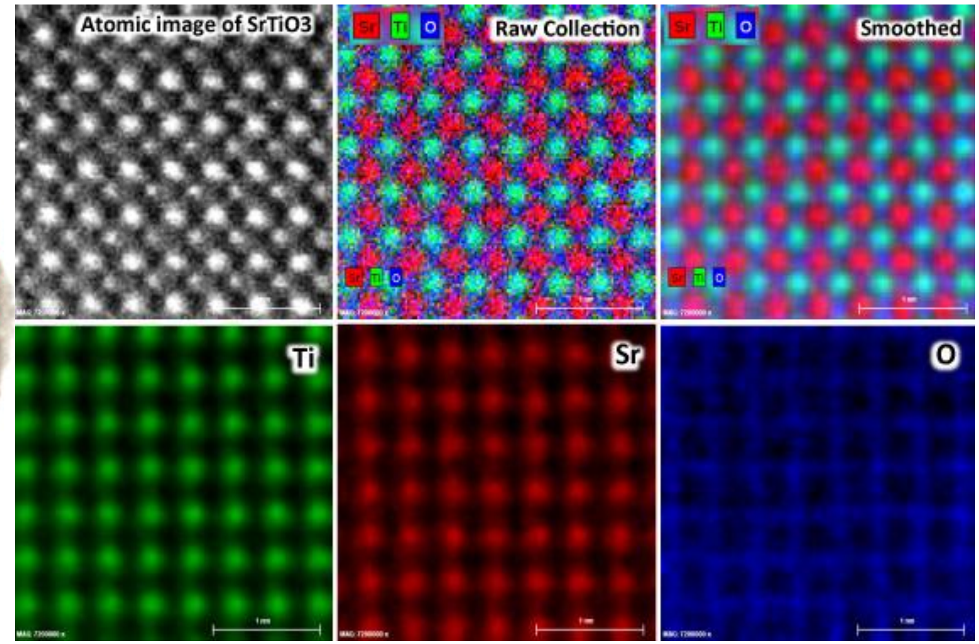
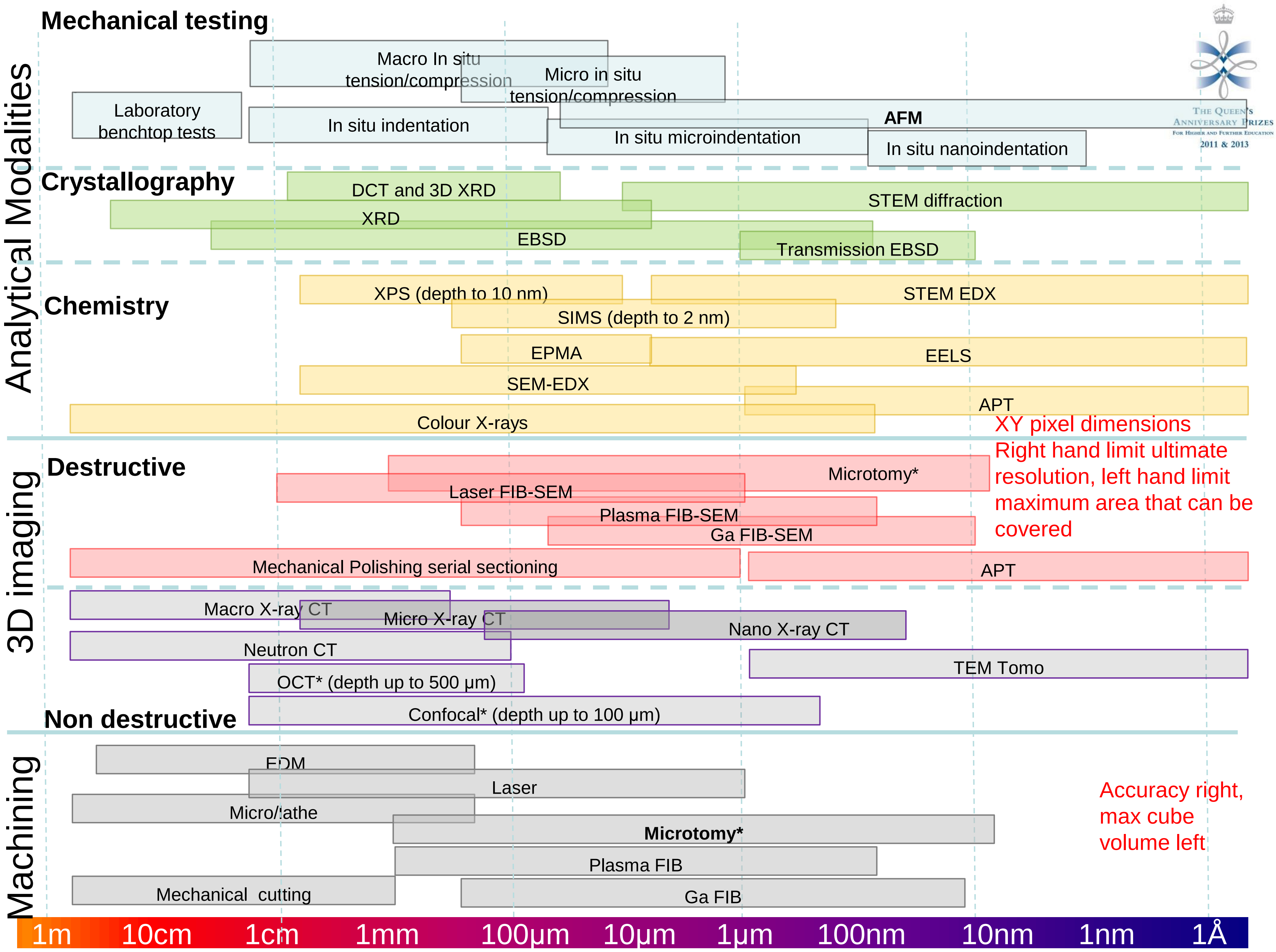


Overlay 3D atom probe map and STEM image of pearlitic steel: GB2, (Σ3 coherent twin) shows much lower C segregation than average grain boundary[Herbig, Raabe et al PRL 2014]



Atomic resolution elemental map of $SrTiO_3$ crystal by Super X EDS on Titan 80-300 Aberration Corrected STEM [NC State Univ]



Analytical Modalities
Mechanical testing
Macro In situ tension/compression
Micro in situ tension/compression
Laboratory benchtop tests
In situ indentation
AFM
In situ microindentation
In situ nanoindentation
Crystallography
DCT and 3D XRD
STEM diffraction
XRD
EBSD
Transmission EBSD
Chemistry
XPS (depth to 10 nm)
STEM EDX
SIMS (depth to 2 nm)
EPMA
EELS
SEM-EDX
APT
Colour X-rays
XY pixel dimensions
Right hand limit ultimate resolution, left hand limit maximum area that can be covered
3D imaging
Destructive
Microtomy*
Laser FIB-SEM
Plasma FIB-SEM
Ga FIB-SEM
Mechanical Polishing serial sectioning
APT
Macro X-ray CT
Micro X-ray CT
Nano X-ray CT
Neutron CT
TEM Tomo
OCT* (depth up to 500 µm)
Confocal* (depth up to 100 µm)
Non destructive
Machining
EDM
Laser
Micro/lathe
Microtomy*
Plasma FIB
Mechanical cutting
Ga FIB
Accuracy right, max cube volume left
1m
10cm
1cm
1mm
100µm
10µm
1µm
100nm
10nm
1nm
1Å
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To see the value of a correlative multiscale framework lets consider pitting corrosion of a stainless steel wire

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Connecting timescales: Macroscale X-ray CT

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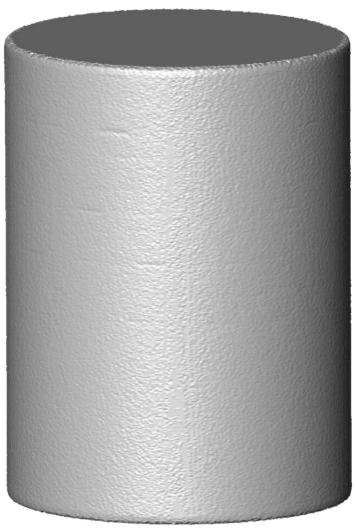
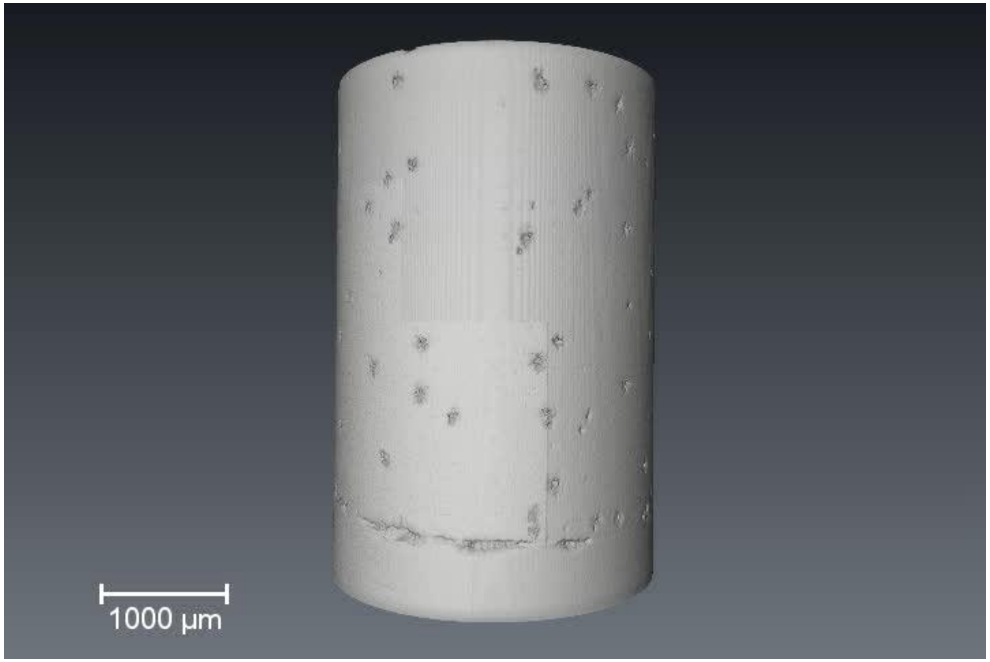
Sample immersed in Chloride Solution and polarized

- Time lapse images show nucleation and growth of corrosion pits
- Identification of the fastest or slowest growing pits

But we need to better characterize the pit morphology….





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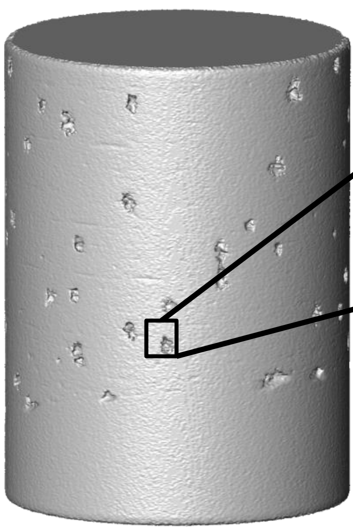
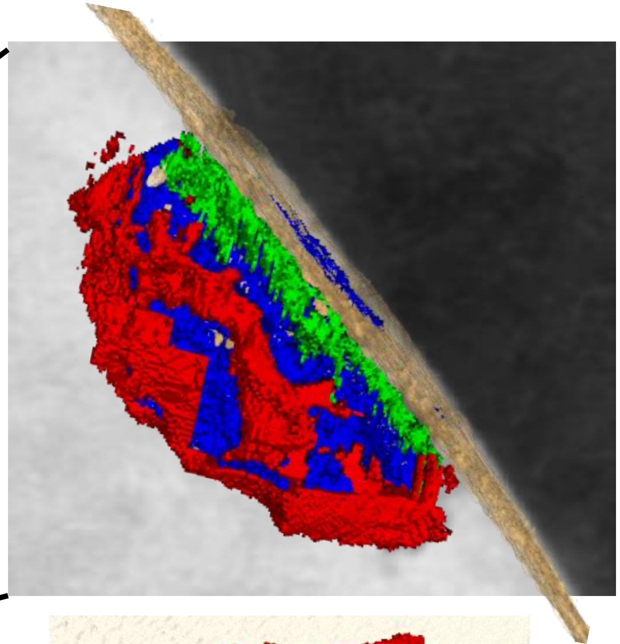
Connecting Scales: high-resolution X-ray CT

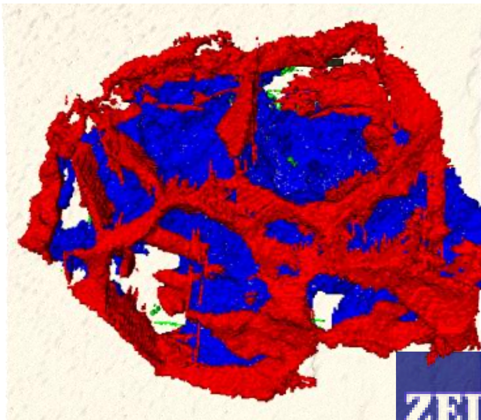
Use macroscale X-ray CT as a 3D map to find RoI for microscale X-ray CT

- We can now see the detailed morphology of the corrosion pit, revealing a network of ‘intergranular’ corrosion surrounding the pit

We now need to explore the microstructure around the pit in more detail….







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Correlative Tomography: Linking X-ray CT to SEM

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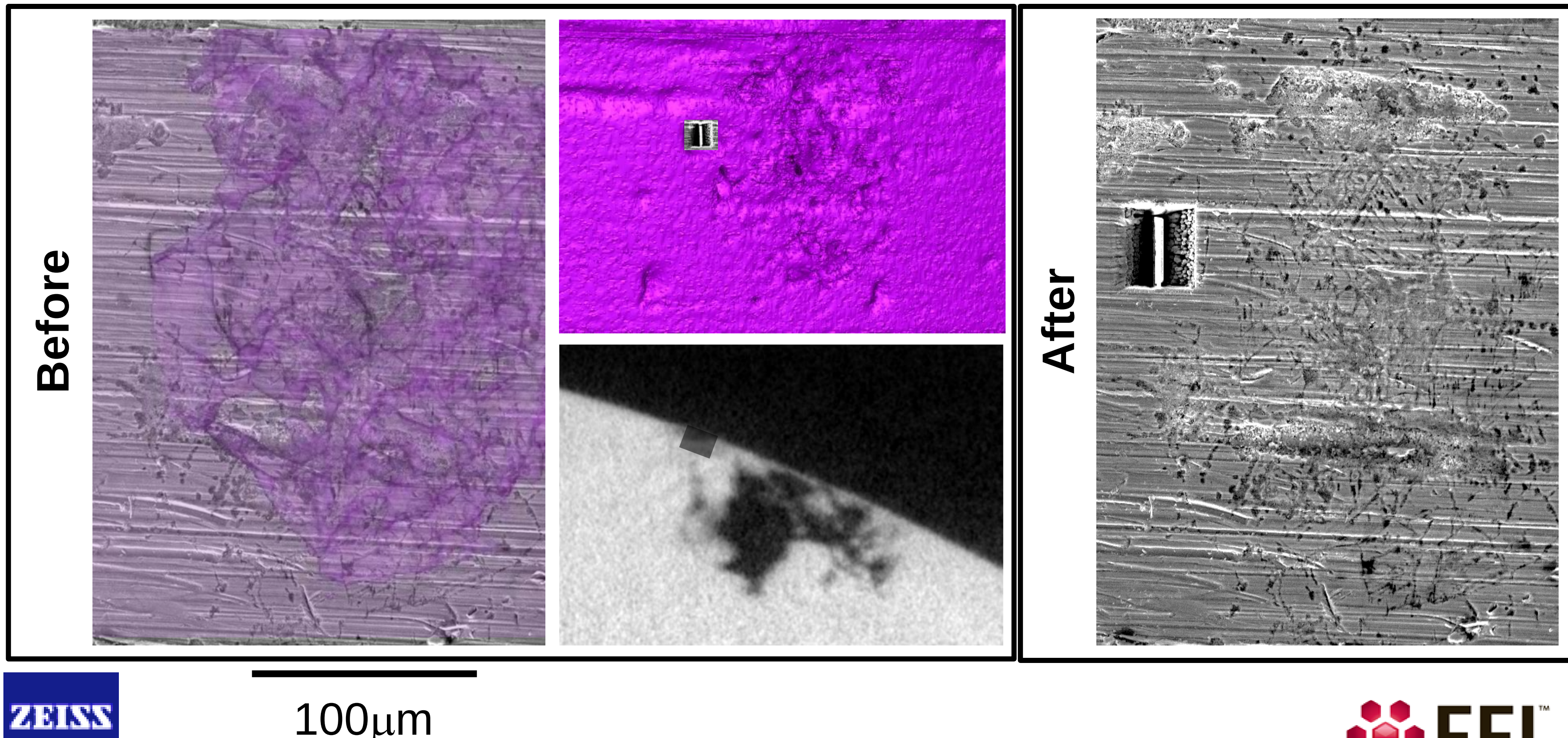
Manually register the surface as rendered from X-ray CT to the SEM image to locate periphery of pit obscured by a lacy cover:



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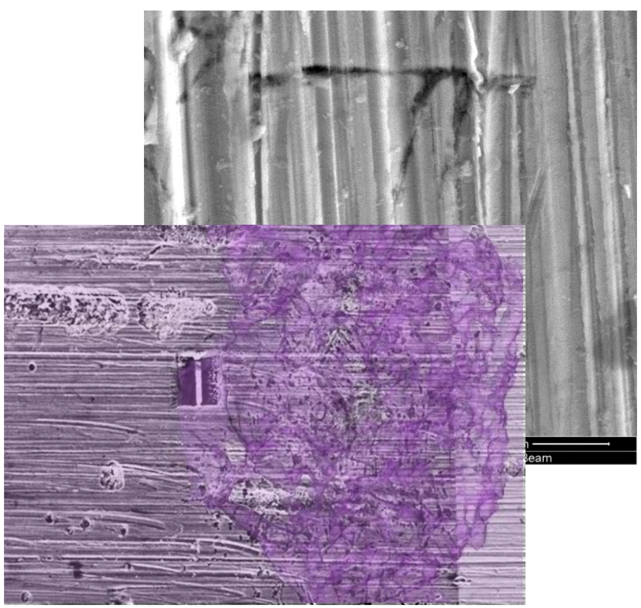
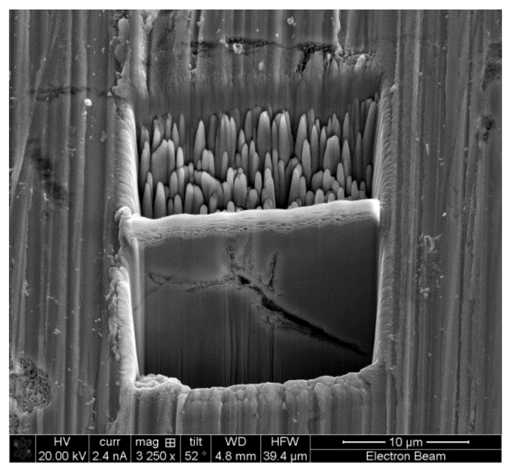
Correlative Tomography: FIB-SEM serial sectioning

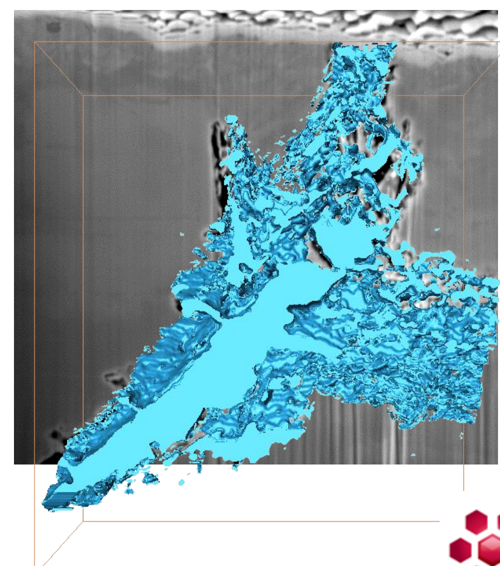
3D analysis at the nanoscale using Slice and View

- Destructive but very high resolution and SEM imaging reveals contrast from grain boundaries
- Characterize the shape, extent and direction of the corrosion fronts

But we need to understand the crystallography to identify the corrosion fronts….





