

Application Note

Lab-based DCT validated against synchrotron grain mapping

Grain mapping at synchrotrons

Grain mapping is a common name for a variety of non-destructive tomographic techniques originating from synchrotron x-ray facilities to access the grain-based 3D polycrystalline microstructure of bulk samples. Grain mapping comes in several variations, such as 3D x-ray diffraction (3DXRD) microscopy [1], diffraction contrast tomography (DCT) [2], and high-energy diffraction microscopy (HEDM) [3]. These techniques are available at a very limited number of synchrotron beamlines worldwide.

Lab-based DCT

Inspired by the developments at leading synchrotrons and motivated to enable access to the broader research community, the lab-based DCT variants LabDCT Pro™ and CrystalCT™ were introduced as the very first commercial solutions providing non-destructive crystallographic imaging on conventional Zeiss Xradia x-ray microscopes in the laboratory [4].

Lab-based versus synchrotron DCT

Fig. 1 shows the comparative results of lab-based and synchrotron DCT on a coarse-grained polycrystalline titanium alloy (Timet 21S) sample with an average grain size of 120 μm . Good correspondence is found between the synchrotron and lab-based DCT mappings of the titanium alloy for both grain orientation and morphology accuracy. Analyzing 93 matched grains gives mean grain pair misorientation of 0.031° and mean grain boundary distance of 7.1 μm (less than 2 voxels).

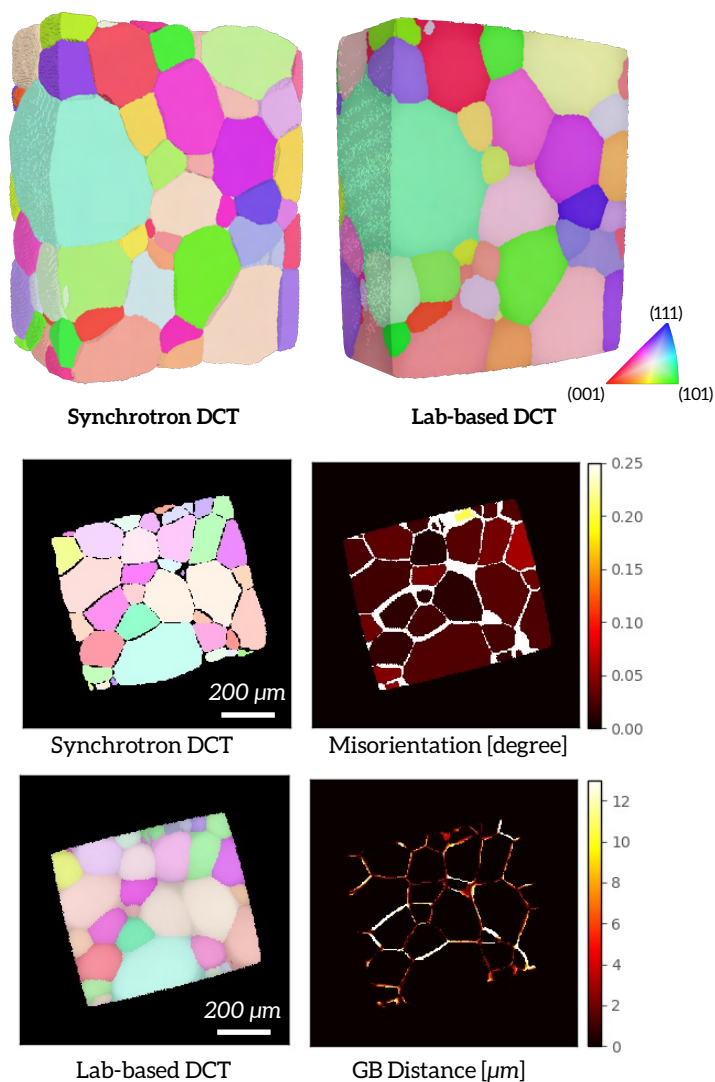


Fig. 1 Titanium alloy. Top: Reconstructed 3D grain maps from synchrotron and lab-based DCT with grains colored according to crystallographic orientation (inverse pole figure). Bottom: Virtual cross sections through the same grain maps and point-to-point misorientations (scale max 0.25°) and grain boundary distances (scale max 13 μm) between the two.

Lab-based DCT versus synchrotron HEDM

Synchrotron HEDM in the near-field variant for mapping grain morphologies and crystallographic orientations uses a planar-focused beam with a height of a few microns. Large microstructure maps are then assembled from a series of sequentially collected layers. This approach provides improved confidence in the spatial resolution of the indexed grains, particularly in the direction normal to the layer, but significantly increases the scan time and hence limits the accessible sample volume.

