doi:10.1017/S1431927618014964
Microsc. Microanal. 24 (Suppl 2), 2018

Quantitative non-destructive 3D Crystallographic Imaging of Microstructures using Laboratory X-ray Diffraction Contrast Tomography

Hrishikesh Bale[1*], Ron Kienan[2], Stephen T Kelly[1], Nicolas Gueninchault[3], Erik Lauridsen[3], Ashwin Shahani[2]

[1.] Carl Zeiss Microscopy, Pleasanton, CA, USA.
[2.] Dept. of Materials Science and Engineering, University of Michigan, Ann Arbor, MI, USA
[3.] Xnovo Technology ApS, Køge, Denmark
[*] Corresponding author, *hrishikesh.bale@zeiss.com*

Knowledge of the grain structure and crystallographic orientation distribution within polycrystalline samples is critical to the understanding of the mechanical and electronic properties of a material. Scanning Electron Microscopy (SEM) with Electron Backscattered Diffraction (EBSD), reveals the crystallographic microstructure of a material in great detail, but is limited to only 2D surface information. Extending the EBSD capabilities to 3D requires destructively milling the sample surface with a focused ion beam (FIB). A few synchrotron beamlines began expanding the non-destructive capabilities of x-ray tomography beyond just the conventional absorption/transmission contrast by utilizing diffraction signals [1,2]. Since then, the diffraction contrast tomography (DCT) technique has gained considerable traction over the years as a complementary method to EBSD due to its ability to uniquely obtain bulk, 3D grain structures in a non-destructive fashion, opening the door for large-scale grain structure analysis or time-dependent evolutionary studies. Due to the exclusive presence of DCT at the synchrotron, however, its accessibility to the wider research community has been inherently limited.

In this work, a lab-based adaptation is presented, termed lab diffraction contrast tomography (LabDCT), the technique operates within a lab-based X-ray microscope with a polychromatic divergent beam (as opposed to the typically collimated, monochromatic beam at synchrotron beamlines) [3,4]. Mounting a sample in a Laue focusing condition, individual grains produce diffraction spots on a specialized high resolution detector. The polychromatic beam provides a unique advantage wherein a majority of the grains within a sample simultaneously satisfy the Bragg condition due to the wide spectrum of wavelengths, yielding crystallographic information including grain orientation, location of center of mass, and morphology for a large number of grains within the sample. This information can be utilized to complement microstructural features such as voids, inclusions, or secondary non-crystalline phases that are observed in traditional absorption-based tomography.

We present here exemplary results from integrated imaging of polycrystalline silicon in three dimensions using X-rays which provides new insights on grain boundaries, particles, and their correlations in polycrystalline silicon [5]. Polycrystalline silicon (poly-Si) is an important material for photovoltaic device fabrication, and the efficiency of poly-Si photovoltaic devices is critically dependent on the nature of the grain boundaries and foreign metal impurities in the bulk. The process of electron-hole recombination is known to occur at grain boundaries and precipitates which are characteristically present in poly-Si and hence strongly impact the performance of devices made from poly-Si. For instance, less than one parts-per-billion concentrations of iron can drastically reduce the minority carrier diffusion length in poly-Si [5]. Here, we probe the characteristics and distributions of these defects in three dimensions by using an integrated, non-destructive imaging approach, combining both absorption and DCT imaging on a laboratory X-ray microscope. Absorption contrast tomography (ACT) data

resolves the high-density impurities and precipitates, while diffraction contrast data reveals underlying grain boundaries and orientations. Utilizing the complete morphological information of grains revealed by LabDCT, a five-parameter grain boundary analysis was performed and juxtaposed with the information of the spatial location of impurities from ACT. Using this unified analysis approach, we determined that the location of the impurity particles is non-random in the bulk and strongly dependent on grain boundary character, leading to their predominant presence along grain boundaries. Among the grain boundaries identified, Σ9 {221} boundaries feature a higher density of the impurities compared to Σ3 {111}. The dependence of precipitate decoration on coincident site lattice type may be due to energetic factors, e.g., disordered atomic structure along Σ9, or kinetic factors, e.g., faster impurity diffusion in the core of Σ9. The correlative workflow developed in this work bridges the gap between different imaging modalities, thereby providing a more unified description of the microstructural landscape and the potential for non-destructive materials diagnostics directly from the laboratory.

