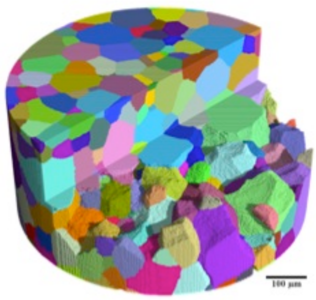
Mapping polycrystalline materials in 3D:

Diffraction contrast tomography and related techniques



Andrew King

Synchrotron SOLEIL

W. Ludwig, P. Reischig, L. Nervo, S. Schmiederer, N. Viganò, Y. Guilhem, G. Johnson and many others…

Psiché

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Outline

- **Introduction**
- Motivation
- Underlying techniques
- Diffraction contrast tomography
  - Setup, data acquisition and processing
- Application examples
- Developments in progress
- Related techniques

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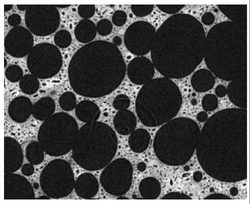
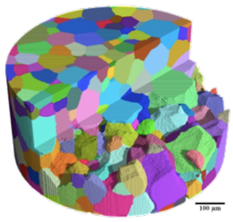
Myself

- Diffraction for strain, stress and damage characterisation
  - Manchester 2001-2005
- Tomography for 3D morphology of porous and granular materials
  - INSA de Lyon 2006
- *Diffraction Contrast Tomography for mapping polycrystals and materials applications.*
  - *Manchester/ESRF/HZG 2009-2013*
- At SOLEIL since 2013…
  - PSICHE beamline, tomography for materials science





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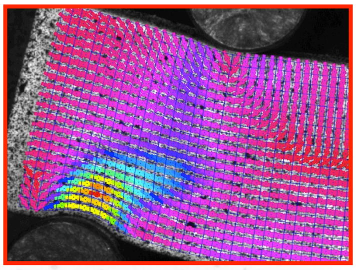
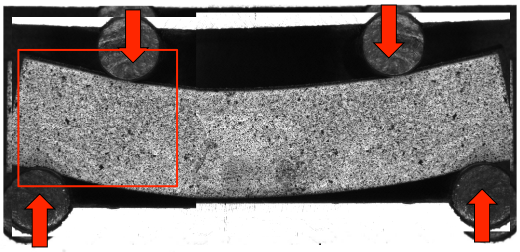
What are we trying to do, and why?

- Imaging materials
  - How are they structured?
  - How does the structure determine properties and behaviour?
  - How does the structure change during use?



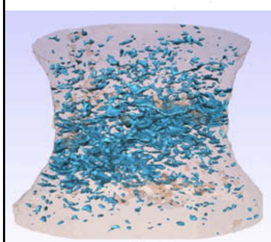


- This covers a lot of materials science!

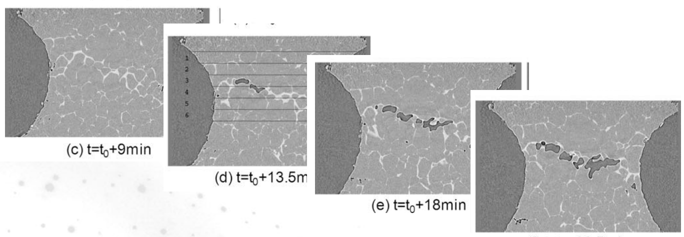
More specifically

- **Polycrystalline materials**
  - Many important and everyday materials

- **3D, in-situ measurements**
  - Most "real" problems are 3D
  - Need techniques to *see inside* evolving systems



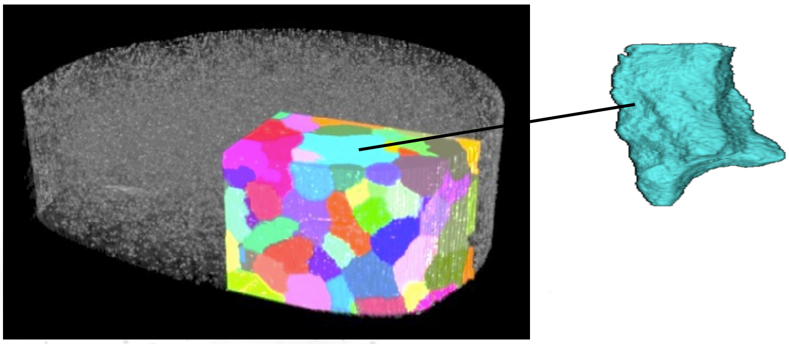
$t=t_0+9.9$min



(c) $t=t_0+9$min (d) $t=t_0+13.5$m (e) $t=t_0+18$min (f) $t=t_0+22.5$min

Diffraction Contrast Tomography

- X-ray technique
- Tomographic concepts and algorithms
- 3D, non-destructive mapping of:
  - Grains (shapes, orientations…)
  - Sample structure (cracks, pores, inclusions…)



