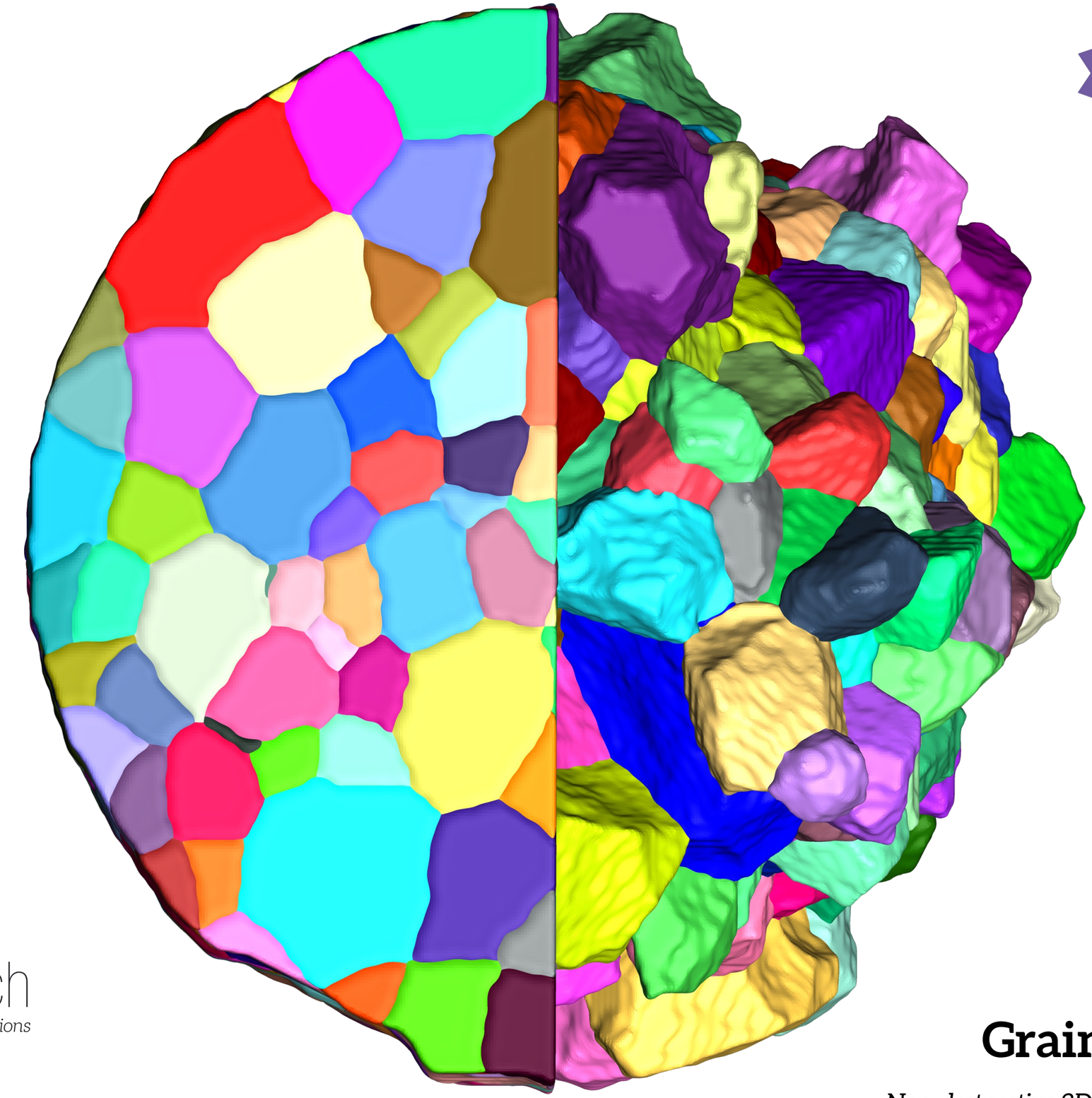
xnovotech



xnovotech

Innovative 3D X-ray Imaging Solutions

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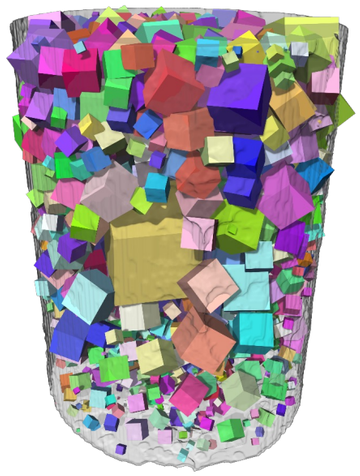
GrainMapper3D™

Non-destructive 3D Grain Mapping Solution for Lab-based Diffraction Contrast Tomography

GrainMapper3D™ is the engine powering Lab-based Diffraction Contrast Tomography solutions offered on ZEISS Xradia Versa **LabDCT Pro™** and **CrystalCT™**, enabling non-destructive 3D crystallographic imaging of polycrystalline samples in your home laboratory. **GrainMapper3D** offers the users seamless diffraction data collection over representative sample region of interest through **DCT Acquisition Wizard**, and guides the users to the final 3D grain map of the sample through an intuitive workflow. The 3D grain map provides comprehensive information of the polycrystalline sample structure, including grain centroid position, grain orientation, grain size, grain morphology as well as grain boundary characters.

