Master Thesis

# Development of Laser Powder Bed Fusion parameters and their effect on microstructure formation in Low Alloyed Steels

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# Preface

This work was done in the frame of the Centre for Additive Manufacturing – Metal (CAM$^2$) at Chalmers University of Technology, supported by the Swedish Governmental Agency of Innovation Systems (Vinnova). It was connected to the PhD student project of William Hearn, which is focused on the development of ferrous alloys for Laser Powder Bed Fusion. The presented MSc Thesis provided support to the LPBF process development trials done by William Hearn, by means of conducting metallography evaluation of the produced components.

# Abstract

Additive Manufacturing (AM) is a process that allows for the production of 3D parts based on a CAD-model. This provides noticeable freedom of design that cannot be realized when using traditional subtractive manufacturing (i.e. machining) or casting processes. One of the most prominent AM technologies is Laser Powder Bed Fusion (L-PBF), which uses thermal energy to selectively fuse regions of a powder bed together. Currently in L-PBF there are fewer than ten ferrous alloys that have been approved for commercial use, and of those alloys they are either stainless or tool steels. Meaning that there is a lack of ferrous alloys that can be used for structural applications. This work focuses on the processability of a variety of Low Alloyed Steels via L-PBF. The goal was to define the printability of said alloys by determining the optimal process windows that allow for the production of high density, defect-free parts. To do so the microstructures of the mentioned Low Alloyed Steels were examined to identify the types of defects that can form, as a function of both, the processing conditions and alloy composition. By doing this analysis a process window can be obtained that allows for the production of high density, defect-free ferrous alloys that can be used in structural applications.

## Zusammenfassung

Über den Prozess der additiven Fertigung können 3D-Teile direkt auf Grundlage eines CAD-Modells gefertigt werden. Dadurch ergeben sich Designmöglichkeiten, die mit klassischen subtraktiven Fertigungstechniken oder Gusstechniken nicht umsetzbar sind. Eine der am weitest verbreiteten Techniken ist die Laser Powder Bed Fusion (L-PBF). Dabei wird Material durch die thermische Energie eines Lasers verschmolzen. Derzeit gibt es weniger als zehn Eisenlegierungen, die für die kommerzielle Nutzung in L-PBF von Equipment Herstellern zugelassen sind und bei allen handelt es sich um rostfreie oder Werkzeugstähle. Aufgrund des Fehlens niedrig legierter Stähle behandelt diese Arbeit die Verarbeitbarkeit einiger kommerziell erhältlicher niedrig legierter Stahlpulver. Es wurde der Einfluss der Legierungszusammensetzung und der Prozessparameter auf die Mikrostruktur der produzierten Teile untersucht. Dadurch wird der Zusammenhang zwischen Porosität und anderen Defekten zur volumetrischen Energiedichte geschaffen, welcher in weiterer Folge verwendet werden kann, um Prozessparameter von einer L-PBF Maschine auf eine andere zu übertragen. Es wurden durch diese Analyse Prozessfenster für die untersuchten Legierungen gefunden, in denen hochdichte Teile ohne Defekte produziert werden können. Außerdem konnten die Einflüsse der Zusammensetzung des Stahlpulvers und des Transfers zwischen L-PBF Maschinen auf das Prozessfenster gezeigt werden.

## Acknowledgements

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Lastly, I must thank my family, friends and particularly Julia for encouraging me throughout the process of researching and writing this thesis. Special thanks to my fellow master students at Chalmers for welcoming me as one of their own from day one. Thank You!

# Contents

# 1    Introduction

Additive Manufacturing (AM) builds three-dimensional parts by progressively adding material, layer-upon-layer, using a digital model as a guide. This allows for production of parts directly from design without the need for expensive tools, like dies or forms. Furthermore, the process capabilities of AM circumvent many of the design restrictions that govern conventional manufacturing, giving the opportunity to realize internal structures and complex geometries [1].

Various AM techniques have been developed that can process polymer, ceramic and metallic materials, like vat polymerization, binder jetting, powder bed fusion and many more. For the processing of metal powders, laser powder bed fusion (L-PBF) is one the most industrially adopted processes. This method of L-PBF is an additive manufacturing (AM) process that uses thermal energy to selectively fuse regions of a powder bed together.

Although many metal alloys are available in powder form, many are either not suitable or have not been approved for use in L-PBF. To date, only 24 materials are approved by the original equipment manufacturers, with 6 of the mentioned 24 being iron based alloys. Not only are ferrous alloys for AM rarely commercially available, but they are also lacking research effort, especially higher carbon compositions like carbon steels and low alloyed steels. In Figure 1 current (2018) published research for ferrous alloys in L-PBF is depicted, separated into the investigated steels grades.



*Figure 1: Relative emphasis of different ferrous alloys in L-PBF research (total amount: 155 papers); redrawn after Fayazfar et.al. [2]*

To date, most research on ferrous alloys in L-PBF has dealt with either stainless steels or other high alloyed compositions that have low carbon contents (e.g. 316L SS and 17-4PH SS). The low carbon contents of these materials make them unsuitable for use in some structural applications. As such, the addition of low alloyed steels to the portfolio of approved materials for L-PBF would noticeably expand the capacity and applications of L-PBF ferrous alloys.

This thesis investigates the influence of L-PBF processing parameters on the processing window and microstructure of various low alloyed steels, with an emphasis on relating the types of defects that formed to the examined processing parameters.

The main questions sought to be answered in this thesis are as follows:

1. What are the dominant types of defects in low alloy steels? And can they be described as a function of the processing conditions?
2. Is there an ideal processing window for the investigated set of low alloy steels?
3. Can process parameter development be transferred between L-PBF machines? How does this change affect the processing window?

# 2 Background

Additive Manufacturing (AM) is a process that produces 3D components, layer by layer, based on a CAD-model. The layer by layer addition of material provides freedom of design that is not possible when using conventional manufacturing techniques. In conventional manufacturing designs are restricted by the shape of tools (casts,…), machining (round holes, straight cuts,…) and commercial shapes (sheets, tubes,…)[1]. Since in AM tools are unnecessary, those restrictions are lifted, and even complex and irregular shapes can be realized. Internal structures and complex geometries can be implemented allowing new functionality impossible to produce with other processes, like ultra-low-density components [3]. Furthermore, AM facilitates faster iterations of design processes, through its use in rapid prototyping, which allows for products to be more quickly introduced to its intended market [2].

However, AM is not only used for prototyping and can be used for the fabrication of critical components as well. For example, AM produced parts are currently used in both the medical and aerospace industries. This includes, for example,  printed implants for hip replacements and fuel injector nozzles in aeroplanes [1]. Additionally, AM products need little to no post processing, helping to reduce the lead time and overall cost [4].

In recent years Additive Manufacturing (AM) with metals has drawn a lot of attention due to the numerous applications of metals in industry, ranging from basic steel beams for construction to highly specialized nickel alloys for turbine blades in aircrafts. While still not being as refined as AM of polymers, some materials have been certified by equipment manufacturers and can be used in adequate quality for industrial application.

## 2.1   Laser – Powder Bed Fusion

Laser Powder Bed Fusion (L-PBF) is an additive manufacturing (AM) process that uses thermal energy to selectively fuse regions of a powder bed together, layer-by-layer, allowing for the production of near-net-shape parts, with complex geometries and a unique microstructure due to the rapid solidification during the process [2].

In L-PBF, a laser beam is used to selectively melt powder particles together and once a layer is completed the platform lowers, a new powder layer is applied, and the process repeats once more until the part is completed. After production there are some post-processing steps that are required to remove remaining unbound powder [5]. A sketch of the L-PBF process is shown in Figure 2.

Many materials have been studied using the L-PBF process. Some of the most extensively examined materials include Ti6Al4V [6]–[9], Inconel 718 [10]–[16] and 316L Stainless Steel [17]–[20]. On one

hand, these materials have very good properties and are important for a variety of applications. However, in some cases they are unable to provide a solution for L-PBF produced materials that can be used in either structural or automotive applications.



