

LASER POWDER BED FUSION AND SELECTIVE LASER MELTED COMPONENTS INVESTIGATED WITH HIGHLY PENETRATING RADIATION

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Abstract

Methods of incremental manufacturing, i.e. 3D printing, have been experiencing significant growth in recent years, both in terms of the development of modern technologies dedicated to various applications, and in terms of optimizing the parameters of the process itself so as to ensure the desired mechanical and strength properties of the parts produced in this way. High hopes are currently being pinned on the use of highly penetrating types of radiation, i.e. synchrotron and/or neutron radiation, for quantitative identification of parameters characterizing objects produced by means of 3D printing. Thanks to diffraction methodologies, it is feasible to obtain input information to optimize 3D printing procedures not only for finished prints but also to monitor in situ printing processes. Thanks to these methodologies, it is possible to obtain information on parameters that are critical from the perspective of application of such obtained elements as stresses generated during the printing procedure itself as well as residual stresses after printing. This parameter, from the point of view of tensile strength, compression strength as well as fatigue strength, is crucial and determines the possibility of introducing elements produced by incremental methods into widespread industrial use.

Keywords: neutron diffraction, synchrotron diffraction, residual stress measurements, additive manufacturing, SLM, LPBF **Article Category:** Research Article

INTRODUCTION

Rapid prototyping also known as 3D printing has been used for almost 30 years. Its milestone was marked by Charles W. Hull who in 1983 constructed a stereolithography apparatus SLA-1 [1]. The machine used liquid photopolymer curable via UV light to additively produce 3D objects layer by layer. Today the number of competing additive



This work is licensed under a Creative Commons Attribution-NonCommercial-NoDerivatives 4.0 International License technologies is so high that it is quite difficult to establish a valid classification nevertheless, efforts already have been made [2] to do so. Various additive manufacturing methods were already adopted in different industries [3]. In automotive, aerospace and medical sector for example laser powder bed fusion (LPBF) based technologies such as selective laser sintering (SLS) and selective laser melting (SLM) have already proven to be reliable and cost-effective means of producing high quality, elaborate shape designed components. Still many challenges lie ahead in terms of process optimization for either SLS or SLM to become truly disruptive technologies for the manufacturing industry. Focusing on the SLM, it is to be noted that two of the major hurdles to overcome are residual stress buildup and defects formation during SLM parts fabrication. Process parameters optimization can be done through numerical simulations of residual stress state and defect formation, employing finite element methods (FEM) approach to reduce time-to-market however, non-trivial thermomechanical interactions between physical phenomena occurring during the SLM AM component production are not feasible to be fully incorporated into the models to be tested since computational costs are to be considered. A complementary approach of combining FEM with nondestructive testing (NDT) or more recently non-destructive evaluation (NDE) have already shown to be effective both in mitigation of faults plaguing some of the SLM fabricated components and gaining a better insight into SLM process itself. The X-ray diffraction methodology performed with laboratory X-ray diffractometers and applying low energy radiation has its limitations with regards to mapping complex stress fields present in intricate shaped SLM manufactured components as only near surface stresses can be studied. Low energy X-ray radiation penetrate to a depth of over a dozen of micrometers while synchrotron X-ray diffraction and neutron diffraction (ND) methods provide penetration depths of ~10 μ m up to 5 mm and ~1 mm up to 10 cm respectively [4]. Moreover, both aforementioned measurement techniques possess wider capabilities for conducting in-situ experiments that enable real time monitoring of materials properties during samples LPBF fabrication. Below, recent state of the art on diffraction methods using synchrotron or neuron radiation employed in residual stresses investigation of LPBF and SLM components is presented.

Application of synchrotron radiation diffraction in laser powder bed fusion for residual stress measurements

Three-dimensional residual strain and stresses in LPBF Ti6Al4V bridge shaped component were studied by Strantza et al. (2018) with synchrotron radiation diffractometry and computational methods employed [5]. Bulk, compressive strains were found in transversal plane with respect to build direction of examined parts. They were balanced by tensile strains on samples sides. Experimental results were compared with the simulation, and it was demonstrated that strain values determined with both experimental and computational methods have the same trends, although the applied model over-predicted the strain in scanning and hatching directions in the studied parts boundaries.

Mishurova et al. (2019) investigated LPBF Ti6Al4V cuboid shaped samples to probe stress dependence on different, not commonly considered LPBF process parameters

[6]. A varying decrease in residual stress values was observed for different combinations of laser scanning speed, laser power, hatch distance etc. used for calculation of volumetric energy density. Additionally, samples surface roughness and the effect of support structure were studied, together with the influence of the argon flows direction on the residual stress state. The authors concluded that although the support structure had no influence on the subsurface residual stresses, samples orientation in the LPBF production chamber with regards to the argon flow direction on affected stresses and should be taken into consideration, as one of important process parameters in LPBF.

