ESRF	**Experiment title:** Understanding the nucleation of twins in hexagonal close packed (HCP) polycrystalline materials	**Experiment number**: MA-3613
Beamline: ID-11	**Date of experiment**: from: Feb, 21, 2018 to: Feb, 27, 2018	**Date of report**: March 2020
Shifts: 18	**Local contact(s)**: Jonathan Wright, Marta Majkut	*Received at ESRF:*

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Report:

Hexagonal closed-packed (HCP) metals have been extensively used in various industrial sectors, eg, zirconium in nuclear industry, magnesium in transportation industry, and titanium in aerospace industry. Understanding deformation mechanisms of HCP metals is crucial for developing predictive models that can be used for performance and failures analysis of engineering components. The aim of this research is to employ a novel experimental technique, three-dimensional synchrotron X-ray diffraction, on two different HCP metals and acquire a statistical data that can help us understand fundamentals of deformation twinning. By updating and developing new procedures for grain matching, more than 19000 grains are investigated individually. It is shown for the first time that twin variant selection is preferential in the plastic zone, yet not at the early stages of plasticity.

3D-XRD experiment and setup

The in-situ 3D-XRD experiment was conducted at a high-energy X-ray beamline ID-11 at the European Synchrotron Radiation Facility (ESRF) in Grenoble, France. The experimental set up is shown in figure 1. The laboratory coordinates of the sample are defined based on the positioning of the sample in the uniaxial tensile rig and axes were labelled as loading direction (LD), X-ray beam direction (BD), and transverse direction (TD). The final dimensions of the targeted scanning range are 1mm height, 0.78mm width, and 0.67mm

thickness for zirconium sample and 1mm height, 0.95mm width and 0.93mm thickness for magnesium sample. The experiment is carried out by illuminating a planar beam of monochromatic X-rays.
For the first specimen (Zirconium), the beam energy was set at 78.395 keV, calibrated at platinum's K-edge, and its height was 40 microns in the z-direction, LD. This means that 25 scans for 25 layers are required to retrieve the microstructural data from the entire 1mm height of sample and the specimen is translated such that scanning starts from bottom to top. Toward the end of the experiment, the height of the beam is reduced to 25 microns and the number of layers increased to 40 to cover the entire 1mm scanning range. The reason for this modification was to avoid overlapping of broadened diffraction spots coming from highly plastically deformed zirconium grains.