*Figure 2: Sketch of an L-PBF machine setup with powder dispenser, building platform and powder collector.*

## 2.1.1   Process Parameters

For L-PBF there are over 130 parameters than can influence the final component [21]. A list of some of the most important parameters for L-PBF are listed in Figure 3. This vast quantity of factors makes



*Figure 3: Main process parameters for L-PBF*

it troublesome to optimize every parameter when determining the ideal processing conditions and makes it difficult to transfer developed processing parameters between different L-PBF machines.

### 2.1.1.1   Introducing Volumetric Energy Density

Combining laser power together with layer thickness and scanning speed into what is known as volumetric energy density describes the thermal energy that gets inputted per volume of material during L-PBF. This collective parameter is widely used and can be described by following formula:

$$VED = \frac{LP}{v * h * d}$$

- Laser Power (LP) is the power with which the laser is operated.
- Scanning Speed (v) is the speed of the laser moving over the powder bed.
- Layer Thickness (h) is the thickness of the applied powder layer before melting.
- Hatch Spacing (d) is the distance between scan tracks. Usually it is chosen so that the melt pools overlap.

A schematic depiction of all mentioned parameters on a powder bed can be found in Figure 4.



*Figure 4: Representation of basic process parameters in L-PBF (drawn after* [22]*); in reality 3-5 layers will be remolten with each scan*

Volumetric energy density can be connected to internal porosity in the final part and can be used to compare energy input into the powder bed with different sets of parameters [7]. This allows for the determination of a range of volumetric energy densities that facilitate the production of full density parts. Furthermore, it can be used to compare processing parameters between different L-PBF machines. However, there are some limitations when using it as the sole-design parameter. In Figure 5 work by Scipioni et al. showed that even when the same VED was used there will be noticeable differences in process stability of the melt pool [23]. The reason for that is because the volumetric energy density is unable to describe how the inputted energy affects both the shape and the stability of the formed meltpool. As the volumetric energy density does not consider factors such as fluid

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dynamic effects in the melt pool (like the Marangoni flow), Plateau-Rayleigh instability and the recoil effect. Which all drive heat and mass transfer within different regions of the melt pool [23].

Marangoni flow is radial convection within the melt pool that is caused by surface tension gradients, that



Figure 5: Single melt track morphology at consistent volumetric energy density with different sets of parameters [23]

become present when there are noticable thermal gradients [24]. Usually the edge of the melt pool is cooler than the center since thermal energy is supplied in the center through the laser and is "lost" to the surrounding material at the edge. The higher surface tension at the edge pulls liquid towards it and causes this convection in the melt pool [24].

The Plateau-Rayleigh instability comes into effect when the melt pool reaches a critical ratio of length to width. With increasing laser power, the melt pool gets broader and longer even if the scanning speed is increased (to keep energy input constant) [24]. The liquid is compelled to reduce its overall surface tension and the continuous melt pool will instead collapse into groups of spheres [24]. This effect is referred to as balling [23]. This balling effect leads to surface inconsistencies on the component that can cause disturbances in the powder spreading in the subsequent layers, and in some cases causing the build to fail by stopping the movement of the recoating mechanism.

The last of those 3 factors, recoil pressure, is caused by evaporating material directly in the laser spot. When the energy input becomes high enough, recoil pressure is the reason a keyhole is formed in the melt pool [25]. During L-PBF the keyhole can collapse and leave behind a pore, causing lower part density. Since volumetric energy density generally is a thermodynamic quantity, the beforementioned effects and complex physics are not captured [23].

### 2.1.1.2   Scanning strategy

Scanning strategy is an important parameter for many properties of the specimen. It can be used to decrease the anisotropy of properties due to the minimizing effect of columnar grain growth. Additionally, an optimized scanning strategy can help with the reduction of residual stresses [26].

Microstructure is also heavily influenced by the chosen scanning strategy. Depending on the energy input, and the time between initial melting and remelting of consecutive layer, you can either have a

fine microstructure, tall columnar grains that are oriented in the building direction or a mixture of both [1], [2].

### 2.1.1.3    Powder properties

Beyond the processing parameters of the L-PBF process another important factor to consider is the powder itself. The morphology of the powder is preferred to be as spherical as possible, as this greatly improves the flow properties of the powder. This makes it easier to recoat a homogeneous powder layer onto the building platform, in comparison with irregularly shaped powders [27]. Since gas atomized powders are more spherical than water atomized powders they are typically more preferable for L-PBF [28]. However, new research indicates that water atomization can also be optimized for the use of the powder in AM, resulting in similar properties as gas atomized powder [29].

Many other properties are also tied to the size and microstructure of the initial powder, such as the mechanical strength and residual stresses [30]. Due to the complex thermal variations during the L-PBF process, even small deviations in the initial powder characteristics can noticeably influence the final microstructure and physical properties [30]. Even though the cooling rate in L-PBF is initially very high ($10^3$-$10^6$ K/s) [31] when a layer is first deposited, said layer will have many consecutive layers deposited on top of it. Leading to remelting and reheating, that facilitate diffusion processes similar to tempering, relaxation or precipitation within the material [32].

### 2.1.1.4    Atmospheric conditions

During L-PBF the printed part needs to be protected from oxidation. As formed oxides can change the surface energy in the melt pool and as a result the wettability of the molten material, leading to Lack of Fusion (LOF) defects [26]. Additionally, the absorptivity of the powder will change with surface oxides [25]. To protect components from oxidation during the L-PBF process, a protective gas is used within the build chamber. With gases such as argon, nitrogen and helium being the most commonly used.

The chosen atmosphere will also influence the density of the produced specimen. As discussed by Zhang et al., the important factor to consider when examining the protecting gas is the ionization energy [18]. When a gas has a low ionization energy a plasma plume will be formed above the melt pool instead of on it. This can severely obstruct the input of energy to the powder from the laser.

## 2.2    Microstructure of ferrous alloys in L-PBF

Currently, base Fe-C steels are being investigated by William Hearn at Chalmers University of Technology to help develop a knowledge base of ferrous alloys in L-PBF. The main goal in said work is

to achieve full density of the printed component in order to get properties that are comparable to traditionally manufactured parts. As high or full density is crucial for mechanical properties like yield strength, ultimate tensile strength, resistance to fatigue crack propagation and fracture toughness [33].

### 2.2.1 Porosities

In the work on Fe-C steels [34] there were three main types of porosity that affected the part density of the printed component, which each type of porosity having different origins:

    I.      Lack of Fusion

    II.     Gas Porosity

    III.    Keyholing pores

Lack of fusion porosity can be characterized by its distinctive shape – large, irregular pores elongated perpendicular to the building direction (Figure 6) [35]. It is caused by insufficient energy input to completely melt the powder layer and fuse the material with the substrate or the previous layer. Since the layers are separated at those areas, physical properties deteriorate with high amounts of LOF defects [33]. The irregular shape and sharp edges of the pores promote stress concentrations and can act as crack initiation sites



*Figure 6: Example picture of lack of fusion porosity [35]*

when exposed to cyclic loading [36]. When increasing energy input into the material, LOF defects are reduced due to better fusion of the powder layer with previous layers.

Gas porosity are small spherical pores, originating from gas bubbles trapped in the melt pool during solidification (Figure 7) [6]. If the material solidifies before the gas can escape from the melt, it is enclosed and remains in small spherical pores. Additionally, constituents with low boiling point are evaporated and can be trapped in the solidified melt pool the same way as other gases [36]. Since gas porosity is connected to evaporation of material, its



*Figure 7: Example of spherical porosities [6]*

occurrence increases with volumetric energy density. Small, spherical pores are not as big of a problem as LOF defects. As they are far less likely to be crack initiation sites than irregular defects with sharper edges. However, they still reduce the final part density which is not desired.

