Investigation of the effect of Laser Shock Peening in Additively Manufactured samples through Bragg Edge Neutron Imaging

M. Morgano¹, N. Kalentics², C. Carminati³, J. Capek¹, M. Makowska⁴, R. Woracek³,⁵, T. Maimaitiyili⁴,⁶, T. Shinohara⁷, R. Loge², M. Strobl¹,³,⁸*

¹ Laboratory for Neutron Scattering and Imaging (LNS), Paul Scherrer Institut (PSI), 5232 Villigen, Switzerland
² Thermomechanical Metallurgy Laboratory – PX Group Chair, Ecole Polytechnique Fédérale de Lausanne (EPFL), 2002 Neuchâtel, Switzerland
³ Nuclear Physics Institute, Czech Academy of Science (CAS), 250 68 Husinec—Rež, Czech Republic
⁴ Laboratory for Condensed Matter and Materials Science (LSC), Paul Scherrer Institut (PSI), 5232 Villigen, Switzerland
⁵ Neutron Instrument Division, European Spallation Source ERIC (ESS), 22100 Lund, Sweden
⁶ SWERIM AB, 16440 Kista, Sweden
⁷ J-PARC Center, Japan Atomic Energy Agency (JAEA), Tokai, Ibaraki 319-1195, Japan
⁸ Niels Bohr Institute (NBI), University of Copenhagen, 2100 Copenhagen, Denmark

*Corresponding author: markus.strobl@psi.ch

Abstract
Additive manufacturing is a promising and rapidly rising technology in metal processing. However, besides a number of key advantages the constitution of a part through a complex thermo-mechanical process implies also some severe issues with the potential of impacting the quality of products. In laser powder bed fusion (LPBF), the most applied metal additive manufacturing process, the repetitive heating and cooling cycles induce severe strains in the built material, which can have a number of adverse consequences such as deformation, cracking and decreased fatigue life that might lead to severe failure even already during processing. It has been reported recently that the application of laser shock peening (LSP) can counteract efficiently the named issues of LPBF through the introduction of beneficial compressive residual stresses in the surface regions mostly affected by tensile stresses from the manufacturing process. Here we demonstrate how lattice strains implied by LPBF and LSP can efficiently be characterized through diffraction contrast neutron imaging. Despite the spatial resolution need with regards to the significant gradients of the stress distribution and the specific microstructure, which prevent the application of more conventional methods, Bragg edge imaging succeeds to provide essential two-dimensionally spatial resolved strain maps in full field single exposure measurements.

Introduction
Laser powder bed fusion is an additive manufacturing process in which a component is built through layer-wise addition of material [1]. It is a powder bed technique, where a laser selectively melts the powder in a scanning procedure covering the cross section of the component in consecutive layers. After constituting a layer through this procedure, the base is lowered and a new powder layer is deposited and the process repeats. This way the material goes through complex local thermo-mechanical cycles and the solidification of a new layer on an already existing solid layer implies the build-up of detrimental tensile residual
stresses due to shrinkage during cooling of the top layers [2,3]. Despite competitive material properties achieved nowadays in many cases through process optimization [4-10], these complex stress states can cause distortions, cracking and even delamination and complete process failures during the build process [2,3,11].

A variety of in-situ and post-processing techniques have been developed and applied to circumvent the issues. Post-processing cannot prevent process failure and most techniques have severe limitations and drawbacks. For example, pre-heating and laser re-melting can reduce tensile residual stresses and improve geometrical accuracy, but they are limited in introducing compressive stresses and improving fatigue life, hardness, microstructure and crack density [12]. Similar is true for post process heat and hot isostatic pressing (HIP) treatments, which, while improving density through crack closure, can even negatively affect the microstructure and hence properties such as yield strength through promoting significant grain growth [13-15].

