3D grain structures from X-ray diffraction contrast tomography

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Metal Structures in 4D

Improve our understanding of how structural materials react to external stimuli

- deformation
- annealing
- chemical environment

Focus on metallic alloys (polycrystalline)

Spanos, Rowenhorst, Lewis, Geltmacher
Combining serial sectioning, EBSD and Finite Element Modeling, MRS Bulletin

Topotomography of Al grain during recrystallization

Outline:

Non destructive mapping of polycrystals in 3D

Diffraction contrast tomography:
- principle
- analysis strategy
- current possibilities
- limitations

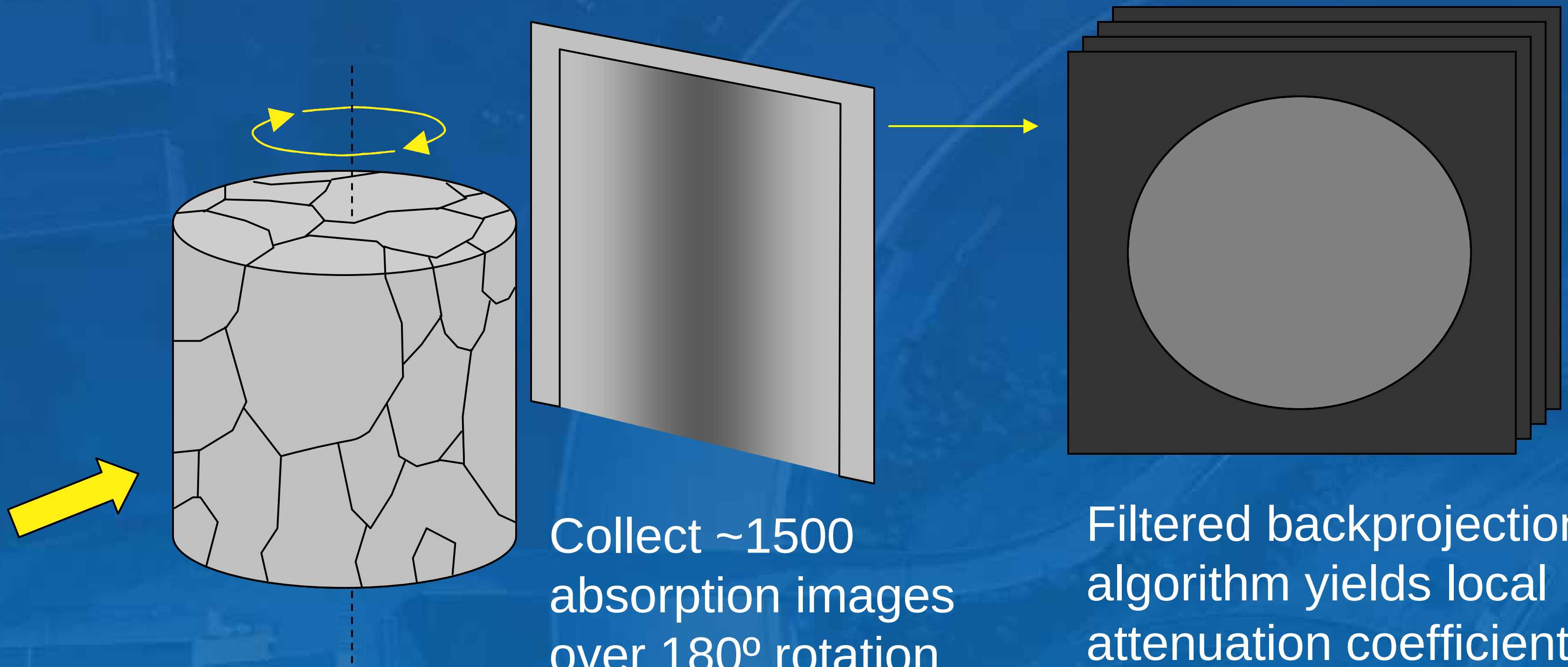
Applications

Conclusions

Conventional absorption tomography

Synchrotron or lab X-rays, neutrons, etc

- No contrast between grains of the same phase
- No crystallographic orientation or strain information



Collect ~1500 absorption images over 180º rotation

Filtered backprojection algorithm yields local attenuation coefficient

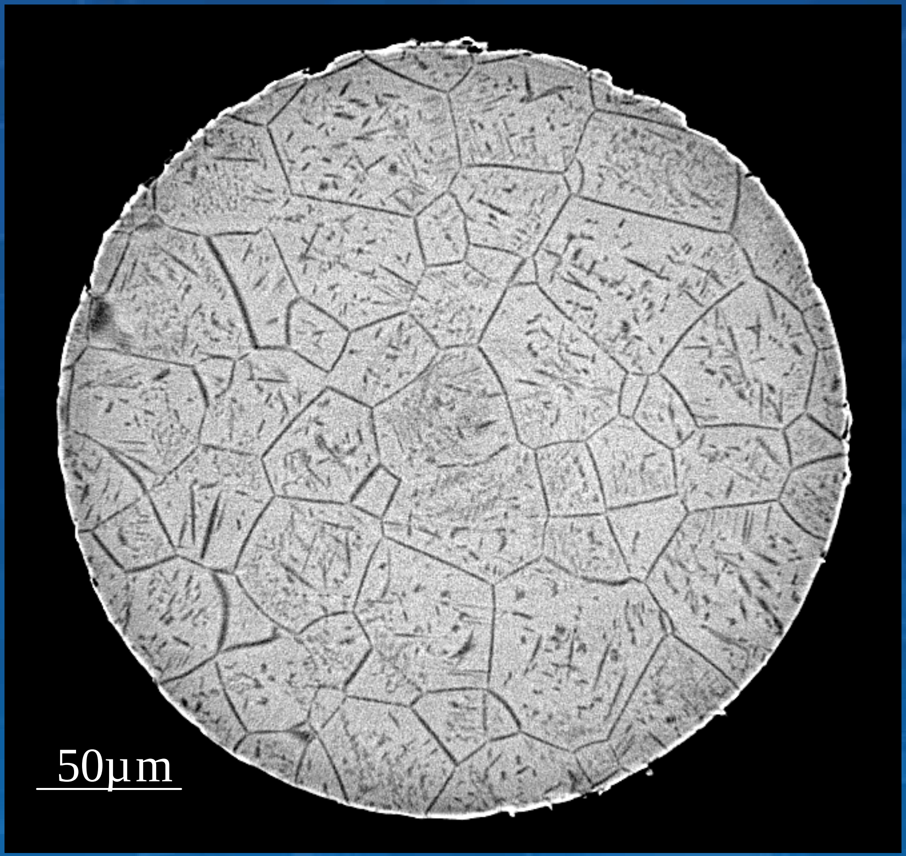
…some exceptions

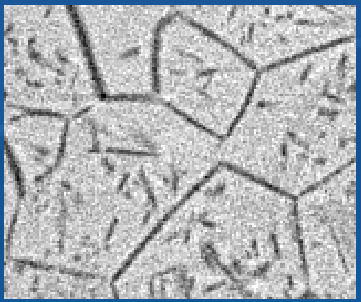
- Segregation and/or precipitation at grain boundaries
- Duplex microstructures
- Liquid metal penetration (Al - Ga)

X-ray absorption or phase contrast imaging may reveal 3D grain structure in these cases

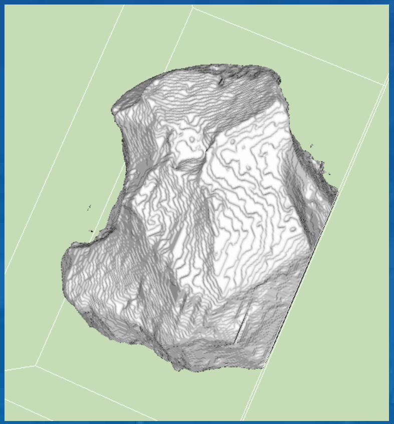
However: crystallographic orientation unknown

