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In situ heat accumulation by geometrical features obstructing heat flux and by reduced inter layer times in laser powder bed fusion of AISI 316L stainless steel

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Abstract

Material qualification for laser powder bed fusion (L-PBF) processes are often based on results derived from additively manufactured (AM) bulk material or small density cubes, although it is well known that the part geometry has a tremendous influence on the heat flux and, therefore, on the thermal history of an AM component. This study shows experimentally the effect of simple geometrical obstructions to the heat flux on cooling behavior and solidification conditions of 316L stainless steel processed by L-PBF. Additionally, it respects two distinct inter layer times (ILT) as well as the build height of the parts. The cooling behavior of the parts is in-situ traced by infrared (IR) thermography during the built-up. The IR signals reveal significant differences in cooling conditions, which are correlated to differences in melt pool geometries. The acquired data and results can be used for validation of computational models and improvements of quality assurance.

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Keywords: selective laser beam melting; thermography; inter layer time; geometry; heat accumulation; preheating temperature; melt pool depth

1. Introduction

Laser powder bed fusion (L-PBF) is the most prevalent additive manufacturing (AM) technology to produce metallic components [1, 2]. Selective Laser Melting (SLM) and Laser Beam Melting (LBM) are widespread synonyms for L-PBF [3, 4]. The process offers huge potentials for the production of complex and lightweight structures and the integration of functional designs as well as the potential for mass customization and lead time reduction [4-6].

Nomenclature

L-PBF laser powder bed fusion ILT inter layer time RAE ratio of area exploitation

Recently, concerns about inconsistent or inhomogeneous mechanical properties of AM parts produced by L-PBF of the same material could be noticed in the relevant literature [3, 7]. Variability in microstructure of AM components as well as variability in defect existence and characteristics has been identified as a major challenge, especially for safety-critical

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components [8]. The comparability between L-PBF real part components which have often complex geometries containing different sized cross sections as well as volume leaps and standard test specimens is controversial [6, 8, 9]. There are many factors which are able to significantly affect the thermal history of a L-PBF component: not only variations in processing parameters, scanning strategies and support design affect the thermal history, also geometrical variations itself and variations in number of parts per build process do, which directly affects the ratio of area exploitation (RAE) and in turn the inter layer time (ILT) [6, 7, 10].

The thermal history of a part governs the development of the microstructure and is able to affect the occurrence and distribution of defects as well as residual stresses [6, 7, 11]. Hence, the mechanical properties depend on the thermal history during manufacturing [7, 12]. Temperature gradients and melt pool sizes as "inherent characteristics" of the L-PBF process depend on thermal behavior [13]. Consequently, the call for reliable and well documented relationships between process and properties, which includes the consideration of the real part geometry or structure, is expressed by many authors [3, 6, 9, 14, 15]. Additionally, small ranges of variations of process parameters in many studies, attempting to investigate the correlations between parameters and microstructure evolution, were criticized as they limit the process-microstructure correlations to a narrow band [16]. There are many studies focusing on the influence of process parameters and/or scanning strategies or post processing treatments on defect densities, microstructural development and mechanical properties [17-19]. However, only very few respect further aspects during manufacturing as inter layer times and part geometry on a large scale in terms of relevant built height for L-PBF processes.

To extend the existing picture of the process-propertyrelationship of the widely used and intensively studied stainless steel AISI 316L, this study investigated the rarely considered aspect of ILT and heat flux obstructing variations in geometry at still tall and massive specimens at once. The in-situ usage of

an infrared (IR) camera gave valuable insights into the thermal history of the growing parts.

2. Material and methods

2.1. Material and L-PBF processing conditions

A commercial AISI 316L stainless steel powder of spherical morphology in a sieving fraction of approx. 10 μ m to 45 μ m was processed on a commercial L-PBF single laser system SLM280 HL (SLM Solutions Group AG, Lübeck, Germany). The system was equipped with a 400 W continuous wave ytterbium fiber laser emitting at a wavelength of 1070 nm. The spot size was approximately 80 μ m in focal position. The laser melting process ran in argon gas atmosphere with oxygen content below 0.1 %. All parts were manufactured on a stainless steel substrate plate at a platform preheating temperature of 100 °C. Detailed information about the L-PBF system and the powder properties can be taken from [7], as the same configurations were used in this study.