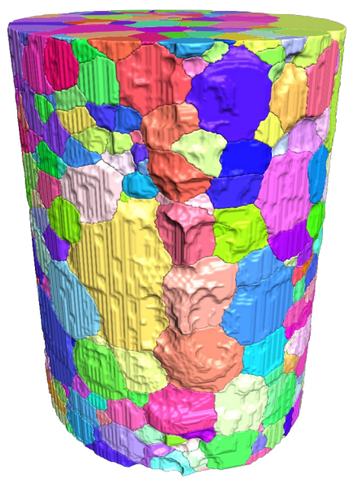
Available information from 3D grain map represented for an individual grain: (left to right) grain centroid position, grain orientation, grain boundary misorientation angle, grain boundary plane inclination and grain boundary curvature.

Take a look inside...



Jul. 2015

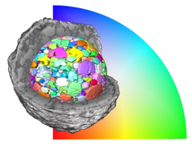
LabDCT Launch
GrainMapper3D v1
Enabling grain mapping in the lab



Jun. 2018

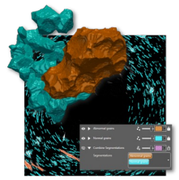
GrainMapper3D v2.0
Grain shape reconstruction

Jul. 2020



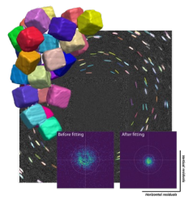
GrainMapper3D v2.3
All crystal symmetry

Dec. 2019

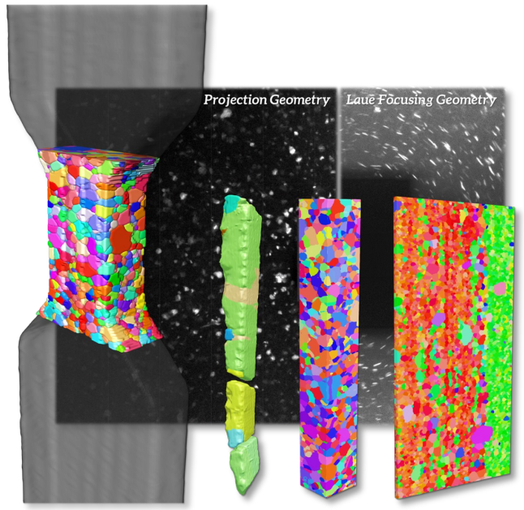


GrainMapper3D v2.2
Advanced segmentation

Jun. 2019



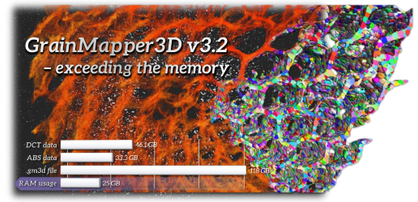
GrainMapper3D v2.1
Forward simulation



Mar. 2021

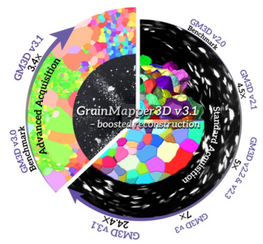
GrainMapper3D v3.0
Projection geometry & advanced acquisition

Mar. 2023



GrainMapper3D v3.2
Exceeding the memory

Mar. 2022



GrainMapper3D v3.1
Boosted reconstruction

Lab-based DCT: A brief history

Bringing the sophisticated diffraction contrast tomography crystallographic imaging technique from the large-scale synchrotron facilities to the laboratory platform has been a challenging and exciting journey.

Since its launch in July 2015 as LabDCT™, lab-based DCT has been the only commercially available non-destructive 3D grain mapping solution for the home laboratory. The reconstructed 3D grain structure was originally reproduced by colored cubes representing the sizes, centroid positions and crystallographic orientations of the grains [1-2].

GrainMapper3D v2.0 released the full 3D grain morphology reconstruction [3], which was a substantial contribution as lab-based DCT now enabled grain boundary characterization capabilities.

