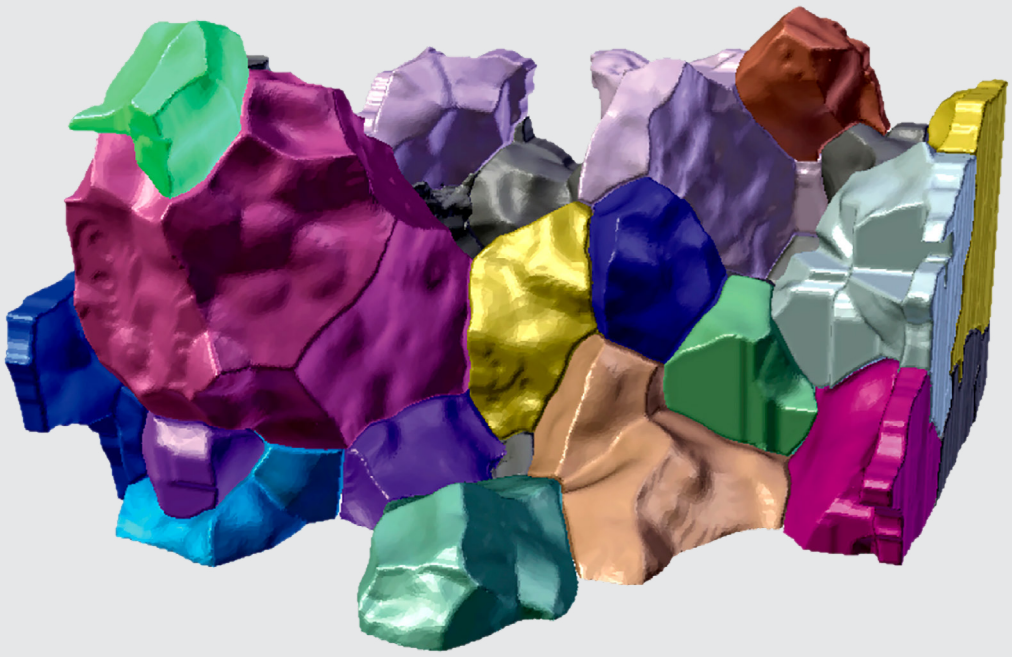
Technical Note

Diffraction Contrast Tomography

Unlocking Crystallographic Information from Laboratory X-ray Microscopy



ZEISS

Seeing beyond

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X-ray tomography has operated under two primary contrast mechanisms for 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, etc.) do not exhibit any significant contrast using absorption or phase mechanisms. Synchrotron-based XRM has demonstrated results in this area for about a decade with diffraction contrast tomography (DCT), which provides crystallographic/diffraction information from poly-crystalline samples, non-destructively, in 3D. Now, advancing laboratory X-ray microscopy (XRM) one step further, we describe here the capabilities of laboratory-based DCT on the ZEISS Xradia 620 and 630 Versa 3D X-ray microscope, and the new research and characterization capabilities this enables.

Introduction

In the continued spirit of transferring synchrotron capabilities to laboratory XRM systems, ZEISS in partnership with Xnovo Technology, has implemented the ability to obtain crystallographic (diffraction) contrast information on ZEISS Xradia 620 and 630 Versa [1,2].

Crystallographic imaging is commonly known from several metallographic techniques, including light and electron microscopy (EM) methods. 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.

However, the destructive nature of 3D EBSD (via examination of sequential slices) prevents one from directly evaluating the microstructure evolution when subject to either mechanical, thermal or other environmental conditions. Understanding this evolution process on the 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.

In response to this constraint, synchrotron-based crystallographic imaging, known as diffraction contrast tomography (DCT) has emerged over the past decade [3,4]. Utilizing non-destructive X-rays, synchrotron users could 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" evolution experiments. However, limited regular access to such synchrotron techniques has constrained the ability to perform thorough, longitudinal studies on materials evolution.

Cover Image: *LabDCT 3D imaging of grain orientations in aluminum alloy – IPF (inverse pole figure) colored.*

Image Courtesy of: *TU Denmark*

ZEISS has taken this established synchrotron modality and successfully transferred it to the ZEISS Xradia Versa family of laboratory-based sub-micron XRM systems. LabDCT may 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 [5]. Following an evolution experiment, the sample may be sent to the electron microscope or focused ion beam (FIB-SEM) for post-mortem complementary investigation of identified volumes of interest. This natural correlative workflow combining multiple lab-based methods on the same sample volume remains an attractive future direction of microscopy to many researchers and is enabled by the ZEISS foundation and breadth of imaging instrumentation.

