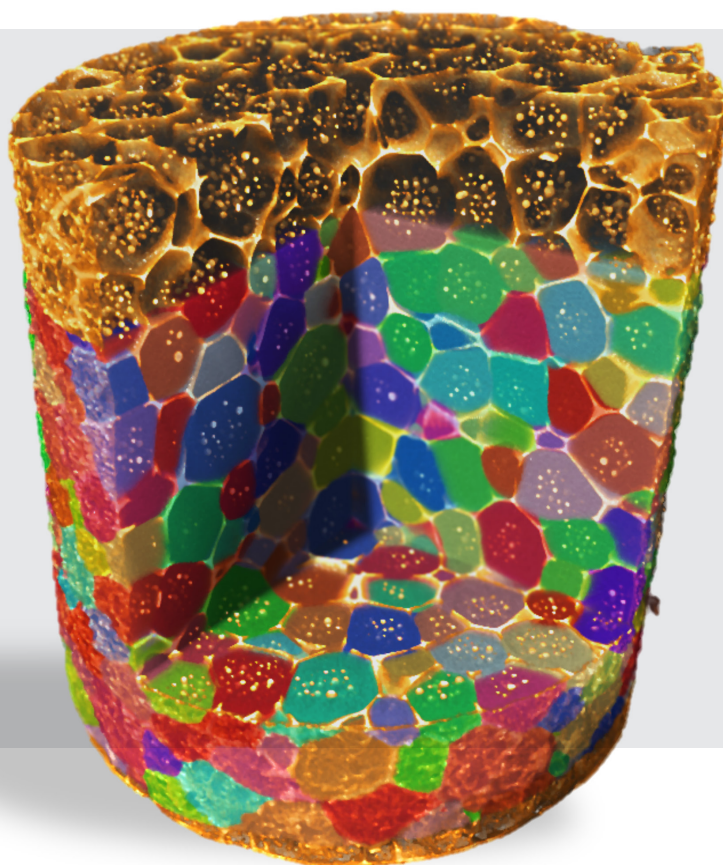# Enabling Premium 3D Crystallographic Imaging in Your Laboratory

Laboratory-based Diffraction Contrast Tomography

ZEISS

Seeing beyond

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ABSTRACT X-ray tomography for 3D non-destructive imaging has been widely adopted and operated under two primary contrast mechanisms for quite some time: X-ray absorption and phase contrast, which both rely on material density differences within the sample. However, single-phase polycrystalline materials (e.g., steels, alloys, ceramics) do not exhibit any absorption contrast that reveals the underlying grain microstructure. Synchrotron-based X-ray imaging methods – such as diffraction contrast tomography (DCT), which provide crystallographic information from the diffraction signals of single-phase polycrystalline samples, non-destructively and in three dimensions (3D) – were the first to successfully demonstrate results in this class of materials almost two decades ago. Now, advancing laboratory X-ray microscopy (XRM) one step further, we describe here the latest capabilities of laboratory-based DCT on ZEISS Xradia 620 Versa and ZEISS Xradia CrystalCT 3D X-ray imaging systems, and present the new research and 3D characterization capabilities this enables.

Introduction

Crystallographic imaging is a metallography technique that commonly uses light and electron microscopy (EM). In recent years, the introduction of 2D and 3D electron back-scattering diffraction (EBSD) techniques have made EM a routine tool for research and/or development related to metallurgy, functional ceramics, semiconductors, geology, etc. The ability to image the grain structure and quantify the crystallographic orientation relationships in such materials is instrumental for understanding and optimizing material properties (mechanical, electrical, etc.) and *in situ* processing conditions.

While 2D EBSD is a surface imaging method and requires careful sample surface preparation, 3D EBSD can provide detailed sub-surface information through a combination of consecutive imaging and ion-milling workflow components. However, the destructive nature of 3D EBSD (via examination of sequential slices) prevents one from directly evaluating the microstructure evolution when subjected to either mechanical, thermal or other environmental conditions. Understanding this evolution process on the exact same sample volume is key to unlocking a more robust understanding of materials performance, along with improved modeling capabilities, and is a key driver of future materials research efforts.

X-ray tomography has traditionally been the 3D non-destructive imaging technique that serves in the investigation of the underlying microstructural information related to voids, defects, and pores, or identifying structural details of phases with different X-ray attenuation. It typically operates under two primary contrast mechanisms: X-ray absorption and phase contrast, which both rely on material density differences within the sample, and produces 3D reconstructed data volumes that can be quantitatively analyzed and used for virtual cross-sectioning to gain microstructural insights in three dimensions. However, typical single-phase polycrystalline materials previously referenced, i.e., steels, alloys, and ceramics, do not exhibit any absorption contrast to reveal the underlying grain microstructure.

In response to this imaging gap, synchrotron-based crystallographic imaging, known as diffraction contrast tomography (DCT), emerged over the past two decades. Using non-destructive X-ray, synchrotron users can quantify grain orientation information in the native 3D environment without physical sectioning. This has led to the logical desire to study the evolution of grain crystallography *in situ* or during interrupted 4D (x, y, z, time) evolution experiments. However, limited regular access to such synchrotron techniques has constrained the ability to perform thorough, longitudinal studies of materials evolution.

Sample Representivity

Virtual materials testing is one of the emerging trends in materials science and engineering, and shows promise in rapid materials discovery, important for continuously evolving industries like aerospace, automotive, energy, and construction. To enable such research, it is crucial to obtain large volumes of real data with increased data representivity and reduced sample specificity that capture the underlying multiscale structure-property relationships while retaining the context of entire length scales. Comprehensive data that capture the intricate mechanisms that operate and govern the mechanical and thermal behavior of a material are vital to validate the accuracy of computational models. The ability to gather information from several cubic millimeters of samples with greater than 10^4 grains and associated grain boundaries, empowers researchers to get an accurate statistical representation of the sample at hand.