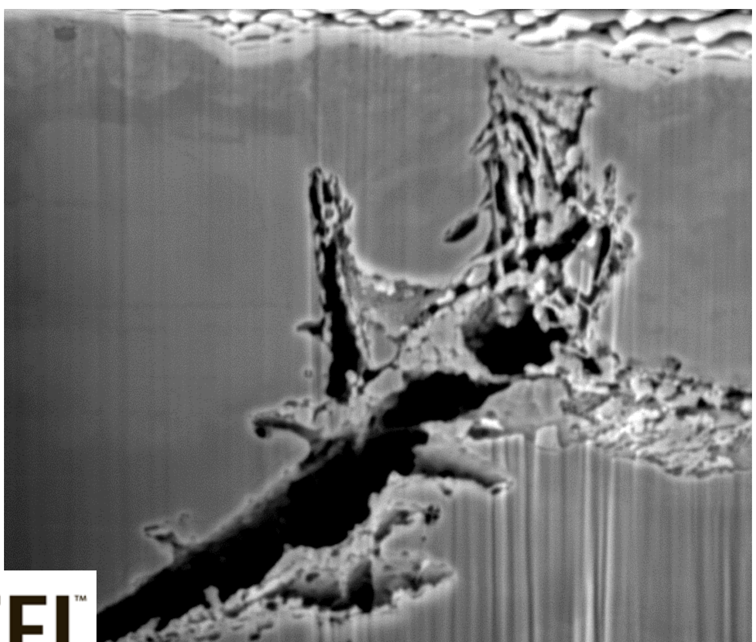


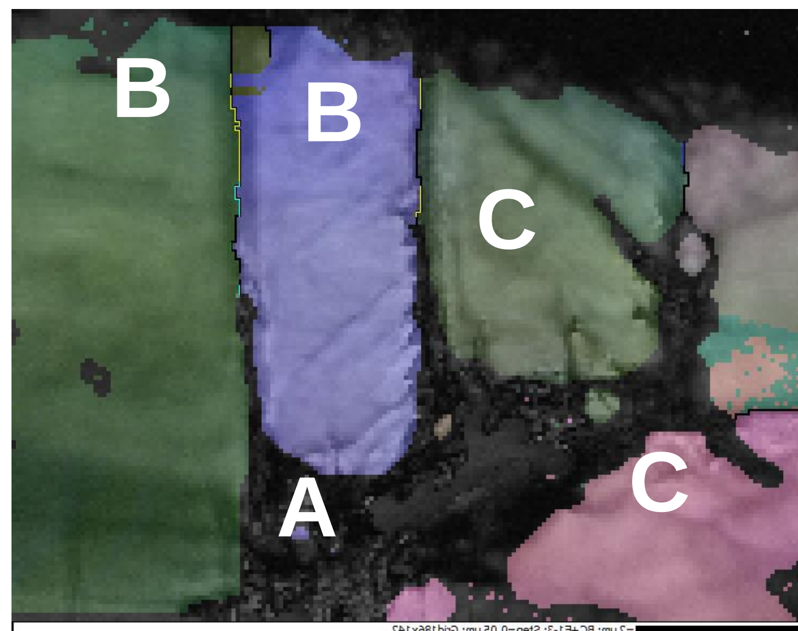
Connecting Modalities: Crystallography

Electron backscatter diffraction has enabled analysis of the crystallography around the corrosion fronts

- We have identified high angle grain boundaries (A), coincidence site lattice (CSL)(B) and slip bands (C)
- The structural disorder of each of these boundaries appears related to the degree of corrosion

But we need to understand the role of the materials chemistry….

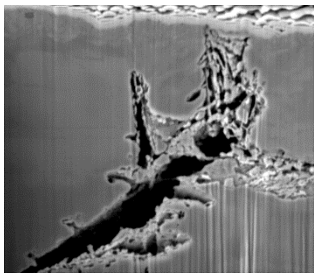


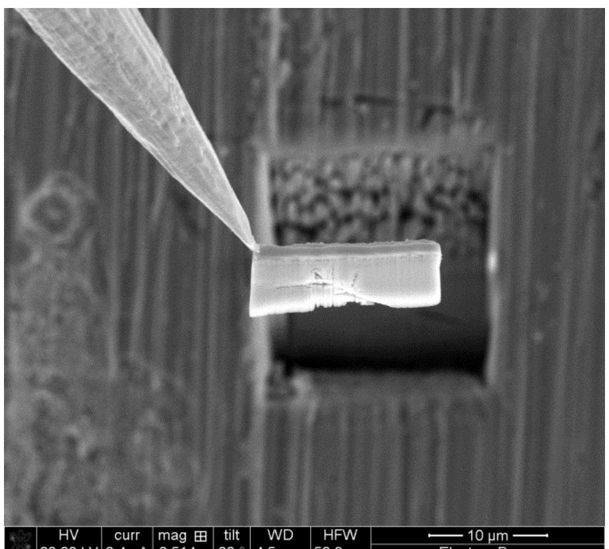


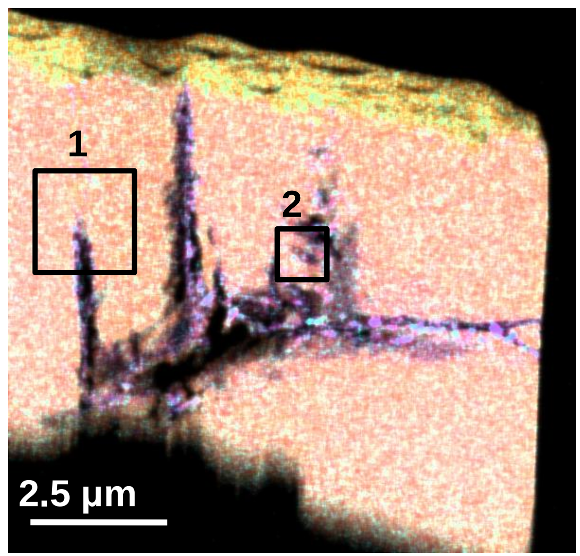
Connecting modalities: Nanoscale Chemical Analysis

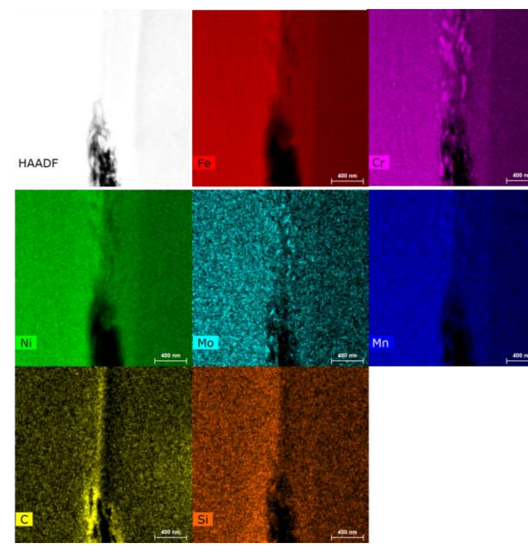
Chemical Mapping with Titan ChemiSTEM-EDS

- GB and CSL are associated with chemical segregation
- Slip bands have not yet shown any chemical segregation

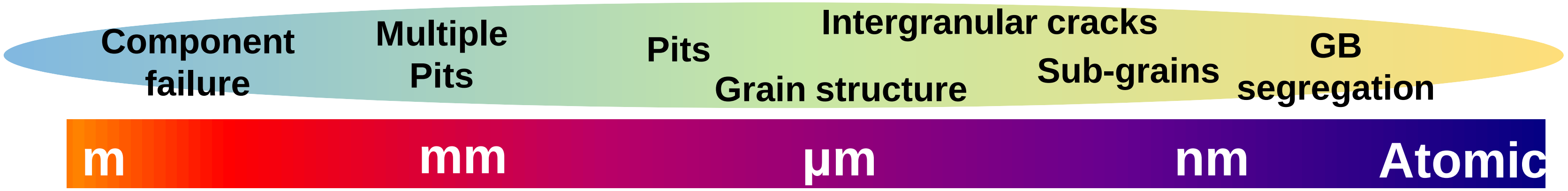


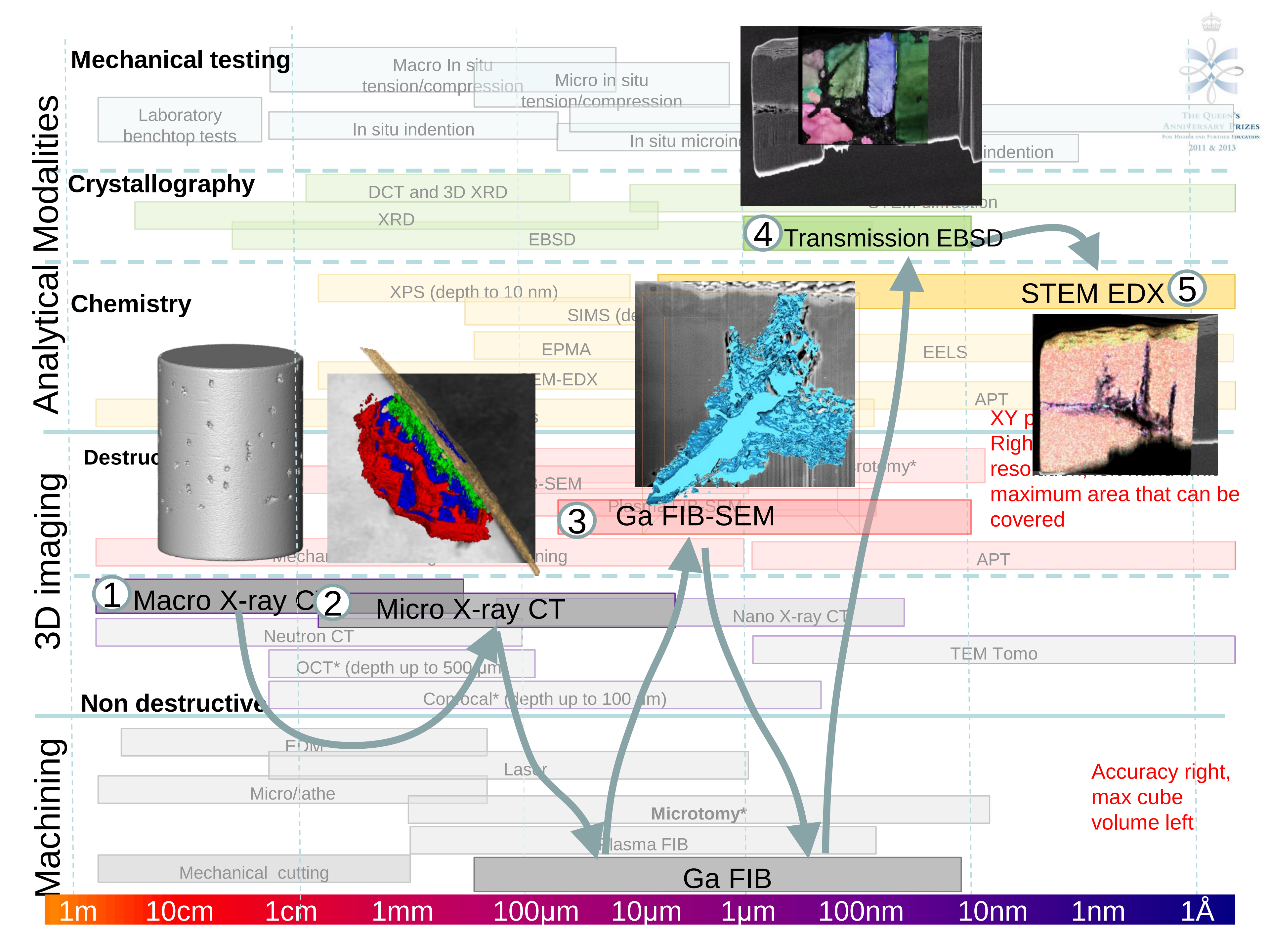






Correlative Tomography





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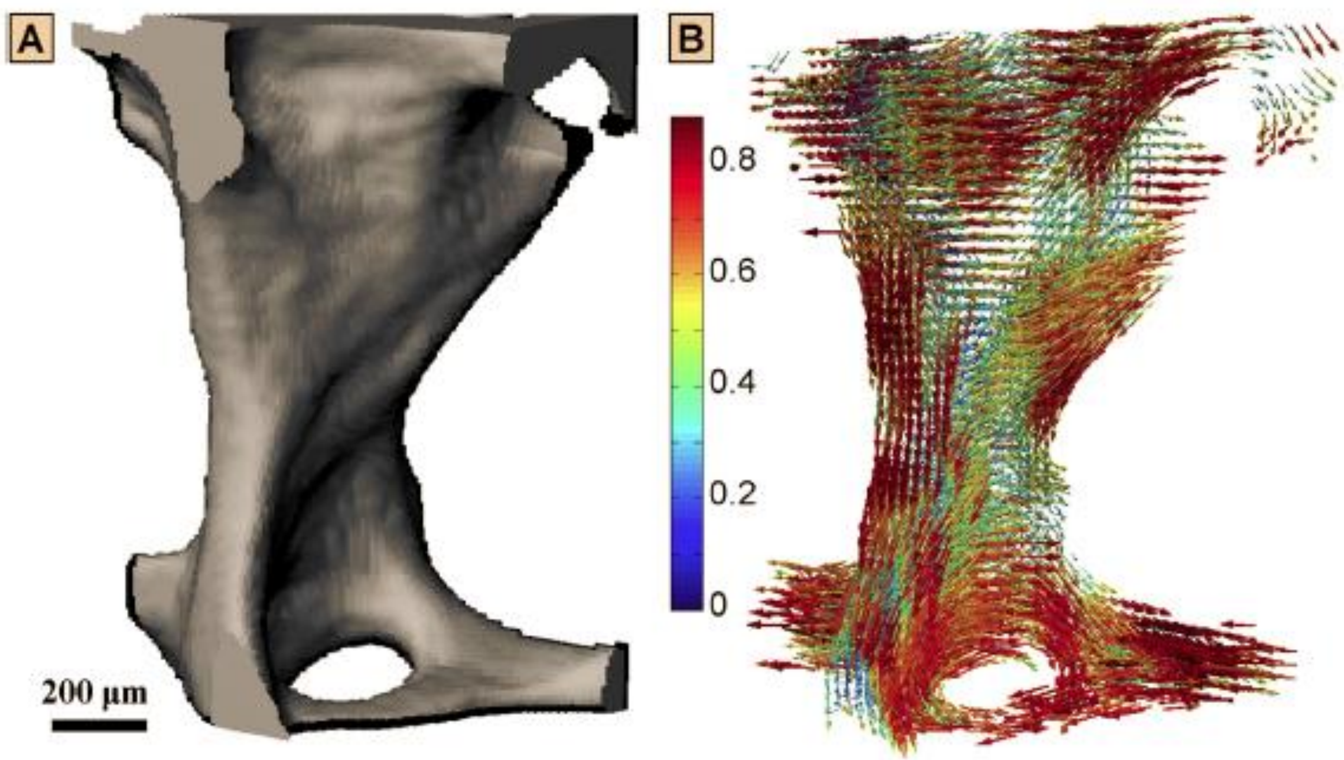
XY plane: Right resolution,
maximum area that can be covered
3D imaging
Ga FIB-SEM 3
APT
Macro X-ray CT 1
Micro X-ray CT 2
Nano X-ray CT
Neutron CT
TEM Tomo
OCT* (depth up to 500 µm)
Non destructive
Machining
Laser
Micro/lathe
Microtomy*
Plasma FIB
Mechanical cutting
Ga FIB
Accuracy right, max cube volume left
1m 10cm 1cm 1mm 100µm 10µm 1µm 100nm 10nm 1nm 1Å

We now have advanced tools to map microstructures across the scales:



3D map of crystalline orientation in bone trebecula [Georgiadis et al Bone 2014]

Challenge is to correlate these to mechanical properties across the scales

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Mechanical characterisation at the millimetre (polycrystal) scale…….