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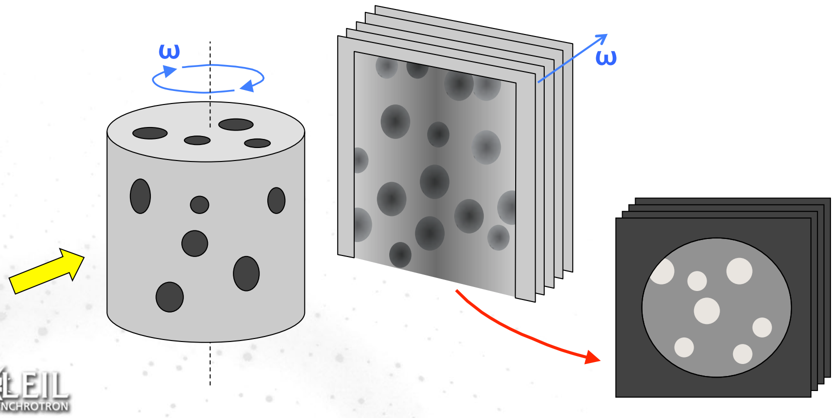
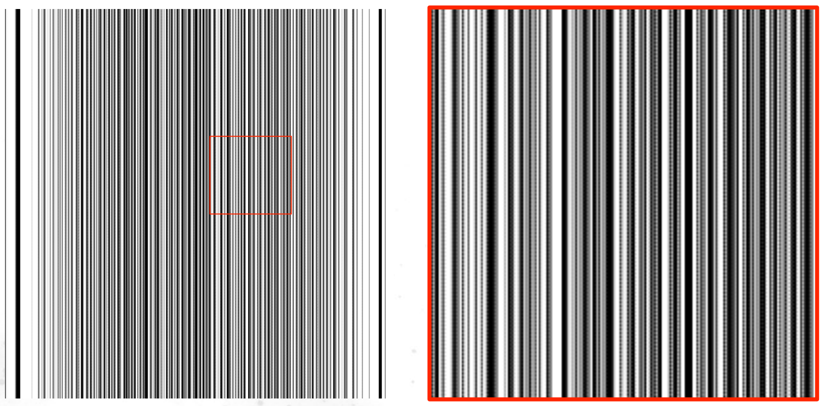
Standard tomography

- Absorption or phase (refraction) contrast
  - Full field radiography + rotation
- Reconstruction of ~density



Standard tomography: Reconstruction

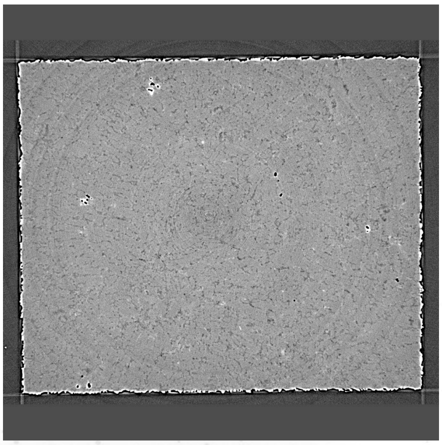
- Filtered back projection
  - most standard, basic reconstruction technique
- 1 / 2 / 4 / 8 / … / 1024 / 1800 projections

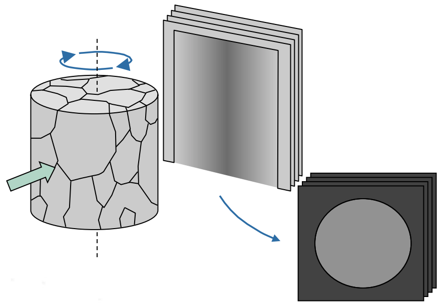


Aluminium alloy for DVC. M. Fregonese, MATEIS, INSA de Lyon

Standard tomography: Reconstruction

- Can see pores and precipitates, but not grains

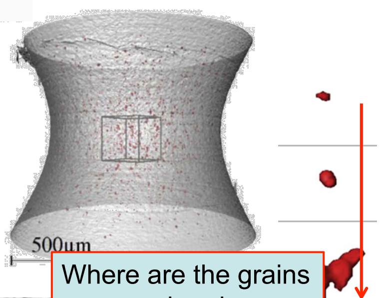




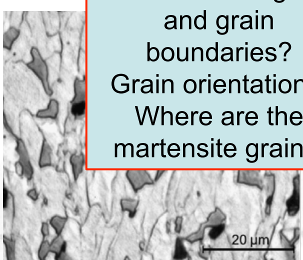
Aluminium alloy for DVC. M. Fregonese, MATEIS, INSA de Lyon

Recent tomography work

- Ductile failure of dual phase steel

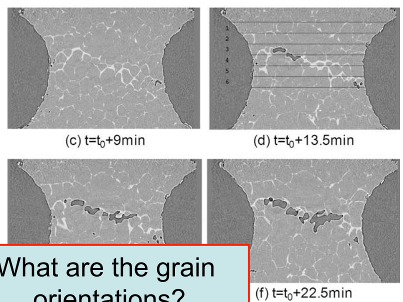


Where are the grains and grain boundaries?
Grain orientations?
Where are the martensite grains?

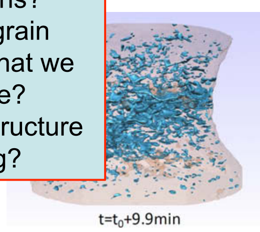


C. Landron *et al*, *Acta Mat.* **59** (2011) 7564-7573

- Hot tearing in Al-Cu



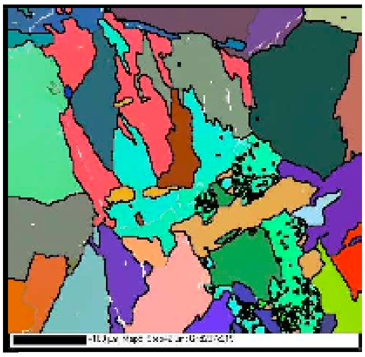
What are the grain orientations?
Are there grain boundaries that we can't see?
Is the grain structure evolving?