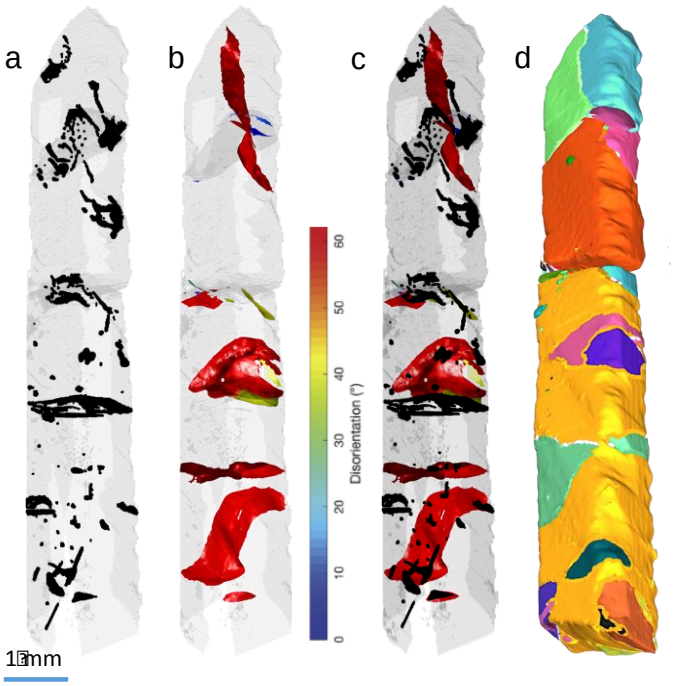

Figure 1. Multimodal imaging in three dimensions: (a) ACT reconstruction showing dense metal impurities (black) in the bulk; (b) grain boundaries retrieved from LabDCT colored according to disorientation angle; (c) ACT and DCT datasets registered. (d) Surface rendering of grains from LabDCT reconstruction showing multiple grains (colored randomly) along the rod sample.

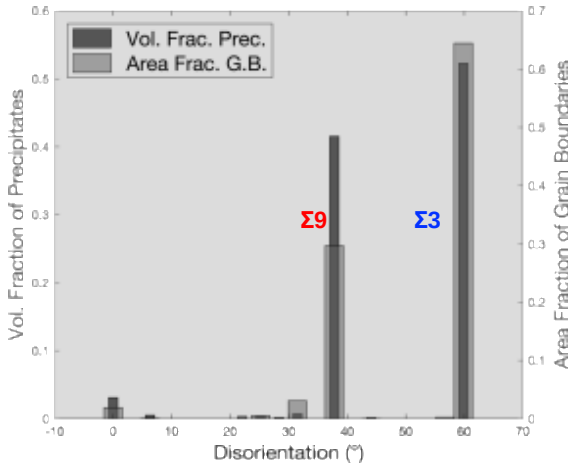

Figure 2. Particle-associated disorientation distribution (i.e., volume fraction of particles located on grain boundaries) and grain disorientation distribution. A higher fraction of impurities per unit area are located on boundaries with disorientation angle of 39° vs. 60°.

[1] U Lienert et al., Journal of the Minerals, Metals & Materials Society, **63** (2011), p. 70.
[2] D Juul Jensen and H.F. Poulsen, Materials Characterization, **72** (2012), p. 1.
[3] C Holzner, *et al*, Microscopy Today, **24** (2016), p. 34.
[4] SA McDonald *et al*, Scientific Reports, **7** (2017), p. 5251.
[5] R Keinan *et al*, Acta Materialia, 148, (2018), p. 255.

https://doi.org/10.1017/S1431927618014964 Published online by Cambridge University Press