Evaluation of thermomechanical model established for LPBF residual stresses prediction together with synchrotron diffraction measurements of residual elastic strains in LPBF Ti6Al4V bridge shaped samples was carried out by Ganeriwala et al. (2019). The research took into account two types of scanning strategies: continuous serpentine and island. Layer agglomeration or lumping approach was introduced to shorten computation times [7]. Synchrotron diffraction measurements were taken at several points on middle cross section plane transverse to samples-built direction and proved to be in good agreement with the simulation results obtained using the proposed numerical model, however uncovered certain limitations of the employed approach. Experimental and numerical simulation data showed stress gradient of compressive nature in the center of each bridge shaped samples balanced by tensile stresses towards samples boundaries which is a typical characteristic of LPBF builds. On the other hand, performed calculations could not fully reveal the effect of adopted scanning strategy on the residual stress values with respect to simulations carried out taking into account a scanning strategy and those relying on beam and layer agglomeration techniques. The authors concluded that since a significantly higher stress values towards the sample's boundaries were observed through measurements in samples fabricated employing the island scan strategy with higher fidelity, yet computational time constrained simulations of the LPBF process should be developed.

Bonder et al. (2020) investigated Inconel 625 and stainless steel 316L (SS316L) multilayer structured cuboid shaped parts built on SS316L base plate. Studied components were fabricated using liquid dispersed metal powder bed fusion technique [8]. Synchrotron diffraction, nano-indentation, as well optical, scanning electron microscopy and transmission electron microscopy were employed to examine the IN625-SS316L multilayers in different scales to reveal materials microstructure and stress state. The periodicity of IN626 and SS316L layers was correlated with crosssectional hardness increase and decrease together with compressive stresses decrease and increase. Hatching strategy involving a 66 deg. rotation between subsequent layers resulted in the development of wave-like shaped grain boundaries which were identified to promote wave-like cracks spreading along the built direction. The authors conclude that C-like stress gradient identified in the material along with substantial surface tensile stresses is to be interpreted in terms of temperature gradient mechanism (TGM). Furthermore, they argue that the formation of chromium-metal-oxide nanodispersoids identified through chemical analysis of the studied multilayer system has a potential for prospect nanoscale microstructural design via reactive additive manufacturing.

A non-trivial link between macro stresses and diffraction based strain was investigated for LPBF Ti6Al4V by Mishurova et al. (2020) using in-situ synchrotron diffraction technique combined with tension and compression tests [9]. Microstructure of LPBF samples was also determined, with SEM, energy dispersive X-ray diffraction and X-ray computed tomography. The authors compare results obtained with chosen techniques against available theoretical models, determine diffraction elastic constants (DECs) and demonstrate that LPBF obtained materials possessing different DECs, than those of wrought alloys due to distinct microstructure and mechanical properties. They conclude that the readily available models can only be used to calculate DECs if certain non-trivial characteristics of microstructure are not being taken into account.

Serrano-Munoz et al. (2020) also investigated the relation between residual stresses and microstructure in LPBF fabricated components. LPBF Inconel 718 elongated prisms were produced with three distinct scanning strategies and characterized with low energy laboratory X-ray diffraction and synchrotron energy dispersive X-ray diffraction combined with optical profilometry. The aim was to study residual stress distribution [10]. Microstructure of the samples was determined by SEM and electron backscatter diffraction techniques. The samples were examined in three states: as built state, as built state but with a thinned built platform and removed from built platform. Highest surface residual stress values were observed in components produced employing unidirectional hatching direction scanning strategy, whereas the lowest when 67 deg. rotated scanning strategy was applied. Considerably high residual stress values were observed in hatching and built directions for samples removed from built platform. Built platform thinning resulted in partial release of the of residual stresses in built direction. Most importantly the authors confirmed that microstructures resulting from the AM process pose a challenge to classical models used for DECs calculation essential in diffraction stress analysis. It was found i.e. that the use of Reuss model [11] for different crystal planes is better suited than a more widely adopted Kroner model [12], as the obtained results were less scattered.

Artzt et al. (2020) studied the influence of contour parameters on surface roughness and subsurface residual stresses in LPBF Ti6Al4V with synchrotron diffraction technique and near-infrared melt pool monitoring system [13]. A direct correlation between those two properties was found to be the following: high laser power combined with fast scanning speeds and using two or more contour lines printed from the outside to the inside reduced surface roughness by over a half and concurrently increased residual stresses by over one half. The stress level of about 800 MPa in the LPBF components was produced with the following scanning parameters: laser power 300 W, scanning speed 1575 mm/s and contour hatching distance 90 µm resulted in observation of macroscopic cracks. A Pareto optimum for outside to the inside contour strategy applied to LPBF Ti6Al4V was achieved with process parameters tuned to values of: laser power 100 W, scanning speed 1050 mm/s and contour hatching distance 90 µm, which resulted in a tradeoff between residual stresses in built direction of 625 MPa and surface roughness of 14.2 µm. Additionally, the authors conclude that utilized nearinfrared metl pool monitoring system could be a useful tool for surface roughness, hance residual stress state estimation and development of built strategies for complex LPBF components.