2.2. Thermographic measurement set-up

The processes were partially monitored by a mid-wave infrared (MWIR) thermographic camera of type ImageIR8300 (InfraTec GmbH, Dresden, Germany) through a sapphire window in the ceiling of the building camber and two gold coated mirrors. The measurement set-up is illustrated in Fig. 1.



Fig. 1. Schematic of the thermographic measurement set-up.

The IR camera used a cooled InSb-focal-plane-array as detector and was sensitive in a spectral range from 2 µm to 5 µm. It was calibrated for blackbody radiation by its vendor. No additional filters were used. All optical elements were chosen to provide minimal and constant optical losses over the whole detection range of the camera; e.g., according to supplier's information, the transmission of the commercial sapphire glass (Edmund Optics Inc., Barrington, USA) was constantly greater than 90 % in the detection range of the camera. Due to these losses and the low emissivity of the build part surface or the recoated powder, the observed specimens showed less intensity than a blackbody would do at the same surface temperature. As a result, the apparent temperature values indicated by the camera underestimated the real temperatures systematically. As a correction could not be derived from the available data, the focus was set on a qualitative analysis in this work. To underline the fundamental distinction to the real temperatures the uncorrected apparent temperature values of the camera were referred as IR-signal values in this work. Nevertheless, as the real temperatures could only be underestimated, the real temperatures must be larger than the IR-signal values presented here. This discrepancy was discussed in more detail in [20]. The used camera calibration ranges varied between 125 °C - 300 °C, 300 °C - 600 °C and 450 °C - 1000 °C. Using a 25 mm objective a spatial resolution of approx. 420 µm/pixel was achieved. Using a frame rate of 600 Hz, and a region of interests of 160 pixels x 224 pixels, the observed field was approx. 68 mm x 94 mm large.

2.3. Specimen geometry, scanning strategy and selection of part sections for analysis

As specimen geometry, three types of cuboids of the outer hull dimensions $(13 \times 20 \times 114.5) \text{ mm}^3$ were built perpendicular to the build plate using an alternating meander stripe scanning strategy. The scan vectors proceeded parallel to the edges of the cuboid over the full length of the part without being split into different sections. The scanning pattern was rotated by 90° from layer to layer. The three specimens differed in the lower 30 mm of their build height with regard to their connection to the base plate, as can be taken from Fig. 2. Regarding the size of the cross section of the connection to the base plate the specimens are called "small connection" (1.3 mm x 2 mm), "medium connection" (3.9 mm x 6 mm) and "full connection" (13 mm x 20 mm) throughout the document.



Fig. 2. Specimens' geometry: (a) small connection to the base plate; (b) medium sized connection to the base plate; (c) full connection to the base plate. Yellow arrows sketch the scanning vectors and scanning strategy. Grey parts were taken for metallographic analysis. Green layers were taken for thermographic analysis.

All specimens were heat treated at 450°C for 4 h under argon gas atmosphere in order to relieve stresses. The specimens were cut into different parts, from which the grey parts (Fig. 2) were taken for metallographic analysis. Fig. 2 also highlights three layers in green color, for which comparisons of thermographic intensity measurements were analyzed in the results chapter.

The scanning parameters (laser power $P_L = 275$ W, $v_s = 700 \text{ mm/s}$ and hatch distance scanning velocity $h_s = 0.12 \text{ mm}$) as well as layer thickness (50 µm) were kept constant for all specimens and built processes. The three types of specimens were built in two series: The first one was built in a process, which had an ILT of approx. 65 s. This ILT will be called intermediate ILT. The second series was built in a process, which had an ILT of approx. 18 s. This ILT will be called short ILT. Apart from the differences in ILT between distinct build processes, a constant ILT over the entire build height of the specimens was ensured, despite the difference in RAE caused by the geometry. This was realized by scanning so called dummy parts using $P_L = 0$ W. The three specimens fitted into the field of view of the IR camera. Thermography data was analyzed by using the software IRBIS 3 (InfraTec GmbH, Germany) and Origin 2019 (OriginLab Corporation, USA). IRsignal-time-curves were able to serve as comparison mean between the respective conditions. The average values of five manually chosen pixels located in the center line of the respective cross section were used for each layer of investigation. Within each part, IR-signal-time-data were

analyzed at three distinct layers (layer 752, layer 1506, layer 2260) which represented different build heights during the process. More information on this evaluation method can be found in [7].