The three releases during 2019 and 2020 – GrainMapper3D v2.1, v2.2 and v2.3 – marked solid steps in equipping lab-based DCT to be a powerful tool for materials scientists with more reliability, flexibility and extended applicability [4]. In particular the algorithm speed was optimised, the map fidelity improved and the user friendliness enhanced.

The recent GrainMapper3D v3.0 took lab-based DCT to the next level of throughput, versatility and representative volume mapping. This was done by introducing three advanced scanning schemes – helical phyllotaxis, helical phyllotaxis raster and helical phyllotaxis HART [5], aimed towards much larger sample volumes and varied sample geometries, and by the release of CrystalCT™, a dedicated microCT system for 3D grain mapping.

GrainMapper3D v3.1 released a major enhancement in the reconstruction algorithm and the introduction of reconstruction boosting. The benchmark testing results show a 3.4× speed up in reconstruction time compared to v3.0 and a 24× speed up compared to v2.0.

This basically means that the reconstruction times for conventional and advanced DCT scans should now be counted in hours and days, respectively, rather than in days and weeks.

Along the path of advanced data acquisition, the current GrainMapper3D v3.2 implementation features a further step in enhancing the users' capability to process large datasets. By advanced memory handling, more than 10,000 DCT projections can now be accommodated with the RAM freed up for 3D grain map reconstruction.

With significantly enhanced versatility built into each new version, the GrainMapper3D reconstruction engine for lab-based DCT continues to expand capabilities and enables addressing a widening range of scientific and engineering problems.

[1] S. A. McDonald et al., Sci. Rep. 5, 14665 (2015)
[2] C. Holzner et al., Micros. Today 24, 34-43 (2016)
[3] F. Bachmann et al., J. Appl. Crystallogr. 52, 643–651 (2019)
[4] S. Niverty et al., JOM. 71, 2695–2704 (2019)
[5] J. Oddershede et al., Integr. Mater. Manuf. Innov. 11, 1-12 (2022)

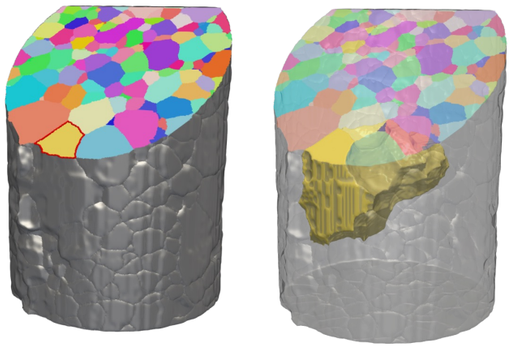


Figure. The abnormal grain exhibits as a normal grain at certain characterization surface, with its majority of the volume in the bulk of the sample.

Sample courtesy of B. R. Patterson, University of Florida, USA.

Structural heterogeneity is frequently observed in thermo-mechanically treated materials. The non-homogeneous microstructure poses a challenge for 2D examination on sectioned sample surfaces, while 3D characterization approaches have the natural advantage in capturing the structural anisotropy present over large length scales. With 3D grain map from Lab-based DCT, unbiased quantitative analysis of grain statistics is guaranteed.

The complex nature of materials structure sets sophisticated demands for sample characterization – usually no single approach is sufficient to fully reveal the necessary information to interpret observed phenomena. Integrated multimodal imaging can build the correlation among different micro-structural features. The complementarity of DCT and other imaging modalities such as absorption CT integrated on the same x-ray microscope enables correlation of the resulting grain map with complementary information on various microstructural features such as cracks, porosities, particles or secondary phases.