Calta et al. (2020) studied melt pool fluid dynamics and post solidification microstructural evolution in LPBF Ti6Al4V (Ti64) and Ti5Al5V5Mo3Cr (Ti5553) employing in-situ synchrotron diffraction [14]. Melt pool fluid dynamics were found to be similar for both alloys and melt pool area was proportional under the same LPBF laser power and scanning speed conditions. While more pores were observed to form in Ti64, with regards to Ti5553, although the authors stated that a comparison between sets of fully built components should be made to statistically verify their finding. Additionally, diffraction pattern evolution was investigated to gather information about phase development during the LPBF. Timescales and the magnitude of peak shifts and intensity modulation were observed to vary with correlation to laser power, nevertheless all the studied samples exhibited comparable behavior. Ti64 was found to show linear relation between the beta phase lifetime and LPBF laser power. Peak broadening was also observed at high laser powers for both alpha and beta phases, this was attributed to simultaneous non-uniform solute segregation and a microstrain induced by increased dislocation density. Ti5553 subjected to high laser powers displayed lattice contraction in the alpha phase together with an lattice expansion in the beta phase that could potentially be contributed to the buildup of isotropic stresses, nevertheless the authors were not able to rule out if solute segregation during solidification should be additionally taken into consideration to explain the observed phenomena. It was concluded that the obtained results can be used as a valuable input for numerical models as well as for LPBFs further process parameters optimization.

A novel approach for predicting residual stresses in LPBF produced components was proposed by Aminforoughi et al. (2021). Similarly, to sin²psi approach used in reflection mode the newly introduced sin²alpha methodology was based on linear regression but could be used in transmission mode meaning different residual stresses were able to be probed locally in the studied samples [15]. Firstly, simulated synchrotron two-dimensional images containing full Debye-Scherrer rings were analyzed to calculate residual stresses with Hooke's and sin²alpha approaches respectively. It was shown that the sin²alpha approach was clearly a more robust one with regards to Hook's. In the next step the proposed sin²alpha approach was evaluated experimentally via in-situ tensile tests where a traditionally produced steel 100Cr6 and LPBF Inconel 718 samples were examined. The gathered experimental data were evaluated, again employing both Hook's and sin²alpha approaches. The conducted analysis proved the sin²alpha methodology to be a more able-bodied in calculating stresses in the studied materials. Lastly stress measurements of LPBF Inconel 718 sample fabricated adopting bidirectional scanning strategy together with 90 deg. rotation for consecutive layers were carried out enabling simultaneous detection of full Debye-Scherrer of different lattice planes. The authors concluded that the proposed sin²alpha approach could be very useful in in-situ measurements during the LPBF leveraging on high-intensity beams and area detectors for fast multiple, complete Debye-Scherrer rings detection.

Application of synchrotron diffraction in selective laser melting residual stress measurements.

Synchrotron diffraction technique was used by Mishurova et al. (2017) to study SLM Ti6Al4V bridges fabricated under different scanning speeds to correlate laser energy density on the state of residual stresses [16]. It was found that high tensile gradients were present on the top surface in the build direction plane for as built samples and the geometry of the samples promotes increasing strains towards the bridges pillars. Process parameters responsible for lower lattice strains were observed to be high laser energy densities. Heat treatment in vacuum conditions at 650°C for a period of 3h performed on selected samples resulted in the lattice strains relief. The authors concluded that synchrotron energy dispersive mode together with confocal microscopy were suitable methods for residual stresses and shape distortion measurement in SLM Ti6Al4V components.

Mishurova et al. (2018) also studied with synchrotron diffraction the effects of support structure affixture and of build platform removal in SLM Inconel 718 components [17]. Two types of samples in the shape of elongated prisms, with and without the support structure were studied. Stress gradient along the hatching direction was found along with high stress values of tensile nature in the built direction plane subsurface, adding that the highest stresses were present in the scanning direction. Build platform removal resulted in stresses redistribution in the sample fabricated with the support structure, whereas samples directly built on the platform exhibited stress relief. Additionally, shape distortion on the built direction planes surface was found with contact profilometry. The sample built with the support structure showed larger shape distortion with regards to the sample built directly on the build platform. The authors concluded that the support structure was a factor that enabled some extent of stress relief.

Selected new aspects aimed at optimizing the SLM Ti6Al4V were investigated by Mishurova et al. (2019). Residual stress state studies combined with porosity measurements were conducted with the use of synchrotron radiation and X-ray computed tomography respectively [18]. The most relevant SLM process parameters were demonstrated to be reduced hatching distance together with shorter scan speeds resulting in the stabilization of the SLM produced elongated prisms microstructure without impact on materials porosity, in conjunction with an approximately four-fold reduction of the subsurface residual values. Both increased laser power and scan speed affected pore shape and the presence of keyhole pores. On the other hand, the position of the sample on the build platform influenced the stress state due to uneven heat dissipation mechanisms taking place in the fused and unfused areas of the powder bed. Moreover, it was proven that the laser focus distance from the powder bed can reduce the residual stress state.

