EUROPEAN SYNCHROTRON RADIATION FACILITY

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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

Reports supporting requests for additional beam time

Reports can be submitted independently of new proposals – it is necessary simply to indicate the number of the report(s) supporting a new proposal on the proposal form.

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.

Deadlines for submission of Experimental Reports

- 1st March for experiments carried out up until June of the previous year;
- 1st September for experiments carried out up until January of the same year.

Instructions for preparing your Report

- fill in a separate form for each project or series of measurements.
- type your report, in English.
- include the reference number of the proposal 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:** In situ characterisation of additive manufactured AlSi10Mg post-processed via FSP	**Experiment number**: MA3758
Beamline: ID11	**Date of experiment**: from: 13/9/17 to: 18/9/17	**Date of report**: 7/9/20
Shifts: 15	**Local contact(s)**: Wolfgang Ludwig	*Received at ESRF:*

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Report:

Additive manufacturing is an increasingly popular technique thanks to the design freedom it offers, coupled with the time and material saving potential. Applications of metal AM already exist in highly competitive industries, such as aerospace. However, the use of metal AM for structural purposes is still to become a reality. The AM parts present features that are currently directly related to the manufacturing process and render them unattractive when aiming for outstanding mechanical performance. Porosity, heterogeneity or residual stress are examples of these specific traits that lead to underperformance in mechanical behaviour, specially in fatigue resistance.

Friction stir processing could be used as a postreatment technique to bring a solution to these issues. Derived from friction stir welding, this method consists of using a non-consummable rotating cylindrical tool comprised of a shoulder and a pin that is introduced and traverses the material to treat, producing a surface local thermomechanical effect. It has been proven on cast material that the porosity reduction, microstructural refinement and homogeneisation FSP brings translates in an improvement of mechanical performance, particularly ductility and fatigue resistance.

In order to get a profound understanding of the changes brought about by FSP on a typical AM material, insitu tensile and ex situ fatigue (f=20 Hz, R=0.2, F_{min}=25 N, F_{max}=125 N) tests were performed on the ESRF ID11 beamline at 0.75 µm resolution. These experiments help in the understanding of the damage mechanisms involved and their modification after FSP.

The samples were machined from plates manufactured through selective laser melting (SLM) or casting, the latter serving as reference. Several conditions were tested (see table 1), including different SLM build platform temperatures (35°C and 200°C), as built (AB) or stress relief heat treated (SRHT) SLM samples (i.e. 300°C, 2h), different orientations (all samples are oriented horizontally with respect to the SLM build platform except for the samples labelled "Vertical" that are oriented vertically) or different number of FSP tool passes.

Table 1. In situ tensile and ex situ fatigue tests samples

In situ tensile	**Ex situ fatigue**
SLM 35°C AB	
SLM 35°C AB Vertical	
SLM 35°C FSP 1 pass	SLM 35°C FSP 1 pass
SLM 35°C FSP 3 passes	SLM 35°C FSP 3 passes
SLM 35°C FSP 6 passes	SLM 35°C FSP 6 passes
SLM 35°C SRHT	SLM 35°C SRHT
SLM 35°C SRHT Vertical	
SLM 200°C AB	
SLM 200°C FSP 1 pass	SLM 200°C FSP 1 pass
SLM 200°C FSP 3 passes	
SLM 200°C FSP 6 passes	
As cast	
Cast FSP 1 pass	
Cast FSP 3 passes	
Cast FSP 6 passes	

The obtained tomography images offer information on the following microstructural features: Initial porosity, Si-rich eutectic particles, Fe-rich impurities, cavities originating with the progressively applied load. This information allows for determination of porosity evolution, nucleation, growth and coalescence of cavities, particle tracking, etc.

These different features can be observed on figure 1. Since the microstructure of cast samples is coarser, it can be easily appreciated on a 2D slice of the tomography volume. On the other hand, porosity is readily detected on the SLM 35°C AB samples, but the Si-rich eutectic is too fine to be seen. After 6 passes of FSP, porosity is virtually eliminated (at least at the tomography resolution level), while the Si-rich eutectic has experienced globularisation and is now observable.