3D grain microstructure from X-ray phase contrast tomography





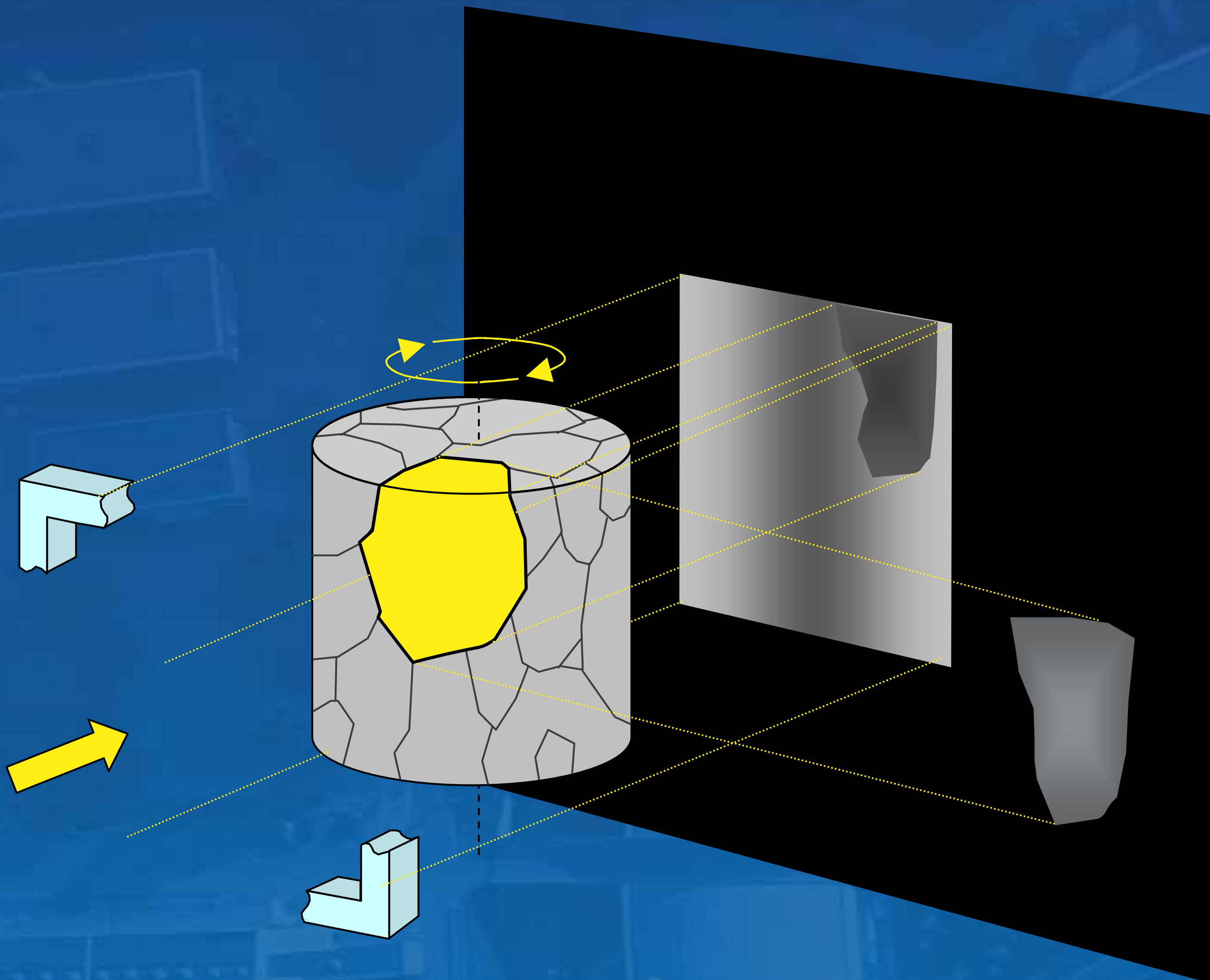
30µm



Ti β alloy (21s) with α phase precipitates

E.M. Lauridsen, R. Fonda, W. Ludwig et al.

Diffraction Contrast Tomography: combined case

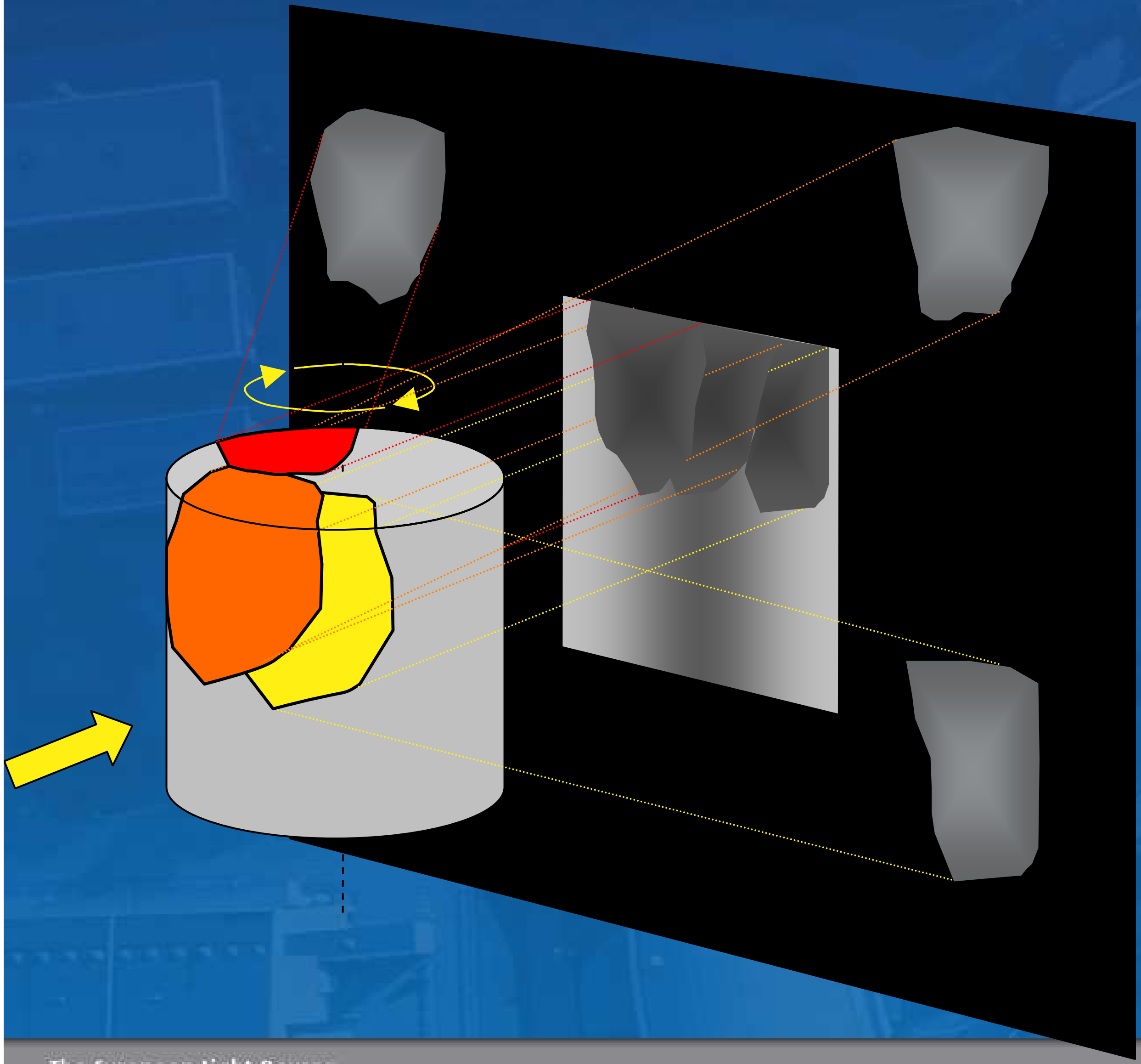


– Conventional tomo-graphy setup
alignments
– Large detector with high dynamic range
– Use slits to confine the beam to sample
– monochomatic beam ($\Delta\lambda/\lambda \sim 10^{-4}$)
– Continuous

– During rotation, grains pass through Diffracting
grains with small spreads in orientation:
“extinction” spot visible in direct beam
– both spots can be approximated as grain projections

G. Johnson, A. King, M. Hoennicke, T. Marrow, W. Ludwig, J. Appl. Cryst. 2008

Diffraction contrast tomography: combined acquisition



During sample rotation should see each grain ~ 20 - 100 times.

Some may be lost

- Overlaps
- Off the detector
- Low contrast

Enough to reveal the 3D grain shape through ART reconstruction

Determination of diffraction vectors

Segment and record diffraction spots in database (~20,000 – 100,000)
Need to determine diffraction angles (θ,η,ω)

Find Friedel pairs of diffraction spots
- (hkl), (–h–k–l) separated by 180° ω

Visualise 180º sample rotation as reversed beam direction, and detector position.