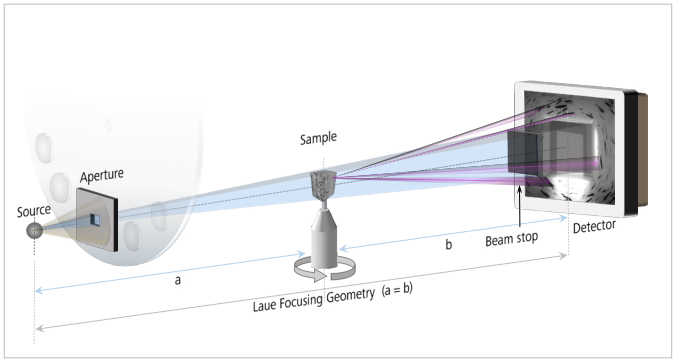

Figure 1 *Schematic of the LabDCT setup. The sample is illuminated through an aperture in front of the X-ray source. Both the sample absorption and diffraction information is recorded with a high resolution detection system. Optionally, a beamstop maybe be added to the set-up in order to increase the dynamic range of the diffraction detection.*

LabDCT: How it Works

Here, we present a method to acquire, reconstruct and analyze grain orientation and related information from polycrystalline samples on commercial laboratory X-ray microscopes (ZEISS Xradia 620 and 630 Versa) that utilizes a synchrotron-style detection system.

A schematic representation of the LabDCT implementation is shown in Figure 1. The X-ray beam is constrained through a specialized aperture to illuminate the sample. Diffraction information is collected with a tailor-made high resolution detector that in addition allows simultaneous acquisition of the sample's absorption information. In order to increase the sensitivity to the diffraction signal a beamstop may be added. The data acquisition is performed in a symmetric geometry, which enables improved diffraction signal strength and allows handling of many and closely located diffraction spots.

With LabDCT a series of projections is taken from which spatially resolved crystallographic information within the sample is derived. Figure 2 illustrates a single such 2D diffraction pattern as acquired with the LabDCT detector. Just a few hundred projections are enough for a complete LabDCT data set.

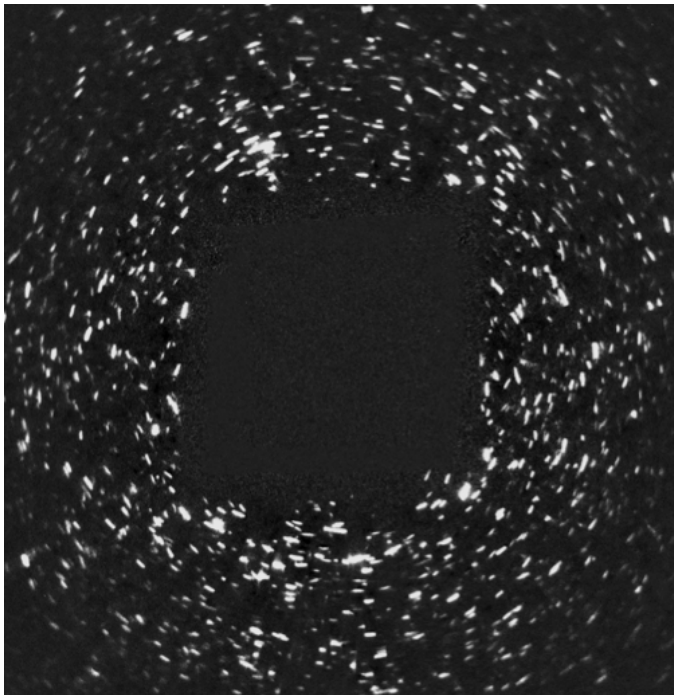

Figure 2 *Example 2D diffraction pattern from a Ti-alloy. The central area in the projection is obscured by a beamstop. This missing data does not impact the diffraction analysis and reconstruction.*

Validation and Application Examples

Polycrystalline Ti-alloy (Timet 21S) and Al-alloy samples have been explored and are used here to demonstrate the results of LabDCT. Successful 3D reconstruction of the diffraction information through Xnovo Technology GrainMapper3D™ software yields a completely voxelated crystallographic representation of the entire sample. The information recovered from a successful reconstruction enables extraction of details of the sample's microstructure, such as grains and their morphology, grain boundaries, grain sizes, grain boundary normals, statistics across the entire sample, pole figures for texture analysis, etc. A 3D grain map of a Ti-alloy sample is shown in Figure 3, where the grains are colored according to their crystallographic orientation in relation to the vertical sample axis (inverse pole figure). A virtual slice through the sample is shown in Figure 3b, whereas a measure for the quality of the LabDCT reconstruction is given to the user through a confidence map as seen in Figure 3c.