Keyholing pores are small, relatively spherical pores found at the bottom of melt pools (Figure 8). At high energy input, a larger fragment of material can evaporate or even form plasma in the shape of a

"keyhole". This so-called keyholing mode is known from welding. The keyhole is stable as long as the vaporization and recoil pressure are consistent but during cooling it can collapse, in some cases trapping gas bubbles [37]. Although, this mechanism seems to be widely accepted Madison et al. suggest that there might be a different mechanism for the formation of pores at high scan speeds [38].



*Figure 8: Example of a keyholing pore*

To get a part with adequate physical properties it is necessary to find to optimal processing parameters that can help eliminate defects, allowing for the production of full density components.

### 2.2.2 Thermal History

Thermal history is one of the most important factors influencing microstructure development in ferrous alloys. In L-PBF very high cooling rates of $10^3$-$10^5$ K/s can be observed during the building process [1], [31]. This results in a very small grainsize and a unique microstructure [2]. Due to the extremely high cooling rates that suppress diffusion, Martensite is formed through an athermal diffusionless phase transformation. During cooling face centered austenite is compelled by thermodynamics to transform to body centered ferrite and cementite. However, if the cooling rate is high enough the C atoms cannot diffuse out of the austenite crystal in high enough quantities to form cementite. As a result, a highly strained body centered tetragonal martensite is formed, which is supersaturated with carbon. Martensite formation is aided by high cooling rates due to the immobilization of carbon and propagation of diffusion less processes [39].

### 2.2.3 Role of alloying elements

Another important factor to consider are the role of various alloying elements in the studied ferrous alloy. Some studies can be found that examine the influence of carbon content on the densification and microstructure of carbon steels manufactured using L-PBF, but other alloying elements have scarcely been looked at [2], [34]. The effect that alloying elements have on the properties of ferrous alloys have can be separated into two groups [40]:

I.     Alpha stabilizers: (Cr, Mo, W, V, Al, P)
II.    Gamma stabilizers: (Ni, Cu, C, N, Mn)

The name of these groups describes the influence of the alloying element on the Fe-C phase diagram. Alpha stabilizers expand the area in which α-iron (Ferrite) is stable, whereas γ-stabilizers expand the stability of γ-iron (Austenite). This greatly impacts the formation of what phase will form and thus affects the mechanical properties. Beyond this effect on ferrite and austenite stabilization some

elements might have a higher oxygen affinity than iron (C, Si, Mn and Cr; based on Gibbs free energy values found in the Ellingham-Diagram) which leads to reduction reactions during melting and can influence wettability of the substrate.

The general effect of alloying elements that are relevant to this thesis are described below:

- **Carbon** [41]**:** Carbon is the most important alloying element. It strengthens iron through strengthening mechanisms, e.g. solid solution hardening. Additionally, it reduces the melting temperature. Since it is a strong reducing agent it can reduce formed iron oxides, while releasing $CO/CO_2$. If high amounts of gas are formed during the processing of the material, pores can be formed. Furthermore, oxidized carbon no longer has a strengthening effect on the material, so oxygen content and the resulting carbon loss must be considered when using ferrous alloys with higher carbon contents.

- **Silicon:** Silicon is part of the group of α-stabilizers and reduces melt viscosity. Like Carbon Silicon is a reducing agent, that has a strong oxygen affinity [42].

- **Manganese** [40]**:** Manganese is an austenite stabilizer. In addition, it has a very high vapor pressure, so it is prone to being vaporized and lost during melting. It is mainly used to capture sulphur and reduce its detrimental effect on cracking [42].

- **Chromium** [40]**:** Chromium is an α-stabilizer. It greatly increases hardenability and in high concentrations can also improve the corrosion resistance of ferrous alloys.

- **Molybdenum** [40]**:** Molybdenum is an α-stabilizer. It increases hardenability.

- **Nickel** [40]**:** Nickel stabilizes the γ-phase. It influences thermal expansion and corrosion resistance.

To summarize, L-PBF is, compared to conventional manufacturing, a very new technology. Its principles are understood only at a basic level and a lot more research needs to be done to help mature the process and make it viable for a broader market. In this thesis, development of our understanding of L-PBF will be done by looking at the processability of low alloyed steels, which are not yet certified by equipment manufacturers for production via L-PBF.

# 3   Methods and Materials

## 3.1   Powders

Table 1 lists the different ferrous alloy compositions that were examined during this investigation. All listed ferrous alloys were pre-alloyed powders, that were produced via gas atomization. With respect to the particle size distribution, the powders either had a particle size distribution 20-53 µm or 15-45 µm, depending on their composition.

*Table 1: Chemical Compositions in wt% of powder alloys used in this investigation*

|  | % Cr | % Mo | % C | % Mn | % Si | % Ni | % P (max) | % S (max) |
|---|---|---|---|---|---|---|---|---|
| AISI 4130 | 1.0 | 0.2 | 0.34 | 0.6 | 0.3 | n/a | 0.01 | 0.006 |
| AISI 8620 | 0.5 | 0.23 | 0.2 | 0.8 | 0.35 | 0.7 | 0.01 | 0.005 |
| AISI 4140 LC | 1.0 | 0.2 | 0.43 | 0.75 | 0.29 | n/a | 0.006 | 0.004 |
| AISI 4140 HC | 1.0 | 0.2 | 0.47 | 0.6 | 0.2 | n/a | 0.011 | 0.007 |
| AISI 4340 | 1.0 | 0.3 | 0.43 | 0.6 | 0.17 | 1.9 | 0.01 | 0.005 |

## 3.2   L-PBF Machines

The L-PBF machines that were used in this investigation were an EOS M100 and an EOS M290. Both machines were equipped with an Yb-fiber laser, that has a maximum laser power of 200 W and 400 W, respectively. The focus diameter of the laser EOS M100 machine was ~40 µm, while the focus diameter of the laser on the EOS M290 was ~100 µm. For both machines, argon was used as the inert shielding gas, with the oxygen content being kept at ~0.1% within the processing the chamber.

*Table 2: Technical Data for EOS M100* [43] *and M290* [44]

|  | M100 | M290 |
|---|---|---|
| Building volume | Ø 100 mm x 95 mm | 250 mm x 250 mm x 325 mm |
| Laser type | Yb-fiber laser; 200 W | Yb-fiber laser; 400 W |
| Scan Speed | Up to 7.0 m/s | Up to 7.0 m/s |
| Focus Diameter | 40 µm | 100 µm |
| Building Plate Preheating | Up to 80 °C | Up to 200 °C |

## 3.3   Analysis

### 3.3.1   Sample Preparation

For metallography, specimens were first sectioned with a Buehler ISOMET 2000 precision saw (Figure *9*-I). This was followed by mounting of the sectioned samples, using a Struers Citopress machine (Figure 9-II). Next, the mounted samples were ground with a Struers TegraPol machine (Figure 9-III),

using SiC-foil papers in following order: #220, #320, #500, #800, #1200, #2000 and #4000. After grinding the samples were polished using a 3 µm and a 1 µm diamond suspension.



*Figure 9: Buehler ISOMET 2000 precision saw (I), Struers TegraPol – 31 polishing machine (II), Struers CitoPress – 20 mounting machine (III)*

### 3.3.2   Porosity Analysis

Based on work of Kasperovich et al. [45] it was decided that porosity measurements would be done using light optical microscopy of sectioned, unetched samples. This is due to its relatively high accuracy of this technique (in relation to µCT scans), along with the relatively quick analysis time of the method. In order to measure the porosity of the sectioned samples, first OM images were taken using a Zeiss Axiovision 7 microscope and then stitched together using the Zeiss Zen 2 Zencore software. Next these images were loaded into an image analysis program (FIJI), where the area fraction of porosity in the cross sections would be measured. To do this FIJI would convert the stitched image (Figure 10-I) into

a binary, black and white, image (Figure 10-II), with a threshold of 68 on a scale from 0 to 255. Next the edges of the image were cropped so the measured area fraction would be related to the bulk material (Figure 10-III). At this point the ratio of black (pores, cracks, etc.) and white (material) could be determined, giving the overall relative density of the specimen.



| Stitched image | Binary stitched image | Cropped binary for analysis |

*Figure 10: Steps of image processing for porosity analysis (the green arrow signifies the building direction)*

### 3.3.3 Hardness

To measure the hardness, indentations were performed with a DuraScan 70-G5 machine (Figure 11-I). The used norm was Vickers hardness, with a load of 10 kg, at a holding time of 10 seconds.



