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APPLICATION OF LABORATORY DIFFRACTION METHODS IN CHARACTERIZATION OF ELEMENTS MADE BY ADDITIVE SLM METHODS – STATE OF THE ART

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Abstract

The greatest challenge of widely developed incremental manufacturing methods today is to obtain, as a result of the manufacturing process, such components that will have acceptable strength properties from the point of view of a given application. These properties are indirectly determined by three key characteristics: the level of surface residual stress, the roughness of the component and its porosity. Currently, the efforts of many research groups are focused on the problem of optimizing the parameters of incremental manufacturing so as to achieve the appropriate level of compressive residual stress, the lowest possible porosity and the lowest possible roughness of parts obtained by 3D methods. It is now recognized that determining the level of these three parameters is potentially possible using experimental X-ray diffraction methods. The use of this type of radiation, admittedly, is only used to characterize the surface layer of elements, but its undoubted advantage is its easy availability and relatively low cost compared to experiments carried out using synchrotron or neutron radiation.

Keywords: X-ray diffraction, residual stress measurements, additive manufacturing, SLM
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1. INTRODUCTION

Additive manufacturing (AM) methods have evolved over the years into technologies enabling industrial production of complex geometry components. One practical AM technique for building metal, ceramic and composite parts i.e., for medical, aerospace, automotive applications is selective laser melting (SLM) [1] which uses high intensity laser beams to melt and fuse regions of powder bed, layer by layer in accordance with numerical code generated from CAD data. The key advantage of SLM is indisputably cheaper and faster production of complex geometries, sometimes not even achievable with traditional machining methods. Furthermore, the deployment of SLM requires a smaller machinery park, when compared to subtractive production methods. This in turn can extremely reduce equipment and logistics costs.

The major tradeoff of the SLM is that the produced parts are prone to residual stresses resulting from large temperature gradients [2] arising from rapid melting and fusing of the powder bed with high power lasers. In some cases, these stresses may lead to geometrical distortions [3] and heavily impact strength and fatigue life of the produced parts. Although SLM technology can produce almost full-dense parts another problem to be addressed is the porosity and surface roughness of the manufactured elements, that again can be induced by process parameters associated with laser operation [4] such as: scanning speed, scanning strategy, operation in keyhole regime, etc. that affect the melt, remelt, solidification and other processes taking place in the scanned powder bed.

To tailor all the process parameters necessary to produce components with the desired properties using SLM lot of experimentation is required, hence extensive resources, such as machine time to be devoted. An alternative approach to process optimization is through application of computational modelling to simulate preset physical phenomena leading to formation of stresses, porosity and surface roughness in such systems. This also has disadvantages, as to model all the physical phenomena and their complex relations, adding that they are taking place at the same time, during the SLM renders high computational costs. A solution may lie in combining simulations with experimental methods aimed at gathering the data about the stresses, porosity, surface roughness, etc. in real existing SLM systems and feeding it as parameters to the SLM process numerical simulation models to substantially lower the computational costs.

2. SCIENTIFIC METHODS USED TO MEASURE RESIDUAL STRESS

Scientific methods for residual stresses measurements can be divided into three categories: destructive testing, semi destructive testing and non-destructive testing. Destructive testing techniques include i.a. sectioning and contour methods [5], [6]. Semi destructive techniques include hole-drilling, ring-core and deep-hole methods [7–9]. Non-destructive methods include diffraction methods with a use of X-ray or neutron radiation [10–14]. The others are ultrasonic and nanoindentation methods or those applying Barkhausen noise phenomenon [15–17]. To cover all of them in detail is far beyond the scope of this work, as its aim is put on laboratory X-ray diffraction techniques, which have proven to be versatile methods for studying residual stresses levels. It should be added that non-destructive techniques such as X-ray and neutron diffraction are both standardized [18, 19], making them suitable for characterization of mission-critical components [20, 21]. In standard laboratory diffractometers the X-rays are generated by tubes with different anode target material [22]. This involves hitting a pure element with a beam of accelerated electrons. In the case of research facilities using radiation of higher penetration, the radiation is produced by means of a nuclear reactor as in the case of neutron radiation or is obtained as a result of bending the path of high-energy charged particles in the ring of the synchrotron [23]. Key advantage of laboratory X-ray diffractometry is that it is cheap, means it does not require neither expensive equipment nor big facilities however, synchrotron diffraction offers better spatial resolution and higher penetration depths. The key trade-off of neutron and synchrotron sources is the need of expensive equipment and big facilities. In the present work focus is put on laboratory, easily accessible equipment, as a relatively cheap powerful method for residual stress measurements.