Examining such massive sample volumes non-destructively remained largely impractical until now. Figure 1 gives an overview of the relative sample volumes that can be investigated by various techniques.

Inspired by the developments at leading synchrotrons and by the motivation to enable access to the broader research community, ZEISS, in partnership with Xnovo Technology, has successfully enabled a 3D grain mapping modality for the ZEISS Xradia family of laboratory X-ray tomography microscopes[1–4]. An example result of non-destructive 3D grain mapping on a ZEISS X-ray microscope is shown in Figure 2. Due to the ease of access to grain mapping within the base X-ray absorption tomography setup, laboratory-based DCT can be efficiently coupled to *in situ* sample environments within the microscope or subject to an extended time evolution 4D experiment (across days, weeks, months) – a unique practical strength of laboratory-based XRM/DCT experiments[3, 5].

Furthermore, laboratory-based DCT can be integrated into many multimodal workflows through the existing correlative ecosystem of ZEISS microscopy products to seamlessly gather unique insights from a range of microscopy techniques and uncover valuable information from the sample[6–8].

Laboratory-based DCT : How it works

The DCT feature is enabled on the ZEISS Xradia Versa 3D X-ray microscope as the optional LabDCT Pro module and the purpose-built ZEISS Xradia CrystalCT microCT (hereafter referred to as "LabDCT" for either). It leverages the robust design and high stability of the X-ray imaging architecture of ZEISS Xradia platforms. Additional hardware consisting of aperture and beam stop assemblies are incorporated in the DCT module and function without compromising on the core tomographic imaging capabilities of the base X-ray microscope.

LabDCT set-ups are shown in Figure 3. The divergent, polychromatic X-ray beam is constrained through an aperture to illuminate a region of interest (ROI) of the sample. A beamstop beyond the sample blocks transmitted X-rays on the detector to increase sensitivity towards the substantially weaker diffraction signals.

Non-destructive
LabDCT Pro and CrystalCT
Volume: >>(1000)³ μm³ and beyond
Isotropic voxels: up to 2 μm
Voxel aspect ratio = 1

Prior Non-destructive DCT
Volume: (1000)³ μm³
Isotropic voxels: up to 2 μm
Voxel aspect ratio = 1

PFIB + EBSD
Volume: (250)³ μm³
Slice thickness: 0.2 - 5 μm
Voxel aspect ratio ≥ 50

Ga-FIB + EBSD
Volume: (250)³ μm³
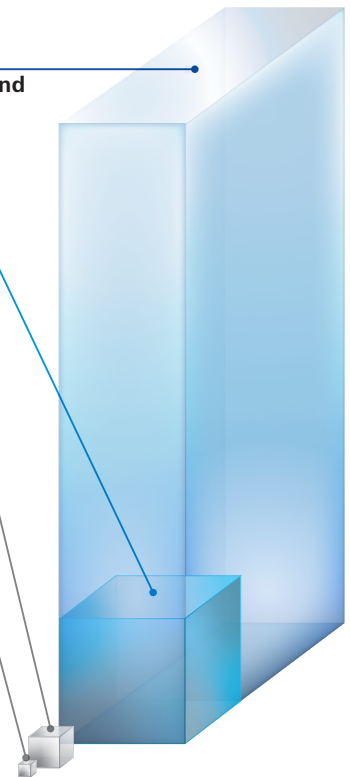Slice thickness: 10 nm
Voxel aspect ratio ≥ 1

Figure 1 *Comparison of the volumes that can be analyzed using various destructive and non-destructive volume imaging methods*
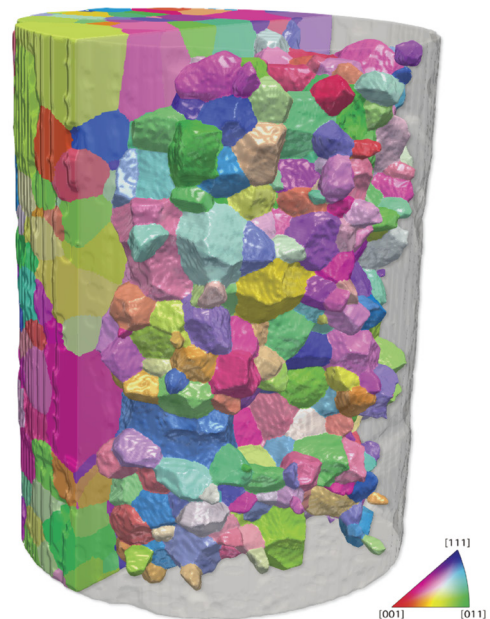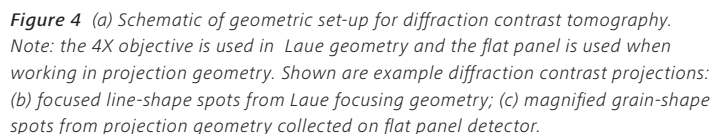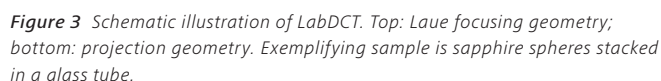

Figure 2 *3D grain map of an Armco iron sample. The grains lying in the outer periphery of half the sample have been rendered transparent to reveal inner grain clusters. The sample, courtesy of Prof. Burton R. Patterson, University of Florida, US, has a diameter of 1 mm and height of 1.6 mm.*

A DCT experiment can now be implemented with two geometries: a Laue focusing geometry and a projection geometry based on the equipped detector system. With the Laue focusing geometry, the sample is placed equidistant of the source and detector ($L_{SS} = L_{SD}$), using the dedicated DCT detector with 4X optical magnification. With the projection geometry, the sample is placed closer to the source than to the detector ($L_{SS} < L_{SD}$), using a flat panel detector and pure geometric magnification.

