ESRF	**Experiment title:** Full field Bragg coherent diffraction imaging of large poly-crystal grains	**Experiment number**: MA 5371
Beamline: ID01	**Date of experiment**: from: Feb 2, 2023 to: Feb 7, 2023	**Date of report**: Sept 9, 2023
Shifts: 15	**Local contact(s)**: Steven Leake	*Received at ESRF:*

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Report: This experiment aimed to demonstrate that the enhanced brightness of the EBS-ESRF source can be leveraged for expanding the reach and applicability of Bragg coherent diffraction imaging (BCDI) towards 3D strain-sensitive images of individual grains within bulk polycrystals. Such a capability will open the possibility of coupling BCDI with x-ray diffraction microscopy methods (e.g. DCT, HEDM) so that lattice structural information of polycrystals can be characterized from millimeters down to the few-tens-of-nanometer scale that reveal sub-grain strain fields. Three major challenges towards this goal were addressed in this beam time:

1) Utilizing an x-ray energy of 33.4 keV (higher than the typical energies used for BCDI) results in the compression of coherent diffraction patterns that requires adaptation of typical measurement strategies in order to fulfill the requirements of phase retrieval. Our team made measurements of an 0.5 micron sized nanocrystal from a dewetted Au film under conditions that allow the testing of one particular strategy of fringe recovery from undersampled Bragg diffraction patterns. A horizontal diffraction geometry was adopted to enable a 111 Bragg reflection from Au to reach the Eiger detector on its long-translation stage. This allowed a rocking curve measurement of a typical nanoparticle to be conducted at a sample-to-detector distance of 6 m such that BCDI sampling conditions were satisfied to serve as a control measurement. A measurement of the same nanoparticle was performed at a detector distance of 3 m, where sampling conditions were just underfulfilled. At each angle of the rocking curve, a raster scan was performed using the translation stages beneath the Eiger such that multiple exposures of the BCDI pattern were recorded by translating the detector translation in horizontal and vertical sub-pixel steps. Initial analysis has indicated that the 6 m BCDI scan is suitable for phase retrieval. We have also performed simulations and data analysis based on the work of [1] to enable upsampling from a sub-pixel raster grid. These fringe recovery scripts are now running and have been used to generate preliminary upsampled images from the data collected at this beamtime, which will be refined and used for phase retrieval in the near future. An important finding was that we have discovered that though 1/6 pixel size steps (12.5 microns) were intended, the vertical motor stage regularly under-translated the detector, while the horizontal translation was accurate. Data points among those available were still suitable for upsampling, but this indicates that we need encoders for future measurements, and this issue motivates

work to design a more general upsampling algorithm that can handle arbitrary in-plane detector shifts, which we will pursue in the future. Figs 1,2 summarize the findings and progress towards goal 1.

2) A second goal of this work was to demonstrate that high-energy (HE)-BCDI at a fourth generation synchrotron is viable for 3D imaging of crystallites in the size range of 3-5 microns, much larger than the typical 0.5 micron crystal size for BCDI. This capability would bridge the length scale gap between HEDM/DCT which typically have spatial resolutions of ~1-2 microns and allow interrogation of grains of interest identified with HEDM/DCT to be interrogated with sub-grain resolution with HE-BCDI. Larger crystallite sizes, as with high x-ray energies, compress the angular fringe spacing of the coherent diffraction patterns needed for imaging, again necessitating strategies for upsampling. During the beamtime, we measured a 3-micron-diameter Au crystallite from the dewetted film. With a 6 meter sample to detector distance in the horizonatl plane, fringes were still undersampled with the Eiger detector. The sub-pixel translation raster scan described above was performed on the Bragg peak from this crystal. The fringe pattern and peak splitting is pronounced (as shown in Fig 2) and upsampling will be performed once a successful strategy is charted with the control data.

