

**FACULDADE DE ENGENHARIA DA UNIVERSIDADE DO PORTO**

# **Development of printing parameters for porous iron structures obtained by Laser Powder Bed Fusion**

**José Miguel Castro da Silva**



**Master's Degree Dissertation in Materials Engineering**

**Supervisor: Jorge Lino Alves**

**Second Supervisor: Pedro Lopes**

**July 31, 2025**





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# Abstract

Pure iron has emerged as a promising material for biodegradable bone implants due to its excellent biocompatibility and gradual degradation in physiological environments. However, its high stiffness (Young's modulus of  $\approx 200$  GPa) compared to bone can lead to stress shielding, where the implant carries too much of the load, reducing mechanical stimulation of surrounding tissue and potentially causing bone resorption. Also, pure iron has a low degradation rate that limits its effectiveness in applications requiring timely implant degradation. Introducing controlled porosity through lattice structures addresses both challenges: reducing stiffness and increasing surface area to enhance degradation. Laser Powder Bed Fusion (L-PBF) provides the precision needed for such geometries, but its use with pure iron remains underexplored and demands parameter development.

This work develops L-PBF processing parameters for pure iron. The study covers: (1) microstructural analysis of specimens with varied bulk parameters, (2) mechanical characterization through tensile tests, (3) contour parameter development in two case studies, and (4) powder characterization for future use.

Microstructural analysis revealed a hierarchical microstructure with elongated grains and a wide variation in grain size. Mechanical testing confirmed that the developed parameters yield properties consistent with pure iron produced by L-PBF, including a tensile strength of approximately 477 MPa and a Young's modulus of around 180 GPa. Case studies showed that Linear Input Energy Density affect contour continuity and porosity. Despite challenges in surface finish and dimensional accuracy, pure iron's mechanical properties and densification were promising for its intended use in biodegradable implants. Additionally, powder characterization showed irregular particle morphology, suggesting water-atomized powder, with a particle size distribution suitable for L-PBF use, though further characterization is needed for a complete understanding.

Overall, the study successfully demonstrates the feasibility of fabricating pure iron cellular structures via L-PBF and enhances understanding of pure iron in this technology, advancing biodegradable iron-based implants with tailored mechanical and degradation behavior.

**Keywords:** Additive Manufacturing, Lattice Structures, Processing Parameters, Microstructure, Biomedical Implants



# Resumo

O ferro puro destaca-se como material promissor para implantes ósseos biodegradáveis, pela sua biocompatibilidade e degradação gradual em ambientes fisiológicos. Contudo, a sua elevada rigidez (módulo de Young de  $\approx 200$  GPa), superior à do osso, pode provocar *stress shielding*, onde o implante suporta carga excessiva, reduzindo o estímulo mecânico no osso e podendo causar reabsorção do mesmo. Além disso, a baixa taxa de degradação do ferro limita aplicações onde a reabsorção do implante deve ocorrer num prazo específico. A introdução de porosidade controlada através de estruturas celulares permite resolver ambos os desafios: reduzir a rigidez e aumentar a área superficial, acelerando a degradação. A tecnologia *Laser Powder Bed Fusion* (L-PBF) oferece a precisão necessária para estas geometrias, embora a produção de ferro puro ainda seja pouco explorada e requeira otimização de parâmetros.

Este trabalho desenvolve parâmetros de L-PBF para ferro puro. O estudo inclui: (1) análise microestrutural de amostras com diferentes parâmetros *bulk*, (2) caracterização mecânica por tração, (3) desenvolvimento de parâmetros de contorno em dois casos de estudo e (4) caracterização de um pó para futuras produções.

A análise microestrutural revelou uma microestrutura hierárquica com grãos alongados e uma ampla variação no tamanho dos grãos. Os ensaios mecânicos confirmaram que os parâmetros desenvolvidos geram propriedades consistentes com o ferro puro produzido por L-PBF, incluindo uma resistência à tração de aproximadamente 477 MPa e um módulo de Young de cerca de 180 GPa. Casos de estudo mostraram que a Densidade de Energia Linear afeta a continuidade e porosidade do contorno. Apesar dos desafios na qualidade superficial e precisão dimensional, as propriedades mecânicas e a densificação do ferro puro mostraram-se promissoras para o seu uso em implantes biodegradáveis. Além disso, a caracterização do pó mostrou morfologia irregular das partículas, sugerindo pó atomizado por água, com uma distribuição de tamanho de partículas adequada para uso em L-PBF, embora mais caracterizações sejam necessárias para uma compreensão completa.

No geral, o estudo demonstra com sucesso a viabilidade de fabricar estruturas celulares de ferro puro via L-PBF e aprimora o entendimento do ferro puro nesta tecnologia, avançando o desenvolvimento de implantes biodegradáveis à base de ferro com comportamento mecânico e de degradação personalizado.

**Palavras-chave:** Fabrico Aditivo, Ferro Poroso, Estruturas Celulares, Parâmetros de Processamento, Microestrutura, Implantes Biomédicos



# Scientific Disclosures

- P. Lopes, J. Silva, and J. L. Alves. "Optimizing Process Parameters for Manufacturing Porous Pure Iron Scaffolds Using Laser Powder Bed Fusion (L-PBF)". 2025. IJUP'25 – 18.º Encontro de Investigação Jovem da Universidade do Porto.
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*“Man can do what he wills, but he cannot will what he wills”*

Arthur Schopenhauer



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# List of Abbreviations

|          |                                     |
|----------|-------------------------------------|
| AM       | Additive Manufacturing              |
| BCC      | Body-Centered Cubic                 |
| CAD      | Computer-Aided Design               |
| C        | Cubic                               |
| CI       | Carr Index                          |
| DED      | Directed Energy Deposition          |
| DLS      | Dynamic Light Scattering            |
| EBM      | Electron Beam Melting               |
| EBSD     | Electron Backscatter Diffraction    |
| ECM      | Extracellular Matrix                |
| EDM      | Electrical Discharge Machining      |
| E-PBF    | Electron Powder Bed Fusion          |
| FCC      | Face-Centered Cubic                 |
| GA       | Gas Atomization                     |
| GND      | Geometrically Necessary Dislocation |
| HA       | Hydroxyapatite                      |
| HR       | Hausner Ratio                       |
| HV       | Vickers Hardness                    |
| IPF      | Inverse Pole Figure                 |
| LD       | Laser Diffraction                   |
| LMD      | Laser Metal Deposition              |
| L-PBF    | Laser Powder Bed Fusion             |
| Micro-CT | Micro-Computed Tomography           |
| OM       | Optical Microscopy                  |
| PBF      | Powder Bed Fusion                   |
| PCL      | Polycaprolactone                    |
| PGA      | Polyglycolic Acid                   |
| PEEK     | Polyetheretherketone                |
| PLA      | Polylactic Acid                     |
| PLGA     | Poly(lactic-co-glycolic acid)       |
| PSD      | Particle Size Distribution          |
| RCO      | Rhombicuboctahedron                 |
| RTCO     | Rhombitruncated Cuboctahedron       |
| SEM      | Scanning Electron Microscopy        |

|              |                                          |
|--------------|------------------------------------------|
| SLM          | Selective Laser Melting                  |
| SLS          | Selective Laser Sintering                |
| TC           | Truncated Cuboctahedron                  |
| TPMS         | Triply Periodic Minimal Surface          |
| UHMWPE       | Ultra-High Molecular Weight Polyethylene |
| VED          | Volumetric Energy Density                |
| WA           | Water Atomization                        |
| $\beta$ -TCP | $\beta$ -Tricalcium Phosphate            |

