TEMPERATURE HISTORY WITHIN LASER SINTERED PART CAKES AND ITS INFLUENCE ON PROCESS QUALITY

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Abstract

The temperature distribution and history within laser sintered part cakes is an important aspect regarding the process quality and reproducibility of the polymer laser sintering process. Especially the temperature history during the build and cooling phase is decisive for powder ageing effects and the development of part quality characteristics. In this work, a measurement system for three-dimensional in-process temperature measurements is set up and the influence of different parameters on the inner part cake temperature distribution and history is analyzed. Important factors are not only geometrical build job parameters like the part packing density and build height, but also process parameters like the layer thickness and bulk powder density. Individual in-process temperature profiles at different positions within a part cake are finally correlated with powder ageing effects. The results of this work help to understand the temperature history dependency of powder and part properties and can therefore be used to develop optimized process controls.

Introduction

Polymer laser sintering is an important additive technology for the rapid production of individual products and complex parts like functional prototypes and end-use parts. However, the process faces challenges regarding the reproducibility of part quality characteristics. A possible reason are temperature inhomogeneities during the process. Thereby, energy input is performed on the powder bed surface by infrared heating and laser energy, while energy output is represented by the cooling process. In literature, the scattering of part quality features is mostly correlated with inhomogeneous temperatures during energy input on the powder bed surface. [1-4]

Although also the cooling process influences part properties, there is only very few knowledge about the inner part cake temperature history, how it is influenced and how it affects the process quality. Since polymer powder has a very low heat conductivity, thermal processes are very slow and temperature gradients during cooling are very high [5-6]. In this work, the inner temperature distribution is analyzed dependent on the used layer thickness and powder bed density. Exemplarily, temperature profiles and histories during the build phase are presented. Individual, position-dependent temperature histories are also correlated with powder ageing effects.

The following chapter about the state of the art describes the influences on the inner part cake temperature distribution and history as well as its impact on part and powder properties. Based on literature studies and previous investigations, the most important influencing factors are figured out. Then, the experimental methods for the measurement of the inner part cake temperatures as well as the determination of powder age by melt volume rate (MVR) is presented. The results of the measurements are shown and discussed afterwards. Finally, this work is summarized and an outlook to further work concerning related topics is given.

State of the Art

In order to analyze the influence of process parameters onto the temperature distribution and history within the part cake, an analysis of the whole laser sintering process chain has been performed. Based on previous investigations and literature studies, the main potential influencing factors are figured out. A summary of these factors is given in figure 1. The parameters are classified into build job layout, process parameters and machine parameters.