3) A third goal we made significant progress towards was to demonstrate that sub-grain structural detail can be attained at multiple Bragg reflections using the focused beam nanodiffraction of a specific grain within a bulk crystal for which grain locations and orientations were determined by high energy diffraction microscopy. A Ti-6Al-4V aerospace alloy sample with a 1 mm X 1 mm cross section for which the lattice orientations of thousands of grains were tabulated from previous experiments at APS was measured at this beamtime. A specific grain within this bulk was successfully located and oriented for Bragg diffraction at the 0002, 10-11, and 10-12 Bragg reflections. At each reflection, a 1-micron focused beam was rastered to collect nanodiffraction data suitable for analysis with STXM methods or Bragg ptychography. In this measurement, undersampling was not an issue so Bragg peaks could be measured with the Maxipix detector on the general-purpose diffractometer detector arm. The major achievement in this measurement was establishing the methodology for measuring multiple peaks from a targeted grain.

Together, these three sets of measurements we performed during the beamtime go a long way to establishing the framework and methodology of the integration of nanoscale structural x-ray microscopy methods (BCDI/scanning nanodiffraction) with structural microscopy methods that characterize tremendous numbers of individual grains within a bulk down to the 1-2 micron scale. Such an integrated multi-scale approach is needed to validate models of elastic and plastic deformation and to discover the local mechanisms that govern macroscale properties via in-situ measurements.

[1] Maddali, et al., "Sparse recovery of undersampled intensity patterns for coherent diffraction imaging at high x-ray energies." Scientific Reports 8, 4959 (2018).

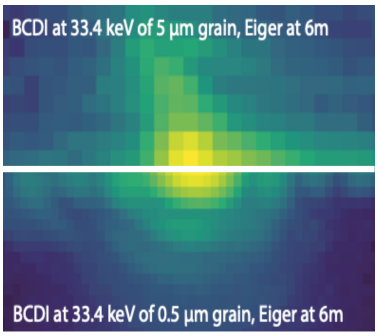

Fig 1: Results from ID01 Feb 2023 showing BCDI signal from a 0.5 μm Au crystal measured at 3 m and 6 m. Detector translation scans were done at the 3 m detector distance for upsampling. Detector translations of 1/6 a pixel (12.5 μm) were performed.

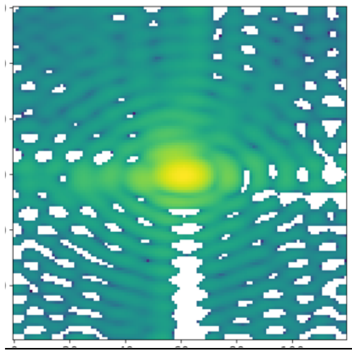

Fig 2: Upsampled diffraction pattern from an 0.5 Au particle from the detector translation data set measured at 3 m. This output pattern has an emulated pixel pitch of 25 μm compared to the native 75 μm pitch of the Eiger on which the data were collected. Upsampling all data as a funciton of rocking cureve will allow 3D phase retrieval.

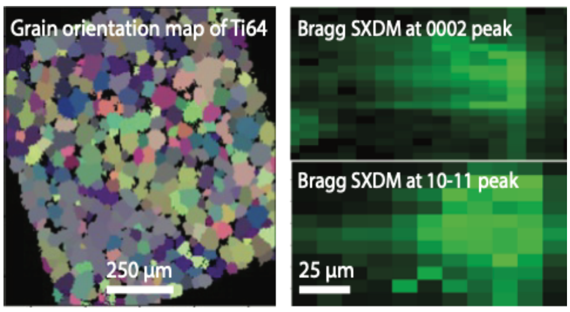

Fig 3: (left) HEDM spatial/orientation map of grains within a cross section of the Ti-6Al-4V sample. (right) A single grain from this population was measured with scanning x-ray diffraction at the 0002 and 10-11 reflections. A map at 10-12 was also measured, not shown. Maps show integrated peak intensity at fixed incident angles of the respective Bragg peaks.