# List of Symbols

|                          |                                                             |
|--------------------------|-------------------------------------------------------------|
| $A$                      | Elongation at Fracture                                      |
| $Cr$                     | Chromium                                                    |
| $d$                      | Laser Spot Size                                             |
| $d_{10}, d_{50}, d_{90}$ | Particle Size Percentiles (10th, 50th, and 90th percentile) |
| $d_{\text{mean}}$        | Mean Particle Size                                          |
| $E$                      | Young's Modulus                                             |
| $E_{\text{int}}$         | Energy Intensity                                            |
| $Fe$                     | Iron                                                        |
| $H$                      | Hardness                                                    |
| $H_h$                    | Hole Height                                                 |
| $H_w$                    | Hole Width                                                  |
| $h$                      | Hatch Spacing                                               |
| $Mg$                     | Magnesium                                                   |
| $P$                      | Laser Power                                                 |
| $P$                      | Porosity                                                    |
| $R_m$                    | Ultimate Strength                                           |
| $R_{p0.2}$               | Yield Strength at 0.2% Offset                               |
| $Ra$                     | Surface Roughness (Arithmetic Average Roughness)            |
| $t$                      | Strut Thickness                                             |
| $t$                      | Layer Thickness                                             |
| $T_m$                    | Melting Point                                               |
| $T_n$                    | Ambient Temperature                                         |
| $T_p$                    | Plastic Point                                               |
| $v$                      | Scanning Speed                                              |
| $Zn$                     | Zinc                                                        |
| $\rho_B$                 | Bulk Density                                                |
| $\rho_T$                 | Tapped Density                                              |





# Chapter 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. It presents a study on the fabrication of porous iron structures via Laser Powder Bed Fusion (L-PBF), with a particular focus on their application in biodegradable bone implants. This research aligns with the goals of the GradImp project, officially titled (“*Biodegradable Implants in Porous Iron Obtained by Additive Manufacturing*”).

GradImp is a research project funded under the project number **PTDC/CTM-CTM/3354/2021** and supported by FCT (Fundação para a Ciência e Tecnologia). The project brings together a multidisciplinary consortium of institutions:

- **Institute of Mechanical Engineering (IDMEC)**
- **Faculty of Medicine of the University of Porto (FMUP)**
- **INEGI – Institute of Science and Innovation in Mechanical and Industrial Engineering**
- **Polytechnic Institute of Setúbal (IPS)**
- **CeFEMA – Center of Physics and Engineering of Advanced Materials**
- **Faculty of Engineering of the University of Porto (FEUP)**
- **Higher Technical Institute (IST)**

The joint effort aims to develop innovative temporary fixation devices based on porous iron scaffolds manufactured via Laser Powder Bed Fusion (L-PBF), combining expertise in materials engineering and biomedical sciences.

This chapter outlines the motivation for the study, defines the main objectives, and provides an overview of the dissertation’s structure.

## 1.1 Background

Additive Manufacturing (AM), and more specifically L-PBF, has emerged as a powerful technique for producing metallic components with intricate geometries and controlled internal architectures. In biomedical engineering, this technology enables the design of lattice structures that closely replicate the mechanical and morphological characteristics of natural bone. Such implants can be engineered to be biodegradable, supporting bone regeneration while gradually degrading *in situ*. This eliminates the need for secondary surgeries to remove the implant, providing both patient benefits and healthcare cost reductions.

Among the materials investigated for biodegradable implant applications, pure iron shows promise due to its mechanical strength and biocompatibility. However, its slow degradation rate under physiological conditions and high Young's modulus (around 200 GPa) are limitations, especially when compared to materials like magnesium or zinc. These issues can lead to stress shielding, where the stiffness mismatch inhibits natural bone remodeling. Introducing controlled porosity can mitigate these drawbacks by reducing stiffness and enabling better regulation of the degradation rate.

The GradImp project focuses on overcoming these challenges by developing porous iron scaffolds with variable density lattice structures tailored for temporary biomedical applications. The project aims to fabricate these scaffolds via L-PBF, optimize their design and processing parameters, and assess their cytocompatibility and biodegradation behavior. This dissertation contributes to these objectives by focusing on the additive manufacturing of pure iron lattice structures, with an emphasis on process parameter development, microstructural analysis, and powder characterization.

## 1.2 Objectives and Scope

The main goal of this study is to support the development of porous iron lattice structures using the L-PBF process within the broader scope of the GradImp project. The specific objectives are:

- Investigate the influence of bulk processing parameters on the resulting microstructure.
- Evaluate the mechanical properties of bulk samples through tensile tests.
- Develop contour parameters through two case studies. In Case Study 1, evaluate the influence of varying linear input energy density on contour continuity and porosity. In Case Study 2, evaluate the porosity of cellular structures produced with different parameters.
- To characterize an iron powder intended for future manufacturing, focusing on morphology, particle size distribution, and density.

## 1.3 Structure of the Dissertation

This dissertation is organized into the following chapters:

- **Chapter 1 – Introduction** outlines the motivation behind the study, presents the research objectives, and introduces the GradImp project. It also provides an overview of the dissertation structure.

- **Chapter 2 – Literature Review** provides the scientific and technological background for the study. It covers key topics including tissue engineering, design of lattice structures for biomedical implants, L-PBF and process parameters, properties and behavior of metallic powders, typical defects in L-PBF produced parts, and the microstructure of pure iron produced via L-PBF.
- **Chapter 3 – Materials and Methods** details the experimental procedures and materials used. This includes a description of the powders and L-PBF equipment, the development of bulk and contour process parameters, and the application of various characterization techniques.
- **Chapter 4 – Results Presentation and Discussion** presents and analyzes the results of the experimental work. This includes the effects of process parameters on microstructure, grain morphology, porosity, surface quality, and the dimensional fidelity of cellular structures. A comparative analysis of pure iron and stainless steel lattices is also discussed, along with a detailed evaluation of powder density, morphology, and particle size distribution.
- **Chapter 5 – Conclusions and Future Work** summarizes the main conclusions drawn from the research, highlighting key results and their implications. It also outlines potential directions for future studies.

## Chapter 2

# Literature Review

### 2.1 Tissue Engineering

Tissue engineering is an interdisciplinary field that merges principles of biology, engineering, and materials science to restore, replace, or enhance the function of damaged or diseased tissues. It involves the use of scaffolds, cells, and signaling molecules to guide tissue regeneration, often aiming to replicate the native structure and function of the target tissue. In the context of orthopedic medicine, tissue engineering focuses on developing bioactive and biodegradable constructs that can support bone repair and regeneration. These engineered solutions offer potential advantages over traditional grafts and permanent implants by promoting biological integration, reducing the need for secondary surgeries, and matching the mechanical and biochemical properties of natural bone [1, 2].