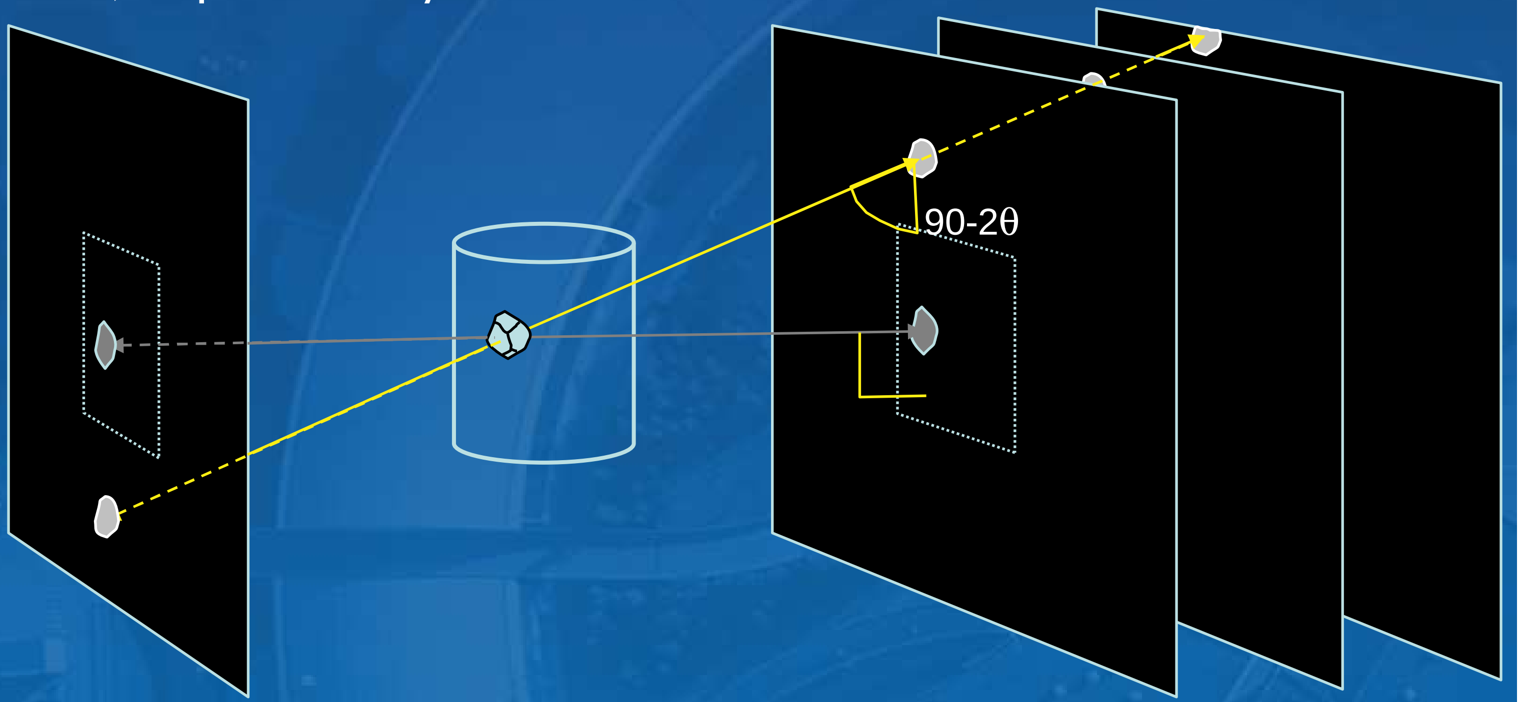
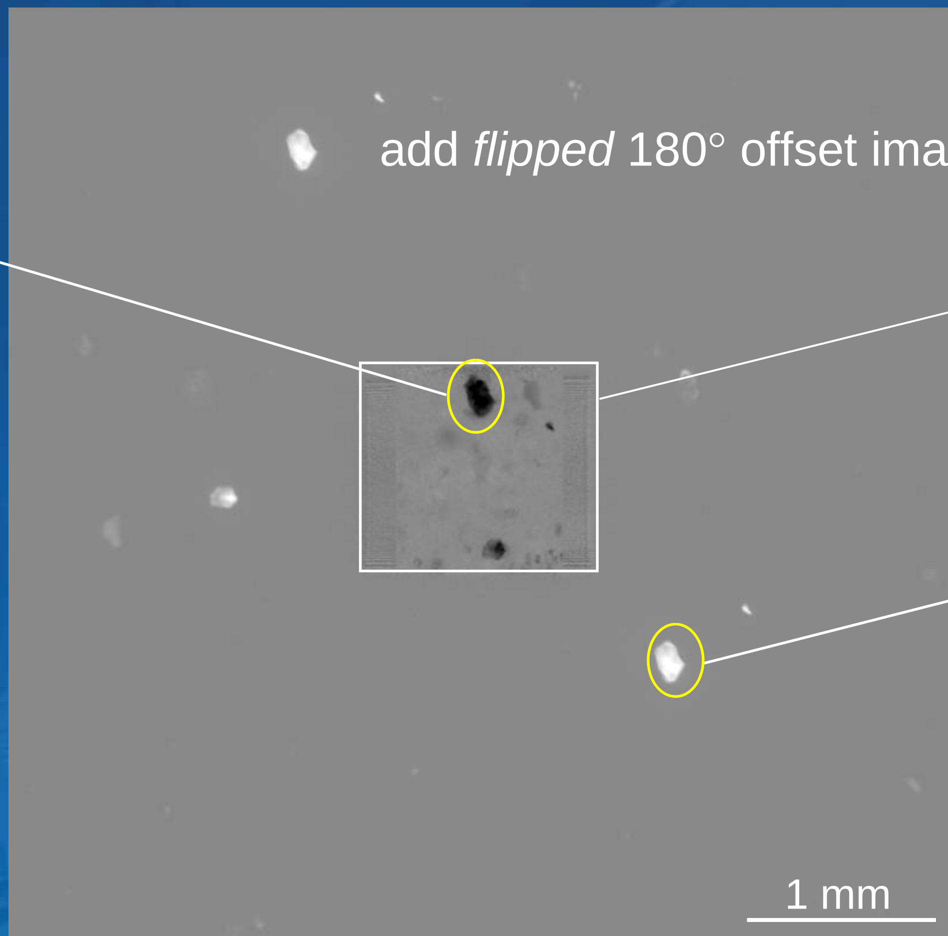


Image processing

Separate diffraction contrasts from images (background removal, filtering)

add *flipped* 180° offset image

Extinction spot



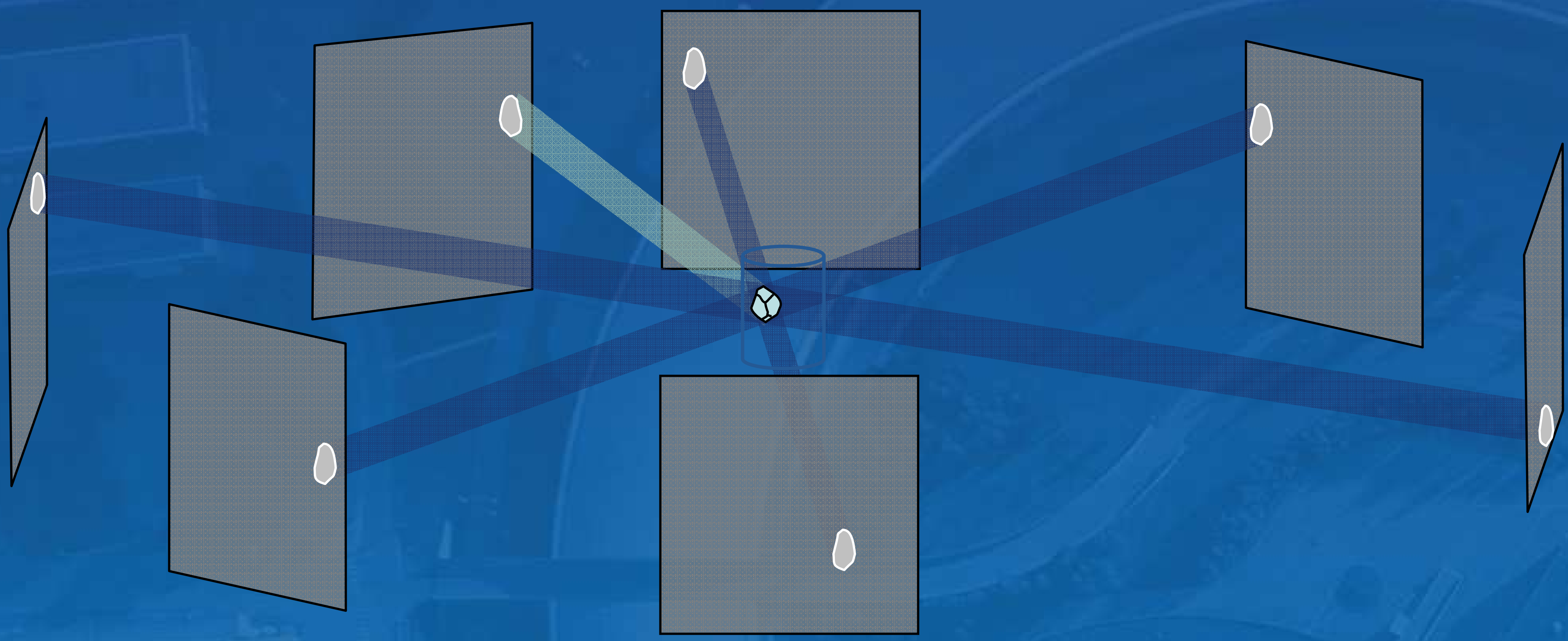
Direct beam area (frequent overlaps)

