EUROPEAN SYNCHROTRON RADIATION FACILITY

INSTALLATION EUROPEENNE DE RAYONNEMENT SYNCHROTRON

ESRF

Experiment Report Form

The double page inside this form is to be filled in by all users or groups of users who have had access to beam time for measurements at the ESRF.

Once completed, the report should be submitted electronically to the User Office via the User Portal:
https://wwws.esrf.fr/misapps/SMISWebClient/protected/welcome.do

Deadlines for submission of Experimental Reports

Experimental reports must be submitted within the period of 3 months after the end of the experiment.

Experiment Report supporting a new proposal ("relevant report")

If you are submitting a proposal for a new project, or to continue a project for which you have previously been allocated beam time, you must submit a report on each of your previous measurement(s):

- even on those carried out close to the proposal submission deadline (it can be a *"preliminary report"),*
- even for experiments whose scientific area is different form the scientific area of the new proposal,
- carried out on CRG beamlines.

You must then register the report(s) as "relevant report(s)" in the new application form for beam time.

Deadlines for submitting a report supporting a new proposal

- 1st March Proposal Round - **5th March**
- 10th September Proposal Round - **13th September**

The Review Committees reserve the right to reject new proposals from groups who have not reported on the use of beam time allocated previously.

Reports on experiments relating to long term projects

Proposers awarded beam time for a long term project are required to submit an interim report at the end of each year, irrespective of the number of shifts of beam time they have used.

Published papers

All users must give proper credit to ESRF staff members and proper mention to ESRF facilities which were essential for the results described in any ensuing publication. Further, they are obliged to send to the Joint ESRF/ ILL library the complete reference and the abstract of all papers appearing in print, and resulting from the use of the ESRF.

Should you wish to make more general comments on the experiment, please note them on the User Evaluation Form, and send both the Report and the Evaluation Form to the User Office.

Instructions for preparing your Report

- fill in a separate form for each project or series of measurements.
- type your report in English.
- include the experiment number to which the report refers.
- make sure that the text, tables and figures fit into the space available.
- if your work is published or is in press, you may prefer to paste in the abstract, and add full reference details. If the abstract is in a language other than English, please include an English translation.

ESRF	**Experiment title:** Residual stress-free Laser Additive Manufacturing	**Experiment number**: MA 5229
Beamline:	**Date of experiment**: from: 27/06/2022 to: 03/07/2022	**Date of report**: 12/09/2022
Shifts:	**Local contact(s)**: HONKIMAKI Veijo	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

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Report:

Research Background:

Laser Powder Bed Fusion (L-PBF) is a technology with a large number of potential applications for aerospace, energy, and biomedical sectors. The adoption of this manufacturing technique has been hampered by the poor performance of certain components, originating from appreciable residual stresses introduced by solidification cycling. For steels that display the phenomena (e.g. M300 steel), a volume expansion associated with displacive phase transformations (martensitic transformation) provides a potential solution to mitigate residual stress development during initial manufacturing. Herein synchrotron X-ray diffraction in ID31 is used to perform a three-step XRD study to understand the complex relationship between residual stress development and phase transformations in this manufacturing process. Both ex-situ and in-situ synchrotron XRD on steels manufactured by L-PBF to elucidate and quantify the mechanism of residual stress reduction by phase transformations. So, a three-step study using synchrotron XRD is completed: (1) mapping the residual stress of as-built samples under different scanning speeds; (2)mapping the residual stress of pre-heat samples, to investigate how the cooling rate can modify the residual stress by manipulating the martensite phase volume fraction and the martensite phase transformation process; (3) study the phase transformation in-situ under very fast heating cooling rates.

The large amount of XRD data collected is still under analysis. More work will be finished and published at the end of 2022.

Experiment completed:

For the no-preheat printing, there are 4 samples fabricated with the same parameters. A total of eight thick cuboid samples were removed from the baseplate and half of the samples were sliced into 2 mm thickness samples. The choice of 2 mm is based on the pre-test in March 2022, a trade-off between the fast cooling rate

and enough material for induction heating. The other half of the thick samples were just removed from the baseplate to study residual stress relief during the removal of the baseplate. The as-built thin sample (2 mm) and one thick sample (10 mm) were left still attached to the baseplate, for the residual stress mapping. The pre-heat samples were manufactured using a relatively small baseplate.