#### 2.1.1 Bone Structure and Function

Bone is a porous composite material composed of minerals, proteins, water, cells, and other macromolecules. Its composition, architecture, and mechanical properties vary with anatomical location, age, diet, physical activity, and diseases that affect it. Bone has various functions, including the protection and support of soft tissues, locomotion, the harboring of bone marrow, and the storage of calcium and phosphate. It is composed of both organic and inorganic components. Type I collagen is the main organic component (90%), but there are also Type III and Type IV collagens, as well as fibrillin. Bone is also composed of an inorganic crystalline mineral such as hydroxyapatite (HA), which is mainly made up of calcium phosphate salts, allowing bone to function as a reservoir of phosphate and calcium that can be mobilized. Collagen molecules provide flexibility, while HA provides strength and toughness. Bone also contains magnesium, citrate, fluoride, and acid phosphate [3–5].

Bone can also be categorized into cortical and trabecular bone (Figure 2.1). Cortical bone, or the cortex, is a thin, dense, and compact outer region. Within the cortex is the marrow cavity, which contains hematopoietic elements, fat, and trabecular bone, an open-celled porous structure made up of slender spicules and interconnecting trabeculae separated by marrow spaces [4].

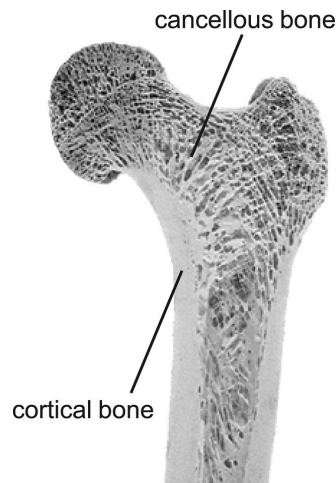


Figure 2.1: Cortical and trabecular (or cancellous) bone in human femur [6].

### 2.1.2 Bone Mechanical Properties

As mentioned in the previous subsection, bone is composed of collagen and HA. Collagen exhibits low stiffness compared to HA, has tensile strength sufficient to support physiological loads, and has limited capacity to resist compressive forces. HA is brittle, provides significant resistance to compression, and primarily contributes to the overall stiffness of the bone. Together, these components form an anisotropic composite material characterized by substantial compressive resistance and intermediate tensile strength [4].

Bone is also organized into two structural types: cortical (compact bone) and trabecular (cancellous bone). Cortical bone exhibits porosity between 3% and 5%, comprises approximately 80% of total bone mass, and primarily supports mechanical loads. In comparison, trabecular bone displays porosity ranging from 50% to 90% and constitutes about 20% of bone mass [3, 7, 8]. Cortical bone effectively resists torsional forces, whereas trabecular bone accommodates compressive and shear loads. The mechanical behavior of bone varies with factors such as age, anatomical location, and structural characteristics. Additionally, the performance of trabecular bone under mechanical load is influenced by the orientation of its interconnected trabeculae. Due to this complex architecture, bone exhibits anisotropic behavior, with direction-dependent stiffness and strength [3, 4, 9].

Table 2.1 provides a general estimation of the typical ranges of mechanical properties found in human cortical and trabecular bone. These values should be interpreted as approximations, as they can vary depending on the testing method and conditions used.

Young's modulus is the mechanical property with the most importance in the design of medical implants, as the use of implants with greater stiffness than bone can cause stress shielding, bone reabsorption, and the inhibition of bone formation [3]. Stress shielding is a mechanical phenomenon that occurs when an implant has a significantly higher stiffness than the surrounding bone, causing it to absorb most of the mechanical load. As a result, the bone experiences reduced mechanical stimulation, which is essential for maintaining healthy bone structure. This reduction in load leads to bone resorption, resulting in a net loss of bone mass. Simultaneously, the lack of mechanical stimulus inhibits new bone formation, compromising osseointegration and potentially leading to implant loosening and long-term failure [18].

Table 2.1: Typical mechanical property ranges of human cortical and trabecular bone [10–17].

| Mechanical Property                      | Cortical Bone | Trabecular Bone |
|------------------------------------------|---------------|-----------------|
| Young's Modulus (GPa)                    | 7 – 30        | 0.05 – 0.5      |
| Compressive Strength (MPa)               | 100 – 205     | $\approx 50$    |
| Tensile Strength (MPa)                   | 50 – 150      | 8 – 50          |
| Elongation at Fracture (Tensile) (%)     | 1 – 3         | 2 – 7           |
| Elongation at Fracture (Compressive) (%) | $\approx 2$   | $\approx 50$    |

### 2.1.3 Permanent and Temporary Medical Implants

Implants are biomedical devices that replace a part of the body, enabling the repair or regeneration of damaged tissues or organs. They are used for orthopedic, cardiovascular, and dental applications. There are important features that an implant must have, like biocompatibility, stability, and functionality [19]. There are two types of implants: permanent and temporary. Permanent implants are designed for long-term stability and durability as they stay in the patient's body for the rest of their life [19, 20]. Table 2.2 presents commonly used materials for permanent implants along with their properties.

Within temporary implants, those can include stents for cardiovascular treatment, scaffolds for bone regeneration (Figure 2.2), and devices used for bone fracture fixation (Figure 2.3). These implants can be biodegradable, allowing for bone regeneration but degrading with time, avoiding an implant removal surgery, which benefits the patient and avoids high societal costs. The porous design of these implants allows the control of degradation rate and the adjustment of mechanical properties as needed [7, 20, 21].

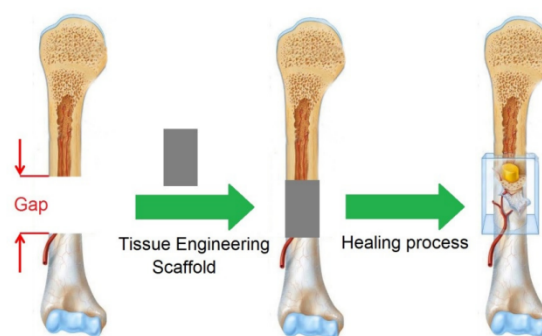


Figure 2.2: Tissue engineering approach for reconstruction of bone fractures [22].