2.4. Metallography

For each specimen two parts were prepared for metallographic analysis (grey parts in Fig. 2), hereafter called upper and lower part. By cutting these cuboids into three sections (first cut: bisection; second cut: bisection of the left half, illustrated in [7]) two metallographic planes of each part were prepared for light-microscopy (embedding, grinding, polishing). Polished but unetched cross sections were taken for optical light microscopy in order to determine porosity by grey value analysis using the software IMS Client V17Q1 (Imagic Bildverarbeitung AG, Glattbrugg, Switzerland). A threshold value of 115 was chosen for all cross sections. Cross sections were then color etched using Beraha II etching detergent for 10 s to 30 s. Melt pool depths were determined at the upper part of the specimens, by measuring ten melt pools of the last exposed layer. To get information about melt pool depths in lower sections of the specimens, further production of these respective specimens is planned.

3. Results

3.1. Macroscopic results

Tempering colors on the upper surface of the specimens were noticed for the specimens of small connection and medium connection in the short ILT process during the manufacturing of the specimens. These effects varied in intensity (color and time of glowing after laser exposing) during the process, which was observed qualitatively by the machine operator. Additionally, the depowdered specimens showed tempering colors in different characteristics at their surfaces. The upper surface of the specimens manufactured with intermediate ILT did not show visible tempering colors. However, the full connection specimen manufactured at short ILT showed a brownish color and the small and medium connection specimens showed a grey-bluish color. Variations in tempering colors over the built height were noticeable.

3.2. Thermographic results

Fig. 3 show the IR-signal-time-curves for all specimen geometries and ILT. For comparability the illustrated IR-signal-time-curves of each individual specimen were shifted in the manner, that the measured maximum IR-signal value (for short ILT) or the first value of the maximum value plateau (for intermediate ILT) were set as t = 0. The following trends could be observed: Reducing the ILT led to a strong increase of the IR-signal values for all analyzed layers and specimens. With increasing size of the connection lower IR-signal values were measured, whereby the effect decreased with increasing layer number. An increase of IR signal values of the full connection specimen could be observed over the complete build height.



Fig. 3. IR-signal-time-plots of complete parameter matrix. Three lines indicate the three different geometries: small connection (blue), medium connection (yellow), full connection (green). Values of three different build heights are presented for two different ILT (left: intermediate ILT, right: short ILT). Grey (white) areas indicate temperatures outside (inside) the used calibration range.

3.3. Metallographic results

Differences in melt pool geometries of the top layer were observed as exemplified in Fig. 4. The mean values of the depth of ten measured melt pools per specimen were plotted in Fig. 5.

For intermediate ILT no significant differences in melt pool depth between the distinct specimens were measured. However, for short ILT specimens, the melt pool depth varied significantly in comparison to the intermediate ILT specimens. Furthermore, within the short ILT specimens a significant variation between the geometrical different specimens was revealed. No clear difference could be noticed between small and medium connection but a huge increase in melt pool depth in comparison to the full connection specimen.

The porosity determination by grey value analysis at two cross sections per part indicated for nearly all parts a very low level of 0.1 % porosity. Only the porosity value in the lower part of the small connection type specimen manufactured at short ILT showed a slightly increased porosity of 0.5 %. Detailed analysis by micro computed tomography will be conducted in a subsequent study.



Fig. 4. Color etched cross sections showing melt pool geometries at the topmost layer for the small connection specimen at intermediate ILT (left) and short ILT (right).



Fig. 5. Measured melt pool depth at the topmost layer, i.e. at built height of 114.5 mm.

4. Discussion

The analysis of the manufactured specimens by means of melt pool measurements, porosity determination and in-situ IR measurements clearly stressed the influence of part geometry and ILT, and as worst case scenario a combination of detrimental conditions of both factors combined, on the thermal history of parts manufactured with a type of standard processing parameters. The heat accumulation could be qualitatively retraced in the tempering colors already. However, inner surfaces of complex real parts cannot be visually analyzed.