On figure 2 the initial porosity on the central part of the SLM 35°C AB and SLM 35°C FSP 1 pass in situ tensile samples can be observed. The initial porosity volume decreases from 0.15% to 0.024% after 1 pass of FSP. Furthermore, the largest pores, which are more critical for mechanical behaviour, are eliminated.
The negative influence of Fe-rich impurities on mechanical behaviour and the effect of clustering can be appreciated on figure 3. After 1 pass of FSP on the SLM material the porosity has been greatly reduced while keeping the microstructure fineness. However, more FSP passes (or variations in other FSP parameters) are needed to complete the breakdown and redistribution of Fe-rich impurities. Further tuning of FSP parameters is needed to reach an equilibrium between excessive globularisation of the microstructure and insufficient dispersion and refinement of Fe-rich particles.

The ex situ fatigue tests also yield very interesting results. 11 tomography images were obtained on the SLM 35°C SRHT fatigue sample, the first before loading (Figure 4 left) and the rest after a certain amount of cycles during the test. Figure 4 centre and right respectively depict the Z-projection of the 6th and 10th tomography volumes. Thanks to this test, the origin of the main fatigue crack could be linked to the presence of a critical pore close to the surface.

The observation of all the phenomena occurring on these tests allows to reach a profound understanding of the damage mechanism nature and its alteration through the thermomechanical effect of the FSP technique. This in turn leads to the possibility of optimising and tayloring the process.

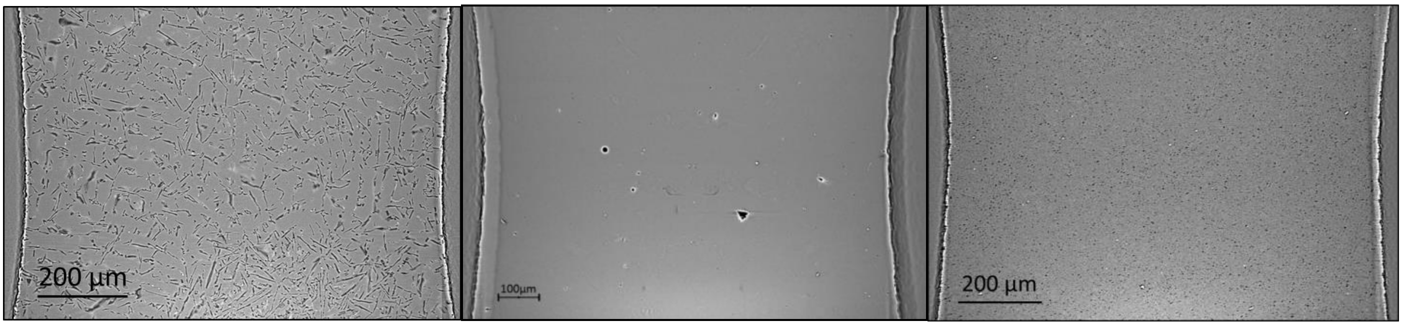

Figure 1: As cast (left), SLM 35°C AB (centre) and SLM 35°C FSP 6 passes (right) samples