Table 2.2: Permanent Implant Materials: Young's modulus, advantages and disadvantages.

| Material Type                             | Young's Modulus (GPa) | Advantages                                                                                                 | Disadvantages                                                                 | Author(s)    |
|-------------------------------------------|-----------------------|------------------------------------------------------------------------------------------------------------|-------------------------------------------------------------------------------|--------------|
| Titanium and Alloys                       | 110–120               | Lightweight, excellent osseointegration, corrosion-resistant, high biocompatibility, minimal encapsulation | High cost, possible metal ion release, imaging interference                   | [23–25]      |
| Stainless Steel (316L)                    | 200                   | Cost-effective, easily manufactured, good mechanical properties                                            | Corrosion risk, metal ion release, stress shielding, fibrous tissue formation | [25–27]      |
| Cobalt-Chromium Alloys                    | 220–230               | Excellent strength, wear/corrosion resistance, fatigue resistance                                          | Expensive, cobalt ion toxicity, high modulus, possible allergies              | [25]         |
| PEEK                                      | 3–4                   | Radiolucent, bone-like modulus, stress shielding reduction, chemical resistance                            | Lower strength, wear debris, limited osteoconductivity                        | [28, 29]     |
| UHMWPE                                    | 0.8–1.6               | Excellent wear, low friction, high fatigue resistance, biocompatible                                       | Limited strength for load-bearing, oxidation risk                             | [30]         |
| Zirconia (ZrO <sub>2</sub> )              | 200–210               | Aesthetic, biocompatible, strong, metal-free, low plaque retention                                         | Brittle, limited long-term data, hard to process                              | [31, 32]     |
| Alumina (Al <sub>2</sub> O <sub>3</sub> ) | 380–420               | Extremely hard, wear-resistant, chemically stable, bioinert                                                | Brittle, low fracture toughness, processing difficulty                        | [32, 33]     |
| Bioglass                                  | 30–35                 | Bonds to bone, promotes growth, high bioactivity, stimulates osteogenesis                                  | Low strength, non-load bearing, low fracture resistance                       | [31, 34, 35] |

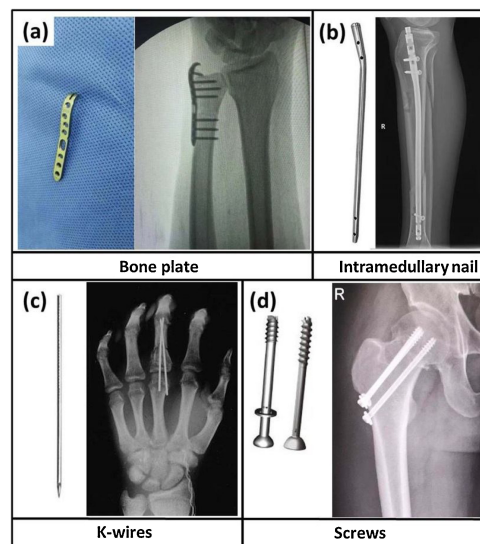


Figure 2.3: Common medical devices used for bone fracture fixation. (a) Bone plate used for the fixation of ulnar fracture ; (b) Intramedullary nail used for the fixation of tibial fracture; (c) K-wire used for the fixation of phalangeal fracture; (d) Screws used for the fixation of femoral neck fracture [36].



### 2.1.4 Biodegradable Materials for Bone Implants

Biodegradable materials used for bone implants include metals, polymers, ceramics, and extracellular matrix (ECM)-based materials. Synthetic polymers like PCL, PLGA, and PLA offer adjustable degradation rates and mechanical behavior, while natural polymers such as collagen, chitosan, and gelatin provide biocompatibility and ECM mimicry. Bioceramics like HA,  $\beta$ -tricalcium phosphate ( $\beta$ -TCP), and bioactive glasses are osteoconductive and support bone bonding. Composite scaffolds, such as polymer-ceramic hybrids and nanocomposites, enhance both strength and bioactivity. ECM-based materials like decellularized bone and ECM-derived hydrogels offer natural templates for bone growth. Smart biomaterials, including piezoelectric scaffolds and ion-releasing glasses, add functional stimulation for healing [37]. Table 2.4 lists common materials used for biodegradable implants and their properties.

A new generation of biodegradable metals capable of *in vivo* degradation has emerged. These materials allow for the development of temporary implants that can elicit a compatible host response and undergo complete dissolution within the body. The most extensively studied biodegradable metals for temporary implant applications are magnesium (Mg), zinc (Zn), and iron (Fe), due to their biocompatibility, tunable degradation behavior, and mechanical characteristics that can support bone regeneration. Magnesium exhibits mechanical properties that can minimize stress shielding but tends to degrade rapidly, leading to non-uniform corrosion and hydrogen gas accumulation. Zinc displays a corrosion rate that falls between that of Mg and Fe, but its mechanical behavior may not meet the structural demands of load-bearing applications [7, 21].

#### 2.1.4.1 Pure Iron Implants

Compact iron, despite its mechanical strength and biocompatibility, is generally unsuitable for load-bearing implants due to its high stiffness. With a Young's modulus of approximately 200 GPa, it significantly exceeds that of native bone, increasing the risk of stress shielding, a condition that disrupts the natural healing process of bone and can lead to adverse physiological responses or the need for revision surgeries [7]. In addition to its mechanical mismatch, iron also degrades much more slowly than other biodegradable metals such as magnesium and zinc. This slow degradation may not align with the timeline required for effective bone regeneration, further limiting its suitability for temporary orthopedic implants. Table 2.3 presents the approximate *in vivo* degradation rates of pure Fe, Zn, and Mg.

Table 2.3: Approximate degradation rates of pure Fe, Zn, and Mg. Rate values vary with sample form and conditions [38–41].

| Metal          | In Vivo Degradation Rate (mm/year) |
|----------------|------------------------------------|
| Magnesium (Mg) | 0.2 – 1.0                          |
| Zinc (Zn)      | 0.05 – 0.2                         |
| Iron (Fe)      | 0.01 – 0.1                         |

To address these challenges, porous iron structures produced via AM have emerged as a promising solution. By introducing controlled porosity, the effective stiffness of iron can be reduced to better match that of both cortical and trabecular bone, thereby minimizing the risk of stress shielding. For example, porous iron with porosity levels between 45.6% and 86.9% can exhibit compressive Young's modulus values ranging from

218 MPa to 845 MPa, aligning more closely with the mechanical behavior of trabecular bone. Moreover, these porous architectures enable enhanced control over the degradation rate of iron, allowing better synchronization with the bone healing process [7].

Table 2.4: Biodegradable Implant Materials: Young's modulus, advantages and disadvantages.

| Material Type                           | Young's Modulus (GPa) | Advantages                                                                                                                 | Disadvantages                                                                               | Author(s) |
|-----------------------------------------|-----------------------|----------------------------------------------------------------------------------------------------------------------------|---------------------------------------------------------------------------------------------|-----------|
| Mg and Mg Alloys                        | 40–45                 | Naturally present, promotes bone growth, avoids second surgery, bone-like modulus, stimulates osteoblasts                  | Rapid degradation, H <sub>2</sub> gas evolution, mechanical loss, risk of premature failure | [42–44]   |
| Zn and Zn Alloys                        | 90–110                | Stronger than Mg, controlled degradation, retains strength at body temp, promising biodegradable strength                  | Limited clinical data, toxicity at high levels, emerging technology                         | [44, 45]  |
| Fe and Fe Alloys                        | 200–210               | High strength, good for load-bearing, excellent mechanical integrity                                                       | Very slow degradation, iron buildup, mismatch with bone regrowth rate                       | [43, 46]  |
| PLA                                     | 1.2–3                 | Biocompatible, predictable degradation (12–24 months), decent mechanics, FDA-approved                                      | Hydrophobic, acidic byproducts, low-load use only, dimensional changes                      | [47–49]   |
| PGA                                     | 6–7                   | Fast degradation (2–4 months), good initial strength, biocompatible                                                        | Strength loss over time, acidic degradation, limited mechanical longevity                   | [47–49]   |
| PLGA                                    | 2–4                   | Tunable degradation (1–12 months), versatile, biocompatible                                                                | Acidic byproducts, inhomogeneous degradation, bulk erosion                                  | [47–49]   |
| PCL                                     | 0.4–0.6               | Long-lasting, great biocompatibility, viscoelastic, low inflammation                                                       | Very slow degradation (2–4 years), weak strength, not load-bearing                          | [42, 50]  |
| Hydroxyapatite (HA, Ceramic)            | 35–120                | Excellent biocompatibility, osteoconductive, bonds to bone, chemically similar to bone mineral, long clinical track record | Brittle, poor fatigue strength, non-load-bearing, slow/incomplete degradation               | [51]      |
| $\beta$ -Tricalcium Phosphate (Ceramic) | ~100                  | Osteoconductive, resorbable, chemically similar to bone mineral, encourages new bone growth                                | Brittle, low tensile strength, degradation may outpace or lag bone growth                   | [51]      |
| 45S5 Bioactive Glass                    | 50–80                 | Bonds directly to bone, bioactive ion release (Si, Ca, P), stimulates osteogenesis                                         | Brittle, fast dissolution, unsuitable for load-bearing use                                  | [34]      |