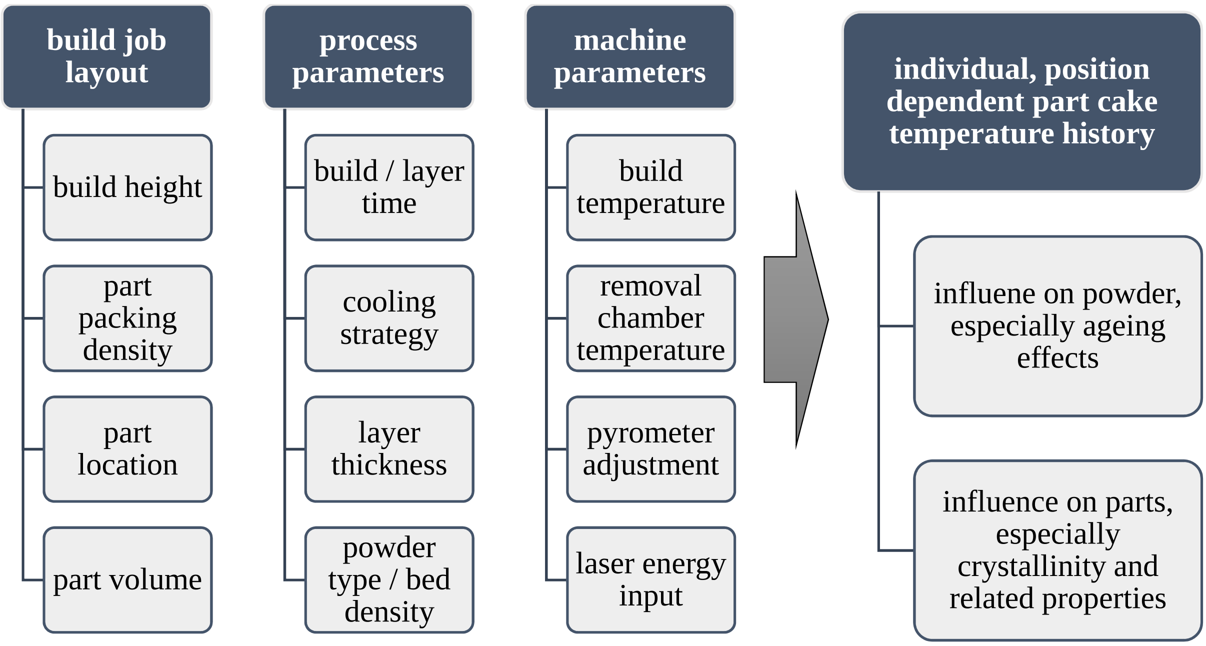

Figure 1: Main parameters influencing the inner part cake temperature distribution and history

Regarding the build job layout, important parameters are the build height and the part packing density (volume of parts compared to the whole part cake volume). The higher the build job, the longer the process and cooling times. Built parts also induce energy into the part cake, which changes the powder temperature especially in close proximity to the parts. The shape and location of the parts also influence the temperature distribution: strong powder caking is especially observed between close and near voluminous parts. Parts located in outer regions also cool down faster than parts located in the part cake center. [6-8]

Considering process parameters, the build and layer time is a direct outcome of the build job layout and selected layer thickness. Another aspect is the cooling strategy, which may be a combination of cooling phases in a controlled environment and cooling phases at ambient conditions. Most intense temperature gradients within the part cake are observed during cooling. Also the core temperature, at which the parts are unpacked, is relevant. One more important factor is the type of the used material: The heat conductivity of the bulk powder is strongly influenced by the powder bed density and thereby also by the recoating mechanism and compaction of the powder layers. Since temperatures within the part cake change only very slowly, also small changes of the heat conductivity may have a significant influence on the temperature history. In addition, the type of the base material itself and additives like aluminum also change the thermal properties of the bulk powder. [5-6, 9-11]

Regarding machine parameters, the setting of the build and removal chamber temperature (or frame temperature) are the most obvious influencing factors since they directly change the boundary temperatures of the part cake, especially during the build phase. In addition, also the adjustment (measurement location, cleanliness) of the pyrometer as basis for the temperature regulation may have impact on the preheating temperature. Lastly, the volume energy of the laser (result of layer thickness, hatch distance, laser power and scanning speed) influences the melt temperature and thereby also the heat transfer into the part bed. [12-13]

In the end, a combination of all described parameters results in individual, position dependent part cake temperature histories during build and cooling. However, these temperature histories have not been analyzed in detail and correlated with the process quality yet.

Concerning the unmolten powder, long exposure times at high temperatures lead to an increase of the average molecular chain length. Used powder has to be refreshed with new powder to ensure a constant material quality for application. A poor powder quality results in surface defects on built parts and also in a slight reduction of mechanical properties. To measure the powder age, several methods have been proven: Due to the increased molecular chain length, the melt flow rate (MVR) decreases and the solution viscosity increases. Ageing effects on the particle level also lead to a reduced flowability of the bulk powder. In literature, the effect of time and temperature on the powder age has been investigated extensively. Thereby, ageing experiments were performed in laboratory ovens and using different laser sintering systems. However, oven and “real” ageing do not correlate well if a constant machine temperature is assumed. Considering machine ageing, it has been shown that the powder age depends on the position within a part cake, with a trend to stronger ageing effects in the center of the part cake and reduced ageing in outer regions. Also the influence of the layer thickness and job height on the powder age has been analyzed, with a trend to stronger powder ageing at longer process times due to higher build jobs and lower layer thicknesses. Nevertheless, the individual temperature history during the laser sintering process has not been analyzed and correlated with powder ageing effects yet. This knowledge is essential to specify and predict individual in-process ageing effects. [9, 14-18]