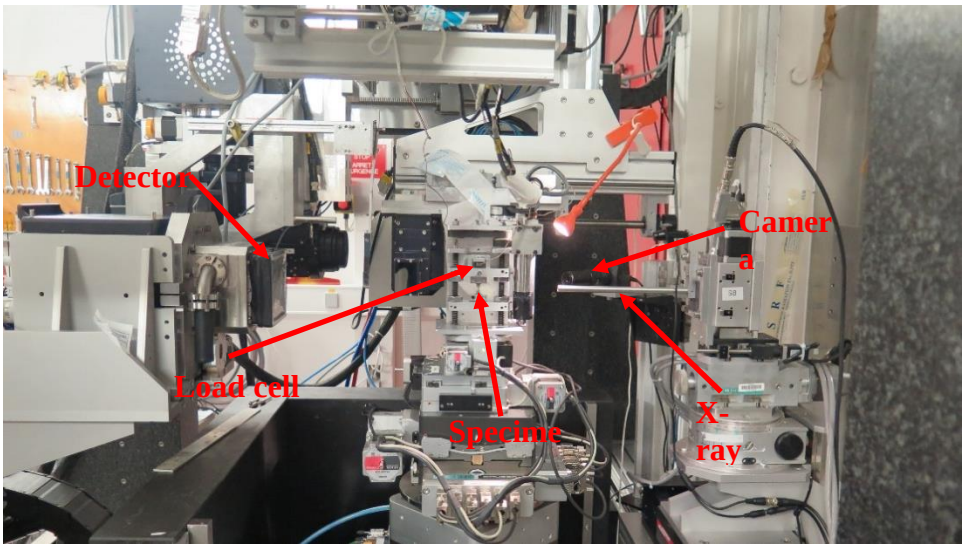

Figure 1: The experimental setup at ID-11, ESRF

The load cell on the tensile rig was used to measure the macroscopic applied stress while the macroscopic strain is measured by mounting two silver wires on the specimen on the plane perpendicular to x-axis, BD. The wires are placed at the maxima of the vertical scanning range and their displacements are tracked using X-ray beam to measure the macroscopic strain. During the in-situ experiment, the zirconium sample was rotated on an omega-stage around the loading direction (LD) and the diffraction patterns were collected in angular ranges of $[-234.5^{o}$ to $-125.5^{o}]$ and $[-54.5^{o}$ to $54.5^{o}]$ in steps of 0.25^{o} to collect the diffraction spots of all grains. The diffraction images were collected on a Frelon2K detector with 2048x2048 pixels with each pixel size of 50x50 μm^2. The distance between the sample and the detector was fixed at approximately 325mm. Likewise, the same experiment was conducted on the magnesium sample. The X-ray beam energy in this experiment was set to 51.996 keV, calibrated Terbium, Tb, K-edge. The dimensions of the sample are 1mm in the vertical direction, LD, 0.95mm width (TD), and 0.93mm thickness (BD). The initial beam height used for scanning is 100 microns. The beam height is relatively large when compared to the beam height used in zirconium due to the large grain size (~60 microns) in the magnesium specimen. After the first two in-situ loading steps, the beam size was halved to 50 microns to capture twins and minimize overlapping of peaks. The total number of layers required to accommodate the full height of the scanning range was initially 10 and then 20 when the beam size dropped. The sample to detector distance was 185 mm for this experiment.

The total number of diffraction images captured from both experiments are slightly close to 300,000 comprising of approximately 4TB size of data. Intensive post-processing was done by coding in Python and using various software by the author of this thesis to index grains.

Results

After indexing grains and performing grain refinement, the COM of each grain is acquired. These layers are used to create the grain map for the entire scanned volume. Since grain morphologies are not available from a 3D-XRD experiment, complexity arises when joining two layers. The main problem that should be tackled in this process is finding grains that exists in two or multiple layers. One way used in finding those grains is setting a misorientation limit between the grains in their distinct layers and distance limit between their COMs.

1. **Misorientation limit**

The misorientation limit between the c-axes of the separated grains and their total misorientation limit are kept equal. Since the zirconium specimen is textured and the magnesium sample is extremely textured, the appropriate limit is required to be chosen such that all the separated grains are picked up and pairing of grains with the "wrong" orientation is avoided. A study is conducted to investigate the number of paired grains (i.e. merging of sectioned grains) as a function of the misorientation limit. This investigation was conducted for the preload step.