To help validate the accuracy of the LabDCT technique, comparisons to the closest related technologies, EBSD and synchrotron DCT and absorption X-ray microscopy were carried out. In doing so, independent measurements of sample microstructures were obtained. Figure 4 shows the comparison of a 3D grain structure derived from LabDCT data (Figure 4a), with the 3D grain structures derived from high resolution X-ray absorption tomography 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 4b 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 4d. Main contributing factors to these differences are the segregation of the Cu itself, leading to an uneven distribution and missing of some grain boundaries (compare Figure 4c); furthermore, the subsequent segmentation of the grain boundary network from the absorption tomography reconstruction will introduce uncertainties in the location of the boundaries.

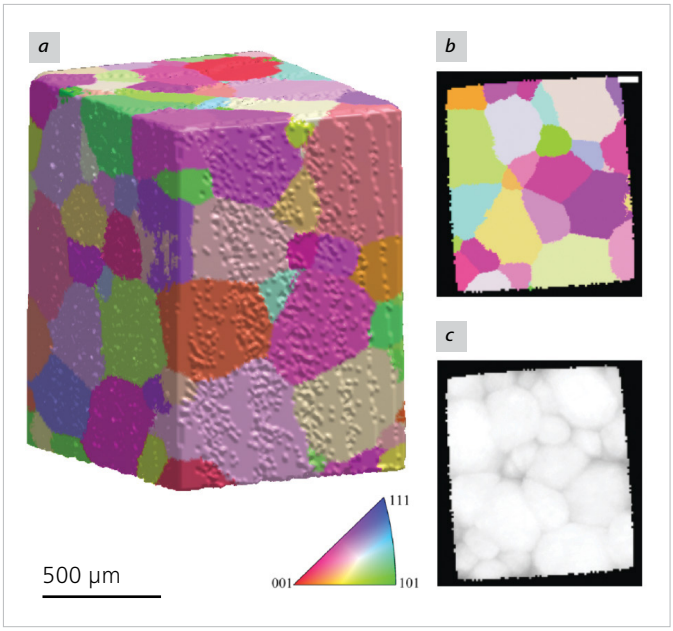

Figure 3 *a) Visualization of a 3D grainmap of a titanium alloy (Timet 21S) from reconstruction with the LabDCT GrainMapper3D software. Inverse pole figure color coding highlights the crystallographic information. b) Virtual cross section through the 3D grainmap; scale bar: 100 µm. c) Confidence map of the reconstruction corresponding to the virtual cross section in b); the gray scale reflects the confidence value of each voxel from 0% (black) to 100% (white) confidence.*

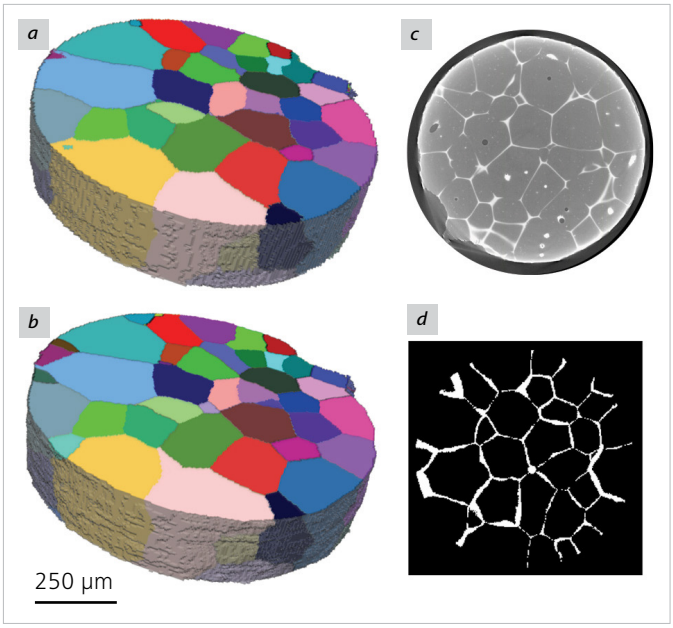

Figure 4 *a) LabDCT reconstruction of Al-Cu sample. b) Volumetric segmentation from absorption tomography. Colors in a) and b) indicate a grain index. c) Virtual slice through the absorption tomography in gray-scale. d) Virtual slice of the difference map between the data of a) and b); the binarized color scale indicates regions of differences in white and regions with no difference in black.*

