Experimental report – MA5180

Title: In-situ observation of crack coalescence during stress assisted grain boundary oxidation in Ni base superalloy 718 (InnovaXN13)

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Beamline: ID11

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Experimental setup

The experimental setup for this experiment consisted in three different parts.

(i) observation of the crack networks formed during a pilot test of accelerated stress corrosion cracking (SCC) tests under autoclave conditions at Framatome;
(ii) *in-situ* observation of stress assisted grain boundary oxidation (SAGBO) in miniaturized dog-bone samples with different microstructures at 650 ^{o}C;
(iii) characterization of pristine samples for further analysis of SCC accelerated tests at Framatome;

Crack networks from pilot SCC autoclave tests

The samples submitted to the accelerated SCC tests are loaded in a three-point bending configuration. The shape of the sample is rectangular, with approximate dimensions of 0.27 x 5.0 x 18.0 mm. The area subjected to higher tensile loads was cut to allow transmission over a range of 360^{o} to perform Phase Contrast Tomography (PCT) measurements.

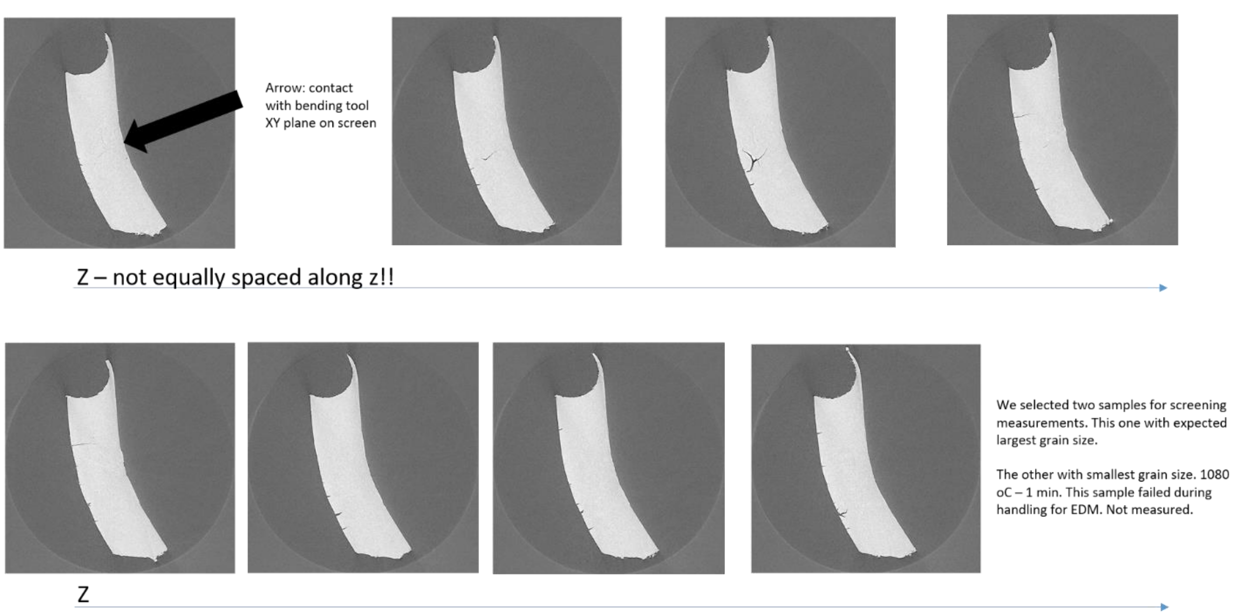

Figure 1 - XY slices of autoclave SCC tests.

The main conclusion from this pilot test was to change the sample dimensions from a rectangle (30 x 4mm) to an elongated dog-bone shape with 0.27 x 1mm cross section) that is still compliant with the autoclave machines, but also compatible with PCT and DCT characterization at 60 keV. Different propagation distances were also evaluated to allow better capturing the cracks without introducing too many streak artifacts in the reconstructions. As these samples were plastically deformed, DCT reconstructions were not successful in this case.