Diffraction spot
Well spaced out
no overlapping spots

Data processing - grain reconstructions

3D backprojection geometry (as opposed to pseudo 2D) for the ART reconstruction has been implemented

- Gives more accurate grain reconstructions



Analysis route

1. Background removal, integration and segmentation of diffraction spots
2. Find Friedel pairs of diffraction spots (pair matching)
3. Find consistent groups of reflections (indexing)

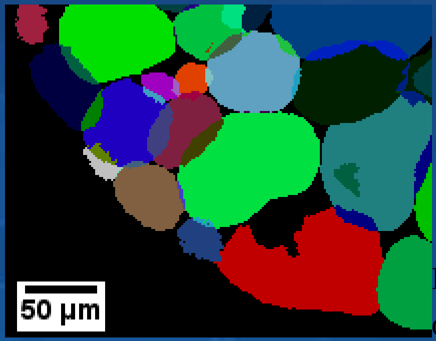
Results:
- Sets of projections belonging to individual grains
- Grain orientation
- Grain position
- elastic strain tensors (optional)

5. 3D grain shape from ART (grain by grain)
6. Assemble sample volume
7. Post-process grain map (remove overlaps, fill gaps)

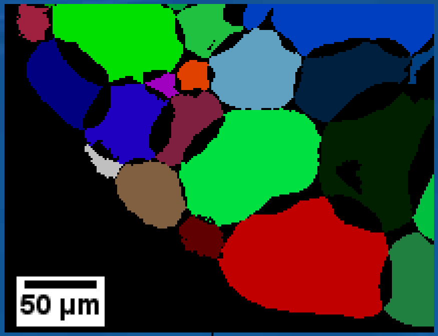
QuickTime™ and a decompressor are needed to see this picture.

200 µm

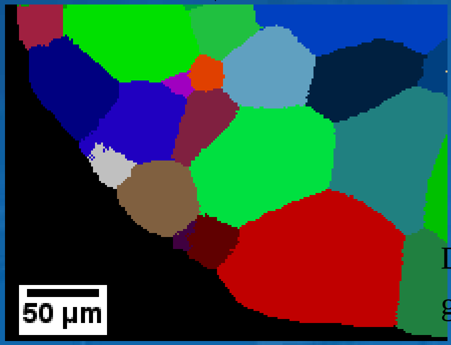
Experimental – Diffraction Contrast Tomography



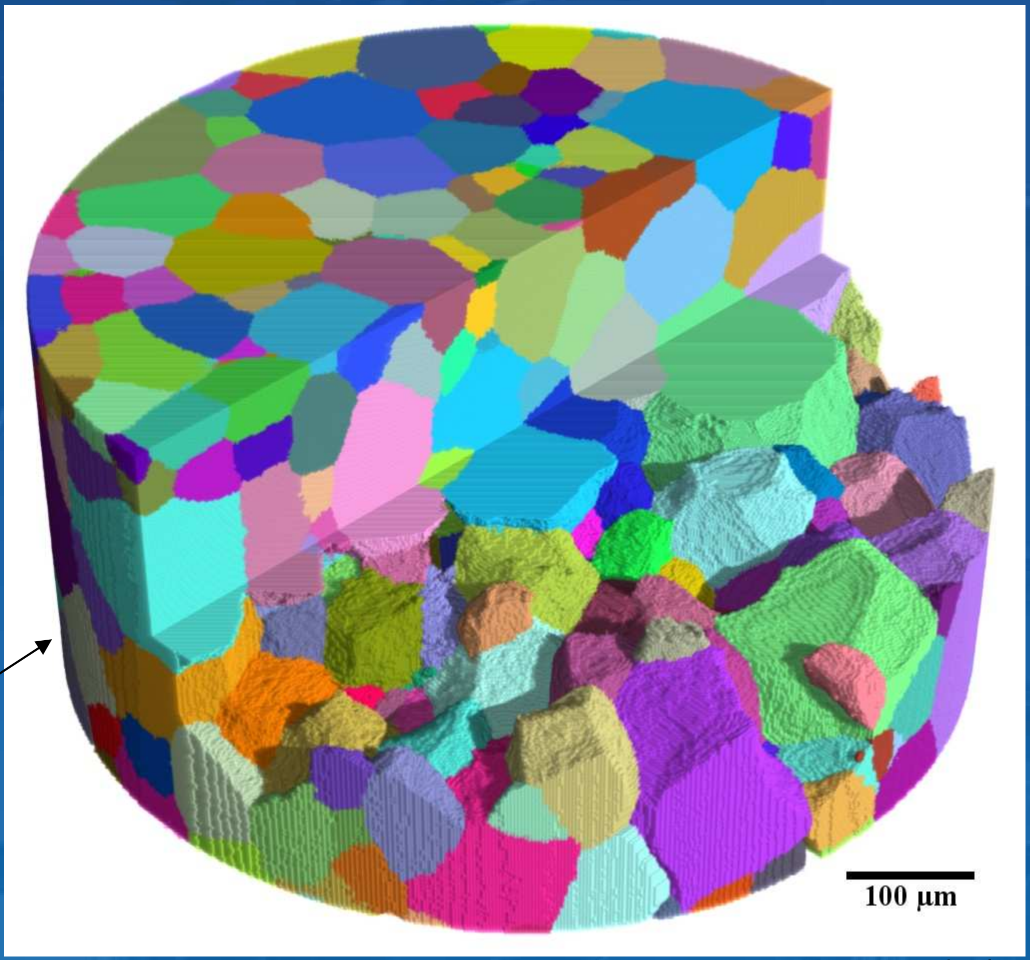
Reconstruction output



Removed overlaps

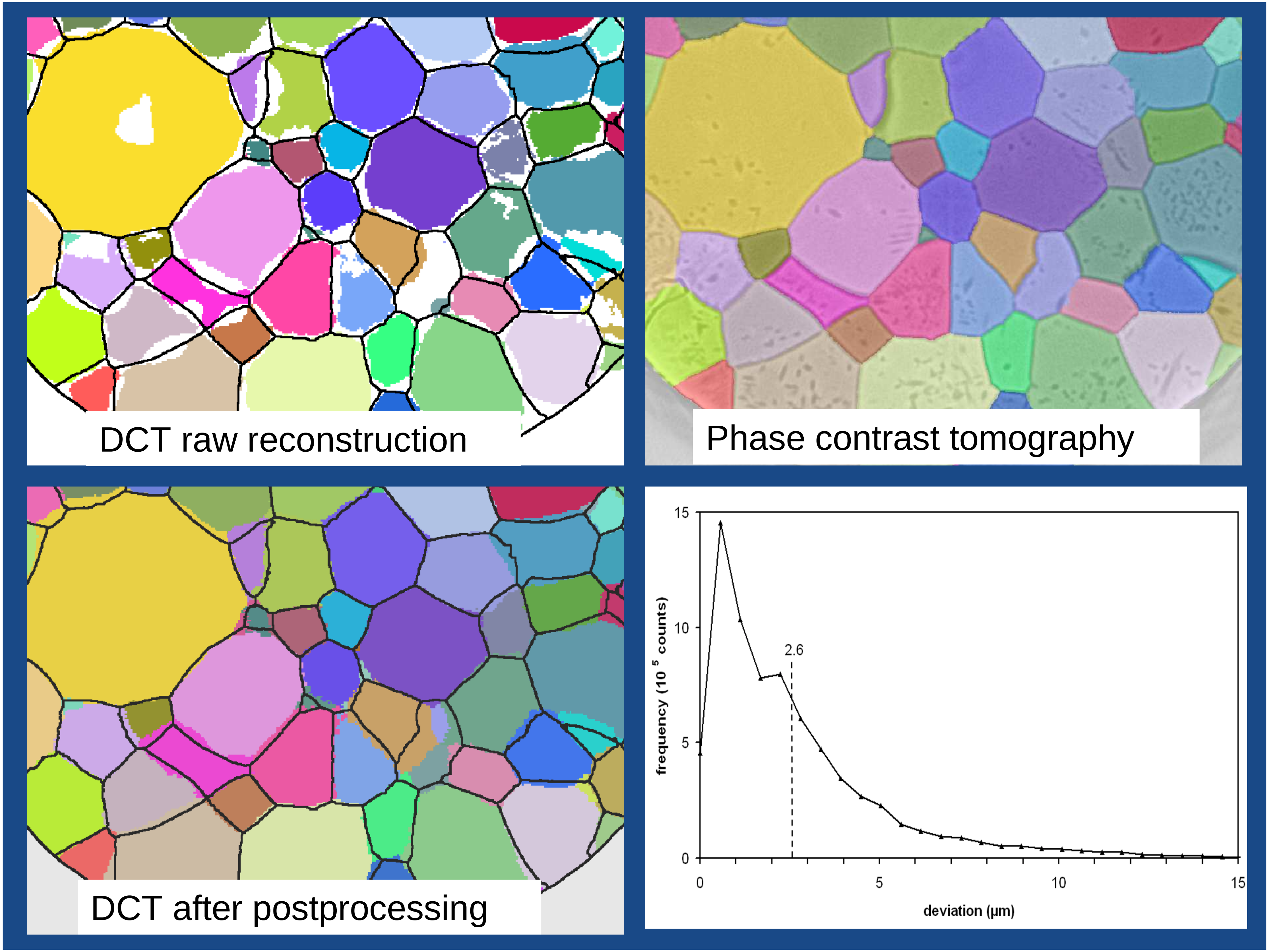


Dilated until grains touch



Reconstructed volume

Question: are grain boundaries correct?