The DCT projection images obtained using the two modes described in the earlier section produce distinct diffraction patterns on the detector due to their focusing geometries and are presented in Figure 4. In the Laue focusing mode, grains produce diffraction spots that are sharp lines as shown in Figure 4b. on the scintillator-coupled objective-based detector. The projection geometry uses the flat panel detector in the magnifying or defocused position to collect diffraction spots, which appear as projected shape profiles of the corresponding diffracting grains. ZEISS Xradia 620 Versa microscope uses the 4X DCT objective and can optionally be equipped with the large area Flat Panel Extension (FPX) for enabling projection mode acquisitions. Both of these modes of data acquisition are supported by LabDCT Pro. ZEISS Xradia CrystalCT uses its dedicated flat panel detector in projection mode for grain mapping.
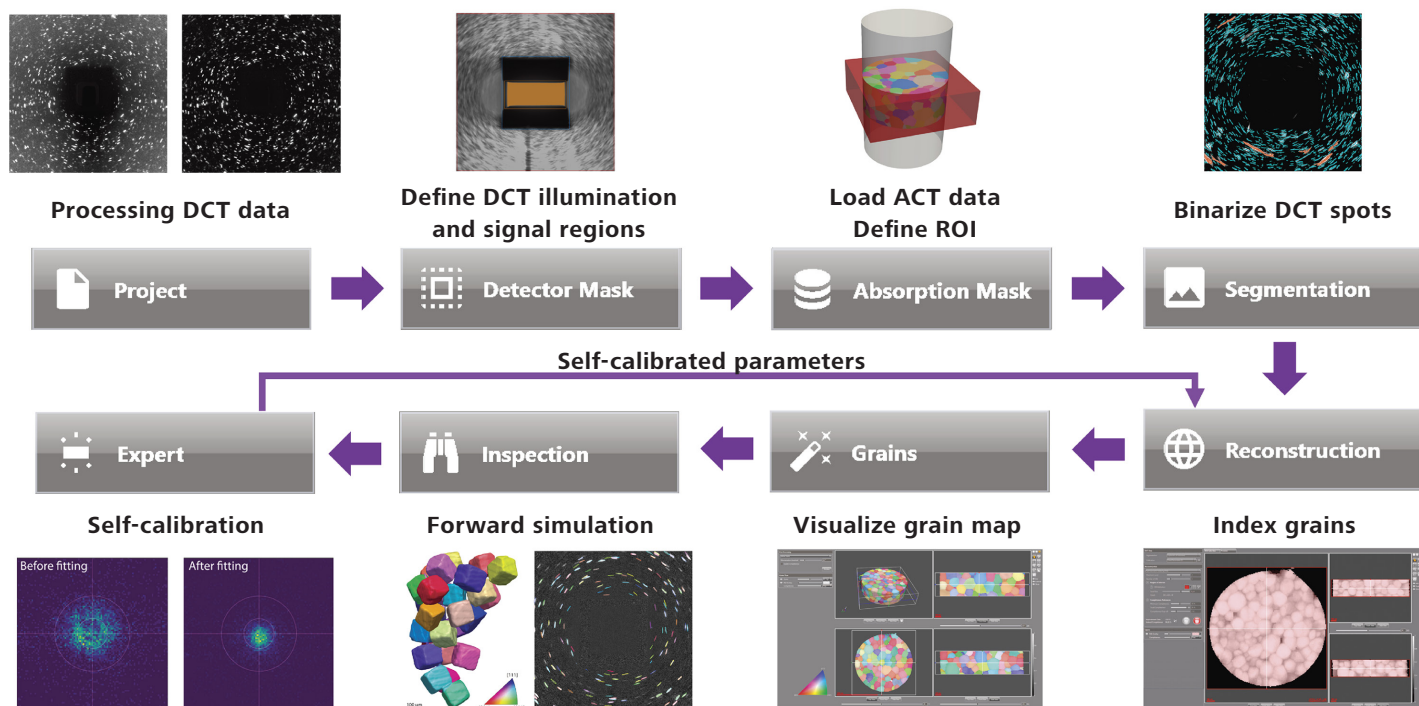
Laue Focusing Geometry

$L_{SS} = L_{SD}$

Projection Geometry

$L_{SS} < L_{SD}$

Figure 3 Schematic illustration of LabDCT. Top: Laue focusing geometry; bottom: projection geometry. Exemplifying sample is sapphire spheres stacked in a glass tube.

Source	Source	Objective	Flat Panel
		Focus position	Magnifying/defocused position

Figure 4 (a) Schematic of geometric set-up for diffraction contrast tomography. Note: the 4X objective is used in Laue geometry and the flat panel is used when working in projection geometry. Shown are example diffraction contrast projections: (b) focused line-shape spots from Laue focusing geometry; (c) magnified grain-shape spots from projection geometry collected on flat panel detector.

Figure 5 *Overview of the intuitive workflow of GrainMapper3D v3 for LabDCT*

The data acquisition workflow in a LabDCT experiment consists of two scans: an absorption contrast tomography (ACT) scan to define the sample outline, and a diffraction contrast tomography (DCT) scan in which a specified number of diffraction contrast projections are collected as the sample rotates and translates. The collected ACT and DCT data are then imported into GrainMapper3D[9] developed by Xnovo Technology for further processing and reconstruction. Information on grain morphology, crystallographic orientation, size and centroid position is available from the reconstructed 3D grain map.

Facing non-expert end users, GrainMapper3D is an embedded intuitive graphical user interface (GUI) and workflow, equipped with advanced functionalities guiding towards high fidelity reconstructions. The processing steps comprising the latest GrainMapper3D v3.0 workflow are headlined and illustrated in Figure 5. The GUI provides a variety of tailored data preprocessing approaches and reconstruction parameters, instant visual feedback on data quality and reconstruction progress, a suite of validation tools, as well as export to a widely accepted 3D data format—making a complex scientific method accessible for a non-expert user.