As opposed to this, both synchrotron and lab-based DCT employs a beam height of the order 0.1–1 mm, giving access to larger sample volumes and superior grain statistics. In addition, the advanced acquisition schemes for lab-based DCT circumvent the layer stitching step, which may still apply for very large sample volumes in synchrotron DCT experiments.

Fig. 2 shows the comparative results of synchrotron high energy diffraction microscopy (HEDM) and lab-based DCT of a fine-grained polycrystalline SrTiO₃ ceramic sample with an average grain size of 25 μm.

A very good correspondence is found between the synchrotron HEDM and lab-based DCT mappings of the SrTiO₃ sample in terms of both grain orientation and morphology accuracy. Because of the better spatial resolution, synchrotron HEDM does detect grains smaller than the 10 μm grain detection limit of lab-based DCT. Analyzing around 1400 matched grains gives mean grain pair misorientation of 0.028° and mean grain boundary distance of 2.7 μm (less than 2 voxels).

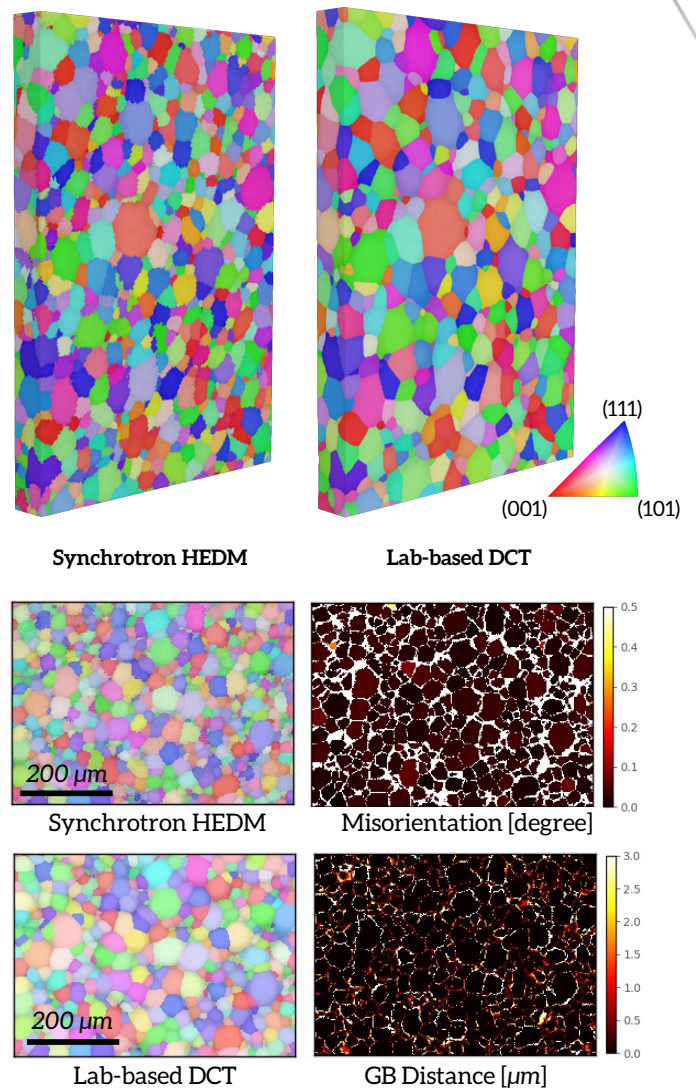


Fig. 2 SrTiO₃ ceramic. Top: Reconstructed 3D grain maps from synchrotron HEDM and lab-based DCT with grains colored according to crystallographic orientation (inverse pole figure). Bottom: Virtual cross sections through the same grain maps and point-to-point misorientations (scale max 0.5°) and grain boundary distances (scale max 3 μm) between the two. SrTiO₃ sample and synchrotron HEDM courtesy of Amanda Krause, University of Florida, USA

References:

- [1] H. F. Poulsen et al., *J. Appl. Cryst.* 34, 751 (2001)
- [2] W. Ludwig et al., *J. Appl. Cryst.* 41, 302 (2009)
- [3] S. F. Li & R. M. Suter, *J. Appl. Cryst.* 46, 512 (2013)
- [4] S. A. McDonald et al., *Sci. Rep.* 5, 14665 (2015)