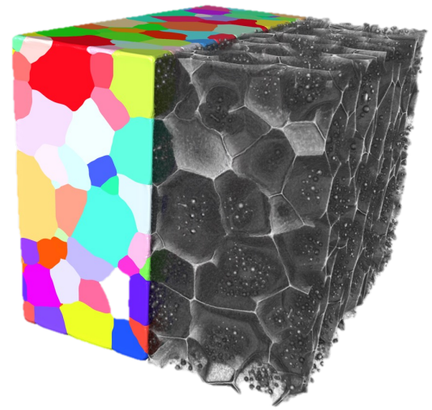
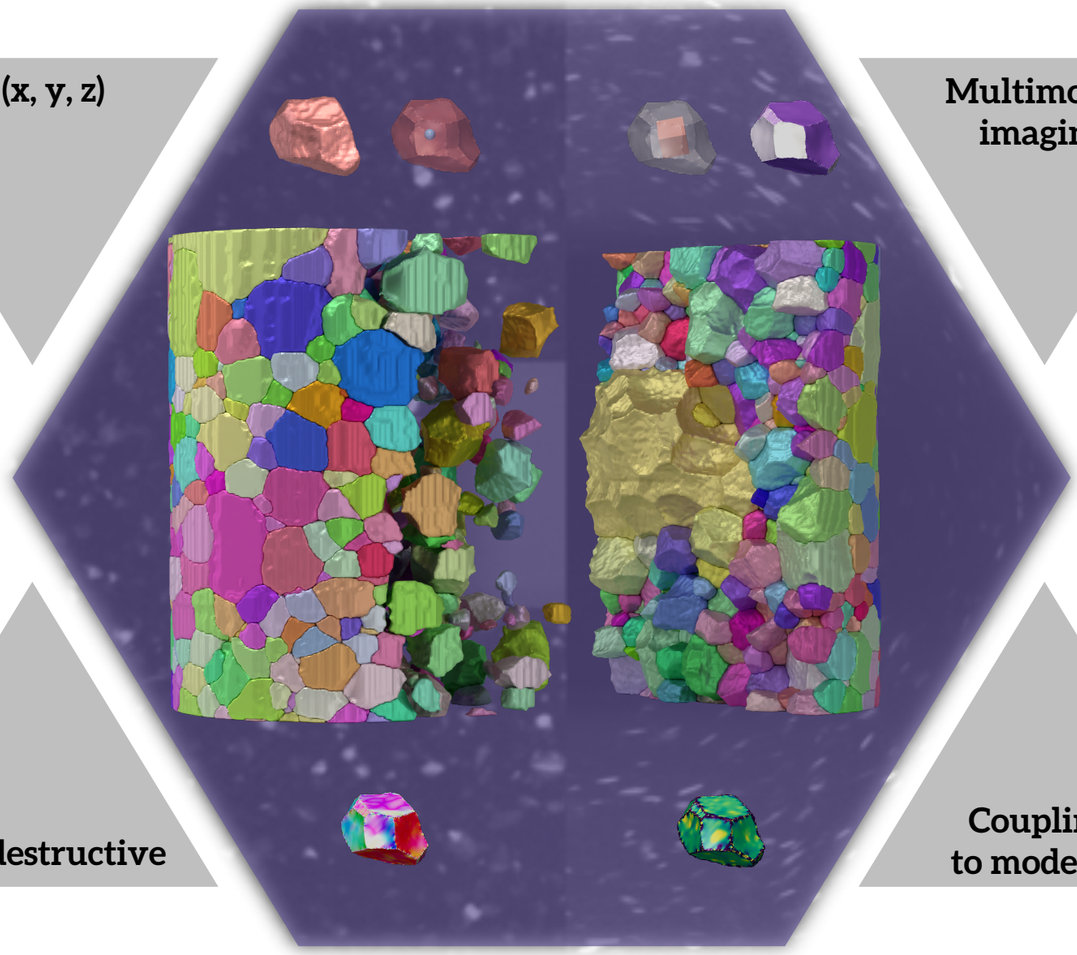


Figure. Al-4wt% Cu sample with Cu-rich phases forming precipitates and segregating to grain boundaries of the Al matrix, which can be characterized from absorption contrast tomography. With Lab-based DCT, the grain structure can be mapped out, allowing a correlative analysis of different microstructure features.

Sample courtesy of J. Dake, University Ulm, Germany.

3D (x, y, z)



Multimodal imaging

Non-destructive

Coupling to modeling

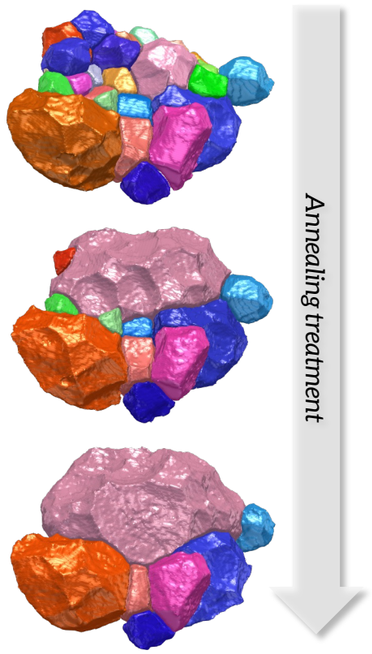


Figure. The evolution of a cluster of ferrite iron grains during a designed annealing treatment. A grain growing in an abnormal manner is captured within the reconstructed 3D grain maps at three annealing stages.

Sample courtesy of B. R. Patterson, University of Florida, USA.

As a non-destructive 3D technique, Lab-based DCT provides the accessibility for in-depth studies of temporal variations in crystallographic grain structure through 4D (x, y, x, time) experiments. Direct interpretation is therefore possible as the grain structural evolution is followed correspondingly.
The non-destructive nature of lab-based DCT allows a variety of subsequent processing to be carried out on the same sample, so phenomena related to materials damage and deformation can be explored with comprehensive crystallographic information as input.