## 2.2 Design of Lattice Structures for Medical Implants

Lattice structures are classified by porosity (open or closed), order (regular or stochastic), and geometry (strut-based or Triply Periodic Minimal Surfaces, TPMS) [52, 53]. Open-porous structures have interconnected pores that allow cells, nutrients, and fluids to pass through, supporting bone regeneration, while closed-porous structures have isolated pores that block flow. Regular structures consist of repeating unit cells, while stochastic structures have random arrangements. Lattice structures are widely used in biomedical applications for their tunable mechanical properties and biocompatibility. Geometry is key to both mechanical and biological performance. The most studied geometries are strut-based lattices and TPMS, discussed next [52–55].

### 2.2.1 Strut-Based Lattices

Strut-based lattices are composed of beam-like elements and include geometries such as body-centered cubic (BCC), face-centered cubic (FCC), octet-truss, and diamond [52]. Beyond these conventional geometries, other strut-based unit cells have also been explored to assess mechanical suitability for biomedical applications. For instance, Nogueira et al. [54, 56] investigated less conventional lattice topologies like Cubic (C), Truncated Cubic (TC), Rhombicuboctahedron (RCO), Truncated Octahedron (TO) (Figure 2.4a), and Rhombitruncated Cuboctahedron (RTCO) (Figure 2.4b), due to their geometric compatibility with AM and potential to mimic bone mechanical behavior. Among these, TO and RTCO exhibited superior structural stability across a broad range of relative densities and Young's modulus values, highlighting their promise as candidates for bioresorbable implants.

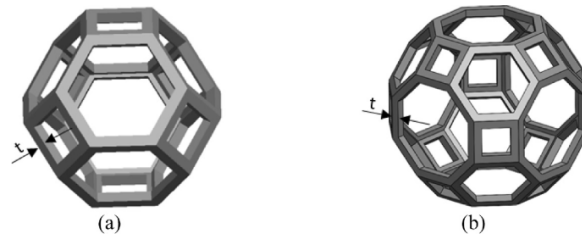


Figure 2.4: Graphical representation of the unit cells for (a) TO and (b) RTCO structures. The parameter  $t$  in the image represents the strut thickness [55].

### 2.2.2 Triply Periodic Minimal Surfaces

TPMS structures are defined by smooth, periodic surfaces with zero mean curvature and interconnected pores. They are generated mathematically and then thickened into printable geometries [57, 58]. TPMS lattices offer high surface-to-volume ratios and controlled porosity, which makes them particularly useful in bone tissue engineering. Their continuous surfaces can promote uniform cell proliferation and support biological functions such as oxygen diffusion and nutrient transport [59].

Several studies confirm that TPMS structures not only achieve sufficient mechanical strength but also better mimic the architecture of trabecular bone, making them promising candidates for bioresorbable implants [58].

### 2.2.3 Functionally Graded Structures

Functionally graded materials (FGMs), such as bone, exhibit a density gradient from dense cortical to porous trabecular bone, enhancing implant integration and functionality. Graded cellular lattices, which feature gradual changes in porosity and strut thickness, replicate bone anatomy through "dense-in" or "dense-out" profiles, as shown in Figure 2.5, optimizing bone ingrowth and structural integrity. Studies by Nogueira et al. [56] highlight that grading improves biodegradation, fluid flow, and stress transfer.

Computational studies by Nogueira et al. [55] demonstrated that graded lattice structures, like TO and RTCO, effectively mimic the mechanical properties of bone while maintaining a lower density. These lattices were designed to replicate the trabecular, intermediate, and cortical regions of bone, with varying densities and corresponding Young's moduli. Despite similar overall densities (around 0.25), the graded lattices performed comparably to non-graded specimens with higher densities, providing equivalent mechanical strength with reduced material. This advantage is particularly beneficial for temporary implants that gradually resorb during the healing process.

In conclusion, porosity control and functional gradation in lattice structures enable the development of advanced implants that meet both mechanical and biological requirements. Among the explored designs, TO and RTCO geometries stand out for their mechanical stability, adaptability across density gradients, and potential to mimic the hierarchical structure of human bone.

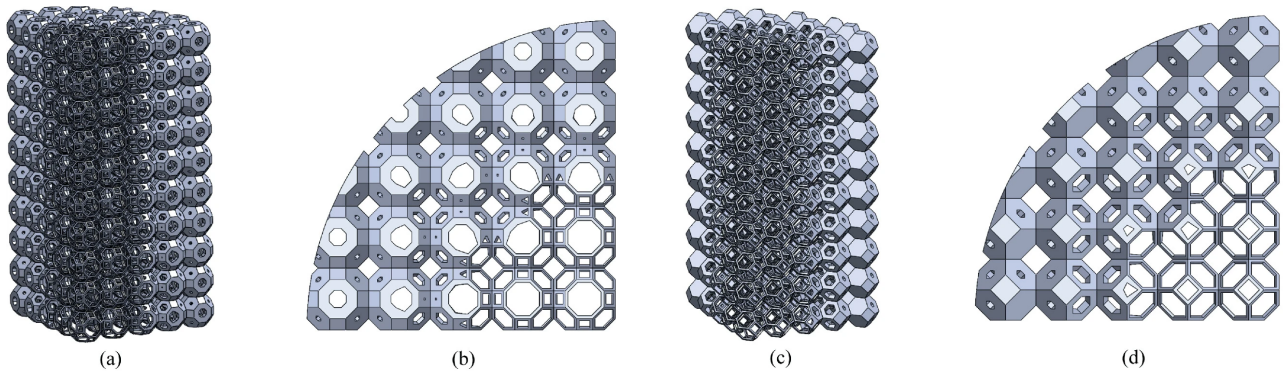


Figure 2.5: CAD of graded lattices: (a) RTCO dense-out, (b) RTCO dense-out (top view), (c) TO dense-out, and (d) TO dense-out (top view) [55].

