	Experiment title: Crack initiation at the micron scale: Unprecedented insights by combined high resolution 3D-DAXM and in situ micromechanics.	**Experiment number**: MA-3607
Beamline:	**Date of experiment**: from: November 8th 2017 to: November 14th 2017	**Date of report**:
Shifts:	**Local contact(s)**: Jean-Sébastien Micha, Olivier Ulrich, Loic Renversade	*Received at ESRF:*

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Report:

<u>Objective:</u>

The aim of this beamtime was to install, test and operate a new feature of the "Differential Aperture X-ray Microscopy" (called DAXM) technique, combined with a nanoindenter (Figure.1). Scientifically, the aim was furthermore to understand the first stages of fatigue crack initiation. Therefore, we performed low cycle fatigue experiments *in situ* on micron sized focussed ion beam (FIB) milled copper microsamples. The samples were single crystalline with a standard size of $5x5x25\mu m^3$. This experiment is directly related to the ANR-DFG XmicroFatigue project (ANR-16-CE92-0024 / DFG KI-1889/3-1).

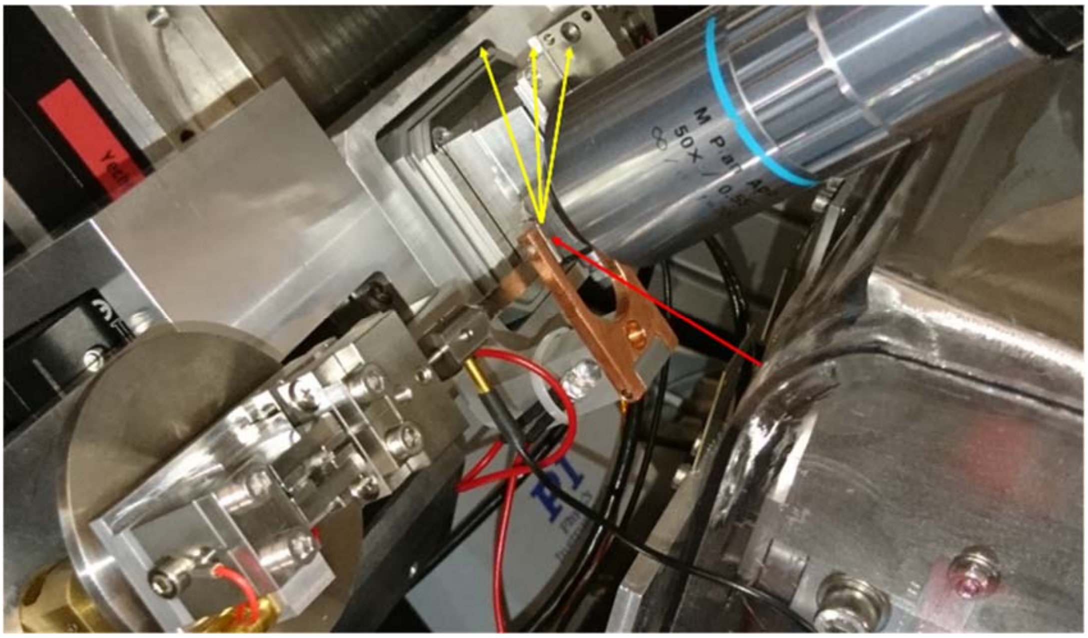

Figure 1: Nanoindenter combined with a DAXM setup installed during MA-3607.

Experiment protocol and achievements:

To our knowledge, this is the first succesful *in situ* 3D submicron volume scan. For the first time we were able to use three tungsten wires (50µm diameter, called W-wire) for DAXM scans with considerable reduced the time for a 3D submicron volume scan and therefore allowed the interrupted *in situ* scans. Those W-wire were moving parallel to the camera, at constant step in order to optimize the scanning time, and the depth resolution of our experiment (Figure 2).