During the residual stress measurement process, the pencil beam (with a beam energy of 75 KeV) was used for the experiment. L-PBF samples are printed layer-by-layer, which might cause residual stress to varying in different layers. So, a relatively small beam size (70*120 μm) was selected when mapping the residual stress distribution. However, in the in-situ fast heating/cooling experiment, only one position was monitored throughout the cycle. So a macroscopically uniform area was preferred using a beam size of 172*172 μm.

As shown in Figure 1 a), the diffraction signal (Debye-Scherrer patterns) was be collected by a PILATU3X detector. The induction furnace (the black box with an induction loop) allows a fast heating rate of ~100 °C/s and a cooling rate of ~20 °C/s. The induction loop is made of a Cooper pipe and keeps cooling with water flowing inside. During the experiment, the blue pipe was used to deliver Argon flow. There are different Argon flow rates for the heating and cooling process. A low flow rate was used during heating just to prevent the samples oxidizing in air. By contrast during the cooling process, an extra large flow (~40 bar) was employed to flush the surface of the sample and accelerate the cooling rate. The extra flow is controlled by an automatic valve, which is synchronous with the cooling control program. Based on the pre-test in March, the induction loop was located at the same height as the thermocouple, which makes sure the induction furnace worked well. The loop was 2 mm lower than the beam (the observation point), ensuring the diffraction signal was not blocked by the loop.

For residual stress mapping, a larger motor in the y direction was used, and continuous scanning used to accelerate the scanning speed. The experimental setup is shown in Figure 1 b). The mapping is based on the central position of the samples, which is determined by coarse scanning in y direction and gives redundancy in every edge. A coarse scanning scheme was applied to the whole sample with fine scanning in the edge area, where a larger residual stress gradient might be expected.

Because of the large penetration of synchrotron X-rays, identifying the stress free lattice spacing d0 is a challenge in contrast to lab XRD. (d-spacing of stress-free samples). In this project, comb samples and powders were both used to determine the d0. In order to calibrate the temperature, a pure iron sheet of the same geometry was heated and cooled in the induction furnace.

Preliminary result and future work plan:

The profile peaks, (position, intensity, and full width at half-maximum) of the SXRD data are being evaluated, allowing quantification of both the extent and temperature of the phase transformation and the induced plasticity and residual stresses. An interesting result is shown in Figure 2, two samples cut from one as-built M300 sample show a very different Austenite reversion temperature. That indicates the L-PBF samples are not uniform, which should be considered carefully during the residual stress mapping analysis.

The integrated d-spacing distribution of the as-built samples under 800 mm/s scanning speed is shown in Figure 3. The d-spacing near the surface area is different and shows fluctuation. The fine scanning data near the edge area is being analysed in order to map the residual strains there. Subsequently we will cake and analyse the full Debye Scherrer cone to determine the residual stress in different orientations.

A very large amount of data was collected. Currenetly the residual stress distributions for all the as-built and preheated samples are being calculated and compared to understand the influence of scanning speed and cooling rate. Following this, the in-situ data will analysed to study the mechanism of residual stress reduction by phase transformations.

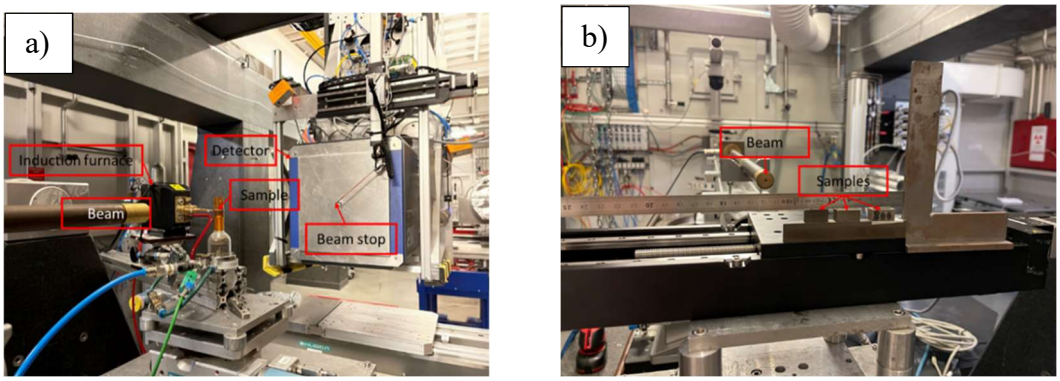

Figure 1 Experimental setup for a) in-situ fast heating cooling test; b) residual stress mapping

