ESRF	**Experiment title:** Linking 3DXRD to High Resolution EBSD: Application to commercially pure titanium	**Experiment number**: MA 2492
Beamline: ID11	**Date of experiment**: from: 1 April 2015 to: 7 April 2015	**Date of report**: 24/02/2017
Shifts: 18	**Local contact(s)**: Jonathan Wright	*Received at ESRF:*

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Report

High resolution electron backscatter diffraction (HR-EBSD) is a novel technique that has been recently develooped at the University of Oxford [1]. With HR-EBSD it is possible to measure orientation, “relaive” elastic strain, “relative” lattice rotation, and the geometircaaly necessary disloacation (GND) density in various grains of a polycrystaline material. Similar to many other diffraction based experimental techniques, measuremenet of elastic strain and rotation require a refrence point which ideally should be stress-free. Since HR-EBSD is based on cross-corrolating diffraction patterns using imaging techniques, the state of the refrence point is un-known, and hence, all of the stresses measured with this technique have so far been “relative”. That is, for each grain a refrence point is selcted where stress, strain, and rotations are reported with respect to this point. In order to solve the refrence pattern problem, in parallel to two other techniques that have been examined at Oxford, 3DXRD are used to determine the state of the refrence point in each grain and compare it to the other techniques. The final goals of this research are (a) to measure state of deformation in various grains of Hexagonal Close-Packed (HCP) polycyrtsline materials with the use of both HR-EBSD and 3DXRD to solve the refrence pattern problem (b) to produce data sets from the same measured volume for validating a crystal plasticity finite elememt code (CPFE) developed by the PI, and (c) by linking CPFE to HR-EBSD and 3DXRD, to develop a technque for characterizing dislocation densities in measured grains.

Experiment

Four samples were brough to the ID11 for running 3DXRD, and DCT experiments: commercially pure titanium (CPTi) with grain size of 100 μm and 300 μm, commercially pure zirconium (CPZr), and a mini-fatigued titanium sample. The first two days of the beamtime were spent on the deformation of the CPTi with smaller grain size (CPTi-100). For this sample, DCT was used to measure grain shape and positions in 1.8 mm of the sample gague. The sample was then mounted on the ID-11 tensile rig for 3DXRD measurment. X-ray beam energy of 40keV was used to measure stress fileds of the grains that were measured with DCT techqnique. Measurements were conducted at the preload, onset of plastoicity, applied strain of 1% and enevtually at the unload. For each measurmenet step, 18 layers of sample were probed by X-ray beam where for each layer, sample were rotated from -234 to -125 and also from -54.4 to 54.5 at the rotation step of 0.25 degress and exposure time of 0.25 seconds.

Two other days of the beamtime were dedicated for measruing deformation in grains of the CPZr sample. For this sample no DCT scan was done as grains were too small for DCT analysis. Instead, 3DXRD were pefromed at 78 keV with measurements at the preload, onset of plasticity, applied strain of 1.2% and unload. More than 1mm of the sample gauge were probed by scanning 15 layers of grains. For each layer, sample was rotated from -234 to -125 and from -54 to 54 at the rotation step size and exposure time of 0.25.

One day of the beamtime was dedicated to measure stress fields close to a cracked mini-fatigued Ti sample. Unlike the rest of the samples, this experiment was done ex-situ as the sample was already fatigued and deformed. State of deformation in the grains close to the cracked and remote areas were studied by probing layers close to the fatigue crack and in the arm of the sample.

The last day of the beamtime was dedictaed to measure stress fields in the CPTi sample with grain size of 300 μm (CPTi-300). For this sample, DCT was conducted before any load was applied. Then the sample was mounted on the tensile rig for 3DXRD experiments. Measurements were conducted at the preload, onset of plasticity, applied strain of 1%, applied strain of 2%, and unload. The test setup was similar to CPTi-100 except for the rotation step which was set to be 1 degree.

PostPtrocessing data

The post-processing of the measured data was mainly done by the use of ImageD11 and the subroutines embedded into Fable (https://sourceforge.net/p/fable/wiki/Home/). After calculating the background and reducing it from diffraction patterns, peak search was done by applying a set of thresholds of 100, 200, 400, 800, 1600, 3200, 6400, 12800 to determine the position of the measured peaks. The peak search was performed on the first 12 rings- the un-complete rings close to the edge of the detector were cut-off. Once diffraction peaks were identified, they were indexed and assigned to grains. This was done by cross correlating peaks from different rings using the hkl tolerance of 0.01. The criteria for determining a grain was to find at least 60 peaks that could be assigned to each. The indexed grains were used to refine parameters to find more grains and index more peaks. It is worth mentioning that the global parameters were tuned by the use of Ceria, i.e. parameters such as detector tilts, ceneter of the detector, and sample to detector distance. Grains with more peaks were used to refine crystal parameters. For final tuning of the parameters, the grain maps measured at the preload

were fed into FitAllB program [2]. Once the final parameters were identified, they were used to re-index grains from preload and to find grains and their state of deformation at the onset of plasticity, and the two other steps.