*Figure 11: Hardness Tester DuraScan 70-G5 (I) and scheme for a 4x4 grid of indentations (II)*

### 3.3.4    SEM

For taking scanning electron microscope micrographs a Leo Gemini 1550 SEM was used, that was also equipped with Energy Dispersive X-ray Spectroscopy (EDS).

# 4    Results

## 4.1    Processing window

In this work the ideal processing window was defined as a set of processing parameters that yielded densities >99.8 %, without the presence of defects.

### 4.1.1    M100 Results

At low volumetric energy densities large pores can be found throughout the sample cross section (Figure 12). When examined in greater detail, un-melted powder particles can be observed within the pores (Figure 13-I). As the volumetric energy density increased, the amount of these large irregular pores decreased. Instead there was the formation of small spherical pores (Figure 13-II). When the volumetric energy density is further increased, the observed spherical pores become more common and a third type of porosity appears. This type of porosity is slightly more irregular than the spherical pores and increase not only



*Figure 12: Stitched light optical micrograph of the entire crosssection of an 4130 specimen produced at (60 J/mm³)*

in number but also in diameter (~10 to ~30 μm) with higher volumetric energy density (Figure 13-III). This general trend can be seen in all investigated alloys, with the only difference being the energy input where these transitions in porosity occur.



*Figure 13: Light optical micrographs of 4130 specimen at low VED (60 J/mm³) (I), at medium VED (140 J/mm³) (II) and at 180 J/mm³ (III); 110 W*

With the 4130 alloy the required part density of 99.8 % can be reached at a volumetric energy density of 120 J/mm³ and above (these results can be found in Figure 14). For a laser power of 110 W the part

density development shows a rapid increase at low volumetric energy densities (60-100 J/mm$^3$) and surpasses the desired part density threshold at 140 J/mm$^3$. Part density decreases below the threshold once the volumetric energy density becomes greater than 160 J/mm$^3$. When using a laser power of 140 W the general trend of density development remains the same. However, the range of volumetric energy densities that reach >99.8 % density shifts to 160 – 180 J/mm$^3$. At 170 W the range further expands to 120-180 J/mm$^3$.



*Figure 14: Part density development vs. VED for the 4130 alloy at laser powers of 110 W, 140 W and 170 W on the M100 machine; the red dotted line signifies the threshold for densities high enough to qualify for the processing window (99.8 %)*

For the 8620 alloy the development of porosity is almost identical to the 4130 alloy. At low volumetric energy densities large irregular pores are found, whose amount and size are reduced when increasing volumetric energy density (Figure 15-I/II). Porosities of type two and three become more prevalent as the energy input is increased (Figure 15-III).



*Figure 15: Light optical micrographs of 8620 specimen a low VED (60 J/mm$^3$; 170 W) (I), at medium VED (120 J/mm$^3$; 140 W) (II) and at high VED (180 J/mm$^3$; 110 W) (III)*

In Figure 16 the density development for the 8620 alloy can be found. At 110 W laser power high density is reached in a range of volumetric energy density between 120 J/mm$^3$ and 140 J/mm$^3$. At a volumetric energy density of 160 J/mm$^3$ a decrease in density can be seen, but since the shown values are singular, no statistical significance can be attributed to this. At a laser power of 140 W the range

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for high density specimens expands to 110 – 140 J/mm$^3$. For a laser power of 170 W this range further expands to 100 – 180 J/mm$^3$.

*Figure 16: Part Density Development vs. VED for the 8620 alloy with laser powers of 110, 140 and 170 W on the M100 machine; the red dotted line signifies the threshold for densities high enough to qualify for the processing window (99.8 %)*

The alloy 4140LC has a higher carbon content than both the 4130 and 8620 alloys but was not found to cause a change in the type of porosities that formed. In Figure 17-I, large, irregular pores are found, identically to previous alloys, which are not present at higher VED, as can be seen in Figure 17-II. Instead, at high volumetric energy densities (Figure 17-III) the smaller and more spherical porosities are observed.



*Figure 17: Light optical micrographs of 4140LC specimen a low (60 J/mm$^3$; 170 W) (I), at medium VED (120 J/mm$^3$; 110 W) (II) and at high VED (180 J/mm$^3$; 110 W) (III)*

Figure 18 shows the results of part density's dependency on the volumetric energy density for the 4140LC alloy. For the laser power of 110 W the range of VED yielding high density parts can be found at 100-140 J/mm$^3$. When using 140 W the range shifts to 110 – 180 J/mm$^3$; At 170 W the range further expands to 100-180 J/mm$^3$.

*Figure 18: Part Density Development vs. VED for the 4140 LC alloy with laser powers of 110, 140 and 170 W on the M100 machine; the red dotted line signifies the threshold for densities high enough to qualify for the processing window (99.8 %)*

In Figure 19 the microstructure of 4140HC specimen cross sections is shown. At low volumetric energy densities large, irregular pores are found for all investigated laser powers (Figure 19-I). With higher volumetric energy density their amount and size diminished until they cannot be found at all (Figure 19-II). However, if the volumetric energy continues to get larger, we start to see the formation of spherical pores (Figure 19-III).



*Figure 19: Light optical micrographs of 4140HC specimen a low VED (60 J/mm³; 110 W) (I), at medium VED (20 J/mm³; 170 W) (II) and at high VED (180 J/mm³; 140 W) (III)*

Figure 20 shows the results of part density and its dependence on the volumetric energy density, for the 4140HC alloy. Although none of the parts made with the 4140HC alloy, using a laser power of 110W, reached the desired >99.,8 % density we saw the typical trends. Initially the part density would rapidly improve as the volumetric energy was increased from 60 J/mm³ and eventually it would start to deteriorate if the volumetric energy became too large. When using a laser power of 140 W the density development shows a rapid increase at low volumetric energy densities and can achieve the desired part density at a volumetric energy density of 160 J/mm³. At 170 W the same general trend is recognized however the range of volumetric energy densities that yielded >99.8 % density expanded to a range of 100-180 J/mm³.

Although the porosities in the 4140HC alloy were acceptable, the alloy displayed cracking at all investigated volumetric energy densities. These cracks were approximately 10 µm to 60 µm in length and were distributed throughout the entire cross section of the specimen seemingly without any preferred orientation (See 4.2.2).



*Figure 20: Part Density Development vs. VED for the 4140 HC alloy with laser powers of 110, 140 and 170 W on the M100 machine; the red dotted line signifies the threshold for densities high enough to qualify for the processing window (99.8 %)*

For the 4340 alloy, large irregular pores can be found at low volumetric energy densities, regardless of used laser power (Figure 21-I). With increments of volumetric energy density this type of porosity in reduced in number and size, peaking where none of this type of porosity can be found (Figure 21-II). As the volumetric energy density continues to increase past this ideal range the number and size of spherical pores increases (Figure 21-III).



*Figure 21: Light optical micrographs of 4340 specimen at low VED (60 J/mm³; 170 W) (I), at medium VED (120 J/mm³; 170 W) (II) and at high VED (180 J/mm³; 110 W) (III)*

The density development of 4340 samples can be found in Figure 22. Starting at low volumetric energy density a rapid increase in density can be ascertained. When a laser power of 110 W is used the desired density of >99.8% is achieved at a volumetric energy density of 80 J/mm³. Afterwards the trend reverses and density slowly degrades as the volumetric energy density increases. When a laser power

of 140 W is used the volumetric energy density range that can achieve high density expands to 110-160 J/mm$^3$, and at a laser power of 170 W the range further expands to 110-180 J/mm$^3$.