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Mapping elastic properties - Spatially resolved acoustic spectroscopy (SRAS)

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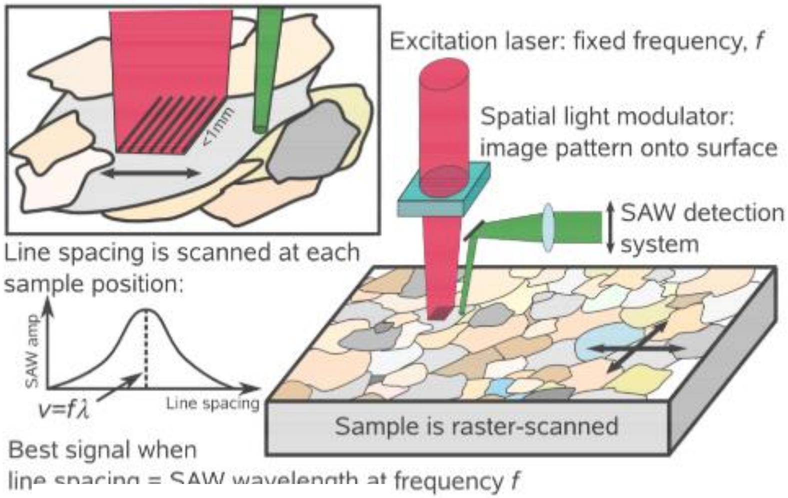
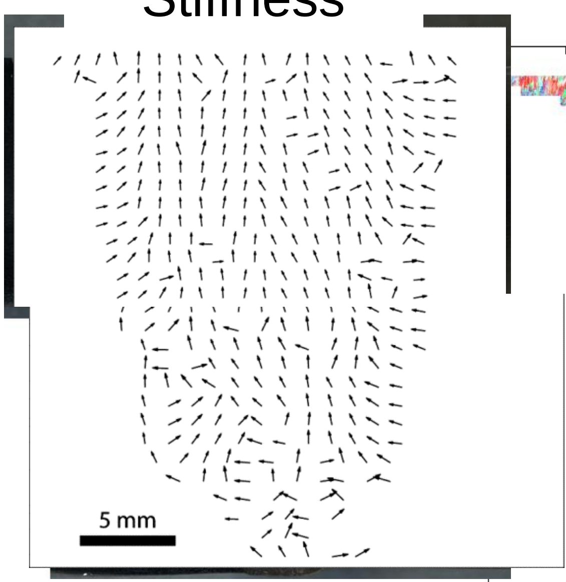
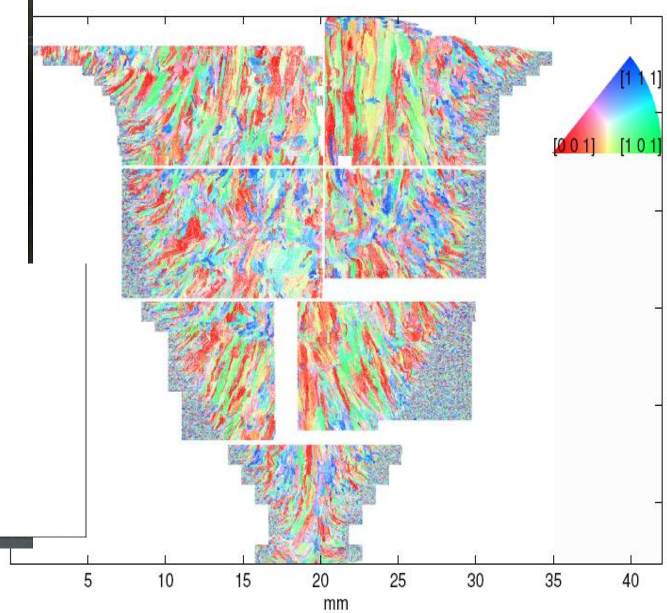


- With Steve Sharples (Nottingham), Lowe (Imperial College)

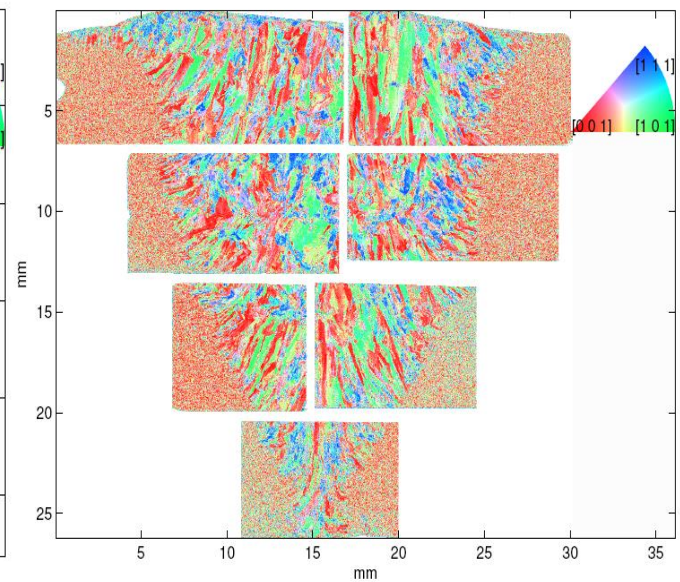
Stiffness



(EBSD:UoM)

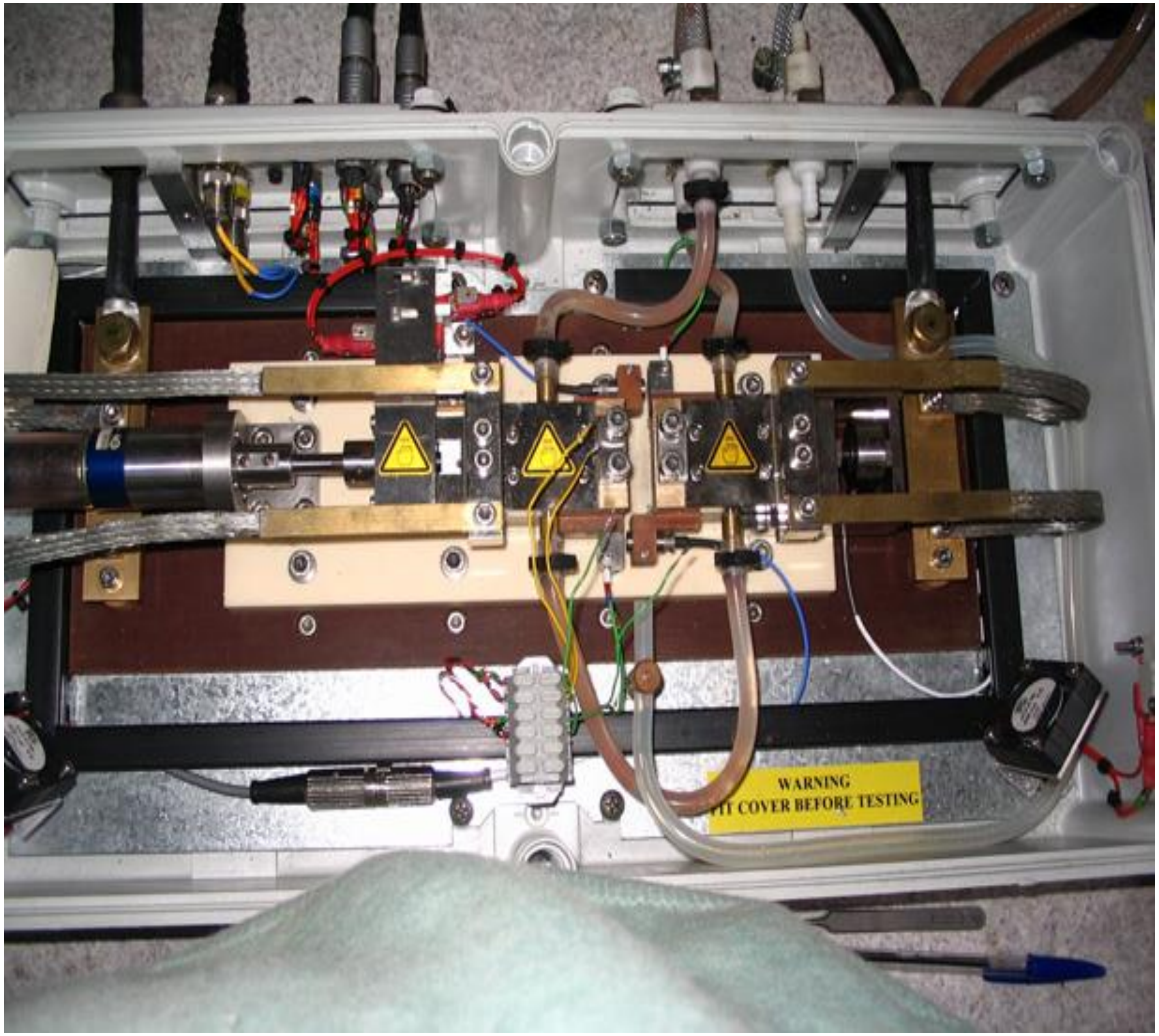


(SRAS:Nott)



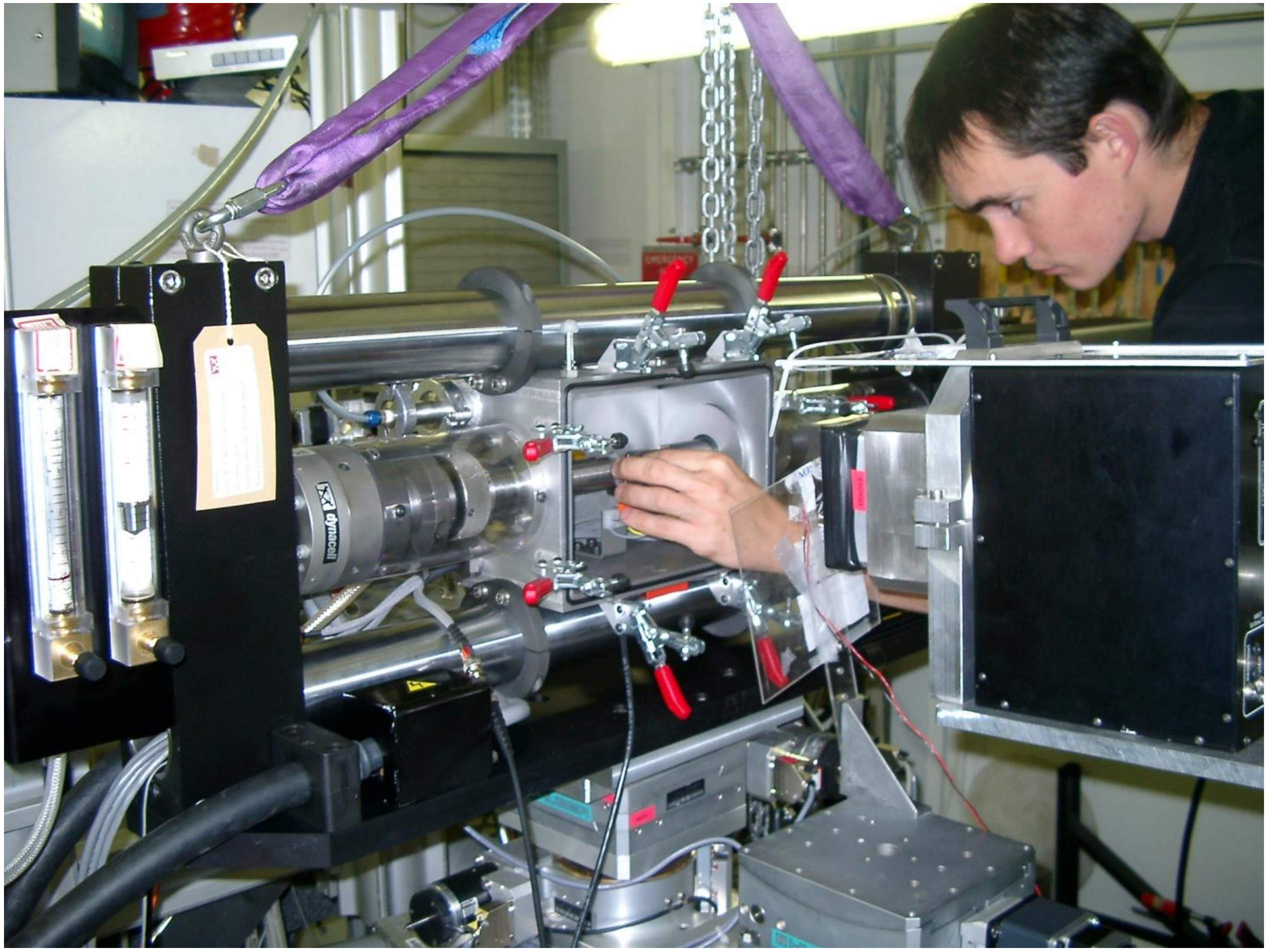
(SS308 TIG weld)

Mapping plastic properties:The ETMT



- Originally developed by Brian Roebuck et al. NPL

In situ monitoring of smart weld filler during weld cooling



Instron version of ETMT

- Experiments conducted at ID11 beamline at the ESRF synchrotron facility

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Evolution of phase fractions

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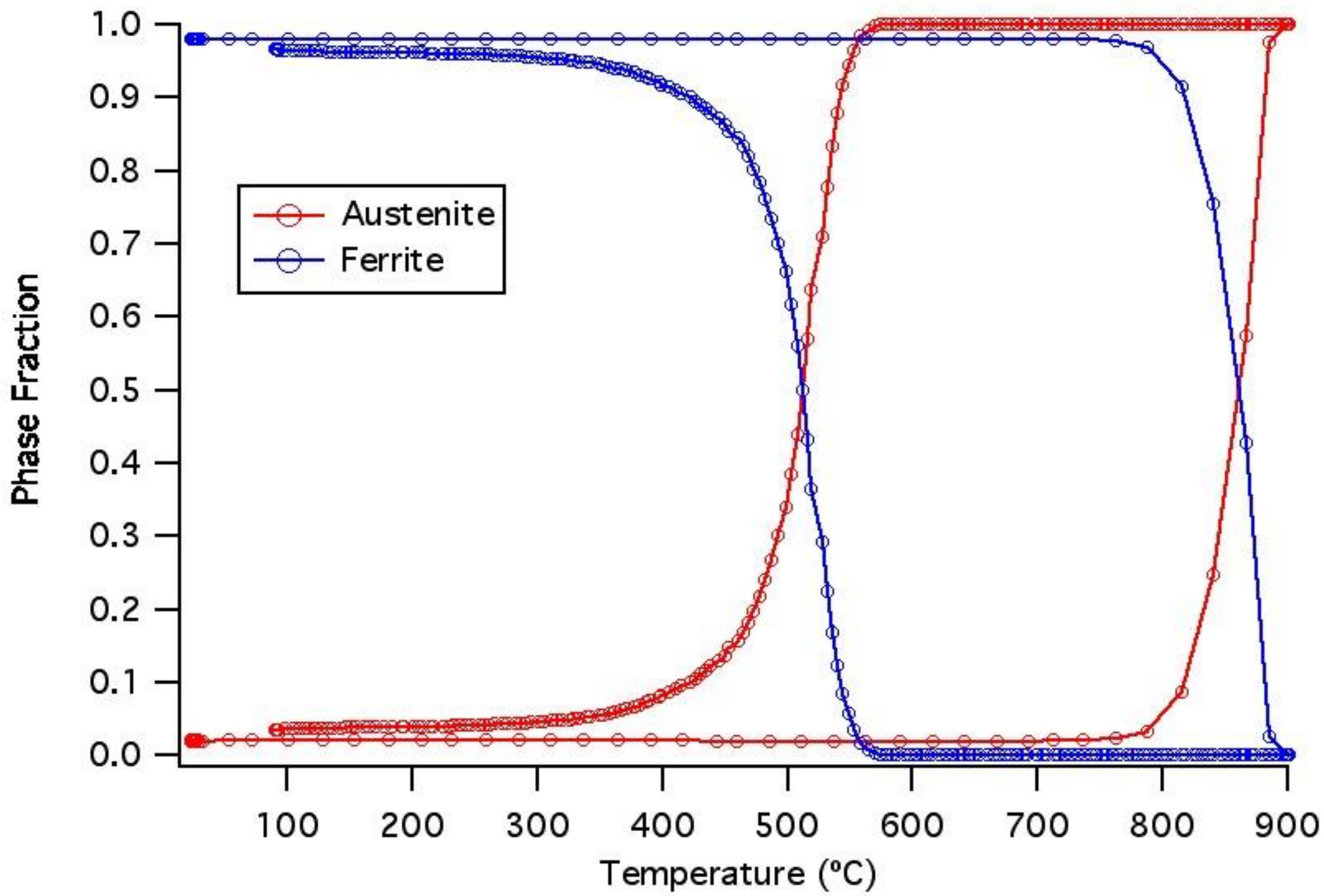

- Full structural (Rietveld) refinement of the diffraction patterns to obtain phase fractions

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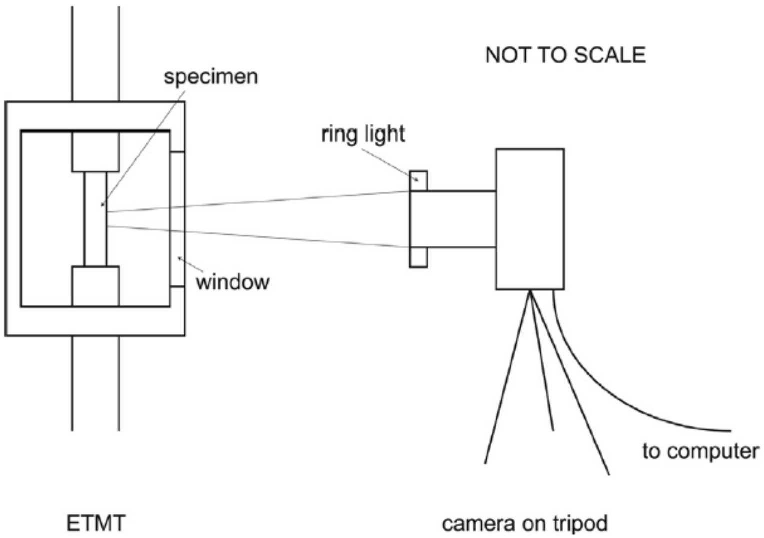
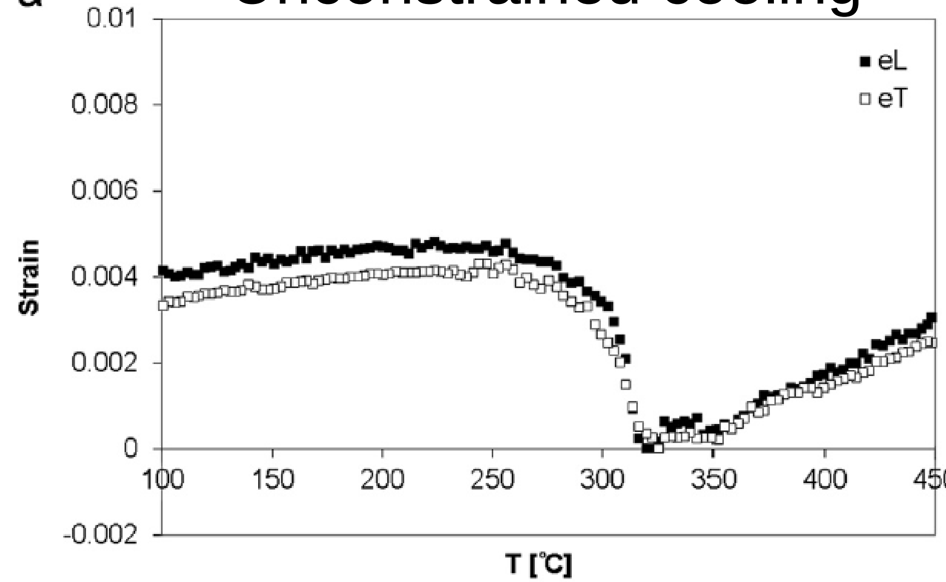
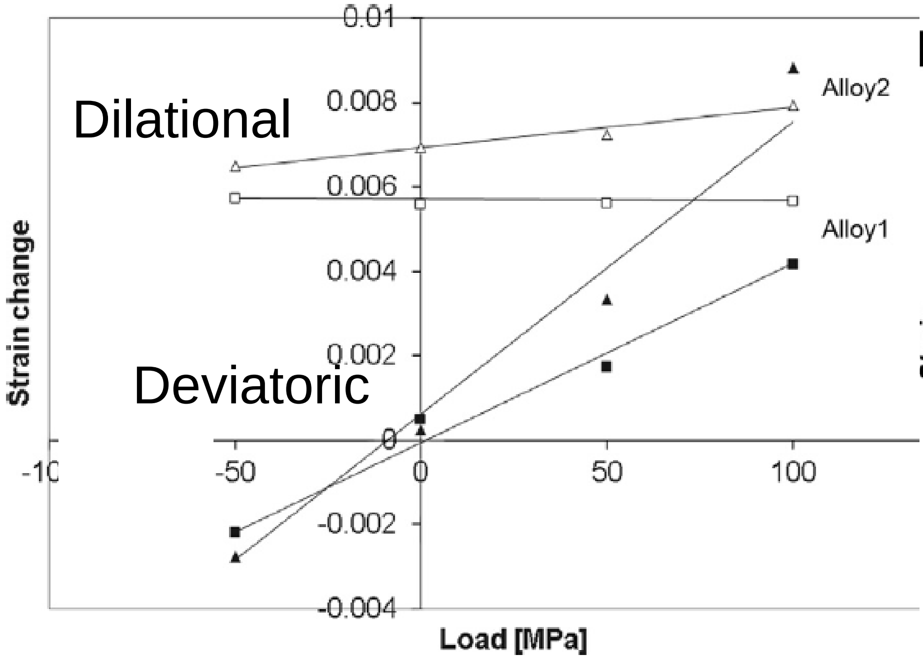
Smart weld fillers

