

**Experiment title:**  
Study of incipient plasticity in Ti7Al alloy by X-ray topotomography

**Experiment number:**  
MA-3921

**Beamline:**  
ID11

**Date of experiment:**  
from: 03/05/2018 to: 09/05/2018

**Date of report:**  
28 Feb. 2020

**Shifts:**  
18

**Local contact(s):**  
W. Ludwig

*Received at ESRF:*

**Names and affiliations of applicants** (\* indicates experimentalists):

Dr. Henry Proudhon\*, Centre des Matériaux MINES ParisTech, PSL Research University

Dr. Jean-Charles Stinville\*, Materials department, University of California Santa Barbara

Dr. Patrick Callahan\*, Materials department, University of California Santa Barbara

**Report:**

The MA1921 experiment took place in May 2018 at the ESRF, Grenoble. The main goal of that experiment was to study the plasticity mechanisms in a Ti7Al alloy using a combination of Diffraction Contrast Tomography (DCT) and Topo-tomography (TT). The method has been previously used to study an AlLi on a single sample deformed in situ with the Nanox stress rig (results published in materials in 2018, see Fig. 1). One of the limitation of the previous work was the small number of grains (3) characterized during the experiment. For this experiment, the goal was to collect more statistics and to scan larger grain clusters to be able to capture both plastic activity and slip transmission events from one grain to the next. Also a different material: Ti7Al with an hexagonal crystal structure was used.

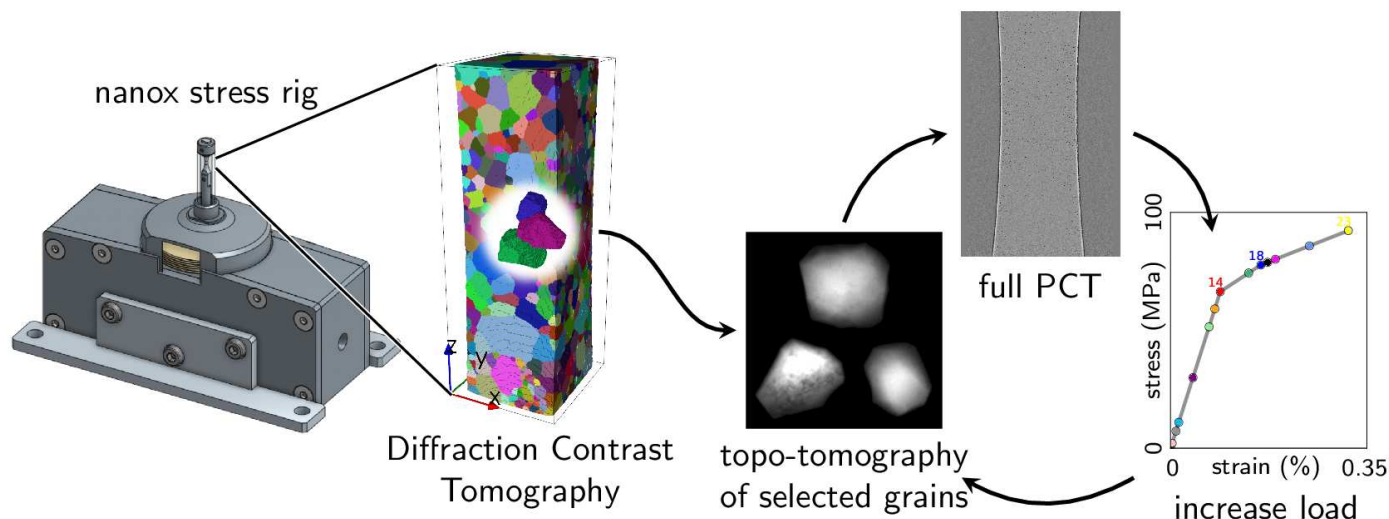


Fig. 1: Experimental procedure combining DCT and TT to study plastic localisation in bulk grains.

## 1 Specimen preparation

Several specimens were prepared from a forged Ti7Al piece with a grain size around 100 microns. Samples were EDM cut, polished, characterized by EBSD. The surface of the gage length received a nano-speckle and some specimens were pre-strained at UC Santa Barbara (United States, see Fig. 2) with various levels of deformation between 0.4 and 1.0%. Specimens were then transferred to ESRF for the synchrotron experiment.

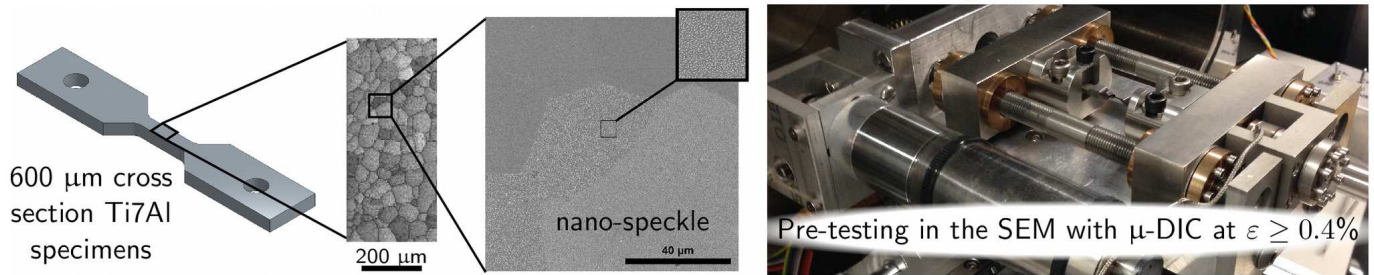


Fig. 2: Specimen preparation and pre-straining at UCSB.