3. RESIDUAL STRESS MEASUREMENTS OF SLM COMPONENTS

In 2014 Yadroitsava et al. studied residual stress levels in 316L steel samples fabricated by one zone strategy with 50% track overlap and found that the stresses along scan direction was about 1.2-1.7 times higher than in perpendicular direction [24]. This was followed in by a work by Yadroitsev et al. (2015) who studied directions and values of residual stresses remaining after printing in stainless steel 316 and Ti6Al4V samples with a combination of diffraction measurements and numerical simulation [25]. The stresses in Ti6Al4V were also studied by Morita et al. (2017) in components produced at 0, 45 and 90 degrees with respect to the build direction. It was found that compressive stresses of relatively small values were induced at the surface of the components but no relationship between them and fatigue strength of the components was found. To clarify if such a relationship existed the authors proposed to study a whole cross-section of the samples [26]. Simson et al. (2017) studied residual stresses in both build direction and in direction perpendicular to it in AISI 316L austenitic stainless steel samples produced with different SLM process parameters [27]. What was found is that stresses in studied materials heavily depend on parameters such as energy density during the laser powder bed fusion (LPBF) process. Furthermore, it was observed that the stresses' values are higher in the built direction of the studied samples. SLM produced Inconel IN718 elongated prisms were studied by Thiede et al. (2018) by both neutron diffraction and with laboratory X-ray diffractometry. The bulk and surface stresses in two directions: along the built direction and in direction perpendicular to it for as-built samples and with base plate removed were studied [28]. While surface residual stresses in the as built condition were proven to be constant, after the removal of the base plate the high gradients of the stresses along the built direction were revealed. The results were later correlated with the distortion maps obtained from tactile probe measurements. Neutron diffraction experiments revealed bulk residual stress gradients in all examined sample directions. These were correlated by the authors with temperature gradients and heat flow associated with SLM process parameters i.e., hatch length. Furthermore, it was observed that the bulk residual stress gradients partially disappeared after platform removal. Inconel 718 SLM produced components with two different support configurations were studied by Nadammal et al. in 2018 with optical microscopy, electron back-scattered diffraction (EBSD) and laboratory X-ray diffraction [29]. Based on the diffraction measurements the authors concluded that surface residual stresses develop in the as built samples dependent on the constraints imposed by both the chosen support configuration and the scanning strategy. Rosenthal et al. (2018) studied the heat treatment effect on the mechanical properties in AlSi10Mg alloys [30]. The authors confirmed that the origin of residual stresses in the AlSi10Mg alloy is related both to high thermal gradients due to fast scanning of the powder bed and an order of magnitude thermal expansion coefficients difference between Al and Si. They also argued that level of residual stresses in the studied alloy is influenced by the thermal history of certain areas like the melt-pool borders that undergo multiple cycles of fusion and refusion. This in turn could be directly correlated with the varying microstructure across the whole sample. A typical T5 treatment showed a substantial reduction of stresses in the studied samples, which was attributed to Al creep that moderated the stresses introduced during the SLM process. High temperature isostatic pressing (HIP) treatment resulted in the uttermost degree of RS relief in both Al an Si, as it erased the microstructure emergent from the SLM. Laboratory diffraction was used to validate the stress measurements obtained by three-dimensional digital image correlation (3D-DIC) by Bartlett et al. (2018) in SLM produced 316 L stainless steel samples [31]. A two-dimensional analytical model was developed in to convert the surface curvature measurements into in-plane stress estimates. A complex in-plane surface stress field was revealed by 3D-DIC by imaging the entire top area of the sample. Diffraction measurements were conducted in several chosen points of the sample. An average error of 6% was reported when comparing the developed model with the diffraction measurement results. Residual stresses generated by single point incremental forming (SPIF) of AlSi10Mg sheets produced by SLM were studied by López et al. (2018). Diffraction was employed for both the