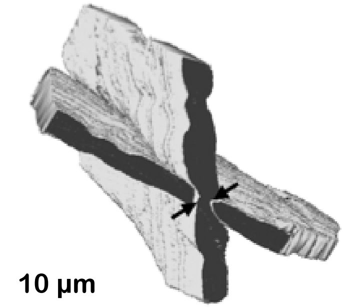


S. Terzi *et al*, *Scripta Mat.* **61** (2009) 449-452

Mapping grains

- Scanning electron microscope technique electron backscatter diffraction (EBSD)
- Electron penetration is very limited (<1μm)
- Good 2D mapping
- Difficult to extend to 3D, and only possible destructively.

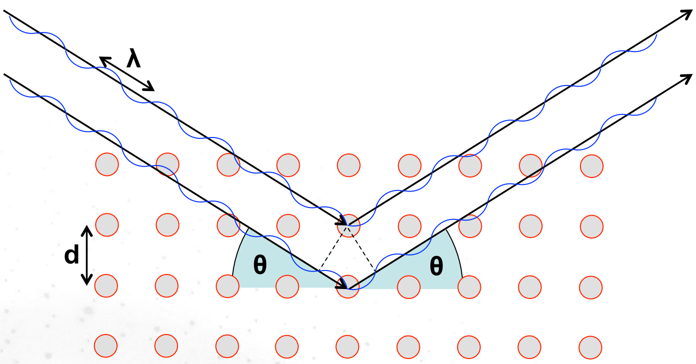




How to see the grains?

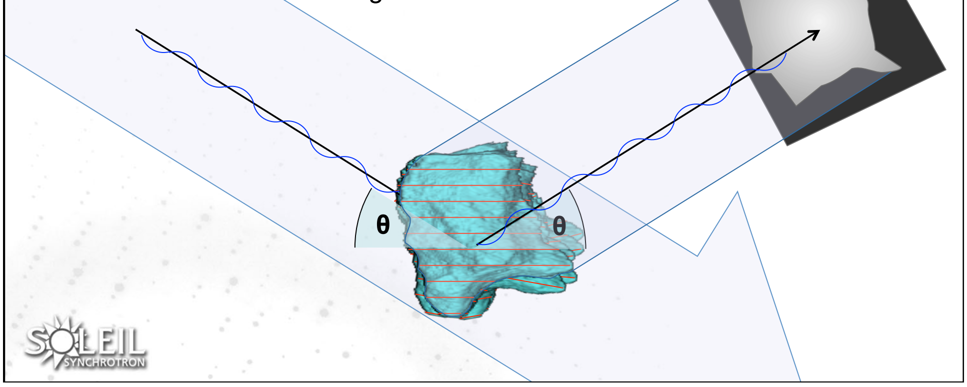
- Absorption and refraction, but X-rays also **diffract**
- Scattering of radiation from periodic structures (crystals)

Bragg's law: $\lambda = 2d \sin(\theta)$



Diffraction

- Monochromatic radiation
  - discrete values of **θ** corresponding to $\mathbf{d}_{hkl}$
- Only occurs when crystal is aligned correctly
  - angle **θ** between incident ray and crystal plane
- For us: All parts of the crystal volume diffract with same efficiency
  - “kinematic scattering”



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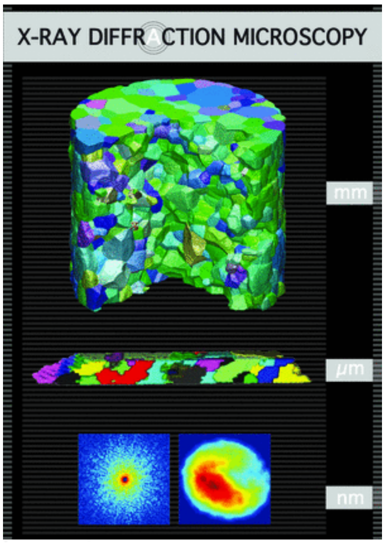
Using X-ray diffraction for grain mapping

- A number of techniques use diffraction of x-rays to image (poly-) crystals
- This talk will concentrate on diffraction contrast tomography
- Talk tomorrow on scanning microscopies – M. Cotte.



Journal of Applied Crystallography, special issue March 2013

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- **Diffraction contrast tomography**
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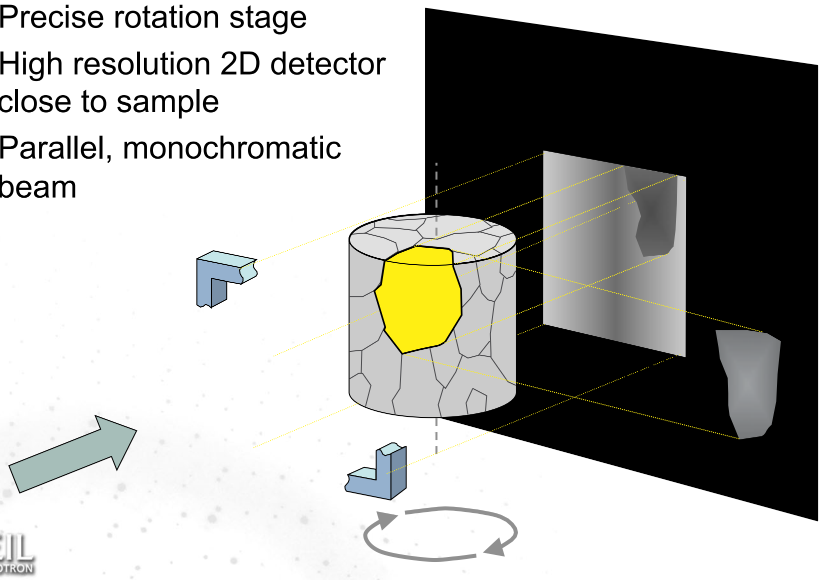
Diffraction contrast tomography