When considering the IR-signals as apparent surface temperatures in °C of the observed solidified parts as in [20], which would be assumed to underestimate the real temperatures, the process inherent preheating effect of the growing part for the short ILT got obvious. As could be seen from IR-signal-time-plots, the parts did not cool down to a level of the pre-set platform temperature in the respective build heights, as the IR-signal values prior to exposing are well above the starting values in these cases. This consideration neglected any differences in emissivity of powder and solidified bulk AM material. However, the general aspect of an elevated surface temperature prior to recoating and prior to exposing remained valid. A quantification is planned in calibration experiments by the authors. As variations in emissivity are one of the most important sources of measurement uncertainty in thermographic measurements [21] and are highly dependent on surface condition [22], the comparison of the IR-signal-timeplots had to be considered very carefully. The observed tempering colors as a result of oxidation might have influenced the emissivity. However, under the assumption of higher emissivity values of oxidized steel surfaces compared to nonoxidized surfaces [23], the revealed differences in apparent temperature would get even increased, as the degree of underestimation would be higher for higher emissivity values.

The observed preheating effect increased by geometrical heat flux obstructions as the small and medium connection geometries constituted. The resulting changes in melt pool sizes represented most likely not the maximum variation within these parts, as the melt pool sizes could be measured only at the topmost layer, which was not the layer of the maximum preheating temperature as indicated by the thermographic results from the two other layers of analysis. The occurrence of more spherical pores in the lower part of the small connection specimen manufactured with short ILT was an additional indicator that the melt pool sizes were probably even stronger increased in these regions. This will also be analyzed in detail in a subsequent study.

Moreover, according to [7], it can be assumed that a change in melt pool size went along with a change in sub grain sizes. This can be explained with changes in constitutional undercooling of the melt during solidification, which changes with varying preheating temperature [24]. A grain size decrease for higher cooling rates was reported by Zitelli et al. [25]. Bertoli et al. [26] pointed out that the size and morphology of the resulting microstructure is a function of temperature gradient within the liquid phase and velocity of the solidification front. At least the former will, therefore, be influenced by preheating temperatures. A detailed analysis of microstructure will be conducted in a subsequent study.

The authors of this study were aware of industrial partners who do not want to conduct recrystallization heat treatments for AISI 316L components. Seifi et al. reported the industrial desire (aerospace and biomedical) for as-built materials even in high cycle fatigue (HCF) critical applications [15]. These industrial inputs emphasized the importance of knowledge about the presumed microstructural variations within a L-PBF part and opportunities of monitoring. Furthermore, recent research goals in L-PBF regarding tailored materials by control of microstructure during the built up of an AM part could be noticed [5, 27-29]. The knowledge about the current surface temperature of the growing part is mandatory for any kind of pre-calculations in this direction. For these examples variations over build height and through changing ILT or geometries has to be taken into account as the results of this study clearly indicated.

Lastly, off-axis thermographic measurements of surface IR radiation of already solidified material seems to be a valuable mean for the definition of critical locations within a part. They do not require a high temporal resolution, which can reduce costs for equipment and computational time as well as data storage. Seifi et al. [15] considered the potential impact of in-situ process monitoring and control as extremely high for qualification and certification of AM parts. In addition, they considered the lack of adequate process monitoring methods as a bottleneck for more widespread applications of AM technologies [15].

The IR thermography appeared to be a useful tool for retracing the thermal history of a part and to give answers to questions regarding the characterization of location-specific properties and the relationship of properties between small laboratory specimens and large real part components. The answers are key for the application of safety critical AM components, as Shresta et al. [12] and Gorelik [9] stated very clearly. Numerical models can be helpful for this duty [13]. Optimization of the manufacturing process as well as quality control are both related to a sufficient determination of temperature profiles and melt pool sizes. The acquired data can assist with the validation of computational models.

5. Conclusions

In this study, the relevance of the interplay between part geometry, build height and ILT for properties and quality of the resulting product was highlighted. A process inherent preheating effect of the growing part, which increased with geometrical heat flux obstruction was experimentally shown. The occurring heat accumulation resulted in raising surface temperatures and affected the melt pool sizes significantly. Some indications of effects on defect density (porosity) were also presented. Assumptions of effects on the microstructure, especially on the sub-grain sizes, were discussed. The knowledge about the current surface temperature of the growing part appeared to be mandatory for a quality assurance. In-situ thermographic imaging demonstrated its ability to retrace the thermal history of a part. Further research questions were identified.

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