## 2.3 Additive Manufacturing

Additive Manufacturing (AM), often referred to as 3D printing, is defined by ISO/ASTM 52900 as the "process of joining materials to make parts from 3D model data, usually layer upon layer, as opposed to subtractive manufacturing and formative manufacturing technologies" [60].

One of the fundamental principles of AM is the layer-by-layer addition of material, where each layer represents a thin cross-section of the final part derived from CAD data. Since each layer has a finite thickness, the final part is an approximation of the original design, with finer layers leading to higher accuracy. AM technologies differ in the materials used, layer formation methods, and bonding techniques, which influence

the accuracy, material properties, mechanical characteristics, production speed, post-processing requirements, machine size, and overall cost [61].

AM presents numerous advantages, including unprecedented design freedom that allows for complex geometries and internal structures, the ability to consolidate multiple components into single parts, improved part properties through controlled material distribution, mass customization, rapid prototyping, material efficiency, and benefits to inventory and supply chain management. However, AM also faces limitations, such as slower production speeds, material constraints (narrower range and higher costs), quality consistency and surface finish issues necessitating post-processing, significant capital investment and operational costs, potential environmental limitations in high-volume scenarios due to energy-intensive processes, health and safety considerations related to powder-based processes, and challenges in dimensional control. The economic viability of AM is context-dependent, influenced by production volume, part complexity, and material selection, with AM generally proving more cost-effective for lower production volumes and complex parts. AM involves many steps that transition from the virtual CAD description to the physical resultant part, with most AM processes involving, to some degree at least, the eight steps illustrated in Figure 2.6 [61–65].

AM is revolutionizing industries like aerospace, automotive, construction, retail, and especially the medical field. In healthcare, AM enables the production of patient-specific implants, medical devices, and models for surgical planning. It is used for customized implants (orthopedic, dental, cranial), medical aids (guides, splints, prostheses), and precision tools for medical devices. Additionally, AM has advanced biomanufacturing, particularly in tissue engineering and implant fabrication for regenerative medicine, which will be addressed next [66, 67].

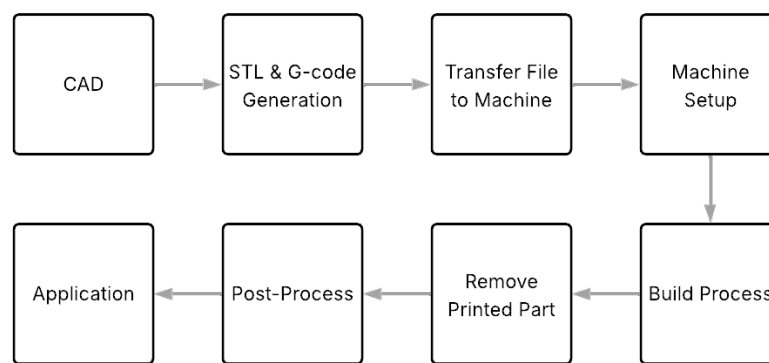


Figure 2.6: CAD to part steps of AM, showing all eight stages. Adapted from [61].

### 2.3.1 Additive Manufacturing for Biodegradable Bone Implants

There are many possible manufacturing techniques to achieve porous metal structures, and each one of them affects the microstructure and mechanical properties of the structure in its way. Next, both conventional and AM methods will be addressed [7].

Various processes can be used to fabricate biodegradable metal porous structures. Among these are direct foaming, spray foaming, chemical vapor deposition, electrophoretic deposition, powder metallurgy, and

metal injection molding. Although these processes control some porosity specifications, they are not capable of controlling the pores' size and organization, resulting in randomly organized porous structures. Another limitation of these processes is their inability to produce parts with complex geometry, which may be needed in the customization of bone implants to respond to the patient's requirements [3, 7, 68].

Besides that, the cell growth into a scaffold depends on how well nutrients can permeate the structure during the cell culture process, and that can only be achieved with the right pore size distribution, high pore interconnectivity, and sufficient porosity [59]. AM has brought about a revolutionary change in medical implant design and production. These technologies make it possible to produce customized implants to the patient's needs, with controlled pore size, interconnectivity, shape, and geometry [7]. Lattice structures produced by AM outperform those with the same porosity but produced by conventional methods, due to the greater geometric control and predictability [52]. Among the AM technologies, Selective Laser Melting (SLM) and Electron Beam Melting (EBM) are the most widely used in metal implant production [7].

### 2.3.2 Laser Powder Bed Fusion

The standard ISO/ASTM 52900 categorizes Powder Bed Fusion (PBF) processes into two types: Laser Beam PBF (L-PBF) and Electron Beam PBF (E-PBF) [60]. The first designation encompasses industrial terms like Selective Laser Melting (SLM) and Selective Laser Sintering (SLS), while the second designation includes mostly Electron Beam Melting (EBM) [69]. Despite variations in conditions, process capability, and part characteristics, all PBF processes share the same working principle [70].

As described in Figure 2.7, the material, in powder form, is spread on the build plate by a recoater. Then, an energy beam scans the powder layer in selected regions that define one of the part slices. When the layer is completed, the build platform drops down, and another layer of powder is added. The cycle repeats itself, layer by layer, until the component is completely built [70]. The process occurs inside the built chamber, under the protection of an inert gas, generally argon or nitrogen, to prevent oxidation [71]. In the case of the L-PBF process, one of the most utilized AM technologies, the energy beam is a laser.

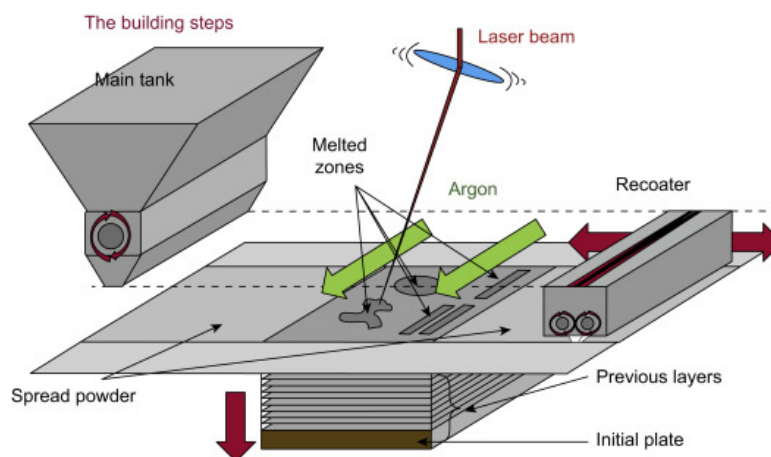


Figure 2.7: Schematic illustration of the laser-based powder bed fusion process [71].



## 2.4 Process Parameters in L-PBF

The optimization and selection of appropriate process parameters are essential to obtain parts with the desired final quality [69, 72]. Next, the main parameters of the L-PBF process, illustrated in Figure 2.8, will be reviewed along with their impact on the process and the resulting part properties.

**Laser power** main function is melting the powder. It can be changed depending on the material's melting point. Under the same conditions, the laser power directly influences the energy density, having an impact on the appearance of defects [73, 74].