- Setup:
  - Precise rotation stage
  - High resolution 2D detector close to sample
  - Parallel, monochromatic beam



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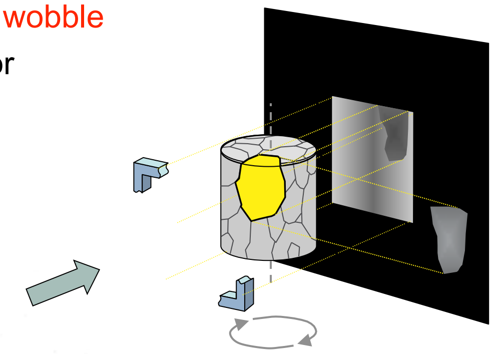
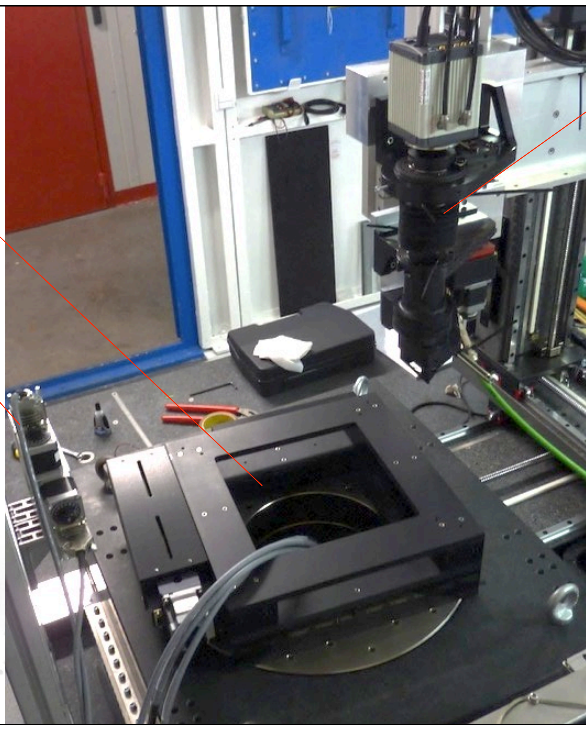
Diffraction contrast tomography - requirements

- Precise rotation stage
  - submicron eccentricity, wobble
- High resolution 2D detector close to sample
  - compact design
  - high dynamic range
- Parallel, monochromatic beam
  - synchrotron source

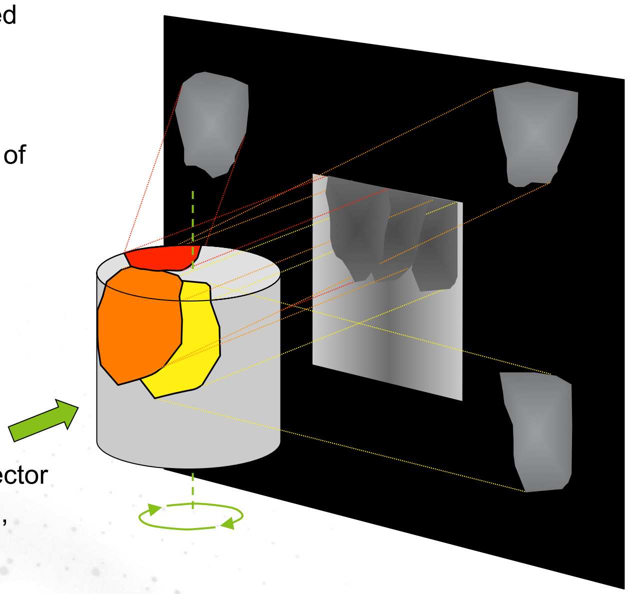


Instrument



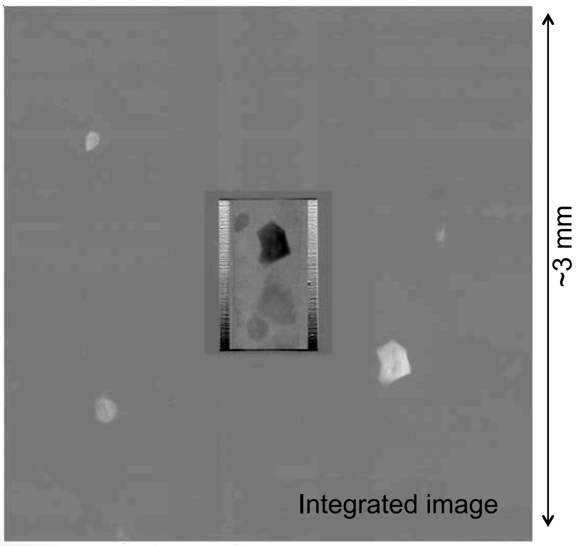
Data acquisition

- 360° rotation, integrated images of 0.05°-0.1°
- Near-field
  - Spots approximate parallel projections of grains
- 50-100 spots per grain
  - Reconstruct grain shapes
- Diffraction geometry
  - Crystallographic orientations
- Absorption contrast image in center of detector
  - Reconstruct cracks, pores, etc



