



PRODUCTION AND CHARACTERIZATION OF 17-4PH STAINLESS STEEL PROCESSED BY LASER POWDER BED FUSION AND SUBSEQUENT HEAT TREATMENT

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MASTER'S DEGREE DISSERTATION IN MATERIALS ENGINEERING SUBMITTED TO
THE FACULTY OF ENGINEERING OF UNIVERSITY OF PORTO

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<i>TÍTULO</i>	Production and Characterization of 17-4PH Stainless Steel Processed by Laser Powder Bed Fusion and Subsequent Heat Treatment	
<i>DATA</i>	28 september 2026	
<i>LOCAL</i>	Faculdade de Engenharia da Universidade do Porto	
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ABSTRACT

17-4 PH stainless steel is widely used in demanding applications due to its combination of high strength, toughness, and corrosion resistance. This study investigates the production and characterization of LPBF-fabricated 17-4 PH stainless steel, with particular emphasis on the influence of post-heat treatment routes and build orientations on its microstructural evolution and mechanical behavior. The gas-atomized powder was characterized in terms of particle size distribution and morphology, showing a predominantly spherical morphology and a particle size distribution suitable for LPBF processing. Specimens were subsequently manufactured by Laser Powder Bed Fusion (LPBF) in horizontal (0°), inclined (45°), and vertical (90°) orientations. The as-built material and heat-treated conditions were characterized using Scanning Electron Microscopy (SEM), Electron Backscatter Diffraction (EBSD), Vickers microhardness (HV0.5), tensile testing, and fractography. Direct aging (H900) and combined solution annealing and aging ($1242^\circ\text{C} + \text{H900}$) were investigated to evaluate their effects on the microstructure and mechanical properties.

The as-built condition exhibited a predominantly martensitic microstructure with approximately 10.1% FCC phase, associated with retained austenite. Solution annealing at 1242°C reduced the FCC fraction to 0.8%, resulting in an almost fully BCC microstructure, while direct H900 aging resulted in 18.9% FCC phase. The combined $1242^\circ\text{C} + \text{H900}$ treatment resulted in 8.9% FCC phase. Microhardness increased from 302 HV0.5 in the as-built condition to 449 HV0.5 after H900 aging, reaching 538 HV0.5 after the combined $1242^\circ\text{C} + \text{H900}$ treatment. Tensile testing revealed differences in mechanical response with build orientation and heat treatment, with the H900 condition showing a favorable strength-ductility balance under the investigated conditions. The combined $1242^\circ\text{C} + \text{H900}$ treatment achieved the highest hardness but resulted in reduced ductility compared with direct aging. Overall, the results demonstrate that the microstructural and mechanical response of LPBF-fabricated 17-4 PH stainless steel is strongly influenced by the thermal history and build orientation.

Keywords

17-4 PH stainless steel; laser powder bed fusion; solution annealing; direct aging; build orientations; microstructure; mechanical properties.

ACKNOWLEDGEMENTS

This thesis marks the end of a chapter that, despite all the work behind it, I know I could never have completed entirely on my own. There were difficult days, moments of doubt, and many times when things did not go quite as planned. Looking back, what stands out the most is not only the work itself, but the people who made this journey easier, more meaningful, and, at times, simply possible.

I am deeply grateful to Professor Aida Moreira, for her unconditional support and guidance throughout this work. Thank you for being present from the beginning to the end, for your availability whenever I needed it, and for the confidence you placed in me throughout this process. More than just guiding this thesis, you were someone I could count on when things became difficult, and that made a greater difference than I can put into words. I am truly grateful to have had you as my supervisor.

To my mother and my sister, for being there through every step of this journey. Thank you for your constant presence, for listening to me talk about things you probably did not understand, for celebrating my achievements as if they were your own, and for being there on the days when I needed nothing more than to come home and feel that everything would eventually be fine. You have always been one of the most important parts of everything I achieve, and this is no exception. Thank you for believing in me, even when I struggled to believe in myself.

To Rui, for being by my side throughout this entire journey. Thank you for listening to me through the stressful days, for putting up with my frustrations, my doubts and all the moments when my mind was somewhere else. Thank you for knowing when I needed advice and when I simply needed someone beside me. Your patience, support and affection made the difficult days easier and the good ones even better. Having you beside me throughout this chapter meant more than I could ever properly put into words.

To Kika, for being there in all the ways that matter. Thank you for every conversation, every piece of advice, every time you listened to me complain, overthink or simply needed to talk. Thank you for helping me whenever I needed it, for celebrating the good moments with me and for always being someone I could count on. So many moments throughout these years have you in them, and I am grateful for every single one. Some people simply become part of your life, and eventually you cannot imagine having lived these years without them.

To all of you, thank you for being part of this chapter of my life. This thesis may have my name on it, but the journey behind it was never mine alone.

INDEX

LIST OF FIGURES.....	vii
LIST OF TABLES.....	ix
LIST OF ABBREVIATIONS.....	xi
1. INTRODUCTION.....	1
1.1. Contextualization and Motivation.....	1
1.2. Objectives and Strategy.....	2
1.3. Dissertation Structure.....	2
2. LITERATURE REVIEW.....	3
2.1. Additive Manufacturing.....	3
2.1.1. Classification of Additive Manufacturing Technologies.....	4
2.1.2. Advantages and Limitations of Additive Manufacturing.....	5
2.1.3. Applications for Additive Manufacturing.....	5
2.2. State of the art on LPBF 17-4 PH.....	6
2.3. Laser Powder Bed Fusion.....	7
2.3.1. Printing Parameters for LPBF processes.....	8
2.3.2. Powder Characterization.....	10
2.3.3. Defects in Laser Powder Bed Fusion Components.....	11
2.4. 17-4 PH Stainless Steel.....	13
2.4.1. Microstructure of 17-4 PH Stainless Steel.....	13
2.4.2. Chemical Composition of 17-4 PH Stainless Steel.....	14
2.4.3. Applications for 17-4 PH Stainless Steel.....	15
2.5. Post-Heat Treatments.....	15
2.5.1. Solution Annealing.....	16
2.5.2. Aging Treatment.....	16
3. EXPERIMENTAL PROCEDURE.....	19
3.1. Material.....	20
3.2. Powder Characterization.....	20
3.3. Laser Powder Bed Fusion: Process Parameters.....	21
3.4. Design and Fabrication of the samples: CAD Model.....	22
3.4.1. Characterization samples.....	22

3.4.2.	Metallographic Preparation and Characterization	24
3.4.3.	Tensile Samples.....	25
3.4.4.	Fracture Characterization.....	26
3.5.	Post-Processing Heat Treatments of LPBF 17-4 PH Stainless Steel: Annealing and Aging	27
4.	RESULTS AND DISCUSSION.....	31
4.1.	Powder Characterization.....	31
4.1.1.	Particle Size Distribution	31
4.1.2.	Morphological Defects of Powder Particles	32
4.2.	Microhardness.....	34
4.3.	Microstructural characterization.....	36
4.3.1.	SEM Analysis	36
4.3.2.	EBSA Analysis	39
4.4.	Tensile Test	43
4.5.	Fracture Surface Analysis	49
5.	CONCLUSIONS	55
5.1.	Future work perspectives	56

LIST OF FIGURES

Figure 1. Sequential steps in AM process flow[8];	3
Figure 2. Scheme of the LPBF process; [15]	8
Figure 3. LPBF process parameters; [18]	9
Figure 4. The schematic of gas atomization process and the formation of internal pores in the large particle; [19]	10
Figure 5. Representative defects observed in LPBF fabricated components: (a) balling phenomenon, showing the formation of spherical droplets due to melt pool instability and poor wettability;[21] (b) delamination between layers caused by insufficient interlayer bonding;[22] (c) swelling effect characterized by surface bulging resulting from excessive heat input and over-melting; [22](d) schematic illustration of warping mechanisms, including distortion due to thermal gradients and residual stress accumulation [23].....	11
Figure 6. Possible phases of 17-4 PH stainless steel represented by a red dot according to Schaeffler Diagram;[12]	13
Figure 7. 17-4 PH components produced by LPBF a) fuel injector with conformal cooling channels; b) helicopter engine part; c) automotive engine gear; d) plastic injection mold with cooling channels; [31]	15
Figure 8. Flowchart summarizing the experimental methodology adopted in this work, including powder characterization, LPBF fabrication of the specimens, post-heat treatments, and microstructural and mechanical characterization.	19
Figure 9. Schematic representation of the zig-zag laser scanning strategy used during the LPBF process, illustrating the rotation of 67° between successive layers. The scanning direction is alternated and progressively rotated from one layer to the next.	22
Figure 10. Schematic representation of the specimens manufactured by LPBF: (a) geometry and dimensions of the cubic specimens used for microhardness measurements; (b) geometry and dimensions of the parallelepiped specimens after removal of the lower 10 mm section, resulting in a final height of 37 mm; (c) arrangement of the parallelepiped specimens on the build platform during fabrication.....	23
Figure 11. Representation of the sixteen samples after the cutting step	24
Figure 12. Distribution and numbering of the sixteen Vickers hardness indentations on the top and lateral surface of the samples	25
Figure 13. (a) Geometry and dimensions of the tensile samples; (b) Printing orientations (0°, 45°, and 90°) relative to the build platform.	26
Figure 14. Schematic representation of the heat-treatment conditions, including the annealing and aging processes and their corresponding temperatures and holding times	27
Figure 15. Particle size distribution of 17-4 PH SS powder used in this study;	31
Figure 16. Powder morphology observed by (a) SEM and (b) optical microscopy (OM).	32
Figure 17. Representative SEM images of powder particle defects: (a) internal pore and oxidation zone; (b) satellite particle	32
Figure 18. Energy Dispersive X-ray Spectroscopy (EDS) analysis of the powder 17-4 PH stainless steel sample: (a) SEM micrograph showing the selected region of analysis, and (b) corresponding EDS spectrum confirming the presence of the main alloying elements (Fe, Cr, Ni, Cu) alongside a oxide region..	33
Figure 19. Average Vickers microhardness (HV0.5) values obtained from the cutting and lateral	

surfaces of the samples, together with the corresponding standard deviations	34
Figure 20. SEM images of the alloy under different processing conditions: as-built state, annealing condition (1071 °C), aging condition (H900), and combined heat treatment (1071 °C + H900), illustrating the microstructural evolution induced by post heat treatment.	37
Figure 21. SEM images of the LPBF 17-4 PH stainless steel after (a) annealing at 1242 °C and (b) annealing at 1242 °C followed by H900 aging (1242 °C + H900), illustrating the microstructural evolution induced by the combined heat treatment.	38
Figure 22. EBSD maps illustrate the microstructural evolution of LPBF-fabricated 17-4 PH stainless steel after different heat treatment conditions. From left to right, the columns present the Image Quality (IQ), Inverse Pole Figure (IPF), grain boundary and phase maps. From top to bottom, the rows correspond to the as-built, solution-treated at 1242 °C, direct-aged (H900), and solution-treated at 1242 °C + H900 conditions.....	40
Figure 23. Stress strain curves of the samples 17-4 PH SS produced by LPBF in the H900 condition for the three build orientations (0°, 45° and 90°).	475
Figure 24. Stress strain curves of the samples 17-4 PH SS produced by LPBF in the combined annealing at 1242 °C + H900 condition for the three build orientations (0°, 45° and 90°).....	477
Figure 25. SEM fracture surface morphology of tensile specimens fabricated in the horizontal, inclined (45°), and vertical build orientations after the H900 and 1242 °C + H900 heat treatments	50

LIST OF TABLES

Table 1. Description of additive manufacturing technologies based on the ISO/ASTM 52900:2015 [8];	4
Table 2. Microstructural and mechanical response of LPBF 17-4 PH under different post-processing conditions.....	7
Table 3. Chemical Composition according to ASTM A747/A747M;[33]	14
Table 4. Standard aging parameters and expected property trends for 17-4 PH stainless steel [33]	17
Table 5. Chemical Composition of the powder provided by the supplier; [37]	20
Table 6. Process Parameters used to produce 17-4 PH stainless steel components;	21
Table 7. Summary of the heat treatment combinations evaluated for the LPBF manufactured 17-4PH stainless steel samples;	28
Table 8. Microhardness values (HV0.5) measured at the side and cut regions across the different tested conditions.	35
Table 9. Summary of the tensile properties of the 17-4 PH SS produced by LPBF in the H900 condition, including yield strength ($R_{p0.2}$), ultimate tensile strength (R_m), elongation and reduction in area (RA) for each build orientation.....	45
Table 10. Summary of the tensile properties of the 17-4 PH SS produced by LPBF in the combined annealing at 1242 °C + H900 condition, including yield strength ($R_{p0.2}$), ultimate tensile strength (R_m), elongation and reduction in area (RA) for each build orientation.	47

LIST OF ABBREVIATIONS

AM	Additive Manufacturing
ASTM	American Society for Testing and Material
BCC	Body-Centered Cubic
CAD	Computer Aided Design
Cu	Copper
DLS	Dynamic Light Scattering
EBSD	Electron Backscatter Diffraction
EDS	Energy-dispersive X-ray Spectroscopy
FCC	Face-Centered Cubic
IQ	Image Quality
ISO	International Organization for Standardization
IPF	Inverse Pole Figure
LPBF	Laser Powder Bed Fusion
Ms	Martensite Start Temperature
OM	Optical Microscopy
PH	Precipitation - Hardening
RA	Reduction in Area
R_m	Ultimate Tensile Strength
R_{p0.2}	0.2% Proof Stress
SEM	Scanning Electron Microscopy
SS	Stainless Steel
STL	Standard Tessellation Language
2D	Two-Dimensional
3D	Three-Dimensional

1. INTRODUCTION

This dissertation was developed as part of the Master's Degree in Materials Engineering at the Faculty of Engineering of the University of Porto. The experimental work was carried out using metallic powder supplied by Sandvik[1]. The present chapter provides an overview of the dissertation topic, highlighting the motivation and objectives of the study. Additionally, it provides a description of the structure and content organization of the document [1]

1.1. Contextualization and Motivation

Additive Manufacturing (AM) has emerged as one of the most significant manufacturing technologies in recent decades, enabling the production of complex geometries that are difficult or impossible to achieve using conventional manufacturing methods. Among the different AM technologies, Laser Powder Bed Fusion (LPBF) has attracted considerable industrial interest due to its ability to manufacture near fully dense metallic components with high dimensional accuracy and excellent geometric flexibility. Consequently, LPBF has been increasingly adopted in sectors such as aerospace, biomedical, automotive, tooling, and energy, where high-performance materials and optimized designs are required.

Among the stainless steels processed by LPBF, precipitation-hardening 17-4 PH stainless steel has gained particular attention because of its excellent combination of high strength, good corrosion resistance, and satisfactory toughness. These characteristics make it suitable for demanding engineering applications, including structural components, molds, valves, shafts, and aerospace parts. However, the rapid melting and solidification cycles associated with the LPBF process generate a microstructure that differs significantly from that obtained by conventional manufacturing routes, leading to residual stresses, microstructural heterogeneity, retained austenite, and anisotropic mechanical properties.

To tailor the final properties of LPBF-produced 17-4 PH stainless steel, post-heat treatments are commonly applied. Aging treatments promote precipitation hardening through the formation of Cu-rich precipitates, while solution annealing aims to homogenize the microstructure, reduce chemical segregation, and dissolve undesired phases prior to aging. Although these treatments are well established for conventionally manufactured materials, their effects on LPBF-produced components remain an active research topic due to the unique microstructure generated during additive manufacturing. Furthermore, the influence of build orientation on the response to heat treatment and on the resulting mechanical performance has not yet been fully clarified. Therefore, a comprehensive understanding of the relationship between LPBF processing, heat treatment, microstructural evolution, and mechanical behavior is essential for the reliable implementation of additively manufactured 17-4 PH stainless steel components in

industrial applications. This dissertation aims to contribute to this understanding by investigating the influence of different post-heat treatment routes on the microstructure, phase evolution, hardness, tensile properties, and fracture behavior of LPBF-fabricated 17-4 PH stainless steel, considering different build orientations.

1.2. Objectives and Strategy

Following the contextualization of the dissertation and relevance of the materials and processes involved, is important to define the main goals established for the present work. The primary objectives of these dissertations are presented as follows:

- (i) Manufacture of 17-4 PH stainless steel specimens by LPBF using selected processing parameters and three building orientations (0°, 45° and 90°);
- (ii) Assess selected direct aging, solution annealing and combined heat treatment routes;
- (iii) Characterize powder and microstructural/ phase evolution by OM/SEM/EDS and EBSD;
- (iv) Evaluate hardness, tensile response and fracture morphology;
- (v) Correlate processing-microstructure-properties.

1.3. Dissertation Structure

This dissertation is organized into five chapters. Following this introductory chapter, Chapter 2 reviews the literature on 17-4 PH stainless steel, Laser Powder Bed Fusion (LPBF), and the influence of post-heat treatments on the resulting microstructure and mechanical properties. Chapter 3 describes the experimental methodology, including powder characterization, specimen fabrication by LPBF, post-heat treatments, and the characterization techniques employed throughout this work. Chapter 4 presents and discusses the experimental results obtained from powder characterization, SEM and EBSD microstructural analyses, microhardness measurements, tensile testing, and fracture surface characterization. Finally, Chapter 5 summarizes the main conclusions drawn from this study and outlines recommendations for future research.

2. LITERATURE REVIEW

This chapter reviews the literature relevant to LPBF processing of 17-4 PH stainless steel, with emphasis on powder characteristics, process parameters and defects, as well as the microstructural and mechanical response to solution treatment, aging and build orientation.

2.1. Additive Manufacturing

Additive Manufacturing (AM), commonly known as three-dimensional (3D) printing, includes a group of manufacturing technologies capable of producing 3D components through the successive addition of material layer-by-layer until the final geometry is achieved. Unlike subtractive processes such as machining, AM builds components layer by layer from a digital model and can reduce material waste while enabling complex geometries [2-5] .

The first additive manufacturing technologies emerged during the 1980s and were developed for rapid prototyping applications. These systems were initially used to fabricate prototypes for visualization, design and functional verification. However, advances in material science, machine capabilities and computational tools allowed AM technologies to go from prototyping into a viable manufacturing technology capable of producing functional components with mechanical properties comparable to those obtained through traditional manufacturing methods [2-5].

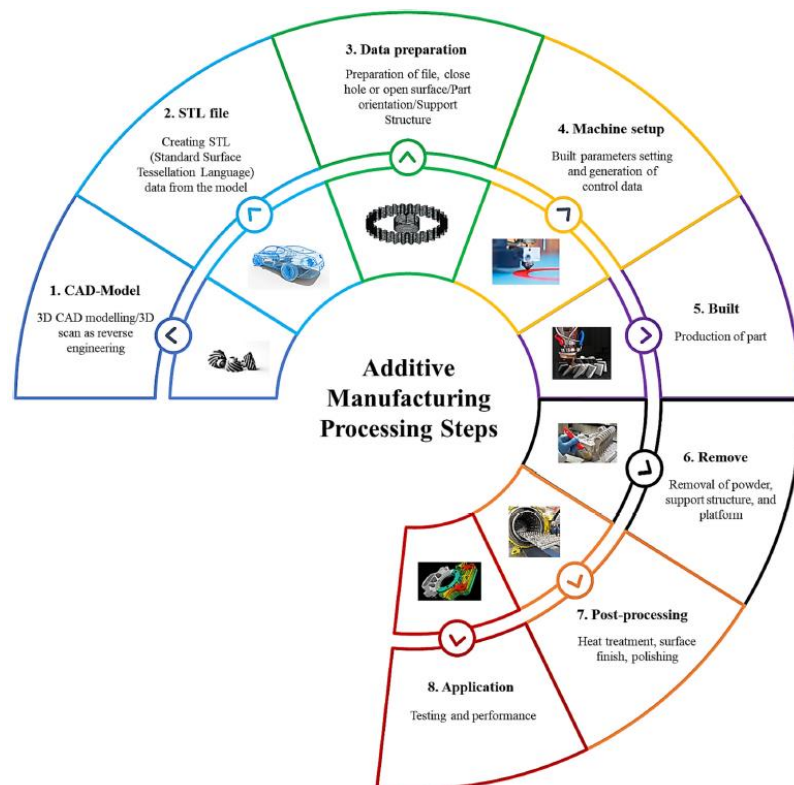


Figure 1. Sequential steps in AM process flow[6];

The additive manufacturing workflow begins with the creation of a 3D model using computer-aided design (CAD). This CAD contains all the geometric information required to produce the component. After the design stage the digital model needs to be converted into a format that can be interpreted by manufacturing system, usually Standard Tessellation Language (STL) format [2-4].

Once the model is prepared, several process parameters must be defined, including build orientation, layer thickness, laser power, scan speed, hatch spacing and preheating conditions [5]. Once these parameters are established the manufacturing process can begin where the component is fabricated through the successive deposition and consolidation of the layers. Depending on the size and complexity of the component, this process can take hours or even days [2,3].