*Figure 22: Part density development vs. VED for the 4340 alloy at laser powers of 110 W, 140 W and 170 W on the M100 machine; the red dotted line signifies the threshold for densities high enough to qualify for the processing window (99.8 %)*

In Table 3 processing windows for the investigated compositions can be found. This represents sets of parameters which yield crack free, >99.8 % density parts. An upper limit of 180 J/mm$^3$ does not represent an actual upper limit, it was just the highest VED that was investigated in this work using the EOS M100 machine.

*Table 3: Processing windows (in respect to a density >99.8 %) for various low alloyed steels printed in an EOS M100 machine;*

| Processing windows [J/mm$^3$] | 110 W | 140 W | 170 W |
|---|---|---|---|
| 4130 | 140 | 160-180 | 120-180 |
| 8620 | 120-140 | 110-160 | 100-180 |
| 4140 LC | 100-140 | 110-180 | 100-180 |
| 4140 HC | - | 160 | 100-180 |
| 4340 | 80 | 120-160 | 110-180 |

### 4.1.2   M290 results

On the EOS M290 machine only 2 of the 5 mentioned alloys were investigated; 4130 and 4140HC. The other powders were not available in large enough quantities. Processing parameters were kept the same as on the M100 machine, except the laser power of 140 W was no longer included in the investigation, so laser powers of 110 W and 170 W were used.

The density development with volumetric energy density (Figure 23) of the 4130 alloy shows a rapid increase in density at low volumetric energy density (60-100 J/mm$^3$) for both laser powers. At higher VED the density consistently stays at density values above 99.8 %.

*Figure 23: Part density development vs. VED for the 4130 alloy at laser powers of 110 W and 170 W on the M290 machine; the red dotted line signifies the threshold for densities high enough to qualify for the processing window (99.8 %)*

For the 4140HC alloy the trend is similar to the 4130 alloy, where the achieved part density will noticeably improve when you increased the volumetric energy density from low values. And as the volumetric energy increases to larger values the density fluctuates, in some cases drops below the desired 99.8 % density (Figure 24). This was the case when using a laser power of either 110 W and 170 W.

*Figure 24: Part density development vs. VED for the 4140HC alloy at laser powers of 110 W and 170 W on the M290 machine; the red dotted line signifies the threshold for densities high enough to qualify for the processing window (99.8 %); the bar at 140 J/mm$^3$ is missing due to a print failure*

## 4.1.2.1    Building Plate Preheating

In further investigations the preheating of the building plate in the M290 machine was varied, to values of 25 °C, 100 °C and 180 °C. For the 4130 alloy the density increases at low volumetric energy densities (60-90 J/mm$^3$) and stays consistently high until a VED of 200 J/mm$^3$ (Figure 25).

*Figure 25: Part Density Development vs. VED for the 4130 alloy with laser powers of 110 W and 170 W and building plate preheating temperatures of 25 °C, 100 °C and 180 °C; the red dotted line signifies the threshold for densities high enough to qualify for the processing window (99.8 %)*

For the 4140HC alloy building plate preheating to temperatures of 25 °C, 100 °C and 180 °C was done as well. Again, the density was found to rapidly increase at low volumetric energy densities and then stays high but fluctuates above and below the desired density of 99.8 % (Figure 26).

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*Figure 26: Part Density Development vs. VED for the 4140HC alloy with laser powers of 110 W and 170 W and building plate preheating temperatures of 25 °C, 100 °C and 180 °C; the red dotted line signifies the threshold for densities high enough to qualify for the processing window (99.8 %)*

The results for the set of processing parameter that yield crack free, high density parts with the 4130 and 4140HC alloys can be found in Table 4 below.

*Table 4: Processing windows for the M290 machine with various building plate preheating temperatures.*

| | M290 25 °C | | M290 100 °C | | M290 180 °C | |
|---|---|---|---|---|---|---|
| | 110 W | 170 W | 110 W | 170 W | 110 W | 170 W |
| *4130* | 120-200 | 100-200 | 120-200 | 120-200 | 100-200 | 90-200 |
| *4140 HC* | 120-180 | 150 | 100-180 | 90-120 | 100-120 | 90-200 |

## 4.2 Microstructure

### 4.2.1 Porosities

Within the sample cross-sections there were three types of porosity that were identified, for the investigated low alloy steels:

I.     Large, irregular pores, containing with unmelted powder particles (Figure 27-I).

II.    Small spherical pores that were distributed throughout the sample (Figure 27-II).

III.   Mainly spherical pores that were located at the bottom of formed melt pools (Figure 27-III).



*Figure 27: Large, irregular pores containing unmolten particles (I); fine spherical porosity (II); pore at the bottom of a melt pool (etched with 3 % Nital) (III)*

The large, irregular pores are observed at low volumetric energy densities, most prevalent below 90 J/mm$^3$. The other 2 types of porosity both have a higher impact at higher volumetric energy densities. Type II is visible at all investigated volumetric energy densities but starts being a major contributor to overall porosity around 90-100 J/mm$^3$, when the first type starts to disappear. Their size and amount increase with volumetric energy density. At around 100 J/mm$^3$ the third type of pores starts appearing. First in minor numbers but the same as type II their size and amount increase with volumetric energy density.

## 4.2.2 Cracks

In this work three types of cracks were identified, which occured in some of the investigated low alloy steels. The first type of cracking consisted of large, horizontal cracks that originated at sample surface or at irregular pores within the material (Figure 28-III). The crack path was perpendicular to the building direction and followed no visible paths, like melt pool boundaries or microstructural features. This can be seen in both Figure 28-I and Figure 28-II. The processing parameters under which those cracks appear are at low volumetric energy densities and in materials that had carbon content of 0.4 wt% carbon or greater.



*Figure 28: Large horizontal cracks originating at the surface of the specimen; stitched image (I), etched (Nital 3%) light optical micrograph (II) and cracks originating from a pore (III)*

The second type of cracking was observed only in the 4140HC alloy, with said cracking being visible regardless of the used processing parameters or the used L-PBF machine. These cracks are predominantly oriented in building direction and are not concentrated in any specific area of the cross section (Figure 29). Rather they are finely distributed throughout the material, with their size ranging from 10 µm to 60 µm in length.

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they are located within the last deposited layer, at a location that is approximately in the middle of the melt pool, as shown in Figure 31.



*Figure 31: Light optical micrographs of top layer cracks in 4140HC parts in unetched and etched condition (3% Nital), on the right image in the lower part a Vickers indentation is visible.*

### 4.2.3 Melt Pools Characteristics

To make the melt pool boundaries visible, the specimens were etched with a 3% Nital etchant. By doing so the melt pool boundaries within the material could be revelated (Figure 32-I). These melt pool boundaries consist of needle-like structures. However, when increasing the magnification (Figure 32-II/III), melt pool boundaries are revealed to be diffuse and not discrete borders.



*Figure 32: Light optical (I, II) and SEM (III) micrographs of the microstructure of etched cross sections (Nital 3 %)*

## 4.2.4  Inclusions

When conducting analysis via optical microscopy it is not possible to distinguish inclusions and gas porosity from one another. In order to do so analysis needs to be done by coupling SEM and EDS analysis. In Figure 33 a SEM micrograph can be found with EDS spectra of the bulk material and a pore, proving it to be a void and not an inclusion.



*Figure 33: SEM image of a pore with corresponding EDS spectra of regions marked on the micrograph* [46]

An example for inclusions in the material can be found in Figure 34. The EDS spectra found several types of inclusion compositions. Two of the displayed inclusions exhibit elevated amounts of oxygen and elements with high oxygen affinity, like aluminium, silicon and manganese. The third inclusion on the other hand shows an elevated amount of carbon compared to the bulk material.

The analysis of gas pores and inclusions by SEM and EDS allows to draw the conclusion that they can be distinguished in light optical microscopy by measuring the size. Both have a mostly spherical shape, but gas pores have a diameter of a round 10 μm, while inclusions have a diameter of just 1 μm and below.