Continuum crystal plasticity modeling is a powerful tool to examine, interpret, as well as predict, the deformation behaviors of polycrystalline materials. Lab-based DCT, particularly now equipped with advanced acquisition schemes, provides a routine solution for experimentally acquiring explicit 3D grain structures, to be used as either input or validation of computational results from modeling. Sample geometries that are routinely used for in-situ experiments like the dog bone in the example can be imaged in their native state, enabling a direct coupling of experimental results and simulations.

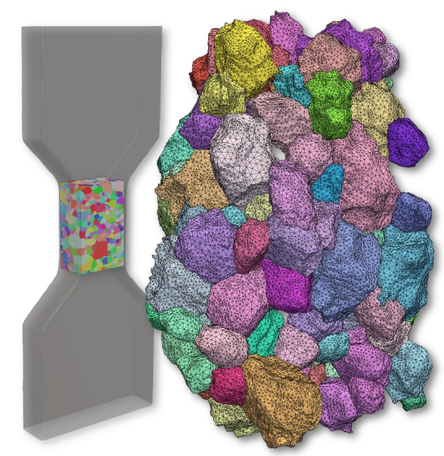
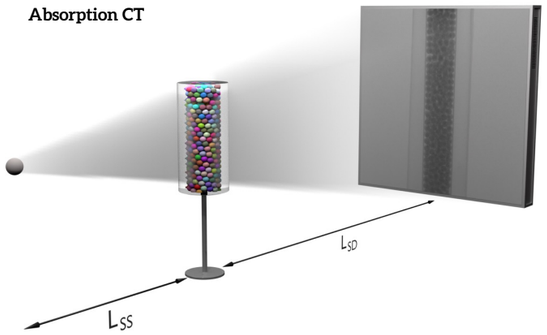
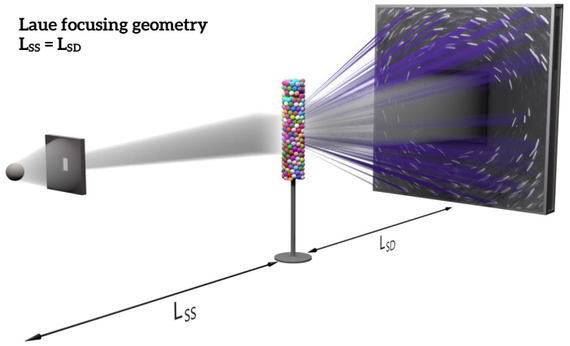
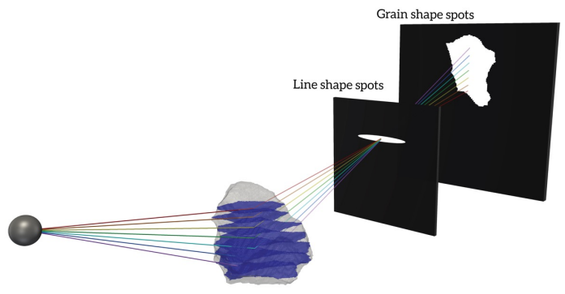


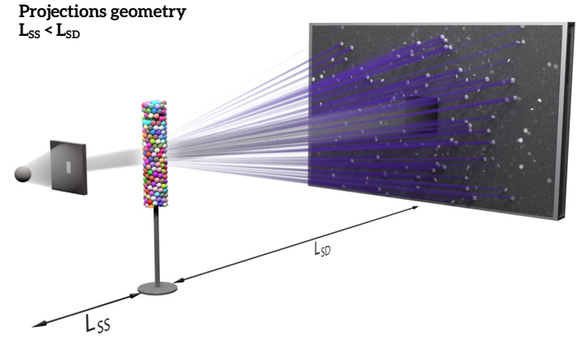
Figure. A dogbone sample with gauge section characterized by Lab-based DCT. The 3D grain map at initial state serves as the basis for interpretating the deformation behaviors during the followed tensile testing.

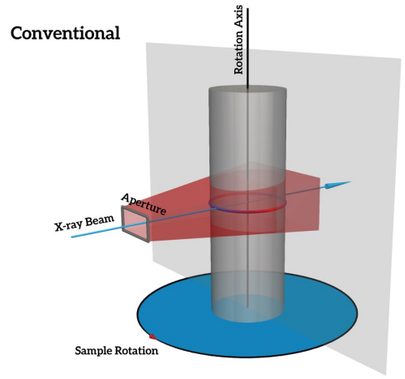
Sample courtesy of M. Kobayashi, Toyohashi University of Technology, Japan.