An inhouse built MiniSLM device in combination with synchrotron diffraction methodology was used by Hocine et al. (2020) to conduct operando experiments on SLM Ti6Al4V fabricated with different process parameters and investigate the high and the low temperature phases evolution in conjunction with heating and cooling rates measurement of powder and the fused solid material [19]. Stress state in the beta phase

were studied and a link was established between materials microstructure evolution and the adopted scanning strategy, in particular a size effect associated with scanning vectors length. The authors conclude that operando diffraction experiments are a useful practical tool for FEM-based models' validation. Furthermore, they argue that a synergic effect would be obtained if operando experiments measurement data could be combined with numerical simulations i.e. via adjusting the X-ray beam profile to match the FEM simulations mesh size or vice versa providing a more thorough insight into aluminum alloys micro-alloying, thus microstructure evolution conveyed by chosen SLM process parameters. Additionally, they propose in-situ heating to be introduced into the experiment for stress relief and microstructure optimization.

Application of neutron diffraction in laser powder bed fusion residual stress measurements.

A study of residual stresses with regards to LPBF process parameters, such as laser scanning speed, scanning strategy and build direction was performed by Wu et al. (2014) in stainless steel 316L (SS316L) with the use of destructive and non-destructive methods [20]. Built platform removal and sectioning together with digital image correlation (DIC) measurements were coupled with neutron diffraction experiments. Results obtained with different methods were in good agreement. It was observed that stresses present as a result of printing technology can be reduced by adopting a scanning strategy of decreased island size with 45 deg, island scan rotation and increased laser energy density. Data gathered with neutron diffraction experiments provided information that in-plane residual stresses changed depending on the islands scanning rotation angle, whereas axial residual strains were simultaneously found not to be altered. It was concluded that a 45 deg rotation of the scanning direction created a favorable misalignment between thermal residual stresses present along this direction and the largest dimension of the LPBF fabricated part however, the stresses along the built direction of produced prisms increased. It resulted in spherical deformation towards the samples top upon removal from the built platform. This was attributed to a constraint imposed onto the built direction plane area reduction by the subsurface layers coupled with thermal effects associated with reheating. The authors showed that the increase of laser power and scanning speed translates into a decrease of the fabricated elements deflection after sectioning. Furthermore, it was found that LPBF process parameters result in multiple thermal effects to occur the subsequent subsurface layers, that influence residual stress values in LPBF fabricated components.

Residual stresses in a LPBF IN625 thin curved wall element were studied with the use of neutron diffraction by An et al. (2017). Additionally, the stresses in the studied part were predicted by a simulation based on a simplified FEM numerical model for both the production process and after cooldown [21]. The neutron experiment data and the simulation generated one were found to be in good quantitative agreement. It was demonstrated that the introduced model has not only correctly predicted tensile circumferential stresses on the built direction planes of the top and bottom surfaces of the sample but also the tensile axial stresses on the edges of the LPBF part, as well as compressive axial stresses in the curved thin walls volume. Therefore, the authors concluded that the simplified numerical model with layer-wise activation could effectively be used for stress state mapping and in turn distortion prediction in the LPBF fabricated components. Furthermore, it was shown that neutron diffraction was a reliable NDT technique for residual stress distribution mapping in bulk metallic samples, that enabled determination of stress-free lattice spacing $- d_0$ required in elastic strain calculation.

Gloaguen et al. (2019) performed neutron diffraction experiments on LPBF Ti6Al4V (Ti64) cubes. To characterize the full stress tensor the measurements were carried out in several positions and depths of the samples [22]. The authors concluded that residual stresses in LPBF produced samples mostly originate due to thermal gradient mechanism. They added that the stresses found in the top and bottom surfaces of the samples were of tensile or low compressive nature, while in the volume of the sample were solely of compressive nature. This finding is consistent with other works investigating residual stresses in LPBF and/or SLM fabricated components using diffraction methods. Moreover, it was noted that to reduce stresses the LPBF process parameters should be optimized, which was also suggested in the works by other authors. Lastly, a non-negligible second order residual stress gradients arising various physical phenomena that occur during the LPBF process should be taken into consideration for normally highly anisotropic hexagonal alloys like Ti64.