After the printing stage is completed, the component is removed from the building platform and usually undergoes post-processing operations. These may include the removal of support structures, heat treatments and surface finish processes to improve surface quality and mechanical properties [2,3,7].

2.1.1. Classification of Additive Manufacturing Technologies

Additive manufacturing technologies can be classified into several categories depending on the material feedstock, energy source, and deposition mechanism used during the fabrication process. According to ASTM International [8], AM processes are divided into seven main categories: powder bed fusion, directed energy deposition, binder jetting, material jetting, material extrusion, vat photopolymerization and sheet lamination [3,4,7].

Table 1. Description of additive manufacturing technologies based on the ISO/ASTM 52900:2015 [8];

ADDITIVE MANUFACTURING TECHNOLOGIES DESCRIPTION	
AM Technologies	Description
Powder Bed Fusion	Selective fusion of powder using a thermal energy source
Directed Energy Deposition	Fusion of a material during deposition by melting using focused thermal energy (laser or electron beam)
Binder Jetting	Selective deposition of a liquid binder to bond powder particles
Material Jetting	Selective deposition of droplets of build material, typically photopolymers or waxes
Material Extrusion	Extrusion of material through a nozzle to build components layer by layer
Vat Photopolymerization	Selective curing of liquid photopolymer using a light source
Sheet Lamination	Bonding of material sheets to form a three-dimensional part

These technologies differ in terms of mechanisms used to deposit and consolidate materials, which allows additive manufacturing processes to be applied on a wide range of materials such as polymers, metals, ceramics and composite materials [3,4].

2.1.2. Advantages and Limitations of Additive Manufacturing

Additive manufacturing offers several advantages compared with the traditional manufacturing methods, such as the ability to produce highly complex geometries that would be difficult or impossible to produce through traditional techniques. Since the components are built layer by layer directly from digital models, AM provides design flexibility, enabling the development of optimized structures with improved functionality and performance [2,3,4,5].

Another important advantage is the reduction of material waste, since the material is only deposited where is required. This characteristic is beneficial when processing high value materials such as advanced alloys used in aerospace and biomedical applications [3,5]. Additionally, additive manufacturing can reduce the number of manufacturing steps required for complex components and simplify supply chains by enabling the production of directly from digital files. This way the production time is also reduced [2,4,9].

Despite these advantages, additive manufacturing also presents some limitations. During fabrication, defects such as porosity, lack of fusion and surface roughness may occur, which can negatively affect the mechanical properties of the final component [2,3,5,6]. These defects are associated with the complex thermal cycle during the layer-by-layer manufacturing process [2,7].

In metal additive manufacturing processes, the repeated cycles of rapid heating and cooling during layer deposition can lead to the development of residual stress and microstructural heterogeneities within the material [2,3,7].

2.1.3. Applications for Additive Manufacturing

Additive manufacturing has been adopted in multiple industrial sectors due to the ability to produce complex and optimized components [3,5,9]. Industries such as aerospace, automotive, biomedical engineering and structural engineering have widely explored AM technologies for prototyping and end-use applications [3,9].

In aerospace industry additive manufacturing is used to produce lightweight components with optimized internal geometries, contributing to weight reduction and improved fuel efficiency. In biomedical applications AM enables the production of customized implants and medical devices that can be adapted to the patient's specific anatomical requirements [3,9].

2.2. State of the art on LPBF 17-4 PH

Laser powder bed fusion (LPBF) of precipitation-hardening 17-4 PH stainless steel drawn significant attention due to its capability to produce complex near-net-shape components with unique microstructural and mechanical attributes [5]. Unlike conventional casting or wrought processes, LPBF involves high cooling rates and localized thermal gradients [10], resulting in a non-equilibrium as-built microstructure predominantly composed of retained austenite (FCC) embedded in a fine-grained lath martensite matrix [4,7,11].

The mechanical performance and microstructural evolution of LPBF of 17-4 PH are dependent on post-processing heat treatments, which are generally categorized into direct aging and solution annealing followed by aging [9]. In the as-built condition, parts typically exhibit high hardness (around 300 and 350 HV), due to solid solution strengthening and high dislocation densities. Alongside moderate yield and ultimate tensile strengths, but with limited ductility and pronounced mechanical anisotropy caused by epitaxial grain growth along the build direction [12].

Applying a direct aging treatment promotes the precipitation copper-rich within the martensitic matrix, driving an increase in hardness and tensile strength [9,13]. However, direct aging preserves the thermal residual stresses and crystallographic textures inherited from the LPBF processes, which sustains directional dependence in yielding and tensile behavior [14]. Conversely, introducing a solution annealing step prior to aging helps to homogenize the microstructure, dissolve micro segregation, and transform the retained FCC phase, improving ductility at the peak strength [15,16,17]. Furthermore, build orientation (0° , 45° and 90°) continuously plays a governing role in mechanical anisotropy, influencing the scatter of offset yield strength, and ultimate tensile strength across different directions [14,18,19]. Table 2 summarizes recent literature findings regarding the microstructural features of, and mechanical properties of 17-4 PH processed by LPBF under different thermal conditions and build orientations.

Table 2. Microstructural and mechanical response of LPBF 17-4 PH under different post-processing conditions

SUMMARY OF LPBF 17-4 PH MICROSTRUCTURAL AND MECHANICAL RESPONSE			
CONDITION	As-Built	Direct Aging	Solution Annealing + Aging
Build Orientation	Various	0°, 45° and 90°	Various
Microstructure / Phase	Retained austenite (FCC) in lath martensite matrix	Martensite + copper rich precipitates	Homogenized microstructure (dissolved microsegregation)
Hardness and Strength Response	300 - 350 HV; moderate yield and ultimate tensile strength	Increased hardness and tensile strength	Balance strength response
Ductility / Elongation	Limited ductility; pronounced mechanical anisotropy	Affected by directional dependence	Improved ductility

2.3. Laser Powder Bed Fusion

Laser Powder Bed Fusion (LPBF) is one of the most used metal additive manufacturing technologies that enables the production of complex metallic components through the melting of the powder, layer by layer, using a high energy laser beam [2,3,18]. A schematic representation of the LPBF process is shown in Figure 2, where the main components of the system can be observed, including the laser source, powder bed, recoater arm and build platform. In this process a thin layer of metal powder is distributed uniformly over the build platform. After this a focused laser will selectively melt a specific region, according to the geometry that was defined on the CAD model [3,20]. After the selective melting of each layer the build platform moves downward, and a new powder layer is deposited by a recoating system. Then the laser scans the next section and re-melt the previous solidified layer to guarantee the bonding between the successive layers [3,9]. The repetition of this process will lead us to the fabrication of 3D metallic components with a high density and near net shape geometry [9,20], that can be used in several industries such as aerospace, automotive, tooling and medical devices.

Laser Powder Bed Fusion is characterized by the rapid thermal cycle during the process. The interaction between the high energy laser beam and the metallic powder creates a localized melting followed by cooling rate that may reach the order of 10^5 - 10^6 , depending on the local thermal conditions [4]. The combination

between the repeated thermal cycling, caused by the successive layer deposition, and the rapid solidification led to the formation of unique microstructure [4,15].

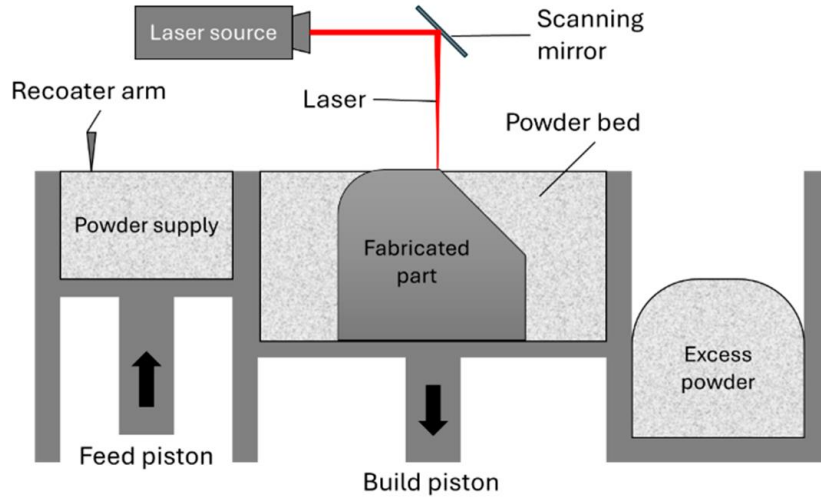


Figure 2. Scheme of the LPBF process; [21]

This technology has been widely used in many industries including aerospace, biomedical engineering and automotive, due to the ability to be used on a wide range of material such as stainless steel, titanium and aluminum alloys and nickel-based superalloys [2,5]. The interest of LPBF has been growing because of the capability to produce complex geometries with high dimensional accuracy and mechanical properties that can be comparable with traditional methods [20].

2.3.1. Printing Parameters for LPBF processes

To achieve an almost fully dense and ensure the functional performance of the components that are produced by LPBF, several process parameters need to be controlled, as they directly influence the interaction between the laser beam and the metallic powder [22]. The most relevant parameters include laser power, scan speed, hatch spacing, layer thickness, scan strategy, build orientation and platform preheating [3,23]. These parameters were described by using a combined parameter that is called volumetric laser energy density E_v (J/mm³), which represents the amount of laser energy per unit volume of printed material during the melting process (see Equation 1) [20].

$$E_v = \frac{P}{v \times h \times t} \quad (\text{Equation 1})$$

This parameter is defined mathematically in Equation 1, where P is the laser power (W), v (mm/s) is the laser scanning speed, t (mm) is the layer thickness, and h (mm) is the hatch spacing (distance between laser scan lines) [22].

The combination of these parameters determines the total energy input delivered to the powder bed during the process. Energy density is important because it guarantees the stability of the melt pool and the quality of the final component. Insufficient energy can lead to an incomplete melting of the powder particles and formation of lack-of-fusion, while excessive energy can result in keyhole porosity and unstable melt pool [3]. The different process parameters of LPBF are present in Figure 3.

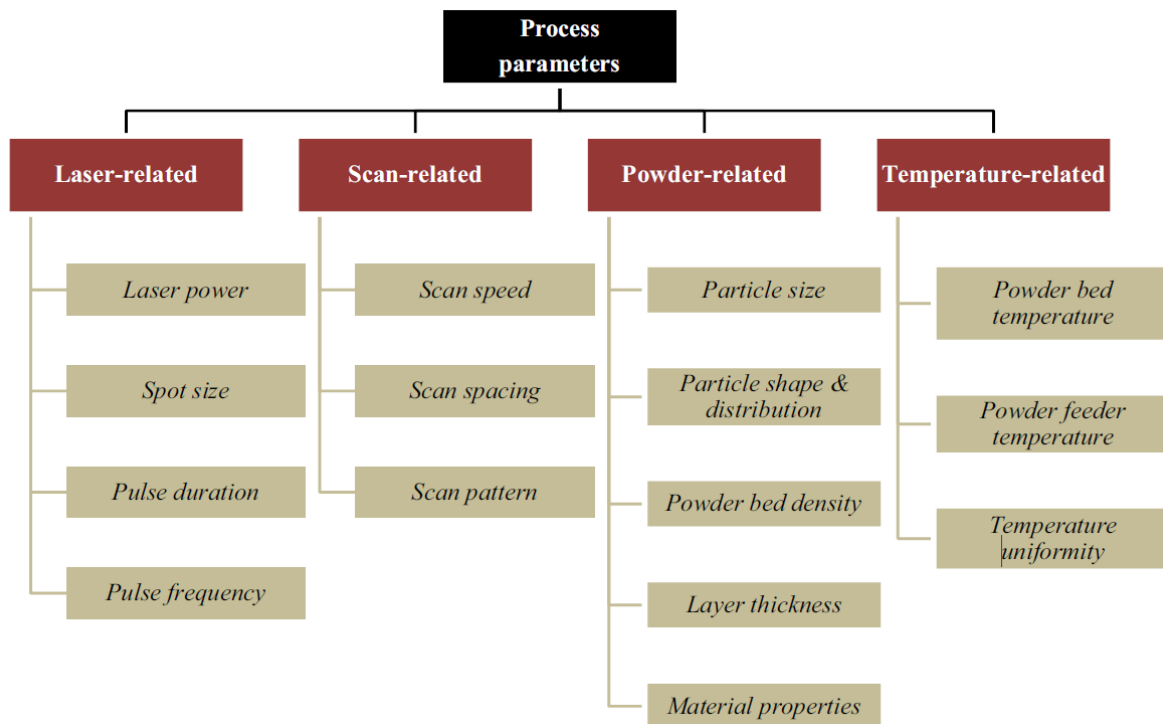


Figure 3. LPBF process parameters; [24]

Laser scanning strategies are important to control the thermal history of the component during the fabrication, since different patterns, such as chessboard, hexagonal or stripe strategies will influence the distribution of thermal gradients and cooling rates across the build platform, affecting the residual stress formation and microstructural evolution [5]. With the alternation of the scanning direction between layers is possible to reduce the heat accumulation and get a uniform thermal field during the process.

Layer thickness and hatch spacing are also important parameters that influence the overlapping of adjacent laser track and bonding between successive layers. These parameters need to be optimized to ensure the formation of a stable melt pool and guarantee a high part density and dimensional accuracy [3,22]. Build orientation

and preheating conditions also affect the mechanical performance and microstructural characteristics of the LPBF components [20,22].

2.3.2. Powder Characterization

In LPBF processes the powder feedstock is fundamental to determine the quality of the produced components. Powder characteristics such as particle size distribution, morphology and chemical composition influences the powder spreading behavior, packing density and melting efficiency during the process.

For LPBF applications spherical powder particles are more often used due to their superior flowability that enables the formation of a homogeneous powder layer during the recoating process. However, during the fabrication of gas atomized powders deviations from ideal spherical morphology can occur, leading to surface irregularities such as satellite particles, that are formed by the adhesion of fine particles onto larger ones during the solidification step.

Powder used in LPBF is typically produced by atomization techniques, such as water or gas atomization. Gas atomization is the most widely used method for high-performance alloys that promote a rapid solidification of molten droplets in an inert gas stream (argon or nitrogen), resulting in a predominantly spherical particles morphology, lower oxygen content, and a better flowability compared to water atomization [3,20]. Nevertheless, the process may still lead to the formation of internal porosity due to the gas entrapment during the droplet solification, as illustrated in Figure 4 [25]. It may also result in residual oxide inclusions, depending on the process atmosphere and chemical reactivity of the alloy.

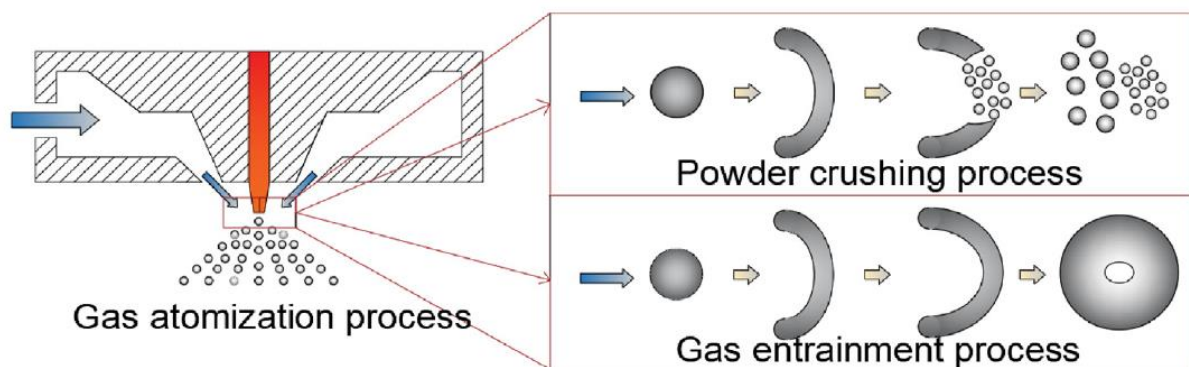


Figure 4. The schematic of gas atomization process and the formation of internal pores in the large particle; [25]

These powder defects can be transferred into the LPBF process and affect the final part quality. Internal porosity on the powder particles if is not fully eliminated during the melting it can contribute to the lack of fusion or residual porosity in the build component. Similarly, surface oxides can reduce proper melting and bonding between layers, leading to more defects and lower mechanical performance.

Satellite particles can also affect the powder flowability and packing density, leading to non-uniform powder layers and increased defect susceptibility in the final part [3,20].

Powders in the tens-of-micrometers range are commonly used for LPBF; the appropriate size distribution depends on the recoating system, layer thickness and machine specification [3,22].

Additionally, powder feedstock is highly susceptible to oxidation due to the high surface area-to-volume ratio. This process is usually conducted on an inert atmosphere, typically argon or nitrogen gas, to prevent oxidation and contamination of molten material during the process [3].

2.3.3. Defects in Laser Powder Bed Fusion Components

Despite the advantages of LPBF, the process is associated with several types of defects that can affect mechanical performance of the produced components. These defects are associated with the complex interaction between process parameters, powder characteristics, and processing environment, as well as the rapid thermal cycle and melt pool instabilities inherent to the layer-by-layer process [2,20,26].

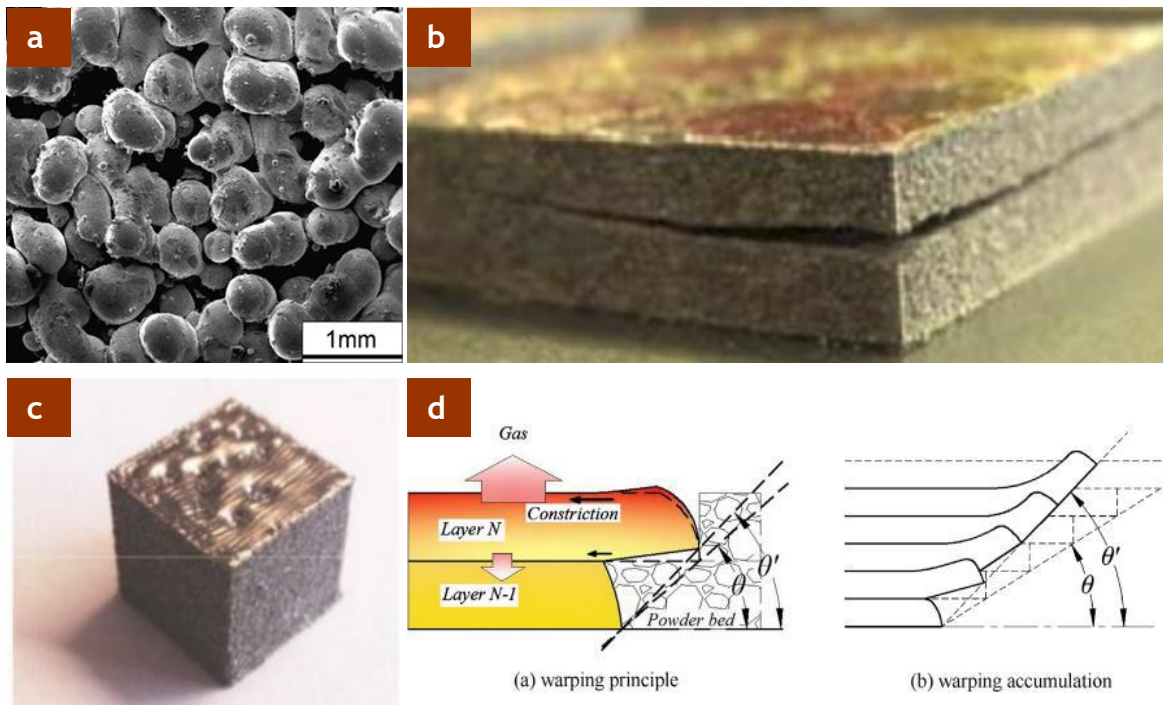


Figure 5. Representative defects observed in LPBF fabricated components: (a) balling phenomenon, showing the formation of spherical droplets due to melt pool instability and poor wettability; [27] (b) delamination between layers caused by insufficient interlayer bonding; [10] (c) swelling effect characterized by surface bulging resulting from excessive heat input and over-melting; [10] (d) schematic illustration of warping mechanisms, including distortion due to thermal gradients and residual stress accumulation [28]

Porosity is one of the most common defects observed in LPBF. It can originate from gas entrapment during melt pool solidification or from lack of fusion caused by insufficient laser energy input, leading to incomplete melting of powder particles. Lack of fusion defects are irregular in shape and can occur between layers due to inadequate bonding, promoting stress concentration and reduction of fatigue resistance and ductility [20,23,26]. In contrast excessive energy input can generate keyhole-induced porosity due to material vaporization and instability of the melt pool [29].

In addition to porosity, several other defects may occur. Balling is a phenomenon associated with the poor wettability of the melt pool, where the molten material forms spherical droplets instead of a continuous track. This defect is influenced by the combination of laser power and scanning speed that are connected to the instability of the melt track formation [29,30]. This phenomenon is shown in Figure 5a [10].