The influence of individual temperature histories on produced parts is more complex due to its interdependency with the laser energy input. Mostly, a poor reproducibility of part quality characteristics is explained by and correlated with inhomogeneities on the powder bed surface. These temperature gradients depend on the machine type and used heating systems. The inhomogeneities on the powder bed surface may also “extend” into the inner part cake. Less focus is on the energy output yet: the cooling of parts inside the part cake. During consolidation, material properties develop dependent on the cooling rate. Since large temperature gradients are expected within a part cake, warpage effects can be explained by inhomogeneous cooling and crystallization rates. Another aspect are cooling rate or position dependent shrinkage effects. [1-4, 19-23]

However, it is still a challenge to differentiate effects that are based on the energy input and effects that have to be traced back to the energy output. Also a validated information, in which state of the process decisive crystallization effects occur, is missing. Knowledge about the temperature history below the build area is expected to help understanding these effects. In past studies, measurements of inner part cake temperatures have been performed at only few selected positions or only during specific phases of the build process [21, 23]. Therefore, the authors implemented an advanced temperature measurement system within a laser sintering system [6].

Experimental methods

To specify the inner part cake temperature distribution and history, a temperature measurement system has been implemented within an EOSINT P395 laser sintering system from EOS GmbH, Krailling, Germany. The system allows a three-dimensional temperature measurement during the build and cooling phase using a high sensor density of 48 sensors within 1/8 of the build area. The measurement principle is shown in figure 2a [6]. Main elements are 6 sensor bars (figure 2b), each equipped with 8 thermocouples, attached to brackets at the bottom of the build frame. Holes are drilled into the build stage to allow the sensor bars to penetrate into the part cake from beneath. During build, the relative position of the sensors changes within the part cake; during cooling, the positions are fixed. The thermocouples are attached to two measurement devices Expert Key 200L from Delphin Technology, Bergisch Gladbach, Germany. An exemplarily temperature history is given in figure 2c. The overall history starts with the recoating of the powder and can be divided into the build and cooling phase.

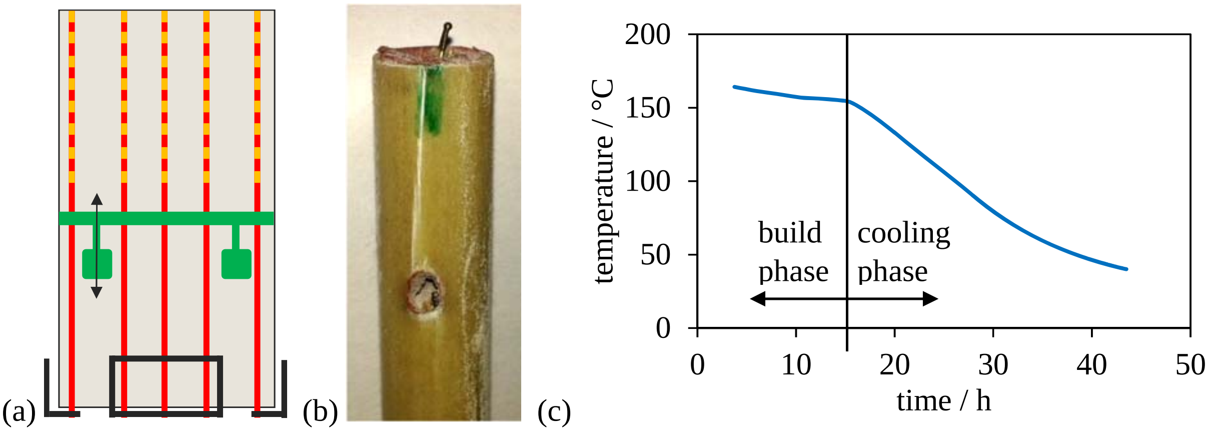

Figure 2: (a) schematic sketch [6], (b) close-up photo of a sensor bar, (c) exemplarily temperature history of the powder during build and cooling