LPBF austenitic stainless steel 316L (AISI316L) prisms were studied by Ulbricht et al. (2020) to investigate residual stress distribution due to heat accumulation [23]. Two different sample wall scanning strategies were employed: from the outside to the inside (O-I) and from the inside to the outside (I-O) to determine the effects of thermal gradient mechanisms influence on solidification shrinkage mechanism. Residual stresses were characterized using neutron diffraction in transversal plane with regards to build direction. Additionally, thermography data obtained during the LPBF process were used to establish a link between heat dissipation and stress distribution in the studied LPBF AISI316L prisms, while optical microscopy combined with micro computed tomography provided insight into residual stress distribution with regards to defects presence in the material. Compressive residual stresses were found in the bulk of the samples along with surface tensile residual stresses, which was consistent with other residual stress studies on LPBF fabricated components, however these values varied depending on the adopted scanning strategy. The authors concluded that solidification shrinkage mechanism was the main factor that determined the residual stress distribution, whereas the thermal gradient mechanism has its effects solely on the residual stresses magnitude, however in-situ thermography measurement data gathered for the O-I prepared sample uncovered highly localized compressive residual stresses due to latter mechanism.

LPBF build process, thermomechanical effects and heat treatment creep stress relaxation were investigated via FEM numerical simulations and later validated trough neutron diffraction experiments in LPBF stainless steel 316L (SS316L) samples by Williams et al. (2020). Large tensile residual stresses predicted by the developed models in vertically produced samples at top and bottom surfaces were in equilibrium with compressive residual stresses predicted in samples center. When compared with FEM predictions for horizontally produced components their values were found to be about

40% lower. Moreover, samples fabrication orientation and geometry were observed to have a profound impact on their distribution after removal of the build platform. Additional heat treatment simulations showed about 10% stress relief in vertically produced samples and 40% stress relief in horizontally produced samples. FEM predicted residual stress values and their distribution were found to be in good agreement when validated via neutron diffraction. In conclusion, it was demonstrated that a validated model could be a practical tool to predict the residual stress in as built state and after relaxation in case of LPBF produced components.

Inconel 718 plate shaped samples fabricated horizontally and vertically with LPBF and electron beam powder bed fusion techniques were studied with neutron diffraction to analyze stresses by Goel et al. (2020) [24]. Process parameters and heat treatment impact on the residual stresses lead the authors to conclusions that as built samples produced with chessboard scanning strategy, when compared with samples fabricated using a bidirectional raster scanning strategy were lower and post-heat treatment further lowered the stress values. Moreover, stresses in LPBF parts were higher than in the electron beam powder fusion parts due to higher temperature gradient occurring during the fabrication process. Lastly, the measured stress free lattice spacing (d_0) was largely influenced by the manner in which the stress-free reference sample was produced, adding that calculated d_0 value could not be valid for thick samples, as it assumed only plane stress state condition.

A valve housing with complex internal three-dimensional features, produced via LPBF from 316L stainless steel (SS316L) was studied by Clausen et al. (2020) using neutron diffraction and contour method to evaluate stresses evolution in two states: as built and removed from base plate [25]. In the as built state the sample showed a typical residual stresses pattern observed in LPBF produced components, that is high tensile in the built direction plane and compressive in the bulk. Neutron diffraction probed residual shear stresses which were found to be relatively low near the built platform region, when compared with their normal values. Built platform removal resulted in substantial stress changes near the bottom of built direction plane and in the vicinity of the geometrical features around which stress concentration buildup was observed by contour method. At the built direction plane the residual stresses were completely relieved however scanning direction residual stresses changes were also found to be of significant values. Moreover, the channels introduced in the structure design spawned highly localized stresses that can be concluded to influence both structural and dimensional properties of the LPBF fabricated component.

L-shaped LPBF Inconel 718 parts fabricated in three different build orientations were studied by Pant et al. (2020) with neutron diffraction technique and optical three dimensional scanning [26]. Neutron diffraction method enabled residual stresses mapping in chosen cross-sections of each of the samples, while 3D scanning was used to evaluate parts shape distortion after removal from the build platform. Additionally, the gathered data was compared with a simplified FE numerical simulation. It was shown that stresses in studied components are distributed in the manner typical for LPBF components, that is are tensile near the surface area and compressive in the sample bulk. Smallest residual stress values were found in the horizontally built samples, whereas the highest were observed in the vertically built ones. Residual stress values in vertically built parts rotated

45 deg. in the built direction plane to accommodate support structure lied in between those of horizontally and vertically built. Nevertheless, these samples were shown to be less deformed after built platform removal, than the samples produced horizontally. Since the component of stress in built direction in both the vertically and horizontally built samples was larger than the component along the scanning direction the authors concluded that proper samples orientation should be considered for different shapes and sizes of the LPBF produced parts. Furthermore, although a simplified numerical model was shown to be in good quantitative agreement with the obtained results it was also concluded that further improvements in the simulation should be introduced.

Zhang et al. (2021) studied with in-situ residual strains, stresses and dislocation density with neutron diffraction in LPBF AlSi10Mg alloy [27]. The authors observed maximum stress values in both the Al and Si phases under plastic deformation in the load direction. Both tensile and compressive stresses were found. Additionally, dislocation annihilation phenomenon was uncovered in the Al matrix, resulting in the reduction of dislocation density during the sample unloading stage, with the amplitude of the decrease rising in the plastic deformation regime. The authors concluded that the uncovered phenomenon was induced by compressive stresses in the Al matrix. Additionally, the annihilation of screw dislocations during unloading was found to promote the decrease in total dislocations density.