Recently, a novel and very promising approach has been introduced. Laser shock peening (LSP) has previously been applied successfully to conventionally produced components to introduce beneficial compressive residual stresses in the surface region and thus increase fatigue life. LSP is a well-known surface treatment. It is used for the purpose of introducing plastic deformation and compressive residual stresses (CRS) into the subsurface region of the treated material. A high energy laser with a pulse duration in the nanosecond range is directed at the surface of the sample. The high energy laser pulse ablates a shallow layer of the surface thus creating a high pressure shockwave directed towards the bulk of the sample. This shockwave plastically deforms the material thus introducing CRS in the subsurface region [16]. Now it could be shown, that LSP applied to LPBF parts has the ability to convert the tensile residual stresses produced by LPBF into beneficial compressive stresses in the processed surface region [17]. In addition to that, it could be proven that LSP can be applied in a hybrid LPBF process referred to as 3D-LSP [18], which allows to apply LSP at any chosen layer of the build process, where it does not only enable stress fields but local microstructure to be tailored to specific requirements [19, 20]. The implied compressive stresses also support crack healing, geometrical integrity and increase in fatigue life [21,22]

Method

Here we introduce an advanced approach for the inspection and investigation of additively manufactured and particularly LSP treated samples. While conventional non-destructive investigation techniques [23] reach their limits due to grain sizes, strain gradients, hence required spatial resolution, and material depths to be assessed, Bragg edge neutron imaging [24] appears well suited to visualize strain fields with the required resolution [25]. Bragg edge imaging is capable to map lattice strains through full field single exposures when sufficient wavelength resolution is achieved in a transmission imaging experiment [24,26]. The method returns through-thickness averages of strains along the beam direction and is based on the wavelength dependent signature of Bragg scattering on the transmitted beam spectrum. Assuming random grain orientation with respect to a considered lattice plane family, the elastic coherent cross section reaches a local maximum, and transmission hence a local minimum, when the Bragg angle reaches \( \theta = 90 \deg \) at the wavelength \( \lambda = 2d_{hkl} \). The cross section and the transmission discontinuously drop and rise, respectively, beyond this wavelength, because no Bragg scattering can take place at the specific hkl lattice planes for longer wavelengths. Therefore, the specific wavelength of such Bragg edge is a precise measure of the corresponding lattice parameter \( d_{hkl} \) of a material and lattice distortions due
to lattice strain can be identified as variation of the edge position in the spectrum. In Bragg edge imaging such information is available for each pixel and hence the strain parameter can be extracted with high spatial resolution, given by the spatial resolution ability of the imaging set-up, which, in the case of this work, is about 100 microns [27].

**Experimental Samples**

In order to investigate the potential of Bragg edge imaging to resolve the strains and thus the strain gradient in the surface region of LPBF and LSP treated LPBF samples with sufficient spatial and strain resolution two additively manufactured samples were chosen for a proof-of-principle experiment. Both samples are cubes of 316 L austenitic stainless steel build by LPBF. The powder was DIAMALLOY 1003, obtained from Oerlikon Metco, Switzerland. The chemical composition is shown in Table 1. Samples were manufactured on a Concept M2 machine (Concept Laser GmbH, Germany) equipped with a fiber laser operated in continuous mode. The laser has a wavelength of 1070 nm and a spot size of 90 microns. The specimen geometry was a 20x20x7 mm$^3$ cuboid with the build height being 7mm. All samples were produced on a 3 mm thick support structure. The processing parameters, i.e. laser power, scanning speed, hatch distance and powder layer thickness were 125 W, 500 mm/s, 0.105 mm and 0.03 mm, respectively. A bi-directional scanning strategy parallel to the part edges was used without a change in scanning direction between layers to deliberately create large residual stresses. Samples were produced under N$_2$ atmosphere and the O$_2$ content was kept below 1 % during the process.

**Table 1: Chemical composition of the 316L stainless steel samples. in wt.%**

<table>
<thead>
<tr>
<th></th>
<th>Cr</th>
<th>Ni</th>
<th>Si</th>
<th>Mo</th>
<th>C</th>
<th>Fe</th>
</tr>
</thead>
<tbody>
<tr>
<td>316L</td>
<td>17</td>
<td>12</td>
<td>2.3</td>
<td>2.5</td>
<td>0.03</td>
<td>Balance</td>
</tr>
</tbody>
</table>

After the production of three equivalent samples by LPBF, they have been removed from the supports and have been subjected to different treatments. One sample (sample A in Fig. 1) serves as a stress free reference for the unstrained lattice parameter $d_0$ in the neutron measurement and has been annealed for 1 hour at 700 deg C in order to release all built in strains. The other two, in contrast, have been subjected to laser shock peening, partially covering the top surface with regards to the building process on an area of approximately 17.2 x 10 mm$^2$. While one of these (sample B) is the main subject of the strain mapping experiment the other sample was locally characterized using the Hole Drilling Method [28]. The LSP treatment was done with a Nd:YAG SAGA HP - class laser from Thales company and water was used as a confining medium during the process. The applied LSP parameters are summarized in table 2.