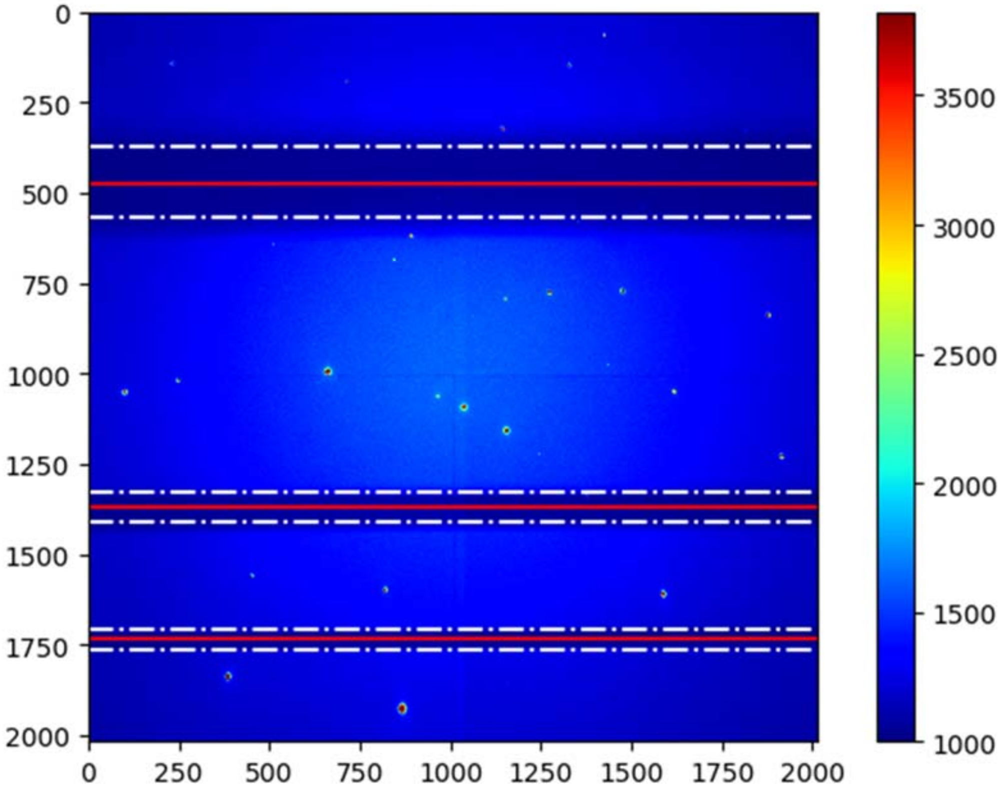

Figure 2: Detector frame with three shadows originating from the 50µm sized W-wires.

Due to the achieved acceleration of a factor four we were able to deform multiple fatigue cycles on these samples. With this regard, unprecedented data on the formation of fatigue damage was recorded. Two cantilevers were cyclically deform during the beamtime. Both of them were submitted to one full cycle fatigue (Figure 3). Mechanical data (force and displacement), and µLaue patterns were recorded simultaneously during each step of the cycle. *In situ* loading was interrupted after ¼, ½, ¾ and 1 cycle in order to thoroughly investigate the local fatigue structure formed. Therefore, a region of 10x10x12µm³ (Figure 4a) was scanned with the DAXM setup with a step size of 1µm and a beam size of 0,9x0,9µm².These scans are further called 3D submicron volume scans. They were performed at the bottom (first 12µm) of the cantilevers where the streaking of the µLaue pattern appears during deformation.