*Figure 34: SEM micrograph of inclusions with corresponding EDS spectra marked on the SEM micrograph* [46]

## 4.3 Melt pool

Melt pool size and shape were evaluated by examining the top layer of sample cross sections that had been etched with 3 % Nital. The dependence of the melt pool depth and shape on volumetric energy density is shown in Figure 35 and Figure 36. With higher VED the molten area expands, and the melt pool can be seen to get wider and deeper (Figure 35-I/II). At high volumetric energy densities, the shape of the melt pool changes to a narrow and elongated form (Figure 35-III).



*Figure 35: Melt pool shape development with increasing VED; specimen etched with 3 % Nital*

As seen in Figure 36, at lower VEDs the increase in melt pool depth on the M100 is somewhat linear and does not vary much with the used laser power. However, starting at a VED of ~100 J/mm$^3$, the differences in melt pool depth become more significant, as a higher laser power leads to deeper melt pools. Starting at 130 J/mm$^3$ the trend between the VED and the melt pool depth start to vary depending on the used laser power. At a laser power of 110W the melt pool depth remains relatively constant when the VED is increased above 130 J/mm$^3$. In comparison an increased in VED leads to an increase in the melt pool depth, when laser powers of 140 W and 170 W were used. At the highest tested volumetric energy density, 180 J/mm$^3$, melt pools were 90 µm deeper when using a laser power of 170 W versus 110 W.



*Figure 36: Melt pool depth for various VEDs and laser powers with 4130*

For the other compositions this trend seems to be consistent, except for the plateauing which is not observed with the other low alloy steel compositions (Figure 37). For the 8620, 4140LC, 4140HC and 4340 alloys no significant plateauing of melt pool depth at higher volumetric energy densities was observed.



*Figure 37: Melt pool depth development with VED for 8620, 4340 and both 4140 modifications*

In the M290 machine the melt pool depth is smaller, at the same processing conditions, than the melt pool depth in the M100 machine. Nevertheless, a linear trend of increasing melt pool depth with increasing volumetric energy density can be observed. The preheating of the building plate does not show any significant impact on the melt pool depth at any VED or laser power (Figure 38).

*Figure 38: Melt pool depth development with volumetric energy density of 4130 specimen produced under different preheating conditions, laser powers in the M290*

## 4.4   Hardness

The hardness development with volumetric energy density, for the 4130 alloy, is shown in Figure 39.

For most of the investigated volumetric energy density range the hardness declines with higher VED.



*Figure 39: Average hardness of 4130 specimen cross sections*

The general trend of the development of hardness versus volumetric energy density is similar for all of the investigated low alloyed steels. First there is an increase in hardness due to a reduction in subsurface porosity (~60-100 J/mm$^3$), followed by a gradual decline in hardness as the volumetric energy density increases (>100 J/mm$^3$). In Figure 40 the hardness results for all investigated alloys can be found.



*Figure 40: Average hardness values for 8620, 4340 and both 4140 modifications as a function of volumetric energy density*

## 4.5  Carbon loss

A measurement of the C loss during processing (for the 4130, 4140LC, 4140HC and 4340 alloys) was done by measuring the C content in the as-built material and comparing it to the original C content of the powder feedstock [46]. It was found that the C loss was relatively consistent, ~16-20%, except for the 4340 alloy, where the C loss between the powder and as-built condition was only 6.3% (Table 5).

*Table 5: Carbon content of the initial powder and the as-built parts with a calculation of relative carbon loss* [46]

| | 4130 | 4140LC | 4140HC | 4340 |
|---|---|---|---|---|
| *Carbon content powder [wt%]* | 0.34 | 0.43 | 0.47 | 0.43 |
| *Carbon content as-built [wt%]* | 0.275 | 0.361 | 0.377 | 0.403 |
| | **-19.1 %** | **-16.0 %** | **-19.8 %** | **-6.3 %** |

# 5 Discussion

## 5.1 Processing window

The density development, with volumetric energy density, can be explained with the formation of different types of porosity.

At low VEDs the energy inputted is insufficient to melt all powder particles. This leads to unmelted powder particles, that can be found in some of said pores. This is an indication that the large irregular pores observed in this work, that occurred at low VEDs, were lack of fusion pores. Similar results at low VEDs had been previously observed in iron carbon systems [34]. With higher energy input this type of porosity cannot be observed. The reason why lack of fusion porosity decreases with increasing VED can be related to the results shown in Figure 37, where the melt pool depth increases with volumetric energy density. This increasing melt pool depth, with increasing VED, helps to ensures sufficient overlap of melt tracks and thus, better fusion between adjacent layers. The addition of more carbon, or other melting point decreasing alloying elements, to the material has a similar effect as it can be observed to shift the processing window to lower volumetric energy densities, as can be seen in Table 3. When looking at phase diagram simulations the influence of carbon on the melting point can be confirmed (phase diagrams can be found in the appendix 8.1). As Nakamoto et al. showed, a higher carbon content increases the wettability of the melt on the previously melted layer. This ensures that no powder can be trapped between two melt tracks, which subsequently cannot be completely molten with the next powder layer, resulting in lack of fusion porosity and explains the observed shift of the processing window [47].

The small spherical pores appearing at higher volumetric energy densities could be gas porosity. The gas can have various origins. Gas can be entrapped in powder particles during atomization prior to processing or insoluble shielding gas and alloy vapours are trapped during processing [1], [48]. Another possibility of gas formation during the melting process could the reaction of carbon with remaining oxygen in the processing atmosphere, oxide contamination in the power or adsorbed oxygen at the surface of the power. All gases try to escape the melt but can be trapped within the material due to the rapid solidification nature of the L-PBF process. It is harder for entrapped gas to escape if the viscosity of the melt is high, resulting in a higher resistance to the movement of the bubbles. Furthermore, the size of the melt pool is very important. The deeper the melt pool the farther the gas bubble has to rise in order to escape the liquid. The higher the temperature in the melt pool the faster carbon is oxidized, potentially aiding the formation of gas and increasing the amount of gas porosity.

The third type of porosity found at the bottom of the melt pool could be keyholing. These pores are larger than gas porosity and more irregular but still mostly spherical (Figure 32). At very high energy inputs the melt pool becomes very deep and narrow. When the recoil pressure of the evaporated material subsides, the melt pool can collapse and entrap gas in the solidifying material [37].

### 5.1.1 Influence of Laser Power on density

For the range of volumetric energy densities that were examined, the laser power is correlated with a widening of the processing window (Table 3). At a laser power of 110 W, the processing window is very narrow. When the laser power increase to 140 W, and further to 170 W, the processing window correspondingly expands. No poor performing area was observed at the highest investigated volumetric energy densities, suggesting that the actual upper limit of the processing windows at 170 W might be even higher than 180 J/mm$^3$. This is mainly connected to the deeper melt pool with higher laser power, which ensures complete melting of the powder layers with a higher number of layers remolten every time the laser passes (Figure 37).

A contradictory influence appears at low volumetric energy densities, below the lower limit of the processing window Figure 42. This is not necessarily connected to laser power but rather to the scan speed. The scan speed required to reach a volumetric energy density of 60 J/mm$^3$, when using a laser power of 170 W, was ~20-50% larger than if one used a laser power of 110 W or 140 W. Due the this extremely high scan speed, when reaching a VED of 60 J/mm$^3$ at a laser power of 170W laser, the material does not have enough time to be fully melted, leading to the noticeable amount of lack of fusion porosity under these conditions.

For 4340 alloy parts produced with 110 W laser power no density higher than 99.8 % could be achieved. The reason for this might be a combination of low laser power, slow scan speed and lower



*Figure 41: Stitched light optical micrograph of a cross section of a 4340 specimen produced with a laser power of 110 W*

melting point resulting in over melting of the material and causing surface waviness and edge swelling (Figure 41). All of which are factors that influence powder spreading. Inhomogeneous powder layers can then influence the melt pool and with it may create high amounts of Lack of Fusion porosity. The density results for the 4340 alloy can be found in Figure 22 and the simulated phase diagram, with calculated melting point, in Appendix 8.1.