In-situ **SAGBO tests**

The samples were initially vertically aligned when already mounted inside the Nanox load frame at the 3DXRD endstation of ID11. A series of 5 slightly overlapping DCT sub-volumes were acquired in order to ensure coverage of the entire gage section of the sample. A reference PCT scan was acquired to serve as a standard for the quality of the reconstruction performed during the high temperature tests.

The experimental setup for the *in-situ* SAGBO tests is depicted in the Figures 2 and 3. It consists in using the load frame Nanox with an extension shaft to allow the use of furnace placed on top of the sample and allows X ray flux and diffractometer rotation when correctly aligned.

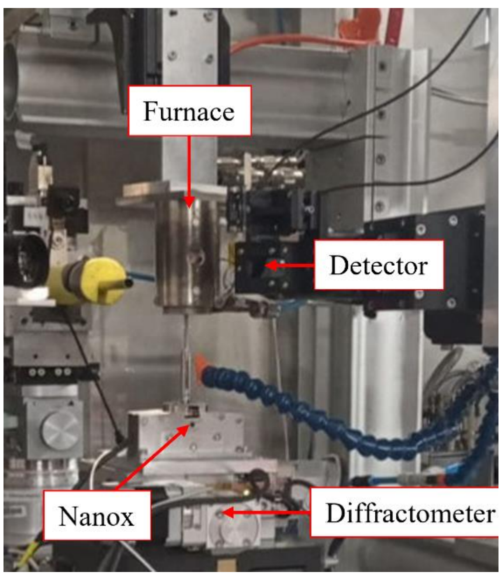

Figure 2 - Experimental setup for SAGBO tests.

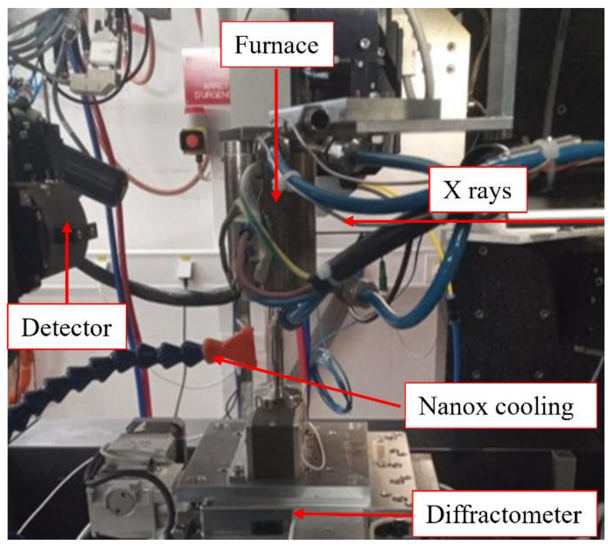

Figure 3 - Detailed view of the SAGBO test with the furnace in position.

The software Nabu, developed at the ESRF, was used to perform the PCT reconstructions. We succeeded to capture the entire process of SAGBO degradation. However, it was noticed that artifacts were introduced by running the tests at high temperature when compared to the reference reconstructions at room temperature. Ring artifacts due to low quality of flat-field correction and error-motion artifacts due to irregular expansion/contraction while the sample rotated were noticed. Also, the noise level was increased at the high temperature tests. These facts were reported in a publication from this experiment (1). In order to account for such errors a more robust data processing pipeline had to be developed including a step of flat-field correction based on Principal Component Analysis (PCA) decomposition and fitting frame by frame to account for the beam profile changes (i.e., monochromator vibrations) during the experiment, implemented internally as described by Jailan *et al.* (2). A projection alignment based on the principle of tomographic consistency was also introduced in the pipeline. Figure 4 depicts the overall type of result obtained by the *in-situ* experiments.