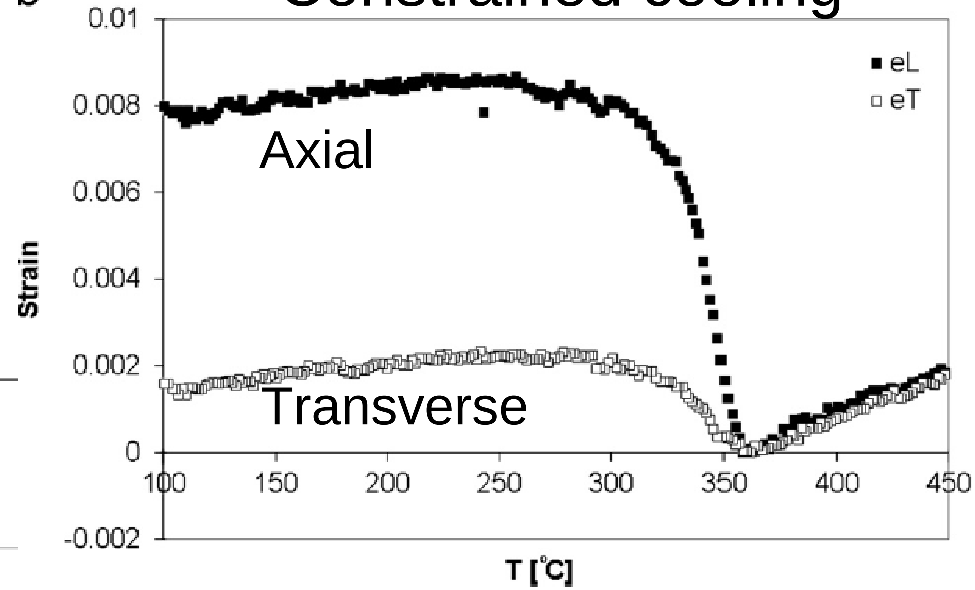


a Unconstrained cooling





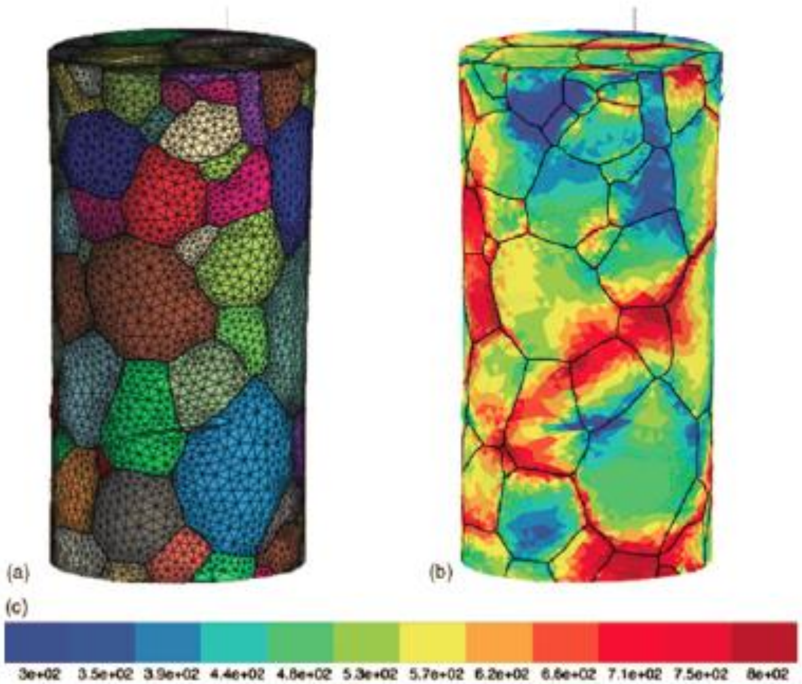
b Constrained cooling



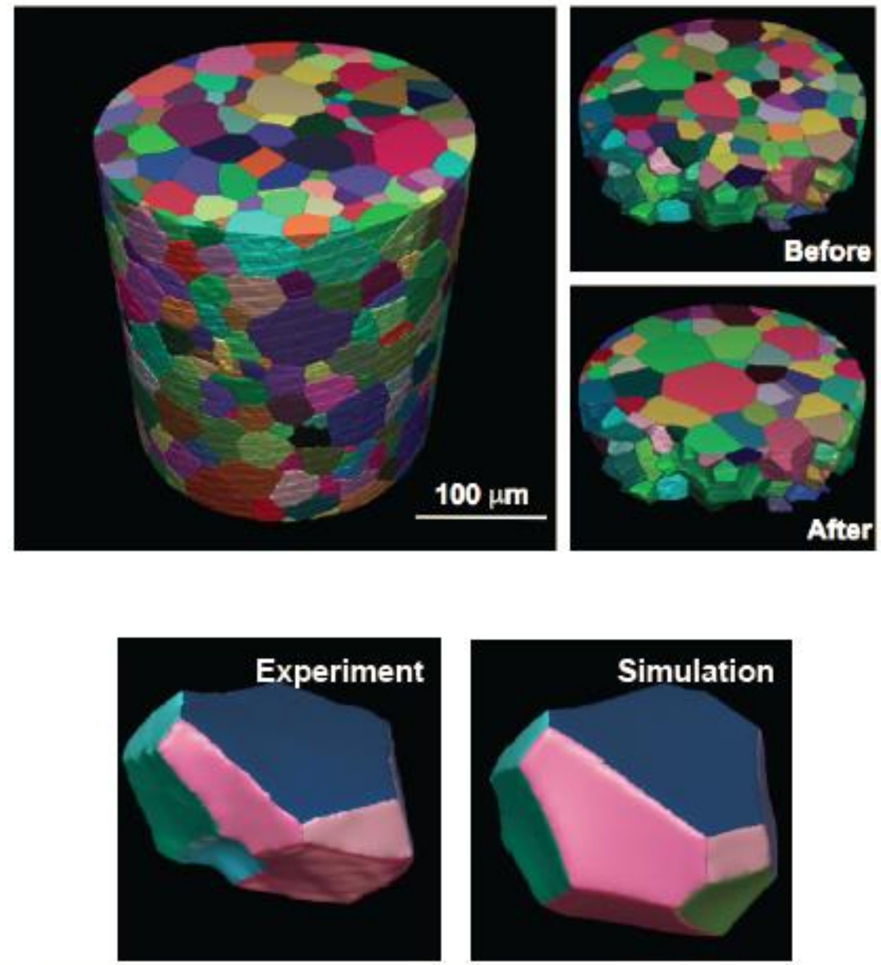
Mechanical characterisation at the tens of micron (grain to grain) scale……

Coupling to 3D simulations

- 3D grain maps are perfectly suited for coupling to 3D computer simulations of microstructure evolution



W. Ludwig, A. King, P. Reischig et al, Materials Science and Engineering A 524 (2009) 69–76

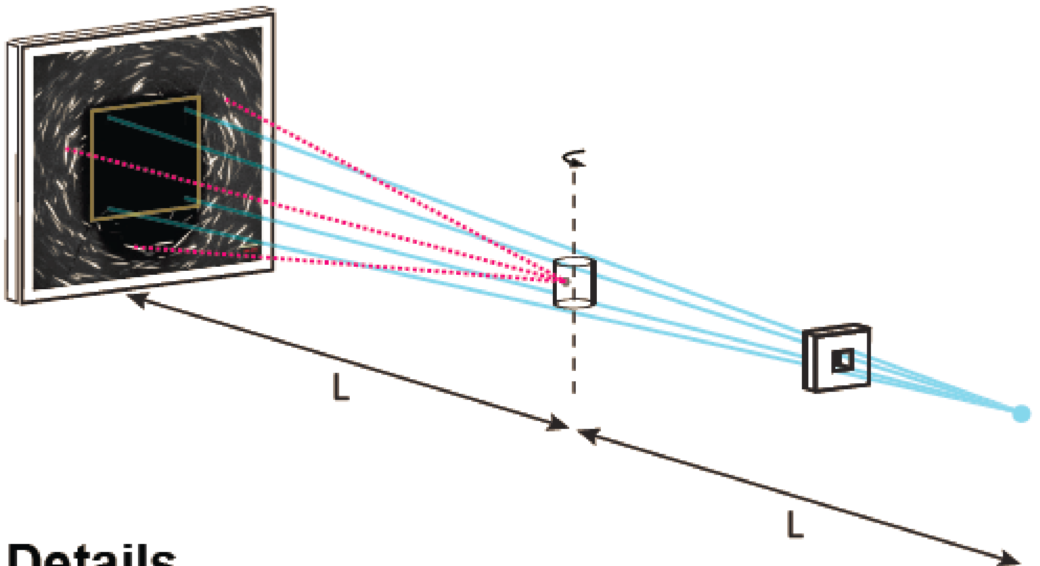


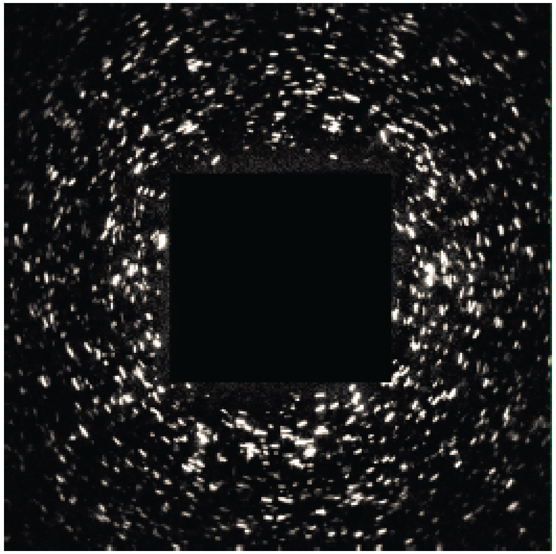
I.M. McKenna, S.O. Poulsen, E.M. Lauridsen, W. Ludwig, P.W. Voorhees, In print

GrainMapper3D™ implementation

New lab-based 3D grain mapper (Zeiss-Xnovo-UoM)

Version 0.5 implementation:
Laue focusing geometry





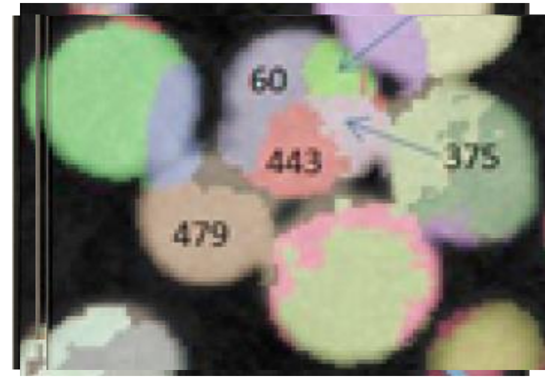
Details

- Add-on module to standard Xradia Versa 520 XRM
- Utilizes white divergent X-ray beam
- Utilizes Laue focusing geometry

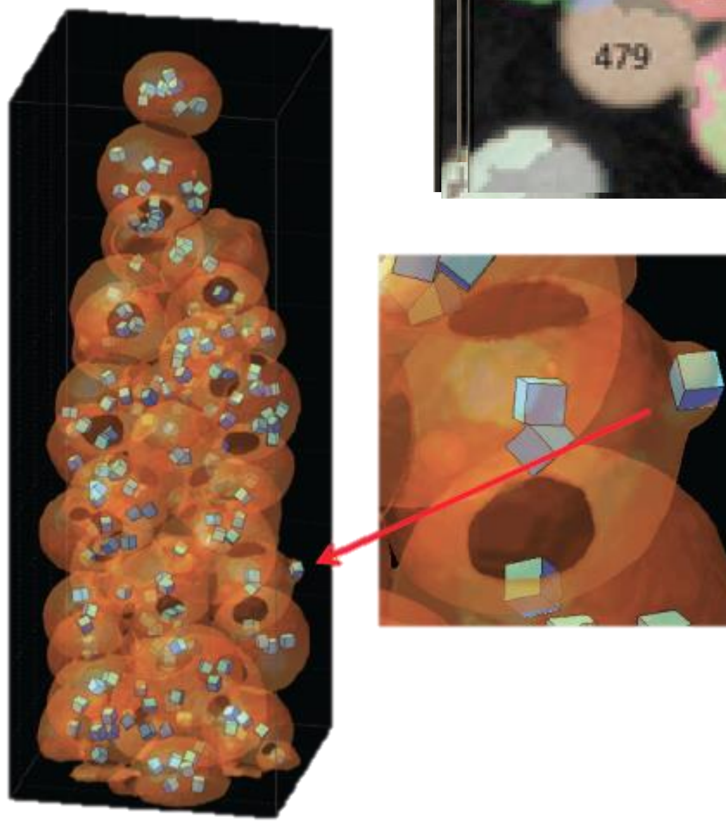
Laboratory Diffraction Contrast Tomography (DCT): crystallographic information

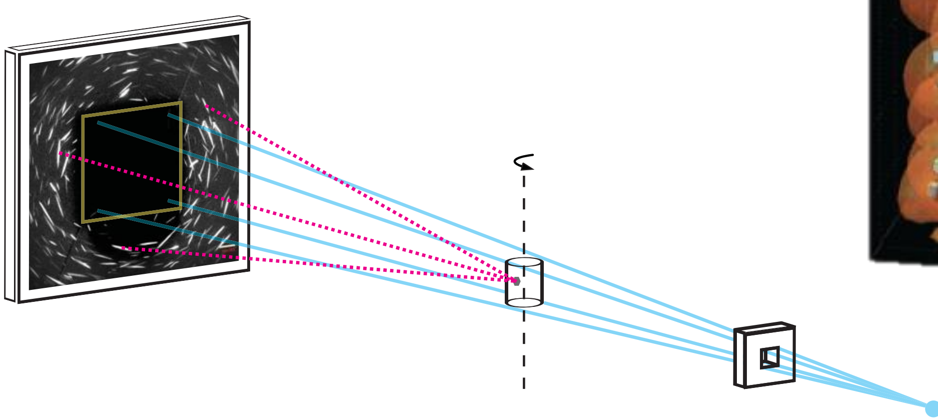
Synchrotron DCT well established
- 3D crystallographic grain maps
- e.g. 3D sintering of Cu particles

2 hours



Bringing synchrotron technology to the laboratory
- 3D crystallographic information obtained on a laboratory XRM (**ZEISS Xradia 520 Versa**)
- Non-destructive 3D grain mapping in lab.



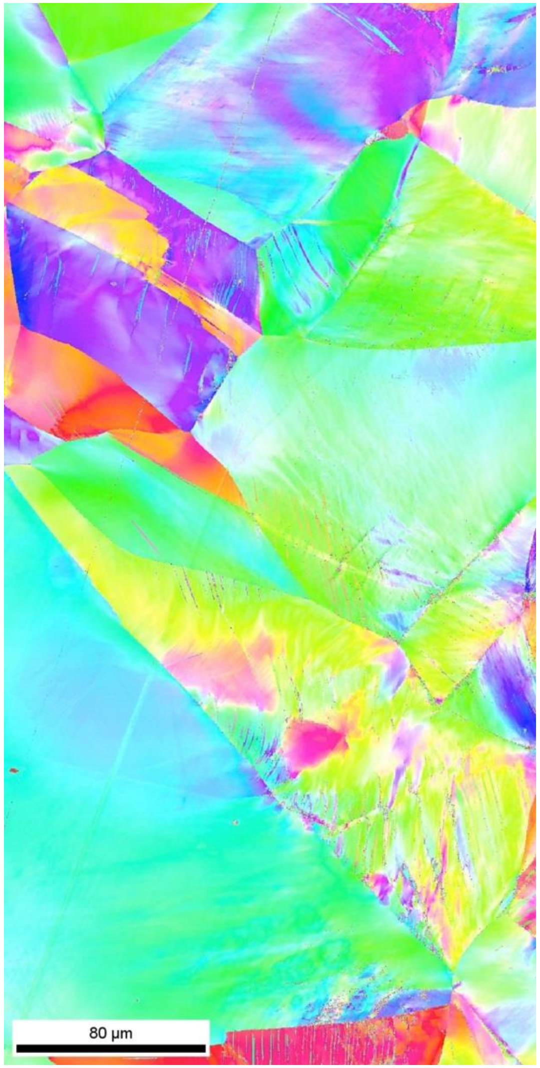


Powered by xnovotech

Mapping grain deformation: EBSD + HRDIC

- Electron Back Scatter Diffraction
  - Before deformation:
    - Grain boundaries
    - Grain orientations (slip traces)
  - After deformation
    - Lattice rotations (local misorientation)
    - Low angle grain boundaries
  - Spatial resolution: 0.2 µm

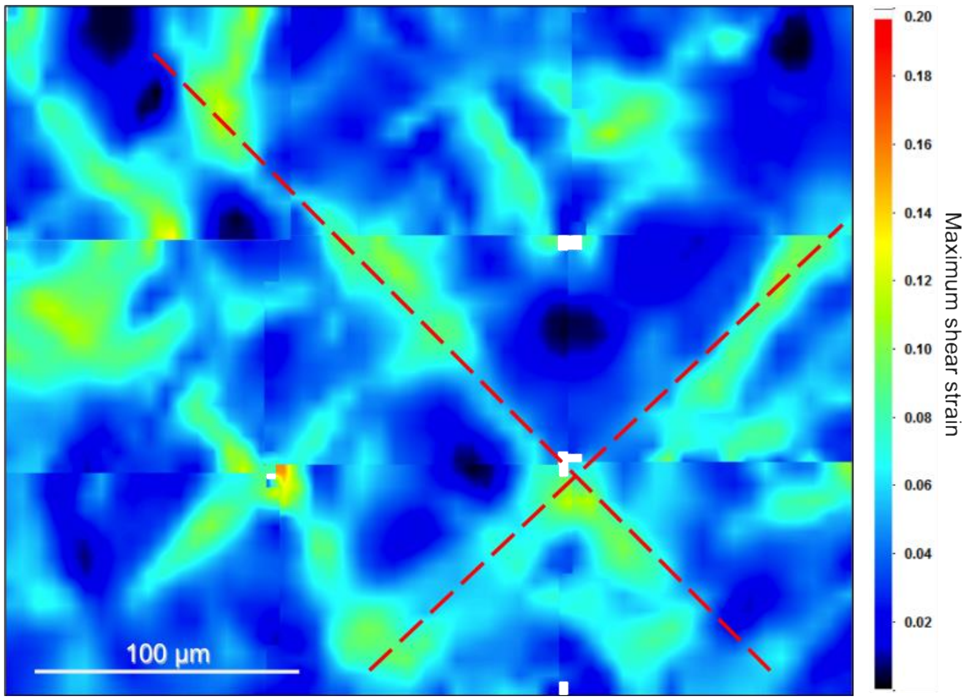
- High Resolution Digital Image Correlation
  - In-plane deformation gradient
    - In-plane strain, rotation and their gradients
  - Spatial resolution: 0.2 µm



