ESRF

Experiment title:
Deformation and fatigue behaviour of polycrystalline nickel based superalloy

Experiment number:
MA-1921

Beamline:
ID19

Date of experiment:
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Report:

The MA1921 experiment took place in November 2013 at the ESRF, Grenoble. The main goal of that experiment was to validate the design of a new in-situ tensile Device called NANOX. The aim of this device is to provide a load capabilty to probe in-situ the mechanical characteristics of materials, essentially thanks to the 3DXRD (3D X-Rays diffraction) technique. The design of this machine allows to load from 0 to 500 N tensile specimens close (2.5 mm) to a FRELON camera (see Fig. 1). It will provide unique capabilities to reach a better understand of the physical mechanisms involved in mechanical phenomenom such as cracking or strain localization, which are active reaserch areas.

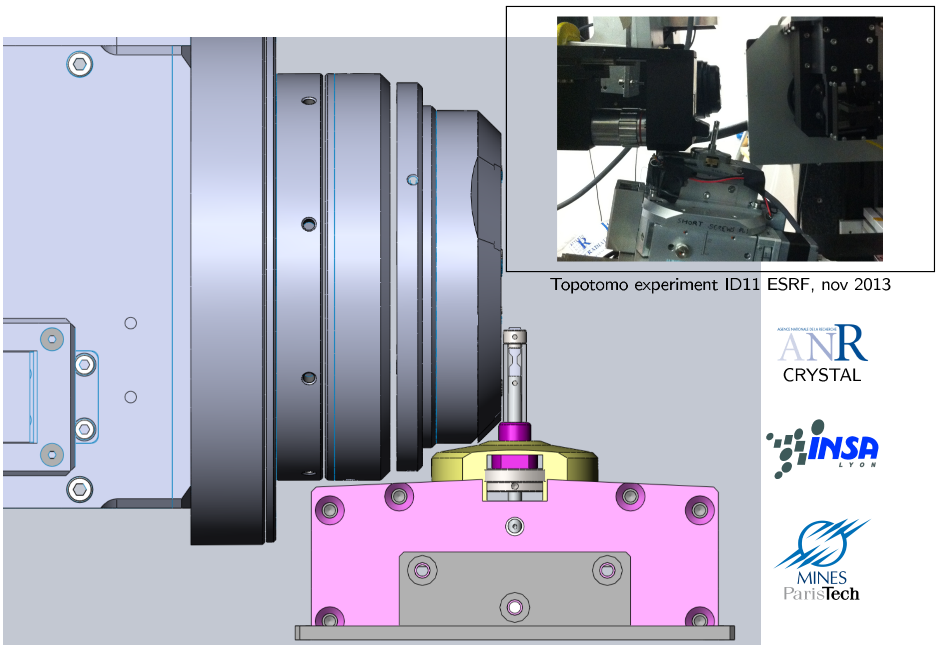

Fig. 1: CAD View of NaNox and a picture taken during the MA1921 experiment, topo tomographysetup (tilted as 13°).

1 The NanoX device

NaNox was designed in collaboration with MinesParistech and INSA Lyon to provide new experimental capabilities for synchrotron based studies at ID11. The sample is loaded thanks to a piezo actuator, which allow to apply load with the needed accuracy, and either statically or dynamically. The elongated design allow users to approach a frelon camera up to 2.5 mm to the center of rotation (0.5 mm from the glass tube), to perform high quality PCT and/or DCT scans. The device is fully compatible with topotomographic scans (see inset in Fig. 1), but a little further to the detector due to tilted surfaces. During the MA1921 experiment, we were able to mount the device on the motorized goniometer on ID11. One of the advantage of this device is to be fully integrated with spec. One could control the piezo motion, and read the load without any action in the hutch, which is a great advantage for optimizing beamtime.