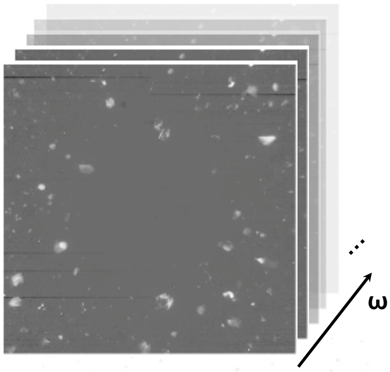
DCT: Closer look at data

- Titanium sample under load, 130 grains
- 0.05° increments
- Showing the diffraction contrasts only
- Strain causes distortion and misorientation in grains
- Causes diffraction spots to spread over several images
- Can also see extinction spots, but significant overlap problems – useful in special cases



DCT: Data processing 1

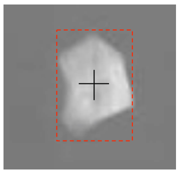
(1) Acquisition



- 3600-7200 images covering 360°.
- Remove background intensity to leave only diffraction contrast.
- Moving median filter

(2) Segmentation

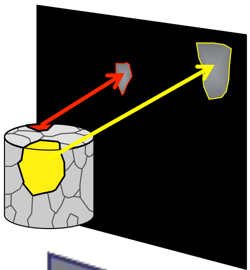
- Identify individual spots
- Spots are 3D objects, because they may spread over a few adjacent images
- 100,000+ spots
- Reduce data volume from 30-60 GB
- Record metadata:
  - **Position (u,v,ω)**
  - **Intensity**
  - **Size (u,v,ω)**
  - **Integrated image**
  - …

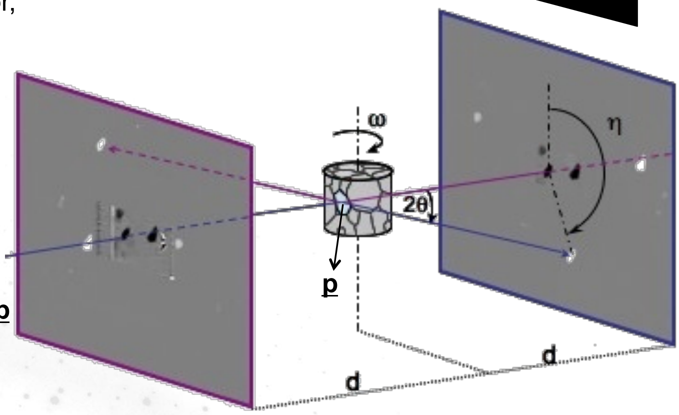


DCT: Data processing 2

(3) Extract diffraction geometry
Pair Matching

- Important step
- With a small, high resolution detector, the spot position does **not** directly tell us the diffraction angles
- Sort spots into pairs, with **2θ**, **η**, and **ω**, and the scattering vector **p** (diffracting plane normal)



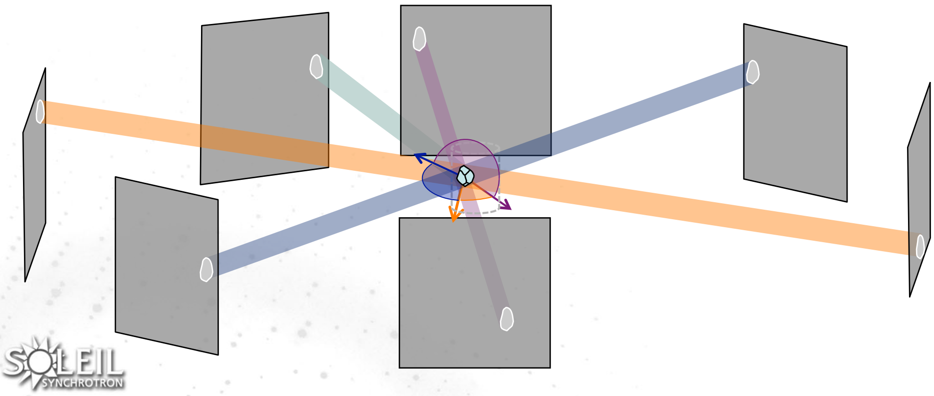


W. Ludwig *et al*, *Rev. Sci. Instrum.* **80**, 033905 (2009), P. Reischig *et al*, *J. Appl. Cryst.* **46**, 297-311 (2013), …

DCT: Data processing 3

(4) Indexing

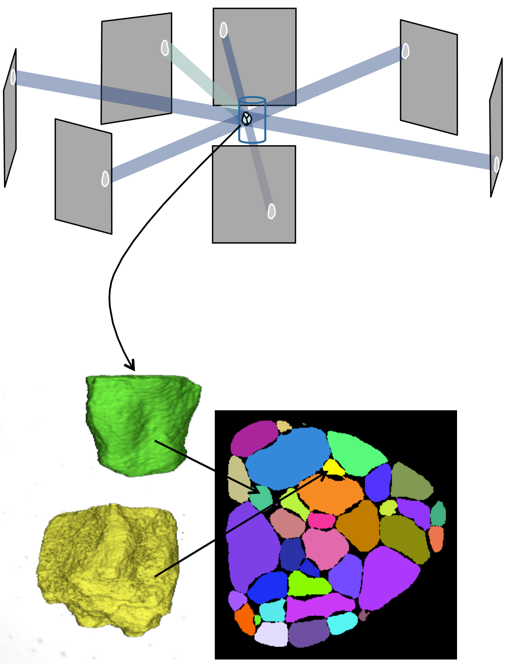
- Assign spots (pairs or individuals) to grains
- INDEXTER algorithm
- Iterative process
- Crystallographic, spatial and shape criteria