LabDCT: A brief history

Bringing this sophisticated crystallographic imaging technique from synchrotron to the laboratory platform has been an exciting while challenging journey. Since its launch in July 2015, laboratory-based DCT has offered the only commercially available non-destructive 3D grain mapping solution for the non-synchrotron laboratory. The reconstructed 3D grain structure was originally represented by colored cubes – with the size, position and rotation of the cubes representing the size, centroid position and orientation of the grains. GrainMapper3D v2.0 released the full 3D grain morphology reconstruction, which was a substantial contribution as LabDCT now enabled grain boundary characterization capabilities. Three releases during 2019 and 2020 – GrainMapper3D v2.1, v2.2 and v2.3 – marked solid steps in equipping LabDCT to be a powerful tool for materials scientists with more reliability, flexibility and extended applicability [10, 11]. LabDCT Pro enables an additional DCT imaging geometry and three advanced data acquisition schemes, unlocking seamless data acquisition and superior sample representivity, aimed towards much larger sample volumes and varied sample geometries. With significantly enhanced versatility built in, LabDCT Pro further expands its capability to address a wider range of scientific and engineering problems.
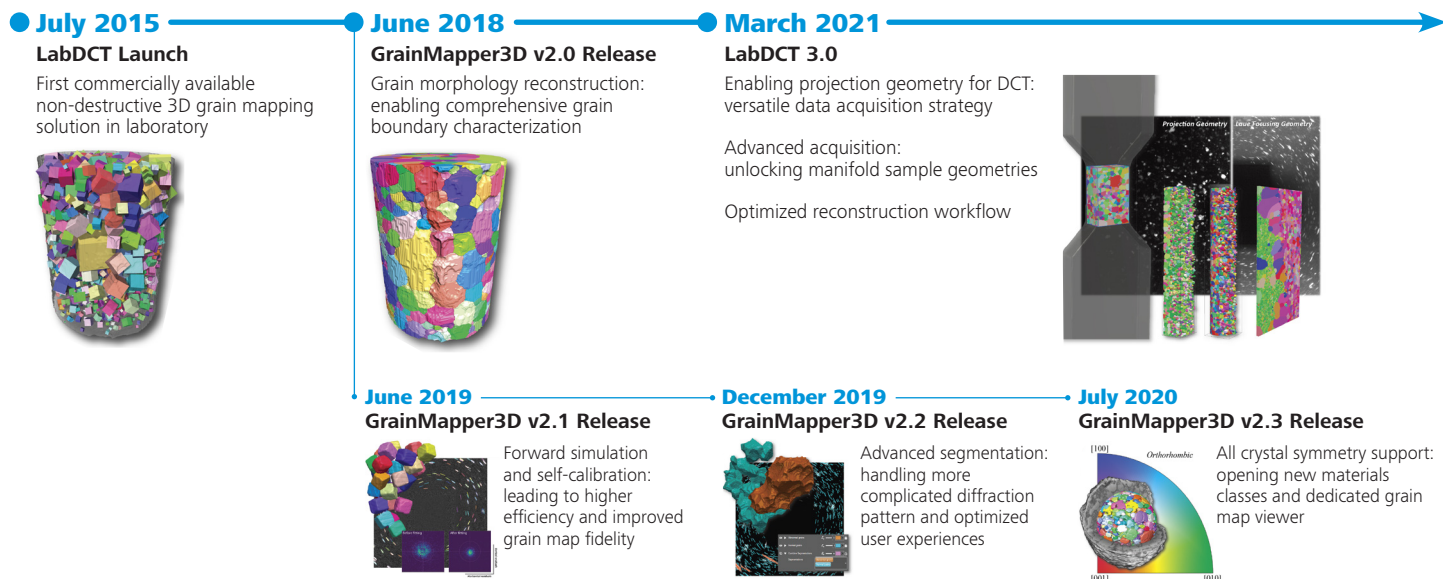
July 2015
LabDCT Launch

First commercially available non-destructive 3D grain mapping solution in laboratory

June 2018
GrainMapper3D v2.0 Release

Grain morphology reconstruction: enabling comprehensive grain boundary characterization

March 2021
LabDCT 3.0

Enabling projection geometry for DCT: versatile data acquisition strategy

Advanced acquisition: unlocking manifold sample geometries

Optimized reconstruction workflow

June 2019
GrainMapper3D v2.1 Release

Forward simulation and self-calibration: leading to higher efficiency and improved grain map fidelity

December 2019
GrainMapper3D v2.2 Release

Advanced segmentation: handling more complicated diffraction pattern and optimized user experiences

July 2020
GrainMapper3D v2.3 Release

All crystal symmetry support: opening new materials classes and dedicated grain map viewer

Figure 6 History of LabDCT development

DCT Results Validation

The results generated by LabDCT have been validated using grain information derived using several different independent imaging modalities such as 2D/3D EBSD, synchrotron-based phase contrast tomography, absorption contrast tomography (of model polycrystalline samples), and synchrotron DCT data.

Two different samples – an aluminum-alloy with copper-decorated grain boundaries (see also cover image) and a polycrystalline Ti-alloy (Timet 21S) – are presented here to demonstrate the grainshape and orientation reconstruction. Figure 7 shows the comparison of a 3D grain structure derived from DCT data (a), with the 3D grain structures derived from high resolution X-ray absorption tomography (b) of an Al-alloy with segregated Cu on its grain boundaries.