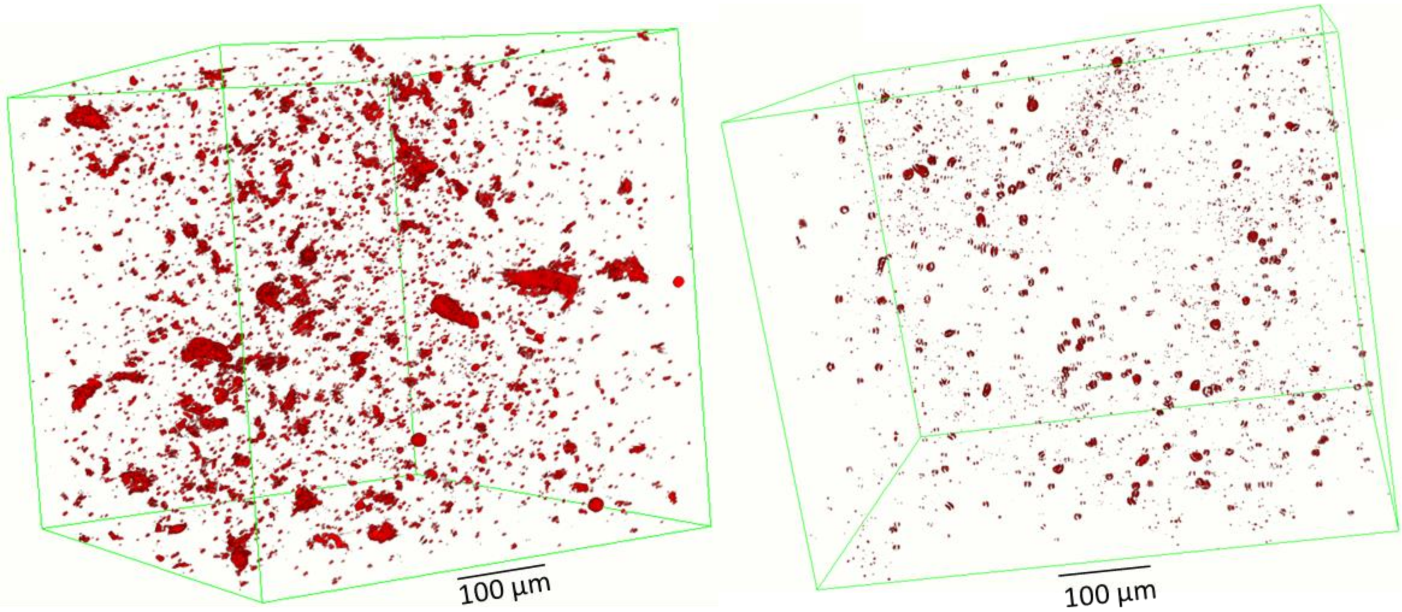

Figure 2: Initial porosity on SLM 35°C AB (left) and SLM 35°C FSP 1 pass (right)

