ESRF	**Experiment title:** Understanding lattice strain distributions within anisotropic FCC and HCP polycrystals – key for materials design and modelling	**Experiment number**: MA5414
Beamline: ID11	**Date of experiment**: from: 26.01.2023 to: 30.01.2023	**Date of report**: 01.03.23
Shifts: 12	**Local contact(s)**: Wolfgang Ludwig, Jonathan Wright	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

*P. Gruber[1], *K. Prikoszovich[1], *C. Kirchlechner[1], *O. Hahn[1], M. Krause[2], T. Böhlke[2]

[1]Institute of Applied Materials - Mechanics of Materials and Interfaces, Karlsruhe Institut für Technologie, Hermann-von-Helmholtz-Platz 1, 76344 Eggenstein-Leopoldshafen

[2]Institute of Engineering Mechanics, Karlsruhe Institut für Technologie, Kaiserstraße 10, 76131 Karlsruhe

Aim of the experiment:

The aim of these experiments was the validation of theoretical micromechanical methods to estimate local stresses for anisotropic BCC and HCP polycrystalline materials, in particular the Maximum-Entropy-Method (MEM) [1], by diffraction contrast tomography (DCT). The MEM formalism allows to predict stresses on the grain level while requiring microstructural information and macroscopic mechanical properties. Using the DCT method at ID11 we obtained all the necessary input data for MEM calculations while also providing the average elastic strain for each grain, which can be used to validate and challange the MEM results. Additionally the DCT data will provide first insights into strain localisation in polycrystalline materials in the elastic region, which has not been investigated yet using 3D X-ray techniques [2] and which is expected to trigger further experiments.

Samples and preparation:

Armco iron and 99,99% pure titanium have been selected for the experiments due to their lack of annealing twins [3] and large amount of literature available. Prerolled sheets of both materials have been purchased from Goodfellow©. After coldrolling the samples have been put in a vacuum oven with the aim to obtain an average grain size of about 50μm, the heat treatment recipes are shown in Table 1.

Material	Annealing recipe	Pressure
Armco Iron	35h at 500°C + 2h at 750°C	10^{-5} mbar
99,99% Titanium	35h at 500°C + 1.5h at 600°C	10^{-5} mbar

Table 1: Annealing recipes for the iron and titanium samples in the vacuum oven at KIT.

After the annealing procedure, the samples were grinded and polished on both sides to remove surface damages and obtain plane material sheets of around 0.5 mm thickness. Two different sample designs were used: (i) Electric discharge machinging (EDM) was performed at KIT and ESRF to produce the dogbone shaped tensile testing geometries required for the Nanox tensile rig [4]. Such samples exhibit an rectangular cross section. (ii) In addition, the company microsample© prepared tensile samples with a circular cross section using a unique grinding procedure [5]. The surface of the EDM-machined tensile samples was carefully grinded with fine (1200+) SiC paper to remove preparation artefacts and surface flaws caused by EDM. All samples were speckled with amorphous SiO_2 spheres soluted in Isopropanol.

Experiments and results:

Before the *in situ* experiments we performed tensile tests for each material and both sample designs to determine the yield strength (see Figure 1).

For each sample we performed similar loading sequences: First, the load was incrementally increased to the high elastic region (close to the yield stress), after which the load was stepwise decreased to a certain point. In the second loading, the sample was brought in to the plastic regime until the plastic deformation lead to non-detectable and distortedDCT diffraction spots with high spot overlap. To better illustrate the loading sequence colorcoded points were added to Figure 1 marking the individual loading steps.