Cracking is another defect that results from high thermal gradients and development of residual stresses. These cracks can propagate through the material, promoting premature failure and compromising mechanical performance [10,26]. Delamination can occur due to insufficient interlayer bonding, that is often caused by inadequate energy input or improper scan overlap, that can get worse by residual stresses [30] (Figure 5b).

Swelling and surface irregularities can be developed due to the excessive heat input, leading to the localized over-melting and formation of material accumulations and surface instabilities. Surface roughness is increased by the adhesion of melted powder particles that are associated with spattering phenomena, vaporization effects or insufficient powder recoating [29]. Distortions and warping are observed because of non-uniform thermal expansion and contraction during heat heating and cooling cycles [28]. These defects are illustrated in Figure 5c and 5d.

Residual stresses are a challenge in LPBF due to the thermal gradients and rapid solidification, that can lead to distortion, cracking and part failure during or after fabrication, this way is often required post processing heat treatments [6,10,29].

Overall defect formation has a direct impact on the mechanical properties of LPBF components. The optimization of the process parameters, scanning strategies and powder characteristics is essential to minimize the formation of defects and guarantee the production of high-quality LPBF components [5,22,26].

2.4. 17-4 PH Stainless Steel

Precipitation - Hardening (PH) stainless steels are a class of alloys that can obtain their mechanical properties through the precipitation hardening mechanism that occurs during the heat treatment. This process involves a high temperature treatment in order to create a supersaturated solid solution, followed by rapid cooling and subsequent aging to form a fine precipitate in the matrix [31].

2.4.1. Microstructure of 17-4 PH Stainless Steel

17-4 PH is one of the most widely used PH stainless steels due to the excellent combination between high strength, toughness and corrosion resistance [4,13]. This alloy is classified as a martensitic PH stainless steel, this is the microstructure, after a conventional process, is mainly martensitic, although small fractions of ferrite or retained austenite can also be present [18,22].

PH stainless steels typically contain three phases: ferrite, austenite and martensite, that can appear in different proportions depending on the alloy composition and thermal history of the material [22]. The phase balance can be estimated using diagrams based on chromium and nickel equivalents, as shown in Figure 6. Based on these diagrams, 17-4 PH stainless steel is typically located in the martensitic region with potential fractions of retained austenite and ferrite depending on the processing conditions. However, the Schaeffler diagram provides only a qualitative indication of the expected phase tendency; LPBF solidification and repeated thermal cycling occur under strongly non-equilibrium conditions [18].

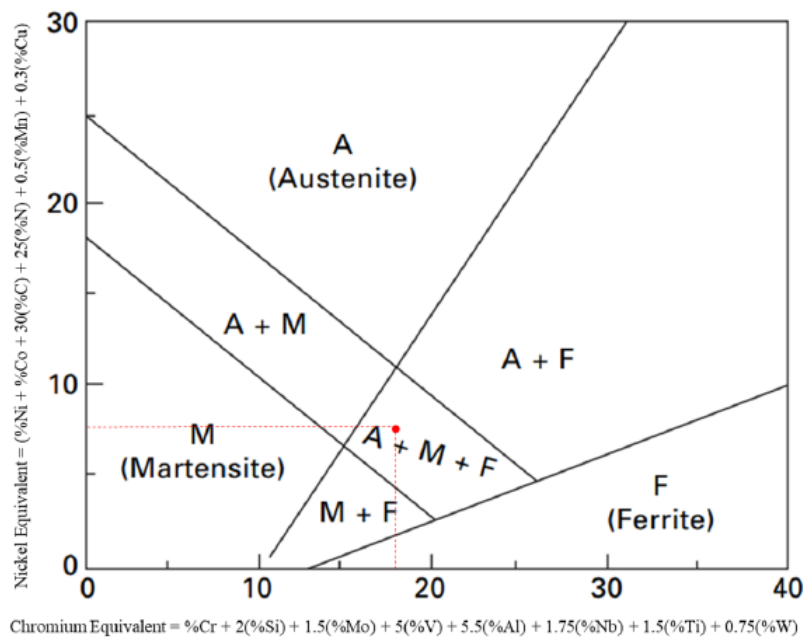


Figure 6. Possible phases of 17-4 PH stainless steel represented by a red dot according to Schaeffler Diagram;[18]

In additive manufacturing processes such as LPBF the rapid solidification rates can influence the microstructure. The high cooling rates can promote the formation of martensite. However, the repeated thermal cycles and localized reheating can lead to the retention of austenite and formation of ferrite, that will result in a non-equilibrium microstructure [18].

The principal precipitation-hardening mechanism in 17-4 PH stainless steels is associated with the formation of nanoscale Cu-rich precipitates within the martensitic matrix during the [13]. At the early stages of aging, fine coherent Cu-rich clusters/precipitates form within the supersaturated martensitic matrix and effectively hinder dislocation motion, resulting in an increase in hardness and mechanical strength. As aging progresses, particularly at higher temperatures or longer holding times, the Cu-rich precipitates undergo structural evolution and coarsening, progressively losing coherency with the matrix. This reduces their effectiveness as obstacles to dislocation motion and can lead to overaging, accompanied by a decrease in hardness and strength. The precipitation sequence reported for Cu-rich particles in martensitic Fe-based alloys involves the evolution from coherent BCC Cu-rich precipitates toward 9R and eventually FCC Cu-rich structures [32].

Due to their high strength and hardness, PH stainless steels are difficult to machine using traditional manufacturing methods. Therefore, additive manufacturing technologies, particularly LPBF, have become an attractive alternative to produce complex components made from these alloys [22].

2.4.2. Chemical Composition of 17-4 PH Stainless Steel

The chemical composition of this material is defined by standards that establish the allowed limits for alloying elements.

Table 3. Chemical Composition according to ASTM A747/A747M;[33]

17-4 PH STAINLESS STEEL: CHEMICAL COMPOSITION										
ELEMENT	C	Mn	P	S	Si	Cr	Ni	Cu	Nb	N
WEIGHT %	≤0.07	≤0.7	≤0.035	≤0.03	≤1.00	15.50-17.70	3.60-4.60	2.50-3.20	0.15-0.35	≤0.05

According to ASTM A747/A747M [33], the material shall conform to the specified chemical composition requirements. The chemical composition requirements are described in section 6 of the standard. For grade CB7Cu-1 (17-4 PH) the chemical composition ranges are present in Table 3.

2.4.3. Applications for 17-4 PH Stainless Steel

Due to the unique combination of high strength, good corrosion resistance and mechanical properties, 17-4PH stainless steel is widely used in a variety of engineering applications [13,34].

Since these alloys can adjust their mechanical properties through heat treatment this allows the material to be used on different service conditions [34]. As a result, 17-4 PH is commonly used in industries where the components are subjected to demanding mechanical and environmental conditions. Typical applications are aerospace industry, such as aircraft structural parts, turbine components and engine elements [18,32]. It is also used in the marine and petrochemical industries, where corrosion resistance and high mechanical strength are essential [5].

In addition, chemical processing equipment, power generations system, injection mold and industrial tooling, due to the high wear resistance and mechanical stability [4]. In Figure 7 is possible to see some examples of applications of 17-4 PH stainless steel.

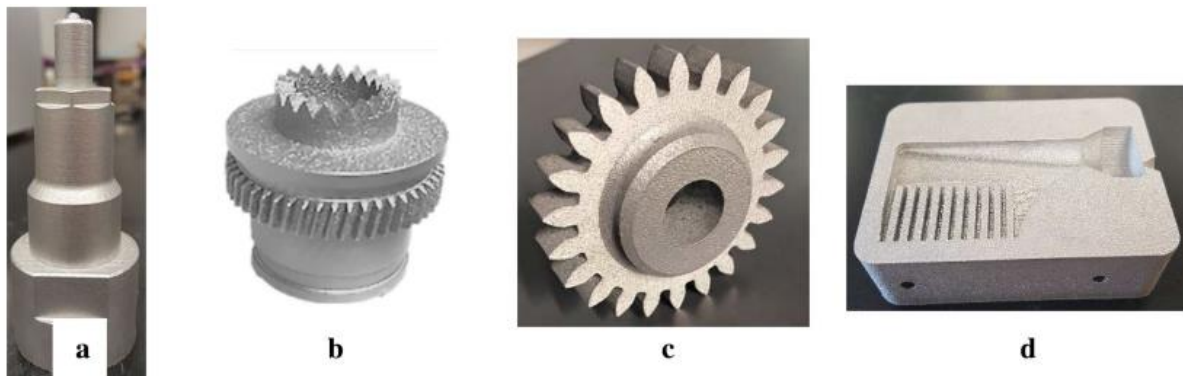


Figure 7. 17-4 PH components produced by LPBF a) fuel injector with conformal cooling channels; b) helicopter engine part; c) automotive engine gear; d) plastic injection mold with cooling channels; [35]

2.5. Post-Heat Treatments

Heat Treatments are important to control the microstructure and mechanical properties of 17-4 PH stainless steel, when is produced by additive manufacturing technologies such as laser powder bed fusion. Due to the rapid melting and solification cycles on LPBF the material often presents microstructural heterogeneities, residual stress and anisotropic properties [22].

Post processing heat treatments are applied in other ways to homogenize the microstructure, reduce the residual stress and improve the mechanical properties of the material. For 17-4 PH the most common post processing treatments are solution annealing followed by aging. In some cases, direct aging treatments can be also applied, depending on the mechanical properties that are needed [20,36].

These heat treatments can promote phase transformation and the precipitation of Cu-rich particles within the martensitic matrix. The formation, size and distribution of these precipitates during aging directly influence hardness, strength and ductility of the alloy [4,36].

2.5.1. Solution Annealing

The first step in the heat treatments of 17-4 PH stainless steel is solution annealing. This treatment will heat the material to a high temperature to dissolve alloying elements into a homogeneous solid solution, followed by a rapid cooling to retain this supersaturated structure [20].

The main objective of this treatment is homogenizing the microstructure and dissolving the precipitates that may have been formed in the previous processes. According to several studies, the temperature for 17-4 PH stainless steel produced by LPBF, for this treatment is around 1040°C with holding times between 0.5 and 1 hour, followed by a rapid cooling such as water quenching or air cooling [4,5,20].

The combination of solution annealing and subsequent aging can recrystallize and homogenize the microstructure of LPBF components, eliminating melt pool boundaries and improving the material properties [22].

2.5.2. Aging Treatment

This treatment is fundamental in the strengthening of 17-4 PH stainless steel, where the supersaturated solid solution obtained after solution annealing is heated until a temperature that can promote the precipitation of strengthening phases within the matrix [20]. Previous studies have shown that direct aging of LPBF 17-4 PH can provide significant strengthening without prior solution treatment. Whether solution treatment is required therefore depends in the as-built microstructure and the target balance of strength, ductility, and dimensional stability.

The aging process leads to the formation of nanoscale Cu precipitates that increase the hardness and strength of the alloy [4,36]. According to several studies the temperature range for 17-4 PH stainless steel is between 480 °C and 620 °C (900 °F to 1150 °F), with holding times varying from a few minutes to several hours, depending on the mechanical properties desired [4,22]. The evolution of microstructure during aging treatment depends on both temperature and time. Lower aging temperatures lead to an underaged condition where insufficient precipitation occurs, while higher temperatures or prolonged holding times can result in over aging, where precipitates get thicker and mechanical strength decreases [36]. Depending on the desired balance between these properties, specific aging conditions are selected, which are designated by the letter “H” followed by the respective aging temperature in Fahrenheit, in accordance with

the standard specifications. For instance, the H900 condition is performed at lower temperatures to promote a fine, dense distribution of coherent Cu-precipitates. This treatment yields the maximum peak strength and hardness, though it results in lower ductility. The H1025 condition acts as an intermediate treatment, offering an optimal balance between mechanical strength, ductility and toughness. Higher temperatures treatments, such as H1150, cause the coarsening of the precipitates, which leads to a decrease in strength that is compensated by a significant improvement in ductility and toughness. Finally, the H1150M condition maximum possible ductility, toughness and extensive stress relief at the expense of overall mechanical strength. A summary of these standard aging treatments, including their typical parameters, microstructure goals and expected trends in mechanical properties are presented in Table 4 [33,36].

Table 4. Standard aging parameters and expected property trends for 17-4 PH stainless steel [33]

STANDARD AGING CONDITIONS FOR 17-4 PH				
CONDITION	TEMPERATURE, °F (°C)	TIME (h)	MICROSTRUCTURAL	EXPECTED PROPERTY
H900	900 (480)	1.5	Fine, dense precipitation of Cu-rich nanoparticles	Maximize strength and hardness; lowest ductility
H1025	1025 (550)	4.0	Moderate, controlled precipitate growth	Balanced compromise between strength and ductility
H1150	1150 (620)	4.0	Coarsening of Cu-rich precipitates	Lower strength; high ductility and toughness
H1150M	1400 (760)	2.0	Deep overaging and extensive stress relief	Minimum strength; maximum ductility and toughness

3. EXPERIMENTAL PROCEDURE

The experimental procedure of this work can be divided into three main stages, as shown in Figure 8. Firstly, the 17-4 PH stainless steel powder was characterized in the as-received condition, focusing on particle size distribution and morphology. The size distribution was evaluated using dynamic light scattering (DLS), while morphology and chemical analysis were performed through optical microscopy (OM), scanning electron microscopy (SEM). Secondly, samples were produced using the laser powder bed fusion (LPBF) process, in which key parameters (laser power, scanning speed, hatch spacing and layer thickness) were defined and controlled, since they influence the final component. Finally, the printed samples were subjected to different solution annealing and aging conditions, followed by microstructural characterization (OM, SEM and EDS) and mechanical testing, including Vickers microhardness and tensile tests.

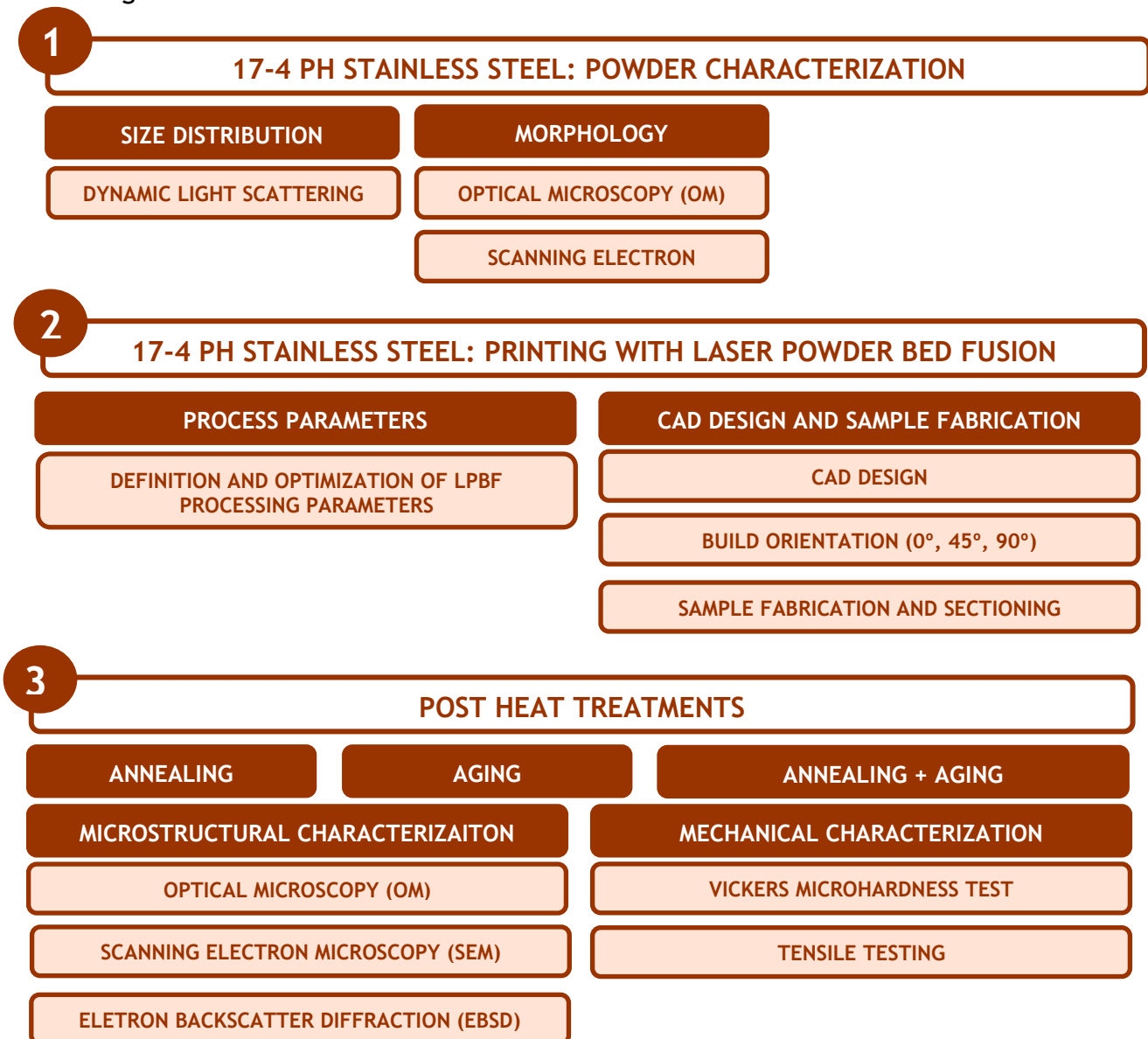


Figure 8. Flowchart summarizing the experimental methodology adopted in this work, including powder characterization, LPBF fabrication of the specimens, post-heat treatments, and microstructural and mechanical characterization.

3.1. Material

Gas atomization 17-4 PH stainless steel powder supplied by Sandvik Osprey® (batch No. 22D0516) was used as feedstock. The powder is manufactured by inert gas atomization, yielding a spherical morphology with a nominal particle size range of 15 - 45 µm, ensuring good flow characteristics and high packing density suitable for Laser Powder Bed Fusion (LPBF) processing. The nominal chemical composition of the alloy is presented in Table 5 [37].

Table 5. Chemical Composition of the powder provided by the supplier; [37]

17-4 PH STAINLESS STEEL: CHEMICAL COMPOSITION											
ELEMENT	Fe	C	Cr	Si	Mn	S	P	Cu	Nb	Mo	Ni
WEIGHT %	Bal.	≤ 0.07	15.5 - 17.5	1.0	≤ 1.0	≤0.04	≤0.03	3.0-5.0	0.15-0.45	≤0.3	3.0-5.0

3.2. Powder Characterization

3.2.1. Powder Morphology

To evaluate the external morphology, size distribution of the as-received 17-4 PH SS powder, scanning electron microscopy (SEM) was performed on loose powder samples. Representative quantities of the powder were directly dispersed onto conductive adhesive carbon tape without prior metallographic mounting to avoid obscuring individual particle features.

3.2.2. Cross-sectional Powder Analysis

To examine the internal features and internal porosity of the powder particles, metallographic cross-sections were prepared. The powder was embedded in a cold mounting resin-hardener system. Following curing, the mounted samples underwent a standard mechanical grinding sequence (320, 400, 600 and 1000 MESH) and polishing sequence, followed by polishing with 6 µm and 1 µm diamond suspensions, and a final polishing with a colloidal silica. The prepared cross-sections were subsequently analyzed using optical microscopy (OM) and scanning electron microscopy (SEM).

3.3. Laser Powder Bed Fusion: Process Parameters

Table 6. Process Parameters used to produce 17-4 PH stainless steel components;

17-4 PH STAINLESS STEEL: PROCESS PARAMETER	
MATERIAL	17-4 PH STAINLESS STEEL
Inert Gas Type	Nitrogen
Platform Heating Temperature (°C)	80
Layer Thickness (µm)	20
Laser Power Inskin (W)	225
Laser Power Border (W)	120
Scanning Speed Inskin (mm/s)	900
Scanning Speed Border (mm/s)	1000
Hatching Distance (µm)	100

The specimens were fabricated using a Print Sharp 150 machine manufactured by Prima Additive. The machine is equipped with a cylindrical build platform with a maximum build size of 160 mm in height and 150 mm in diameter. For this work, a reduced build area of 30 x 60 mm was used, adapted to the size and number of specimens produced. The build preparation and slicing were performed using Materialise software.

The specimens were produced using the selected LPBF parameters summarized in Table 6. These parameters were established in preliminary trials [9,20,32]. The production was conducted under a nitrogen atmosphere to minimize oxidation during processing, and the build platform temperature was maintained at 80 °C. Using the selected LPBF parameters for this study ($P = 225\text{ W}$, $v = 900\text{ mm/s}$, $h = 0.1\text{ mm}$, $t = 0.02\text{ mm}$), the nominal volumetric energy density (E_v) applied to the core (in-skin) region was calculated as 125 J/mm^3 , according to Equation 1. The system utilizes an ytterbium fiber laser with a beam wavelength of 1060 - 1080 nm and an adjustable laser focus diameter (spot size) ranging from 40 to 300 µm.