Due to the density differences between Al and Cu, the grain boundaries can be visualized through conventional X-ray absorption tomography. Figure 7b displays the grain structure of the sample based on segmentation of the absorption contrast dataset. The 3D grain structures agree remarkably well with differences being located at the grain boundaries themselves as shown through the difference map in Figure 7(d). Principal contributing factors to these differences are the segregation of the Cu itself, leading to an uneven distribution and missing some grain boundaries (compare Figure 7(c)). Furthermore, the subsequent segmentation of the grain boundary network from the absorption tomography reconstruction will introduce uncertainties in the location of the boundaries.
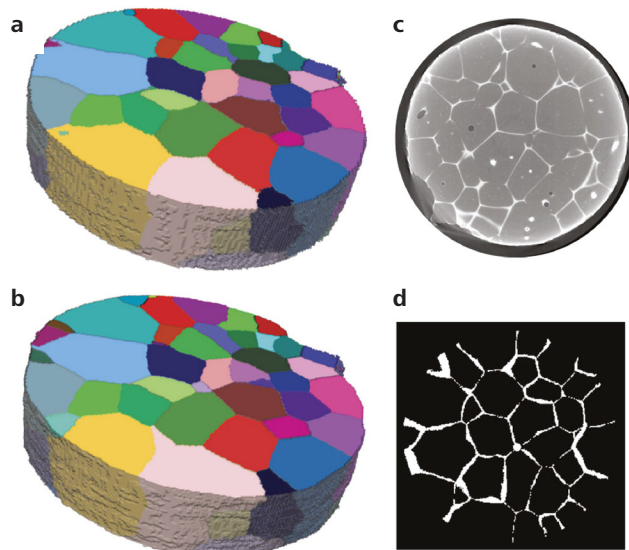

Figure 7 (a) LabDCT reconstruction of Al-Cu sample. (b) Volumetric segmentation from absorption tomography. Colors in (a) and (b) indicate a grain index. (c) Virtual slice through the absorption tomography in grayscale. (d) Virtual slice of the difference map between the data of (a) and (b); the binarized color scale indicates regions of index differences in white and regions with no difference in black.

Shown in Figure 8 is the comparative results of LabDCT and synchrotron DCT of the Ti-alloy sample. Figure 8(a) shows the reconstructed 3D grain map from LabDCT, where the grains are colored according to their crystallographic orientation in relation to the vertical sample axis (inverse pole figure). Good correspondence is found between laboratory-based DCT and synchrotron DCT mapping of the same sample for both grain orientation and morphology accuracy. Analyzing 93 matched grains gives mean grain pair misorientation of 0.031 deg and mean grain boundary distance of 7.1 µm (less than 2 voxels).

Figure 8 (a) Visualization of a 3D grain map of a titanium alloy (Timet 21S) from reconstruction with the LabDCT GrainMapper3D software. Inverse pole figure color coding highlights the crystallographic information. (b) Virtual cross section through a synchrotron DCT 3D grain map of the same sample; (c) corresponding virtual cross section through the LabDCT 3D grain map; (d) misorientations and (e) grain boundary distances between the two independently measured virtual cross sections. All scale bars: 100 µm.

Advanced Acquisition

Conventional DCT data collection assumes that the ROI in the sample is fully illuminated by the aperture field of view (FOV) for all rotational angles of the sample. The conventional method of scanning puts a major constraint on the types of samples that can be imaged in DCT, often requiring the researcher to modify the sample to a smaller cylindrical or "matchstick" sized specimen. This has been a major limitation since it adds cumbersome, time-consuming sample preparation steps and changes the native state of a sample, potentially also introducing some mechanical stresses. In order to avoid this restriction and also to accommodate analysis of more complex, real sample geometries with reduced sample preparation, a new scheme of scanning modes has been introduced, hereafter referred to as advanced scanning. The advanced scanning modes combine complex rotational and translational sample stage movements to enable efficient collection of DCT projections, optimally illuminating non-standard sample geometries.

Advanced scanning of samples that do not fulfill the *Conventional DCT* criterion can be performed with the new DCT Acquisition Wizard, which allows for seamless collection and subsequent reconstruction of DCT data for larger, irregularly shaped sample volumes by combining sample rotation and translations.
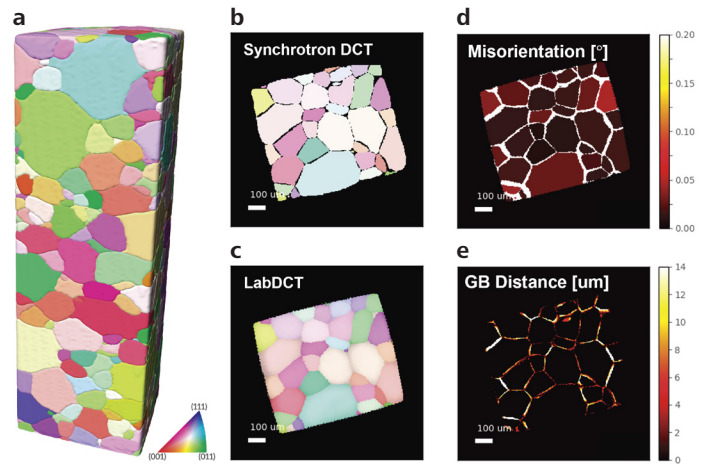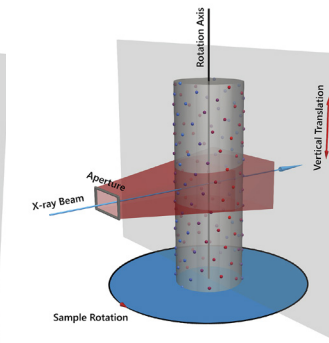
Figure 9 and Table 1 on the next page present an overview of the three advanced scanning schemes offered by the DCT Acquisition Wizard compared to conventional DCT acquisition:

- **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.
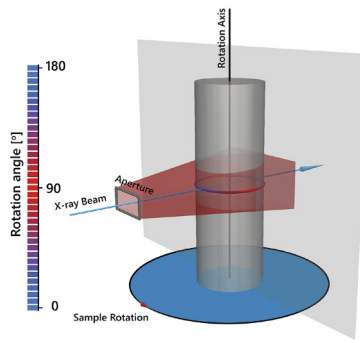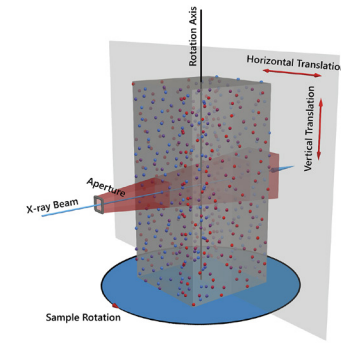
a. Conventional **b. Helical Phyllotaxis** **c. Helical Phyllotaxis Raster** **d. Helical Phyllotaxis HART**
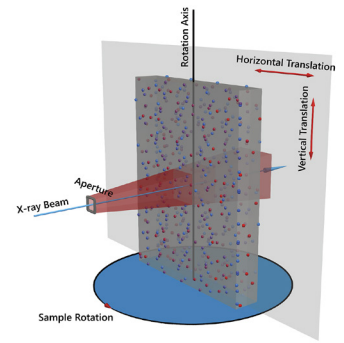
Figure 9 *Schematic illustration of (a) the conventional DCT scanning mode and three advanced DCT scanning modes: (b) Helical Phyllotaxis, (c) Helical Phyllotaxis Raster and (d) Helical Phyllotaxis HART. Points on sample surface mark the position where the center of the beam intersects with the sample surface for an individual diffraction projection, colored by the rotation angle of the sample.*

Scan modes	Conventional	Helical Phyllotaxis	Helical Phyllotaxis Raster	Helical Phyllotaxis HART
FOV vs ROI	ROI fits in FOV	ROI taller than FOV	ROI larger than FOV	ROI larger than FOV
Rotation stepping	360°/N	137.5°	137.5°	137.5°
Vertical translation	No	Yes	Yes	Yes
Horizontal translation	No	No	Yes	Yes – adaptive

Table 1 *Overview of conventional and advanced scanning schemes along with the associated stage motions*

The use of conventional and advanced scanning modes has been demonstrated on a wide variety of engineering materials as presented in Figures 10 to Figure 14, highlighting the unique scenarios in which each of the advanced scanning modes may be used depending on the sample characteristics and geometry.
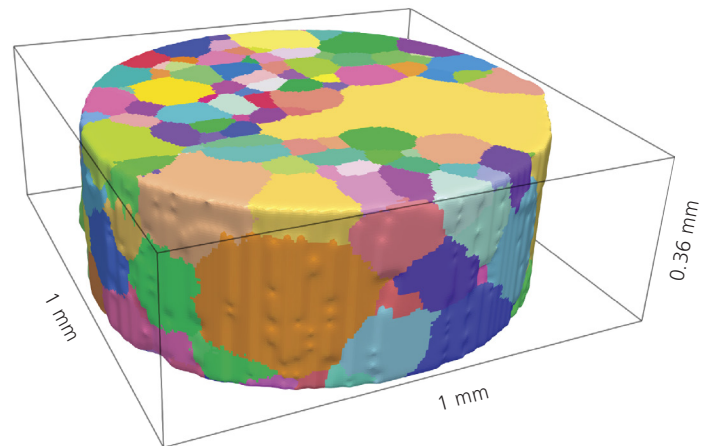

Figure 10 *Example 3D grain maps imaged with conventional scanning mode. Armco iron sample. Courtesy of Prof. Burton R. Patterson, University of Florida, USA*
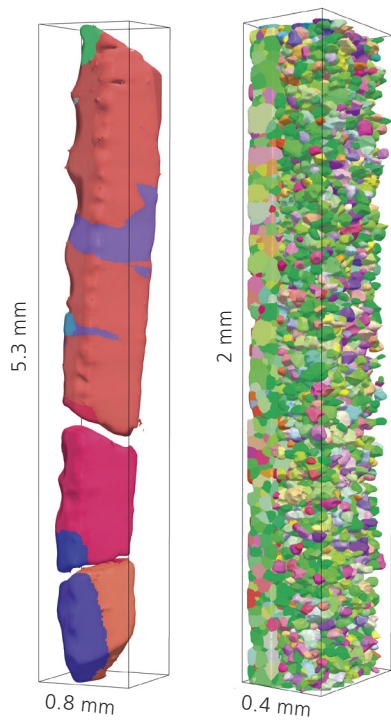
Figure 11 Example 3D grain map imaged with helical phyllotaxis scanning mode. Left: poly Si sample. Courtesy of Prof. Ashwin Shahani, University of Michigan, USA. Right: low carbon steel sample. Courtesy of Prof. Masao Kimura, KEK, Japan.
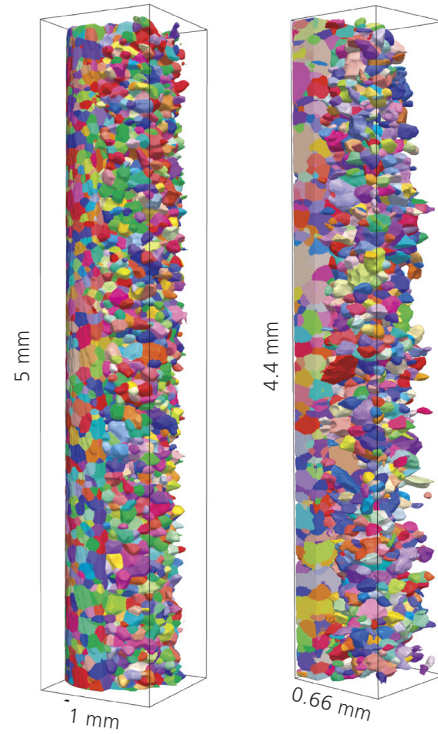