**Table 2: LSP parameters**

<table>
<thead>
<tr>
<th></th>
<th>Wavelength</th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>1064nm</td>
<td>6.3ns</td>
</tr>
<tr>
<td></td>
<td>1mm</td>
<td>0.4J</td>
</tr>
<tr>
<td></td>
<td>8.1 GW/cm$^2$</td>
<td></td>
</tr>
</tbody>
</table>
Sample B was exposed to the neutron beam together with the annealed reference, sample A (Fig. 1) while the third sample, the twin of sample B was investigated by the hole drilling method (HDM) according to Ref. [17].

<table>
<thead>
<tr>
<th>Frequency</th>
<th>5Hz</th>
</tr>
</thead>
<tbody>
<tr>
<td>Overlap value</td>
<td>40%</td>
</tr>
</tbody>
</table>

**Figure 1:** Left: Sample arrangement for neutron imaging measurements. In the experiment, neutrons come from the lower right of the image and impinge the samples on the small vertical face. Right: Photo of twin of sample B, which was subjected to the local hole drilling assessment, showing the LSP area not covering the full surface. (Note, that the size of the treated area slightly deviates from that of sample B.)

**Instrument and measurements**

Neutron Bragg edge strain mapping measurements were performed at the time-of-flight (ToF) imaging instrument RADEN at the JPARC pulsed neutron source facility [29]. The short pulse nature of the source enables to resolve the neutron wavelength according to ToF measurements in the about 20m long instrument. Utilizing a high time resolution neutron imaging detector the pulsed time structure can be exploited for corresponding wavelength resolved neutron imaging in a ToF mode [24-26]. An MCP/Timepix imaging detector [27,30] was installed at 18.5 m from the source with 14 µs time resolution, thus preserving the inherent ToF wavelength resolution of the instrument at the utilized measurement position of about δλ/λ=0.2%. Such ToF resolution has been demonstrated to be capable of resolving <100 microstrain [24-26]. A beam collimation ratio of L/D=300, where D and L are the pinhole size and the pinhole to sample distance, respectively, was chosen to utilize the full 100 micrometer spatial resolution of the ToF imaging detector with 55 µm pixel size [27]. The total exposure time was 6 hours during which both, sample B and reference (A), were exposed simultaneously (Fig. 2). An equivalent open beam measurement (flat field) was taken for 6 hours without the samples as a reference of the incoming beam distribution. The imaging detector produces images of 512x512 pixels for each ToF time bin. The samples, placed right in front of the detector system, occupy a detector area of around 300 x 400 pixels. The neutron source has a repetition rate of 25 Hz and the corresponding 40 ms period defines the spectral width probed with 2804 time bins of 14 µs time by the detector. Thus, 2804 images are recorded accumulating all pulses within the 6 hour total exposure time to produce spatially resolved spectral histograms. Note that the source power, with which the available
flux is scaling close to linearly, was only at around 200 kW at the time of the measurements compared to 1 MW nominal power.

Data analysis
The raw images have been corrected for the dead time of the detector (overlap correction [30]), and were subsequently normalized to the correspondingly corrected flat field images to obtain the transmission spectra for all pixels, containing the Bragg Edge patterns (Fig. 2a). In order to improve the statistics before the fitting process a 3D running average was applied to the 3-dimensional data set \((x,y,\text{ToF})\) according to a size of 5 pixels (horizontal) \(\times\) 9 pixels (vertical) \(\times\) 7 time bins (ToF). This choice of dimensions for the running average is related to the higher spatial resolution need in LSP direction perpendicular to the surface as well as to sufficient ToF resolution and statistics for reliable fitting. The final analyses of the Bragg edge positions which are a measure of the lattice distance \(d\), and thus lattice distortions, has been obtained by analysing the edge position of the spectra in the region of the most pronounced Bragg edge. This edge is the highest wavelength Bragg edge corresponding to the FCC (111) lattice plane located around \(\lambda = 2d = 4.14\ \text{Å}\) (Fig. 2). The discrete numerical derivative of this Bragg edge was fitted with a Gaussian function for each pixel of the image stack (260000) in order to obtain the local d-spacing represented by the fitted peak position.