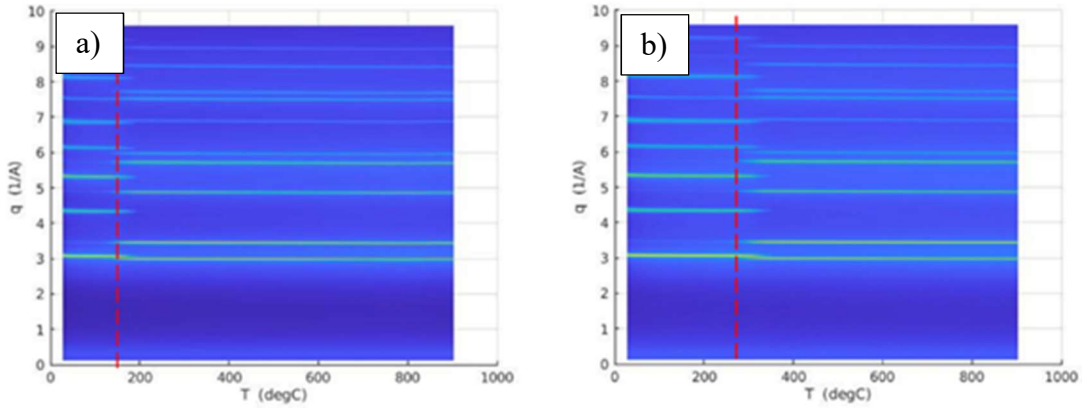

Figure 2 The phase transformation recorded during the fast heating process in different samples cut from one as-built L-PBF M300 under fabricated at 800 mm/s scanning speed. a) sample #1; b) sample #2;

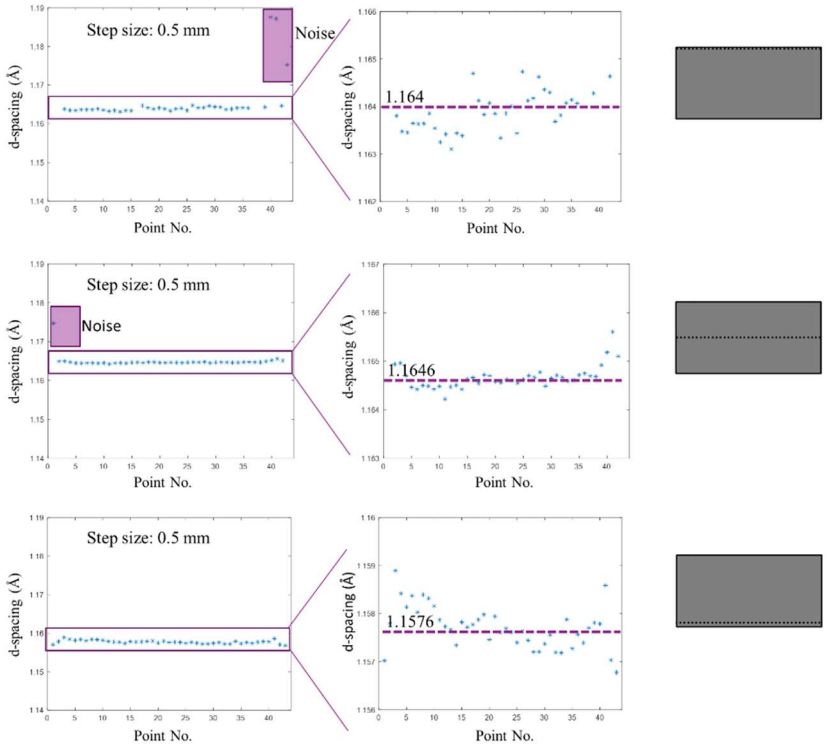

Figure 2 Integrated d-spacing distribution for the as-built samples under 800 mm/s laser scanning speed