In this work, the influence of the layer thickness onto the inner part cake temperature history is analyzed. Therefore, build jobs with a build height of constant 300 mm and the layer thicknesses 60 120 and 180 µm were built. The material used is PA 2200, a standard polyamide 12 laser sintering powder from EOS GmbH, Krailling, Germany. In this work, 100% recycled powder is used, since it has been shown before that the powder quality dies not affect the temperature history significantly. Also, no parts are included in the job so far. The build temperature is adjusted according to EOS working instructions and given in table 1. Due to the different total number of layers, the build time and build speed changes significantly for the different layer thicknesses. Also, different recoater blade shapes are used. It is already known that the combination of different blade shapes and layer thicknesses results in individual powder bed densities. So, the effectively investigated parameters are the build time and the powder bed density – adjusted by layer thickness. Each job is performed twice to reduce statistical deviations.

In addition to the parameter investigation, the influence of individual temperature histories on powder ageing effects is analyzed. This job is performed using a layer thickness of 120 µm and 50 % refreshed PA 2200 powder with an MVR of ~ 31 cm³/10min. After build and cooling, powder samples are removed from the part cake at exactly the same positions of the temperature measurement. No powder boxes were applied; the powder is directly picked out of the part cake. The samples are tested regarding the melt flow rate according to DIN EN ISO 1133 and EOS working instructions on a Zwick Mflow melt flow tester (235 °C, 5 kg, predrying for 30 min at 105 °C (air)).

build height	300 mm		
powder	PA 2200 layer thickness test: 100% recycled, MVR ~ 10.0 cm^3/10min powder ageing test: 50% refreshed, MVR ~ 31.5 cm^3/10min		
layer thickness	60 µm	120 µm	80 µm
build temperature	178 °C	180 °C	182 °C
recoater blade shape	flat	round	pointed
build time	~ 35 h 20 min	~ 14 h 45 min	~ 9 h 15 min
build speed	~ 8.5 mm/h	~ 20.3 mm/h	~ 32.4 mm/h
Removal chamber temp.	130 °C		
cooling phase	10 h within machine (nitrogen) + >24 h outside (standard atmosphere) until <50°C		
part packing density	0% → no parts		

Table 1: Process and job parameters for the test jobs

Results & Discussion

At first, the results of the layer thickness analysis are shown. Figure 3 shows the inner part cake temperature profile in z-direction during cooling. The data is taken from the sensor bar in the middle of the build area. In the diagram, the right three curves represent the initial temperature profile directly after the build job ends (cooling time: 0 h). The curve for the build job with 180 µm is the hottest, while the profile for 60 µm is the "coldest". The differences between the curves are significantly higher than the difference of the build temperatures. So, the effect of different build times is obvious: Since the material has more time to approach the removal chamber temperature, it cools down stronger during the build phase. In contrast, the build job with 180 µm is hotter, but faster. At the bottom, all jobs approach the same temperature during build. Due to the close proximity to the removal chamber, the powder almost reaches its temperature level of ~ 145 °C (set: 130 °C).

During cooling, another effect is observed regarding the cooling rate for the different layer thicknesses: the order of the curves changes. Although the build job with 60 µm starts cooling with the lowest temperature, it continuously moves to higher temperatures compared to the other layer thicknesses. Since the boundary conditions are the same for all jobs, it can be assumed that the heat conductivity has changed due to different powder bed densities induced by different recoater blade geometries. An additional measurement of the powder bed density determined via built powder boxes with a defined volume revealed the following:

60 µm – 415 g/l   120 µm – 431 g/l   180 µm – 428 g/l

Although the differences of the powder bed densities are very small, the effect is significant and reproducible: The build job with 60 µm is up to 10 K hotter during cooling (part cake center) and the required cooling time for cooling down to max. 50 K is about 4 h longer compared to the job with 120 µm. Even though this is only a rough analysis, it hints to a strong influence of the powder bed density on the heat conductivity and thereby also on the cooling rates.