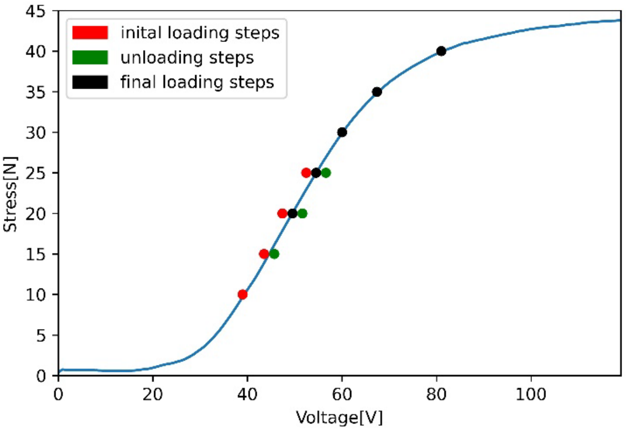

Figure 1: Mechanical data of an EDM-machined iron sample. X-axis shows the voltage applied to the straining piezo. The colorcoded points show the loading steps we chose for the DCT experiments.

At each loading step we applied the same scan sequence consisting of the following scans:

- a Phase Contrast Tomography (PCT) scan of the entire sample gauge to determine the position of the amorphous SiO_2 spheres. **Aim:** Tracking the change of the sphere positions by digital image correlation (DIC);
- 5 DCT scans, each illuminatig a consecutive 100µm thick layer, which will be stitched together after reconstruction. **Aim:** Tracking the position, shape and orientation of each grain as input data for MEM;
- 10 Farfield (FF) Tomography scans, illuminating the same total volume as the DCT scans with each scan covering 100µm, but for easier stitching results each consecutive scan includes the upper half of the previous layer. **Aim:** Measure the average stress tensor (in addition to the orientation and location of the grain) for each grain to validate MEM.

Figure 2 shows an XY slice of a PCT scan, on the right image detail the 5 µm sized microspheres can be seen. The position of these microspheres will be used to determine the macroscopic longitudinal and transvers strains of the tensile sample using DIC.

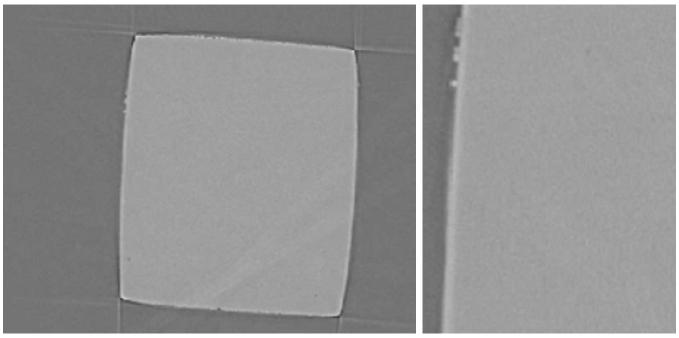

Figure 2: : PCT scan cross section of iron. Right image shows a zoomed in region to better show the micro spheres.

Figures 3 and 4 show cross sections in the XY plane of a DCT reconstruction for the different iron and titanium samples. In case of iron (see Figure 3) we were able to reconstruct all grains, i.e. no grains are missing, documenting the high surface finish with neglgible preparation artifacts. Therefore, we are confident that our sample preparation technique works well for pure iron samples.

The titanium samples in Figure 4 on the other hand had a lot of deformed grains on the surface for both sample designs complicating DCT analysis. For the EDM samples more care and additional steps with finer grinding paper would have helped. For the very soft titanium, further developments in surface preparation are necessary with regard to a good compromise between surface deformation and material removal rate to obtain similar surface features to the iron samples.

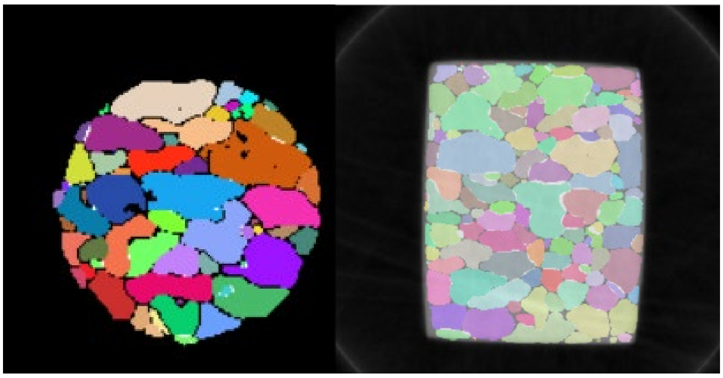

Figure 3: Cross sections of a DCT reconstruction for different iron samples. Left: microsample©, Right: EDM-machined sample with careful grinding steps up to 4000.

