ESRF	**Experiment title:** 3D characterization fo the microstructure of an Al alloy design for 3D printing	**Experiment number**: MA5086
Beamline: ID16B	**Date of experiment**: from: 22/04/2022 to: 25/04/2022	**Date of report**: Janvier 2023
Shifts: 9	**Local contact(s)**: Julie VILLANOVA	*Received at ESRF:*

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REPORT

Aims: New Al alloys designed for 3D printing show heterogeneous microstructures from the macroscale scale down the nanoscale with a bimodal grain structure at the melt pool scale: fine equiaxed grains (FEZ: Fine Equiaxed Zone) at the melt pool boundaries, typically < 1µm; and columnar grains at the melt pool center (CZ: Columnar Zone), typically a few microns in width and >10 µm in length. Second-phase intermetallic particles are also heterogeneously distributed at the microscale. Conventional microstructural characterization techniques suffer from artifacts caused by the 2D nature of the information collected.
Our goal was to:

- Achieve a 3D description of the microstructure using nano-XCT: 3D arrangement of the melt pools, 3D distribution of the regions consisting of fine equiaxed grains and columnar grains, quantification of the volume fraction and morphology of the second-phase intermetallic particles.
- Monitor the microstructural evolutions during a heat treatment performed at 400°C.
- Investigate the 3D microscale strain heterogeneities and damage evolution and establish correlations with microstructural features.

Experiments: Two different sets of experiments were conducted to achieve the objectives described above.

(i) Static X-ray nano-tomography with scans collected at 4 different working distances on different microstructures: as-built (As-B), and after different ageing times at 400°C (400°C/1h, 400°C/4h, 400°C/96h). Scans at two different resolutions were performed: low-resolution using a voxel size of 88 nm^3, and high-resolution using a voxel size of 20 nm^3.

(ii) In situ X-ray nano-tomography scans were acquired during room temperature tensile testing of the various microstructures investigated. Micro-tensile specimens were loaded using the NANOX ID11 micromechanical machine modified to fit with ID16B constraints.
Data were reconstructed using the specific reconstruction procedures developed at ID16B which are relatively time-consuming. Image analysis procedures have been specifically developed to post-process the data, in particular, AI-assisted segmentation was employed to recognize regions consisting of fine equiaxed grains from regions consisting of columnar grains. The AI algorithm for recognition of the various regions is based on local grayscale texture. A framework allowing the determination of the microscale strain heterogeneities using Digital Volume Correlation (DVC) is currently being developed.

Results: The results from the static scans were reconstructed and the image analysis procedures were developed and applied to process the data. The distribution of second-phase intermetallic particles within the

FEZ and CZ respectively can be characterized in 3D. Information regarding the morphology, size, and percolation of the intermetallic network can be obtained, see examples in **Figure 1a-b** in the as-built microstructure.

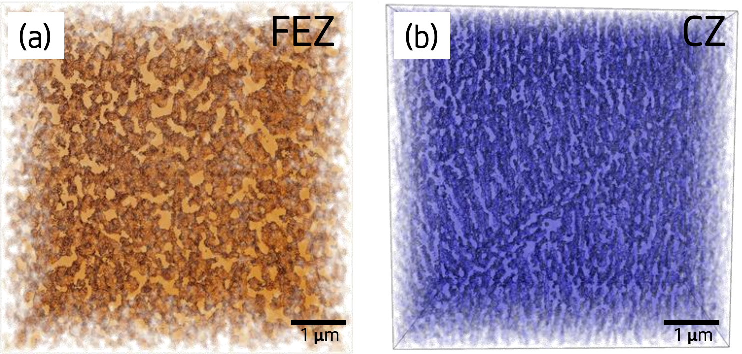

Figure 1. 3D characterization of the second-phase intermetallic in within: (a) the FEZ, and (b) the CZ.