Matching grains

For each loading step, 15 layers of the sample were scanned by the x-ray beam. Once grains of each layer were identified, they were cross correlated with the grains of the below and top layers to merge the repeated ones. Stress and strain of the common grains were calculated based on the weighted average of those found in the neighbouring layers. In order to match grains from each loading step, the rigid body movement of the whole scanned volume was firstly determined and corrected. This was done by comparing the COM of common grains from any two steps. Grains from different loading steps were then cross corrrolated.

Results

An example of the measured and modeled values are shown in Fig.1. Comrehensive explanantions are provided in the several journal papers prepared from these results. In Fig. 1a and 1b the measured 3D micorostruture of the CPZr sample at the preload and 1.2% applied strain is shown. More than 11,000 grains are analysed and post-processed. In Fig. 1b, the grain stress measured along the loading direction is compared for different loading stages. It is clear that stress is higher at the higher applied strains. A comparison between measured textrure from 3DXRD and EBSD is shown in Fig 1d. The measured micro-stuture is also imported into the CPFE model (Fig. 1e) and grain strains are simulated and compared agains measured values (Fig 1f).

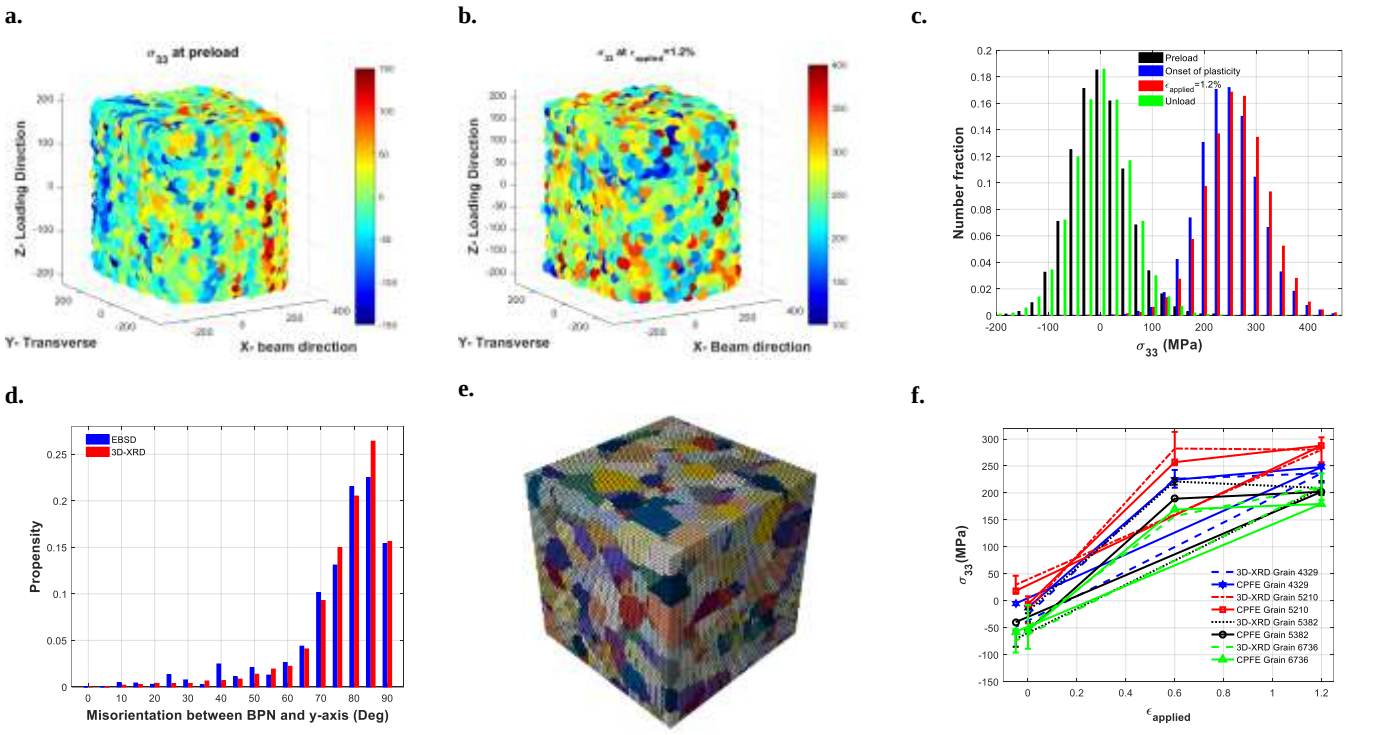

Fig. 1: (a) measured stress fields in more than 11000 grains of CPZr at preload and (b) at the applied strain of 1.2%. (c) a statistical analysis of grains' stress in the loading direction for CPZr (d) comparison between measured texture from EBSD and 3DXRD for CPZr. (e) a crystal plasticity finite element model made based on the measured micro-structure, and (f) comparison between CPFE and 3DXRD

Refrences

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[2] Oddershede J, Schmidt S, Poulsen HF, Srensen HO, Wright J, Reimers W. J Appl Crystallogr 2010;43:539.