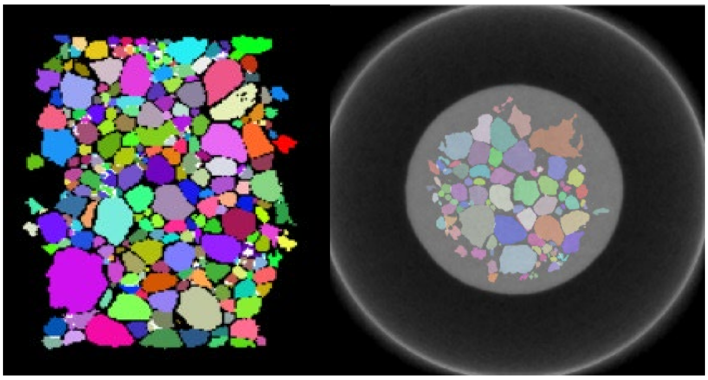

Figure 4: Cross sections of a DCT reconstruction for different titanium samples. Left: EDM-machined sample, Right: microsample©

A statistical relevant number of grains is required to validate the MEM. To achieve this, our goal was to investigate at least 1000 grains for each sample. Table 2 shows the number of grains per layer as well as the total number of grains determined by an individual and the full series of DCT scans, respectively . For all samples, the targeted number of grains was achieved. Only for the Microsample-Fe sample slightly less than 1000 grains have been measured, but this is still enough for meaningful MEM calculations.

Sample type	Grains per layer	Total number of grains
EDM-Fe-ESRF	420	2100
Microsample-Fe	170	850
EDM-Fe-KIT	400	2000
EDM-TI-ESRF	1200	6000
Microsample-Ti	600	3000

Table 2: Number of grains per layer and total number of grains for all samples.

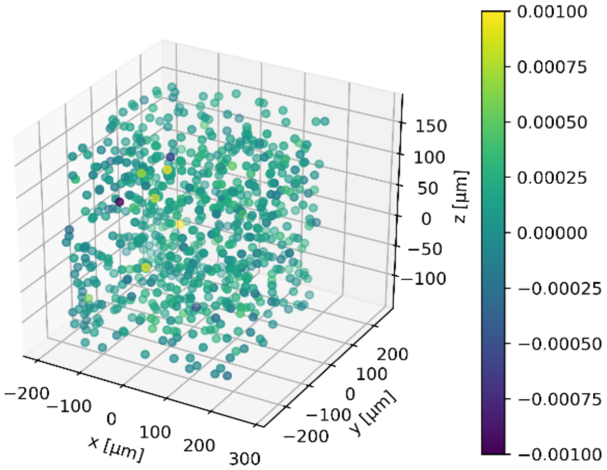

Figure 4: z-component of the average elastic strain tensor for each iron grain.

The grain positions and z-components of the average elastic strain tensor of each grain obtained with the FF scan from the EDM-machined iron sample from Figure 3 can be seen in Figure 5. The load was set to be approximately 5N, most grains are loaded in tension with a few grains having high strain values (up to 10^{-3}) compared to the majority and a few grains even experience compressive strains.

The FF data was used to obtain the inverse pole figures (IPF) for different sample axis as shown in Figure 6 for an EDM-Fe sample. Figure 6 illustrates a mild but noticable texture (see lack of [111] orientations along the straining axis (Fig. 6, right). The mild texture is clearly not in conflict with the aims of expriment MA-5414, however, it rises new questions on the role of textures on MEM and its validation.