Here is the list of the samples, divided into ex situ and in situ, that were prepared for this experiment:

- T1 1.01% strain ex situ EBSD and DIC
- T2 0.58% strain ex situ EBSD and DIC
- T3 in situ EBSD and DIC
- T4 0.49% strain ex situ EBSD and DIC
- T5 in situ EBSD and DIC, labDCT scan
- T6 in situ, no EBSD
- T7 0.64% strain ex situ EBSD and DIC
- Ts1 0.44% strain ex situ EBSD and DIC
- Ts2 0.67% strain ex situ EBSD and DIC
- Ts3 0.83% strain ex situ EBSD and DIC
- Ts4 in situ, EBSD and step0 DIC
- Ts5 in situ, EBSD and step0 DIC

## 2 Initial DCT characterization

Series of DCT scans have been conducted on samples T1, Ts2, Ts3, T4 and T5. Several scans were carried out at different heights to be able to reconstruct a large portion of the gage length similar to the initial EBSD mapping of 1.5 mm. The DCT reconstruction matched very well the surface EBSD information (see Fig. 3). Mapping a large part of the gage length is also important for subsequent crystal plasticity simulations based on the experimental image and carried out using finite elements. Indeed boundary conditions (typically imposed displacements) need to be applied far from the grain of interest in order not to perturb the solution.

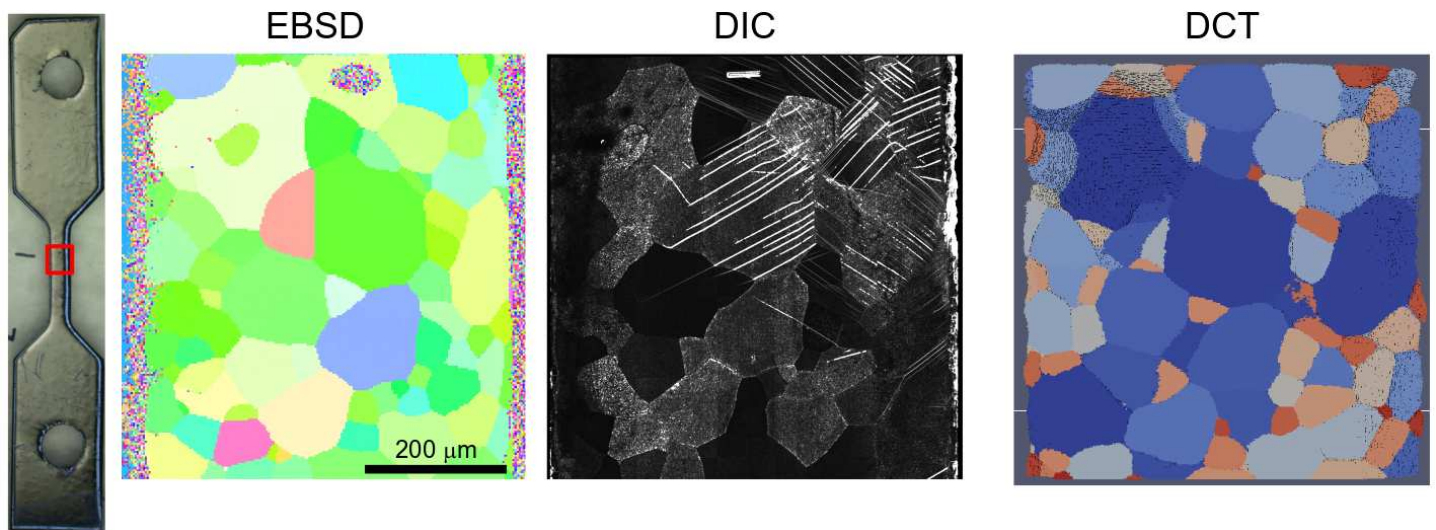


Fig. 3: Comparison of our multi-modal imaging (EBSD, DIC and DCT), when looking at the sample surface.

### 3 Topotomography imaging

DCT scans were used to process the microstructural informations during the experiment to find interesting grains for topo-tomography inspections. First few grains were aligned by a manual process (in particular we needed to verify that our crystallographic calculations with the hexagonal structure were right) and then the alignment procedure has been completely automated from the DCT information alone and proved to be very reliable. In pre-deformed sample Ts3, more than 30 grains were scanned automatically using this procedure. This constitute an important data set were we imaged the plastic activity in both surface and bulk grains showing series of prismatic and basal slip bands in the different grains. The bands developing in the volume of the surface grains could be related to the bands observed at the surface from the DIC measurements.