Lattice structures fabricated from Inconel 625 using LPBF were investigated by Fritsch et al. (2021) to determine residual stress values [28]. The authors also describe ways in which difficulties in correct stress field determination in produced structures could be overcome. Uniaxial stresses could be found along the struts in of the studied lattices, while stresses found in the knots were found to be hydrostatic. In addition, strain measurements were conducted to determine principal stress directions. It was concluded that strain measurements should be taken in nine points of the lattice structure for correct assessment of principal stress direction in the studied lattice structures which is in contrary to textbook knowledge.

Microstructure, texture and stresses development dependence on the scanning strategy was investigated in Inconel 718 LPBF produced elongated prisms by Nadammal et al. (2021) [29] using optical microscopy, scanning electron microscopy, electron backscattered diffraction and neutron diffraction. With the X axis positioned along the samples length, the Y axis positioned along samples width and Z axis representing the built direction plane the X- scanning strategy combined with short hatching length and a serpentine pattern produced a pronounced texture together with columnar grains grown along the built direction, that in turn induced high stress gradients and resulted in high stress values. The Y- scanning strategy on the other hand has been found to produce a different microstructure resulting in lower residual stresses and more uniform stress gradient. The alternating and 67 deg. rotational strategies were observed to produce similar microstructures. Residual stress gradient and its values comparable to the Y- strategy produced sample were found in the alternating strategy produced sample however, lower stress values and the significantly more uniform stress gradient were present in 67. deg rotational strategy built sample. Residual stresses distribution and their values found in the aforementioned two samples were attributed to the redistribution effects due to successive layers rotation, influencing the deposition process. However, the rotational strategy facilitated random orientation grain growth due to more complex thermal fields during the LPBF, that in turn were responsible for decreasing the residual stresses. The authors concluded that residual stresses in the LPBF fabricated samples can be substantially minimized through optimized combination of scanning strategies together with hatching length and spacing.

Scanning strategy influence on residual stresses distribution was also studied was also studied by Serrano-Munoz et al. (2021). The authors used neutron and X-ray diffraction for bulk and surface residual stresses assessment [30]. Additionally, electron backscattered diffraction was used to gather information about samples microstructure and profilometry was employed to study sample shape distortion after built platform removal. Elongated LPBF Inconel 718 prisms were fabricated with 90 deg. alternation and 67 deg. scan rotation strategies, involving long and short scanning vector lengths. X-ray diffraction measurements showed that longer scan vector lengths result in higher residual stress values when compared with short scan vector for the corresponding scanning strategies that were adopted. Furthermore, the 67 deg. scanning strategies were shown to lead to lower stresses than the 90 deg. alternating counterparts. Neutron diffraction measurements were found to show pronounced residual stress gradients in sample bulk. All the samples experienced upward shape distortion upon built platform removal. This was attributed to significant influence of the residual stress component along the built direction, although the samples produced adopting the long vector scanning strategy were seen to possess additional degree of twisting distortion along their length. The dissimilar distortions observed were concluded to originate from scanning strategy effect on stress build-up during the LFPB fabrication process.

A parametric study of laser shot peening (LSP) effects on LPBF stainless steel 361L (SS316L) samples produced with different process parameters were studied with neutron Bragg imaging by Busi et al. (2021) [31]. Large LPBF fabricated SS316L parts were subjected to LSP performed with laser energies of 1J and 1.5J, along with 40% and 80% laser spot overlaps respectively, which resulted in inducing compressive stresses. The most favorable LSP treatment parameters in terms of compressive stresses depth was achieved using laser energy of 1.5J and 80% laser spot overlap. This was concluded to be attributed to a higher overall laser energy density per LSP sample area during the process. A complementary assessment of different LSP strategies was done by comparing AB samples, with surface LSP treated ones and those with a buried LSP treatment layer to develop 3D-LSP strategies that would potentially enable RSs field design in LPBF components. It was found that careful tailoring of the LSP treatment parameters CRSs gain of 50% together with 100% increase in CRS depth can be achieved.

Serrano-Munoz et al. (2021) studied LPBF Inconel 718 samples produced with 67 deg. rotational scanning strategy to evaluate strain-free lattice spacing (d_0) and quantitively determine residual stresses, using neutron and X-ray diffraction methods [32]. A coupon grid and measurement under stress balance approaches were employed for residual stress analysis in the prepared samples. The authors conclude that such methodology, although more time and resource consuming should be applied to address the challenges associated with complex AM components geometries, microstructure and defects distribution in residual stress analysis.