All the samples were fabricated using a zig zag scanning strategy, as shown in Figure 9. In this strategy, the laser follows a continuous back-and-forth scanning pattern across each layer, with the scanning direction alternating between adjacent tracks. Furthermore, the scanning direction was rotated by 67° between successive layers. The rotation promotes a more uniform distribution of thermal effects throughout the material and may contribute to reducing the preferential alignment of melt tracks and the resulting anisotropy in the printed components.

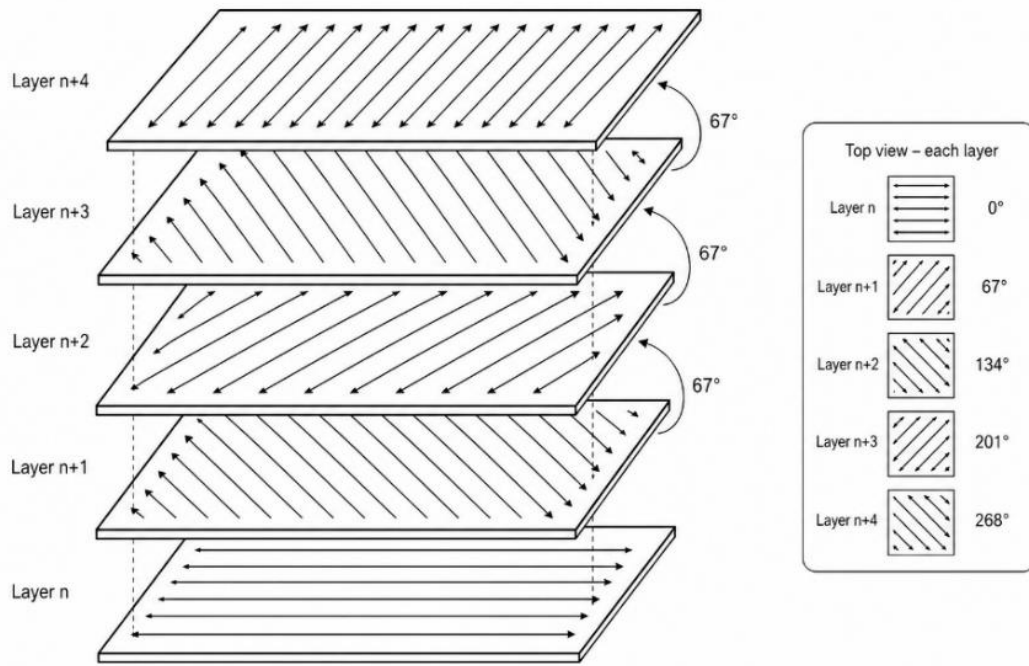


Figure 9. Schematic representation of the zig-zag laser scanning strategy used during the LPBF process, illustrating the rotation of 67° between successive layers. The scanning direction is alternated and progressively rotated from one layer to the next.

3.4. Design and Fabrication of the samples: CAD Model

3.4.1. Characterization samples

This section describes the design and preparation of the specimens prior to the fabrications. The geometries were developed using CAD software (Autodesk Fusion), allowing precise control over the dimensions and configuration of the samples. A single parallelepiped geometry was initially created and subsequently replicated and arranged on the build platform to obtain a total of four specimens. The final geometry was then exported in STL format, as required for the printing stage. After fabrication the samples will be sectioned using an abrasive cutting disc into smaller specimens with approximate dimensions of 1.125 mm, suitable for subsequent characterization.

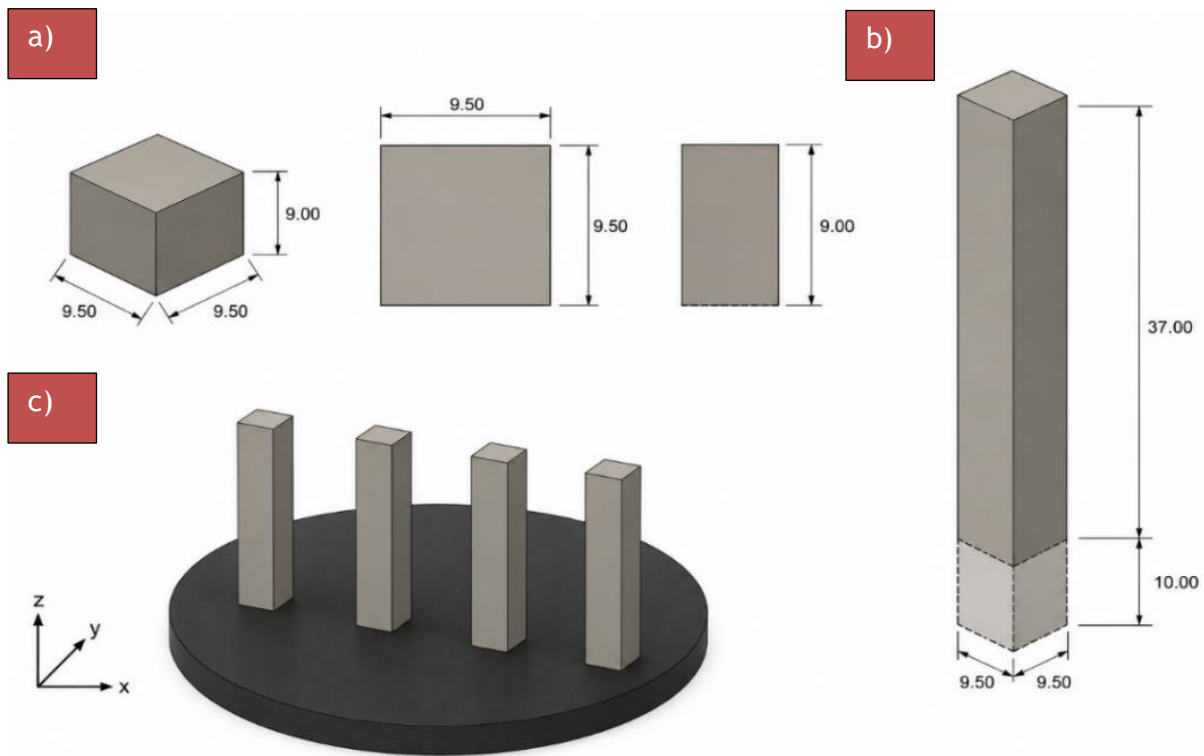


Figure 10. Schematic representation of the specimens manufactured by LPBF: (a) geometry and dimensions of the cubic specimens used for microhardness measurements; (b) geometry and dimensions of the parallelepiped specimens after removal of the lower 10 mm section, resulting in a final height of 37 mm; (c) arrangement of the parallelepiped specimens on the build platform during fabrication.

The design process included the creation of a 2D sketch, also presented in the same figure to show the dimensions of the base edges of the parallelepiped, followed by its extrusion into a 3D model. The main view of the geometry and the 2D sketch is presented in Figure 10. After fabrication, the samples were sectioned using a Struers Accutom-5 abrasive cutting machine. The cutting process was carried out under the following conditions: a rotational speed of 3000 rpm, a feed rate of 0.050 mm/s, medium applied force and continuous water cooling to minimize thermal damage during the cutting.

Following the printing process, a non-functional region of 10 mm was identified as the base of the build. This region corresponds to the initial part of the LPBF process where the thermal conditions and melt pool dynamics are still stabilizing relative to the substrate, and it is also affected by connection to the support structures. Consequently, this portion was excluded from samples extraction, leaving an effective usable height of 37 mm. From this region, four samples of approximately 9 mm were obtained per building direction. In total sixteen individual samples were produced, as illustrated in the corresponding Figure 11, each one with a final dimension of 9.5 mm x 9.5 mm x 9.0 mm.



Figure 11. Representation of the sixteen samples after the cutting step;

3.4.2. Metallographic Preparation and Characterization

After the cutting step these samples were subject to the polishing steps, starting with After the cutting step these samples were subject to the polishing steps, starting with grinding (180,320,400,500,600 and 1000 MESH), followed by the 6 μ m and 1 μ m diamond suspensions to obtain a mirror-like surface suitable for microstructural analysis. The samples selected for EBSD characterization were subject to a final polishing with colloidal silica of 0.06 μ m for 40 min on each surface.

SEM and EDS analysis were performed using a FEI QUANTA 400 FEG (FEI Company, Hillsboro, OR, USA). EBSD analyses were performed using a field-emission scanning electron microscope equipped with an EDAX Pegasus X4M EBSD system (AMETEK BV, Tilburg, The Netherlands). Orientation maps were acquired at an accelerating voltage of 15 kV, with the step size selected according to the magnification used for each analyzed region. The acquired EBSD data were indexed and analyzed using TSL OIM Analysis 5.2 software (EDAX Inc., Mahwah, NJ, USA). A dilation clean-up procedure was subsequently applied, using a grain tolerance angle of 15° and the minimum grain size of 10 points, to remove isolated misindexed points and improve the reliability of the indexed maps.

The polished surfaces were subsequently etched using Kalling's reagent (3 g CuCl₂, 15 g FeCl₃, 50 mL HCl, 25 mL H₂O) in order to reveal the microstructural features of the material [38]. The samples were immersed in the etching solution for approximately 1 second and immediately removed and rinsed in agitated water to prevent over-etching. The etched samples were examined using MO and SEM to evaluate the influence of the different heat treatment on the microstructural evolution.

Vickers microhardness measurements (HV0.5) were subsequently performed on the characterization samples using an applied load of 0.5 Kg (approximately 4.903 N) with a dwell time of 15 seconds. The evaluation was conducted on two distinct planes to assess potential mechanical anisotropy, the lateral surface, which is parallel to the build direction (Z-axis) revealing the layer-by-layer boundaries, and the sectioned top surface, which corresponds to the plane perpendicular to the build direction (X-Y plane) showing the laser scan tracks. On each of these surfaces per specimen, a 4 x 4 indentation matrix was adopted, resulting in 16 measurements per plane. This grid layout ensured that the hardness

values covered the different microstructural deposition regions generated during the printing process (Figure 12). Furthermore, in compliance with standard micro indentation testing guidelines, the distance between the centers of two adjacent indentations was maintained at a minimum of three times the mean diagonal length, and the distance between the center of any indentation and the sample edge was at least 2.5 times the mean diagonal length to ensure the validity of the measurements [39].

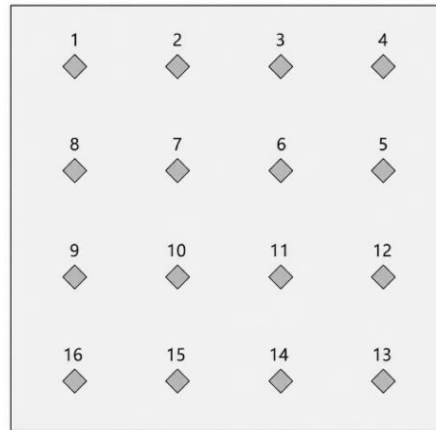


Figure 12. Distribution and numbering of the sixteen Vickers hardness indentations on the top and lateral surface of the samples

3.4.3. Tensile Samples

To investigate the influence of build orientation and post heat treatment on the mechanical behavior of the printed material, tensile samples were designed and manufactured using three different build orientations relative to the building platform (0° , 45° and 90°). The samples dimensions were selected in order to provide a well-defined gauge section and, at the same time, ensure compatibility with the build volume and manufacturing constraints of the additive manufacturing system used. The geometry of the tensile samples and the build orientation are present in Figure 13 [40].

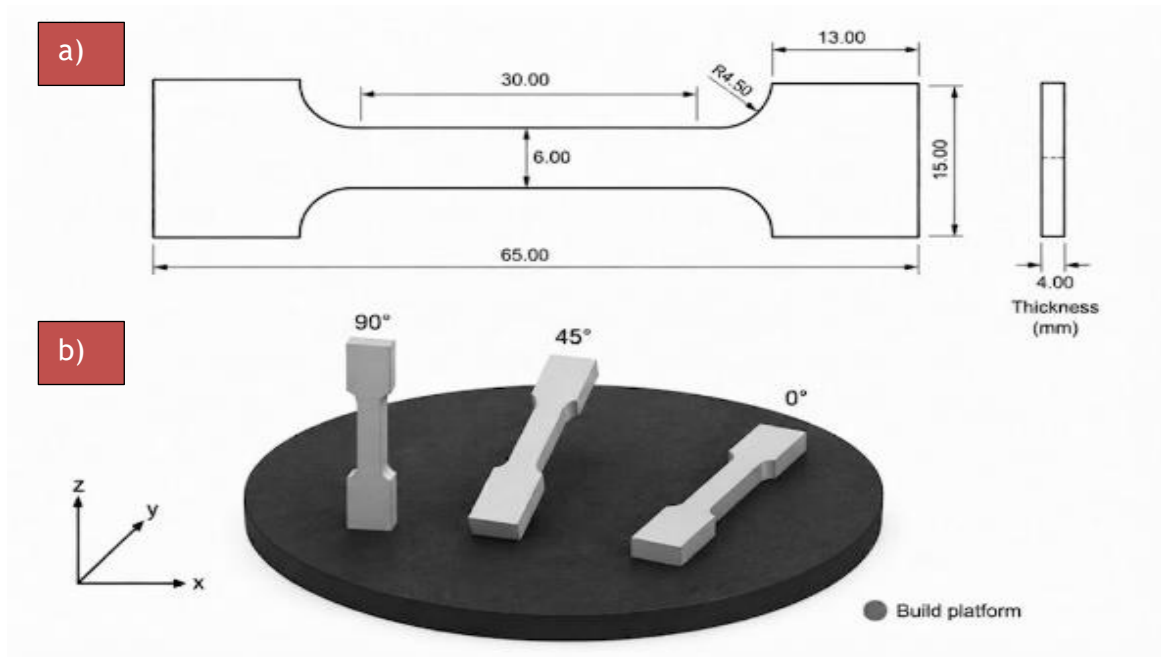


Figure 13. (a) Geometry and dimensions of the tensile samples; (b) Printing orientations (0° , 45° , and 90°) relative to the build platform.

Figure 13a shows the tensile-specimen geometry, with an overall length of 65 mm, a gauge length of 30 mm, a gauge width of 6 mm and a thickness of 4 mm. The Figure 13b shows the three build orientations, horizontal (0°), inclined (45°) and vertical (90°) with respect to the build platform. These orientations were chosen to analyze the influence of the build direction on the mechanical response of the printed material. Different building orientations change the relationship between the loading direction and the layer interfaces, scan-track/melt-pool morphology and crystallographic texture.

Both the tensile and characterization samples were subjected to the heat treatment conditions that are described in Figure 14 and Table 7. This approach enables the evaluation of the influence of the build orientation and heat treatment on the tensile properties, as well as on the hardness characterization.

3.4.4. Fracture Characterization

After tensile testing, the fracture surfaces were characterized by scanning electron microscopy (SEM) to examine the fracture morphology and identify microstructural features, including processing-induced porosity and defects. The analyses were conducted using an accelerating voltage of 15 KV, a working distance of approximately 6.9 mm, and a mixed detector configuration (50 % secondary and 50 % backscattered electrons). To ensure data reliability for quantitative correlation, only valid specimens that fractured within the gauge length and without gripping anomalies were selected to evaluate the relationship between the different build orientations, heat treatment conditions, and the resulting fracture behavior.

Furthermore, Vickers microhardness measurements were performed on one tensile sample from each heat treatment condition and build orientation (0°, 45°, and 90°). Ten indentations were made on each specimen to evaluate the hardness distribution and assess the influence of the different processing conditions.

3.5. Post-Processing Heat Treatments of LPBF 17-4 PH Stainless Steel: Solution Annealing and Aging

After the LPBF fabrication process, both tensile and characterization samples were subjected to post-processing heat treatments to investigate the influence of thermal processing on the microstructure and mechanical performance of 17-4 PH stainless steel.

The heat-treatment conditions were selected using ASTM A747/A747M-16a [33] as a reference and supplemented with experimental high-temperature solution treatments. Four standard aging conditions were selected: H900, H1025, H1150 and an experimental overaging condition at 760 °C for 4 hours (OA760). Among these, the H900 condition was also used by the powder supplier, allowing a direct comparison between the experimental results obtained in this work and industrially established condition. In addition, solution annealing treatments were performed to evaluate the influence of the prior treatment on the subsequent aging response and resulting material properties. The actual furnace set-point temperatures used in the experimental work were 1071°C, 1152°C and 1242°C.

Figure 14 illustrates the individual heat treatment adopted, including the selected temperatures and corresponding holding times for both solution annealing and aging processes.

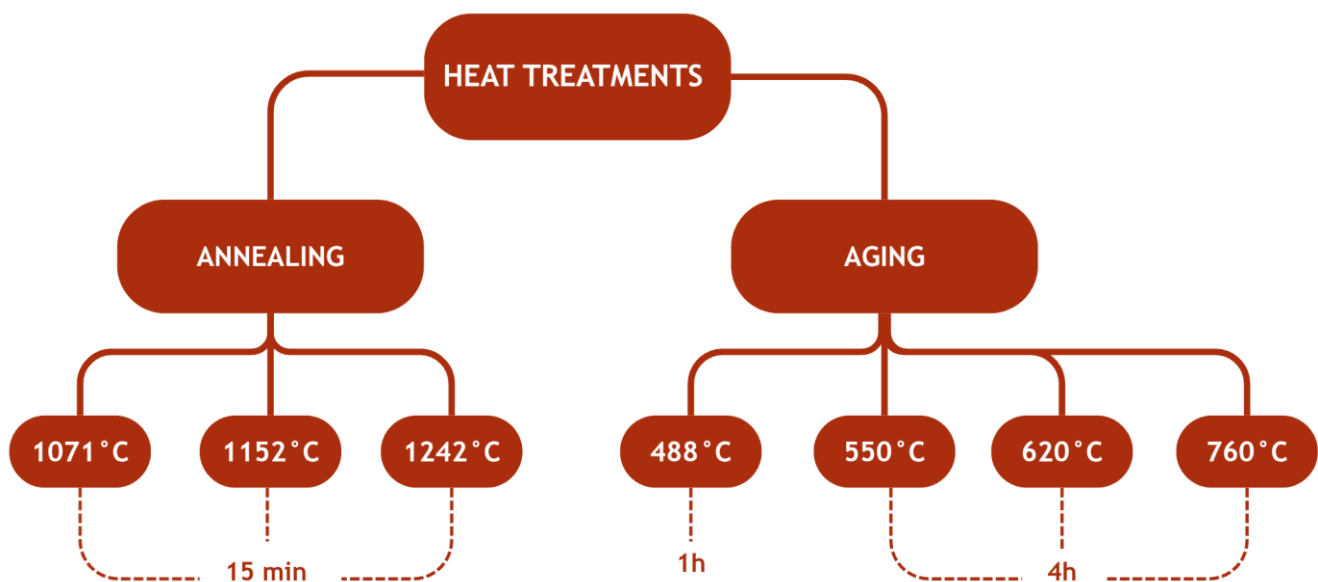


Figure 14. Schematic representation of the heat-treatment conditions, including the annealing and aging processes and their corresponding temperatures and holding times

Besides the evaluation of individual solution annealing and aging treatments, combined heat treatments were also considered in order to assess the influence of solution annealing

prior to aging. In total, fourteen experimental conditions were analyzed, including the reference (as-built) condition that was used for comparison. The table presented below summarizes the combinations of heat treatments applied. The complete set of heat treatment combinations applied to the printing samples is summarized in Table 7.

Table 7. Summary of the heat treatment combinations evaluated for the LPBF manufactured 17-4PH stainless steel samples;

SOLUTION ANNEALING AND AGING PARAMETERS				
	SOLUTION ANNEALING		AGING	
CONDITION	TEMPERATURE (°C)	TIME (h)	TEMPERATURE (°C)	TIME (h)
As-built	-	-	-	-
2	1071	0.25	-	-
3	1152	0.25	-	-
4	1242	0.25	-	-
5	-	-	488	1
6	-	-	550	4
7	-	-	620	4
8	-	-	760	4
9	1071	0.25	488	1
10	1071	0.25	550	4
11	1071	0.25	620	4
12	1071	0.25	760	4
13	1152	0.25	488	1
14	1242	0.25	488	1

The selected heat treatment conditions, summarized in Table 7, were used to provide a comprehensive evaluation of the influence of thermal processing on LPBF samples of 17-4 PH stainless steel. By varying both solution annealing temperatures (1071 °C, 1152 °C, and 1242 °C) and the subsequent aging treatment (ranging from 488 °C to 760 °C), it is possible to investigate the individual and combined effects of these processing steps on the material's microstructural evolution and mechanical behavior.