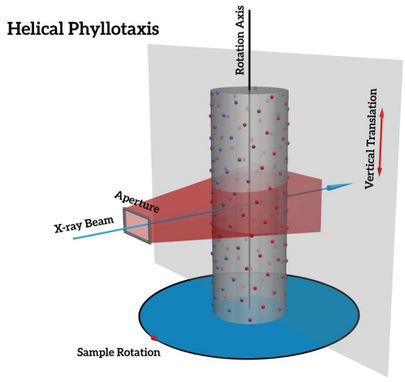


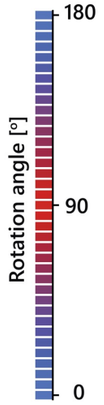


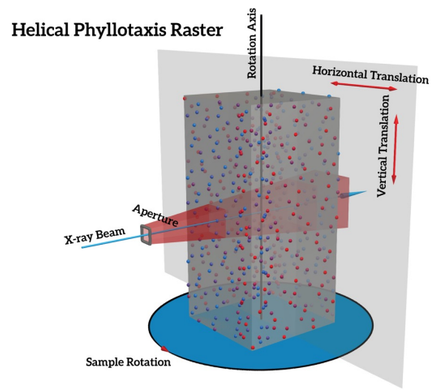


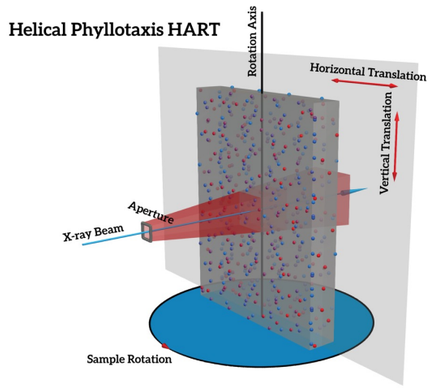












Lab-based DCT experimental setup

A Lab-based DCT experiment consists of two scans:

- **Absorption CT**, where contrast is gained by the x-ray attenuation coefficients of materials, enabling a 3D reconstruction of phase differences, *i.e.* features like cracks, defects and inclusions. Projections are collected through a 360° rotation of the sample illuminated by the direct beam and used to define the sample outline.

- **DCT**, where contrast is gained by Bragg diffraction of individual grains inside the sample, enabling a 3D reconstruction of crystallographic orientations and shapes of individual grains, *i.e.* a 3D space-filling grain map. Projections are collected while the sample is rotated (and translated for advanced acquisition), to cover the sample region of interest (ROI) uniformly. For DCT the divergent, polychromatic x-ray beam is constrained by an aperture to illuminate a volume in the sample defined by the field of view (FOV). A beamstop after the sample blocks transmitted x-rays on the detector to increase sensitivity towards the substantially weaker diffraction signals.

The setup in **Laue focusing geometry**, exploits that for a divergent point source of x-rays, a grain diffracts such that the x-rays are focused one-dimensionally in the Laue focal plane at a sample-detector distance (L_{SD}) equal to the source-sample distance (L_{SS}). The typical working distance is 12-20 mm. A high-resolution detector is placed at this distance, and the Laue focusing effect makes the diffracted signals appear as line-shaped spots optimizing the signal-to-noise ratio, minimizing spot overlap and allowing to record larger volumes or more grains.

The **projection geometry** setup, employs a flat panel detector at a typical working distance of 200 mm < L_{SD} < 550 mm while the source-sample distance is kept significantly shorter, typically 10 mm < L_{SS} < 100 mm. These working distances and the detector efficiency offer some potential advantages for larger samples. The projection geometry, $L_{SS} < L_{SD}$, means that the shape of the grains is projected into the shape of the diffractions spots as can be seen on the schematic.

DCT Advanced Acquisition

Conventional DCT data collection assumes that the sample ROI is fully illuminated by the aperture FOV for all rotation angles. This puts a major constraint on the types of samples that can be imaged, often requiring researchers to modify the sample to a smaller cylindrical or "matchstick" sized specimen. In order to accommodate analysis of more complex, real sample geometries with reduced sample preparation time, new advanced scanning modes have been introduced.

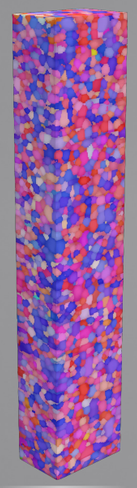
Advanced scanning of samples that do not fulfil the criteria for a conventional DCT scan can be performed with the DCT Acquisition Wizard. The advanced scanning schemes allow seamless collection of DCT data for larger, irregularly shaped sample volumes with a uniform sample illumination both angularly and spatially. This is done by combining complex rotational and translational sample stage movements to enable optimal and efficient collection of DCT data. Reconstruction of the corresponding data is equally seamless, simultaneously using all data to reconstruct the full illuminated volume without the need for stitching of data subsets or sample subvolumes.