**Spot size** corresponds to the laser beam diameter and influences the energy density. A smaller spot size allows for finer resolution and is typically used for detailed features or contour scanning, where precise edge definition is required. Conversely, a larger spot size can be used for bulk infill due to its higher coverage area, contributing to faster build rates but potentially lower resolution [75].

The **scanning strategy** is the moving pattern of the energy beam and affects heat transfer and powder melting and solidification, playing an important role in the microstructure, defect location and distribution, mechanical properties, and residual stresses of 3D printed parts. The uni-directional, bi-directional (zigzag scan), and cross-hatching are some commonly used scanning strategies [72, 76].

The **scanning speed** has a significant influence on the porosity of the parts. If it is low, the relative density is higher, but if it is high, the number and size of the pores increase, decreasing the relative density. In extreme cases, if it is too high, the laser may not have enough time to fuse the powder, leading to a lack of fusion defects and a lot of porosity [74].

The **hatch space** is defined as the distance separating two adjacent scan lines. If the hatch space is larger than the width of the melting pool, overlapping between layers is not achieved, causing some powder between them not to be fused, causing lack of fusion defects, and causing a wavy surface. If the hatch space is smaller than the width of the melt pool, accumulation will occur, and the surface morphology will be uneven. In general, a hatch space equal to or slightly less than half the width of the melt pool achieves the best results in terms of surface smoothing [74].

The **layer thickness** is defined as the height of the powder layer that is spread on top of the previously solidified material. Also, the lower the layer thickness, the higher the density and the better is dimensional accuracy of the part. However, a smaller layer thickness increases manufacturing time. If the layer thickness is too thick, the powder may not be fully melted, leading to poor bonding between layers and other defects [74, 77]. A homogeneous powder layer thickness across the platform will improve the quality of the final part [71].

**Protective gases** such as argon, helium, and nitrogen are essential in the L-PBF process to prevent metal oxidation and undesirable reactions. The presence of oxygen in the atmosphere during L-PBF, particularly when processing metals, can lead to oxide formation and balling, which negatively affect the final part quality. Therefore, maintaining a controlled, inert atmosphere is critical to ensure process stability and optimal material properties [73].

**Pre-heating** the powder bed helps reduce thermal gradients, which in turn lowers residual stresses and limits thermal distortion in the final part. In modern L-PBF systems, pre-heating is usually done by warming the build chamber, heating the base plate, or using multiple lasers. Typical pre-heating temperatures range from 200°C to 500°C [75].

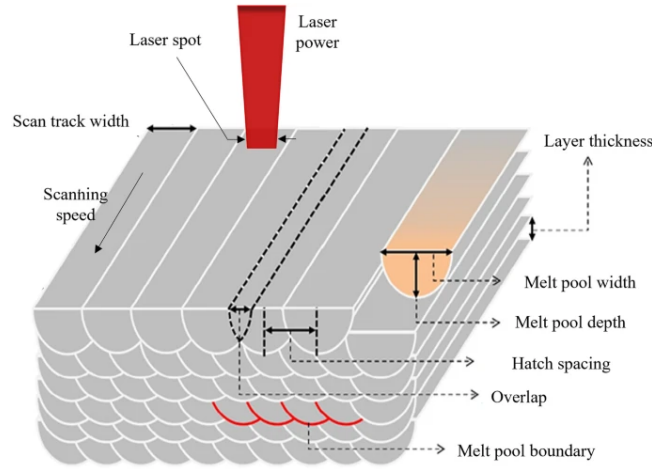


Figure 2.8: Schematic diagram of the main processing parameters of the L-PBF process [78].

### 2.4.1 Equations and Parameter Interdependence

Laser energy input influences the melt condition of metal powders and the flow of molten metal, and therefore significantly impacts the type and size of production defects. The energy input is directly related to process parameters such as laser power, scanning speed, hatch spacing, and layer thickness [76]. A set of variables defined by the processing parameters can be used to characterize the process [71].

Volumetric energy density or just energy density (VED) ( $\text{J}/\text{mm}^3$ ) [71, 76]:

$$E_v = \frac{P}{v \times h \times t} \quad (2.1)$$

where  $P$  is laser power (W),  $v$  is scanning speed (mm/s),  $h$  is hatching space (mm), and  $t$  is layer thickness (mm) [71]. The use of this equation is essential for optimizing parameters in the L-PBF process, helping to select the most appropriate values for laser power, scanning speed, hatch spacing, and layer thickness, which are the parameters with the greatest influence on the appearance of defects [76].

Linear input energy density ( $E_l$ ) ( $\text{J}/\text{mm}^3$ ) [71]:

$$E_l = \frac{4P}{\pi v \times d^2} \quad (2.2)$$

where  $d$  (mm) is laser spot size.

### 2.4.2 Bulk and Contour Parameters

In L-PBF, process parameters, already described in the previous section, are usually divided into bulk and contour types, each with a specific role. Using separate parameter sets for the bulk and contours allows for achieving both strong internal structures and precise outer surfaces. Bulk parameters are used for building the inner part of the component. These settings are chosen to ensure the material melts properly, remains dense, and has as few internal defects as possible. The goal is to achieve strong and consistent results throughout the bulk of the part [79].



Contour parameters, on the other hand, are used for the outer edges and surfaces of the part. These are often adjusted separately to improve surface quality and dimensional accuracy. These parameters, especially offset value, laser power, scanning speed, and spot size, directly affect mechanical properties, surface roughness, defects, and dimensional accuracy [79]. A schematic representation of contour scanning and inner/bulk surface scan is represented in Figure 2.9.

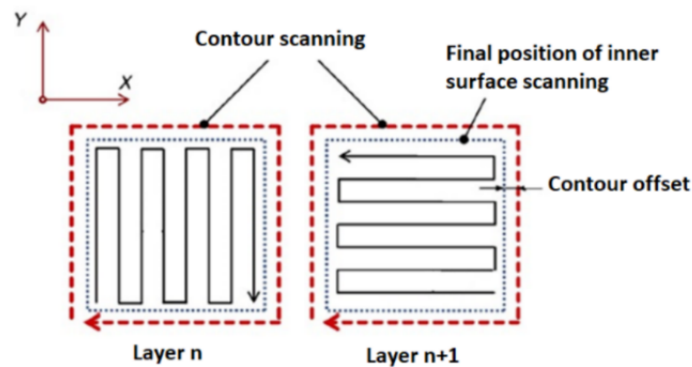


Figure 2.9: Schematic representation of contour scanning and inner surface scan [80].

While most research has focused on bulk parameters, Baykal et al. [80] investigated the effects of these contour parameters on AlSi10Mg alloy. The study found that an optimal parameter set (0.140 mm offset, 200 W laser power, and 280 mm/s scanning speed) resulted in improved tensile and yield strengths, minimized surface roughness ( $R_a = 2.011 \mu\text{m}$ ), and high dimensional accuracy. Increasing the offset value or scanning speed led to a reduction in mechanical properties due to lower energy density and poor fusion quality. Among all parameters, offset had the most significant influence on all quality metrics except surface roughness, which was more sensitive to laser power. The study concluded that proper optimization of contour settings is essential for reducing defects such as porosity and residual stresses. Ren et al. [81] highlighted that the sequence of contour scanning (pre- vs. post-contour) can also affect surface roughness due to differences in laser absorption and melt pool behavior. Their results support the importance of scan strategy in contour quality, but they did not explore mechanical or dimensional outcomes in detail. These results underline the critical role of contour parameters in achieving consistent and high-quality L-PBF components, particularly in applications requiring tight dimensional tolerances and superior mechanical strength.