2 A Pb balls virtual extensometer

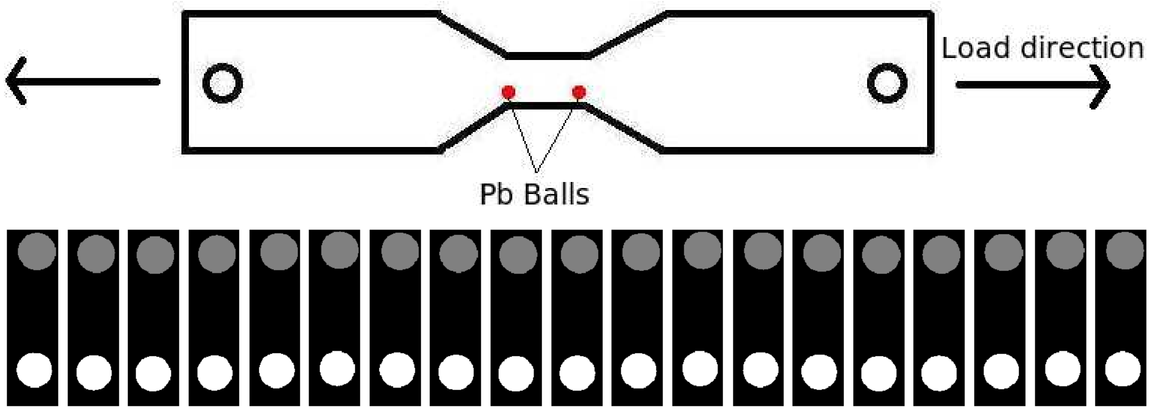

Fig. 2: small 316L tomographic sample with one 50 microns lead balls glued at each end of the gage length.

In order to use Id11 experiment as useful data for mechanical models, one need to know the macroscopic strain applied to the sample. Regarding the tiny design of the device, we have decided to test a virtual extensometer consisting in 2 Pb balls glued on the sample surface (see fig. 2) and tracked them to know how much the sample have been elongated. Images are taken in the direct beam. The radios are automatically processed by a python script: the two balls are labelled and the center of mass are computed. It is a digital images correlation (DIC) like method.

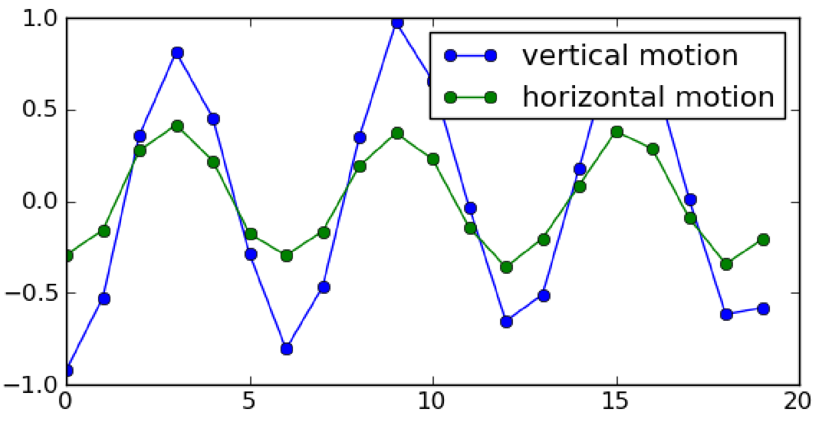

Fig. 3: 100 N cyclic load is applied at 0.1 Hz. X-ray radiographs are recorded every 0.2 sec and are processed automatically.

The test was performed with a 316LN steel mounted on the device, and one can use those values to verify the calibration / measure Young's modulus. for instance in our case, the load was 100N with a section of 0.45×0.45 mm^2. The measured displacement is 1.8 micrometer over a gage length of 169 pixels which results in a modulus of E=210 GPa (Fig. 3), very close to the known value for this material. These results allowed us to validate this method and generalize it to further experiments.

3 DCT scans

A Ni based superalloy was initially planned for those DCT experiments but finally turned out to be inappropriate (too wide grain size distribution). Instead, the material adopted to conduct our experiments is a binary Al-Li alloy, which allow fast scans due to his low average (12.73) Z number (high flux thanks to low absorption). We have tested several samples which appears to be to much deformed, but we were able to find one suitable for a DCT scan (see Fig. 4).

