2312
doi:10.1017/S1431927622008881

Microsc. Microanal. 28 (Suppl 1), 2022

Robust Measurements of Functional Material Properties using *in situ* 4D-STEM

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Conventional scanning transmission electron microscopy (STEM) imaging experiments record only a few values per probe position, generating bright field or dark field images, consisting of unscattered or scattered electrons respectively. The contrast in these images is highly sensitive to changes in local structure and the resolution is sufficient to resolve individual atoms. Unfortunately, many real-world samples in materials science studies are too thick or too complex to easily measure the structural properties of interest in this manner. An alternative to conventional STEM imaging is to use a high-speed direct electron detector, which records a full image (2D data) of the diffracted electron probe scanned over the sample (2D grid of positions), producing a four-dimensional measurement known as 4D-STEM [1]. These 4D-STEM measurements of millions of diffraction patterns are extremely rich in information, but require efficient and robust software in order to measure sample properties. We have developed the py4DSTEM open source analysis toolkit to perform these analyses [2].

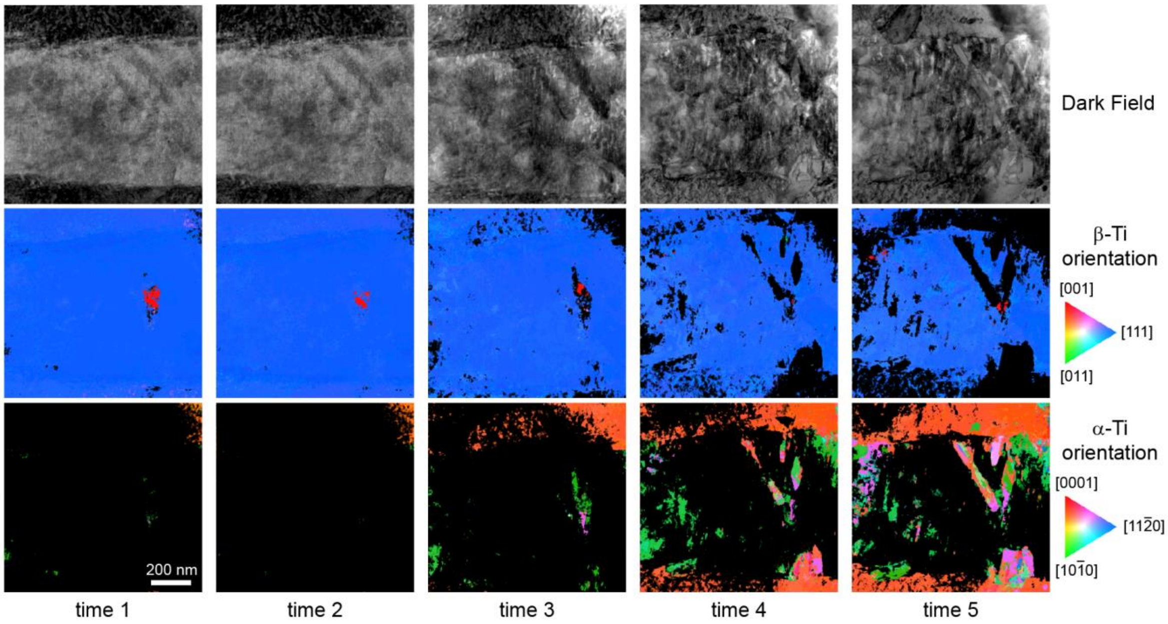

Figure 1. An *in situ* 4D-STEM experiment where a titanium alloy sample is annealed at 1050°C. (top row) A series of dark field images generated by inverting the contrast of a virtual bright field image. (center row) Zone axis orientation maps of cubic β-Ti, and (bottom row) orientation maps of α-Ti. Analyzed using py4DSTEM, where masks are generated from the matching score of the cubic phase [3].

A common *in situ* electron microscopy experiment is sample annealing, which can be performed using a heating stage. Figure 1 shows a 4D-STEM experiment where a heating stage was used to anneal a titanium alloy sample. Initially, the sample consists primarily of a large β-Ti grain aligned close to a [111] zone axis. Due to previous mechanical processing, this grain contains many defects which serve as nucleation sites for secondary α-Ti precipitates. Over time, these precipitates grow with a few well-defined orientations relative to the parent β-Ti grain. This automated crystal orientation mapping (ACOM) is performed using an updated module of py4DSTEM [3]. In this talk, we will describe both the experimental setup and analysis method for ACOM experiments.

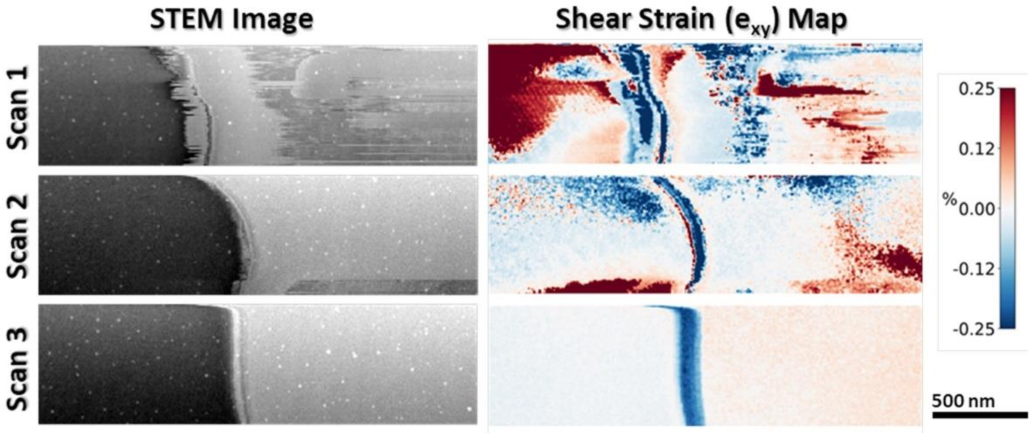

Figure 2. An *in situ* 4D-STEM scan of a domain wall in a boracite sample. (left) Simultaneous dark field image, and (right) shear strain measured using 4D-STEM [2].

Another example 4D-STEM *in situ* experiment is shown in Figure 2. In this experiment, we image the strain field around a domain wall present in an improper ferroelectric boracite sample. Charging induced by the beam produces motion of the domain wall, which is highly dependent on the scan direction and speed. Initially, the measured strain maps are extremely chaotic, as the boundary is reconfiguring during the scan. However, with careful positioning of the electron beam, we can quantitatively measure the strain fields around quasi-stationary domain walls. Figure 2 shows both the domain wall strain profile, and the slightly different bulk strains of the two sides of the domain wall induced by their different polarizations. For these experiments, we use patterned bullseye STEM probes to improve the accuracy of the measured strains by removing systematic errors in the diffraction patterns induced by multiple scattering or mistilt of the sample [5]. In this talk we will describe these and other in situ experiments in detail, as well as the calibration and analysis steps required for accurate property measurements [6].

References:

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[6] Work at the Molecular Foundry was supported by the Office of Science, Office of Basic Energy Sciences, of the U.S. Department of Energy under Contract No. DEAC02-05CH11231.