High resolution digital image correlation

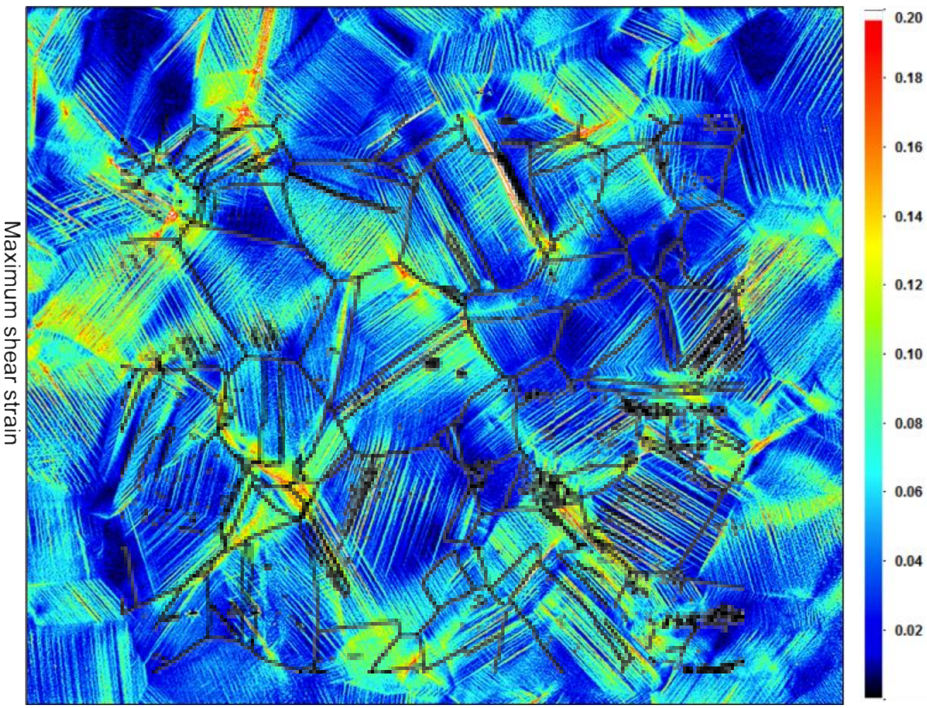
- Sub-micron spatial resolution strain maps

400 VECTORS PER 100×100 µm^2



Natural contrast

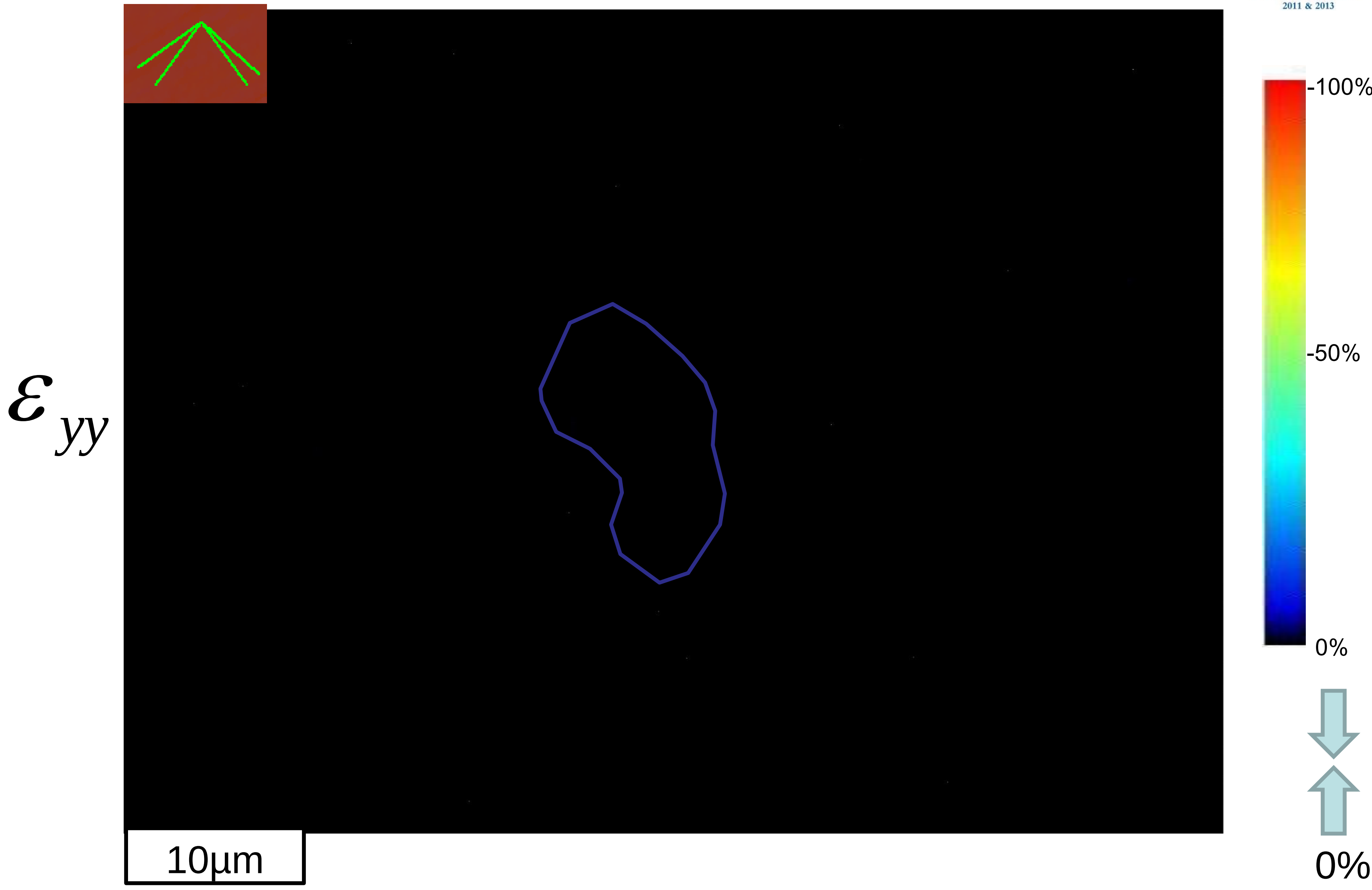
200,000 VECTORS PER 100×100 µm^2



Gold speckle

(LAVision DIC)

Al-Si Local strain evolution



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Al-Si Local strain evolution

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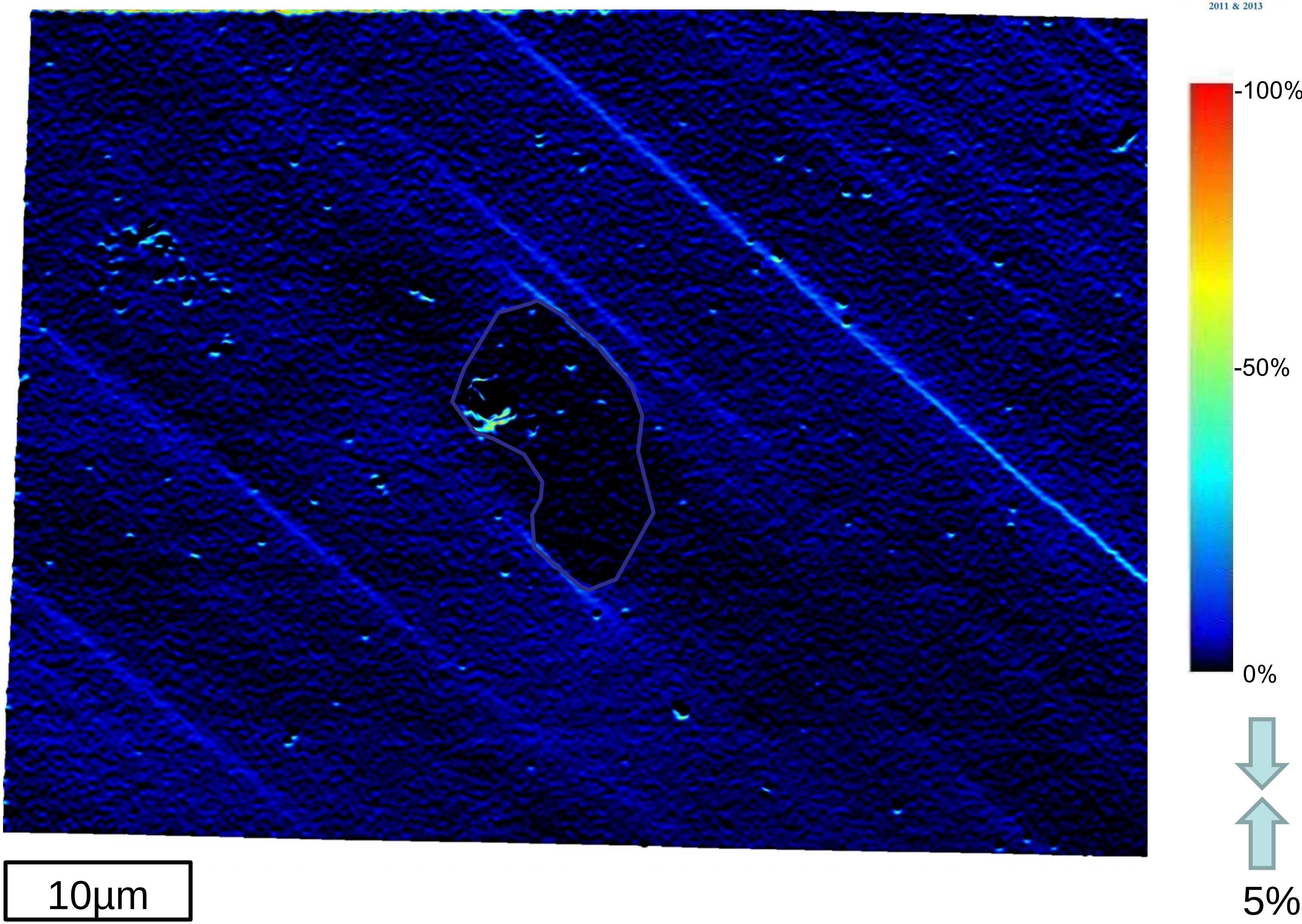
ε_{yy}



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Al-Si Local strain evolution

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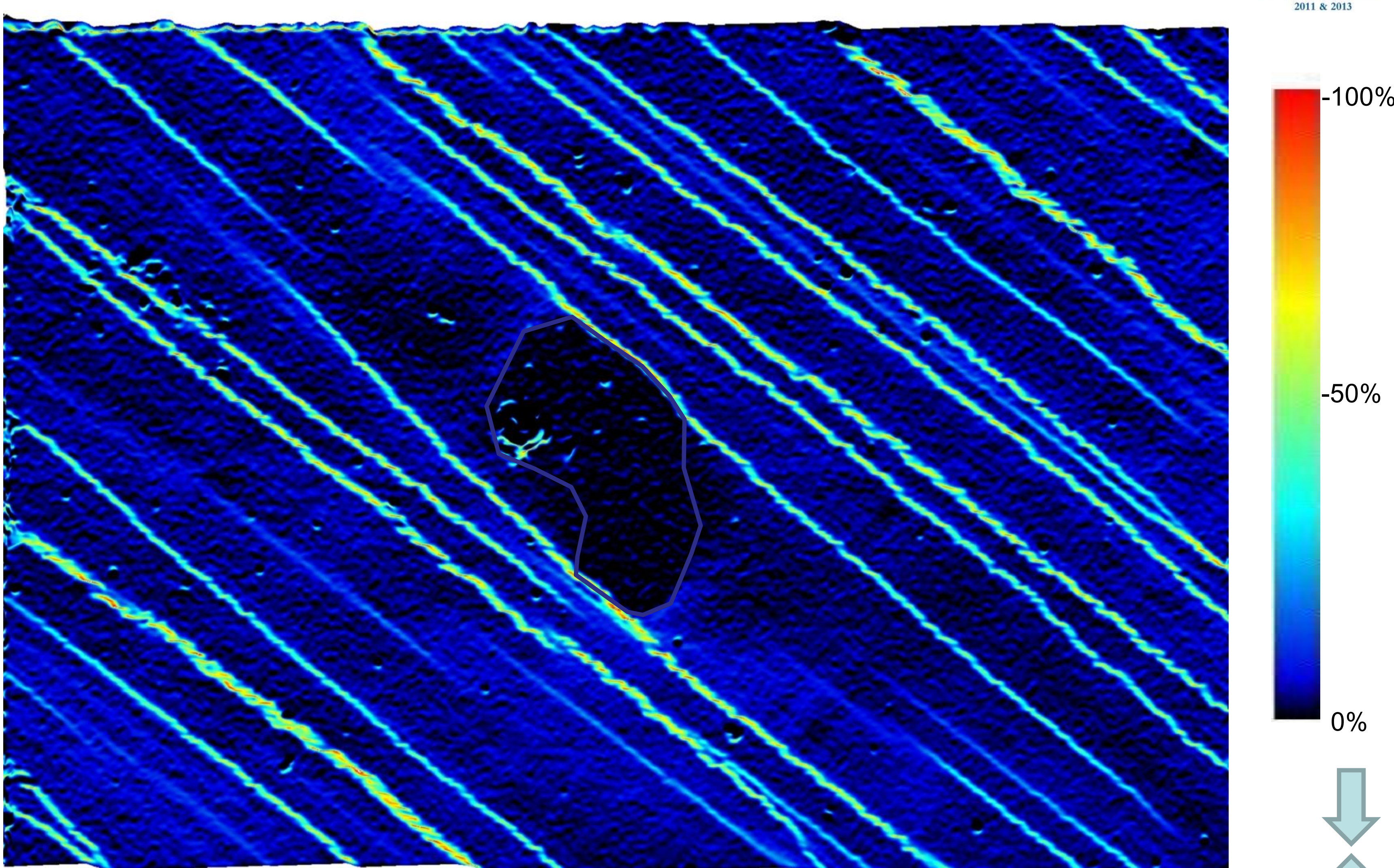
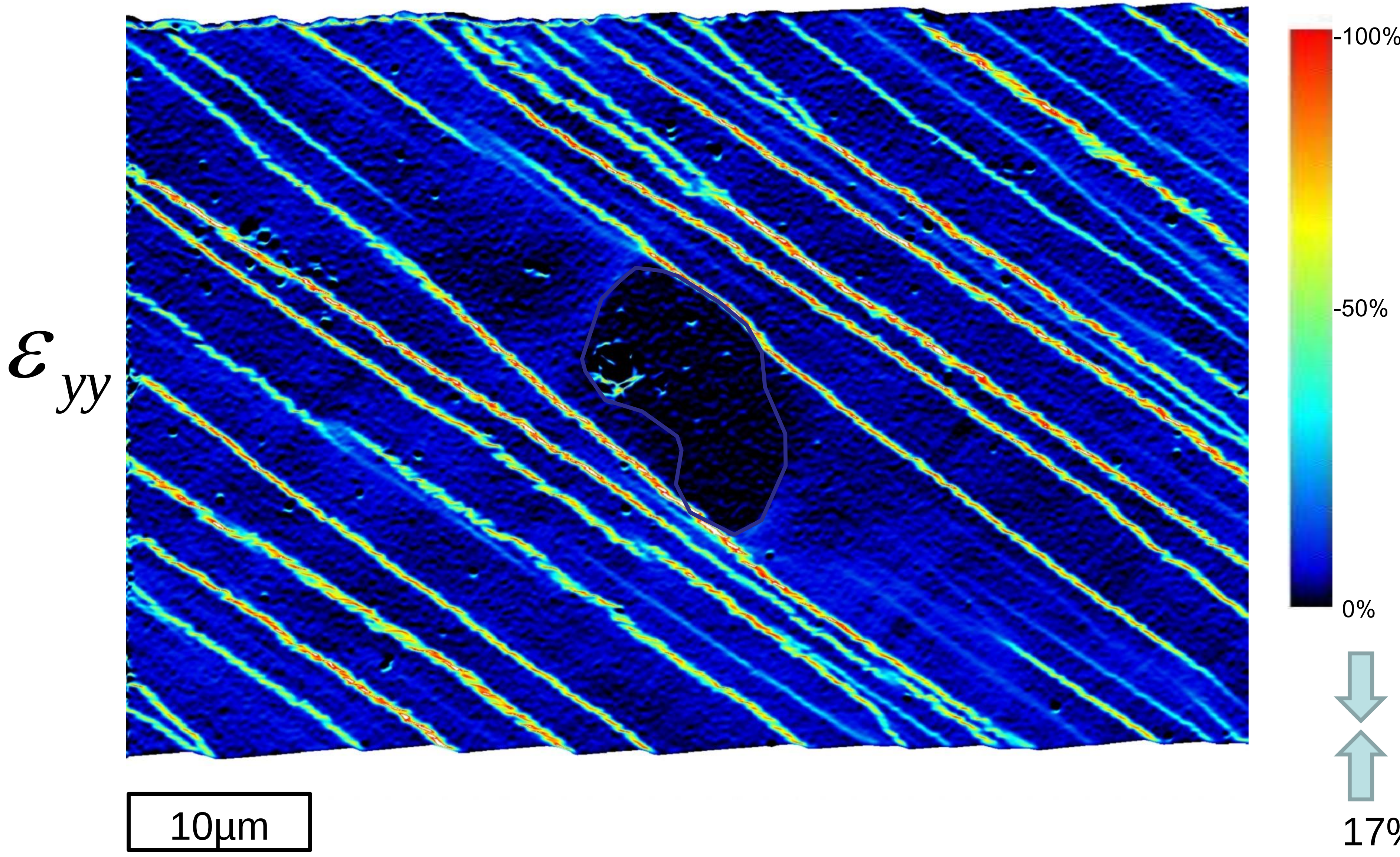
ε_{yy}



10μm

12%

Al-Si Local strain evolution



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Al-Si Local strain evolution