DCT raw reconstruction
Phase contrast tomography
DCT after postprocessing
frequency (10^5 counts)
deviation (µm)
2.6
15
10
5
0
0
5
10
15

3D grain reconstruction from DCT

QuickTime™ and a decompressor are needed to see this picture.

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100 µm

- Acquisition time @ ID11: 2 h
- 1008 reconstructed grains
- 2 days of processing (30 nodes)
- ~ 3 µm accuracy

W. Ludwig et al., Rev. Sci. Instrum. **80** 033905 (2009)

Known limitations…

1. Sample requirements
   - low mosaicity (< 1 degree: recrystallisation or solidification)
   - grain size bigger than about 20 times pixel size
   - sample diameter less than 20 times grain size
   - strong texture and high mosaicity reduce these numbers

2. Significant orientation & strain distributions inside grains violate assumption of parallel projections

3. Assumption of kinematical scattering may be violated:
   Absorption, multiple scattering & dynamical diffraction effects
   alter intensity distributions

Application examples

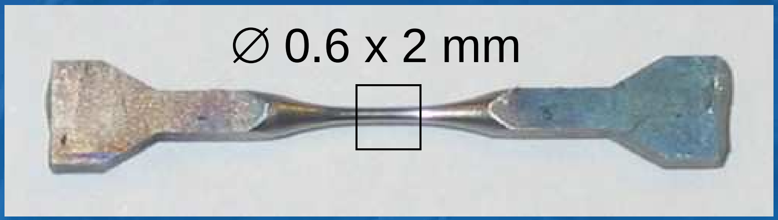
Determination of elastic strain tensors during a tensile load test
(master thesis P. Reischig, TU Delft 2008)

Analysis of short fatigue crack propagation in Ti alloy

(PhD thesis project of M. Herbig)

Measurement device

- 360 degree visibility
- 500N load cell
- mechanical loading (fine thread)

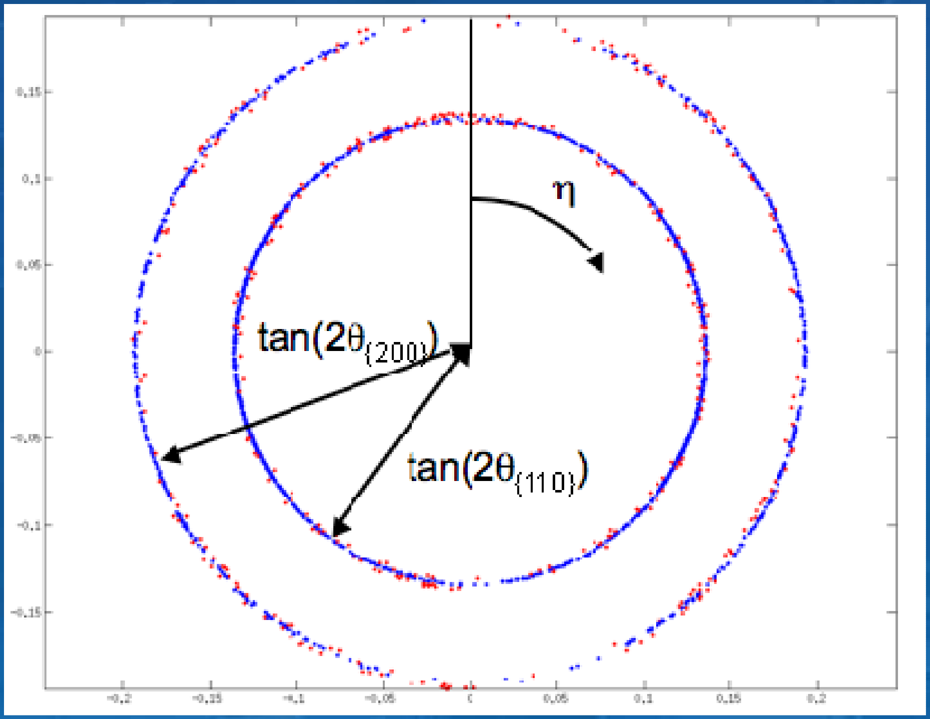


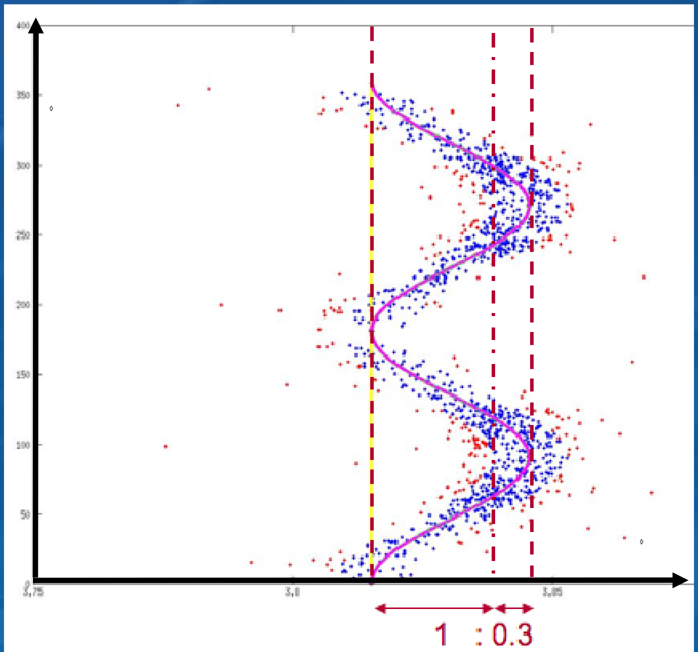
25 mm

Material: Ti β alloy 21S (bcc)
electrical discharge machining

“Artificial” powder diffraction pattern

extracted diffraction angles from the Friedel pairs
no dependence on grain position





clear angular shift
due to applied strain

Fit of strain tensors using linear elasticity model

Combines:

change in interplanar angles
change in interplanar distance

$$\begin{bmatrix} \Delta(n_i \cdot n_j) \\ \epsilon_{n_i}^{rev} \end{bmatrix} = \begin{bmatrix} M_{(n_i,n_j)} \\ L_{(n_i,n_i)} \end{bmatrix} \cdot \langle \epsilon^{rev} \rangle$$

Input

Measured variables: **n** (plane normals), Bragg angles
Reference lattice parameter

Theoretical interplanar angles in unstrained state (bcc)