measurement of residual stresses induced during the SLM process and SPIF [32]. Varied levels of stress were observed in the studied samples which were attributed to effects induced by bending. Only compressive stresses were found both on the top and bottom surfaces of the AlSi10Mg as built samples. SPIF processing changed stress values from compressive to tensile in the volume of the sample. It was observed that after the samples reached 12 mm forming depth small cracks would appear due to SLM processes defects i.e., pores and overlapping melt-pools however the authors argued that the forming ability of the studied AlSi10Mg sheets could be further increased by eliminating the defects through adjusting the SLM process parameters and subjecting the sheets to the HIP densification process. Modification of stress in SLM produced AlSi10Mg cuboids through ultrasonic peening technique was studied by Xing et al. (2019) [33]. It was shown experimentally by diffraction that this technique can significantly change the nature of stresses in the additively produced AlSi10Mg alloy from tensile to compressive. Additionally, thermo-mechanical numerical simulations were caried out to model stress distribution in samples in as built state. Parameters used in the simulation were obtained with ultrasonic detector method. The effect of ultrasonic peening technique on stress distribution was also simulated, using the Johnson-Cook model fed with parameters obtained from the Split-Hopkinson pressure bar experiment. Diffraction stress measurement results were shown to be in good agreement with the finite element model simulations. The authors concluded that the influence of peening time and amplitude are the two major factors with which stresses can be influenced in the studied SLM parts at a depth from samples surface of a 2 mm maximum. Yakout et al. (2019) studied density and mechanical properties of Invar 36 and 316L stainless steel SLM cantilever beams, cylinders and cubes [34]. Diffraction method was used to determine stress values in the center points on the top and lateral surface of the cubes produced with at the same laser energy. 316L stainless steel samples showed higher stress values than Invar 36 ones in both measurement directions. All the measured stresses were of tensile nature, which suggested that they were of thermal origin, that were induced during the SLM build process and associated with the scanning lasers energy density. Stresses in both measurement directions increased with the increased laser energy density, which directly translates into higher temperature gradients, hence higher stress values, were observed. Horizontal, thermally induced stresses in the components produced by scanning the powder bed at higher, than critical laser energy densities exceeded yield stress values in both materials. This resulted in shape distortion taking a form of edge deflection, but it was demonstrated that the observed deformations were lower in Invar 36, when compared to 316L stainless steel samples due to lower residual stresses in Invar 36 SLM produced parts. A procedure for a more accurate determination of residual stresses in SLM produced components using laboratory diffraction was proposed by Fang et al. (2020) [35]. In their work the authors present a method of samples pretreatment with mechanical polishing and chemical etching to reduce surface roughness of SLM AlSi10Mg components, which can lead to lower stress values obtained via diffraction due to stress relaxation on the spiked surface, when spikes height is comparable to X-ray penetration depth. They argued that the proposed pretreatment method could be effectively used for other materials besides the studied alloy. What was also shown is that stresses were not evenly distributed on AlSi10g samples surface, given that the stresses in the built direction were from 1.5 to 2.0 times larger than in the hatching direction. Li et al. (2020) measured stresses in SLM Ti6Al4V alloy blade using X-ray diffraction in several points on the edges of the blades body pressure surface [36]. It was found that both the stresses on the surface determined by the built direction and on the lateral surface are of tensile nature however the latter were lower. Their maximum value was observed to be in the joint between the tenon and the leading edge of the blade. The minimal value of the stresses was observed at the blades top. Residual stresses in the blades body on the other hand, were reported to be compressive. Measured values were in good agreement with a thermal elasto-plastic model employed to calculate thermal stress distribution in the studied component. Influence on surface mechanical attrition treatment (SMAT) of SLM AISI 316L stainless steel was studied by Portella