Residual stress relaxation due to heat treatment and relevant changes in microstructure were studied in LPBF austenitic stainless steel 316L (AISI316L) with neutron diffraction, scanning electron microscopy and electron backscatter diffraction techniques by Sprengel et al. (2021). Three heat treatment (HT) strategies of 450°C, 800°C, 900°C were utilized [32]. Samples HT in the 450°C for 4 h and 800°C for 1 h were found to exhibit both high tensile and compressive residual stresses, whereas in the sample subjected to 900°C annealing stresses were observed to be completely relieved. The authors concluded that near total residual stresses relief could be linked to a close degree with the evolution of characteristic subgrain solidification cellular microstructure.

An in-situ surface heat treatment effect on residual stresses behavior in LPBF stainless steel 316L (SS316L) was investigated in several chosen sample planes by Smith et. al (2021) with contour method and neutron diffraction employed. Conducted measurements revealed residual stresses relief in the vicinity of sample base in three distinct orthogonal directions in addition to residual stresses relief due to diode heat treatment performed every 5 subsequent layers. Complementary numerical simulation showed to be in very good agreement with the gathered experimental data. The authors concluded that further LPBF process optimization in terms of decreasing stresses build-up could be achieved by tunning the number of subsequent layers to be heat treated together.

Application of neutron diffraction in selective laser melting residual stress measurements.

Neutron diffraction was used by Reid et al. (2017), who studied residual stresses in SLM Ti6Al4V components of different geometry notched samples fabricated under the same SLM process parameters [33]. Three samples with notches 60, 90, and 120 deg. were examined. Compressive stresses were found to decrease with the increase of notch angle, this was explained by greater constrain at the notch tip, preventing stress relaxation. Unexpected tensile stresses were observed on one side of the notches in the hatching direction of the built direction plane and virtually no stresses on the other, while the scanning direction stresses were found to be well in good agreement. Kim et al. (2017) evaluated stress-strain relationship of Al and Si phases in SLM AISi10Mg with crystal plasticity finite element method. The evolution of lattice strains in both phases, along scanning and built directions were simulated by this method using elasticplastic constitutive law [34]. Obtained results were verified by fitting residual stresses response and lattice strain measured in-situ with neutron diffraction to determine crystal plasticity for constituent phases of the studied alloy. Additionally, HR-TEM observations proved the existence of plastic deformations originating from stacking faults and mechanical twins in hard Si nanoparticles adjacent to the Al matrix.

SLM Inconel 718 elongated prisms produced with varying hatch length were studied by Nadammal et al. (2017) using electron back-scattered diffraction to determine the crystallographic microstructure of the obtained material, as well with neutron diffraction for assessing the residual stresses [35]. Upon their experimental results the authors drew a conclusion that hatch length used during the SLM process heavily impacts the microstructure and texture development. If increased by a factor of ten, the hatch length reduced texture intensity by a factor of two. This was explained by varying heat transfer rates depending on hatch length, one of key factors responsible for microstructure and texture development during the SLM process. Additionally, longer hatching length was observed to induce large, bulk residual stress gradients along scanning direction, the built direction residual stresses component on the other hand were highly compressive for shorter hatch length. The authors associate the thermal gradients accompanying the melting and solidification mechanisms with the development of a particular microstructure in the studied SLM components. In summary it was demonstrated that depending on hatch length one could tailor SLM components texture and that the use of shorter hatch lengths could be a suitable method for producing textured SLM parts.

Anderson et al. (2018) studied residual stresses of rectangular SLM Ti6Al4V parts of various layers thickness [36]. They showed that the increased layer thickness corresponds to reduced stress gradients in the studied samples but also scanning direction in increasing number of vector orientations can influence the stress field, making it a more uniform. Syed et al. (2019) studied residual stresses and direction dependent fatigue crack growth behavior in SLM Ti6Al4V in as built and stress relieved samples [37]. Results obtained by both neutron diffraction and contour methods stress measurement in compact-tension shaped samples were comparable, showing tensile stresses in the notch root area and at the free edges, with compressive stresses in the middle of the sample. The tensile stresses proved to produce higher fatigue crack growth rate for the samples in the as built condition, when compared to those after stress relief procedure. Heat treated ones in which tensile residual stresses were found to decrease by about 90%, followed by a decrease in crack growth rate. Furthermore the built direction also influenced growth rate but the trend was different in as built and stress relieved conditions. The samples built vertically exhibited the highest stress values, tensile residual stresses near the surface and compressive ones in the center were observed respectively. On the other hand samples built horizontally exhibited lower stress values, when compared to the vertically built ones. In addition microstructure of the SLM TiAl4V parts was determined using optical and scanning electron microscopy. The as built samples microstructure consisted of prior-beta grains aligned along built direction with a needle-like acicular martensite alpha that partially decomposed due to heat treatment into a more stable alpha+beta. Going further the heat treatment would render a crack growth rate determined by the microstructure and fatigue cracks to propagate along columnar prior beta grains. The authors concluded that residual stresses are a major factor behind crack formation in SLM Ti6Al4V but can be minimized by building the parts horizontally or/and heat post-treatment.