The heat treatments were carried out in dedicated furnaces, solution annealing treatment were performed without a protective atmosphere, whereas aging were conducted under an argon atmosphere to minimize oxidation. In all thermal cycles, the specimens were placed directly in the middle of the furnace once it had reached the target set-point temperature. Following solution annealing, the samples were rapidly water-quenched to preserve the desired microstructure. For aging conditions, the samples were air-cooled to room temperature. The effectiveness of each heat treatment was subsequently assessed through microstructural characterization, hardness and tensile testing, establishing the

relationship between processing conditions, microstructure and the resulting material properties.

Additionally, for the treatments involving an aging temperature of 760 °C (conditions 8 and 12), the holding time was set to 4 hours as a factor to consider for an overaging condition. Consequently, rather than adopting traditional standard designations such as H1150M, these variants must be referred to as OA760 since they do not share the exact standard parameters. The effectiveness of each heat treatment was subsequently assessed through microstructural characterization, microhardness, and tensile testing, establishing the direct relationship between processing conditions, microstructural features, and the resulting mechanical properties.

4. RESULTS AND DISCUSSION

This chapter presents and discusses the experimental results, following the sequence established in the experimental methodology (Figure 8).

4.1. Powder Characterization

17-4 PH stainless steel powder particles were analyzed in their as-received condition, being characterized in their particle size distribution and particle defects.

4.1.1. Particle Size Distribution

The powder particle size distribution of the powder was determined by Dynamic Light Scattering (DLS), revealing a mean particle size of $34.65\ \mu\text{m}$, with characteristic distribution of $D_{10} = 21.35\ \mu\text{m}$, $D_{50} = 34.65\ \mu\text{m}$ and $D_{90} = 46.84\ \mu\text{m}$, as illustrated in Figure 15. These diameters indicate 10%, 50%, and 90% of the particle volume is composed of smaller particles than the corresponding diameters.

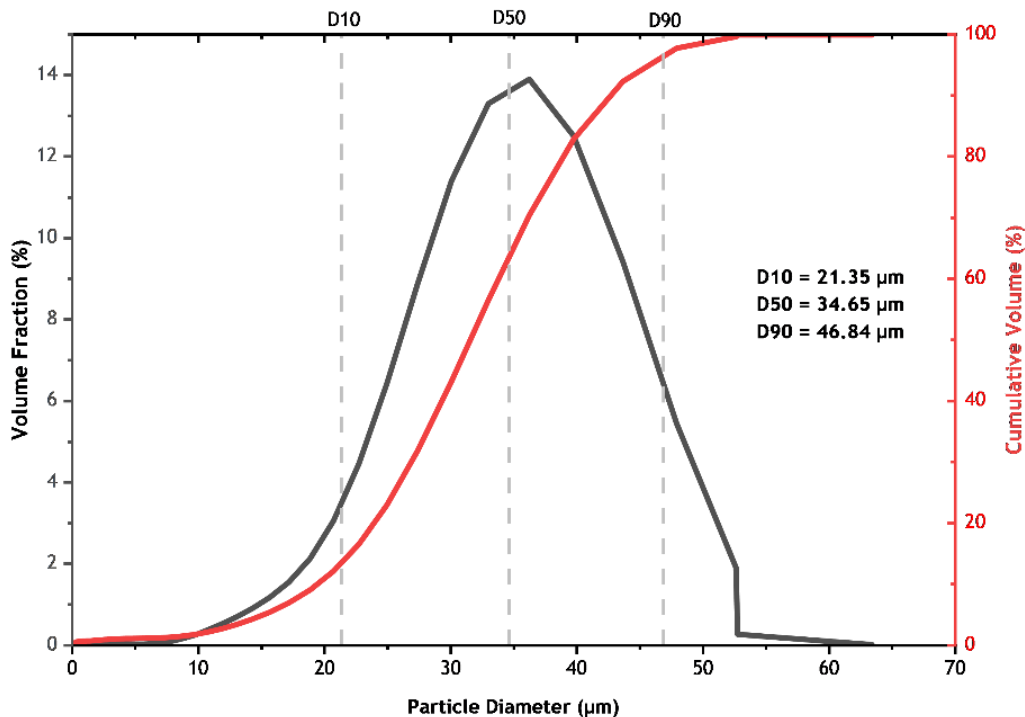


Figure 15. Particle size distribution of 17-4 PH SS powder used in this study;

The particle size distribution shows that there is only a single dominant peak in the center, indicating that most particles sizes are concentrated on a relatively narrow size range. As shown in Figure 15. The cumulative volume curve illustrates a sigmoidal profile, with a fast increase around the D_{50} value, confirming that most powder sizes are between 30 and 40 μm , although particles with different size are also present.

To support these results, complementary observations were used scanning electron microscopy (SEM) and optical microscopy (OM). These observations qualitatively corroborate the presence of particles spanning the size range measured by the particle-size analysis and showed a predominantly spherical morphology, as illustrated in Figure

16. The spherical shape is advantageous for powder processing, because it improves powder flowability and ensures a more homogeneous distribution during subsequent processing.

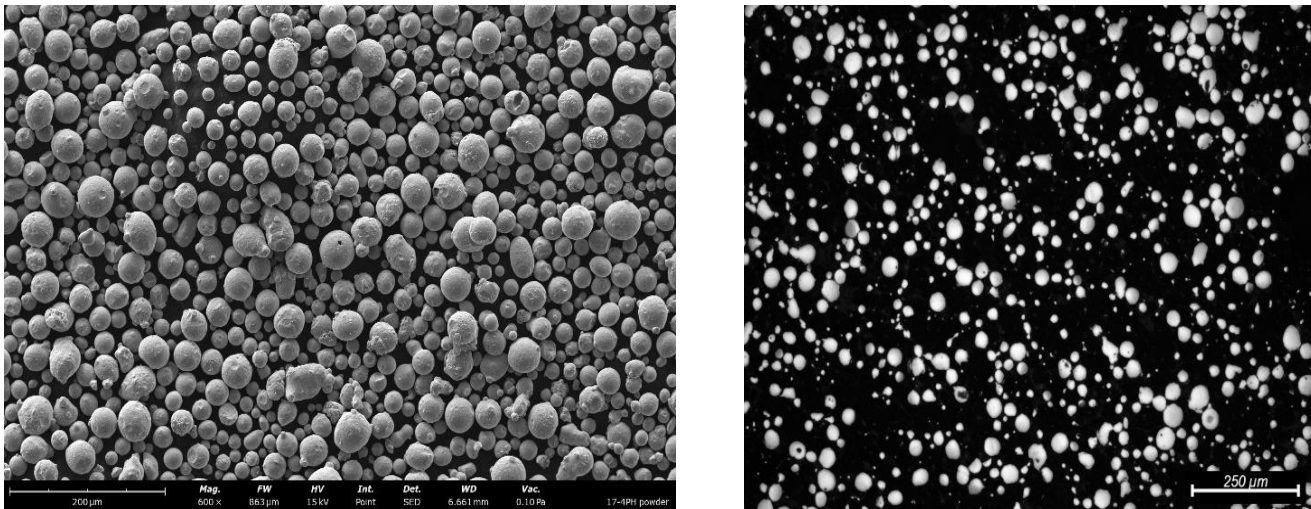


Figure 16. Powder morphology observed by (a) SEM and (b) optical microscopy (OM).

The agreement between the DLS results and SEM and MO observations confirms that the analyzed powder consists predominantly of spherical particles with a moderate particle size distribution centered around 35 μm. These characteristics are generally considered favorable for powder spreading in LPBF; however, flowability and apparent density were not directly measured in the present work.

4.1.2. Morphological Defects of Powder Particles

Furthermore, SEM analysis allowed the observation of several morphology defects in the powder particles. As shown in Figure 17, some particles exhibit satellite particles and minor agglomerations [7,16,22]. These features may reduce powder flowability and negatively affect the quality of the powder bed, leading to a non-uniform powder spreading during the LPBF process and potentially increasing of lack of fusion defects.

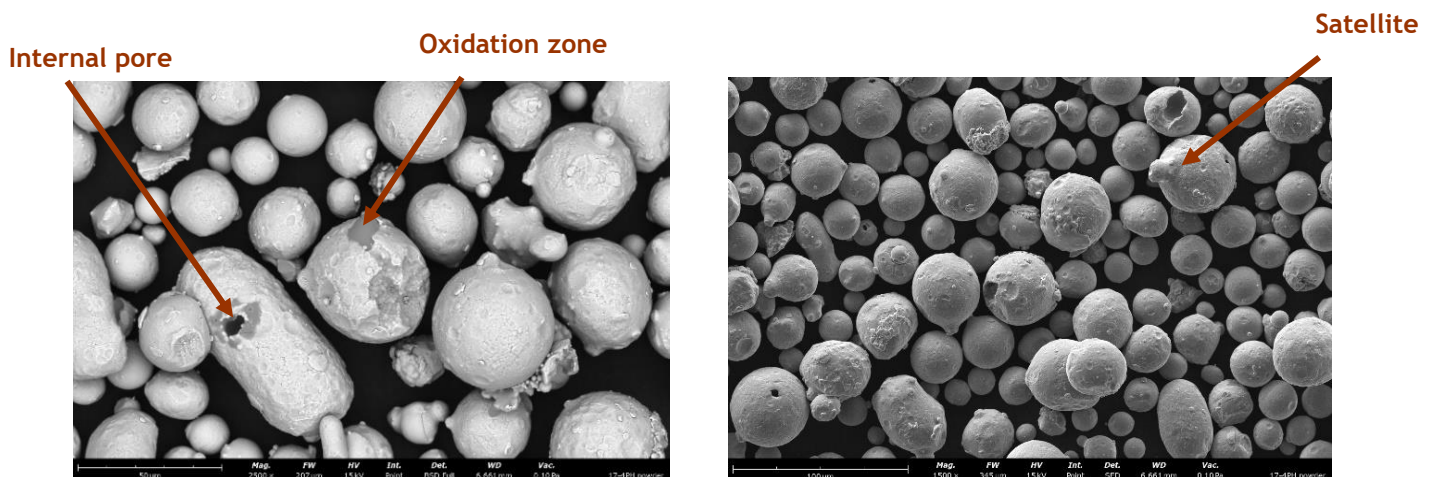


Figure 17. Representative SEM images of powder particle defects: (a) internal pore and oxidation zone; (b) satellite particle

In addition, internal porosity was observed in some particles, as illustrated in Figure 17a). This feature is associated with the rapid solidification and gas entrapment mechanisms inherent to the atomization process [23]. Gas pores present in the feedstock may contribute to residual porosity in the consolidated material if gas entrapment is not fully eliminated during melting and solidification.

Additionally, localized features were observed in the analyzed areas. To investigate their chemical nature, particularly the possible presence of oxide-rich regions, energy-dispersive X-ray spectroscopy (EDS) was performed on selected areas using a Phenom scanning electron microscope. The analysis was conducted in point mode at an accelerating voltage of 15 kV, with a field width (FW) of 207 μm and a working distance (WD) of 6.7 mm, using a full backscattered electron (BSD Full) detector. As shown in Figure 18, the EDS spectra identified the characteristic peaks of the main alloying elements of 17-4 PH stainless steel, namely iron (Fe), chromium (Cr), nickel (Ni), and copper (Cu). In addition, oxygen (O), silicon (Si), phosphorus (P), manganese (Mn), and niobium (Nb) were detected. The quantified composition showed a high oxygen content of 43.56 wt.%, together with 18.08 wt.% Si, 12.59 wt.% Mn, 1.40 wt.% P, and 1.80 wt.% Nb. The high oxygen content indicates an oxygen-rich region and is consistent with the presence of oxide-containing features in the analyzed area. The detection of Si, Mn, P, and Nb at concentrations above the nominal composition specified by the powder supplier may be associated with the localized nature of the analyzed feature rather than representing the overall chemical composition of the 17-4 PH stainless steel. Therefore, the EDS results support the identification of the analyzed feature as an oxide-rich or chemically heterogeneous region.

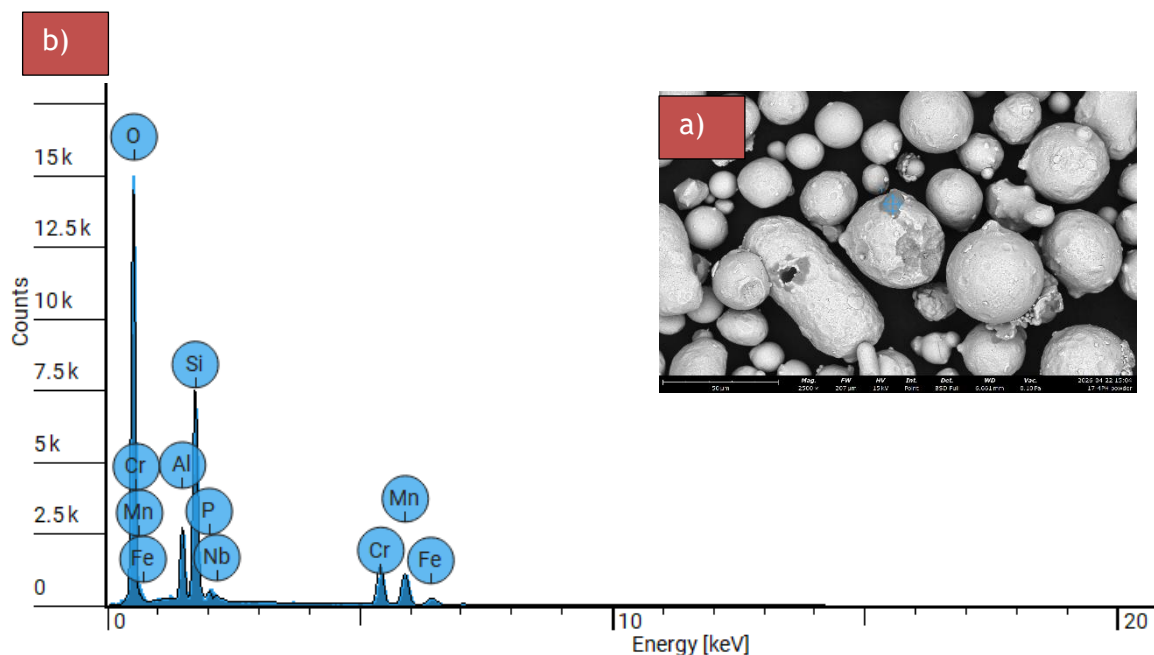


Figure 18. Energy Dispersive X-ray Spectroscopy (EDS) analysis of the powder 17-4 PH stainless steel sample: (a) SEM micrograph showing the selected region of analysis, and (b) corresponding EDS spectrum confirming the presence of the main alloying elements (Fe, Cr, Ni, Cu) alongside a oxide region.

4.2. Microhardness

The microhardness tests were performed using a Vickers load of 0.5 KgF (≈ 4.903 N), reported as HV0.5, with a dwell time of 15 seconds, following the ISO 6507-1 [39]. As previously described, the measurements were carried out on both the cutting surface and the lateral surface of the samples to guarantee that all regions of interest were adequately characterized. A total of sixteen indentations were performed on each surface, in a matrix 4 x 4 matrix.

Figure 19 illustrates the average microhardness values that are calculated from the mean values obtained from the cutting and lateral surfaces, together with their corresponding standard deviations, providing an indication of the variability of the measurements. The individual microhardness values obtained for each analyzed surface are presented in Table 8. As no significant differences were observed between the lateral and sectioned surfaces within the experimental dispersion, the results were combined to provide a clearer overall comparison between the different heat treatment conditions and build orientations.

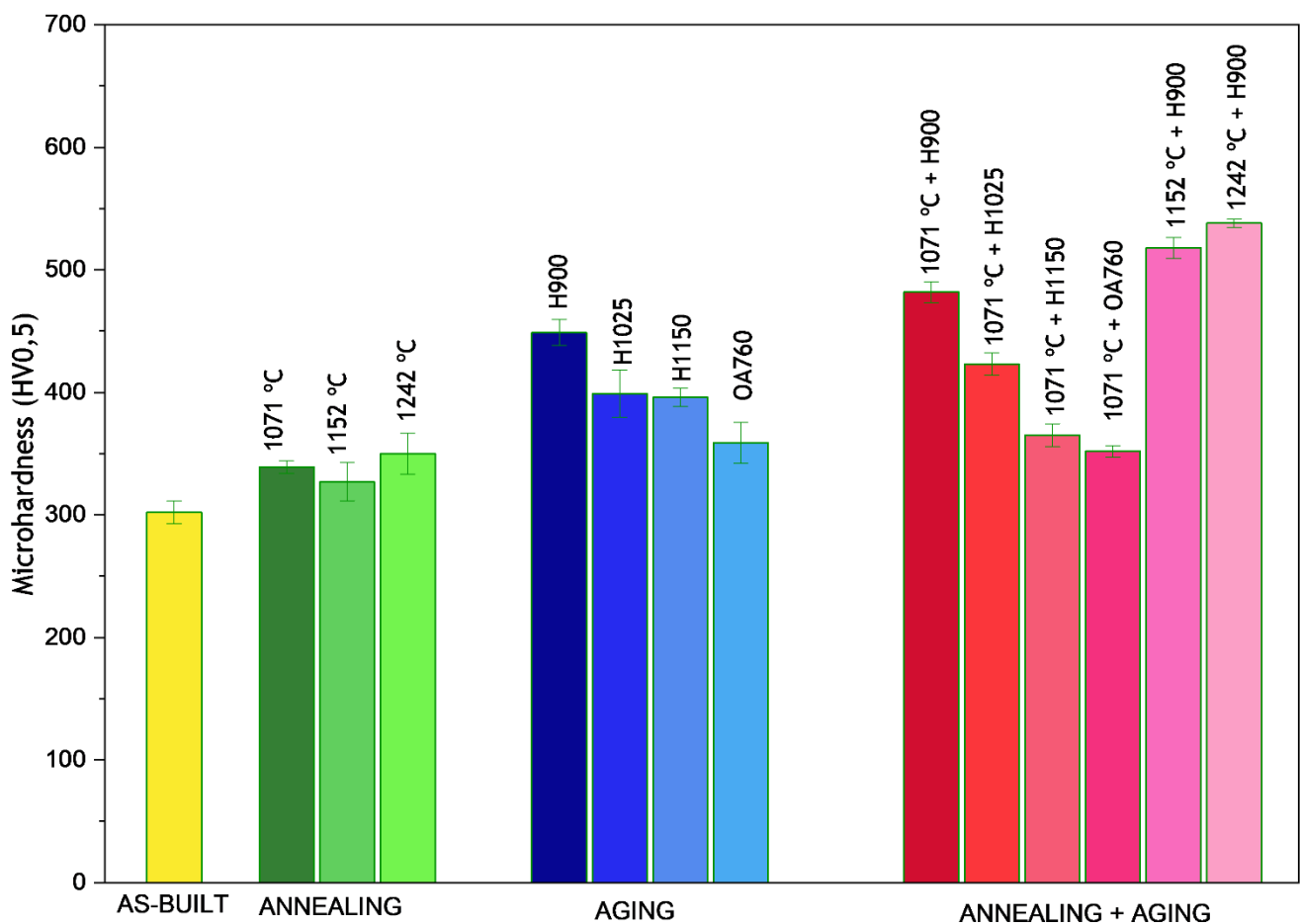


Figure 19. Average Vickers microhardness (HV0.5) values obtained from the cutting and lateral surfaces of the samples, together with the corresponding standard deviations

Regarding the samples that were exclusively subjected to solution annealing, a general increase in microhardness was observed compared with the as-built condition ($302 \pm 9\text{HV}$), regardless of the temperature of the treatment. For the solution annealing at $1071\text{ }^{\circ}\text{C}$ the hardness increased to $339 \pm 5\text{ HV}$. A slightly lower value of $327 \pm 16\text{ HV}$ was obtained at $1152\text{ }^{\circ}\text{C}$, followed by an increase to $350 \pm 17\text{ HV}$ for the samples treated at $1242\text{ }^{\circ}\text{C}$. However, considering the overlap between the standard deviations, particularly for the two highest temperatures, no clear relationship between the treatment temperature and hardness can be established. These results indicate that within the temperature range used the influence of solution annealing on hardness is limited, although all annealed conditions exhibit higher hardness than the as-built material.

Table 8. Microhardness values (HV0.5) measured at the side and cut regions across the different tested conditions.