Figure 12 Example 3D grain map imaged with helical phyllotaxis raster scanning mode. Left: Armco iron sample Courtesy of Prof. Burton R. Patterson, University of Florida, USA. Right: Austenitic stainless-steel sample. Courtesy of Prof. Grethe Wither, Technical University of Denmark
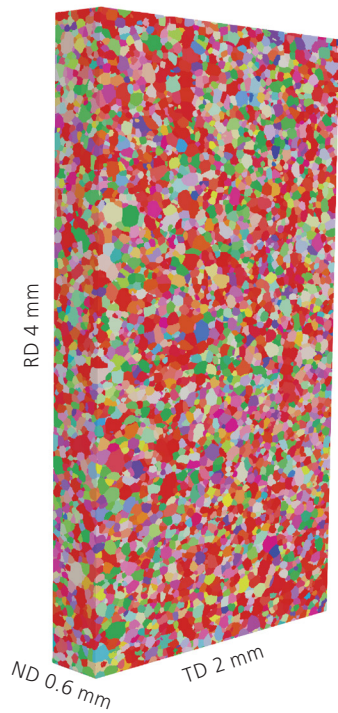
Figure 13 Example 3D grain map imaged with helical phyllotaxis HART scanning mode. AA5657 sheet sample. Courtesy of Dr. Robert Sanders, Novelis, USA.
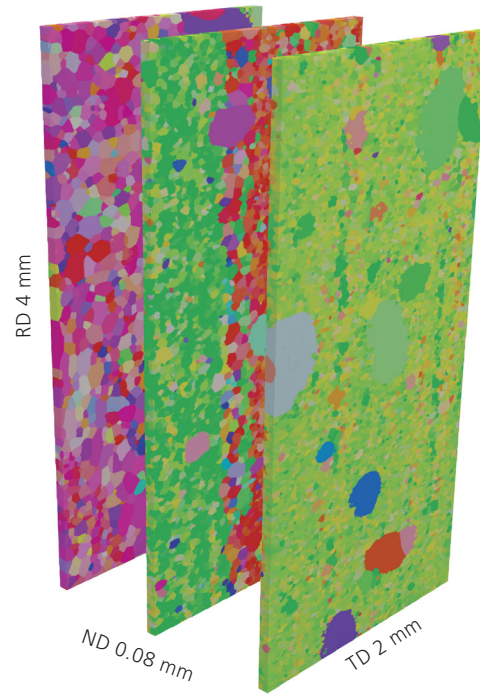

Figure 14 Example 3D grain map imaged with helical phyllotaxis HART scanning mode. Three pieces of oriented electrical steel sheet samples. Courtesy of Dr. Li Meng, China Iron and Steel Research Institute, China.

Application advantages

Complementary 3D Characterization

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 large-scale structural anisotropy. With the grain morphology and crystallographic orientation, the grain boundary can be characterized. The grain boundary characteristics, illustrated in Figure 15, are essential information to analyze grain boundary related behaviors such as preferential precipitation and grain boundary embrittlement. Obtaining the necessary parameters to fully describe a grain boundary in a polycrystalline structure is beyond the reach of 2D characterization techniques and is only achievable through a 3D approach.

Multimodal Imaging with LabDCT

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 (see example in Figure 16) can build the correlation among multiple micro-structural features.

The complementary nature of diffraction contrast tomography and other imaging modalities such as absorption contrast tomography integrated on the X-ray microscope can be used to provide unprecedented insight into the structure of materials, enabling correlation of the resulting grain map along with information on various microstructural features such as cracks, porosities, particles or secondary phases.

Multimodal imaging combining LabDCT with absorption contrast tomography has been employed to reveal the grain structure evolution during densification of a sintered copper sample [3, 12], explore precipitation location in polycrystalline silicon material [6] and investigate the preferential penetration path of gallium in an aluminum matrix [7].
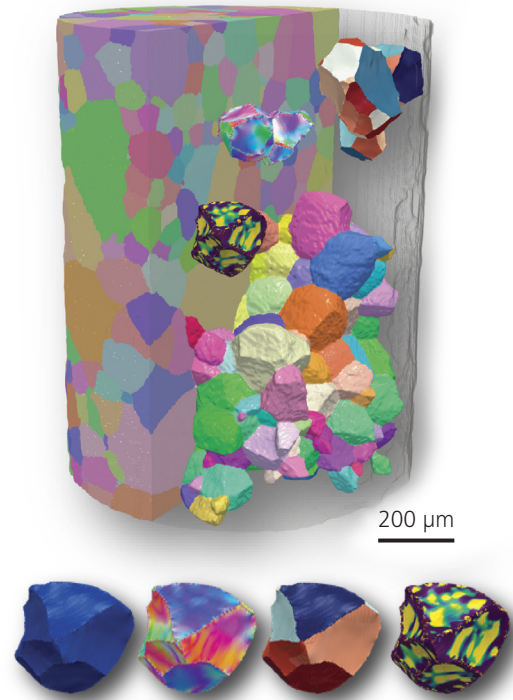

200 µm

Figure 15 3D grain map of an Armco iron sample, an iron grain and the grain boundary characters. Half the sample volume is removed to reveal inner grain (clusters). Faces of a selected grain color-coded (left to right) by IPF color, grain boundary normal direction in crystal reference system, misorientation to neighboring grains and grain boundary curvature.
Courtesy of Prof. Burton R. Patterson, University of Florida, USA.
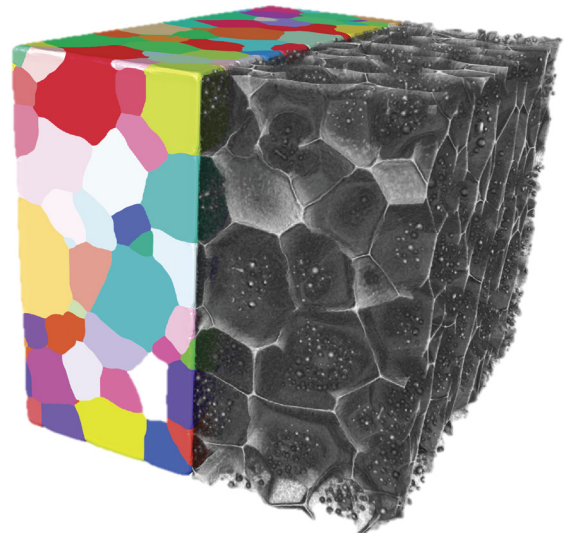