P. Reischig, master thesis

Average elastic strain tensors

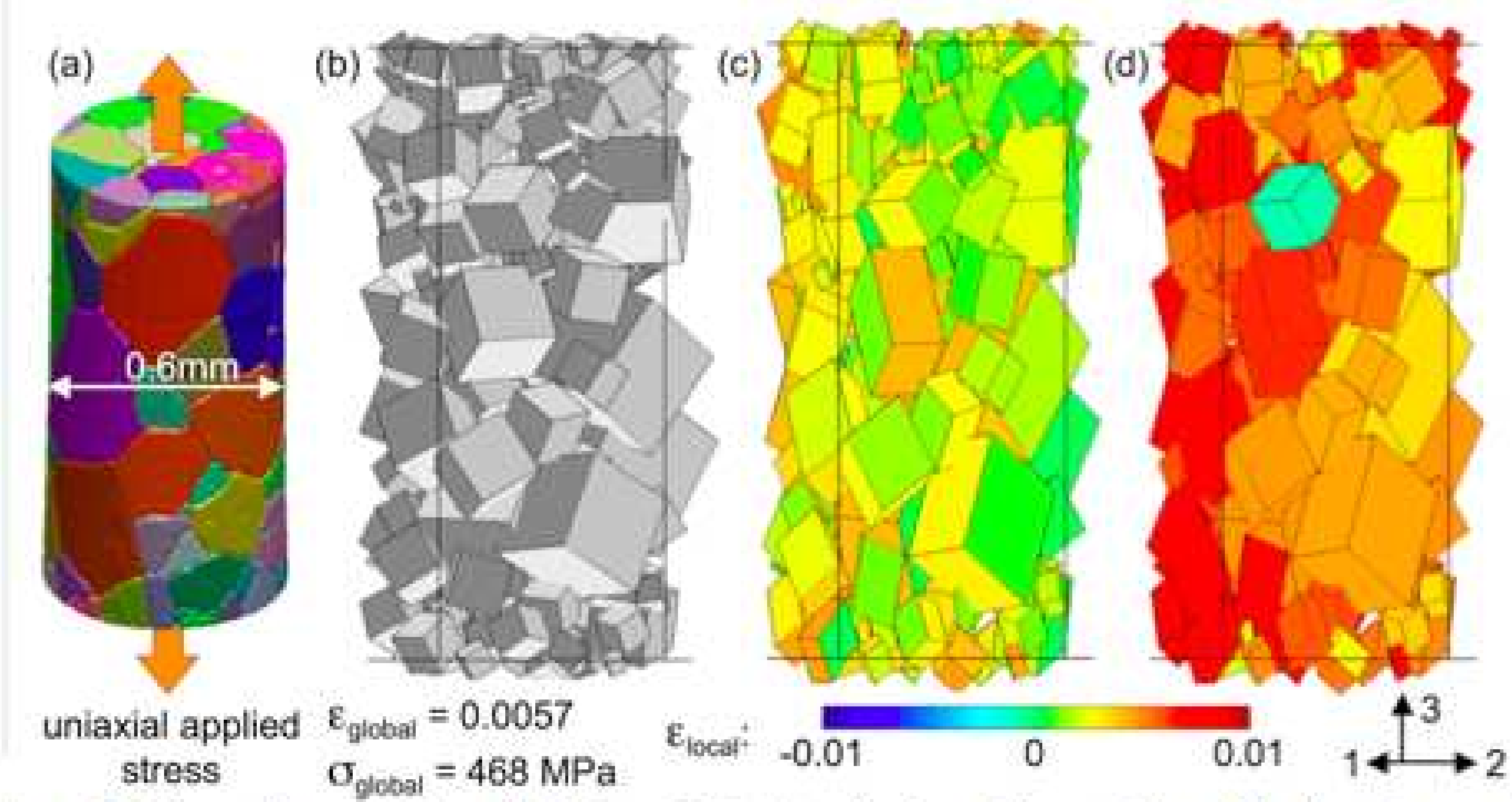


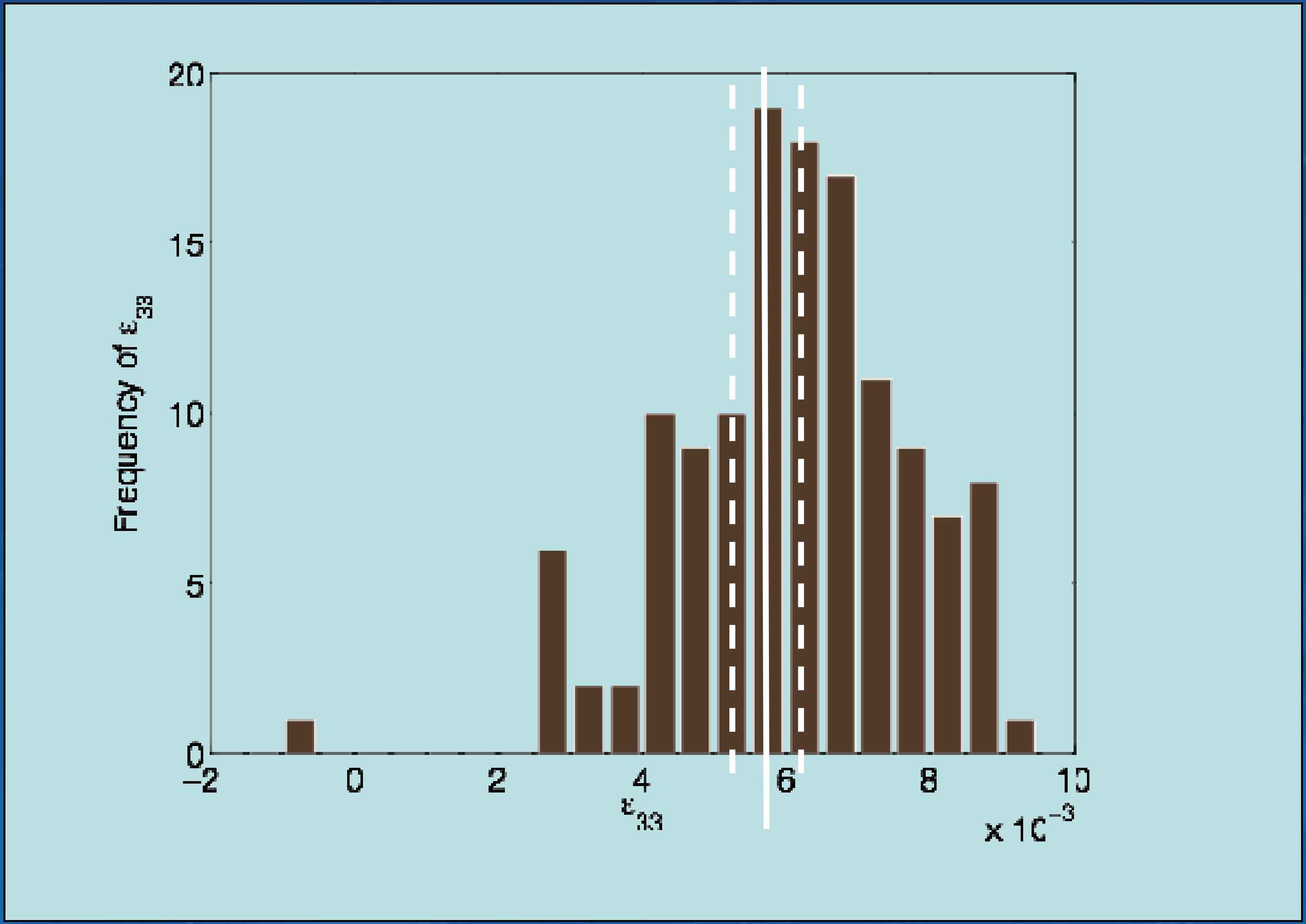
Figure B. Information currently available from DCT: (a) grain shapes (b) crystallographic orientations, and strain tensors, shown (c) resolved onto {002} lattice planes and (d) strain parallel to tensile axis.

Measured strain tensor components

Elongation along the load axis (ε_{33})