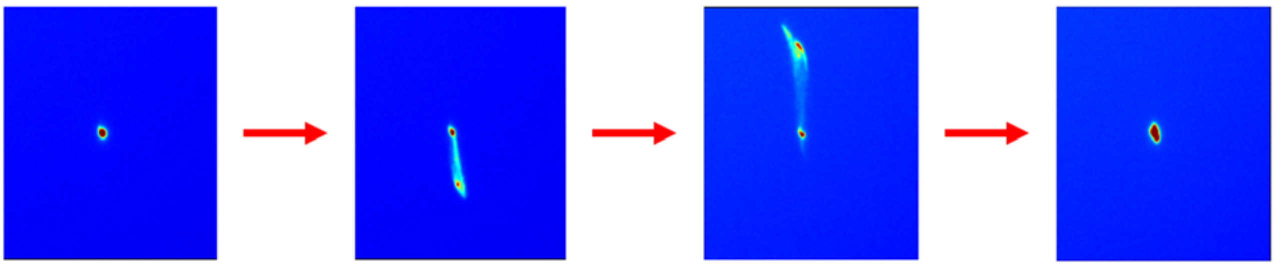

Figure 3: Evolution of one diffraction peak during cycle fatigue, recorded in situ. Representation of one full cycle.

Data analysis is still ongoing, but the first quantitative results show the tremendous success of this experiment. The data contains for instance unique information on the formation on dislocation cells, the local density of GND's and local deviatoric strain fields in this cells, as well as their evolution during fatigue.

An example is show in Figure 4b. It shows a representation of the deviation of the experimental peak position with respect to an unstrained reference in 3D, and therefore can be interpreted as a strain. The sample geometry is well represented in Figure 4b. There is a possibility to represent this parameter in full 3D, as shown Figure 4c.

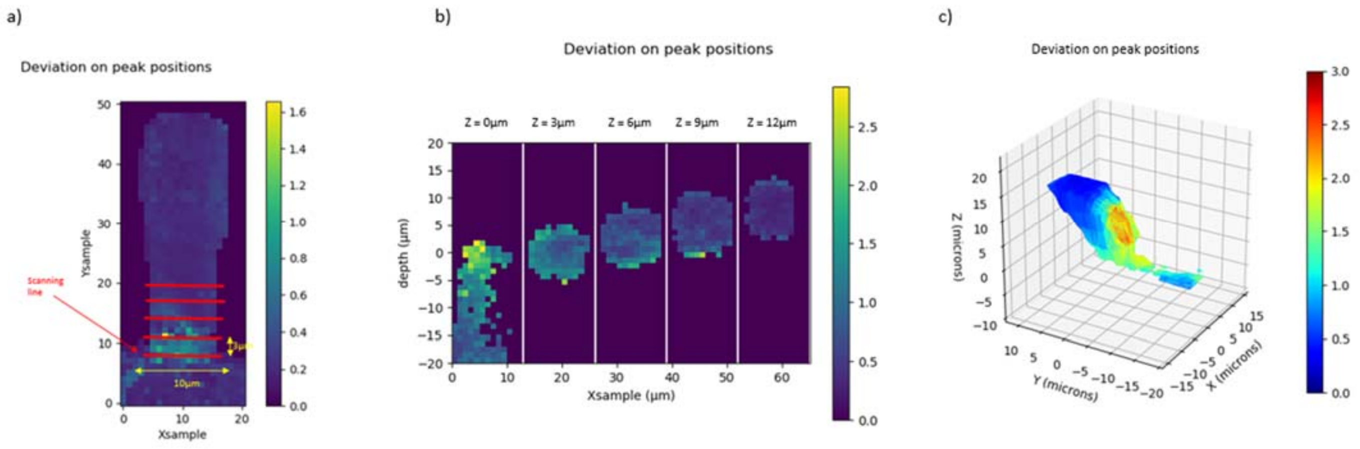

Figure 2: Representation of the cantilever. a) Scanning protocol of the cantilevers after full cycle deformation. And representation of the deviation on peak position in the sample, b) in 2D, c) in 3D.

<u>Conclusion:</u>

The combined nanoindentation DAXM setup is now ready to be used. The first experiments showed promising results for single crystals which will soon be published in a well recognized journal of the field. In the future, this approach will offer unique data to better understand fundamental properties like dislocation slip transfer through grain boundaries or the formation of fatigue damage at grain boundaries.