	Experiment title: **Pores volume evolution and damage nucleation during hot rolling: investigation of opening mechanical fields by in situ X-ray nano-tomography high temperature tensile tests.**	**Experiment number**: **MA5083**
Beamline:	**Date of experiment**: from 10th of November 2021 to 13th of November 2021 and from 20th of April, 2022 to 22nd of April, 2022	**Date of report**: 19/09/2022
Shifts:	**Local contact(s)**: Julie Villanova	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

Julie Villanova: ESRF - The European Synchrotron

Anthony Albert Harrup: ESRF/ SIMAP Laboratory University Grenoble Alpes

Pierre Lhuissier: SIMAP Laboratory University Grenoble Alpes

Luc Salvo : SIMAP Laboratory University Grenoble Alpes

Alexandre Barthelemy: C-TEC Constellium

Mélanie Ollat: C-TEC Constellium

Report:

1. Overview

This experiment has as a main objective to research the damage evolution in aerospace alloy Al 2050. Aluminium 2050 is an alloy widely used for aerospace internal structural components because its high properties combined with its particular low density. During the manufacturing process aluminum alloys undergo various thermomechanical steps, such as hot rolling. In particular, hot rolling enables to close the segregation pores that may form during casting. Exploring new process windows for this process may lead to extreme conditions where tensile states and large strain generate pore nucleation and pore growth. It is essential to learn how the pores evolve during similar conditions. The objective of this study is to mimic these extreme states by in situ tensile tests and track the porosity appearing during the deformation.

2. Measurement/Data

The experiment took place in two sessions as during November 2021 there was a beam-loss that translated in losing at least two shifts, that were re-attributed in April 2022. The material scanned at ID16B was aerospace alloy aluminium 2050. The specimens scanned were small cylindrical samples of no more than 400 µm in diameter, especifically designed to meet the requirements of ID16B. The chemical composition of aluminium 2050 aerospace alloy is based on Al-Li-Cu.

The experiment was performed at an energy of 17.4 keV with a high flux of 5.35x10^12 ph/s. During this session at ID16B, *in situ* high-temperature (400-520 °C) interrupted tensile tests were performed on the specimens and imaged at each deformation step with two different resolutions: low resolution, 324nm/pixel, and high resolution with 100 nm/pixel. The two resolutions allow determining the global strain during the deformation steps and tracking the pore nucleation and growth.

Each scan consists in 1003 projections and takes less than 40 seconds to complete. We successfully

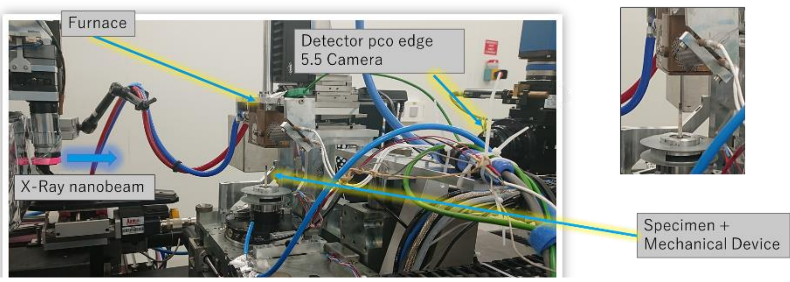

Figure 1 Experimental setup assembled at ID16B. The in house designed furnace. The micro-tensile device was especially designed to fit the rotation stage and the furnace of ID16B [1].

scanned 10 samples during the overall beamtime. This generated a vast database which contains thousands of pores nucleating and growing.

3. Results

The 3D volumes are already all reconstructed and segmented to distinguish the porosities and the intermetallic particles as shown in figure 2a. The main information extracted are: (i) the evolution of the number of porosities vs. strain (figure 2b), (ii) the localization of those pores in relation to the intermetallic particles (figure2 c), (iii) pores shape and morphology evolution (figure 2 d). The analysis of the 3D images using Digital Volume Correlation [2] to track the nucleation and growth of the porosities is still under progress. Finally, local deformation, pore shape and pore morphology will be linked to global strain information and compare with simulation to feed numerical modelling and improve the new hot rolling processes following the workflow described in Gravier et al. article [3].