DCT: Data processing 4

(5) Reconstruction

- Reconstruct grains individually
- ART/SIRT reconstruction from the diffraction spots
- Oblique, parallel beam geometry
- Few 1000 individual grains
- Assemble the full 3D grain map
- Plus absorption reconstruction from direct beam images
    - Mask for grain map



Resulting data (DCT)

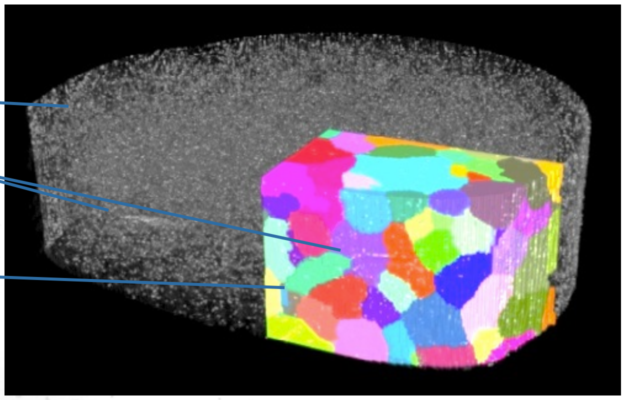
- DCT grain map (few 1000 grains), combined with tomographic reconstruction of sample acquired simultaneously.
- Rendering to show all the information: Mg fatigue test specimen.

Absorption contrast tomography data:

High Z precipitates

2 x FIB notches (implanted Ga)

Part of the grain map from DCT - shapes; orientations.

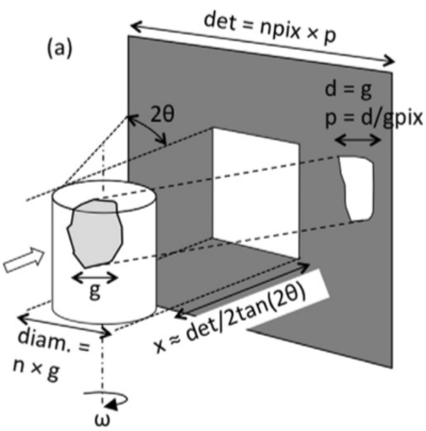


- All acquired non-destructively, so now can perform an in-situ experiment

Limitations

Synchrotron DCT case

- Access to synchrotron
- Sample size
  - Sample size, grain size, and pixel size all scale together
- Number of grains
  - ~5000 grains illuminated at once
- Orientation spread
  - low mosiacity (<0.1°-1° intragranular orientation spread)
  - Main limitation in real materials ➔ Developments in progress



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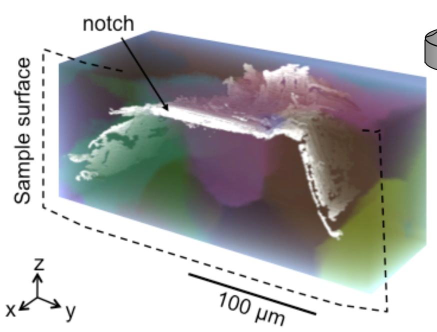
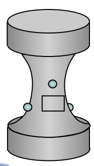
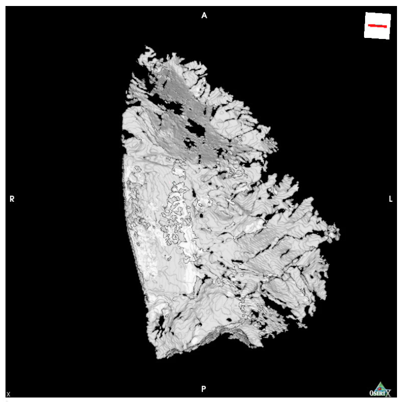
Short fatigue cracks

- Microtomography to observe short fatigue crack growth in-situ in a grain mapped sample.
  - FIB notches placed in specific grains
  - In-situ fatigue using machine from INSA de Lyon
  - Use radiographs to monitor crack
  - Use tomograms to record crack evolution in 3D



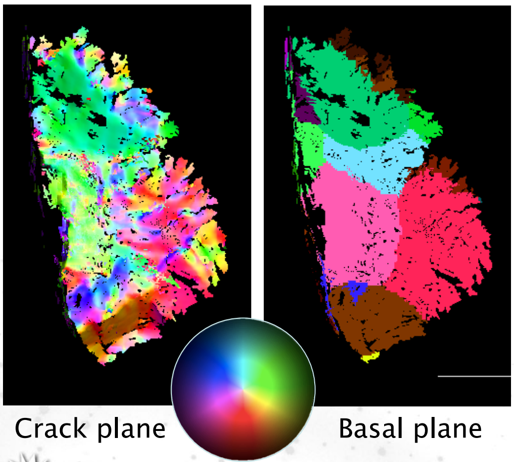




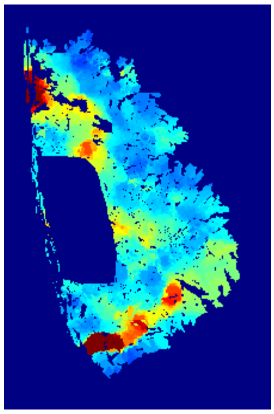
A. King *et al.*, *Acta Mater.* **59** (2011) 6761–6771

Crack and microstructure

- Look at final crack shape compared to microstructure
  - How does this correspond to the crack growth rate?