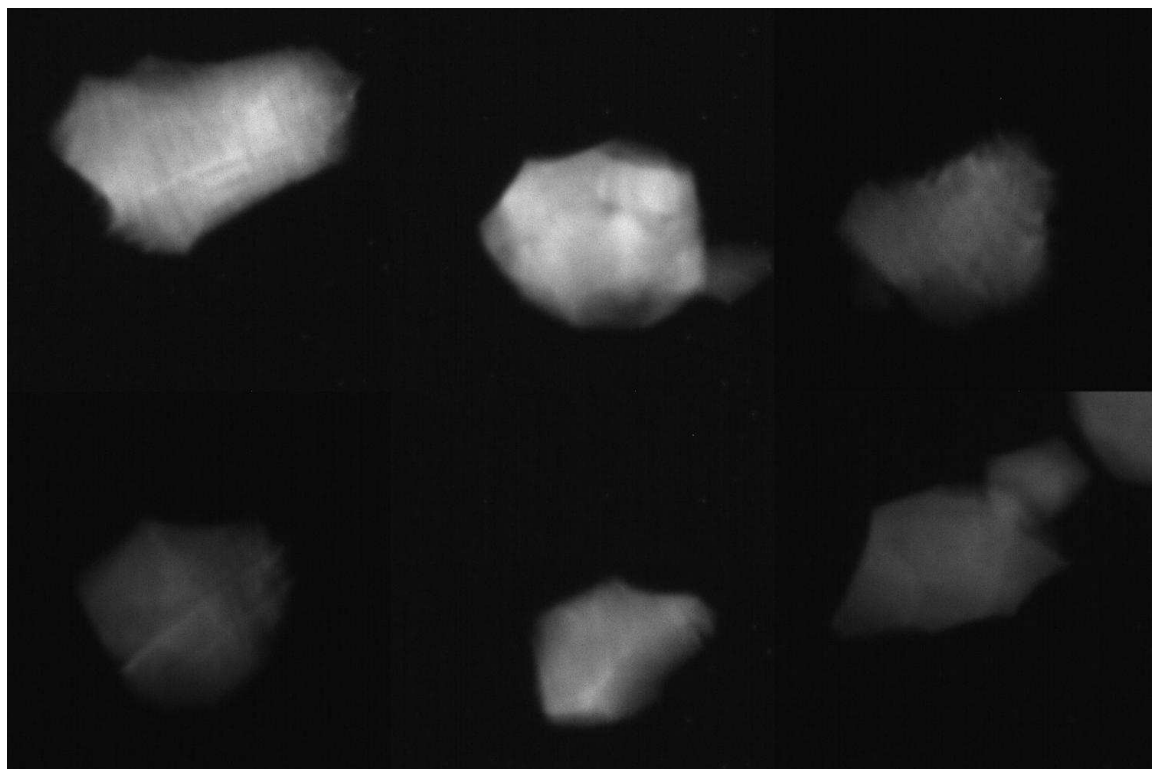


Fig. 4: Examples of topographs recorded in 6 different grains of sample Ts3.

## 4 In situ topo-tomography study

Sample T5 was loaded into the Nanox stress rig (see Fig. 5) and subjected to a series of load-imaging loops (see Fig. 1).



Fig. 5: Photograph of the in situ setup with sample T5 mounted into Nanox and installed on the ID11 diffractometer.

The automation scripts proven to work with a larger number of grains and a cluster of 10 grains around a well oriented grain for plasticity was followed using TT imaging at each load step. This large data set is still under analysis but it appeared that depending on its orientation, each grain showed a different behaviour. Both basal and prismatic slip activity have been captured in situ (see Fig. 6 for an example). The in-depth analysis will show if any slip transmission event in the bulk could be captured.

Edge-on configuration for grain 09 and plane (-12-10)

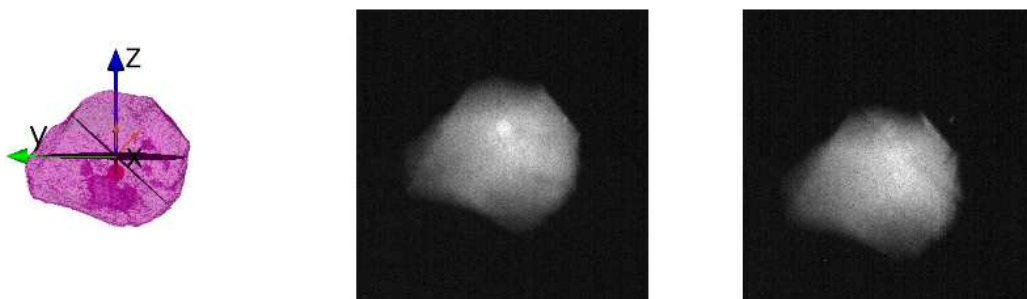


Fig. 6: Bulk grain from sample T5, oriented for basal slip which was shown to depict slip activity as observed in the topographs recorded.