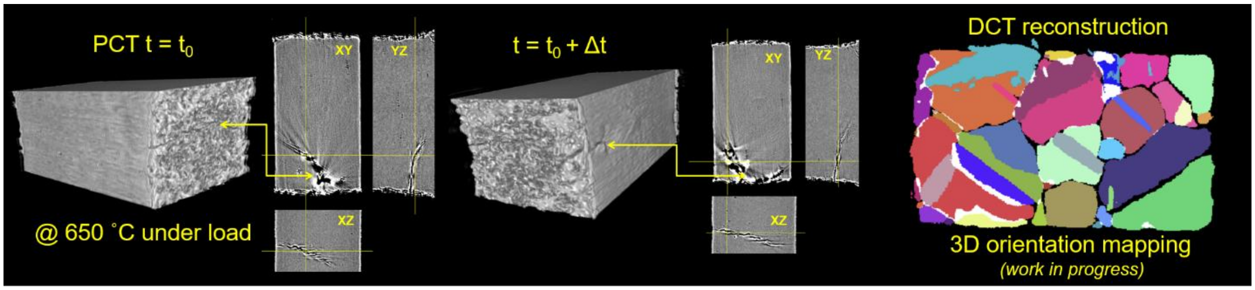

Figure 4 - Representative results from a SAGBO in-situ test.

The aim of these experiments was to use the resulting images as input to a Digital Volume Correlation (DVC) routine to determine the displacement fields within the sample at each step of loading. Overall, the internal contrast of the samples (a second phase precipitated at the grain boundaries) was not enough to enable reliable DVC calculations. Moreover, the grain size was too large, having a dominant grain across the gage length. One of our conclusions is that smaller grain sizes, as well as internal contrast inside the grains would enhance our chances of running good DVC analysis. Figure 5 depicts the dominance of one grain in the cross-section of the sample and presents clear evidence of the grain rotation being the main source of deformation of the sample at the experimental conditions. At the undeformed state (DCT map) the twin boundaries along {111} planes are aligned at an angle of around 49.6^o around the axial loading of the sample. For the uniaxial loading the maximum shear stress will be located at an angle of 45^o with the vertical axis, corroborating the theory of a dominant deformation along such big grain. The angle of the twin boundaries, shown as extinction spots, reach an angle of 54.2^o at the last reconstruction.

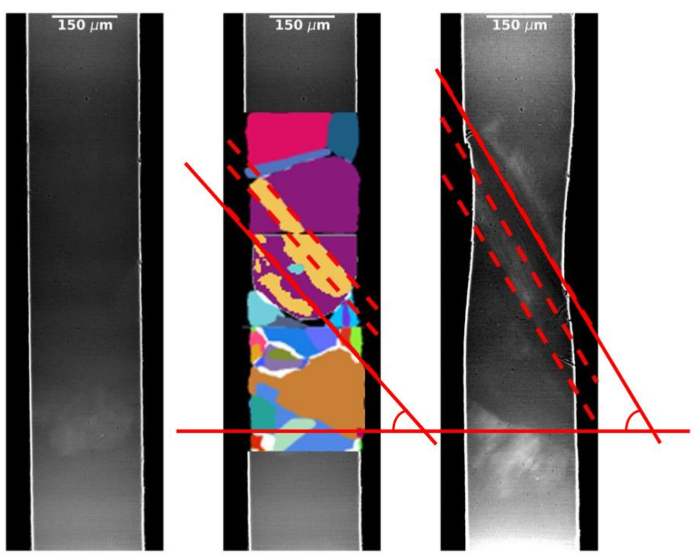

Figure 5 - Combination of DCT slice and projection as evidence of grain rotation during plastic deformation.

Figure 6 shows a vertical slice varying over the time of the experiment and present the clear correlation between the onset of plasticity and the measured load curve as a function of time.