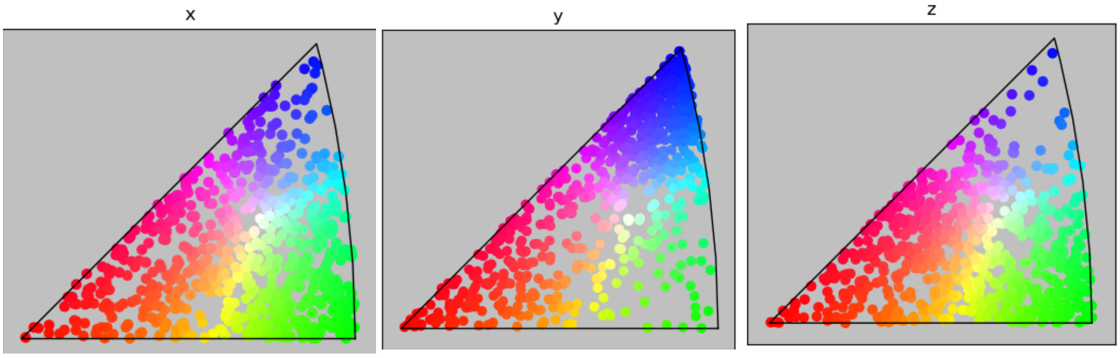

Figure 6: IPF of the EDM-machined iron sample in Figure 3 projected onto the different sample axes. The sample z-axis is parallel to the straining axis.

Modification of the experimental protocol with respect to the initial proposal:
Compared to the initial proposal we have changed two major experimental protocols:

1) Instead of using face centred cubic (FCC) we used bodycentred cubic (BCC) metals, specifically iron. This modification was motivated by the beamline crew, because of their negative experience with twinning in FCC metals, which currently can not be handled easily during data analysis.
2) Instead of using optical microscopy for tracking sample shape canges and feature tracking, we used PCT to track the position of amorphous SiO_2 micorspheres applied to the sample surfaces . This enabled a 3D-reconstruction of the macroscopic sample strains with superior spatial resolution and easened our experimental setup (no implementation of an optical microscope in the experimental setup needed).

Open question(s) and current work: From an experimental viewpoint we have succesfully conducted all experiments to achieve the primary goals of MA5414. Currently, data analysis and post mortem sample characterization in our home-lab at the Karlsruhe Institute of Technology is ongoing and could not be completed in the short interval between the experiment and the due date of this report (4 weeks). Already now, several new puzzling questions are identified based on MA5414 which will be part of a new proposal.

Conclusion:
Four weeks after the beamtime we are still performing an in-depth analysis of all the data and currently start the comparison with MEM approaches. However, already at this early stage, we can conclude:

1) Our sample manufacturing protocol, particularly the EDM-sample design, is well-suited for the intended aim. The sample quality allows for DCT and FF experiments with the required resolution.
2) The PCT on silica microspheres dispersed on the sample surface – with which we replaced the initially proposed optical inspection – proofed to be ideal to track the macroscpic strains in all directions in an accurate, fast and efficient way.
3) The number of grains obtained within one loading step (see right column in Tab. 2) allows for a validation of the MEM, even though this requires stacking of consecutive layers. Per load step we achieved the required number of at least 1000 grains for all samples.
4) The established experimental methodology yields all macroscopic and microscopic parameters which are necessary to accomplish the comparison of the experimental data with MEM appraoches.
5) The first implementation of the experimental data into the MEM framework yields three major observations: (i) Sample eigenstrains are much larger than expected for fully recrystallized and coarse-grained metals, (ii) an informed MEM considering the eigenstrains can predict the evolution of the overall strain distribution in the samples, however, (iii) even for 1000 grains, the variation of the local neighbourhood of individual grains with specific orientation is still low.
6) Based on these observations, we would suggest to investigate samples with different eigenstrain distribution produced by specific annealing conditions and to increase the number of illuminated grains to about 10000. The acquisition rate of DCT at ID11 allows for this increase in sample size and simulations show that this would be sufficient to investigate and incorporate neighbourhood effects.

References

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