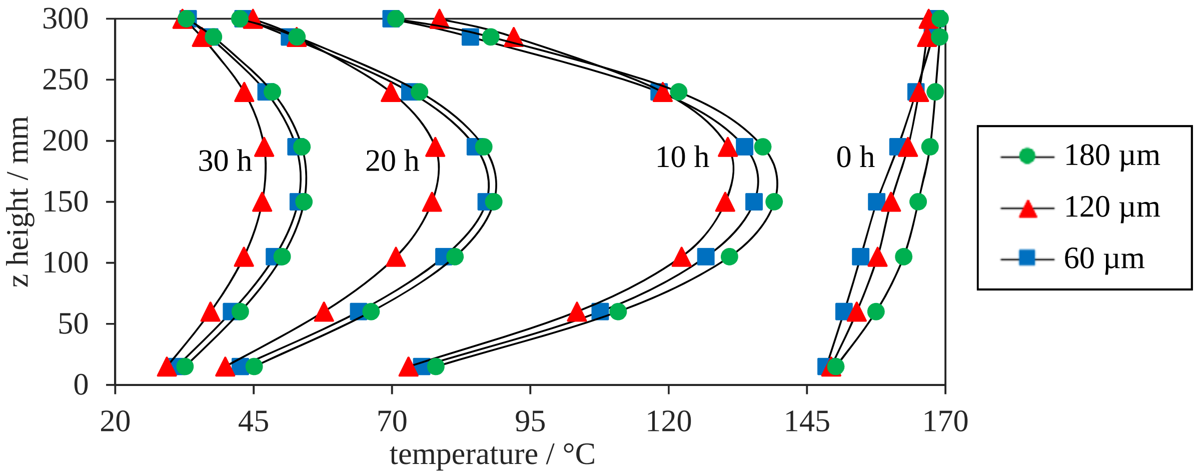

Figure 3: Inner cooling profile in z-direction for different layer thicknesses

Analyzing the temperature profile within the X/Y plane during the build phase, figure 4 shows the temperature history exemplarily for 120 µm layers 60 mm above the build stage. The top curve starts at a total build height of 75 mm, meaning that the powder already cooled down for 45 min or 15 mm build height respectively. The trend shows a slight increase of the temperature from the X/Y center (position 1) until a diagonal distance of 85 mm (position 2). Closer to the frame edge, a drop of temperature is detected (position 3). The trend in the center area fits to the temperature distribution onto the powder bed surface, where the temperature at position 1 is about 2 K cooler than at position 2, while the temperature at position 3 is heavily influenced by the environment.

When the powder cools down during build, the slope of the temperature profile drops continuously from the center to the frame edge. This means that inhomogeneities on the powder bed surface only extend until a defined time (or further build height) into the powder bed. Then, the influence of the environment is stronger due to in-build cooling effects. This effect is also detected for temperature profiles in other build heights. Here, the top surface inhomogeneities seem to be equalized after about 15…60 mm at temperatures of about 162 °C.

Another finding is that the total temperature drop during the build phase is about 10 K within the examined area 15 mm below the powder bed surface. To have a closer view, figure 5 shows the temperature histories at position 2 for different z heights. The bottom curve is the one of the powder that is applied after 15 mm build height. Compared to the build temperature, the temperature decreases quickly and then slowly approaches the removal chamber temperature (~145 °C). Powder applied in higher z heights cools down significantly slower and remains for a longer time at high temperatures. A possible reason is be the built powder acting as isolation to the cooler build frame. As a consequence, the temperature 15 mm below the surface (first data point of all curves) depends on the total build height and is only constant after about 105 mm. This effect may explain why warpage of parts is most intense in the lower area of the part cake. Temperatures within the top 15 mm of the part cake are not known and therefore marked dashed.