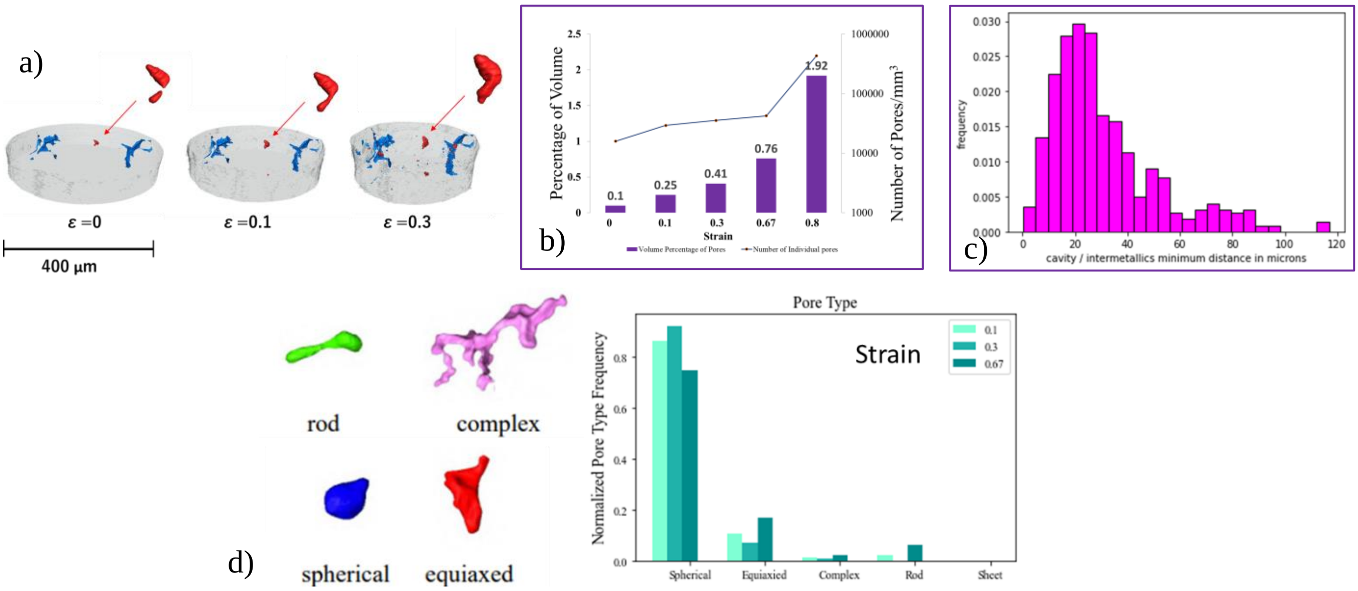

Figure 2 a) Rendered view of a subvolume (pixel size: 324 nm/pixel) in one sample at different deformation steps. Pore is in red and intermetallic particles in blue. b) Evolution of the percentage of porosity volume in a scanned specimen in function of the strain. C) The distance between the intermetallic particles and the porosities in a given strain state. d)Left: Morphology classification of different pores extracted from high resolution images. Right: morphological evolution of the pores for different global strain in the sample.

4. Conclusion

During the shifts awarded for this experiment we performed multiple successful scans. This was possible because of the experimental setup was highly reproducible. The experiments were the main part of communication presentations at multiple conferences: Oral Presentations at: ICTMS 2022 (Grenoble, France), ICAA18 (Toyama, Japan) and ESRF's USER MEETING 2022. These experiments are also the central experimental work for the project InnovaXN XN2019-ESRF04 and will be published in the manuscript of Anthony Harrup's PhD thesis and in papers published in peer-reviewed journals.

5. References

[1] J. Villanova, R. Daudin, P. Lhuissier, D. Jauffrès, S. Lou, C. L. Martin, S. Labouré, R. Tucoulou, G. Martínez-Criado, and L. Salvo: Materials Today, 2017, vol. 20, pp. 354–59.
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[3] P. Gravier, F. Mas, A. Barthelemy, E. Boller, L. Salvo, and P. Lhuissier: Journal of Materials Processing Technology, 2022, p. 117509