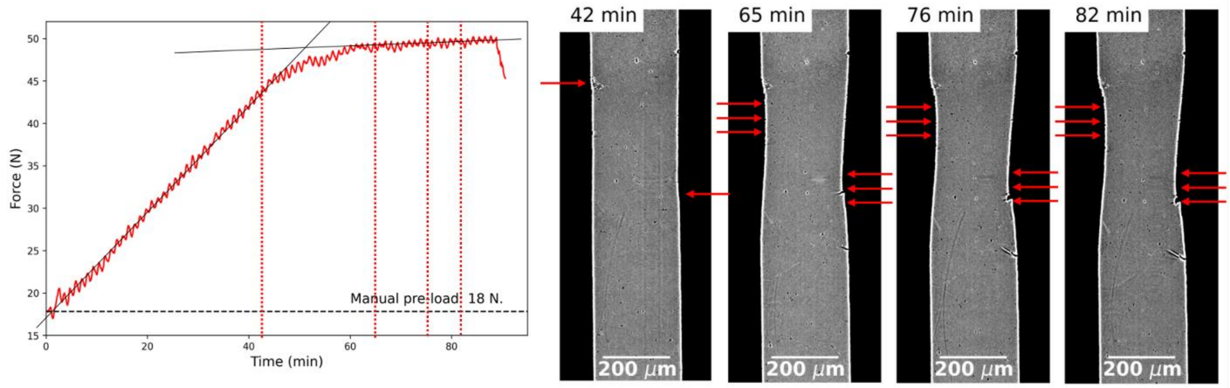

Figure 6 - Load curve as a function of time and the correspondent timesteps at the onset of strain localization.

Characterization of pristine samples for autoclave SCC tests

For this part of the experiment different groups of samples, with varying grain sizes, were grain mapped by DCT. The central part of the samples has been mapped by 7 overlapping DCT scans. Quick reconstructions were performed for the central volume of each sample to confirm data quality and feasibility of grain reconstruction for the different microstructural conditions. The samples were sent to the industrial partner (Framatome) where they are submitted to accelerated SCC tests. Once they are returned to the ESRF they will be scanned again in a similar way to what was shown in the first section of this report and the DCT sub-volumes containing the relevant (damaged) parts of material will be further analysed. Figure 7 shows a preliminary reconstruction from what should be the most difficult sample to reconstruct as it has smaller grain size.

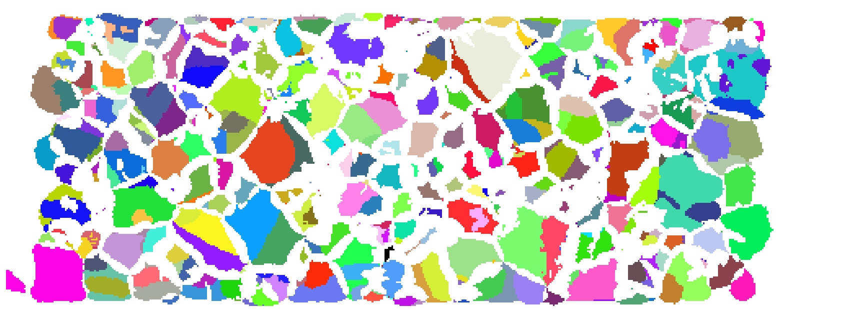

Figure 7 - Preliminary reconstruction of one of the samples sent to accelerated SCC tests at Framatome.

References

1. Resende PD, Viganò N, Bardel D, Réthoré J, Ludwig W. Advanced time-resolved characterization of Stress Assisted Grain Boundary Oxidation of 718 Ni superalloy. IOP Conf Ser Mater Sci Eng. 2022 Jul 1;1249(1):012046.

2. Jailin C, Buffière JY, Hild F, Poncelet M, Roux S. On the use of flat-fields for tomographic reconstruction. J Synchrotron Radiat. 2017;24(1):220–31.