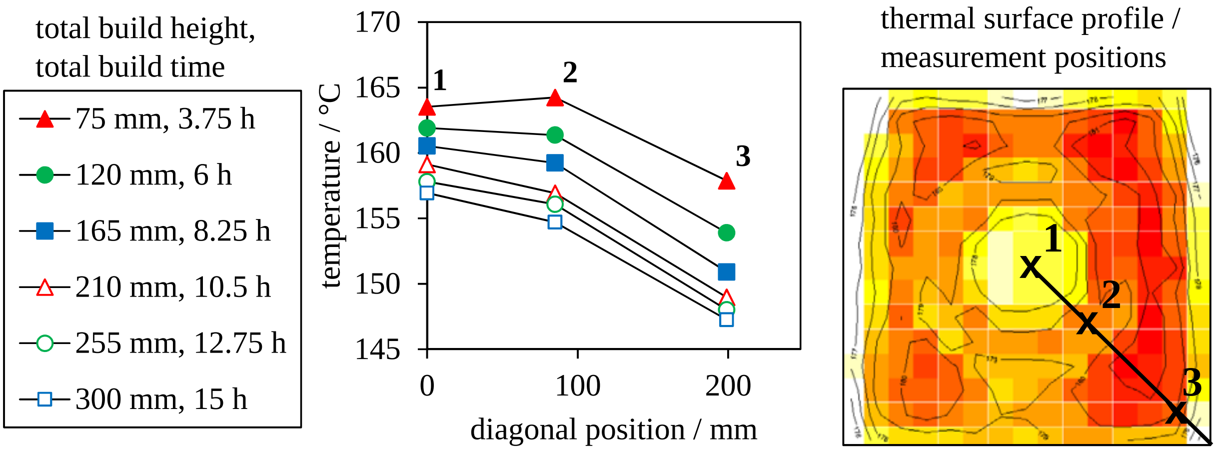

Figure 4: Inner X/Y cooling profile during build (constant distance from build stage: 60 mm)

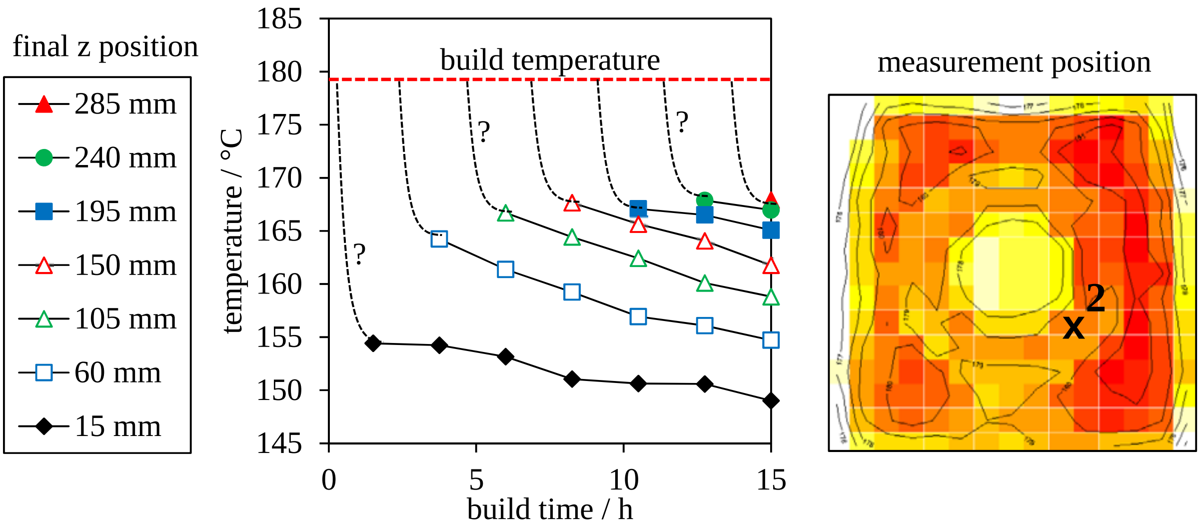

Figure 5: Powder temperature history during build as function of z height

Finally, individual temperature histories are correlated with powder ageing effects. Figure 6 shows the melt volume rate for different positions within the part cake. The powder was extracted at the exact positions of the temperature measurement. In general, the MVR decreases from the initial (mixed powder) value of 31.5 cm^3/10min down to a range of 2…14 cm^3/10 min, meaning that the sample position has a significant influence on the resulting ageing effect.