At the macroscale, it is also of interest to get a 3D description of the spatial distribution of the FEZs and CZs because the resulting mechanical behaviour can be strongly affected if one of the region is found percolating in 3D. This idea is of utmost importance because such microstructures can be considered as a composite materials with FEZs and CZs having different mechanical behaviour. The AI-assisted sgementation algorithm, developed specifically in this work, was successfully applied to the as-built microstructure to distinguish FEZs from CZs in 3D images. This is possible because of the high resolution of the nano-XCT scans, see comparison between a SEM-BSE image (**Figure 2a**) and a 2D cross-section extracted from a 3D reconstructed image (**Figure 2b**). An example of the 3D spatial distribution of the FEZs and CZs is given in **Figure 2c**. A similar approach is being developed to be able to process larger 3D images with low-resolution images (voxel size of 88 nm^3).

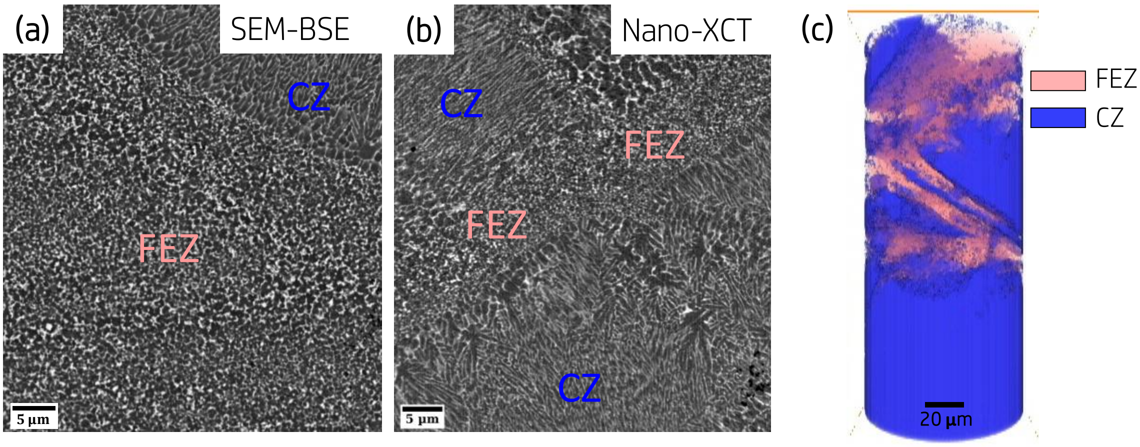

Figure 2. (a) SEM image of the microstructure using the backscattred contrast. (b) 2D cross section extracted from a reconstructed volume obtaned using X-ray nano-tomography (voxel size 25 nm3). (c) 3D view of the distribution regions consisting of fine equiaxed grains (in pink) and columnar grains (in blue) determined using AI-assisted segmentation.

DVC based on the local contrast offered by the microstructure itself is expected to provide new insights into the development of strain heterogeneities. Preliminary results are encouraging. We should thus soon be able to evidence the evolution of the difference in mechanical behaviour between the FEZs and CZs upon ageing at 400°C.

Publications:

The results generated in MA5086 were analyzed by Maxence Buttard (PhD student at SIMaP) and Pierre Lhuissier (CNRS Researcher at SIMaP). Some results were included in the following publication:

[1] Maxence Buttard et al., *Towards an alloy design strategy by tuning liquid local ordering: What solidification of an Al-alloy designed for laser powder bed fusion teaches us,* **Additive Manufacturing** vol 61 (2023) 103313

Two other articles will take advantage of the results collected during MA5086, one of them will be submitted soon while the other one is under preparation.

[2] Maxence Buttard et al., *New insights into the elastoplastic transition of an additively manufactured Al alloy*, **Acta Materialia**, *To be submitted*

[3] Maxence Buttard et al., *3D charcaterization of an additively manufactured Al alloy using X-ray nano-tomography*, *Under Preparation*.