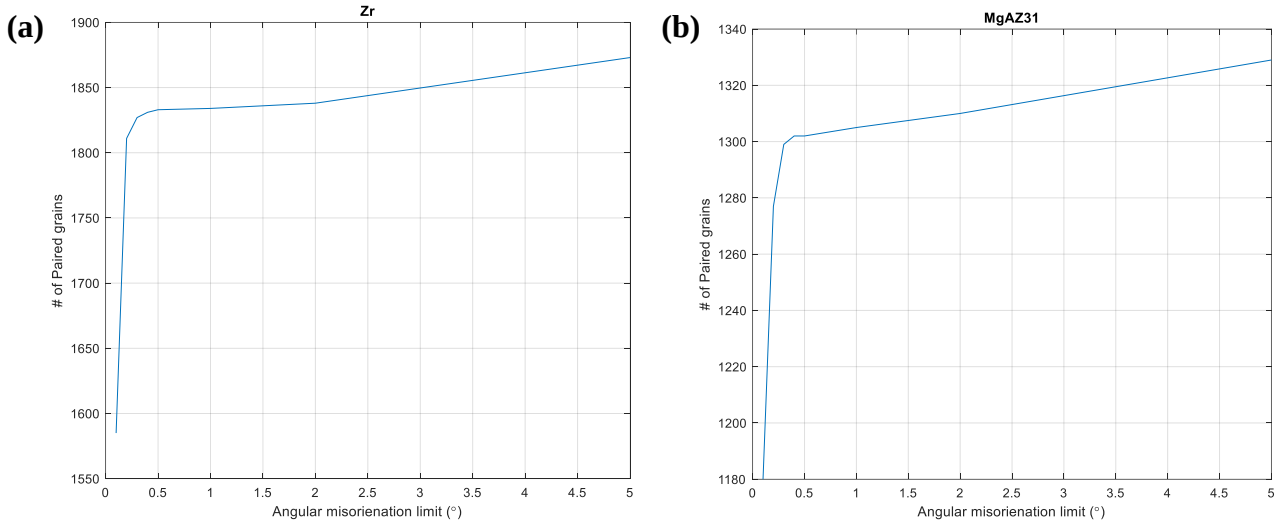

Figure 2: Variation of number of paired grains with angular misorientation limit in pairing grains during grain map construction for (a) Zr and (b) MgAZ31

For both zirconium and magnesium samples, the observed trends, in figure 2, are similar. The number of paired grains rises significantly, with a small change in the misorientation limit, and then converges at a specific number of paired grains followed by a linear positive slope. In the initial rise, the separated grains are picked up since their misorientation is quite low. The convergence indicates that most of those separated grains have been picked up and then the linear slope portrays grains that have been paired up with the "wrong" grain due to their similar orientation. Hence, the appropriate limit that can be used for both specimens is the point of convergence, which is 2° for zirconium and 0.5° for magnesium. However, due to orientation distribution within grain with plastic deformation, with increasing the applied strain, an additional 0.5° is added to the stated limits leaving the final limits as 2.5° and 1° for zirconium and magnesium respectively.

Since EBSD measurements were conducted prior to 3D-XRD experiment, measured grain sizes were used to validate our merging approach. The main measured EBSD was firstly sectioned using the thickness of 40 microns, as shown in figure 3, to be consistent with the X-ray beam size used in the 3D-XRD experiment.

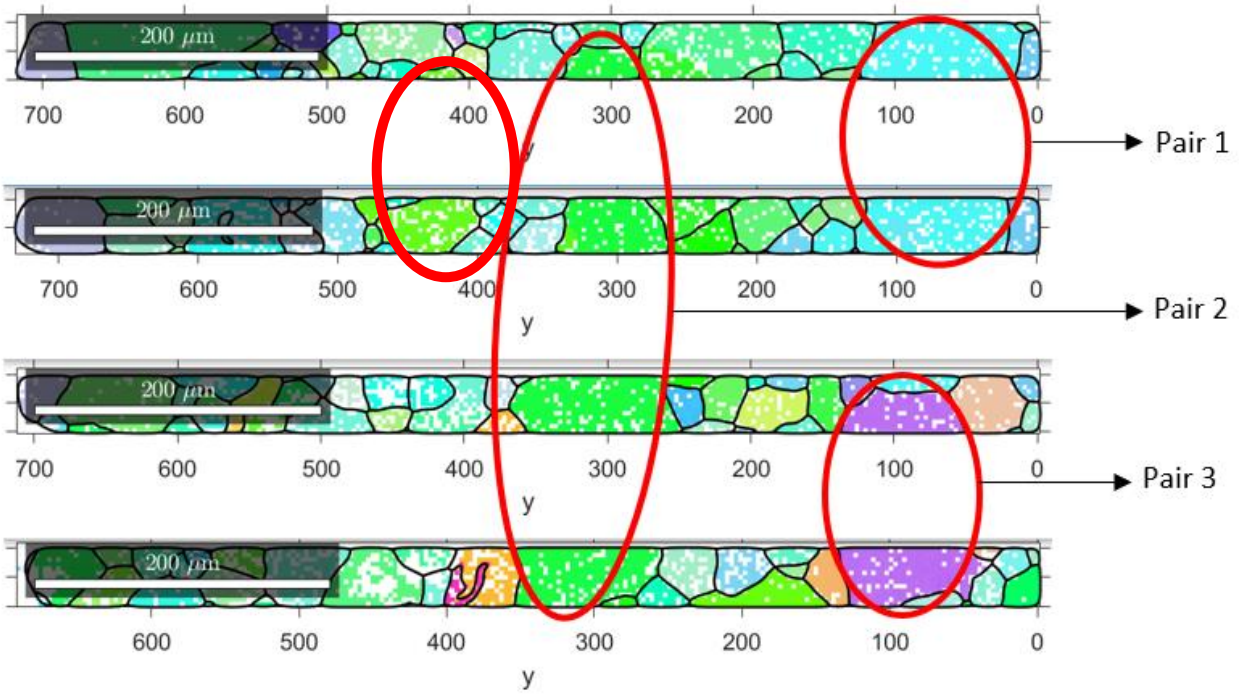

Figure **Error! No text of specified style in document.**: EBSD map sectioned into layers to illustrate the divided grains in Pairs 1, 2, 3