Also the angular accuracy of the crystallographic orientations were compared to those obtained using synchrotron DCT and EBSD. Some of the measurements are shown in Figure 5a along with the corresponding misorientations determined from the Lab-DCT measurement. The absolute values of the misorientation obtained by the two techniques matches well within approximately one degree. Figure 5b shows a histogram of the difference in misorientation measured with EBSD versus LabDCT as well as the difference in misorientations as measured by LabDCT versus synchrotron DCT. The DCT techniques – both being X-ray based – differ in crystallographic orientation by less than 0.3 degrees, whereas the differences between LabDCT and EBSD approach one degree consistent with the lower angular resolution of the EBSD technique [6].

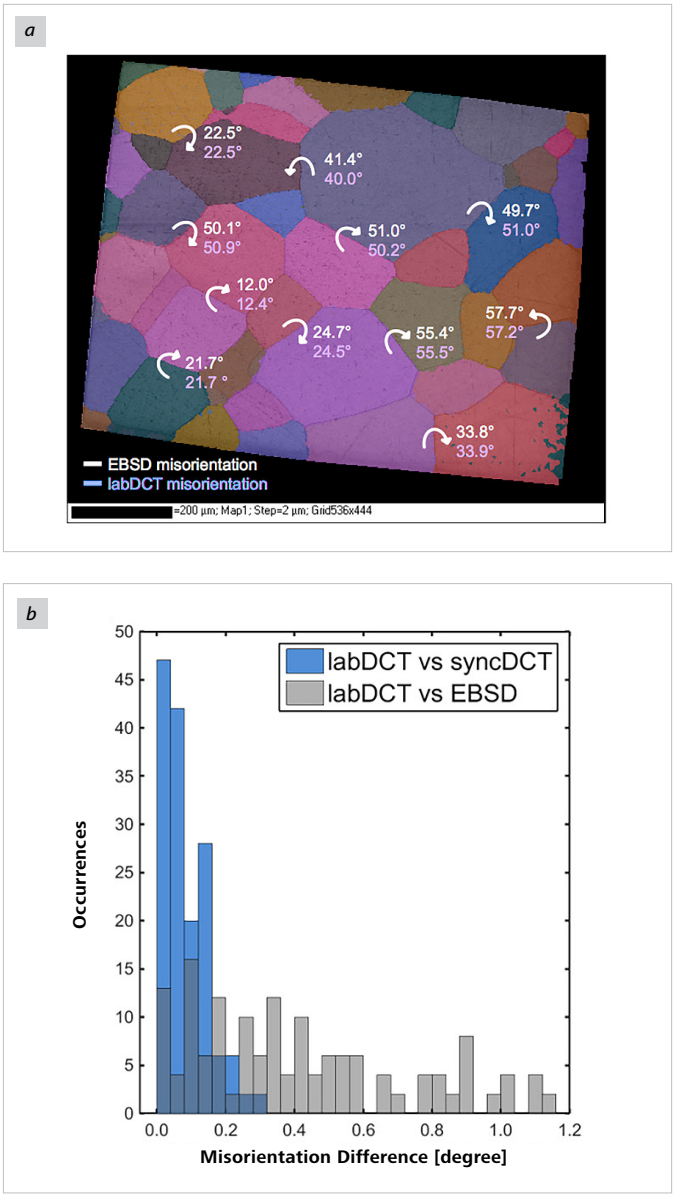

Figure 5 *a) EBSD data from Ti-alloy sample with indications of misorientation angles as determined by EBSD and LabDCT. b) Histogram of the differences in misorientation as determined by LabDCT vs EBSD, and by LabDCT vs synchrotron DCT.*

Conclusion

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 ZEISS Xradia 620 Versa and 630 XRM hardware architecture enables data acquisition in powerful combination with advanced reconstruction and analysis capabilities powered by Xnovo Technology and their experience in the field of DCT. 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.

References:

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