Figure 16 A multimodal imaging example: an Al-4% (wt) Cu sample imaged with both diffraction (left half) and absorption (right half) contrast tomography. Cu-rich grain boundaries and particles are revealed as a brighter phase due to the larger attenuation coefficient of Cu compared to Al.
Sample courtesy of Prof. Carl E. Krill III, Ulm University, Germany

Non-destructive 3D imaging modality

Materials in service experience diverse stimuli from the surrounding environment, under which the materials' internal structure may evolve and result in degraded performance. Even with the modern modeling approaches that can simulate many dynamic conditions that materials undergo, the variables in the actual, complex environment can readily exceed the predictions in the simulation. Experiments designed to investigate the structural evolution of materials with specific external stimuli are thus of vital importance to reveal the underlying mechanisms of material behavior.

As a non-destructive 3D imaging technique, LabDCT provides the accessibility for in-depth studies of, for example, temporal variations in crystallographic grain structure through 4D experiments. Direct interpretation is therefore possible as the grain structure evolution is followed correspondingly [5, 13].

Figure 17 presents the evolution of a cluster of ferrite iron grains during a designed annealing treatment [14, 15]. A grain growing in an abnormal manner is captured within the reconstructed 3D grain map at three annealing stages. The non-destructive nature of LabDCT allows a variety of subsequent processing to be carried out on the same sample.

Coupled with the information from complementary imaging modalities such as absorption contrast tomography, phenomena related to materials damage and deformation can be explored with comprehensive crystallographic information input [5, 7, 16, 17]. The 4D experimental datasets can then be used either as input or validation of models simulating dynamic materials process, or to accelerate the understanding and optimization of materials across a wide range of materials science and industrial applications.

Explicit grain structure for modeling

Continuum crystal plasticity modeling is a powerful tool to examine, interpret, as well as predict, the deformation behaviors of polycrystalline materials. Compared to synthetic grain structure, a full field experimentally acquired 3D grain map has an unprecedented advantage as it represents the inherent materials anisotropy, such as texture and abnormal grain size distribution that can be challenging to synthetize through simulation. LabDCT Pro, particularly now equipped with advanced acquisition schemes and support for faster acquisition using the high throughput large area flat panel detector, provides a routine solution for experimentally acquiring explicit 3D grain structures over more practical sample geometries, which can further be used either as input or validation of computational
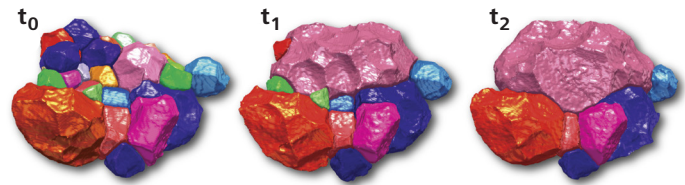

Figure 17 *Partial 3D grain map of an Armco iron sample imaged at various annealing steps. t0: initial state; t1: after annealing at 880 ºC for 8 hours; t2: after annealing at 880 ºC for 16 hours. By imaging the sample at three temporal states, the abnormal grain growth of the top, pink-colored grain is captured. Courtesy of Prof. Burton R. Patterson, University of Florida, USA.*
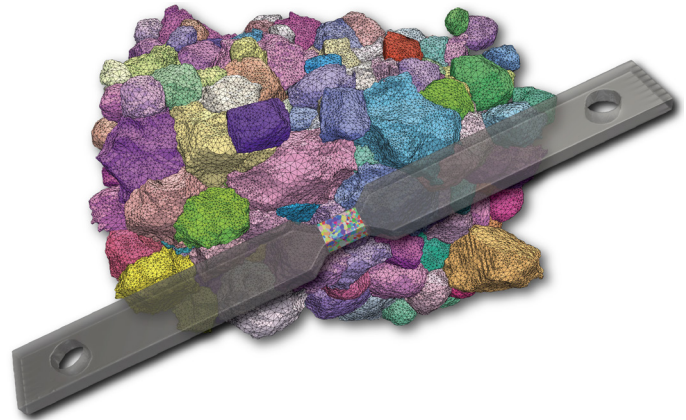

Figure 18 *An aluminum tensile specimen with its gauge length mapped out using ZEISS Xradia CrystalCT. A volumetric meshing of the 3D grain map is shown behind. The 3D grain map can be used as input for, e.g., continuum crystal plasticity modeling. Sample courtesy of Prof. Masakazu Kobayashi, Toyohashi University of Technology, Japan.*

results from modeling. Sample geometries that are routinely used for experimental *in situ* runs can be imaged in their native state (example shown in Figure 18), enabling a direct coupling of experimental results and simulations.

Summary

We have introduced the principles of diffraction contrast tomography and its application to determining crystalline grain structure in samples. This illustrates the continued progress of laboratory XRM to increase the diversity of imaging modalities that are inspired from synchrotron origins to solve problems in materials research and related fields.

The unique hardware architecture on the ZEISS Xradia Versa and CrystalCT platforms enables data acquisition in powerful combination with advanced reconstruction and analysis capabilities powered by Xnovo Technology and their experience in the field of DCT. LabDCT Pro supports use of a high resolution scintillator coupled detector or high throughput large area flat panel detector for acquiring diffraction data, bringing the flexibility to scan a wide variety of polycrystalline sample types catering to a broad range of applications. The continued use and applications development of this technique will accelerate the way 3D and 4D science is pursued for non-destructively studying polycrystalline materials.

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