![Figure 2](image_url)

**Figure 2** Local attenuation spectrum. (a) The transmission spectra as contained in every pixel of the image stack (averaged over a tensile, orange, and compressive, blue, region of interest); (b) the (111) Bragg Edge extracted from the measured spectra in blue (orange) corresponding to an area of compressive (tensile) strain; (c) representative example of fitting of the (111) Bragg edge contained in a single pixel to consistently identify the edge position;

Results and discussion
The application of pixel-wise fitting of the Bragg edge in the attenuation spectrum enables a mapping of the local (111) lattice parameter \(d_{111}\) with high resolution (Figure 3). It clearly displays the expected features of a relatively homogeneous heat treated reference sample, and larger lattice spacings in particular at the edges of the as built (non heat treated) sample where tensile stresses are to be expected. At the lower left edge of the as built sample, where the LSP treatment has been applied a clear deviation from this behaviour is remarkable and can clearly be associated to the treatment.
In order to better quantify the results and compare to conventional characterisation, the mapped lattice parameters of sample B are referenced and divided by the average lattice parameter of the annealed reference sample A ($d_{111,0}$), according to $\varepsilon_{111} = \frac{d_{111} - d_{111,0}}{d_{111,0}}$. This results in a map of the local lattice strains as shown in Fig. 4a. Subsequently regions of interest are defined as displayed in Fig. 4a and averaged line profiles are extracted from these. The profiles are plotted in Figure 4b. While it appears that the reference sample (orange curve) is still not fully homogeneous but displaying some tensile tendencies towards the edges, the effect of the laser shock peening can be assessed in more detail (black curve). Firstly, compressive strain is found throughout the first about 300 $\mu$m from the treated surface and tensile strain has been reduced to a depth of nearly a millimetre. While the tensile strains close to the untreated surfaces (red line) range up to around 1500 $\mu$ε, the maximum tensile strains in the peened region reach a maximum of only about half that value where the primary treatment effect vanishes 0.9 mm below the surface. In the bulk the behaviour is nearly the same with maximum compressive strains of around -1300 $\mu$ε. The red line profile corresponds to the as-built sample in the area not LSP treated (highlighted in red). The measured tensile strain in the surface regions range again about 1 mm deep into the sample. The strains in the bulk turn compressive towards the centre where they appear to equalize the opposite sign strains at the surfaces. Beyond 0.9 mm the compressive effect of the LSP treatment is reversed and up to about 3 mm into the sample the strains under the treated surface range slightly above those of the untreated area balancing to some extend the compressive surface effect induced.
The extracted profiles can be directly compared to conventional measurements performed locally with the hole drilling method (HDM) on the twin sample of the partially LSP treated sample B. The hole drilling technique measures the strains on the surface of a sample after drilling small holes at increasing depths. From the measured strains the residual stresses can be calculated. A RESTAN – MTS 3000 (SINT Technology, Italy) measuring device was used, and the measurements were done according to the ASTM standard E837. Further details on this measurement technique are provided in Ref. [3,17,18] and [28]. A total of 36 measurement points were performed over a depth of 1 mm. In order to achieve more precise measurement results in the near surface region, a variable depth increment was applied. From the surface up to the depth of 100µm, measurements were made every 10µm. From 0.1mm up to 0.5mm in depth, measurements were made every 25µm, and from 0.5mm up to 1mm every 50µm. From the stresses measured with the HDM we could calculate the corresponding lattice strain using Hook’s law and compared with the neutron data (Fig. 4c). In contrast to the neutron measurements this method is not only destructive but also local, i.e. representing a single position. The HDM equipment is usually limited to measuring residual stresses to the depths of up to 1mm since the measurement accuracy decreases at increased depths [28]. Data has been acquired for the first millimetre from the surface in an LSP treated region (black dots) and in an untreated surface region (red dots). In Fig. 4c these two sets of data are directly compared to corresponding line profiles from the neutron measurement (black and red lines respectively). A qualitative agreement is obvious while the local values contain substantial deviations. Peak values close to the surface appear more distinct in the hole drilling data than in the strain map profiles. The spatial depth resolution appears superior with the conventional method and corresponding limitations of the neutron images tend to smear out such features. This might in particular be due to the binning in this direction, which hides the drop of strains towards zero on the surface and thus also shifts the peak strain value, especially for the untreated surface region seemingly further away from the sample edge, while behaving smoother. Peak values of tensile and compressive strains in the treated and untreated surface regions appear lower for compressive and tensile peaks, respectively. Again, this is partially an effect of the resolution smearing. However, in the LSP treated region the difference in magnitude is significant and cannot be explained by spatial resolution only. A reason for this deviation was found to be that the projection of the treated surface also includes untreated regions at the edges of the surface (see Fig. 1), which display high tensile stresses. It has to be noted that the neutron technique is averaging along the beam in the projection image. For the specific case where the strains are, to a first approximation, invariant throughout the thickness, as is supported by the quantitative results in the other region, the given deviation in the (partially) treated region leads to the lower net compressive strain found in the neutron data. The transmitted front and back face of the sample might contribute to a slight offset on the strain axis. Further away from the surface beyond about 0.7 mm, the hole drilling method suggests an increase in tensile strains, while the imaging data suggests a falling trend towards strain free and later compressive strain regions, beyond the depth reached by the hole drilling method. In both cases the trends are consistent for treated and untreated regions. However, the hole drilling method loses significantly in accuracy at the corresponding depths. The depth
up to which the LSP treatment has effect in terms of reducing tensile strain is suggested to be about 0.9 mm with respect to the neutron data. These results are consistent with literature [17,25].