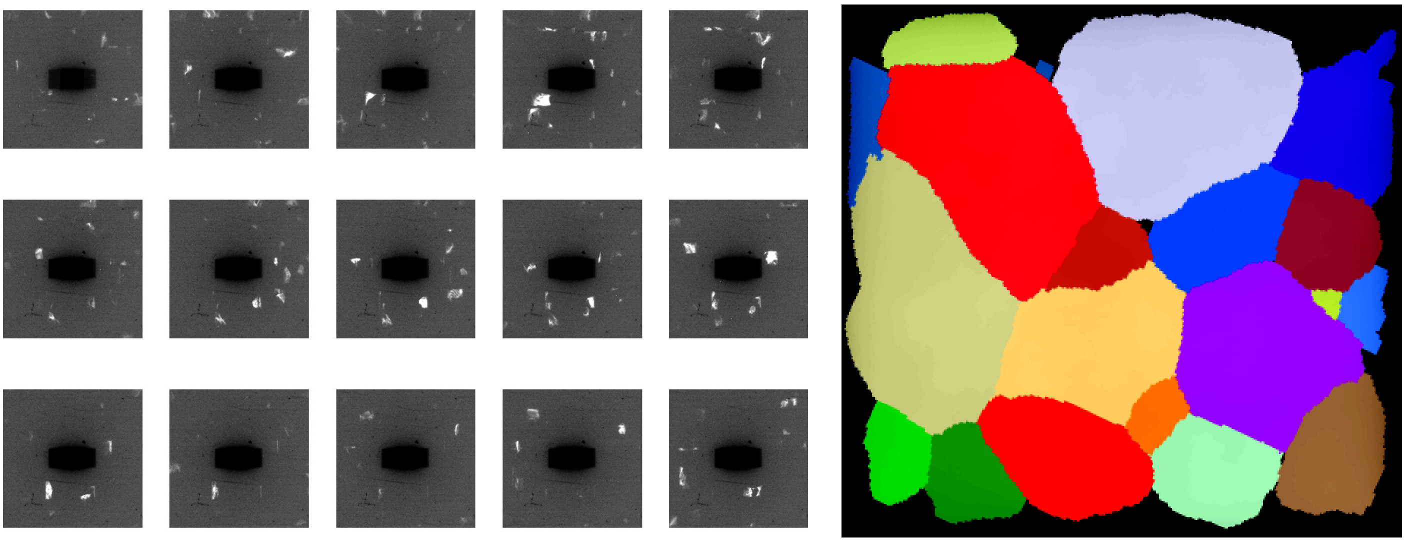

Fig. 4: Raw images of the detector showing diffracting grains (left) and a slice of the reconstructed volume, different colors represent different grains (right).

This phase was essential to the device validation, as a basic step. Furthermore, we were able to get better ideas of how difficult it could be to mount tiny sample without breaking them or deforming them too much to perform accurate experiments. Finally, recording DCT scans before performing topotomo acquisitions is essential, the basic idea for further experiments is to choose the best grain in the volume for the topotomoraphy. This choice has to be made automatically, by a macro and based on the DCT data. The next part of this report will deal with the topomography scans made during the MA1921 experiment.

4 Topotomography scan

Our main idea was, that DCT scans have a lot of advantages, but if we want to study submicrometer phenomenon, the resolution is to scarce to visualize them. We decided to couple a DCT scan to know the microstructure, which is essential as grain neighbourhood has a strong effect on crack initiation, and a topotomography scan to probe with a higher resolution the behavior of the grain of our choise, a grain expected to be subject to plastic strain localization and crack initiation.
Before the MA1921 experiment, we did not know that it could be such a difficult task to align a grain spot *i.e. aligning the scatering vector and the axis of rotation.* One of the main difficulties is that there is no landmark which is easyly recongnizable of the same grain at two different orientations. We suppose now that we have tried to align two different spots. After several tries we were able to perform topotomography scans of a moderately deformed grain (see Fig. 5).