The three advanced scanning schemes offered by the DCT Acquisition Wizard are [5]:

- **Helical Phyllotaxis** – This scan covers samples with a vertical extent larger than the illuminating x-ray beam height by means of a golden angle rotation of ~137.5° combined with a vertical translation on the order of ~1–5 µm between consecutive projections.

- **Helical Phyllotaxis Raster** – This scan combines the sample rotation and vertical translation of the helical phyllotaxis scan with a fixed number of horizontal translation steps for all projection angles to cover samples wider than the FOV.

- **Helical Phyllotaxis HART** (high-aspect ratio tomography) – This scan mode is tailored to specimens with plate-like geometries. It is similar to the helical phyllotaxis raster scan, except the number of horizontal steps is adapted to tightly fit the ROI at every projection angle.



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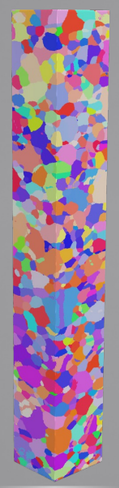

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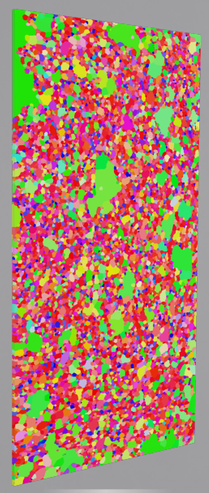

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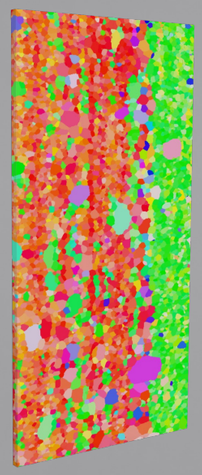

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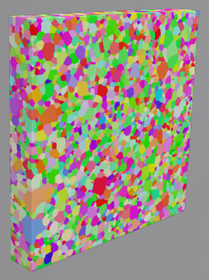

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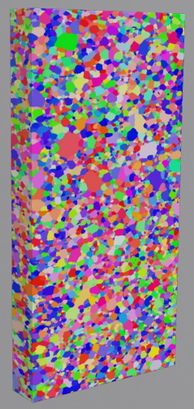

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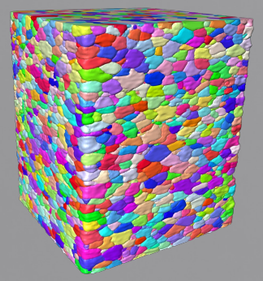

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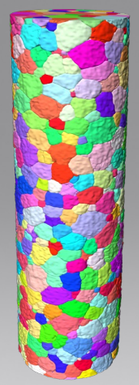

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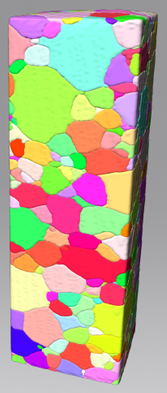

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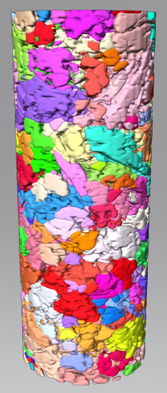

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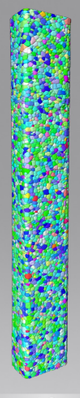

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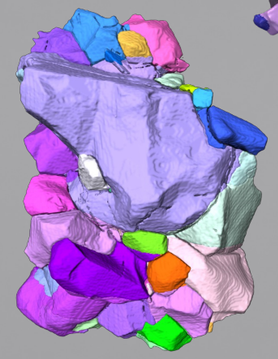
Application Gallery



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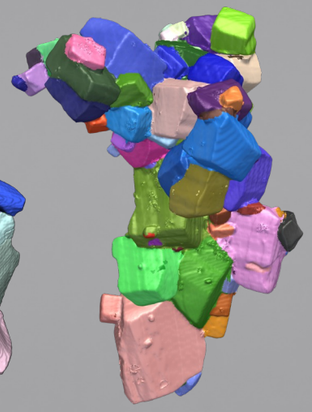

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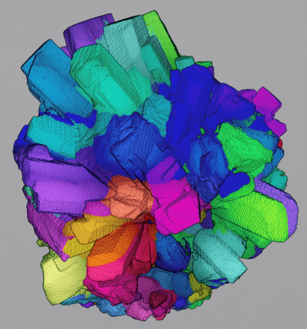