In figure 4, the separated grains in layer 11 and layer 12 of the first step of the zirconium specimen are shown. From the same figure, the COM coordinates in the x-axis and y-axis are similar in both layers but different in the z-axis due to the shift in the z-direction during scanning.

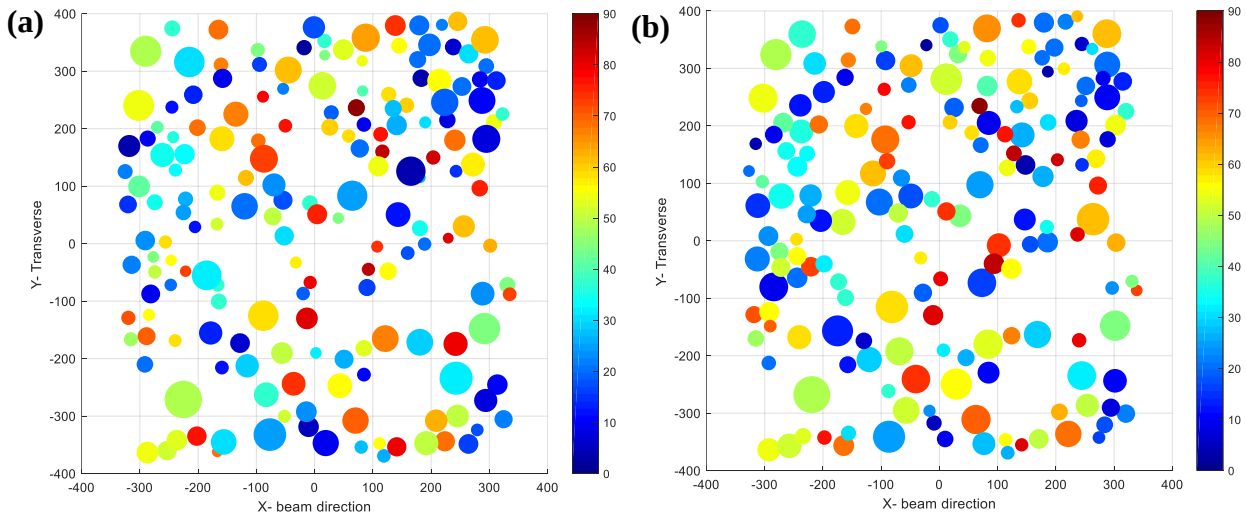

Figure 4: X-Y Plane of the paired grains in Layers (a) 11 and (b) 12 in the 1st step of Zr. The figure demonstrates that all the grains have the same x- and y-coordinates

Concluskon: The results from 3D-XRD were in agreement with EBSD measurements conducted on the same sample.

2. **The evolution of grain resolved stresses**

Due to limited space, selected results are shown here. A reconstructed volume of grains that are matched in steps 2 & 8 in the zirconium samples is displayed in figure 5 (c) and compared with all the grains in step 8, displayed in figure 5 (d). The color bar represents the c-axis misorientation with BD (x-axis).