MICROHARDNESS VALUES FROM THE SIDE REGION AND CUT REGION FOR EACH CONDITION				
	CONDITION	SIDE REGION (HV0.5)	CUT REGION (HV0.5)	AVERAGE VALUES (HV0.5)
SOLUTION ANNEALING	As-built	303 ± 15	300 ± 10	302 ± 9
	$1071\text{ }^{\circ}\text{C}$	329 ± 8	350 ± 5	339 ± 5
	$1152\text{ }^{\circ}\text{C}$	317 ± 35	336 ± 9	327 ± 16
	$1142\text{ }^{\circ}\text{C}$	344 ± 32	356 ± 8	350 ± 17
AGING	H900	458 ± 15	441 ± 13	449 ± 11
	H1025	415 ± 36	383 ± 16	399 ± 19
	H1150	378 ± 13	414 ± 7	396 ± 8
	OA760	348 ± 17	370 ± 5	359 ± 9
COMBINED TREATMENT	$1071\text{ }^{\circ}\text{C} + \text{H900}$	483 ± 9	481 ± 10	488 ± 8
	$1071\text{ }^{\circ}\text{C} + \text{H1025}$	424 ± 6	422 ± 15	423 ± 9
	$1071\text{ }^{\circ}\text{C} + \text{H1150}$	364 ± 19	366 ± 4	365 ± 9
	$1071\text{ }^{\circ}\text{C} + \text{OA760}$	356 ± 6	349 ± 10	352 ± 5
	$1152\text{ }^{\circ}\text{C} + \text{H900}$	517 ± 17	519 ± 5	518 ± 9
	$1242\text{ }^{\circ}\text{C} + \text{H900}$	534 ± 5	541 ± 7	538 ± 3

For the directly aged samples, hardness decreased from $449 \pm 11\text{ HV}$ in the H900 condition to $399 \pm 19\text{ HV}$ and $396 \pm 8\text{ HV}$ in the H1025 and H1150 conditions, respectively. The OA760 condition resulted in the lowest hardness value ($359 \pm 9\text{ HV}$). However, OA760 should not be interpreted simply as a linear extension of the aging-temperature trend, since it involves its specific thermal sequence. The lower hardness obtained after OA760 is consistent with a more advanced precipitation state and precipitate coarsening, which reduce the effectiveness of Cu-rich precipitates as obstacles to dislocation motion and are characteristic of an overaged microstructure.

To evaluate the effect of the combination of solution annealing and aging the results were analyzed using two complementary approaches. First, the solution treatment temperature was fixed at 1071 °C, while different aging conditions were applied. Under these conditions the microhardness followed the same overall trend observed for the direct aging treatment, decreasing progressively as the aging treatment become more severe. The hardness decreased from 488 ± 8 HV for 1071°C + H900 to 423 ± 9 HV for 1071 °C + H1025, followed by 365 ± 9 HV for 1071 °C + H1150, reaching the lowest value of 352 ± 5 HV for 1071 °C + OA760. In second approach, the aging condition was maintained at H900, while the solution annealing treatment temperature was varied to evaluate the influence of the previous thermal treatment. In contrast to the previous analysis an increasing trend in hardness was observed. The hardness increased from 488 ± 8 HV for 1071 °C + H900 to 518 ± 9 HV for 1152 °C + H900, reaching the highest value measured in this work of 538 ± 3 HV for the 1242 °C + H900 condition.

Overall, the combined heat treatments produced higher hardness values than either solution annealing or direct aging. Furthermore, when the aging condition was constant at H900, increasing the solution annealing treatment temperature resulted in progressively higher hardness values. Conversely, when the solution annealing treatment temperature was maintained at 1071 °C, increasing the aging temperature led to a progressive reduction in hardness. These values demonstrate that both processing stages influence the final hardness. However, direct aging treatment alone was also capable of producing substantial increase in hardness compared with the as-built condition. Although the highest hardness values were achieved after the combined solution annealing and aging treatment, these findings suggest that a prior solution annealing treatment may not always be necessary when a significant increase in hardness is the primary objective. In these cases, direct aging may represent the simplest and potentially more efficient heat treatment route.

In summary, the microhardness results demonstrate that different heat treatments produced different mechanical response, with the highest hardness achieved after the combined solution treatment and aging condition. Therefore, a detailed microstructural characterization was performed using SEM and EBSD to investigate the morphological evolution and phase constitution associated with each heat treatment condition.

4.3. Microstructural characterization

4.3.1. SEM Analysis

For the morphological characterization via Scanning Electron Microscopy (SEM), a representative sample from each of the four primary processing conditions was selected: the as-built state, the solution annealing condition at 1071 °C, the direct-aging condition with highest measured hardness (H900), and representative combined heat-treatment conditions (such as 1071 °C + H900). These conditions were selected as they represent the key reference states and standard baselines for this material.

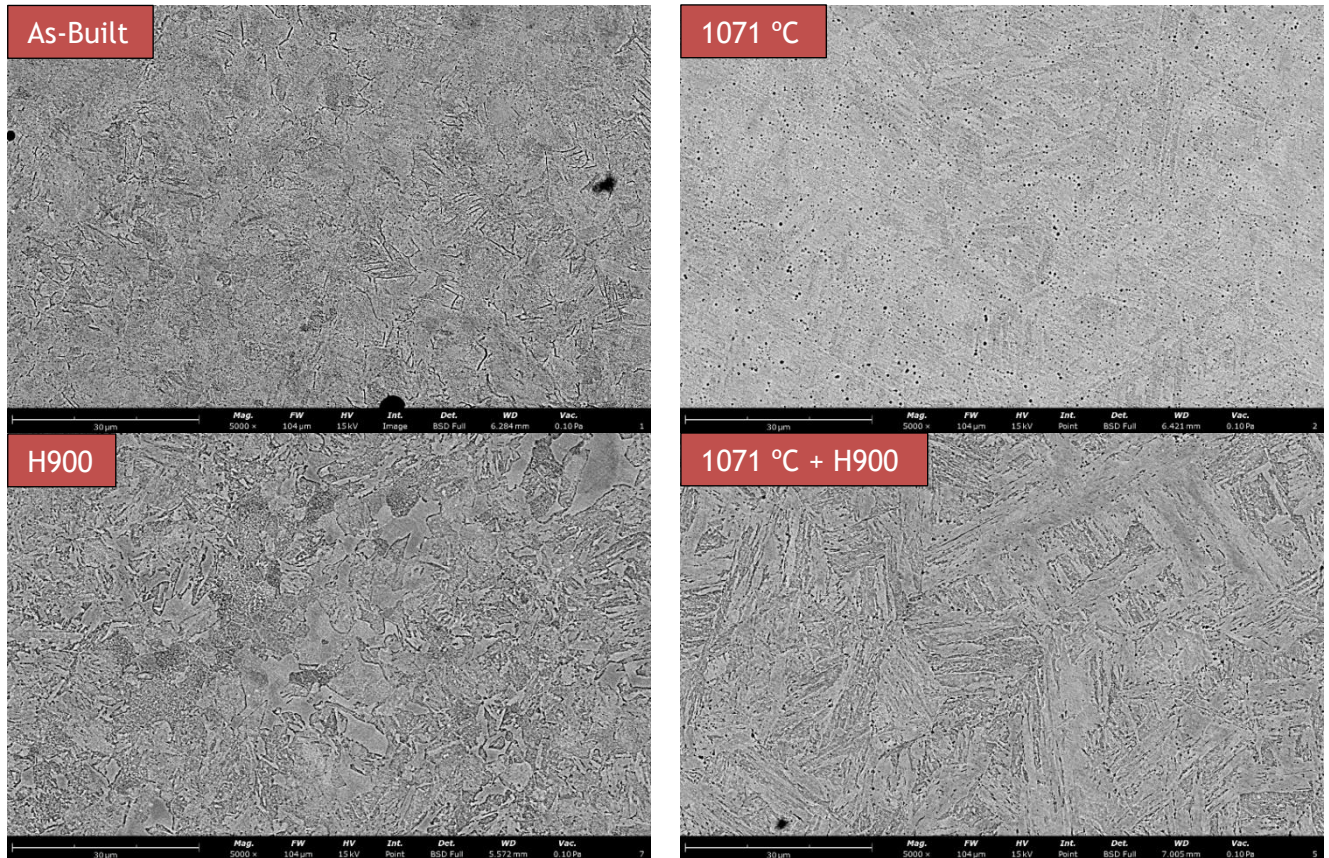


Figure 20. SEM images of the alloy under different processing conditions: as-built state, annealing condition (1071 °C), aging condition (H900), and combined heat treatment (1071 °C + H900), illustrating the microstructural evolution induced by post heat treatment.

The as-built microstructure of LPBF 17-4 PH SS is characterized by a highly refined and heterogeneous morphology resulting of the rapid solidification and the severe thermal cycles inherent to this manufacturing process. SEM revealed a fine lath-like morphology, consistent with the martensitic microstructure reported for LPBF 17-4 PH and subsequently assessed by EBSD [16,41]. Furthermore, Moyle et al. [12] described the hierarchical microstructure produced during LPBF process, consisting of a refined martensitic matrix and melt pool boundaries that are a result of a localized rapid solidification.

Compared with the as-built, the microstructure after the solution annealing treatment 1071 °C exhibits a substantially more homogeneous appearance, indicating the effective thermal relaxation of the features resulting from LPBF solidification. The melt-pool/cellular contrast became substantially less discernible after solution treatment, indicating microstructural homogenization. A reduction in microsegregation is consistent with the literature but not quantified directly here [12, 41, 42].

The application of a direct aging at 488 °C for 1 h (H900 condition) without a prior solution annealing treatment results in a modification of the as-built microstructure. Although the material retains the characteristics solidification features of the LPBF process, the matrix exhibits a noticeably different contrast in the SEM image. Sadhukhan et al. [16] and Salaheldin et al. [42] reported that the H900 treatment promotes the precipitation of Cu-rich nanoprecipitates within the martensitic matrix. Owing to their nanometric dimensions (typically 5 to 40 nm), these precipitates cannot be seen by SEM. Nevertheless, they recognized them as the primary strengthening mechanism associated with the H900 condition.

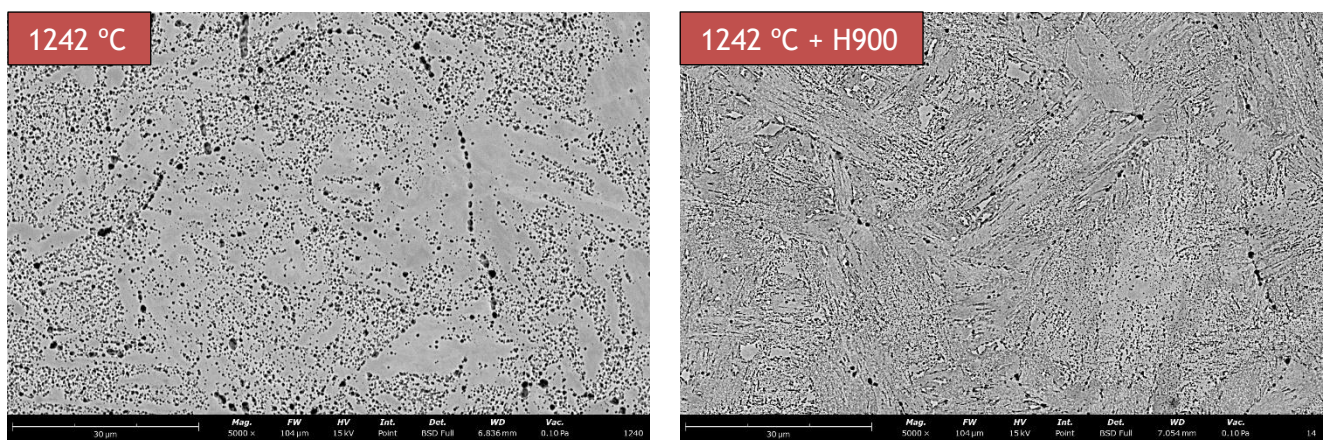


Figure 21. SEM images of the LPBF 17-4 PH stainless steel after (a) annealing at 1242 °C and (b) annealing at 1242 °C followed by H900 aging (1242 °C + H900), illustrating the microstructural evolution induced by the combined heat treatment.

Following the solution annealing at 1242 °C, the microstructure showed a homogeneous appearance, similar to that observed after solution annealing at 1071 °C. Melt-pool boundaries were no longer discernible at the SEM scale used, and the microstructure appeared coarser than after the lower-temperature treatment [12,41]. Although this apparent coarsening is consistent with the higher thermal energy promoting grain growth, a detailed quantification should be supported by EBSD or grain-size analysis.

After the subsequent H900 aging treatment (1242 °C + H900), the overall microstructure remains essentially unchanged, indicating that aging does not modify the morphology previously established during the solution annealing. Nevertheless, the martensitic lath structure becomes more clearly distinguishable, producing a higher contrast throughout the matrix. The contrast change is consistent with an aging-induced microstructural response; Cu-rich precipitation is established strengthening mechanism for this condition but not directly resolved by SEM. Consequently, although the 1242 °C and 1242 °C + H900 conditions exhibit similar overall morphologies, their mechanical behavior is expected to differ due to the precipitation phenomena occurring during aging rather than to significant changes in the observable microstructure.

Overall, the SEM observations can provide qualitative evidence of the microstructural evolution induced by the different heat treatments, demonstrating the progressive homogenization of the LPBF microstructure after solution annealing and the increased definition of the martensitic lath structure following aging. However, SEM alone cannot provide quantitative information regarding the phase constitution or crystallographic characteristics of the material. Therefore, EBSD analysis was performed to determine the martensite and retained austenite fraction, as well as to provide a more detailed characterization of the microstructure associated with each heat treatment condition.

4.3.2. EBSD Analysis

EBSD analysis was performed to complement the SEM observations by providing quantitative information of the microstructure of 17-4 PH SS produced by LPBF, as shown in Figure 22. The EBSD measurements were performed using a FEI Quanta 400 FEG ESEM scanning electron microscope equipped with an EDAX Pegasus X4M EBSD system.

Data acquisition was conducted using an accelerating voltage of 15 KV, a spot size of 5.5, a diaphragm opening of 3, and a working distance ranging between 8.7 mm and 11.5 mm depending on the analyzed specimen. The patterns were collected with an exposure of 0.05 s, a gain of approximately 8.85, and a binning of 4 x 4 (336 x 252 pixels). A hexagonal grid with a step size of 0.2 μm was employed over map areas of 38 x 38 μm acquired at a magnification of x5000.

The acquired EBSD data were indexed and analyzed using TSL OIM Analysis 5.2 software (EDAX Inc., Mahwah, NJ, USA). The phase identification and quantification were performed using a phase file corresponding to Austenite and Martensite. Post processing data clean-up and grain boundary reconstructions were subsequently applied to ensure reliable indexing confidence level before calculating phase fractions (such as reverted austenite quantification). This information is necessary to understand the microstructural evolution induced by the different heat treatments and to establish a direct correlation between the phase constitution and mechanical response of the material.

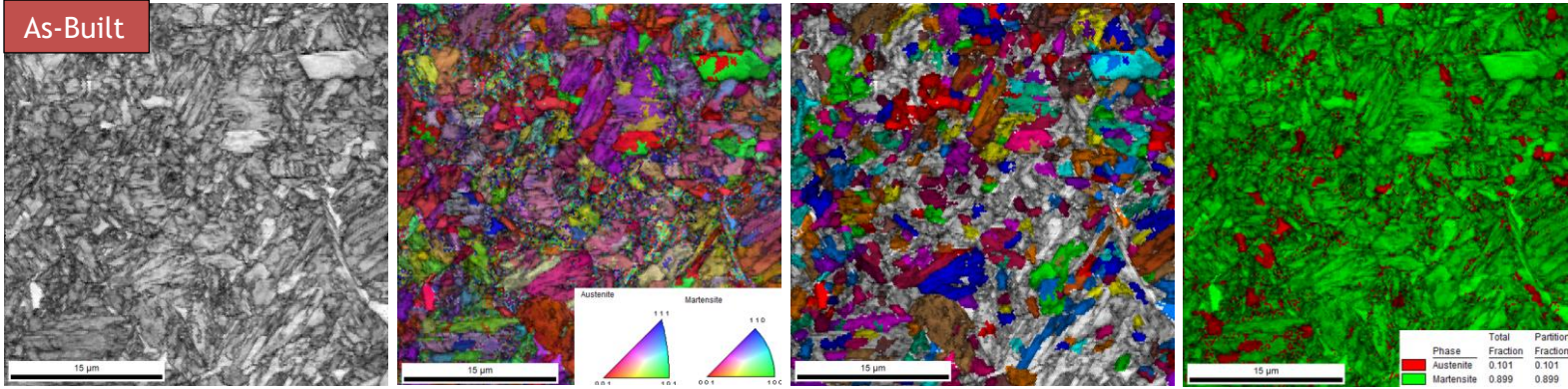
IQ Map

IQ + IPF Map

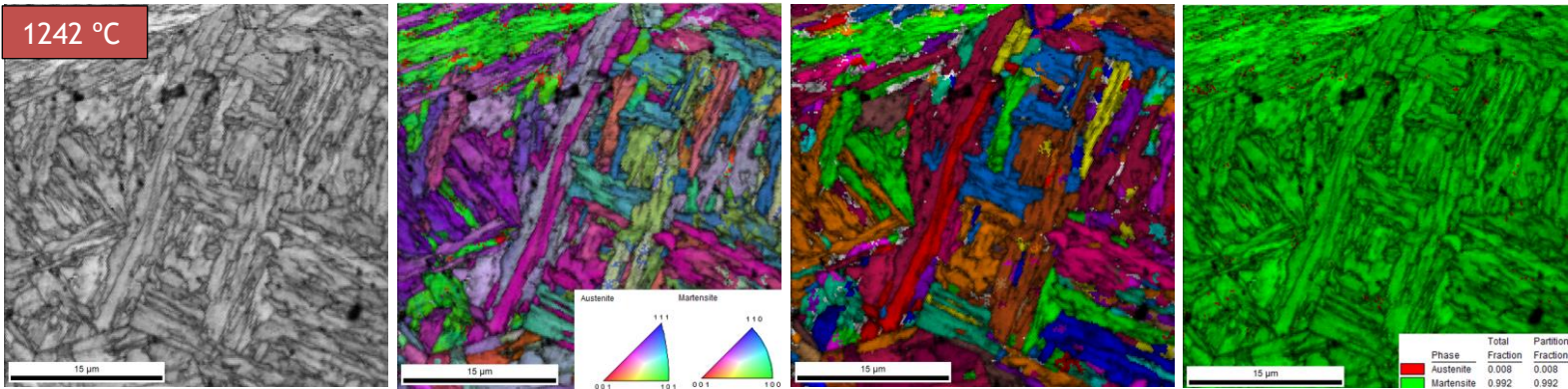
IQ + Grain Map

IQ + Phase Map

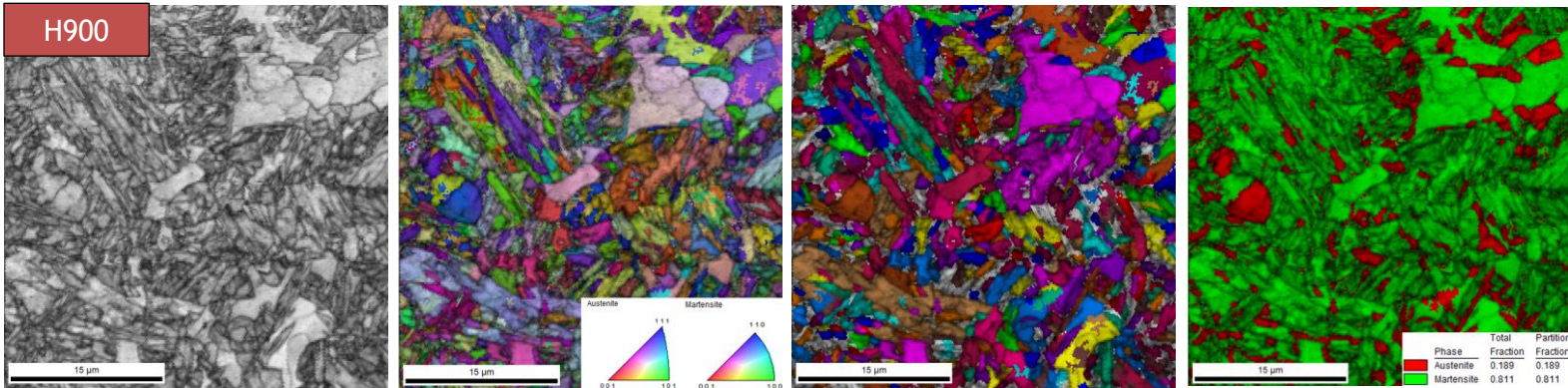
As-Built



1242 °C



H900



1242 °C +H900

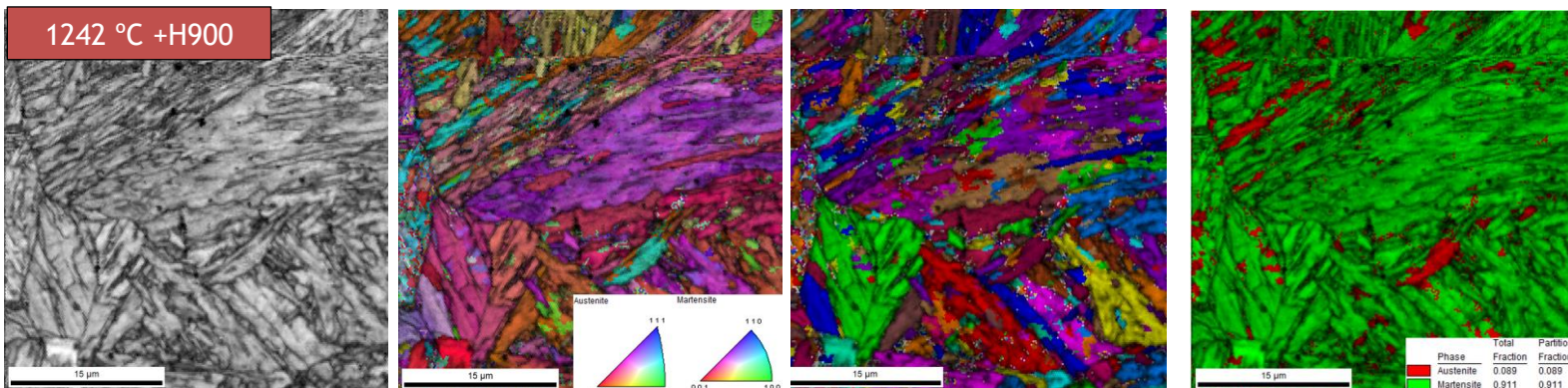


Figure 22. EBSD maps illustrate the microstructural evolution of LPBF-fabricated 17-4 PH stainless steel after different heat treatment conditions. From left to right, the columns present the Image Quality (IQ), Inverse Pole Figure (IPF), grain boundary and phase maps. From top to bottom, the rows correspond to the as-built, solution-treated at 1242 °C, direct-aged (H900), and solution-treated at 1242 °C + H900 conditions.