*Figure 42: Comparison of density values of different material compositions at 60 J/mm³*

### 5.1.2    Influence of Composition

The influence of composition on the processing window is observed in a shift towards lower volumetric energy densities (Table 3). This was explained by the increased wettability of the alloy as the carbon content increased [47]. As mentioned before, for most investigated parameters the poor performing region above the processing window lies higher than the highest tested VED (200 J/mm³). For other alloying elements, most importantly nickel, no clear trends were observed. Generally, a higher alloying element content seems to reduce the lower limit of the processing window to lower volumetric energy densities. Below the lower limit an even higher influence of carbon content was found. In Figure 42 the density values of all compositions are compared, showing an increase of up to 3 % between the lowest (4130) and highest alloying content (4340), when using the same processing conditions.

Based on the results of carbon loss nickel potentially has an influence on the retention of carbon in the material during processing, but since the analysis of 8620 did not yield any useable results, no clear trends can be established.

### 5.1.3    Comparison of M100 to M290

#### 5.1.3.1    *Processing window*

From this examination it was found that the specimens built using the EOS M290 yielded higher part densities, than the specimens produced using the EOS M100 (Figure 43). The main influence on that is the difference of laser parameters e.g. the spot size of the laser. The M100's laser has a spot size of 40 µm while the M290's has 100 µm. Further contributing factors could be the improved recoating mechanism and the improved gas flow of the M290 machine. Comparing the melt pool depths of the two machines, the M290 exhibits a wider and shallower melt pool when the same processing parameters are used (Figure 47). The reason for this is the larger spot size, which cause the inputted

energy to become distributed over a larger area. Decreasing the intensity of energy at the center and changing the shape of the melt pool to help decrease the risk of keyholing porosity. The increased width of the melt pool gives a higher overlap to the adjacent one, resulting in a more homogeneous layer, with less irregularities and less lack of fusion porosity (Figure 47). Preheating of the building plate helps increase part density by there being a more homogeneous input of thermal energy throughout the part.

The processing window of the 4130 alloy the EOS M290, when compared to the EOS M100, is shifted to lower VEDs especially when base plate preheating is used. As here, part density values greater than 99.9% are common over the entire processing window.



*Figure 43: Density development with VED for 4130 produced at the M290 with different building plate preheating temperatures and M100 results to compare*

While still reaching relatively high densities, 4140HC fails to consistently meet the density criterion of 99.8 %. As was already discussed and shown in Figure 29 4140HC has noticeable cracking throughout the specimen. The amount and size of cracks increased when using the M290 instead of the M100. Results for density measurements of 4140HC alloy specimens can be found in Figure 44.

*Figure 44: Density development with VED for 4140 HC produced at the M290 with different building plate preheating temperatures and M100 results to compare*

The most important trends when comparing the processing windows of the two machines and when comparing the build plate preheating temperatures are as follows. Firstly, the process on the M290 machine has less variations, most likely due to the improved gas flow and powder spreading, giving a wider processing window especially when comparing low laser powers. Secondly, the building plate preheating further expands the range due to the additional heat input. The actual processing windows

for 4130 and 4140HC on the M100 and the M290 at different building plate preheating temperatures can be found in Table 6.

*Table 6: Processing windows for M100 and M290 with various building plate preheating temperatures*

| | M100 | | M290 25 °C | | M290 100 °C | | M290 180 °C | |
|---|---|---|---|---|---|---|---|---|
| | 110 W | 170 W | 110 W | 170 W | 110 W | 170 W | 110 W | 170 W |
| *4130* | 140 | 120-180 | 120-200 | 100-200 | 120-200 | 120-200 | 100-200 | 90-200 |
| *4140 HC* | - | 100-180 | 120-180 | 150 | 100-180 | 90-120 | 100-120 | 90-200 |

### 5.1.3.2 Melt pool characteristics

Comparing the materials directly, a relatively consistent trend can be seen in Figure 45. For the investigated materials the melt pool depth is largely independent of alloying content.



*Figure 45: Direct comparison of melt pool development between compositions*

Comparing the measured melt pool depths between machines (Figure 46) the M290 shows a significantly shallower melt pool than the M100. At a laser power of 110 W, the melt pool depth behaves differently between the two machines. For the M100 the penetration depth increases linearly at low volumetric energy densities and plateaus at high volumetric energy densities, whereas for the M290 the melt pool depth increases mostly linear for the entire investigated range of energy input. That means the melt pool depth of M100 and M290 at 110 W first drifts apart and then start converging. At 170 W melt pool depth at the M100 does not start plateauing but both machines show a linear increase with volumetric energy density. Here the ratio between the melt pool depths of the machines at the same processing parameters is about 2:1. Micrographs of etched specimen showing differences in melt pool depth between M100 and M290 can be found in Figure 47.

*Figure 46: Comparison of melt pool depth of 4130 specimen produced under different preheating conditions, laser powers and machines*

When looking at the homogeneity of the molten layer, specimens produced with the M290 show superiority regarding the curvature of the top surface. This waviness, as it is seen in Figure 47-I, can lead to inconsistent spreading of the powder layer followed by process instabilities Due to the larger focus diameter of the laser in the M290 the melt pools are about half as deep as in the M100. This is observed when comparing Figure 47-III and IV. Another characteristic that can be seen in those images is the consistency of the melt pool shape. On the left, the sample produced in the M100, no melt pool looks like the other, when in contrast the melt pools on the right (M290) are almost indistinguishable.

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*Figure 47: Comparison of melt pools of specimens produced at same processing conditions with different machines; M100 (I/III) and M290 (II/IV)*

## 5.2    Investigation of the cracking mechanisms

The density, or in other words the amount of porosity, is one aspect of the definition of the processing window, cracking is the other. As was mentioned, three different types of cracks were observed in this investigation. Large horizontal cracks, shorter horizontal cracks in the top layer and short vertical cracks.

The large horizontal cracks originate at irregular pores at the edge of the or in the bulk material. Those pores or irregularities at the sample surface are strong stress concentrators, that help provide the initiation point of said cracks. Furthermore, the high amount of residual stresses and the high hardness of the material make it even more prone to cracking [1]. This type of cracks can only be seen at low VEDs. The hardness of the material decreases with VED, meaning the susceptibility to cracking is at its highest in the samples produced using a low volumetric energy density. As the VED is increased the hardness of the material decreases, and with-it the probability of these cracks occurring.  Additionally, as the VED is increased the amount of irregular lack of fusion porosity will noticeably decrease, which also reduces the number of potential sites for crack initiation.

The short, vertical cracks can be found only for high carbon modification of 4140 alloy and seem to be cracks that occur during solidification. The increase in amount and size of the cracks comparing the M290 and M100 produced specimen supports this idea of solidification cracking. The larger melt pool cross section at the M290 supplies a higher amount of material in one direction that is contracting during cooling. This increases stresses in the material causing cracks. Carbon cannot be the reason for this type of cracking. No similar cracks were found in the compositionally very similar 4140 LC, neither in the 4340 alloy. The specimens were produced in 3 prints at the M100 in volumetric energy density ranges of $60 - 80$ J/mm$^3$, $100 - 120$ J/mm$^3$ and $130 - 180$ J/mm$^3$. This eliminates the possibility of a build gone wrong. Also, the cracks were observed in specimens produced at the M100 as well as the M290.

Many of the cracks exhibit a distinct form consisting of a head and a tail (Figure 30). The crack seems to initiate at the head and the propagate from there. In some cases, an oxide can be found in the head of the cracks. This potential oxide particle could be the initiation site of the crack, but further investigation is needed to confirm this and eliminate other potential sources of those cracks, like solidification cracking and liquation cracking.

## 5.3 Hardness

The hardness of the specimens produced via L-PBF is higher than conventionally produced quench and temper versions of the same material (Figure 48). The extremely high cooling rates ($10^3$-$10^5$ K/s) during L-PBF process cause the high hardness of the materials [31]. Although high hardness is generally a desirable property, it also makes the material more prone to cracking. This can be seen with some higher carbon samples. Major cracks perpendicular to the building direction can be found even before removing specimens from the building plate (Figure 28).