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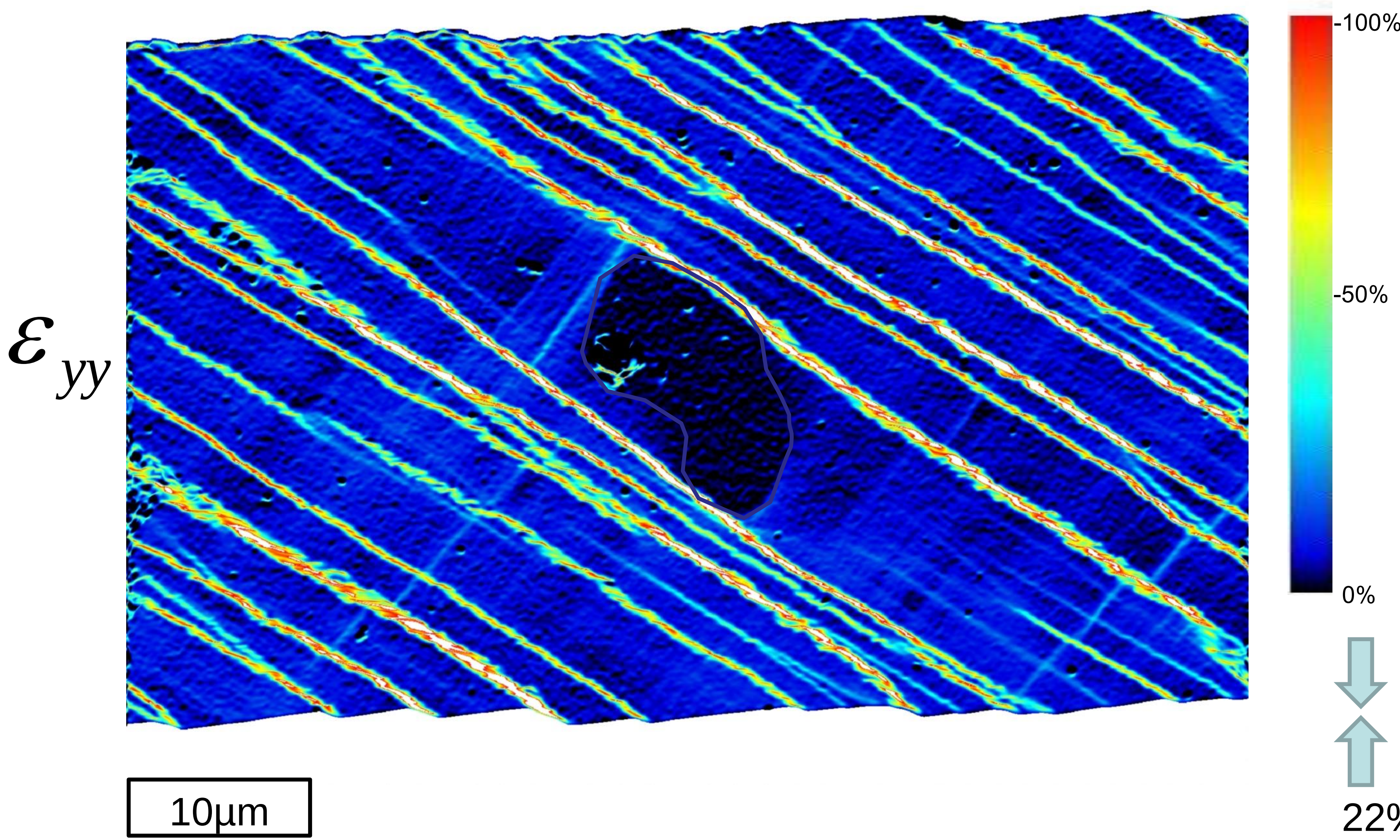
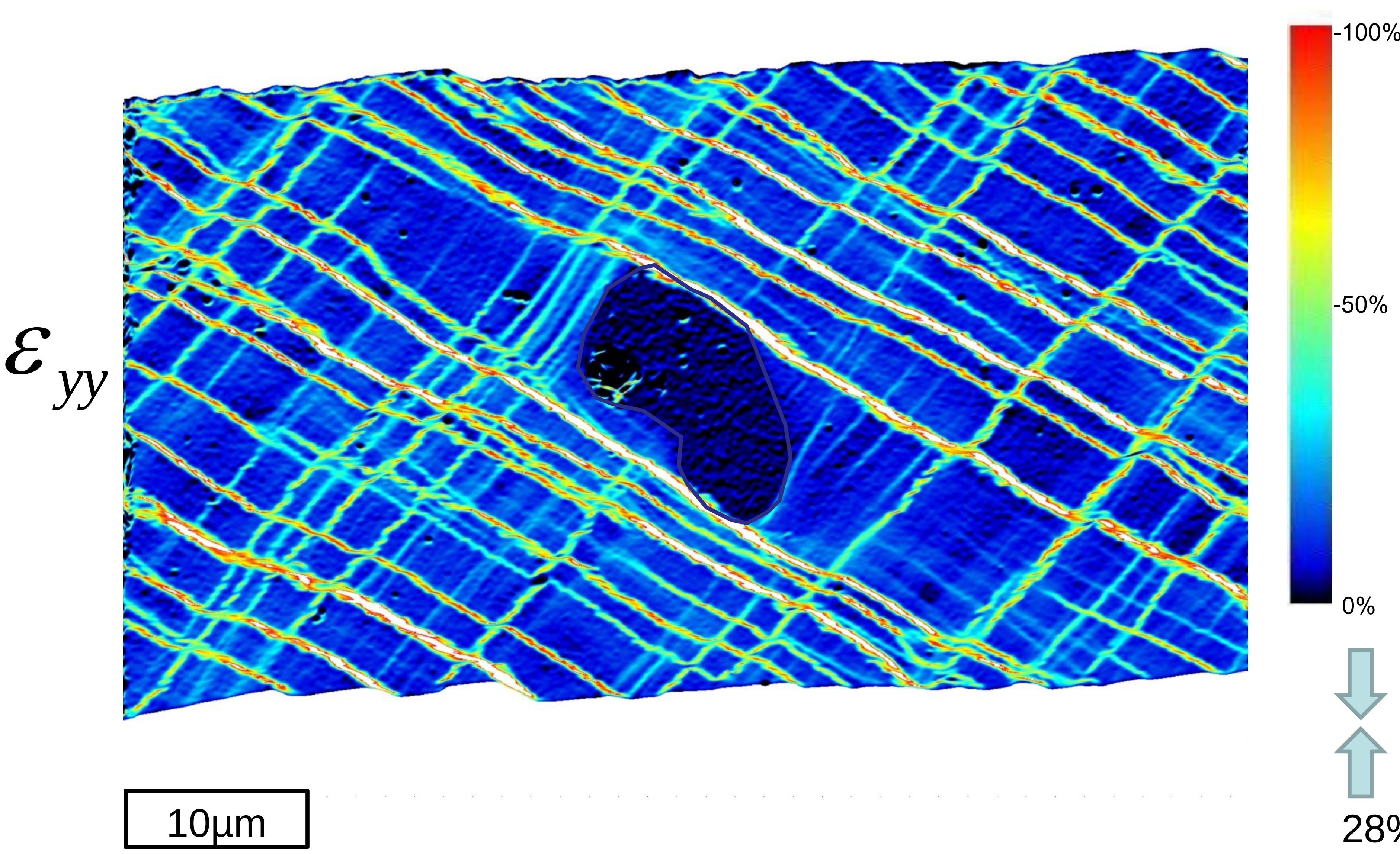
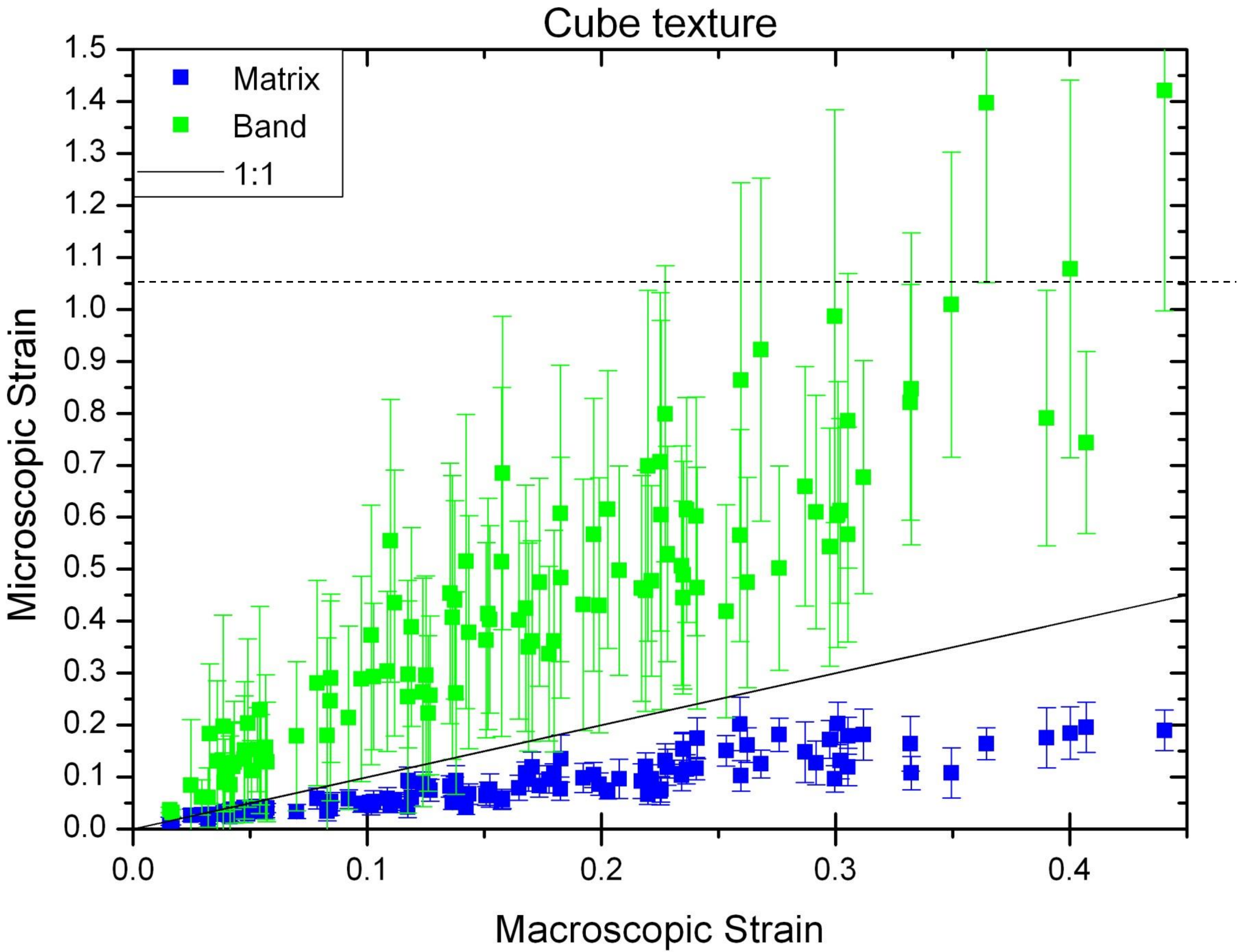


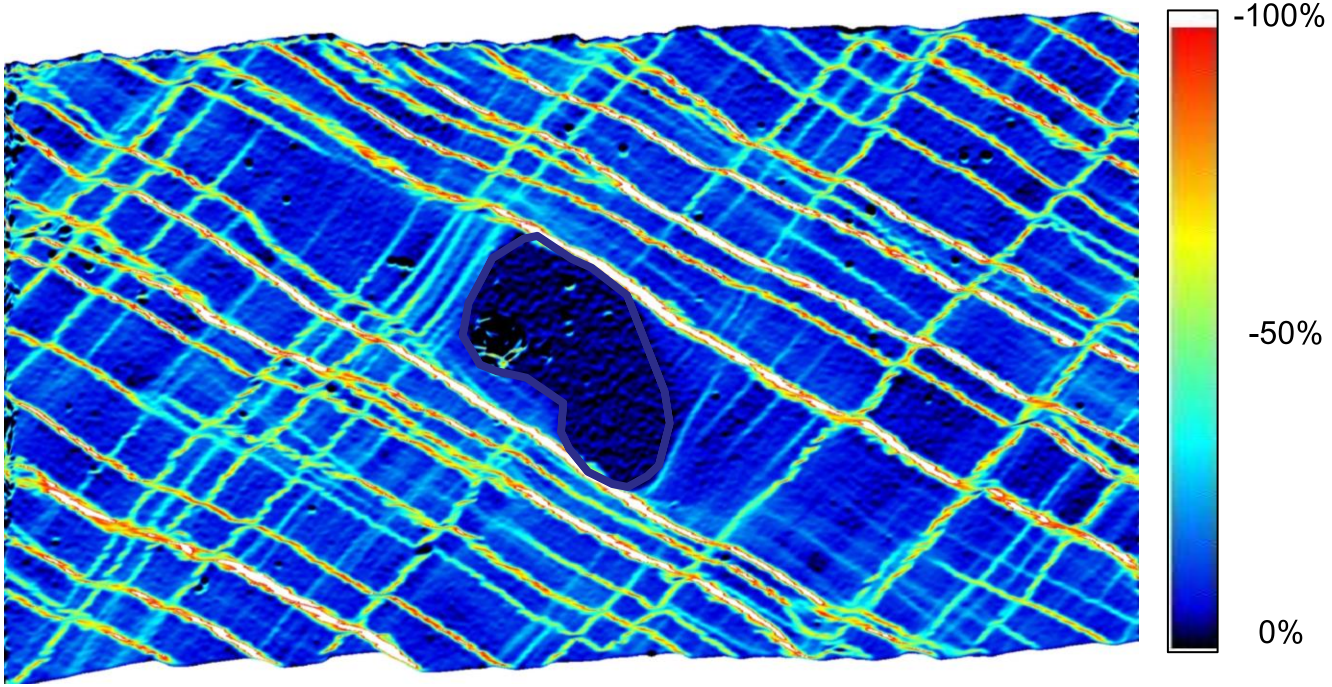
Al-Si Local strain evolution



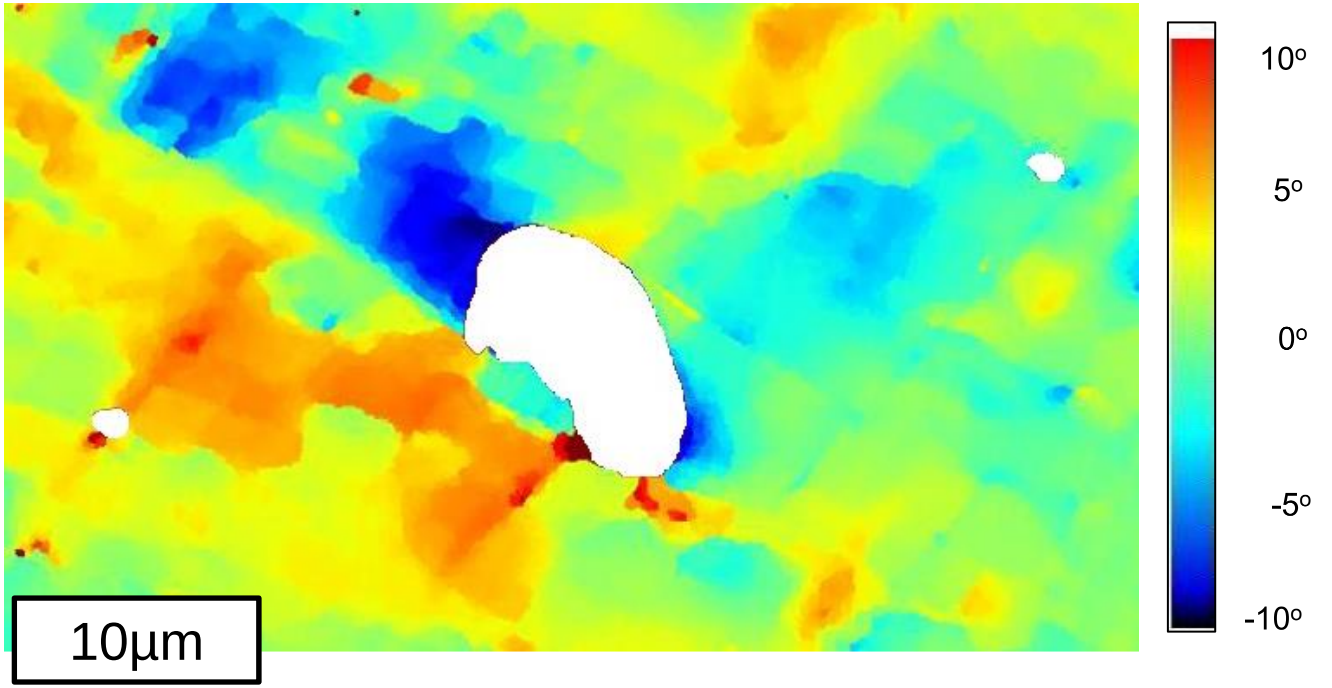
Al-Si strain heterogeneity – Bands vs Matrix



DIC strain

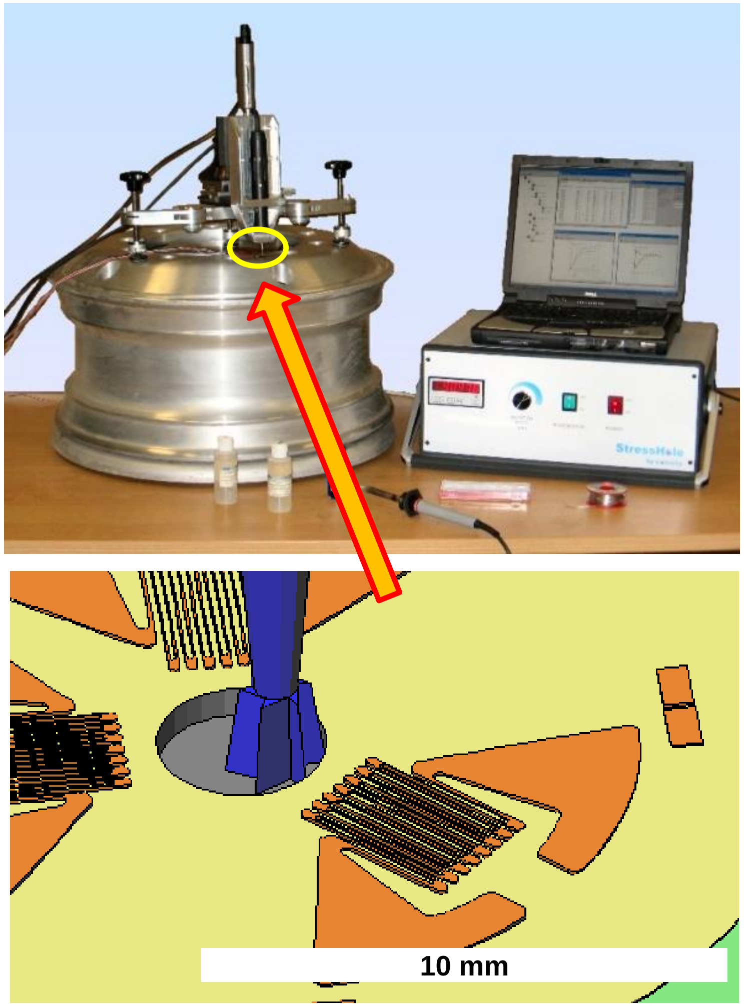


EBSD rotation



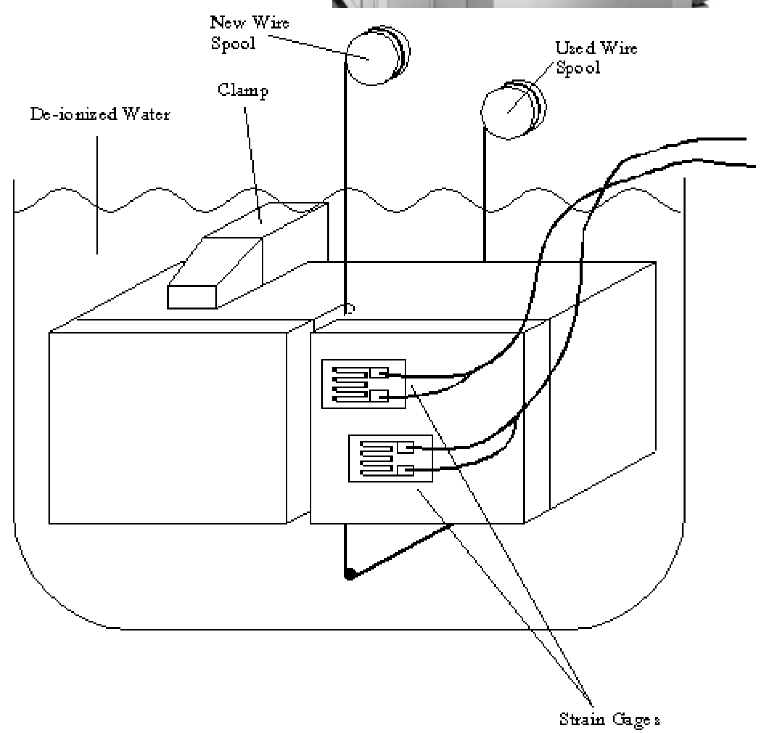
Stress can be measured at the mm scale…

IHD (incremental hole drilling)



