

## Integrated Imaging in Three Dimensions: The Sum is Greater than the Parts

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**Summary:** Recent developments in laboratory-based diffraction contrast tomography (LabDCT) have shown its capability to non-destructively map the 3D morphology and crystallographic orientation in the bulk of a polycrystalline sample. Using a combination of LabDCT and attenuation-based tomography, we present here the first experimental results from the imaging of a polycrystalline silicon sample and demonstrate the application of this integrated approach in obtaining crucial microstructural and grain related crystallographic information.

### 1. INTRODUCTION

Imaging combined with diffraction, or other analytical methods, allow one to observe and understand a material in all its complexity. For instance, in recent years, X-ray tomography has enabled the visualization of the local behaviour of working devices (e.g., batteries and fuel cells) [1]. However, as powerful as this approach can be, usually no single measurement is sufficient to fully characterize a material. Integrated imaging in three dimensions (3D) offers the possibility to bridge the gap between different characterization modalities.

In our talk, we will present recent efforts on integrating results from attenuation and diffraction contrast X-ray tomography experiments in order to provide a unified description of 3D microstructures. The attenuation-based experiments (denoted ACT) enable the identification of different phases in the volume based on their density differences, e.g., pores and inclusions; meanwhile, the laboratory diffraction contrast tomography (LabDCT) experiments allow one to reconstruct the intrinsic grain structure based on the crystallography [2]. Thus, using these two complementary sources of data, we will be uniquely positioned to correlate the distributions of secondary phases (via ACT) with the underlying crystallography of the host material (via LabDCT), in 3D. Due to the non-destructive nature of both experiments, it is possible to further characterize the same specimens at the highest possible resolution by combining different modalities through a correlative approach, e.g., transmission electron microscopy (TEM), energy dispersive X-ray microanalysis (EDX), and atom probe tomography (APT). In this manner, new insights can be obtained by exploring over six orders of magnitude in length-scale!

To demonstrate our fully integrated approach, we have analysed the 3D microstructure of polycrystalline Si (poly-Si), including the distributions of inclusions and grain boundaries within the bulk material. Poly-Si is widely used as the substrate for thin-film photovoltaic (PV) cells. To be commercially relevant, the efficiency of poly-Si cells should reach 12% offered by other thin-film PV devices. However, electron-hole recombination at grain boundaries and inclusions impacts strongly the performance of these devices [3]. Thus, a fundamental understanding of the structure of poly-Si will guide the development of future polycrystalline PV technologies.

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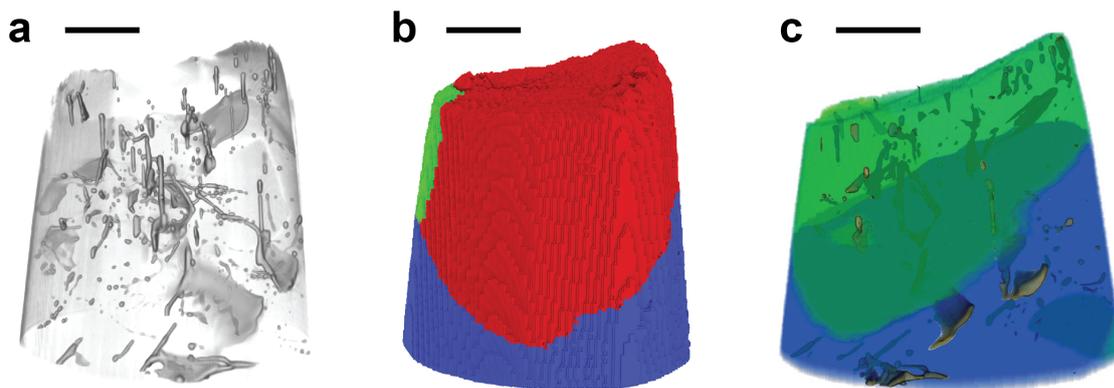
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## 2. EXPERIMENTAL METHOD

Bulk pieces of commercial-grade poly-Si were obtained from American Elements (Los Angeles, CA, USA). The poly-Si was produced by the manufacturer through a modified Siemens process. Smaller 1.5 mm diameter pillars were laser machined from the bulk specimen and imaged in attenuation and diffraction contrast mode at a 1.5  $\mu\text{m}$  resolution on a laboratory X-ray microscope (ZEISS Xradia 520 Versa). The X-ray projection data was reconstructed using the microscope's built-in filtered back projection algorithm and the diffraction dataset was processed using a separate 3D grain reconstruction software (GrainMapper3D, Xnovo Technology ApS, Denmark). The grain reconstruction data contains information related to the crystal orientation, grain volume and shape which were further analysed using the MATLAB toolbox MTEX. Based on the 3D orientation information, we were able to not only reconstruct the shapes of the individual grains, but also calculate the five-parameter grain boundary character distribution (GBCD) [4] in poly-Si. These five parameters include the grain misorientation (i.e., three degrees of freedom) as well as the grain boundary inclination (i.e., two degrees of freedom). Importantly, the measured grain boundary populations are inversely correlated to the grain boundary energies that are available in the literature. To cross-check the DCT results, the same poly-Si specimen was later sectioned and analysed via EBSD.

## 3. RESULTS

Preliminary results are given in Fig. 1. The ACT reconstruction in (a) indicates, perhaps surprisingly, that the monolithic Si sample possesses a high degree of heterogeneity. Specifically, the presence of a high density of inclusions and cracks can be observed. Separately, the LabDCT results in (b) reveal the underlying grain structure of poly-Si. The red and blue grains pictured have a  $\Sigma 3$  (twin) coincident site lattice (CSL) relationship. These ACT and LabDCT results were then superimposed on each other (c), so as to identify the interaction between the inclusions and the grain boundaries. Interestingly, the defects appear to lie both *along* the grain boundaries and *through* the grains themselves. Further characterizations are well underway to shed light on the mechanisms underpinning the intergranular and transgranular segregation of these defects in poly-Si. It is anticipated that our integrated approach can be extended to other complex microstructures with minimal sample-specific tuning.



**Figure 1:** (a) ACT reconstruction of inclusions and cracks within poly-Si sample; (b) LabDCT grain map showing three grains (red, blue, and green); and (c) superposition of ACT and LabDCT results. Scale bar is 250  $\mu\text{m}$ .

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