The use of synchrotron and neutron radiation for residual stress measurements in selective laser melted and laser powder bed fused components.

Both synchrotron X-ray diffraction and neutron diffraction can be used to determine residual stresses in SLM Inconel 718 alloys, as demonstrated by Kromm et al. (2018). In addition, the authors also used the laboratory X-ray diffraction method, as well

characterized the microstructure of the studied SLM parts [38]. Their work showed significant differences between obtained residual stresses measurement results depending on the technique employed. This in turn was suggested to be directly correlated with nonidentical penetration depths of each kind of radiation used in the conducted experiments, hence lead to a conclusion that a complex stress field exists in the studied SLM Inconel 718 elongated prisms. The surface residual stress values obtained by both methods for both the scanning direction and the hatching direction were found to be in good agreement with each other. Additionally, the synchrotron diffraction method enabled to uncover a stress gradient in the build direction plane along to distinguishable components in scanning and hatching directions. Going further neutron diffraction was used to determine bulk residual stress values in the studied samples and revealed a decreasing towards sample boundaries scanning direction residual stress component of tensile nature. The built direction component on the other hand was found to be compressive, while the value of the hatching direction component was found to be negligible. The authors concluded that the significantly lower values of neutron diffraction measured stresses present in the bulk were present due to stresses redistribution during subsequent layers deposition in the SLM process.

Elastic residual strain and stresses together with part deflation was investigated by Phan et al. (2019) in LPBF Inconel 625 bridge-shaped structures [39] by neutron and synchrotron diffraction and contour method. Part deflation was characterized using a coordinate measurement machine after leg separation from the build platform. Neutron diffraction enabled strain measurement along three principal directions, that were scanning direction, hatching direction and built direction. Strains along scanning direction, built direction and 45 deg. off the built direction were measured with synchrotron diffraction. Residual stresses in the scanning direction were measured using contour method both for the LPBF fabricated part and the built platform. Tensile strains were found in the built direction plane along scanning direction, which was consistent with the contour method results measurements of the regions separated from the build platform. Synchrotron diffraction measurement results revealed high value strains along the built direction, along with compressive strains along the scanning direction. Neutron diffraction measurements provided information facilitating calculation of residual stresses in the volume of the sample however it was noted that the values of the unstrained lattice spacing were prone to a slight systematic error due to zero normal stresses assumption in the near the surface region. Other than that, results obtained with all the stress measurement techniques showed to be in good agreement. The authors concluded that fabricated components properties such, as fatigue may be dependent on near-surface strains, stresses and techniques, such as XRD or contour method would be better suited for examining samples in those regions.

SUMMARY AND CONCLUSIONS

Additive manufacturing using techniques changed the way components are being produced giving unprecedented freedom of design combined with reduced costs and faster time-to-market delivery. Although revolutionary in a way that complex geometry parts can be produced still challenges lie ahead in better understanding the processes taking place during manufacturing of components for various mission-critical applications. One of the main issues to be resolved is residual stress state formation due to high thermal gradients while metallic powders get solidified. Residual stresses formation can lead to shape distortion of the produced components that in turn can result in other defects formation, such as cracks and delamination therefore it is necessary to carefully tailor AM process parameters, which has been demonstrated to be a non-trivial task. Materials of interest under active study included the following: Ti6Al4V (Ti64), used mainly in aerospace due to its high fracture toughness and corrosion resistance but also in biomedical applications due superior biocompatibility [40], Inconel type alloys [41] used in a wide range of high temperature applications due to excellent wear, fatigue and hot corrosion resistance combined with favorable weldability, stainless steels (SS316L), austenitic stainless steels (AlSi316L) due high tensile strength and low cost [42], but also studies on Ti553 could be found in the literature due to materials unprecedented tensile strength. It has been shown by numerous researchers that diffraction methods were a powerful tool for non-destructive testing of LPBF produced parts. Laboratory X-ray diffraction has been widely adopted for this purpose although it has some limitations, as only surface stress values could be studied. This of course could be potentially overcome by addition of electropolishing to map stresses layer, by layer. Fortunately, diffraction measurement applying highly penetrating radiation could be used to probe AM produced components material properties enabling both in-situ and ex-situ stress mapping capabilities at different sample depths. In-situ AM process parameters monitoring with synchrotron radiation also gained interest in recent years as it is shown by quite extensive body of work on the topic. Furthermore, synchrotron and neutron diffraction were employed to gather information later used in optimizing numerical simulations aimed at FE modeling of complex thermo-mechanics of the LPBF to reduce computational costs and time. Several process parameters were observed to influence produced part quality. These included laser scanning speed, laser energy density and the scanning strategy itself. Some of the works on the other hand, were focused on the role of support structures and parts build direction in the investigation of residual stresses, as well as stresses evolution upon build platform removal. Post heat treatment and in-situ heat treatment influence on distribution RSs were also studied.

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