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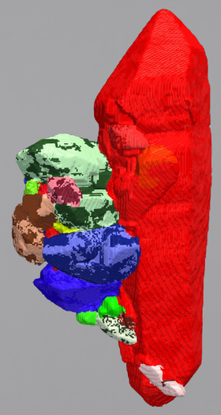

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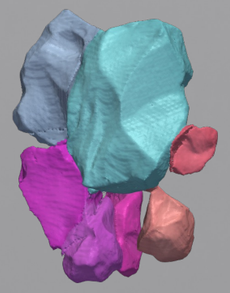

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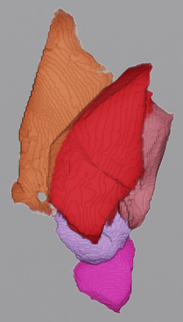

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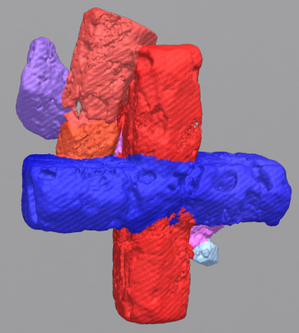

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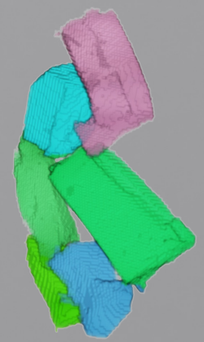

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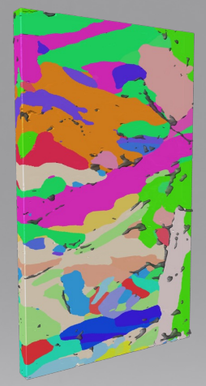
22



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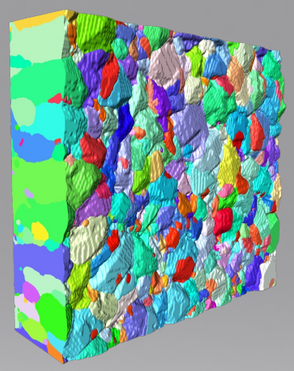
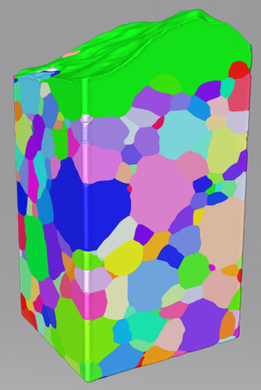


24



25

1 Low carbon steel, cubic, 2×0.4×0.4 mm (Prof. M. Kimura, KEK)
2 316L stainless steel, cubic, 4.4×0.7×0.7 mm (Prof. G. Winther, DTU)
3 Electrical steel, cubic, 40×20×0.4 mm (Prof. P. Yang, USTB)
4 Electrical steel, cubic, 4×2×0.08 mm (Dr. L. Meng, CISRI)
5 Electrical steel, cubic, 3×3×0.4 mm (Dr. I. Petryshynets, SAS)
6 Electrical steel, cubic, 4.4×2×0.5 mm (Prof. L. Chang. NSYSU)
7 Casted Al alloy, cubic, 3.5×2.7×2.7 mm (Ms F. Xue, BaoWu Steel)
8 AA5657, cubic, 4×2×0.6 mm (Dr. R. Sanders, Novelis)
9 Al alloy, cubic , Ø~1.4×5.5 mm (Dr. J. Dake, Ulm Univ.)
10 Ti alloy, cubic, 3×1×0.8 mm
11 Ti alloy, hexagonal, Ø~0.6×2 mm, (Dr. Y. Yang, IPE-CAS)
12 Ti alloy, hexagonal, 20×3×0.4 mm (Dr. C. Ribart, Mines ParisTech)
13 Ni superalloy, cubic, Ø~2×9 mm (Dr. A. Barbeau, SAFRAN)

14 Peridotite, orthorhombic, height - 6 mm
15 Pyrite, cubic, height - 10 mm
16 Aragonite, orthorhombic, heigh - 15 mm
17 Quartz, trigonal, height - 30 mm
18 Olivine, orthorhombic, height - 3 mm
19 Zircon, tetragonal, height - 2 mm
20 Staurolite, monoclinic, height - 15 mm
21 Rhodonite, triclinic, height - 2 mm
22 Sapphire spheres, trigonal, Ø~0.2 mm in capillary Ø~1×2 mm
23 Cast Si, cubic, 12×7×0.6 mm (Prof. A. Shahani, Univ. Michigan)
24 CVD diamond, cubic, 4×4×1.5 mm (Dr. L. Kirste, Fraunhofer IAF)
25 $SrTiO_3$, cubic, 1.2×0.8×0.8 mm (Prof. A. Krause, Univ. Florida)