Based on the microhardness results four conditions were selected for EBSD analysis. The H900 condition was chosen as the representative aging treatment since it exhibited the highest hardness among all the aging conditions. The combined 1242 °C + H900 condition, which produced the highest hardness measured in this work, was selected to evaluate the corresponding microstructure. To assess the individual contribution of the solution annealing on the microstructure, the corresponding solution annealing condition at 1242 °C was also included in the analysis. The as-built condition was selected as the reference state for comparison. This selection enables a systematic evaluation of the individual and combined effects of solution annealing and aging treatments on the microstructural evolution of the 17-4 PH SS produced by LPBF.

The EBSD characterization of the four conditions reveals a significant change in phase distribution and microstructural morphology, that demonstrate the strong influence of the heat treatment on the evolution of 17-4 PH SS produced by LPBF. Representative Image Quality (IQ), Inverse Pole Figure (IPF), grain boundary and phase maps are presented in Figure 22.

In the as-built condition, the phase map contained 89.9 % BCC phase, interpreted as predominantly martensitic, and 10.1 % austenite (FCC). The austenite was mainly observed as thin films and elongated regions distributed between martensitic structures, while the IQ maps exhibited heterogeneous patterns quality, which may be influenced by local lattice distortion and the fine heterogeneous as-built microstructure. The IPF maps further revealed the characteristic fine lath martensitic morphology inherited from rapid solidification. These observations are consistent with the SEM images presented in the previous section, which revealed the fine lath martensitic morphology together with the characteristic LPBF cellular microstructure were also identified. The rapid solidification associated with LPBF promotes cellular solidification and micro segregation of elements that stabilized the austenite, in particular the Ni and Cu, within interdendritic regions [43,44]. The local enrichment of these elements decreases the martensite start temperature (M_s), allowing part of the austenite to remain stable after cooling at room temperature. Consequently, the retained austenite observed is associated to heterogeneity generated during the LPBF process [38,39].

After solution annealing treatment at 1242 °C, a substantial modification of the microstructure was observed. The phase fraction of retained austenite decreased from 10.1 % to 0.8 %, resulting in an almost fully martensitic microstructure (99.2 % BCC). Simultaneously, the IPF maps showed the development of coarser martensitic laths, while the IQ maps exhibited a more homogeneous contrast, suggesting a reduction in lattice distortion compared with the as-built condition. These observations are consistent with the SEM images presented, which showed the disappearance of the characteristic LPBF melt pool and cellular solidification features together with a more homogeneous microstructure after solution annealing. While SEM qualitatively revealed the homogenization of the microstructure, EBSD confirms that this evolution is accompanied by an almost complete martensitic transformation and the development of coarser martensitic laths. These changes indicate that the solution annealing removed the

heterogeneity generated during the LPBF process. At this temperature the diffusion becomes sufficiently extensive to homogenize the alloying elements throughout the matrix, dissolving the cellular segregation that were formed during the solidification [11,44]. Consequently, homogenization can reduce local compositional gradients that stabilize austenite and depress M_s , thereby favoring a more complete martensitic transformation during cooling. Similar reductions in retained austenite were reported by Haines et al. [11] at high temperatures, who demonstrated that homogenization suppresses the chemical gradients responsible for austenite stabilization.

The direct H900 aging condition exhibited 81.1% BCC phase and 18.9% FCC phase. Therefore, this condition presented the lowest BCC fraction and the highest FCC fraction among the four conditions analyzed. Compared with the as-built condition, the FCC fraction increased from 10.1% to 18.9%. The phase map showed that the FCC phase remained distributed within the predominantly BCC matrix. The IQ maps retained a heterogeneous appearance, while the IPF maps continued to reveal the fine morphology associated with the LPBF microstructure. Compared with the solution-annealed condition, the microstructure therefore remained more heterogeneous after direct aging. This is consistent with the SEM observations, where features associated with the original LPBF microstructure remained identifiable. The H900 treatment promotes precipitation hardening of the material; however, the EBSD results indicate that the phase distribution was also affected by the thermal history of the as-built material [43,44].

Following the combined 1242 °C + H900 treatment, the EBSD phase map indicated 91.1% BCC phase and 8.9% FCC phase. The BCC fraction was therefore higher than that measured in the direct H900 condition, while the FCC fraction was considerably lower. Compared with the solution-annealed condition, however, the FCC fraction increased from 0.8% to 8.9% after the subsequent H900 aging treatment. The FCC phase was mainly observed as thin and discontinuous regions within the BCC matrix. The IPF and IQ maps also showed a microstructure different from that of the as-built condition, reflecting the effect of the preceding solution annealing treatment. These results indicate that the microstructural condition established during solution annealing influenced the subsequent response to H900 aging.

The comparison between the two aged conditions is particularly relevant. Although both conditions were subjected to H900 aging, their phase distributions were different. The direct H900 condition contained 81.1% BCC and 18.9% FCC, whereas the combined 1242 °C + H900 condition contained 91.1% BCC and 8.9% FCC. Thus, the presence of the preceding solution annealing step resulted in a different final phase distribution after aging. This difference demonstrates that the response to the H900 treatment depends not only on the aging step itself but also on the initial microstructural condition established before aging.

Overall, the EBSD results demonstrate that the phase distribution of LPBF-produced 17-4 PH stainless steel is influenced by the applied thermal history. The as-built condition contained 89.9% BCC and 10.1% FCC phase, while solution annealing at 1242 °C resulted in 99.2% BCC and only 0.8% FCC phase. Direct H900 aging resulted in 81.1% BCC and 18.9%

FCC phase, whereas the combined 1242 °C + H900 treatment produced 91.1% BCC and 8.9% FCC phase. The results therefore show that the phase distribution obtained after aging differs depending on whether the material is aged directly from the as-built condition or after prior solution annealing. EBSD observations complement the SEM analysis by providing quantitative information on the BCC/FCC phase distribution and crystallographic characteristics. The observed phase distribution should be considered together with the mechanical results, since the hardness and tensile behavior are affected by the combined effects of phase constitution, precipitation, microstructural morphology, and LPBF-induced features.

4.4. Tensile Test

To investigate the influence of build orientation on the mechanical behavior of the material tensile samples were produced in three different orientations: horizontal (0°), inclined (45°) and vertical (90°). Two treatment conditions were selected: direct aging H900 and solution annealing followed by aging (1242 °C + H900). H900 was selected as an industrially established aging reference, whereas 1242 °C + H900 was selected because it produced the highest hardness in the present experimental matrix.

Three specimens were initially assigned to each orientation/treatment combination. However, the number of valid tests was subsequently reduced in several conditions due to experimental issues such as grip slippage or invalid subjected to surface grinding, reducing their thickness from 4 mm to 2 mm to improve gripping compatibility. Therefore, results from the 4 mm and 2 mm specimens were kept separate, and this thickness variation is acknowledged as an experimental limitation.

In addition to the tensile testing, Vickers microhardness was performed on the tensile samples following the same procedure previously performed for the characteristic samples. The measurements were made using a Vickers load of 500 gf (HV 0.5) applied for 15 seconds, with ten indentations performed at random locations on each sample. The purpose of this step was to verify the hardness values previously obtained from the characterization samples were representative of the tensile samples and to assess whether the initial hardness influenced the mechanical properties obtained from the tensile samples. This additional characterization provided a more reliable basis for interpreting the relationship between hardness and tensile behavior.

Stress and strain curve were calculated using the initial dimensions of each sample. For the initial samples an initial area of 24 mm² and a gauge length of 30 mm were used, whereas for the surface ground samples an area of 12 mm² and a gauge length of 25 mm were adopted. The shorter gauge length was selected to maximize the effective gauge section, considering the reduced dimensions of the samples. Results obtained from re-ground specimens were retained only as supplementary/indicative data and were not assumed a priori to be statistically equivalent to the original geometry.

It should be noted that the initial offset stress observed at the beginning of all stress strain

curves corresponds to the preload force applied before the start of the tensile test. This force was necessary to properly secure the samples within the grips, ensuring adequate clamping and preventing sample displacement or slippage during testing. Therefore, the initial stress recorded in the curves includes the contribution associated with the gripping force applied before the actual tensile loading of the samples.

Although the surface grinding procedure improved the gripping conditions it was only partially successful, with only three of the re-tested samples fractured successfully. In total five samples required re-testing, two horizontal samples in the H900 condition fractured successfully, one vertical H900 sample within the gripping region, however this sample already exhibited a defect in the gripping section originating from the manufacturing process that can introduced local stress concentrations and promote premature failure. The remaining two samples correspond to the horizontal and vertical 1242 °C + H900 condition that continued to slip during testing and did not fracture.

Is important to notice that at least two valid tensile tests were successfully completed from each condition. The stress strain curves for the H900 condition are presented in the following section, together with the corresponding value of $R_{p0.2}$, ultimate tensile strength (R_m), elongation (E) and reduction of area (%RA).

As shown in Figure 23 the stress-strain curves reveal differences in the tensile response according to the build orientation, influencing both the strength and deformation behavior of the material. The corresponding tensile properties extracted from the curves are summarized in Table 9.

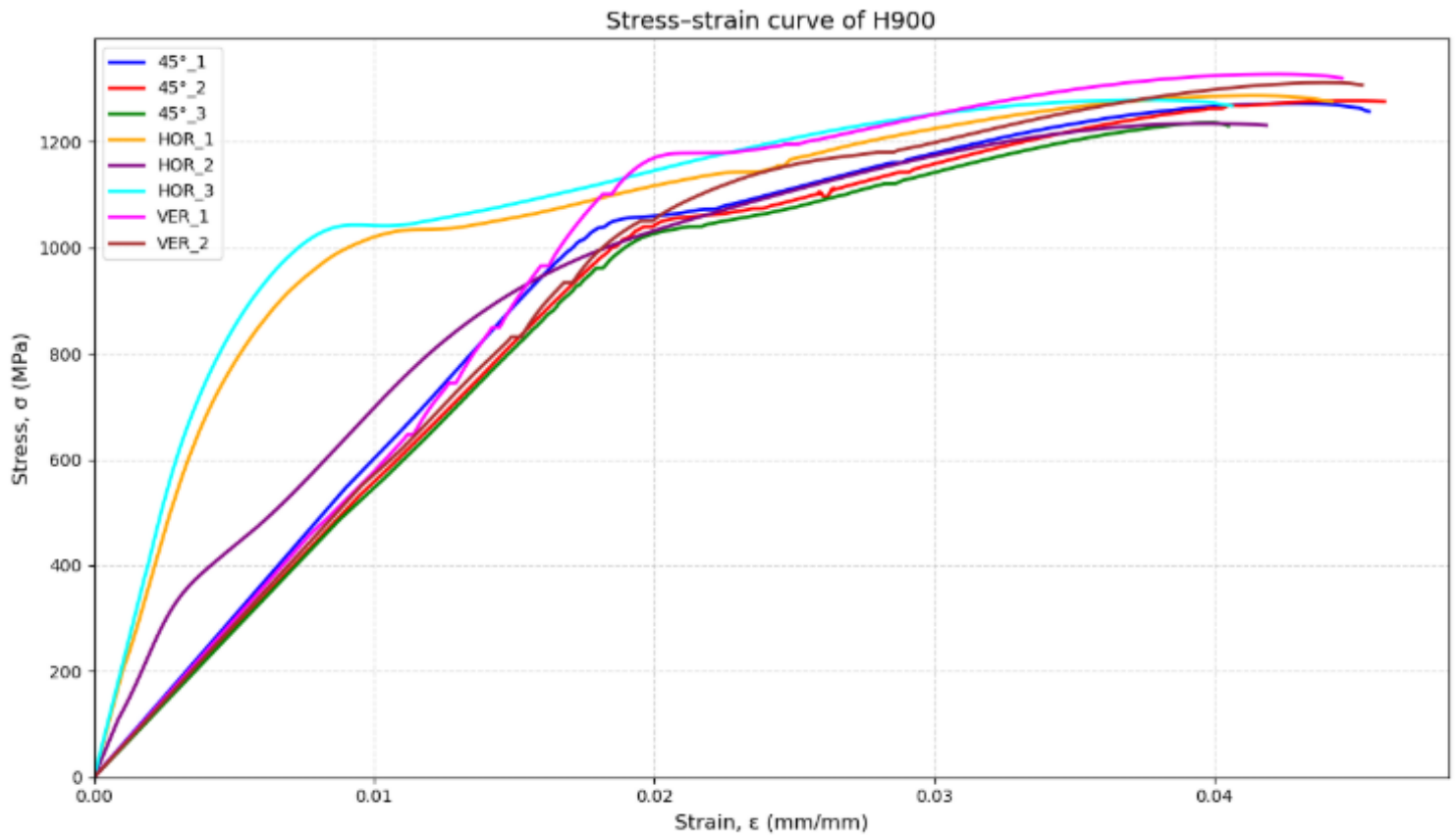


Figure 23. Stress strain curves of the samples 17-4 PH SS produced by LPBF in the H900 condition for the three build orientations (0°, 45° and 90°).

Table 9. Summary of the tensile properties of the 17-4 PH SS produced by LPBF in the H900 condition, including yield strength ($R_{p0.2}$), ultimate tensile strength (R_m), elongation and reduction in area (RA) for each build orientation.

SUMMARY OF THE TENSILE PROPERTIES FOR THE H900 CONDITION						
CONDITION		R_m (MPa)	$R_{p0.2}$ (MPa)	Elongation (%)	RA (%)	HV 0.5
H900	0°	1268 ± 28	1005 ± 5	17 ± 0	8 ± 14	419 ± 18
	45°	1263 ± 23	986 ± 69	14 ± 6	23 ± 2	420 ± 10
	90°	1320 ± 12	1044 ± 73	17 ± 0	21 ± 6	454 ± 15

The tensile behavior of the samples subjected to H900 condition suggests a possible orientation-dependent tensile response; however, this trend should be interpreted with caution considering the limited number of valid tests and pending verification of the strain/offset method for yield strength evaluation. As summarized in Table 9, the samples that were produced with a vertical orientation (90°) achieved the highest strength with a yield strength ($R_{p0.2}$) of 1044 ± 73 MPa and an ultimate tensile strength (R_m) of 1320 ± 12 MPa. The horizontal samples (0°) exhibited an intermediate value ($R_{p0.2} = 1005 \pm 5$ MPa and $R_m = 1268 \pm 28$ MPa), whereas the inclined samples (45°) showed the lowest tensile

properties ($R_{p0.2} = 986 \pm 69$ MPa and $R_m = 1263 \pm 23$ MPa). In comparison with the horizontal orientation the vertical samples exhibited an increase of approximately 3.9 % in yield strength and 4.1 % in ultimate tensile strength, indicating that the build orientation has a more pronounced influence on yielding than on the ultimate tensile response.

The stress strain curves shown in Figure 23 highlight the influence of the build orientation on the deformation behavior. While all the samples exhibited comparable strain hardening characteristics after yielding, noticeable differences were observed in the plastic deformation stage and at the fracture. In particular, the 45° samples showed the largest scatter among the tested samples, that means that have a greater sensitivity at this orientation to local microstructural heterogeneities or process induced defects. Conversely the 90° samples exhibited a more consistent mechanical response and higher stress levels before fracture.

From the ductility point of view, the influence of build orientation is also noticeable. Although the total elongation remained similar for the 0° and 90° samples (17%) the reduction in area differed substantially increasing from 8 ± 14 % in the horizontal samples to 21 ± 6 % in the vertical samples. The 45° orientation exhibited the lowest ductility with an elongation of 14 ± 6 % and a reduction area of 23 ± 2 %. This pronounced variation in reduction of area suggests that build orientation primarily affects the localization of plastic deformation immediately before the fracture rather the overall uniform elongation. The orientation dependence may reflect the combined influence of crystallographic texture, residual process-induced defects, and the relationship between the loading direction and the inherited LPBF microstructural architecture [14].

From a microstructural perspective the mechanical behavior observed in the H900 condition can be correlated with the microstructural features previously identified by the SEM and EBSD. Although the H900 aging treatment promotes precipitation hardening through the formation of nanoscale Cu precipitates on the martensitic matrix, the characteristic morphological anisotropy produced during the LPBF remains evident. Consequently, the alignment of the columnar microstructure and melt pool boundaries relative to the loading direction continues to influence the tensile response, contributing to the superior strength and ductility observed for the vertical built samples [19,46].

The tensile behavior of the samples after the solution annealing at 1242 °C followed by the H900 aging treatment. The effect of build orientation on the tensile response is evident from the differences in yielding behavior, plastic deformation and fracture characteristics, as illustrated in Figure 24. The corresponding properties are shown in Table 10.

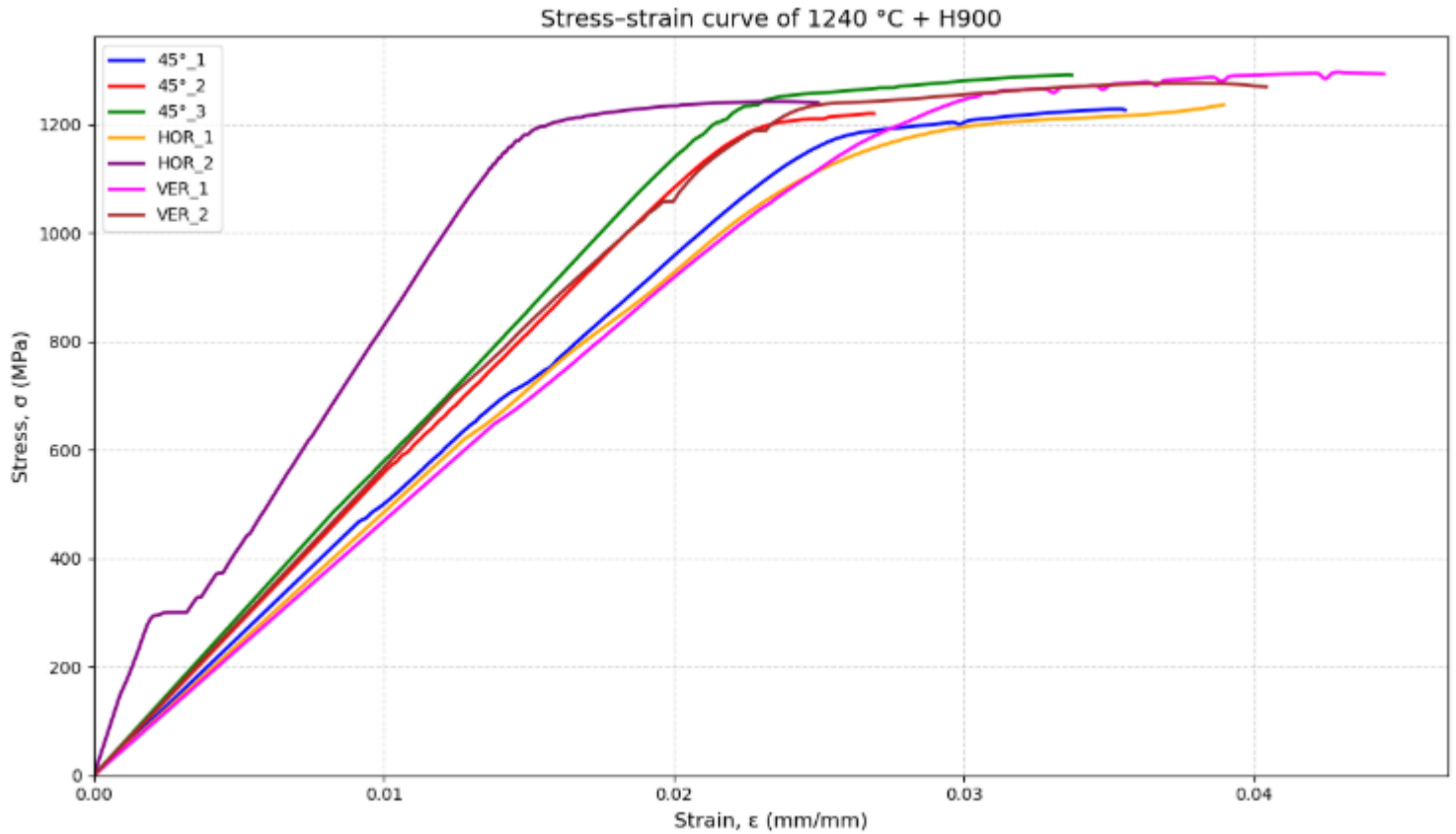


Figure 24. Stress strain curves of the samples 17-4 PH SS produced by LPBF in the combined annealing at 1242 °C + H900 condition for the three build orientations (0°, 45° and 90°).