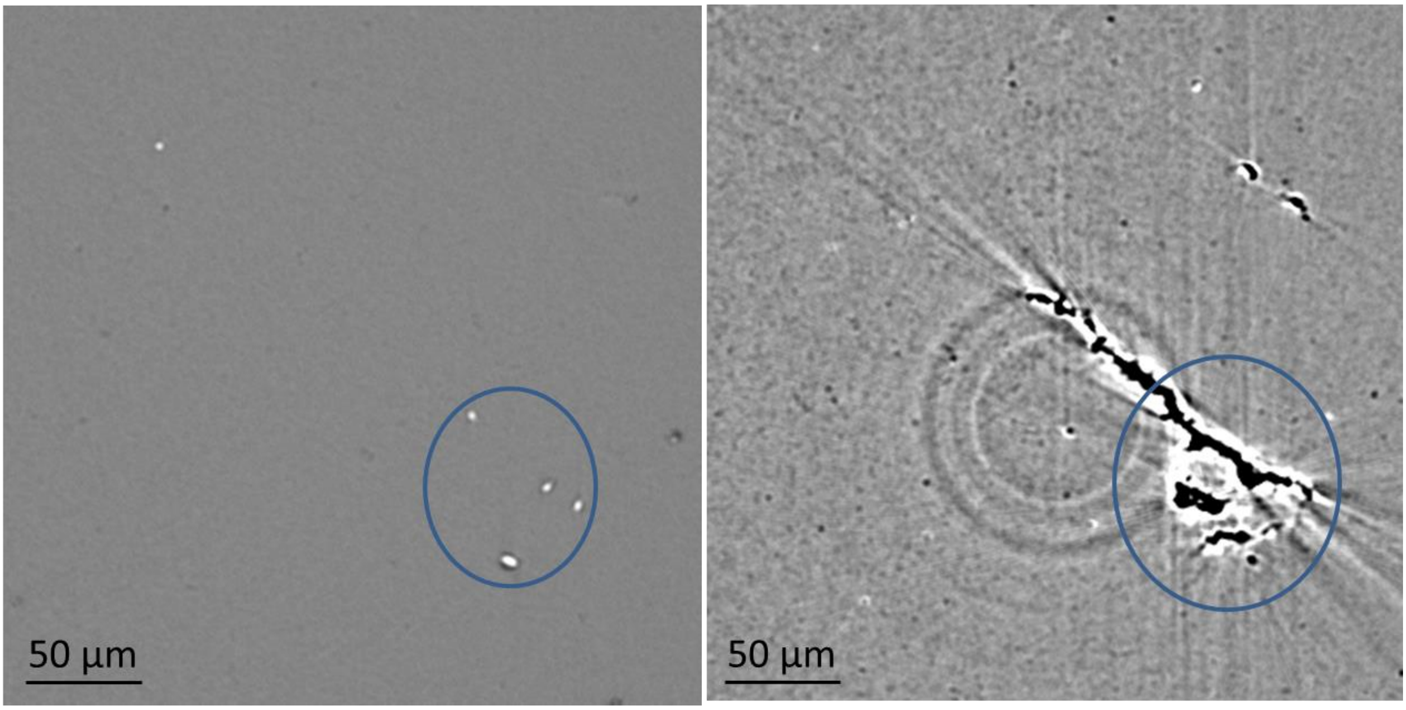

Figure 3: Fe-rich particles cluster on SLM 35°C FSP 1 pass (left) leading to crack nucleation upon loading (right)

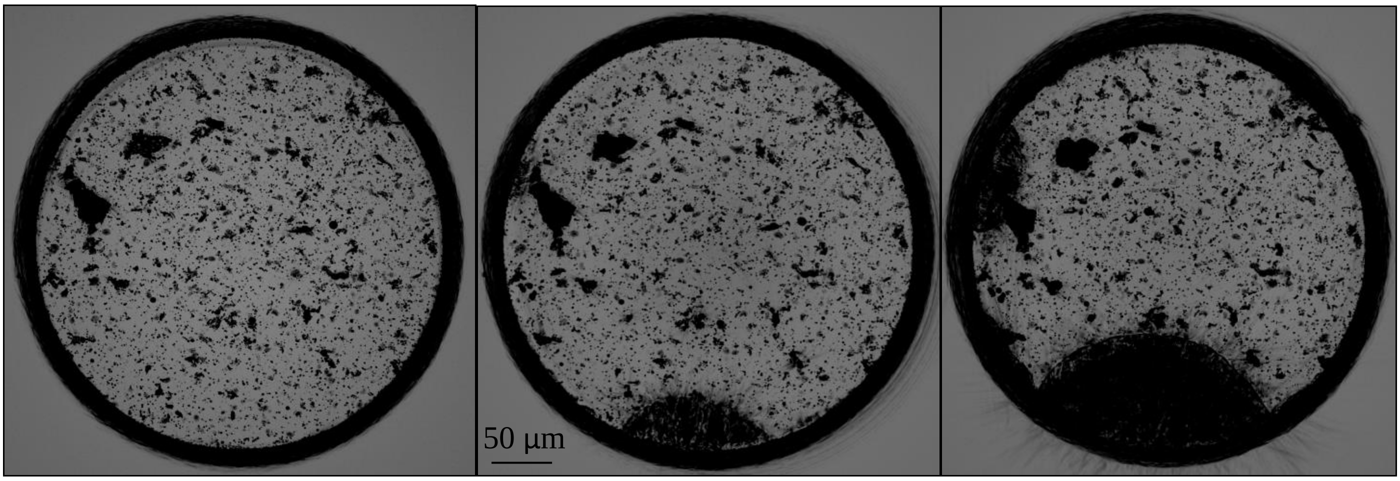

Figure 4: Ex situ fatigue SLM 35°C SRHT minimum intensity Z projection of tomography volumes before loading (left), after 16000 cycles (centre) and after 21000 cycles (right)

Published work

J.G. Santos Macías, C. Elangeswaran, L. Zhao, B. Van Hooreweder, J. Adrien, E. Maire, J.Y. Buffière, W. Ludwig, P.J. Jacques, A. Simar, Ductilisation and fatigue life enhancement of selective laser melted AlSi10Mg by friction stir processing, Scripta Materialia 170 (2019) 124-128.

Abstract: In the effort of expanding the already existing applications of additively manufactured parts, improving mechanical performance is essential. Friction stir processing (FSP) is a promising post-treatment solution to tackle this issue. FSP of selective laser melted AlSi10Mg leads to the globularisation of the Si-rich eutectic network, microstructural homogenisation and porosity reduction. These features have been characterised using in and ex situ mechanical testing and different imaging techniques. A significant modification of the damage mechanism is reported with an increase of the fracture strain from 0.1 to 0.4 after FSP and an enhancement over two orders of magnitude in fatigue life.