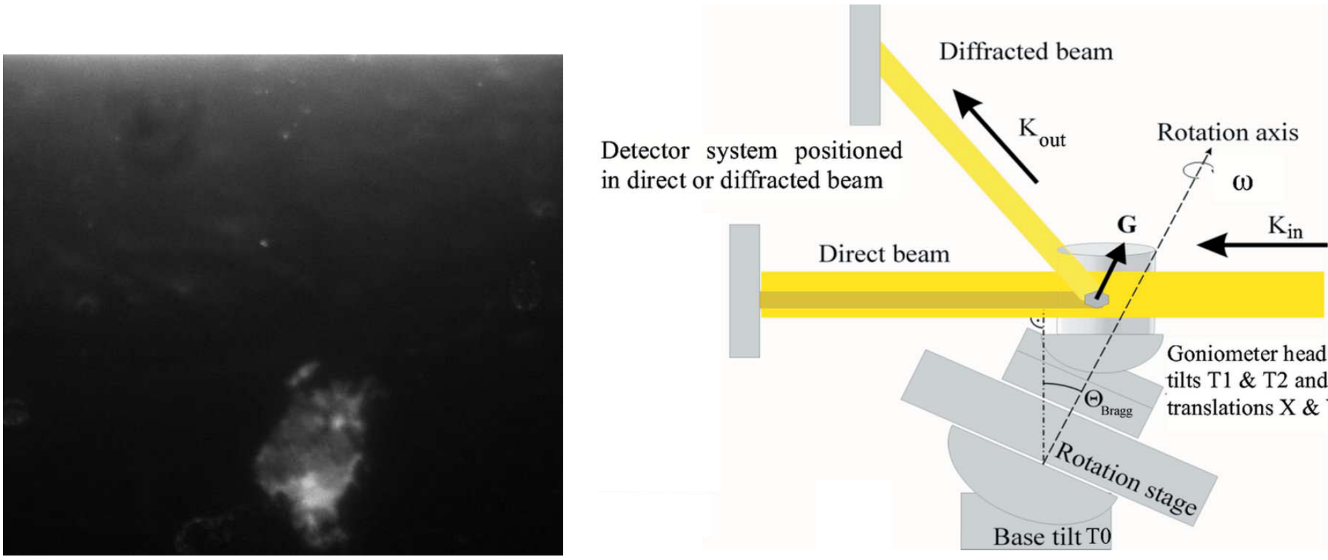

Fig. 5: Radiograph of the grain aligned (left) and scheme of principle of a topotomography setup (right, Ludwig & Al. 2007).

After that time consuming step, we were able to perform several scans. Thanks to NaNox, we have made a sequence of loading the sample then scanning then loading five times. The Al-Li alloy exhibited a PLC (Portevin Le Chatelier like effect and strong lattice rotation with shearing bands (Fig. 6).

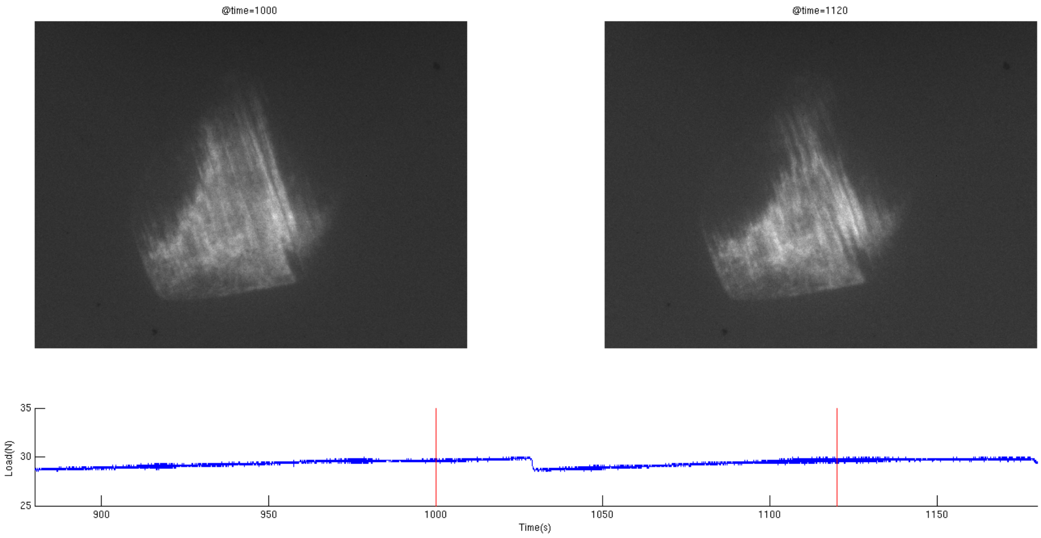

Fig. 6: Two radiographs of the grain of interest before and after the PLC like effect are showed with the evolution of load as a function of time. The red lines represents the time at which the radiographs were taken.