Table 10. Summary of the tensile properties of the 17-4 PH SS produced by LPBF in the combined annealing at 1242 °C + H900 condition, including yield strength ($R_{p0.2}$), ultimate tensile strength (R_m), elongation and reduction in area (RA) for each build orientation.

SUMMARY OF THE TENSILE PROPERTIES FOR THE 1242 °C + H900 CONDITION						
CONDITION		R_m (MPa)	$R_{p0.2}$ (MPa)	Elongation (%)	RA (%)	HV 0.5
1242 °C + H900	0°	1236 ± 10	1160 ± 38	12 ± 2	0 ± 0	477 ± 24
	45°	1247 ± 39	1101 ± 33	9 ± 5	9 ± 13	520 ± 16
	90°	1286 ± 15	1134	13 ± 0	17 ± 2	530 ± 8

The tensile behavior of the samples produced by LPBF subjected to the combined heat treatment that consists in the solution annealing at 1242°C followed by the H900 aging that exhibited a substantial modification of the mechanical response and anisotropic behavior. As summarized in Table 10 the yield strength ($R_{p0.2}$) displayed considerable scatter with average value of 1160 ± 8 MPa, 1101 ± 33 MPa and 1134 MPa, for the horizontal (0°), inclined (45°) and vertical (90°) build orientations respectively. The exceptionally

low value measured for the 45° samples was strongly influenced by pronounced plastic instabilities during the early stages of deformation and should therefore be interpreted with caution. Additionally, significant variability was observed among the vertical samples, mainly due to the behavior of one individual sample (Ver_2). The corresponding stress strain curve exhibited an initial stress offset caused by the higher clamping force applied during gripping to prevent sample slippage. This preload was recorded by the load cell before the beginning of tensile, like in at other sample, that contributed to the scatter observed in the calculated yield strength values for this orientation. In contrast, the ultimate tensile strength (R_m) remained consistent across all building orientations, ranging from 1236 ± 10 to 1286 ± 15 MPa [17, 46].

Although the ultimate tensile strength was independent of build orientation, a pronounced deterioration in ductility was observed. The reduction in RA decreased to 0 ± 0 % for the horizontal samples and only 6% for the inclined samples, whereas the vertical samples retained an RA of 17 ± 2 %. These results indicate a significant loss of plastic deformation capability prior to fracture, particularly for the non-vertical orientations.

The observed mechanical response is consistent with the microstructural evolution identified by SEM and EBSD after the combined condition. The treatment led to the elimination of melt pool boundaries from the LPBF process. The resulting homogenized microstructure, combined with intense precipitation hardening, acts as an effective barrier to dislocation movement, which explain the macroscopic yield strength observed after this heat treatment [19, 46]. In addition the EBSD analysis indicated the presence of retained austenite after the solution annealing treatment. A strain-induced transformation of metastable austenite is a possible contributing mechanism reported in the literature, but it was not directly measured in the present tests [17, 19].

A direct comparison between the direct aging (H900) condition and the combined (1242 °C + H900) condition demonstrates that the addition of solution annealing treatment does not provide an overall improvement in the tensile performance. Although the combined treatment produced a more homogeneous microstructure by eliminating the chemical segregation and defect cellular structure characteristic of LPBF, this homogenization severely compromised the material's ductility. The yield strength actually increased compared to the direct aging condition, but the pronounced reduction in ductility can be associated with the reduction of reverted austenite and the lower defect density resulting from solution annealing. Compared with the direct aging condition, the 1242 °C + H900 sample contained a lower FCC phase fraction, reducing the contribution of the ductile austenitic phase to plastic accommodation during deformation.

When considered together with the hardness measurements and the microstructural characterization these results provide a more comprehensive understanding of the mechanical response. Exhibiting the highest hardness values when compared with the others heat treatments condition investigated, the 1242 °C + H900 condition also showed the highest yield strength. The high hardness obtained after the combined treatment is therefore consistently associated with the high yield strength, more homogeneous martensitic structure and precipitation strengthening. However, it does not reflect the

overall tensile performance, which is severely limited by premature brittle failure. The SEM and EBSD analyses showed that the high-temperature treatment produced a more homogeneous and apparently coarser microstructure; the extent of recrystallization was not quantified in the present work, but this microstructural modification ultimately led to reduced ductility. This demonstrates that hardness cannot be used to predict the overall mechanical performance of LPBF 17-4 PH SS.

Building orientation is also important and plays a significant role in determining the tensile response. The vertical (90°) samples consistently exhibited the most favorable combination of strength and ductility, whereas the horizontal (0°) and inclined (45°) samples showed a greater reduction in plastic deformation before fracture. This orientation dependence is related to the anisotropic microstructure inherited from LPBF, where the alignment of melt pool boundaries, martensitic laths and crystallographic orientations relative to the loading direction affects the activation of plastic deformation mechanism.

Overall, direct aging (H900) provides the most balanced mechanical performance among the heat treatments conditions investigated. By retaining part of the refined defect LPBF structure while promoting effective precipitation hardening and reverted austenite formation, this treatment achieves a superior balance between strength and ductility. Furthermore, within the limitations of the present tensile dataset, the 90° H900 specimens exhibited the most favorable combination of measured strength and ductility.

4.5. Fracture Surface Analysis

The fracture surfaces of the tensile samples were analyzed by SEM to investigate the fracture mechanisms associated with the different heat treatment conditions and build orientations. This analysis was performed to identify the characteristic morphological features of the fracture surfaces and to assess their relationship with the mechanical behavior obtained from the tensile and hardness test. Particular attention was given to the presence of dimples, smooth regions, pores, and other discontinuities, as well as the differences observed between the direct aging (H900) and the combined condition (1242 °C + H900). The influence of the horizontal, inclined and vertical build orientation on the fracture morphology was also evaluated.

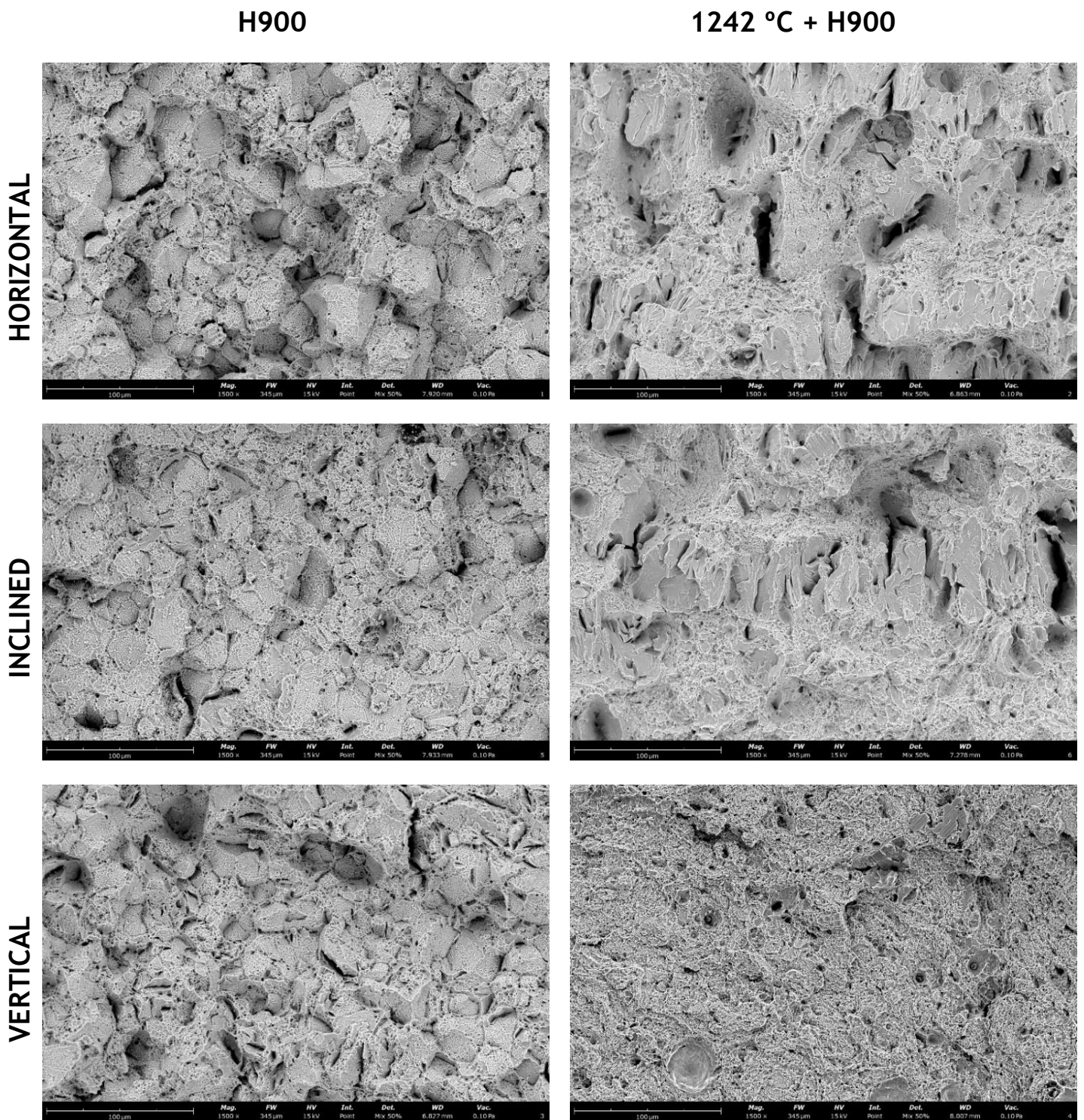


Figure 25. SEM fracture surface morphology of tensile specimens fabricated in the horizontal, inclined (45°), and vertical build orientations after the H900 and 1242 °C + H900 heat treatments

Figure 25 presents the fracture surfaces obtained of the samples subjected to direct aging (H900) and the solution annealing at 1242 °C followed by H900 aging, considering the horizontal, inclined, and vertical build orientations.

Overall, differences in fracture morphology can be observed between the two heat treatment conditions. The samples subjected to the direct H900 treatment exhibited a fracture morphology characterized by numerous cavities of varying sizes, along with microvoid nucleation and coalescence, which are characteristic mechanisms of ductile fracture in metallic materials [5].

In contrast, the samples subjected to the 1242 °C + H900 treatment shown in the second column exhibit a noticeably finer and more uniform fracture appearance compared to the direct aging condition. As previously supported by EBSD analyses, high-temperature solution annealing drastically reduces the retained austenite fraction and homogenizes the martensitic matrix, whereas direct aging preserves a higher fraction of reverted austenite. Similar variations in fracture morphology, including the coexistence of ductile regions and regions with a more brittle character, have been reported in AM of 17-4 PH SS [13].

The differences observed between the two heat treatment conditions can be related to the changes in microstructure induced by the thermal treatments. Solution annealing at high temperature promotes the homogenization of the microstructure and modifies the phase distribution resulting from the LPBF process. Subsequent aging promotes precipitation hardening, which can significantly affect the mechanical response and fracture behavior of 17-4 PH stainless steel [13]. Therefore, the variations in fracture morphology observed between the H900 and 1242 °C + H900 conditions reflect the distinct microstructures developed during these respective heat treatments.

The fractographic features observed in both conditions confirm that local ductile fracture, driven by microvoid nucleation and coalescence, is dominant [5]. However, local visual variations on the fracture surface should be interpreted with caution and not be directly equated with macroscopic mechanical performance, as elongation measured during tensile testing represents the bulk plastic deformation capacity, whereas SEM analysis describes the local mechanisms operating immediately prior to final fracture.

This distinction is particularly relevant when comparing the fractographic observations with the mechanical results obtained. Although the 1242 °C + H900 condition exhibited higher hardness values, ranging from 477 to 530 HV0.5, compared with 419 to 454 HV0.5 for the H900 condition, it presented lower elongation values, ranging from 9 to 13%, compared with 14 to 17% for the H900 condition. The ultimate tensile strength was also slightly lower after the combined heat treatment. This indicates that the higher resistance to localized plastic deformation measured by hardness did not translate into higher macroscopic tensile ductility.

The observed behavior can be associated with the microstructural modifications induced by solution annealing and subsequent aging. The evolution of the martensitic matrix and the formation or dissolution of reverted austenite during heat treatment can influence the capacity of the material to accommodate plastic deformation and resist crack propagation [13]. The presence and stability of reverted austenite affect the deformation and fracture response of 17-4 PH stainless steel, contributing to the accommodation of plastic strain and influencing crack propagation mechanisms [36].

Consequently, the higher hardness observed for the 1242 °C + H900 condition does not necessarily indicate higher tensile strength or ductility. Instead, the increased hardness may reflect a stronger resistance to localized plastic deformation, while the lower elongation indicates a reduced capacity for macroscopic plastic deformation before failure. The relationship between precipitation hardening, hardness, and tensile properties reported for 17-4 PH stainless steel supports the importance of considering the microstructural state and phase balance when interpreting the mechanical response [13].

The build orientation also influenced the fracture morphology and tensile behavior. In the H900 condition, the vertical samples exhibited the most favorable combination of strength and ductility, reaching an ultimate tensile strength of 1320 MPa and an elongation of 17%. The horizontal and inclined samples presented ultimate tensile strengths of 1268 and 1263 MPa, respectively, with elongations of 17 and 14%. These differences indicate that the anisotropic microstructure inherited from the LPBF process continues to influence the mechanical response after heat treatment. The influence of build orientation on the mechanical and fracture behavior of LPBF-produced 17-4 PH stainless steel has been associated with the orientation of melt pool boundaries, crystallographic texture, and process-induced defects relative to the loading direction [36].

After the 1242 °C + H900 treatment, the ultimate tensile strength remained relatively similar between the three orientations, whereas the elongation decreased to 12%, 9%, and 13% for the horizontal, inclined, vertical orientations, respectively. Unlike the direct aging condition, the fracture surfaces obtained after the combined heat treatment appeared finer and more uniform across the investigated build orientations.

The presence of pores and discontinuities observed on the fracture surfaces may also have contributed to crack initiation and propagation. In LPBF-produced materials, process-induced defects such as porosity and lack-of-fusion regions can act as stress concentrators and preferential sites for crack initiation, particularly when their morphology and orientation are unfavorable with respect to the applied loading direction [36]. Nevertheless, the specific nature of the discontinuities observed in the present SEM images cannot be conclusively identified solely from the fracture surfaces. Therefore, they should be described as pores or discontinuities unless their chemical composition or morphology has been confirmed through complementary characterization techniques.

Overall, the fractographic analysis demonstrates that the heat treatment significantly influences the fracture behavior of LPBF-produced 17-4 PH stainless steel. The H900 condition exhibited a more heterogeneous fracture morphology combining rough regions with smoother areas, whereas 1242 °C + H900 condition displayed a finer and more uniform appearance [5]. When correlated with the tensile and hardness results, these observations indicate that the higher hardness achieved after solution annealing and aging did not result in improved macroscopic ductility. Instead, fracture behavior appears to be governed by the combined effects of the microstructural state, phase balance, precipitation hardening, build orientation, and the presence of process-induced defects [13,36].

5. CONCLUSIONS

The present work investigated the production and characterization of LPBF-fabricated 17-4 PH martensitic stainless steel, with particular emphasis on the influence of post-heat treatments on the microstructure and mechanical performance of the alloy. By combining powder characterization, SEM and EBSD analyses, microhardness measurements and tensile testing, it was possible to establish clear relationships between processing conditions, phase evolution and the resulting mechanical behavior.

The characterization of the gas-atomized powder confirmed that the feedstock exhibited a predominantly spherical morphology with a relatively narrow particle size distribution ($D_{50} = 34.65 \mu\text{m}$), which is suitable for LPBF processing. Satellite particles, internal pores and localized chemically heterogeneous regions with high oxygen content, consistent with oxide-containing features, were occasionally observed in the examined powder fields.

Microstructural characterization demonstrated that the as-built LPBF material consisted predominantly of a refined lath martensitic structure containing retained austenite, resulting from the rapid solidification and cyclic thermal history inherent to the LPBF process. SEM observations revealed the characteristic melt-pool and cellular solidification features, while EBSD analysis quantified the phase distribution and confirmed the presence of approximately 10.1% retained austenite in the as-built condition.

Solution treatment reduced the visibility of the LPBF cellular/melt-pool features and produced a more homogeneous microstructural appearance. EBSD results showed that solution annealing at 1242°C reduced the retained austenite fraction to approximately 0.8%, resulting in an almost fully martensitic microstructure. Following subsequent H900 aging, reverted austenite was formed; however, its fraction remained significantly lower than that obtained after direct aging.

The evolution of reverted austenite was strongly dependent on the previous microstructural condition. Direct aging (H900) produced the highest reverted austenite fraction (18.9%), since the LPBF cellular structure and high dislocation density were preserved, promoting Ni redistribution and austenite reversion during aging. In contrast, prior solution annealing homogenized the alloy, reduced the defect density and suppressed chemical segregation, thereby decreasing the driving force for reverted austenite formation. These results demonstrate that the response to aging is governed not only by the aging parameters but also by the microstructural state established before aging.

Microhardness measurements showed that all heat treatments increased the hardness compared with the as-built condition. The highest value ($538 \pm 3 \text{ HV0.5}$) was obtained after the combined $1242^\circ\text{C} + \text{H900}$ treatment, demonstrating the beneficial effect of high-temperature solution annealing followed by precipitation hardening. Nevertheless, hardness alone was not representative of the overall mechanical performance.

The tensile test results demonstrated that the direct aging condition (H900) provides the most favorable strength-ductility balance. In contrast, the combined heat treatment (solution annealing at 1242 °C followed by H900) resulted in a significant loss of ductility despite achieving high yield strength. This embrittlement is attributed to the microstructural modifications introduced by the high-temperature solution annealing treatment, including the development of a more homogeneous and coarser microstructure, alongside a lower amount of reverted austenite available to accommodate plastic deformation.

The tensile results also indicated that build orientation continues to influence the mechanical response of the LPBF material. Differences in strength and ductility were observed between the horizontal, inclined and vertical specimens, suggesting that the anisotropy introduced during layer-by-layer fabrication remains an important factor even after heat treatment.

SEM examination of the fracture surfaces revealed predominantly ductile fracture characteristics for both heat treatment conditions, although clear differences in fracture morphology were observed. The 1242 °C + H900 specimens exhibited rougher fracture surfaces with more pronounced plastic deformation, whereas the H900 specimens showed smoother regions together with pores and manufacturing-related defects, indicating that fracture behavior was influenced by both the heat treatment and the LPBF processing route.

Overall, this work demonstrates that the mechanical performance of LPBF-produced 17-4 PH stainless steel results from the combined effects of build orientation, heat treatment and phase evolution. While the combined solution annealing and aging treatment maximized hardness, direct aging (H900) provided the most favorable balance between strength and ductility while avoiding the additional processing time and cost associated with solution annealing. These findings provide experimental insight into the processing-microstructure-property relationships of LPBF 17-4 PH and support the design of future, statistically robust heat-treatment studies.

5.1. Future work perspectives

Future work should first focus on validating the tensile response using a standardized or fully documented specimen geometry, an extensometer or digital image correlation for strain measurement, and gripping conditions that eliminate slippage. A larger number of valid specimens should be tested for each build orientation to allow statistical assessment of anisotropy. The influence of process-induced defects should be quantified through density/porosity measurements, preferably complemented by X-ray micro-computed tomography. Additional phase characterization by X-ray diffraction would provide an independent assessment of the BCC/FCC phase balance, while EBSD analyses over multiple areas, with quantified grain size, texture and local-misorientation metrics, would strengthen the interpretation of the heat-treatment response. Finally, transmission

electron microscopy could be used to directly characterize the Cu-rich nanoscale precipitates responsible for precipitation hardening and to clarify their interaction with dislocations and boundaries.

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