Conclusions
It has been demonstrated, that high resolution Bragg edge imaging can provide full field maps of strain distributions in additively manufactured samples. The method is capable of visualizing the inhomogeneous strain distribution from the surface to the bulk of the material, from tensile to compressive strains. In particular an application to a sample partially treated by laser shock peening on one surface demonstrates the potential of the non-destructive spatially resolved mapping of strains through wavelength resolved neutron imaging, enabling to visualize and characterize the changes in the strain distribution induced by the post built treatment. This analysis technique can thus be applied to efficiently investigate the residual strains in series of as-built samples, as well as to gauge the effectiveness and range of post-process treatments such as heat treatment and LSP applied to the very same samples. In this context, we demonstrated that the effect of LSP, while strongest within a layer of ~100-300 µm from the surface, where strains are turned to compressive, extends much further beyond the surface and can still be measured up to almost 1 mm from the interface. Inverse effects and deviations from the untreated state can be found down to even about 3 mm. Such studies can therefore be utilized in the context of 3D-LSP to optimize parameters (laser energy, overlap value, spot size, frequency of LSP treatments during a 3D LSP process) and to design treatments in order to achieve desired mechanical properties and variations of properties. While the single exposure method is effectively limited to observations of strains in the beam direction it provides an invaluable overview of variations throughout the sample and returns even quantifiable results as is underlined by the verification with a conventional but destructive and local assessment technique, namely the hole drilling method. The immediate non-destructive characterization appears to have significant potential for the assessment of large series of samples in particular for large parametric series of additive manufacturing. Advancing technology and in particular pulsed neutron source power might even enable in-situ studies during specific treatments.

Acknowledgement
The work was partially funded through the OP RDE, MEYS, under the project “European Spallation Source—participation of the Czech Republic—OP”, Reg. No. CZ.02.1.01/0.0/0.0/16 013/0001794, MS, JC, RL, TM additionally thank for financial support from the Strategic Focus Area Advanced Manufacturing (SFA-AM), an initiative of the ETH Board, and JC also for funding from the European Union’s Horizon 2020 research and innovation programme under the Marie Skłodowska-Curie grant agreement No 701647. The neutron experiments at the Materials and Life Science Experimental Facility of J-PARC, BL22, were performed under project number 2017B0208.

References