et al. (2020) in terms of microstructural, mechanical and tensile properties [37]. Stress measurements on parallel pipe-shaped samples were conducted with the combination of electropolishing and diffraction methods and revealed tensile stresses along the longitudinal and transverse directions of the built direction plane for as built samples to a depth of 200 μm. The authors showed that the SMAT treatment introduced compressive stresses in the studied samples. The values of compressive stresses peaked at a depth of 30 μm from the surface. Parameters of the SMAT process such as: time, diameter of the shots used and Almen intensity, when increased were confirmed to increase the compressive stress value and depth in the samples. Vishwakarma et al. (2020) explored the microstructure and tensile behavior of SLM M300 maraging steel with regards to the build orientation [38]. Both as built samples and samples subjected to solution and aging treatment were studied. In the as built samples significant amount of compressive residual stresses were found by diffraction. Residual stresses in heat treated samples were reported to be relieved due to thermal recovery. In addition, it was shown that with increased build orientation stresses increase due to smaller contact area with the build platform, hence worse heat dissipation. Nagesha et al. (2021) took a thermo-mechanical modelling approach to predict stresses in SLM Inconel 718 high pressure nozzle guide component HPNGV and compared their results with X-ray diffraction stress measurements [39]. Both FE model and diffraction results were shown to be in accordance however, a marginal difference was observed, which was attributed to simulated elements mesh size. Build platform removal resulted in increase of the stresses in the HPNGV component. This could be explained by the substantial thickness preventing shape distortion combined with stresses relief in the simulated and SLM produced part after base plate removal. Takase et al. (2021) provided information on lattice distortion effect and stress values in SLM unstable β-type Ti15Mo5Zr3Al by diffraction measurements performed in high resolution mode [40]. Obtained data revealed surface tensile stresses and numerical simulation of the SLM process pointed towards rapid cooling as being their cause. Partially stress relieved SLM samples were compared to the electron beam melted (EBM) ones that are known to possess negligible stress state, as reported in [41], which supported the suggested stress levels caused by rapid cooling during the SLM process to be the inducing the lattice distortion in the SLM Ti15Mo5Zr3Al. The authors point out that their finding is not consistent with the understanding of stresses as eliciting factors for lattice parameter change without lattice distortion. SLM Titanium grade 23 was studied to determine its mechanical properties and microstructure by Nikiel et al. (2021). Very high compressive stresses with strong anisotropy were revealed in the studied samples in the built direction plane. Thermal treatment almost completely erased stresses and weakened samples crystallographic texture. Additional mechanical polishing of the heat-treated samples enabled to obtain a surface with average roughness of ~1 μm and greatly reduced compressive stresses.

4. SUMMARY AND CONCLUSIONS

Additive manufacturing technologies enable unprecedented freedom of design and fabrication of custom parts for economy sectors where mission-critical components are required. However, still many challenges lie ahead before 3D prototyping and production methods such as SLM can truly disrupt industrial scale manufacturing ways of operation known for the last decades. This in turn heavily underlines the necessity for fast, reliable, efficient and inexpensive methods aimed at AM components material properties characterization promoting for a substantially better understanding and even higher degree SLM fabrication process optimization. Non-destructive testing (NDTs) techniques e.g., diffraction methods fulfill those requirements especially if applied to RSs mapping and analysis in SLM produced parts, as it was demonstrated that a substantial body of work has been published to date on the topic. It is to note that although laboratory diffraction techniques are very versatile tool for material properties characterization and can be used for other tasks besides stresses investigation i.e., phase composition analysis they have their limitations. Nevertheless, several complementary measurement techniques exist that can be successfully employed to gain a more thorough insight into the SLM process and the resulting components materials properties, which was briefly highlighted.

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