ESRF	**Experiment title: Microstructure evolution due to solid-state thermal cycling during AM of 316LSS**	**Experiment number**: ME-1578
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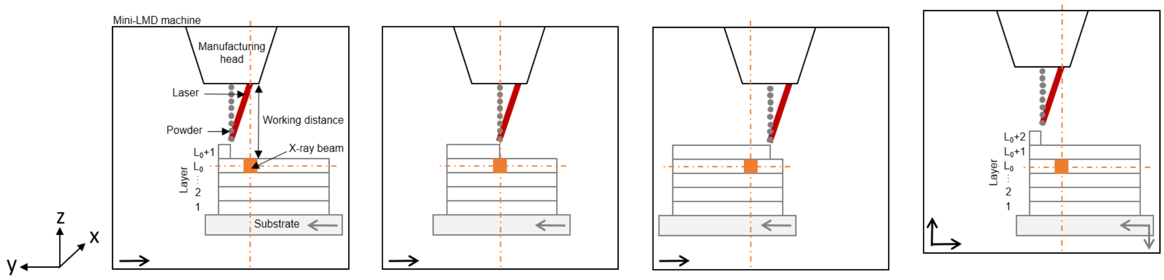
Report: Investigations were conducted with a new miniature Laser Metal Deposition (mini-LMD) additive manufacturing (AM) machine, based on the the directed energy deposition technique, designed and developed by the group of the proposer and dedicated to synchrotron diffraction and radiography experiments. The machine and its equipment (cooling system, powder filtration system, laser, etc.) had been brought and installed in the experimental hutch of the ID31 beamline at ESRF without any problems. The setup was used to study microstructure evolution of an austenitic 316L stainless steel during additive manufacturing via in-situ powder diffraction as well as residual stresses at the end of each building at room temperature. The experimental procedure was as follows : i) define a layer to investigate (e.g., the 20th) and position the mini-LMD machine in order to investigate it (this involves fixing the distance between the substrate and the X-ray beam), and ii) build the wall up to a total of 100 layers. During building, the substrate holder is moved down after addition of each layer in order to keep the working distance between the built wall and the nozzle (laser and powder). Because of this, to keep the same distance between the substrate and the X-ray beam in order to study the defined layer, the mini-LMD machine was moved up after each layer addition with the Heavy Duty Micro Diffraction (HDMD) instrument available at the beamline.

The initial measurement strategy proposed, involved fixing the distance between the heat source (laser) and the X-ray beam along the printing direction to study different positions in the wall during the building, has been changed. The ID31 scientist in charge, Veijo Honkimaki, proposed to move the machine with the HDMD instrument in the opposite direction with respect to the motion of the substrate holder (perpendicular to the optical axis on horizontal plane). Figure 1 shows a schematic representation of the motions during the addition of one layer. The X-ray beam position is fixed in space and in time during the experiment. The substrate is moved along the y direction in order to print one layer while the entire machine is moved in the opposite direction allowing to study an area instead of a single position (Figure 1 a, b and c). Then, after the layer addition, the substrate is brought back to its original position but is moved down to keep the same working distance between the manufacturing head and the latest added layer. In order to keep the X-ray beam at the same layer (i.e., L_0), the entire machine is moved up by a layer thickness distance (Figure 1 d). These steps are repeated until the end of the experiment. This strategy allowed to obtain much more information from each building sequence than the initial one, and with a better time resolution while reducing the measurement times and data quantity. In addition, the mini-LMD machine was connected with the computers available at the control hutch (with the help of beamline and ESRF staff) allowing to synchronize the data acquisition with the machine. This allowed to stop the acquisition during dead times and reduce the quantity of data collected.



a) b) c) d)

Figure 1: Schematic representation of substrate/wall and mini-LMD machine motions during the experiments. The X-ray beam position is fixed in space and time. The substrate is moved along the y direction to add one layer, while the entire machine is moved in the opposite direction allowing to scan an area starting in front and ending behind the laser

To conduct the study, the high-resolution monochromator setup available at ID31 was used to provide a beam with an energy of 77 keV. Pilatus 3X CdTe 2M detector located at 0.8 m was used for the investigations. The machine was fixed to the HDMD instrument with the help of the beamline staff. Argon gas was provided in enough quantity for the experiments.

Figure 2 shows an example of the 111 scattering vector evolution during additive manufacturing of an austenitic 316L stainless steel. The 20th layer (L_0) was followed during this experiment, thus 80 layers were added on top (L_0+N where N_{max} = 80). Few of them are presented in Figure 2 (N = 1, 5, 10, and 20). Considering layer L_0+1, the scattering vector remains constant up to 1-2 mm in front of the laser. Then, close to laser position, a strong decrease of the scattering vector is observed because of the temperature increase. Then, the scattering vector continuously increases with the temperature decrease behind the laser. The sharp change observed close to position 0 mm, is due to the melt pool (not crystalline), which was illuminated by the beam. However, the scattering vector measured at this position is related to the un-melted feedstock (powder) going through the beam. Similar behaviour is observed for the next added layers but with lower intensities because of the highest distance between the investigated layer and the melt pool (i.e., laser position in the vertical axis).

These preliminary results show solid-state thermal cycling occurring during additive manufacturing. In a first approximation it can be assumed that the scattering evolution (i.e., lattice spacing of the austenitic phase) is solely due to thermal expansion and contraction. Thus, high temperature rates are observed close to the laser position and they decrease with the addition of layers. The maximum temperature reached also decrease with the number of added layers. Behind the laser (positive distance), temperature decreases but remains higher than the room temperature. However, the scattering vector is similar at the beginning of each new added layer and similar to the one measured at room temperature, showing that the sample had time to reach room temperature between two layers addition. These evolutions will induce thermo-mechanical driving forces that can trigger a plethora of mechanisms such as precipitation, dislocation dynamics, etc., which result in microstructural changes via evolution of phase fraction, lattice strain, and dislocation density for this austenitic 316L stainless steel.

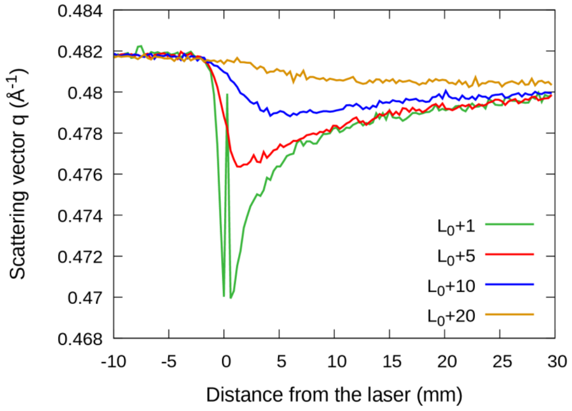


Figure 2: Scattering vector evolution of the 111 plane during AM of a austenitic 316L stainless steel as function of the laser position and added

In order to go further in the investigations, interplanar spacing distances, stresses and dislocation density will be studied during in situ experiments. Results, will be compared and discussed with residual stress measurements acquired at room temperature after each building.

The aim of the proposed experiment has been successfully achieved during the allocated beam time. The results will lead to a publication on the impact of SSTC during additive manufacturing. In addition, the experiments conducted have shown the feasibility to install and use without strong difficulty our developed mini-LMD machine at the ID31 beamline. This, is encouraging to study more complex material systems such as titanium alloys, which we will do in the upcoming beamtime. The higher complexity of such alloys comes from the high reactivity of titanium alloys with oxygen (experiments must be conducted under inert atmosphere) and solid phase transformations (e.g., β to α during the cooling) occurring during SSTC.