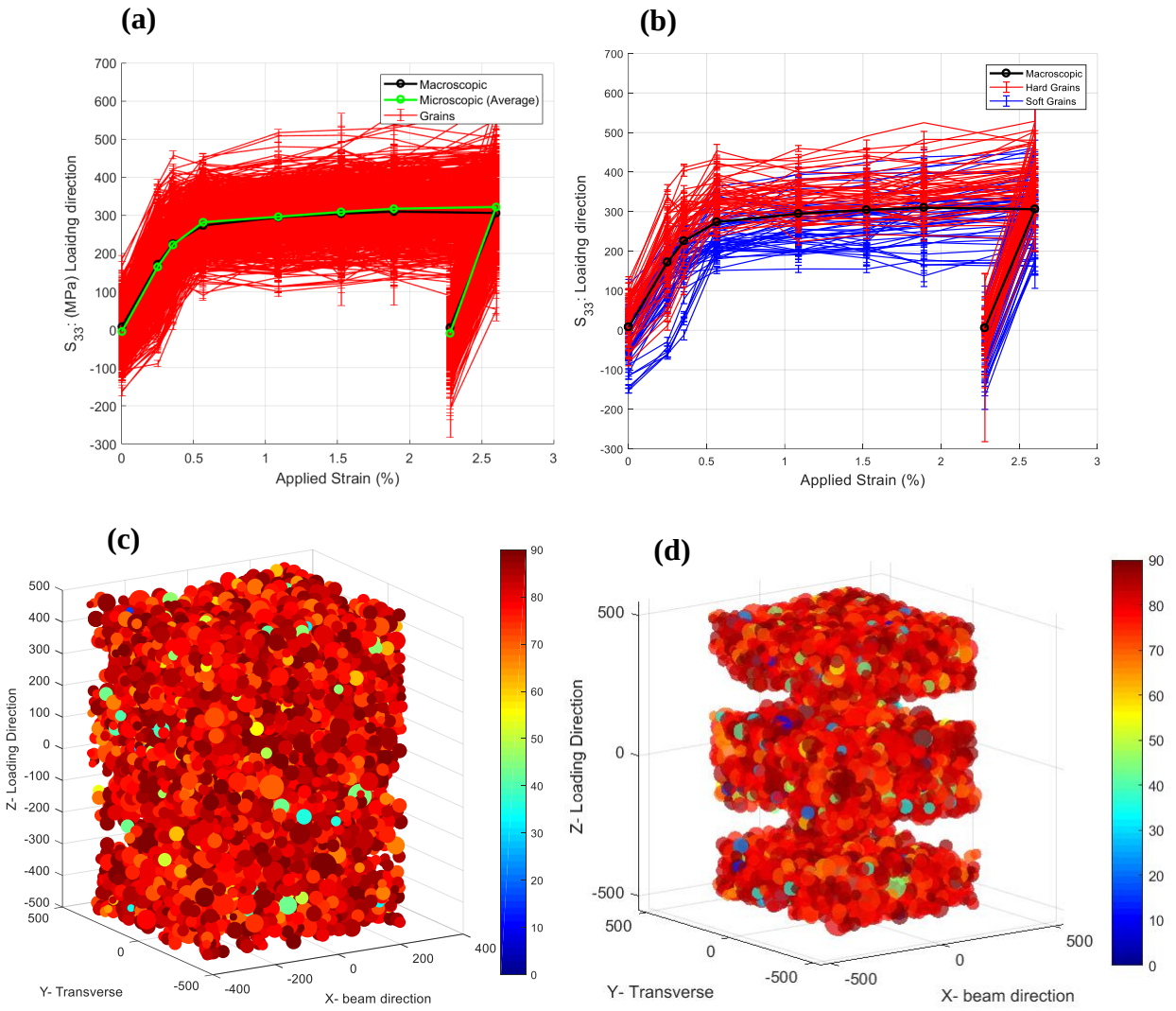

Figure 1: (a) Macroscopic stress-strain plot and stress-strain plots of all grains that are matched in all steps for the zircoium sample, (b) Macroscopic stress-strain plot and stress-strain plot of hard and soft grains, c-axis misorientation with BD (c) of matched grains between step 2 and 8 plotted in step 2 and (d) sliced grain map in step 8

The stress-strain graph in figure 5a shows the stresses in all matched grains in red and the black line is the applied stress measured by the load cell. The green plot represents the weighted average of the stresses measured for all grains in the probed volume. This trend matches applied stress, i.e. macroscopic stress verifying our multi-scale approach. In figure 5b, stress-strain plots of hard and soft orientation grains are plotted and the graphs clearly displays that the stresses in most of the hard grains are larger than soft grains. In order for the hard grain to attain plasticity, a higher stress is required to exceed to the higher CRSS value.