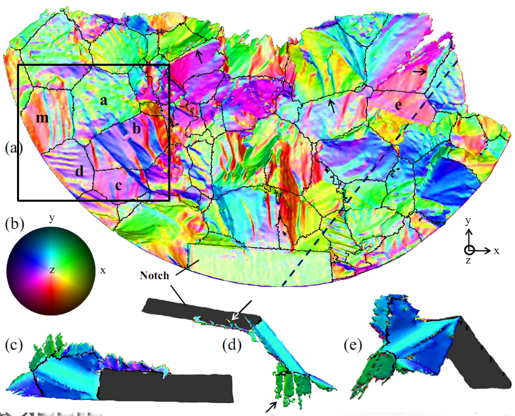
Crack plane Basal plane

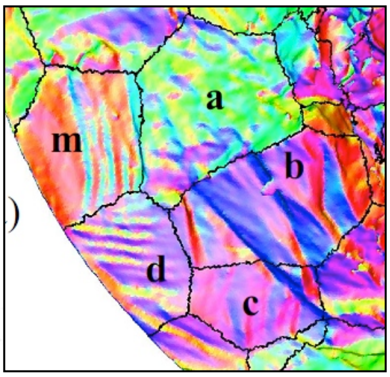


Crack growth rate

Titanium fatigue crack

- Similar concept from PhD thesis of Michael Herbig
  - In Ti-21S, β-Ti alloy (bcc)
  - Explain facetted crack growth in terms of combinations of slip systems
  - Ultimate goal of predicting crack behavior in polycrystals

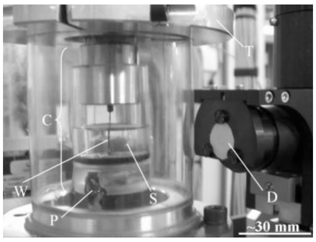


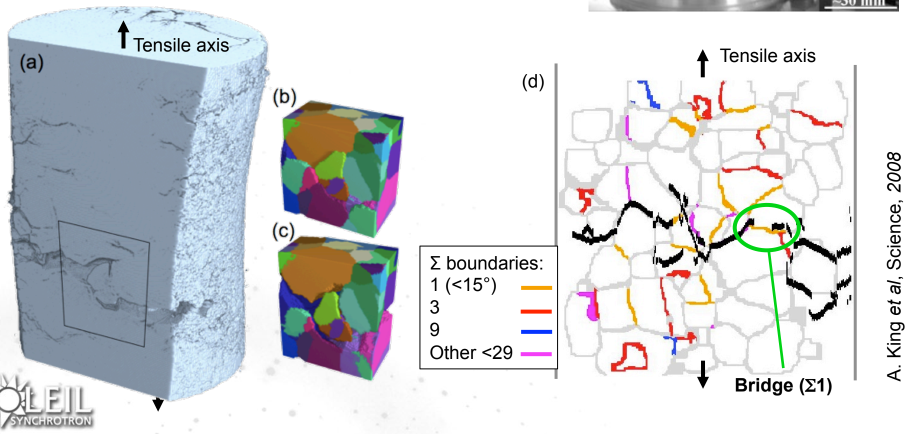


M. Herbig *et al*, *Acta Mater.* 59, 2011, 590-601

Stress corrosion cracking

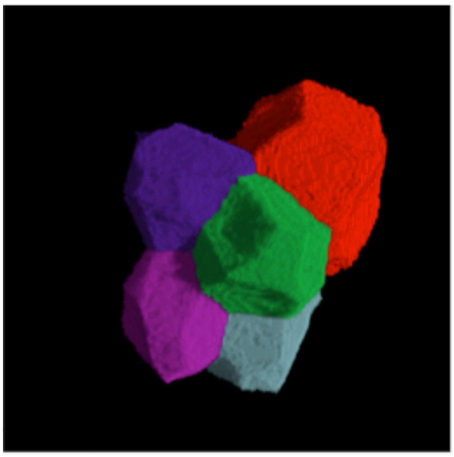
- Interaction of stress, sensitised material, and corrosive environment
  - Identify boundaries which resist cracking
  - Bridging boundaries include low sigma boundaries and low index plane boundaries





Other applications

- Wide range of applications
  - Fatigue
  - IGSCC
  - Deformation
    - Plasticity
    - Twinning
  - Sintering
    - Particle rotations
    - Grain growth
  - Snow dynamics
  - Paleontology/Biology



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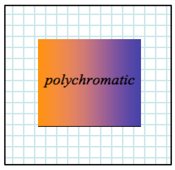
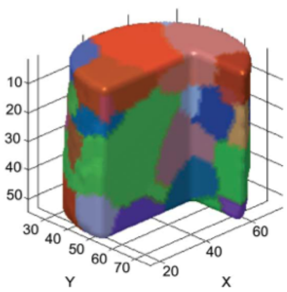
Laboratory DCT

- Synchrotron
  - high flux
  - monochromatic beam
  - parallel beam
- Laboratory X-ray tube
  - Low flux
  - polychromatic beam
  - cone beam
- Commercial development (Xnovo, Zeiss)
- Similar developments using cold neutron source at PSI (Switzerland)