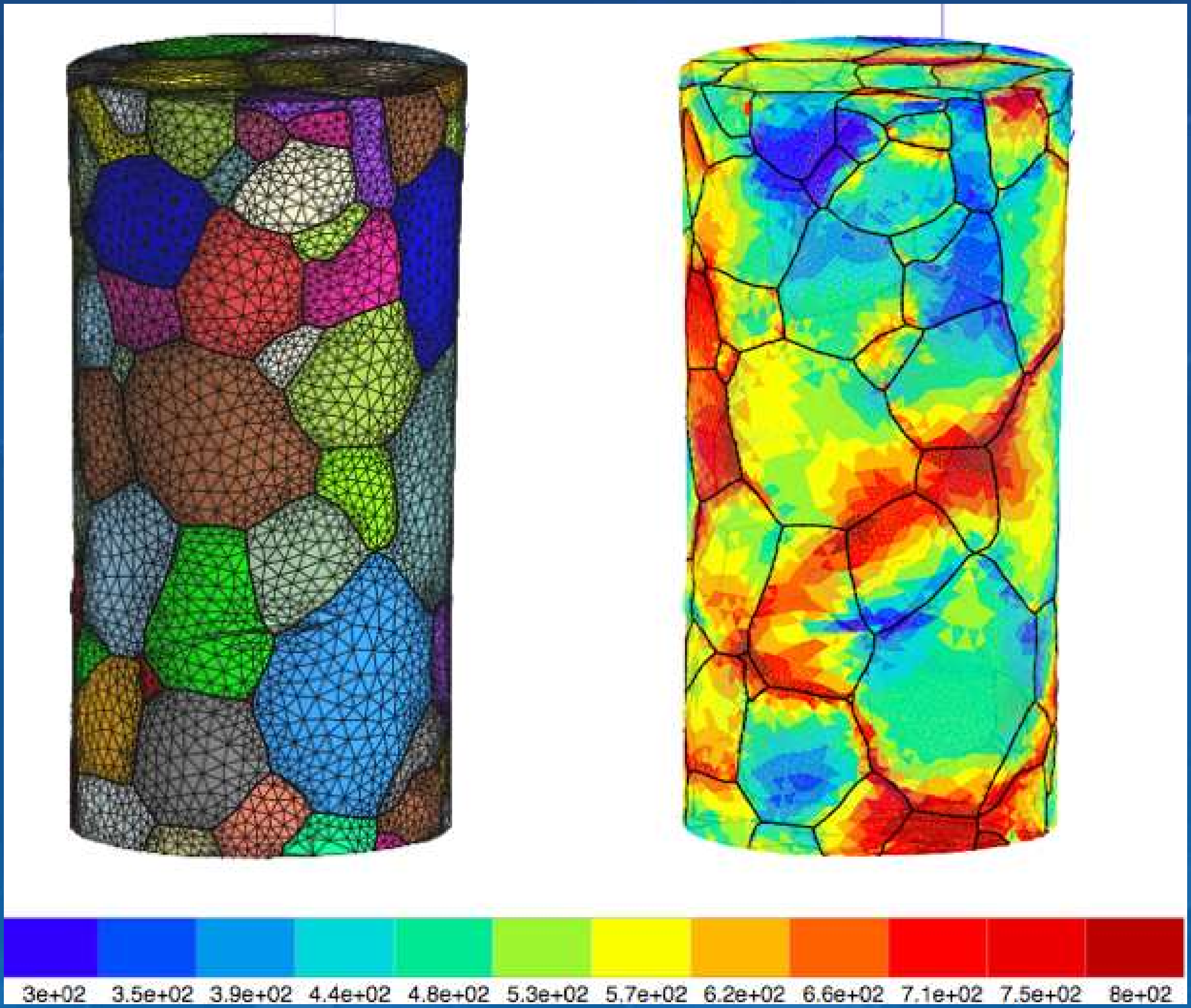
= 467.9 Mpa

= 0.00572



… analysis in progress

CFEM simulation of elastic deformation



Strong variation
of local stress
& strain distribution

Comparison of grain
average values:
…in progress

Need to develop
local characterization
techniques
(e.g. combine DCT &
scanning µ-diffraction)

Collaboration with H. Proudhon, S. Forest, ENSMP

Characterization of short fatigue crack propagation

"Short fatigue crack problem"

- strong variability
- no reliable prediction / simulation
- need for more fundamental work
- experimental challenge

QuickTime™ and a
decompressor
are needed to see this picture.

SEM: Fatigue fracture surface

Experiment: - DCT of FIB notched sample (ID11)
- in-situ crack propagation (25 time steps, ID19)

Heat treatment (grain boundary decoration)

- holotomography (ID19)

In situ crack propagation at ID19

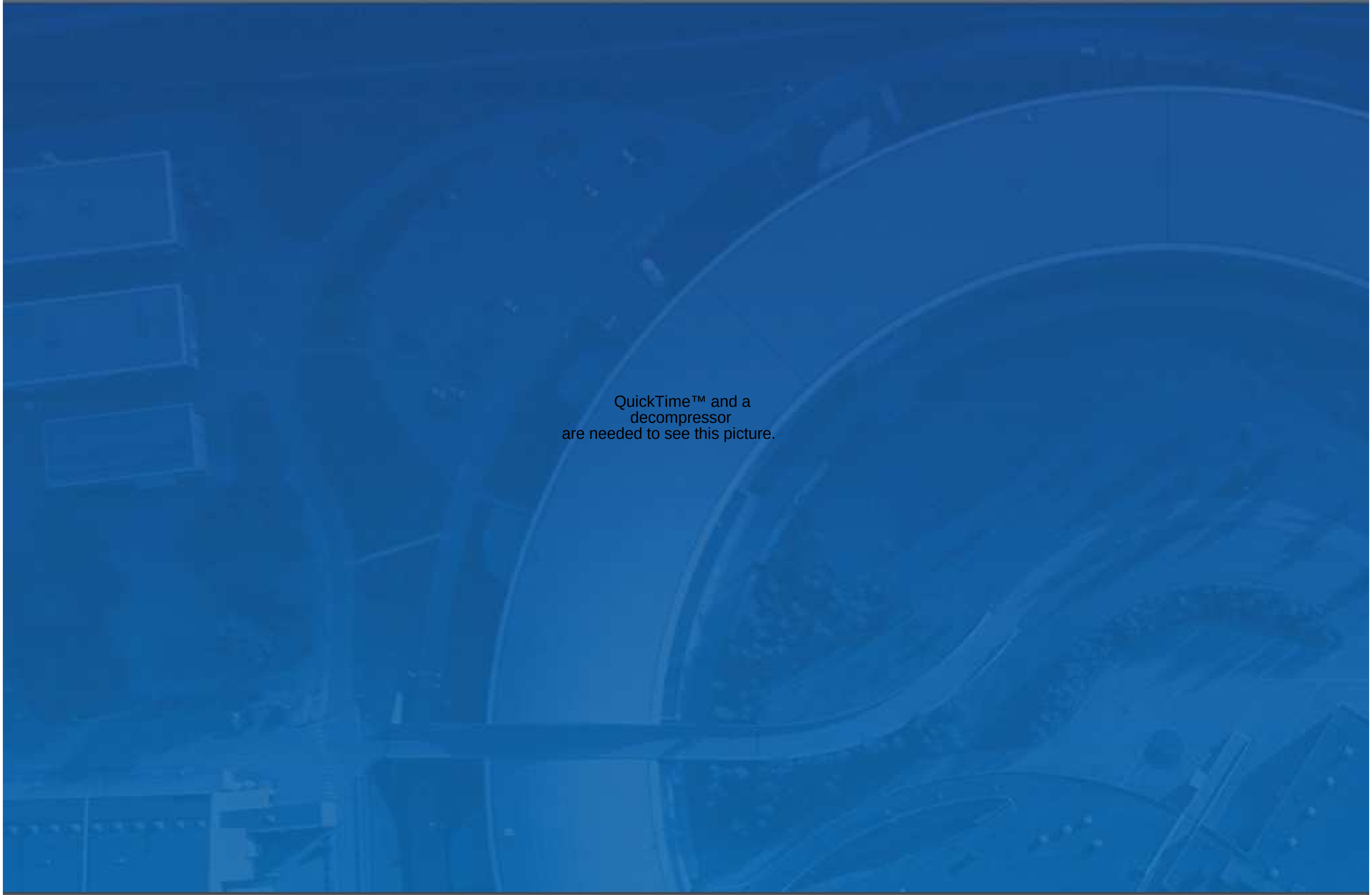
QuickTime™ and a
decompressor
are needed to see this picture.