*Figure 48: Hardness range of 10 mm cubes produced with L-PBF (striped area) compared to conventional quench and temper hardness values (cross); 4130 [49]: water quenched (855 °C), temper (540 °C); 25 mm round; 8620 [50]: quenched (900 °C), tempered (540 °C); 4140 [51]: oil quenched (845 °C), temper (540 °C), 25 mm round; 4340 [52]: oil quenched (800 °C), temper (540 °C), 25 mm round*

The apparent increase in hardness, at low VEDs, is only a material response caused by the used load. Here sub-surface porosity will lead to lower hardness values, even if it looks like a "good" hardness indentation. However, once a high enough volumetric energy density is used this lack of fusion porosity disappears and with it the subsurface porosity that affects the measured hardness. Once the influence of subsurface porosity has been eliminated an almost linear trend between the hardness and the VED can be found, the higher the VED the lower the hardness. An explanation for this trend could be tempering of the material during processing. With higher VED and laser power the melt pool depth increases, resulting in more re-melting and reheating of the previously deposited layers. This causes the material to be at elevated temperatures for a longer period of time, leading to an increased tempering effect that reduces the hardness significantly. In a similar way, tempering could also explain the hardness gradient from top to bottom of a sample cross section. This is discussed in detail in 5.3.3 Influence of Orientation.

### 5.3.1  Influence of Laser Power

When the laser power is increased the hardness of the material decreases, as can be seen in Figure 49, even if the same VED is used. A possible explanation for this could again be tempering. Due to the higher energy input the there is a deeper melt pool resulting in more re-melting and reheating of previous layers.



*Figure 49: Hardness development for 4130 at different laser powers and VEDs; the error bars represent one standard deviation*

### 5.3.2  Influence of Composition

Out of the examined alloying elements the one that had the largest impact on the hardness was carbon. In Figure 50 the development of hardness, with volumetric energy density and increasing

carbon content, can be seen. 4340 has a similar carbon content as 4140 but a significantly higher nickel content, which could explain the even higher hardness. A more likely possibility is the loss of carbon during processing. In Table 5 the carbon loss for several alloys is calculated. Even though the initial carbon content of the 4340 alloy is lower than the one of other alloys, the final components have the highest carbon content resulting from a smaller carbon loss during processing. This can explain the higher hardness of 4340 compared to 4140LC and 4140HC since the actual carbon content in the 4340 part is higher than the in the processed 4140 parts. Since 4340 is the only alloy containing nickel whose carbon loss was measured nickel could help prevent carbon loss, but this still needs to be investigated further.



*Figure 50: Influence of composition on hardness for various compositions; the error bars represent one standard deviation*

### 5.3.3 Influence of Orientation

From the hardness measurements it was found that there is a hardness gradient within a single specimen that depended on the orientation of the sample (Figure 52). It was found that the hardness will decrease as you move from the top to the bottom of the specimen. This trend could result as the initially deposited material spends more time at higher temperatures than the material deposited nearing the end of the process. The elevated temperature could cause the material to be more tempered, leading to a lower hardness. Horizontally the hardness gradient is dependent on the direction of the gas flow. The section of the sample that is located closer to the gas outlet shows the highest hardness, which could be explained through a higher cooling rate in this part of the material. This region is in direct contact with the inert gas while the other side is not. The gradient explains the high standard deviation when taking an average over all indentations. From top to bottom the hardness differences on average are small, 10-20 HV. However, the differences following the gas flow direction can exceed 30 HV. Another explanation could be varying heat dissipation based on the location on the building plate. The origin was not proven in this work.

### 5.3.4  M100 vs. M290

The trends of the hardness development with volumetric energy density seem the same for both, the M100 and the M290. However, when comparing the actual hardness values between the two machines, the hardness of the M290 samples was 40 HV higher for samples produced without building plate preheating, 20 HV higher with a preheating of 100 °C and approximately the same when using a preheating of 180 °C (Figure 51).



*Figure 51: Comparison of average hardness of 4130 specimen produced under different preheating conditions, laser powers and machines*

Using the M290 machine the hardness gradient over the cross section of the specimens decreases as can be seen in Figure 52. The hardness decrease along the z-axis is reduced when heating the build plate. Initially a 20 HV difference between the lowest and highest indentation was measured, when no build plate preheating was used. At a build plate preheating of 180 °C no significant hardness

# 6 Conclusion

In conclusion, processing windows for 4130, 8620 4140 LC and 4340 alloys were established for an EOS M100 machine and a processing window was established for the 4130 alloy when using an EOS M290 machine. In terms of takeaways the main ones are as follows:

1. The dominant types of porosity are lack of fusion (at low VED) and gas and keyholing porosity (at high VED).

2. For the alloys 4130, 8620, 4140LC and 4340 an ideal processing window was found within the constraints of fixed parameters on the M100 and for 4130 on the M290.

3. Process parameters developed on the M100 can be used as an estimation for the M290 but have to be optimised for the other machine. In the case of M100 and M290 the processing window of the M290 is wider and provides a higher process stability.

The results presented in this thesis start to lay the groundwork for understanding the behaviour of more complex ferrous alloys during L-PBF. This understanding can lead to the development of ferrous alloys specifically designed for the use in AM opens up possibilities for a new category of processable material in L-PBF. Although, a limited set of parameters were tested and processing is not optimised, it was shown that despite of relatively high carbon content, crack free parts can be produced. Furthermore, it was shown that transferring processing conditions between different machines can be done with limited success. This presents a problem when wanting to optimise processing conditions in a smaller machine and then transfer the results to industrial size, also when comparing research results obtained with various machines.

## 6.1 Suggestions for Further Research

In terms of future work, the processing windows of the 8620, 4140 LC and 4340 alloys should be investigated when using an EOS M290 machine. For the 4130 alloy more advanced parameters, like up- and down-skin parameters and contour scanning, should be developed to help further optimize the chosen processing conditions.

Mechanical properties like yield strength, ultimate tensile strength, elongation and impact toughness should be tested to compare the capabilities of L-PBF to conventional manufacturing processes and compare the produced properties when the processing conditions are changed.

The reason for the observed cracking in 4140 HC alloy should also be further investigated. This could involve a more detailed chemical analysis of the material and more accurate chemical analysis than EDS should be used for investigation of inclusions to determine the composition and origin.

A closer look on the microstructure should be taken as well. Determining the phases present and their ratios, for example via transmission electron microscopy.

Given the results for the processing windows the parameters can be further improved regarding the building time. With increasing the layer thickness and hatch spacing, printing time could be significantly reduced. Especially at high VEDs the potential to increase layer thickness is given.

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# 8   Appendix

## 8.1   ThermoCalc phase diagram simulations

All simulations were performed with the TCFE8: Steels/Fe-Alloys v8.1 database in ThermoCalc.

*Melting temperatures of initial powder compositions*

| 4130 | 1446 °C |
|------|---------|
| 8620 | 1471 °C |
| 4140 LC | 1429 °C |
| 4140 HC | 1429 °C |
| 4340 | 1425 °C |



*Figure 53: Calculated phase diagram of the 4130 alloy*

Figure 54: Calculated phase diagram of the 8620 alloy



Figure 55: Calculated phase diagram of the 4140 LC alloy

2019.08.27.14.49.33
TCFE8 : Fe, Mn, Cr, Si, Mo, C
Pressure [Pa] = 100000.0, System size [mol] = 1.0, Mass percent Mn = 0.6, Mass percent Cr = 1.0, Mass percent Si = 0.2, Mass percent Mo = 0.2



*Figure 56: Calculated phase diagram of the 4140 HC alloy*

2019.08.27.15.14.29
TCFE8 : Fe, Mn, Cr, Si, Mo, C, Ni
Pressure [Pa] = 100000.0, System size [mol] = 1.0, Mass percent Mn = 0.6, Mass percent Cr = 0.9, Mass percent Si = 0.17, Mass percent Mo = 0.3, Mass percent Ni = 1.9



*Figure 57: Calculated phase diagram of the 4340 alloy*