	Experiment Title:	Experiment Number:
ESRF The European Synchrotron	In-situ radiography investigations of defect formation during powder bed fusion additive manufacturing	MA4066
	Date of experiment:	
Beamline: ID19	from: 2018/06/27 to: 2018/06/30	Date of report: May 2019
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REPORT:

Aims:

We have developed a homemade additive manufacturing replicator that allows in situ 3D-characterisation to be performed during layer-by-layer construction using synchrotron X-ray microtomography. The main purpose of this study was to understanding the interaction between a laser and a metallic powder bed in the framework of additive manufacturing by combining two characterization approaches :

- in situ radiography during lasing

- in situ tomography between lasing sequences

Experiments:

A dedicated 3D printing bench as been developed for this purpose with varied build plates size and shape. Despite an elongated rectangular build plate (1,5mm x 10 mm), during the first hours of experiments it was shown that radiography could not allow to see the melt pool. It was mainly due to the important motion of powder. On the other side, the tomography approach was shown to be really promising. We thus just focused on this second approach.

Two different sets of experiments were carried out with the help of our custom-designed additive manufacturing replicator:

(i) building walls made of stacked single tracks with varying the laser power;

(ii) building walls made of adjacent and stacked molten tracks.

Scans before (subsequently to powder deposition) and after local laser melting are acquired for every layer.

The laser system used in the present study was developed by the former PHOENIX company, now known as 3D Systems. It consists of a fibre high-energy laser source, a beam expander and a scanning head. The energy is delivered by a 200W ytterbium fibre laser from IPG (YLR 4x200W SN). A single head was employed. The laser wavelength is 1080 nm and the power can be varied continuously from 20 to 200W.

The custom-designed build chamber includes a layering system operating in a controlled atmosphere compatible with X-ray micro-tomography. The developed system consists of a rotating rake and a build plate mounted on a piston that can move up and down along the build direction. An accurate position of the piston is ensured by a home-made micromechanical device. The position of the build plate is controlled at $\pm 5\mu$ m and a maximal displacement rate of 0.5mm/s can be achieved. The build plate mounted

on a piston can be moved away from its raking position: up to 10mm downwards allowing new layers to be built, and up to 20mm upwards to be in a "shadow-free" position to perform in-situ microtomography subsequently to key steps of the layer-by-layer building process.

An indirect X-ray image detector, the so-called TripleMIC (OptiquePeter, Lentilly, France) was used: consisting of a scintillator lens-coupled (Mitutovo long-working distance objectives) to an sCMOS camera (type: pco.edge 5.5, PCO AG, Germany). Several magnifications can be achieved using a high-energy Xray beam. Triple-mic enables rapid switching between the objectives without the need for focus adjustment and with a scintillator optimised for each magnification. In this feasibility study, magnifications of 2, 5 and 10 times have been tested, resulting respectively in a pixel size of 3.64, 1.46 and 0.73µm. However, in this work, image acquisitions were only made with the two-times magnification. Thus, all the reconstructions shown here, have a voxel size of 3.64 µm. Note that all the images were acquired using the absorption contrast. The scintillator was a 250 µm thick LuAg. The experiments were conducted with a ring current of 200 mA. A pink beam was employed using a W150 wiggler with a 40 mm of gap of filtering with 1.4 mm of diamond, 1.4 mm of copper, and 5.6 mm of aluminium. This results in a peak energy of about 78keV and a mean integrated detected energy of roughly 95keV. Beam conditions used in this work would also be suitable for heavier metals such as Ni-alloys or steels for a similar building area (build plate diameter of 6mm). The distance between the sample and the detector was about 150 mm (this can be reduced down to about 100 mm). Exposure time was set to 20ms. 1500 projections were acquired per scan resulting in a scan time of 45s including reference images. A region of interest (ROI) of 2440 x 1700 pixels which results in a field of view of 8.8 x 6.2mm was used. Data were reconstructed using the ESRF fasttomo3 preprocessing and PyHST2 routines using classical filtered back-projection algorithms. Post-reconstruction ring removal was applied using the in-house ESRF-Matlab routine. Data were cropped and converted to 8bits using fixed ROI and gravscale range per sample. Post-processing was conducted using Fiji and inhouse plug-ins.

Results:

We have successfully built walls made of stacked single tracks with varying powers as shown in **Fig 1**. A specific procedure named "virtual powder removal" was developed to only reveal the molten tracks embedded in the powder bed during the process. Such experiments not only validate our approach, but also allow molten track morphology, and internal defect to be monitored when building successive layers. Typical defects inherent to powder bed additive manufacturing such as lack-of-fusion pores have also been revealed when building walls made of adjacent and stacked molten tracks when insufficient overlapping between fusion beads were applied. Possible healing of pores can also be evidenced, see example in **Fig 2**. More illustrative examples can be found in [1].



Fig 1. 3D view of the walls made of stacked single tracks built by laser additive manufacturing using various laser powers. 3D data have been segmented to virtually remove particles of the powder bed. The laser power is increased: P1 = 50W, P2 = 100W, P3 = 125W, and P4 = 150W while the laser scanning speed is kept constant (200mm/s) and the layer thickness set to 100µm (defined here as the powder layer height).



Fig 2. 3D view of defects observed at layer two (a) and which are partially healed at layer three (b) when building a wall made of two adjacent melting tracks. 3D data have been segmented to virtually remove particles of the powder bed.

Valorisation:

The results generated in MA4066 were preliminary filtered by Camille Maestre (Master student from PHELMA) and further analyzed by Pierre Lhuissier (CNRS Researcher at SIMaP). Materials and methods are described in details in a scientific article *under revision* and submitted to *Additive Manufacturing* [1]. Preliminary results to demonstrate the feasibility of our approach are included in our publication *under revision*.

Communications have also been given to present the approach to the research and industrial community of additive manufacturing.

The proposed approach as been pointed out as a key strategy by the 2 french research networks (GDR and GIS) dedicated to additive manufacturing.

[1] Pierre Lhuissier, Xavier Bataillon, Camille Maestre, Julien Sijobert, Elodie Cabrol, Philippe Bertrand, Elodie Boller, Alexander Rack, Jean-Jacques Blandin, Luc Salvo, Guilhem Martin. *In situ 3D X-ray microtomography of laser-based powder-bed fusion (L-PBF) - A feasibility study*, **Additive Manufacturing** (2020), *under revision*.