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| ESRF | **Experiment title:**<br>**Study of solid-state phase transformations during additive manufacturing of Ti6Al4V** | **Experiment number**:<br>ME-5222 |
| **Beamline**:<br>ID31 | **Date of experiment**:<br>from: 22/07/2022 to: 27/07/2022 | **Date of report**:<br>01 March 2023<br><br>*Received at ESRF:* |
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**Report:**

Investigations were conducted with a miniaturized laser metal deposition (mini-LMD) additive manufacturing (AM) machine, based on the directed energy deposition technique, designed and developed by the group of the proposer and dedicated to synchrotron diffraction and radiography experiments. The machine had been brought and installed on the heavy duty micro diffraction instrument available in the experimental hutch of the ID31 beamline at ESRF without any problems. Its auxiliary equipment (cooling system, powder filtration system, laser, etc.) was positioned around in the experimental hutch. The laser of the machine was connected to the interlock system for safety. Argon gas used to inert the printing chamber and protect the melt-pool from oxidation was supplied in enough quantity by the beamline. The setup was used to study the microstructure evolution of a titanium alloy (Ti6Al4V) 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 20$^{th}$) 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) continuously build the wall up to a total of 100 layers and tracked the microstructure evolution at the aforementioned position. This position was kept fixed during the experiment by keeping the substrate stationary and moving only the focusing head of the mini-LMD machine to print the samples. Different printing strategies as well as processing parameters (power, and speed) were used. In addition, the laser has been used to perform heat treatment between each added layer with different energies. Experiments were performed with a photon energy of 75.022 keV and beam size of 300 × 300 µm$^2$. The Pilatus3 X CdTe 2M was positioned at ~ 1 m from the sample and was used with an acquisition rate of 250 Hz. The high-frequency acquisition rate permitted the precise investigation of rapid phenomena (e.g., solidification and solid-state phase transformation) occurring during the AM process. Despite the high acquisition rate, a good signal-to-noise ratio was achieved thanks to the synchrotron upgrade. In order to reduce the amount of data, the start of the recording with the detector was triggered when the laser was turned on.

Figure 1 shows the evolution of the intensity of different diffraction peaks over time and scattering angle ($2\theta$) during the printing of the first layer out of 100 layers. Time 0 s was set when the material has just been deposited, i.e., when the laser position overlaps the investigated zone. At that time, the material is liquid (the amorphous pattern is hardly visible in the figure). The solidification process begins ($t$ ~ 0.1 s) with the nucleation of β phase and finishes at $t$ ~ 0.15 s. Then, the temperature decreases and α' transformation begins at $t$ ~ 0.6 s and finishes at $t$ ~ 1 s. Finally, the investigated area cools down until the next added layer on its top not presented here. While not visible, a low fraction of β phase is retained after cooling at room temperature.

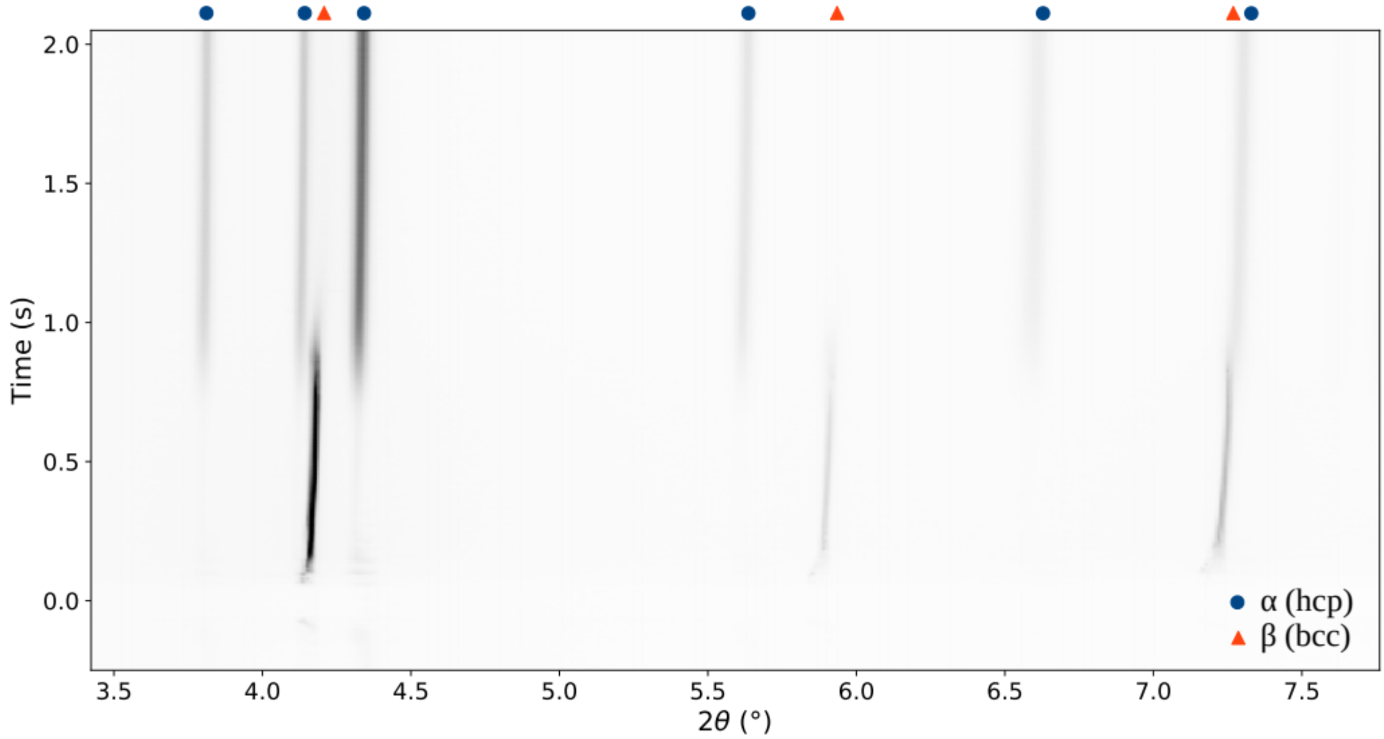


Figure 1: Evolution of the diffraction intensity of different diffraction peaks over scattering angle with a recording rate of 250 Hz showing the liquid to β to α phase transformation over time during the printing of the first layer out of 100 layers of a Ti6Al4V alloy.

In order to go further in the investigations, interplanar spacing distances, phase fractions, stresses, as well as dislocation density are currently being studied. Results will be confronted with complementary experimental analysis such as SEM/TEM and mechanical tests. In addition, the thermal and mechanical contributions and temperature changes that occur during the printing process, as well as the residual strains, will be studied and used to validate a thermo-mechanical model developed in our laboratory. These investigations and results are part of a Ph.D. thesis involved in the ERC project. The aim of the proposed experiment has been successfully achieved during the allocated beam time. The results will lead to publications on the impact of SSTC during additive manufacturing. With these experiments, we will be able to understand the evolution of the microstructure of Ti6Al4V during AM and laser thermal treatment. Throughout the beamtime, the beamline scientist and technician were patient and always available, and we are grateful for their support.