Crack initiation from FIB notch

25 time steps

0.7 µm voxels

3D rendition of short fatigue crack in Ti 21S alloy sample


QuickTime™ and a
decompressor
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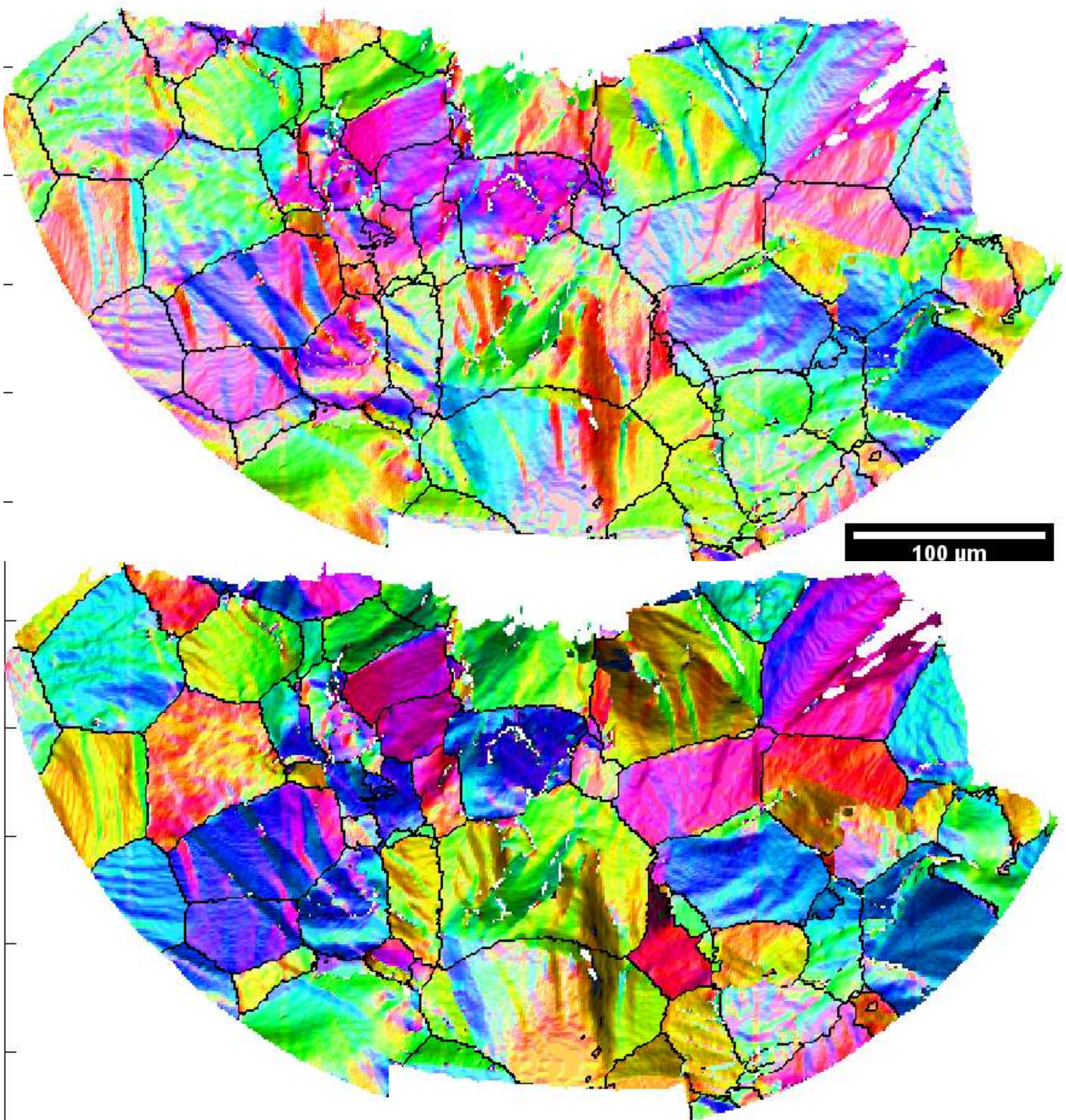
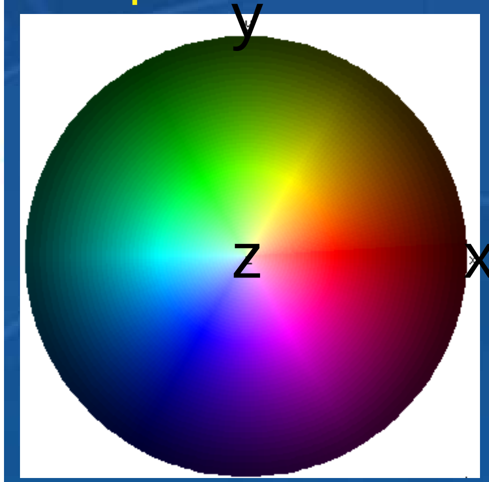
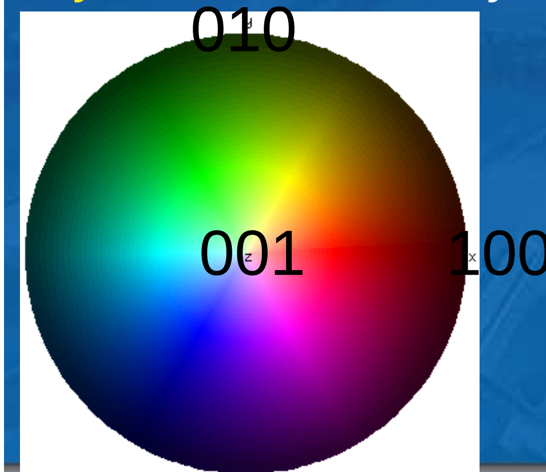


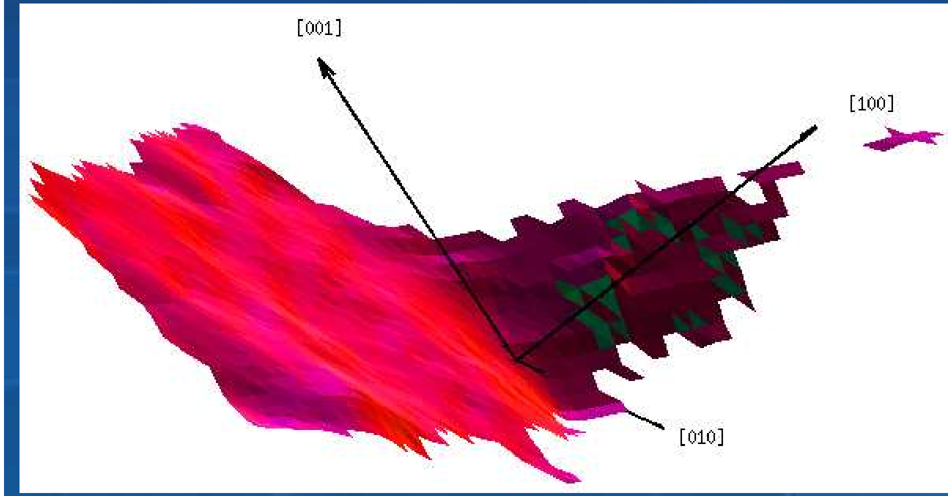
Orientation of crack in sample coordinate system



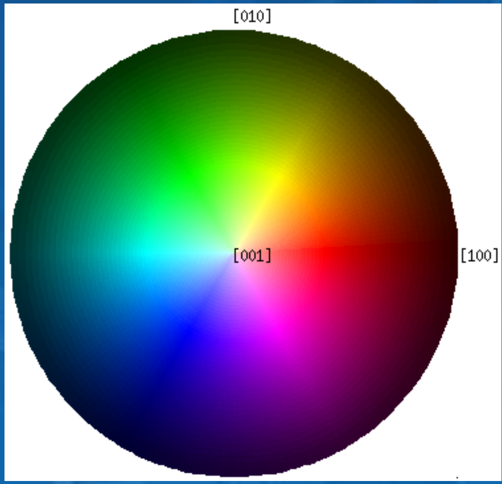
Orientation of crack in crystal coordinate system

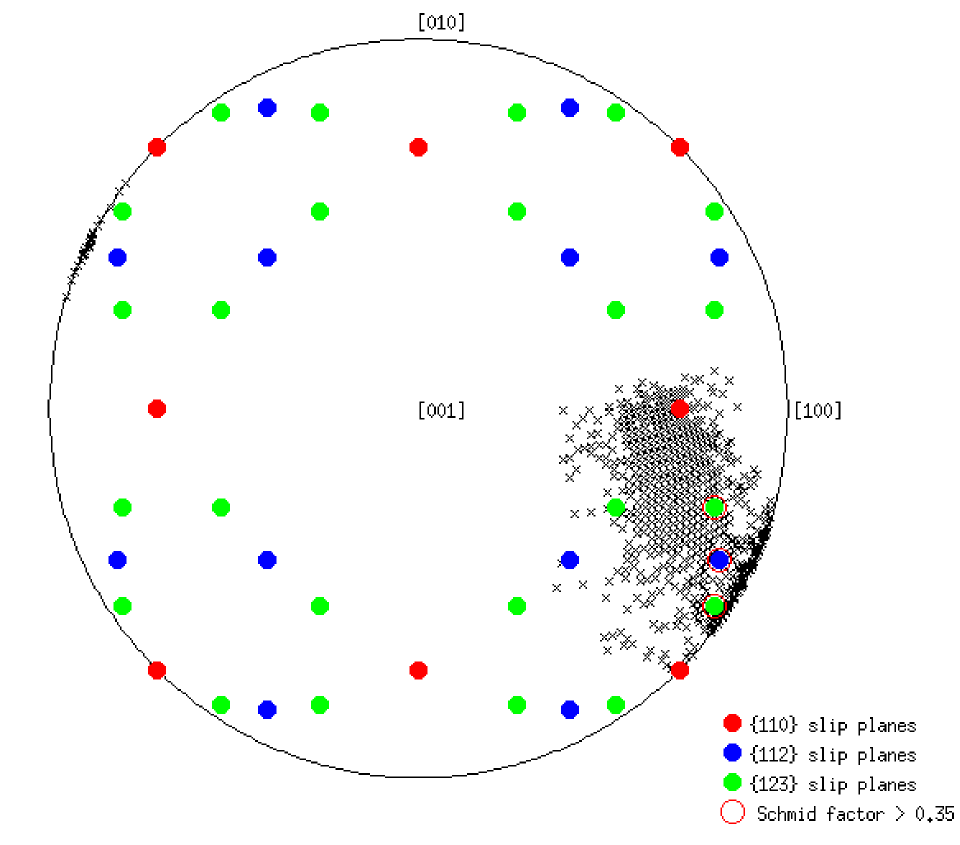


Data Analysis – Orientation of crack in single grain



crack in single grain color coded with respect to crystal coordinate system





Conclusions

X-ray Diffraction Contrast Tomography can provide

- 3D grain shapes	~ 3 µm accuracy
- orientations	~ 0.1 degree
- elastic strain tensors	4.10^{-4} (?)
- attenuation coefficient	

in plastically undeformed polycrystals (single phase) fulfilling some requirements on grain vs. sample size, mosaicity and texture.

The technique can be readily combined with in-situ X-ray tomography observations and provides direct input for 3D Finite Element simulations

… exciting times for 3D Materials Science

Future directions / goals

- Further improve spatial resolution
  - detector point spread function
  - improved reconstruction algorithms :
    - fill gaps based on forward simulation
    - advanced algebraic reconstruction algorithms
- Characterization of local orientation and strain state in *deformed* materials
  - combine DCT & scanning microdiffraction approach
  - use of Monte Carlo simulation techniques

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