Interestingly, powder ageing has been found to be stronger in the top area compared to the bottom area for all tested positions. The strongest ageing is found in the center of the part cake. For the inner two positions 1 and 2, the ageing intensity decreases from the z center to the top and bottom, while the curve for the outer position 3 increases continuously with build height. These effects are in contrast to the expectations and also to other work in the field. Although powder from the bottom remains much longer within the process, it shows a less intense ageing than powder that is applied only shortly before the job finishes. An explanation may be the dependency of the individual temperature history on z height during the build phase (figure 5). While the powder in the lower area cools down quickly to removal chamber temperature (~ 150 °C), powder from the top area remains significantly longer at temperatures of about 160…170 °C. This leads to the assumption that the variation of position dependent in-process ageing intensities has to be traced back to exposure times at temperatures between ~ 150 °C and build temperature; lower temperatures have a less significant impact considering the overall temperature history.

The final MVR of a powder sample is the result of its individual in-process temperature history. Process ageing is therefore difficult to compare with isothermal oven ageing experiments. For a holistic analysis of powder ageing effects, the influence of powder position, build job layout and process parameters has to be taken into account. Other machine types, for example with additional heating systems within the build frame, may show different temperature histories and thereby also other powder ageing distributions.

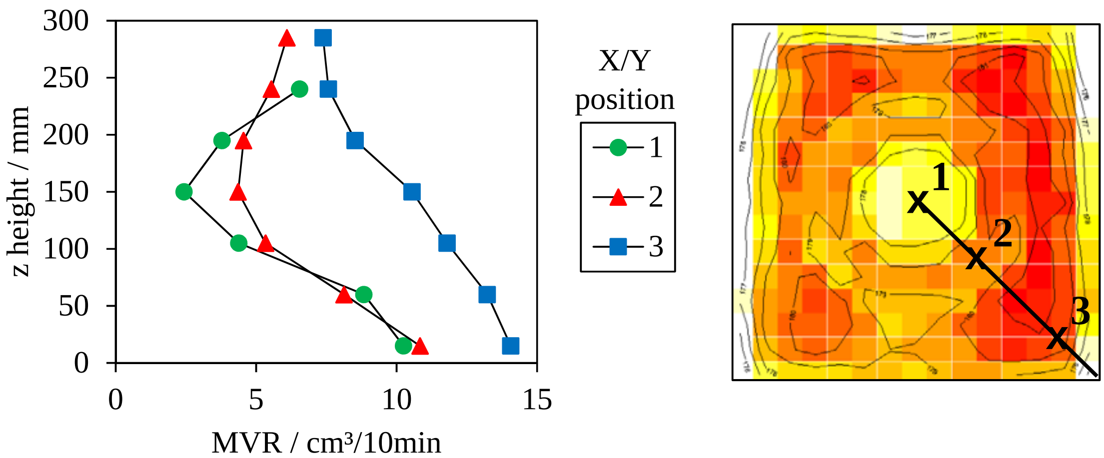

Figure 6: Position dependent in-process powder ageing determined by MVR

Conclusions & Outlook

In this work, the main influencing factors on the inner part cake temperature have been figured out. Experimentally, temperatures have been measured three-dimensionally during the build and cooling process. For example, the powder bed density changed by different layer thicknesses has a significant influence on resulting cooling rates. Temperature inhomogeneities on the powder bed surface are detectable within the part cake, but only until a defined time or additional build height. A combination of process parameters and build job layout finally results in individual, position dependent temperature histories. Furthermore, heat flux through the build stage occurs already during the build phase, resulting in different cooling rates dependent on z height. In-process powder ageing is significantly influenced by this effect: For example, powder from the lower part cake ages less intense than powder from the upper part cake. This leads to the assumption that the most relevant ageing effects occur at temperatures between about 150 °C and build temperature, resulting in completely different ageing intensities within one part cake.

In future work, an investigation of the influence of more parameters on the part cake temperature distribution and history will be performed. Especially the impact of built parts on the part cake temperature is in focus. Also the ageing kinetics will be investigated in greater depth and linked to individual process temperature histories. Another focus is on the impact of individual temperature histories on built parts, in particular the crystallinity and related characteristics like the mechanical properties, dimensional accuracy and warpage.

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