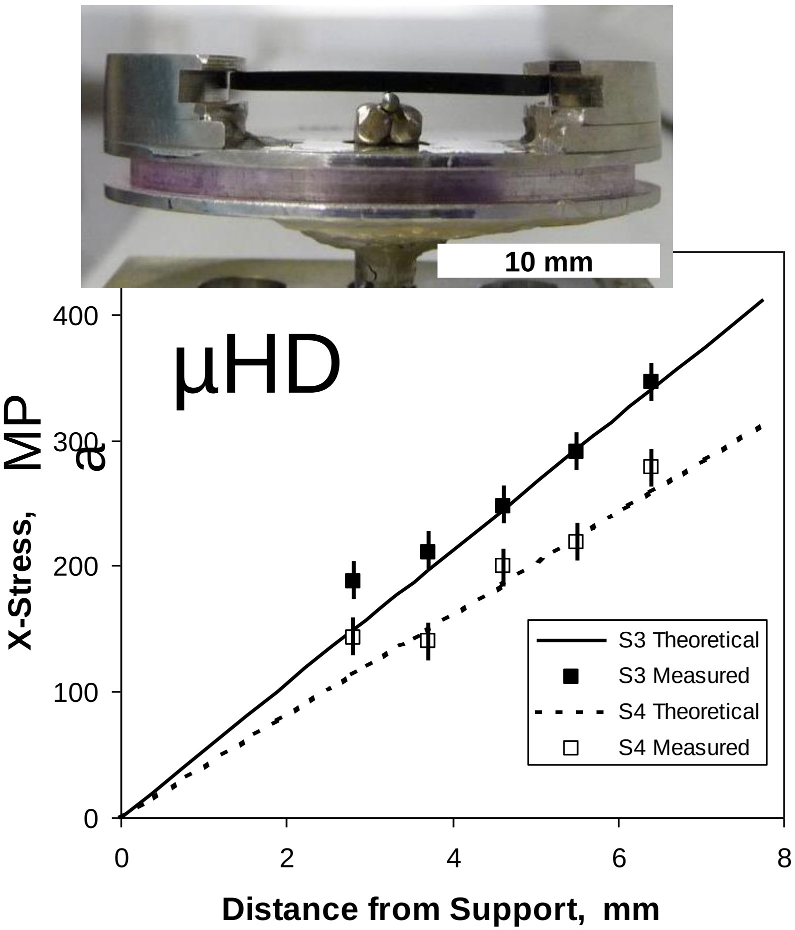
Gary S. Schajer (Uni. British Columbia, Canada)

Slotting (Crack Compliance)

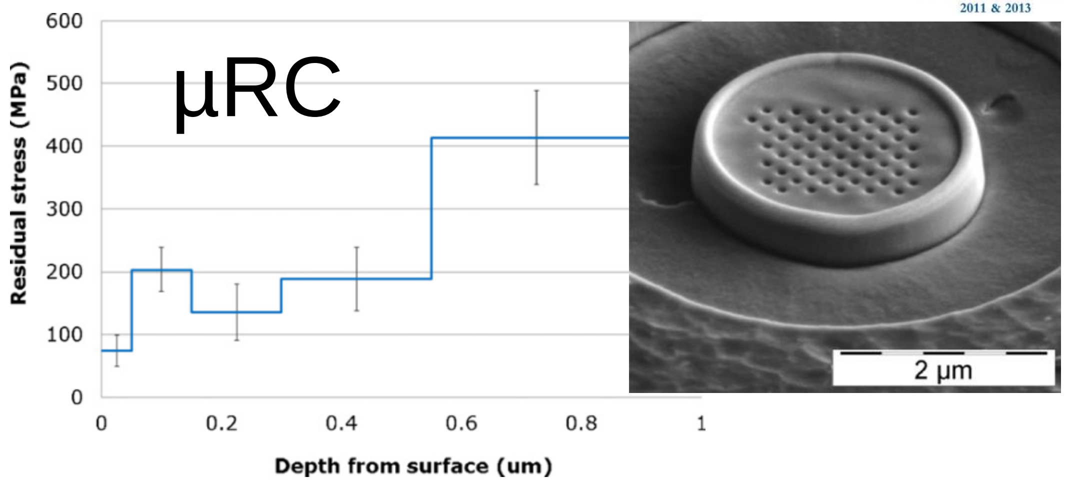


Michael B. Prime (Los Alamos National Lab. USA)

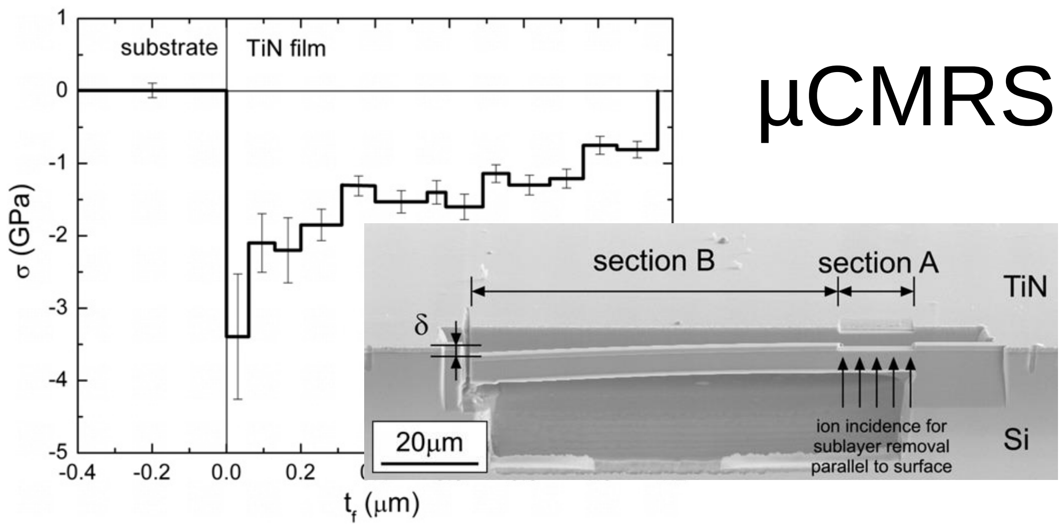
Novel relaxation methods in the micron scale



Schajer GS, Winiarski B, Withers PJ. **Exp. Mech**. 2012



Sebastiani M et al. Mat. Sci. Eng. A 528 (2011) 7901– 7908

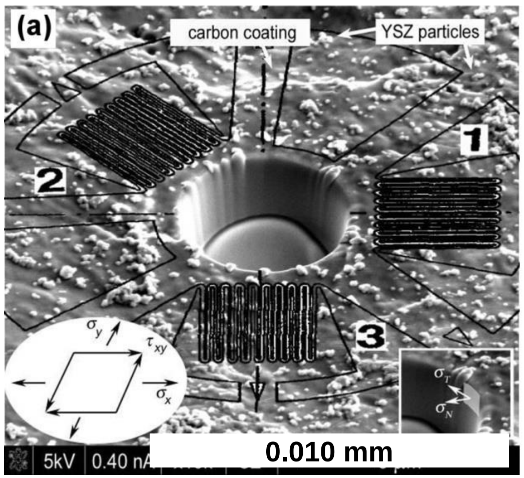


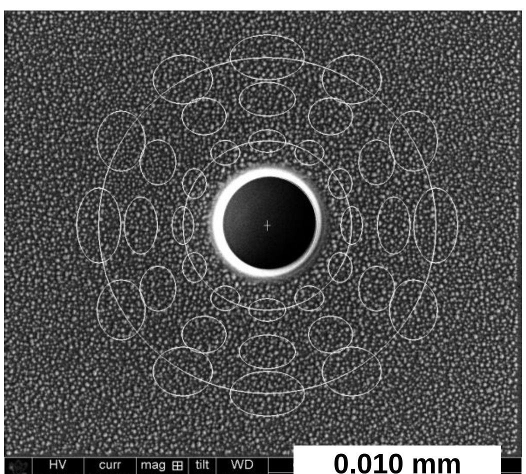
Massl S. et al. Thin Solid Films 516 (2008) 8655–8662

Mapping stress at the micron scale…

$$\varepsilon_x(H) = \int_{h_1}^{h_2} g(H,h)\sigma_x(H)dH$$

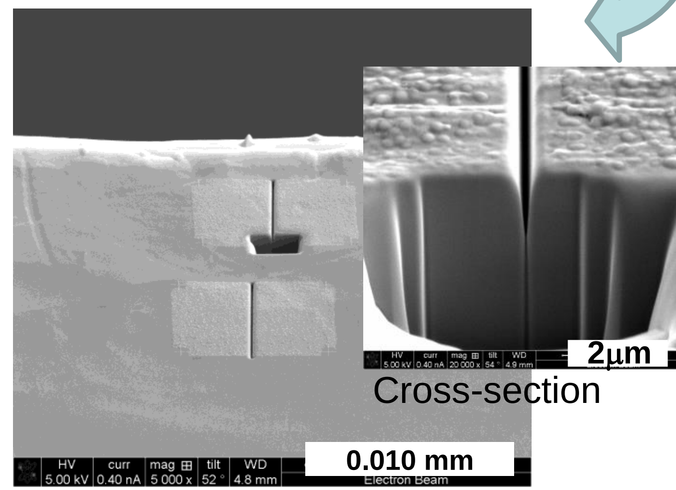
Micro-hole drilling

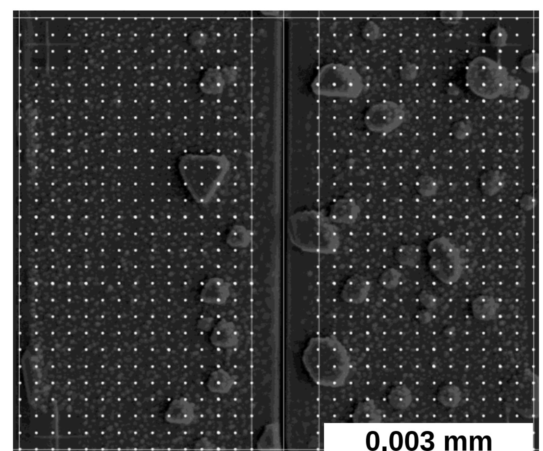




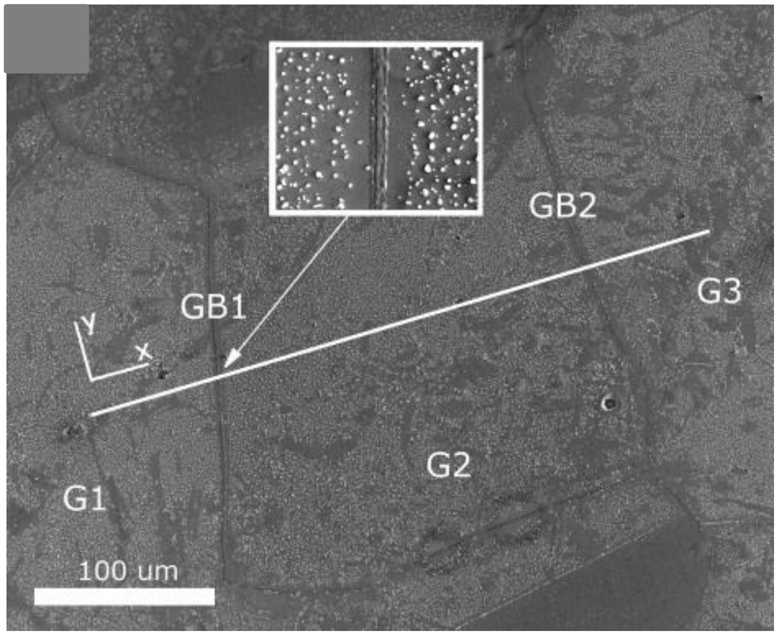
$$\varepsilon_x(H) = \int_{h_1}^{h_2} g(H,h)\sigma_x(H)dH$$

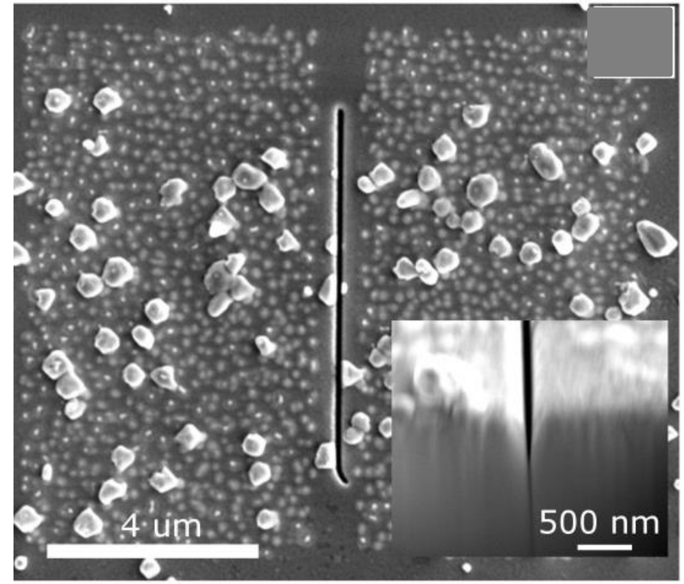
Micro-slotting





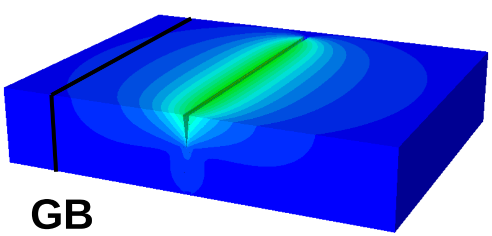
Mapping stresses within Alloy 600 grains

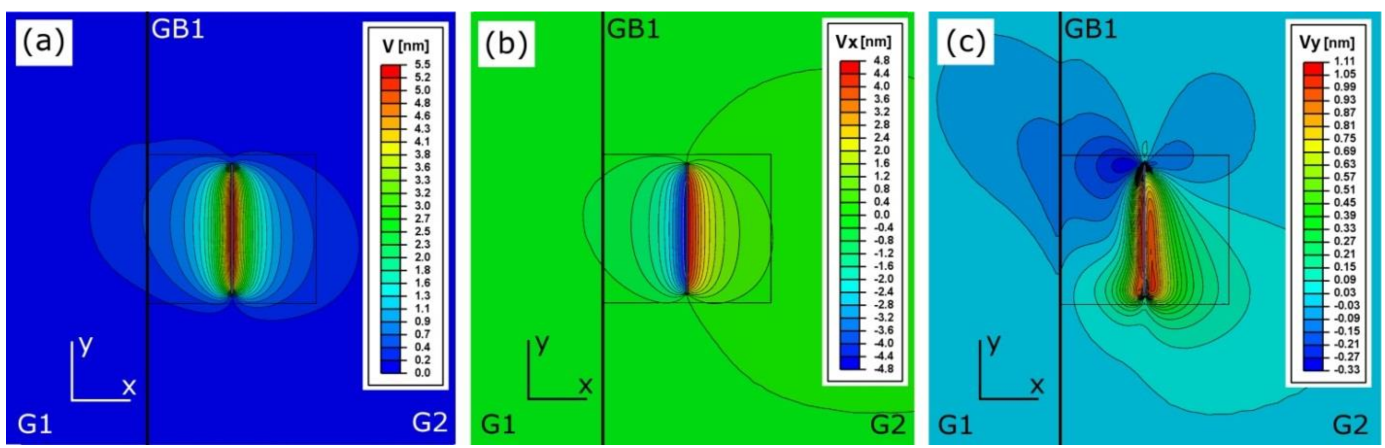




Alloy 600 subjected to low pressure hydrogenated steam

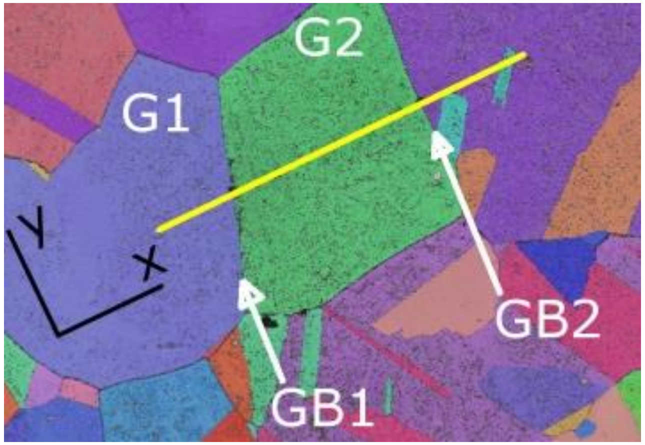
3-D FEA model

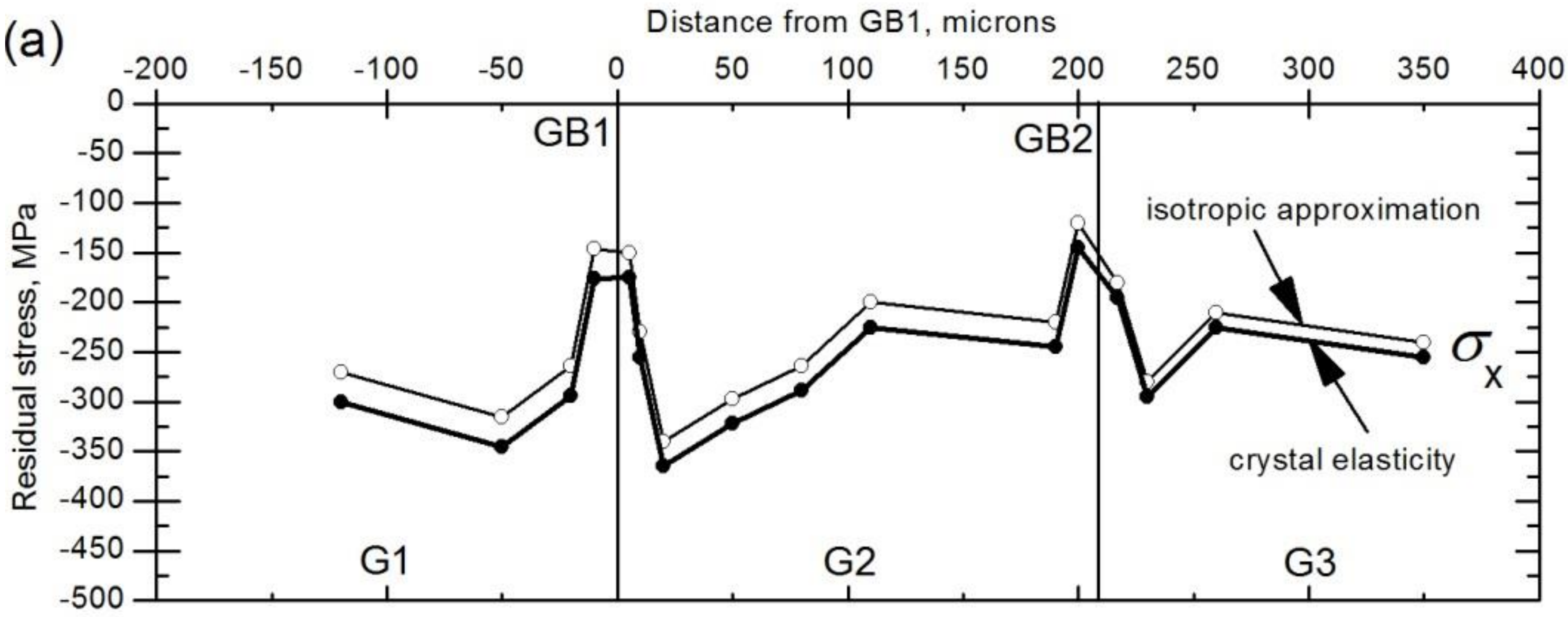




Intragranular RS in Alloy 600

(micro-slotting)