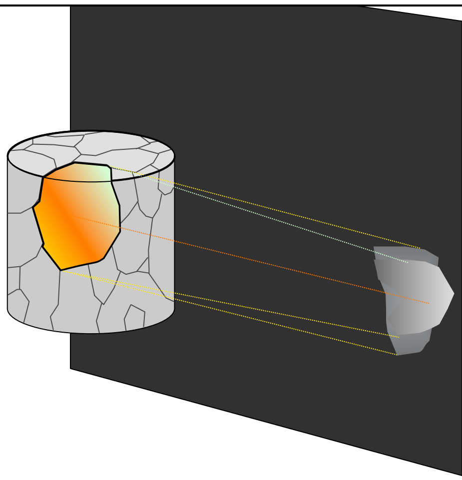


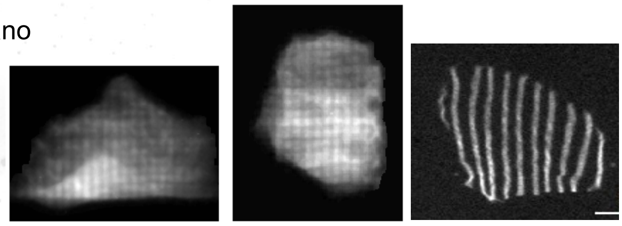


A. King *et al*, *J. Appl. Cryst.* **46**, 2013
S. Peetermans *et al*, *Analyst* **139**, 2014

DCT: 6D reconstruction

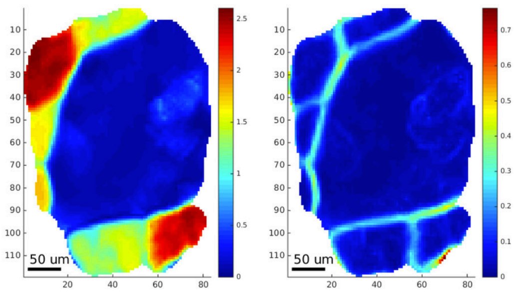
- Main limitation of technique as I have described it is mosaicity
  - Grains are not perfect crystals
  - Intragranular misorientation
  - Assumption that the spots are parallel projections of the grains breaks down
- To deal with this reconstruct both intensity and orientation (rx,ry,rz) as function of position (x,y,z) in each grain
  - Thesis of N. Vigano



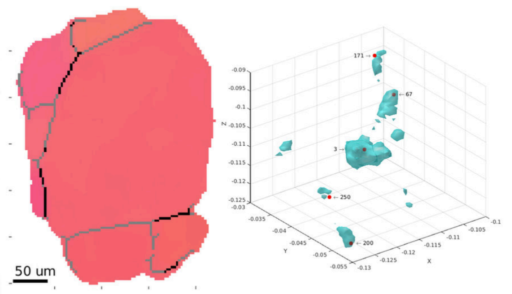


6D reconstruction

- Some 6D reconstruction examples
- Slice through a grain
  - reconstruction of misorientation and subgrains
- Greatly improved grain shape reconstruction
- Good compromise between speed of DCT (reconstruction ~1000 grains), and techniques which solve orientation of every voxel (100 million voxels)



(a) Slice IGM (b) Slice KAM



(c) IPF (d) ODF

N. Viganò, *Scientific Reports* **6**, 20618 (2016)

Outline

- Introduction
- Motivation
- Underlying techniques
- Diffraction contrast tomography
  - Setup, data acquisition and processing
- Application examples
- Developments in progress
- **Related techniques**

A family of techniques

- **Diffraction contrast tomography (DCT)**
- **3D X-ray diffraction microscopy (3DXRD)**
- Original grain mapping technique, from which DCT was derived
- **High energy diffraction microscopy (HEDM)**
  - At APS – recently very successful with deformed materials
- **Diffraction tomography**
  - Point scanning with a focused beam
- **Differential Aperture X-ray Microscopy (DAXM)**
  - White beam, point scanning, analyser wire for 3D spatial info.
- **Topography**
  - Classical diffraction imaging for defects in single crystals
- **Dark field x-ray microscopy**
  - Recent developments analogous to TEM

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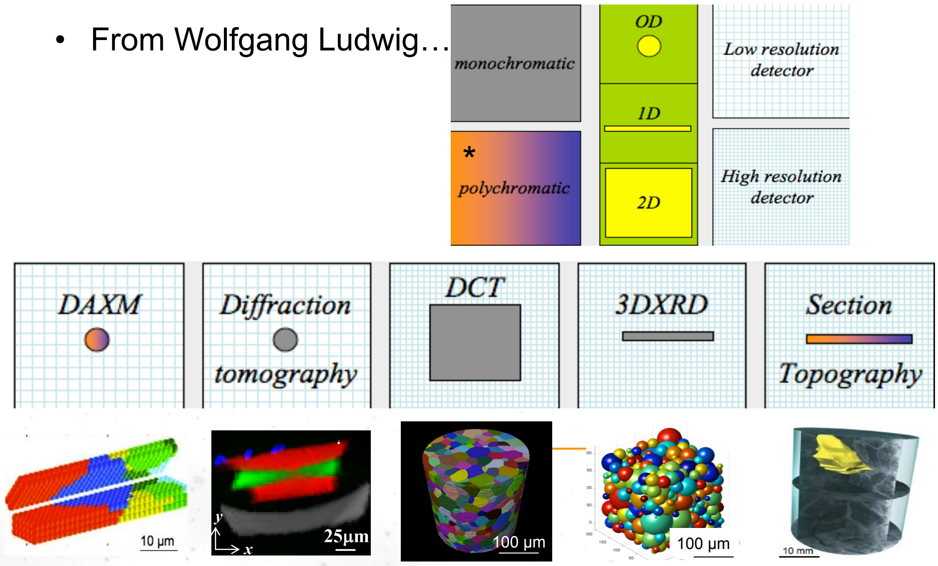
Visual overview

- From Wolfgang Ludwig…



high res.- slow   Cryst. phases   medium res. - fast   single crystals

Related presentations HSC19

- Coherent diffraction imaging talk tomorrow
  - M. Guizar-Sicairos
- Scanning microscopies talk tomorrow
  - M. Cotte
- Wolfgang Ludwig tutorial

SOLEIL SYNCHROTRON