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Mechanical characterisation at the hundreds of nanometers scale……

Nanoindentation

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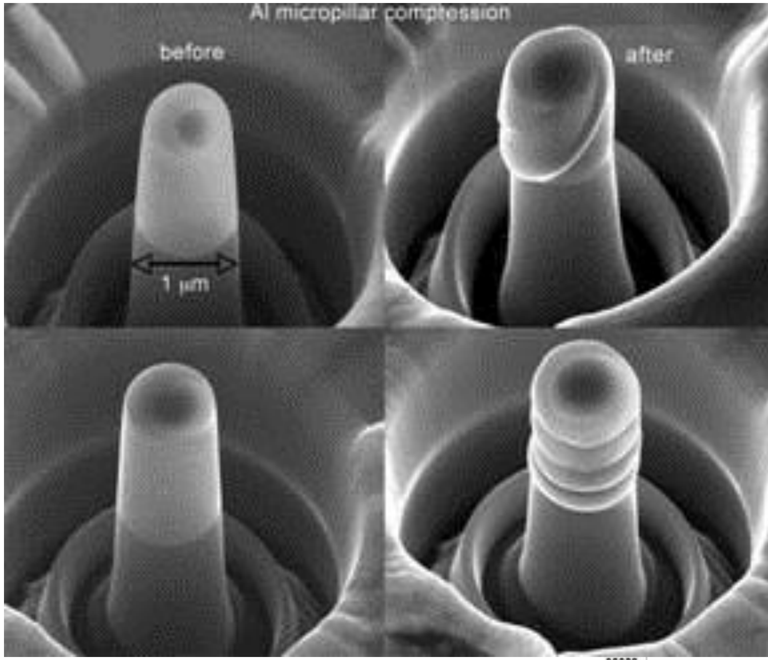
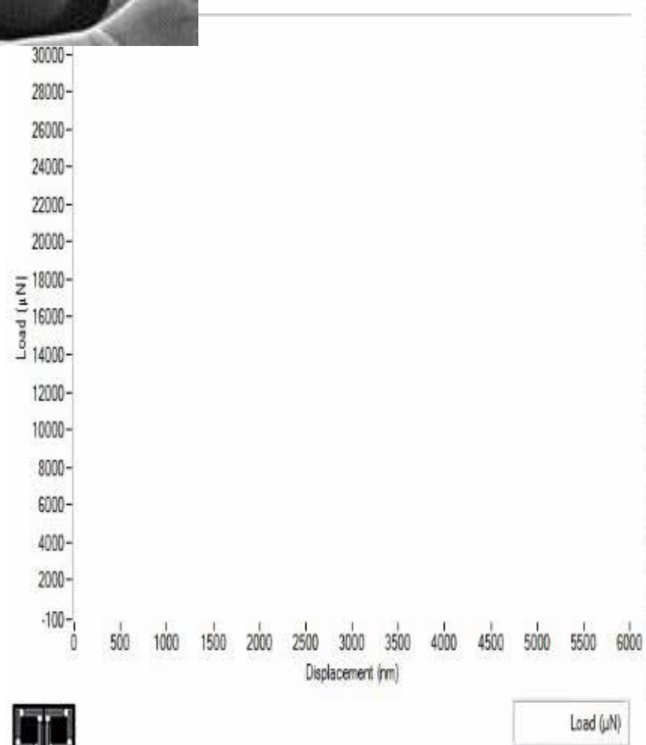
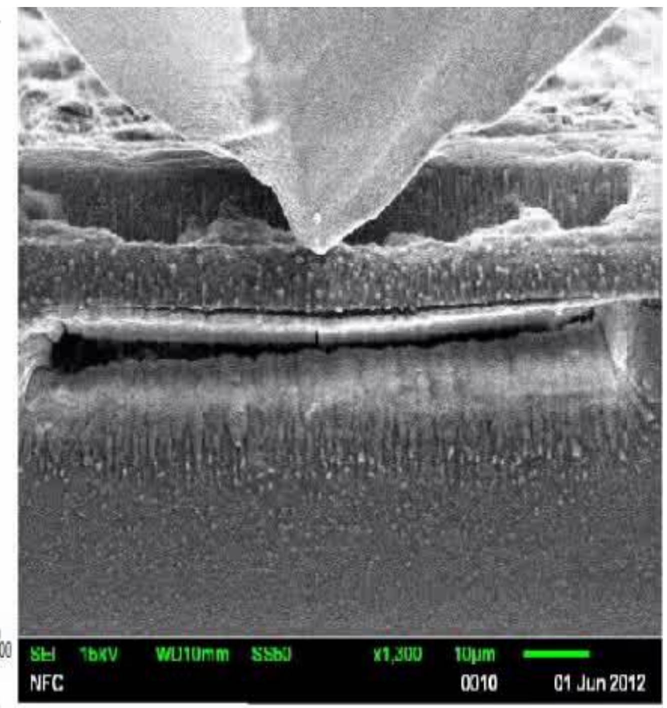


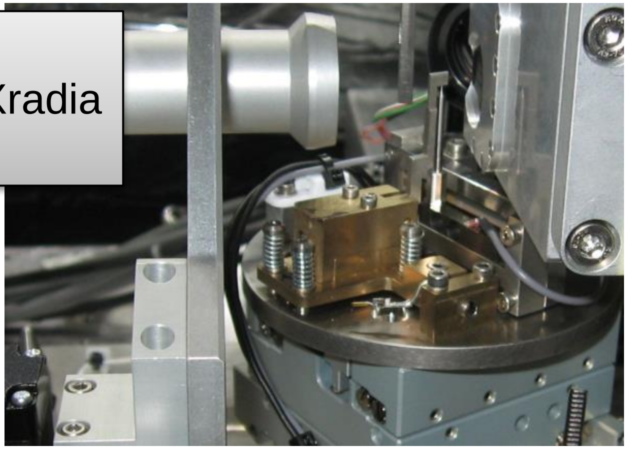


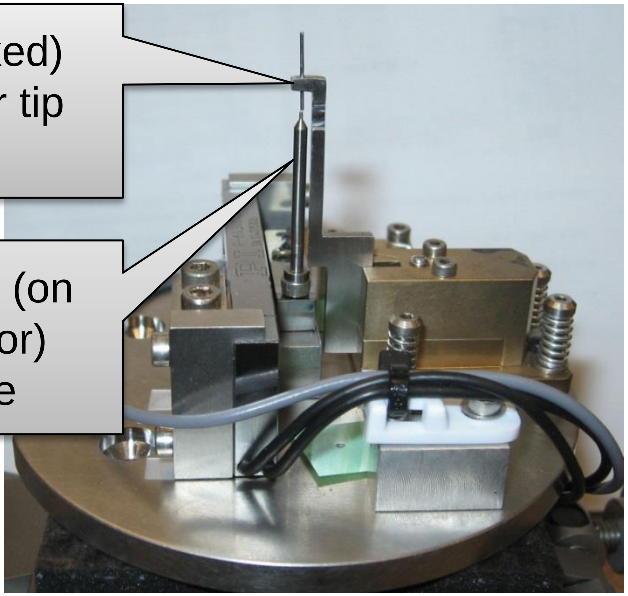
HYSITRON™



X-ray Nanotomography with *in situ* loading

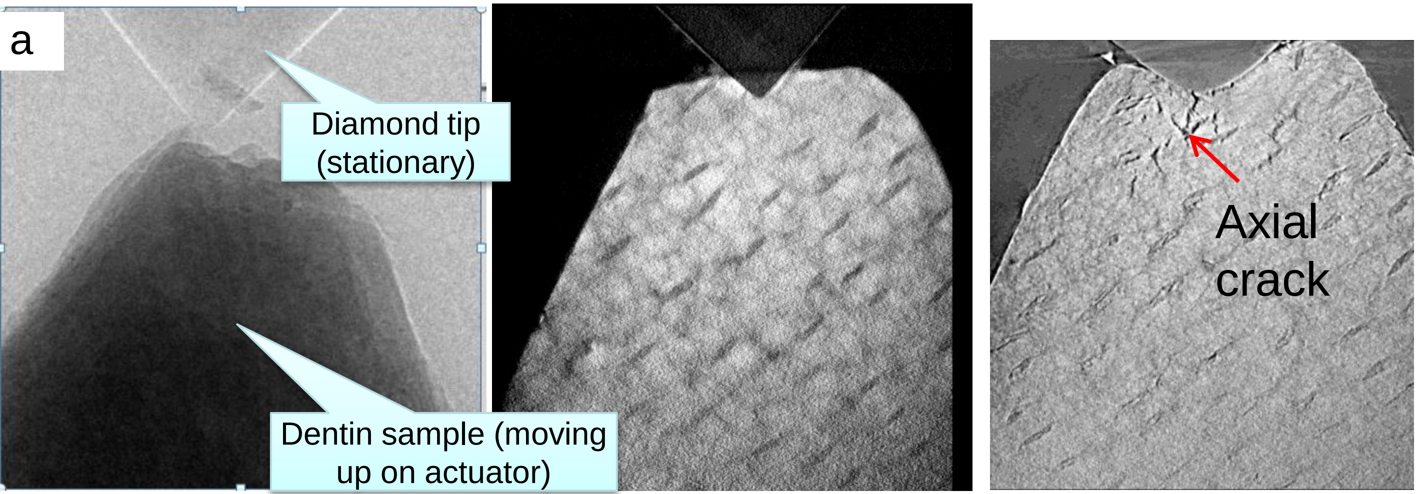
- Mechanical load cell with three operating modes
  - Compression
  - Indentation
  - Tension
- Compatible with Xradia 800/810 Ultra (incl. UltraXRM-L200, nanoXCT-200)
- Preliminary specs:
  - Piezo actuator for closed loop displacement control
  - Strain gauge force sensor, <0.1% sensitivity
    - Option 1: 9 N max force
    - Option 2: 0.8 N max force
  - Allows +/-70° tomography in Xradia Ultra
  - Customizable anvil tips for different operating modes, sample types and experimental designs
- Prototype currently in test phase



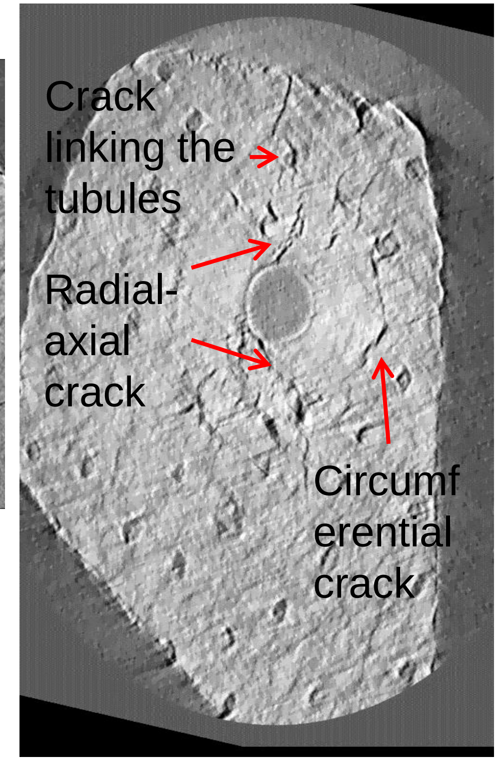


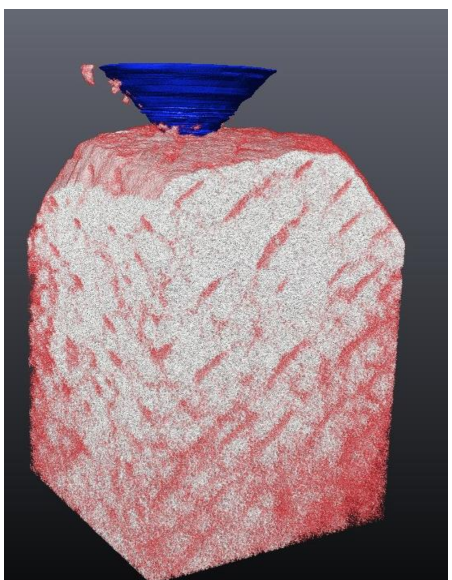
Xradia Ultra load cell – initial results:
In situ indentation / cracking of dentin

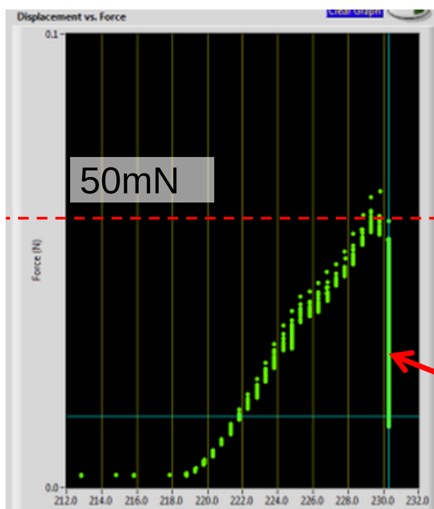
Diamond tip landing on Dentin (2D series, phase contrast, 65 um FOV)



Horizontal section

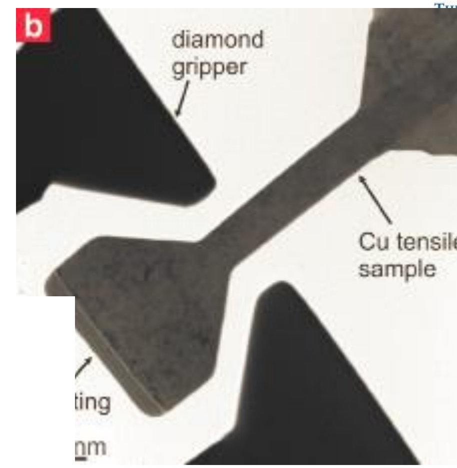


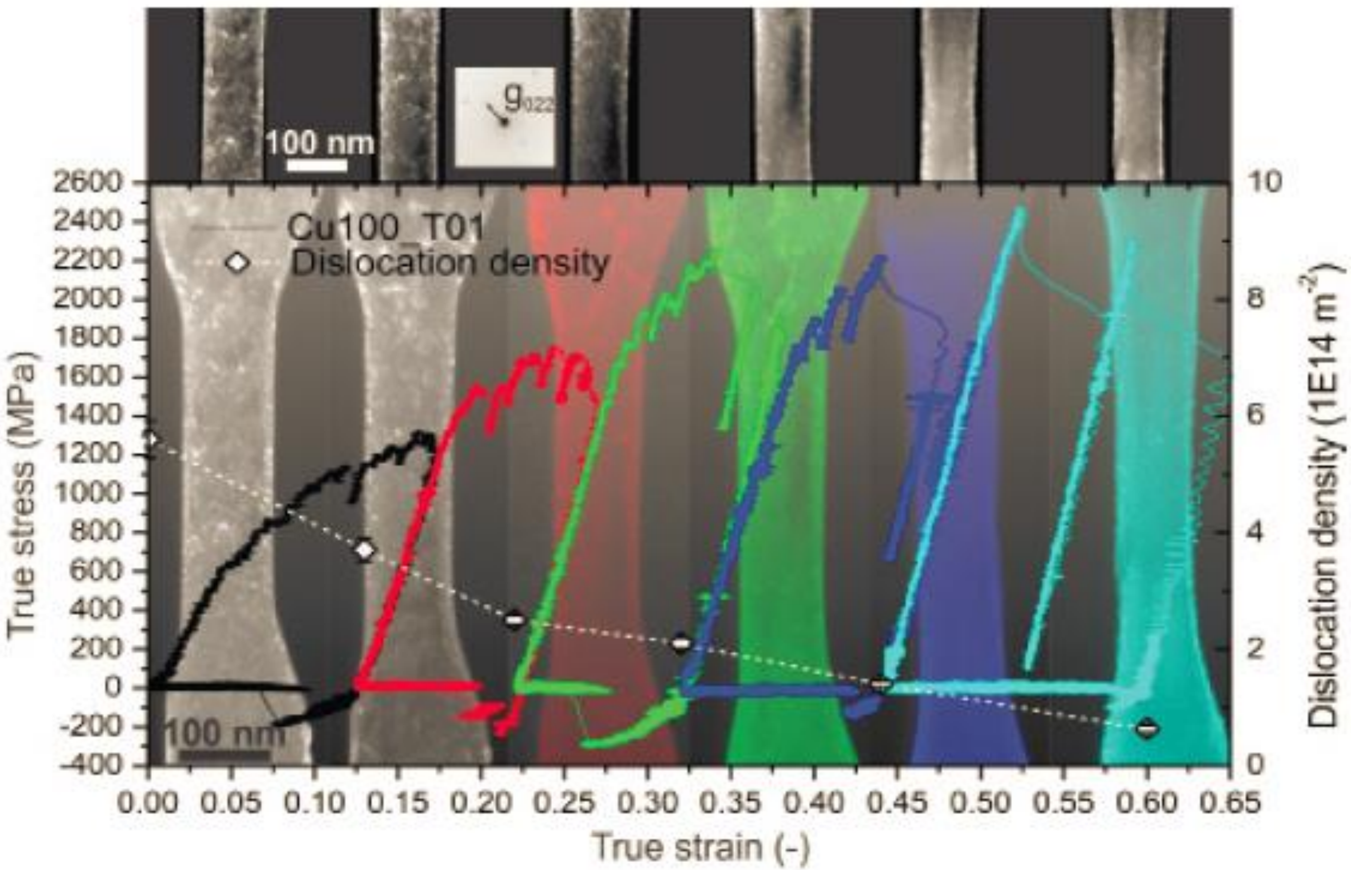




Force–displacement curve: Abrupt fracture event occurred here (c)

Loading in situ within TEM





Courtesy Hysitron

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Concluding remarks

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- Now able to track the same region of interest across time and multiple length scales
- Using a correlative multi-scale framework can now determine for the same region of interest the mechanical properties as a function of length scale
  - Better understanding of microstructure property relationships
  - Better understanding of how micro-structuring can be used to control properties
- Inverse/Virtual fields methods may provide rich spatially variant datasets without the need for idealised test geometries

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Acknowledgements

FEI : Tim Burnett, Remco Geurts, Pybe Faber, Carmen van Vilsteren, Michael Janus, Daniel Lischau, Pascal Doux, Bert Freitag, Trisha Rice

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NPL: Ken Mingard, Mark Gee

UoM : Sam McDonald, Ali Gholinia, Dirk Engelberg, Tristan Lowe, Robert Bradley, Teruo Hashimoto, Tom Slater, Sarah Haigh, Grace Burke, George Thompson, Philip Withers

BP: Ellen Williams

Open University: Hedieh Jazaeri, Shirley Northover, John Bouchard

Luxfer- Henry Holroyd

Innoval- Geoff Scamans

Paper on correlative tomography: http://www.nature.com/srep/2014/140416/srep04711/full/srep04711.html

Video on pitting example: https://www.youtube.com/watch?v=P5oUpiVvZVY

More information: www.imaging.manchester.ac.uk