It is worth noting that no studies in the existing literature have addressed the optimization of contour parameters for pure iron, making the results of this work particularly relevant for advancing knowledge in this area.

## 2.5 Metallic Powders

Metallic powders serve as the primary feedstock for PBF and other powder-based additive manufacturing (AM) processes. Their physical, chemical, and morphological characteristics critically influence all stages of the process, from powder handling and layer deposition to melting behavior and final part performance. Proper understanding and control of these properties are essential for ensuring consistent process reliability, mechanical

integrity, and part quality in laser powder bed fusion (L-PBF) applications. The subsections below examine key aspects of metallic powder behavior, properties and production methods [82, 83].

### 2.5.1 Powder Flowability and Test Methods

Flowability is not an inherent property of the powder, but rather a property that describes the complex behavior of the powder when it is mobilized or subjected to stress, and plays a fundamental role in the homogeneity of the powder layer thickness. This property depends on the testing device used, so it is necessary to specify the conditions under which it was determined. As small variations in flowability can significantly influence process performance, flowability tests must be carried out to verify the quality of the powder. There are various flowability tests, the most commonly used being the Hall flowmeter funnel (ASTM B213) and the Carney funnel (ASTM B964), described in the ASTM F3049-14 standard [82].

Bulk and tapped density are fundamental properties used to assess the packing behavior and flow characteristics of powders. Bulk density ( $\rho_B$ ) refers to the mass of a powder divided by the volume it occupies under untapped, free-settled conditions. In contrast, tapped density ( $\rho_T$ ) is the density measured after the powder has been compacted by tapping or vibration under specified conditions. This process causes particle rearrangement, reduces interparticle friction, and results in a denser packing of the material. These measurements are governed by several standardized methods. For tapped density, the relevant standard is ISO 3953:2011 (Determination of Tap Density of Metal Powders). Apparent (bulk) density, on the other hand, is typically measured using either ASTM B212-21 (Method for Free-Flowing Metal Powders Using the Hall Apparatus) or ASTM B417-22 (Method for Non-Free-Flowing Metal Powders Using the Carney Apparatus) [83].

The flowability of powders can also be assessed using the Hausner Ratio (HR), which is a dimensionless number defined as the ratio of tapped density to bulk density [83]:

$$HR = \frac{\rho_T}{\rho_B} \quad (2.3)$$

where  $\rho_T$  is the tapped density and  $\rho_B$  is the bulk density. A Hausner Ratio close to 1 typically indicates good flowability, while higher values suggest increasing cohesiveness and reduced flow performance [83].

A related metric is the Carr Compressibility Index (CI), which quantifies the same bulk powder behavior but expresses it as a percentage difference between the tapped and bulk densities [83]:

$$CI = 100 \times \frac{\rho_T - \rho_B}{\rho_T} \quad (2.4)$$

Both the Hausner Ratio and the Carr Index are specified in ASTM D6393-14, which provides standard procedures for evaluating powder characteristics such as cohesion, angle of repose, and powder dispersibility [83].

The flowability of the powder is a complex phenomenon that is very relevant to the success and reproducibility of an L-PBF process and is influenced by powder properties like particle size distribution and morphology, which will be addressed in the next section.

### 2.5.2 Powder Properties

Powder packing significantly influences part quality in LPBF by affecting defect formation, surface finish, dimensional accuracy, and mechanical properties. A densely packed powder layer promotes uniform melting and reduces the likelihood of voids, such as gas and lack-of-fusion pores, which can compromise part integrity. High powder packing also results in smoother layer deposition, leading to better surface quality and more accurate geometry. In contrast, low-density powder beds can produce irregular melt tracks, surface roughness, and dimensional deviations due to uneven heat distribution and poor particle bonding. Powder packing is influenced by Particle Size Distribution (PSD) and particle morphology [82–85].

PSD refers to the range and frequency of particle sizes within a bulk powder and plays a critical role in determining both flowability and powder packing in the laser powder bed fusion (L-PBF) process. A wide and multimodal PSD, skewed toward finer particles, enables smaller particles to fill the voids between larger ones, resulting in higher powder packing, enhanced densification, and improved surface quality. In contrast, a narrower PSD typically promotes better flowability due to reduced interparticle interactions. Larger particles ( $60\text{--}75\text{ }\mu\text{m}$ ) often suffer from poor powder packing, while very coarse powders ( $>100\text{ }\mu\text{m}$ ) are generally unsuitable for L-PBF. Conversely, powders with a high fraction of fine particles ( $<10\text{ }\mu\text{m}$ ) exhibit strong cohesive forces, which significantly impair flowability. Pleass et al. [86] reported that powders in which 90% of particles are smaller than  $10\text{ }\mu\text{m}$  display excessive cohesion, rendering them unusable for L-PBF applications. A typical PSD range of approximately  $15\text{--}55\text{ }\mu\text{m}$  is preferred, as it avoids the extremes of fines or coarse particles that cause process issues. PSD can be characterized using techniques such as sieve analysis, Dynamic Light Scattering (DLS), and Laser Diffraction (LD) [82, 83, 87].

The impact of morphology on bulk powder behaviour and final part quality is complex and cannot be captured by simple relationships. For powders in the typical size range ( $\approx 15\text{--}55\text{ }\mu\text{m}$ ), smooth and spherical particles generally exhibit superior flowability compared to those with rough, sharp-edged, or irregular shapes. The presence of satellite particles, smaller particles adhered to the surfaces of larger ones, can further hinder flowability. In contrast, for finer powders ( $<10\text{ }\mu\text{m}$ ), where cohesive and adhesive forces dominate, rougher surfaces may improve flow behaviour. High sphericity is typically associated with better flowability, optimal packing density, better surface finish, and improved performance in the L-PBF process, particularly during layer deposition. Powder morphology is commonly assessed using SEM [82, 83, 86].

The chemical composition of the powder plays a vital role in influencing its melting behavior, the resulting microstructure, and the mechanical properties of components produced using L-PBF. Impurities introduced during powder manufacturing or subsequent handling are inevitably incorporated into the melt pool during processing. These contaminants can contribute to porosity formation and may persist in the final component as inclusions or weakly bonded interfaces. Such defects frequently act as stress concentrators, reducing fatigue resistance by promoting crack initiation. Moreover, the chemical composition affects the physical properties of the material, thereby influencing the interaction between the laser beam and the powder, which ultimately impacts the final characteristics of the produced part [82, 83].

True density (or intrinsic density) is the mass per unit volume excluding pores. It helps evaluate powder packing and part porosity by comparison with apparent density. A higher difference indicates more voids in the powder bed. Besides this, analyzing the true density also provides insight into the internal porosity of the powder. Gas pycnometry, often using helium, is a standard method for true density measurement [88].

In Figure 2.10, it can be seen how each powder property influences in-process performance and affects the properties and quality of the produced part [82].

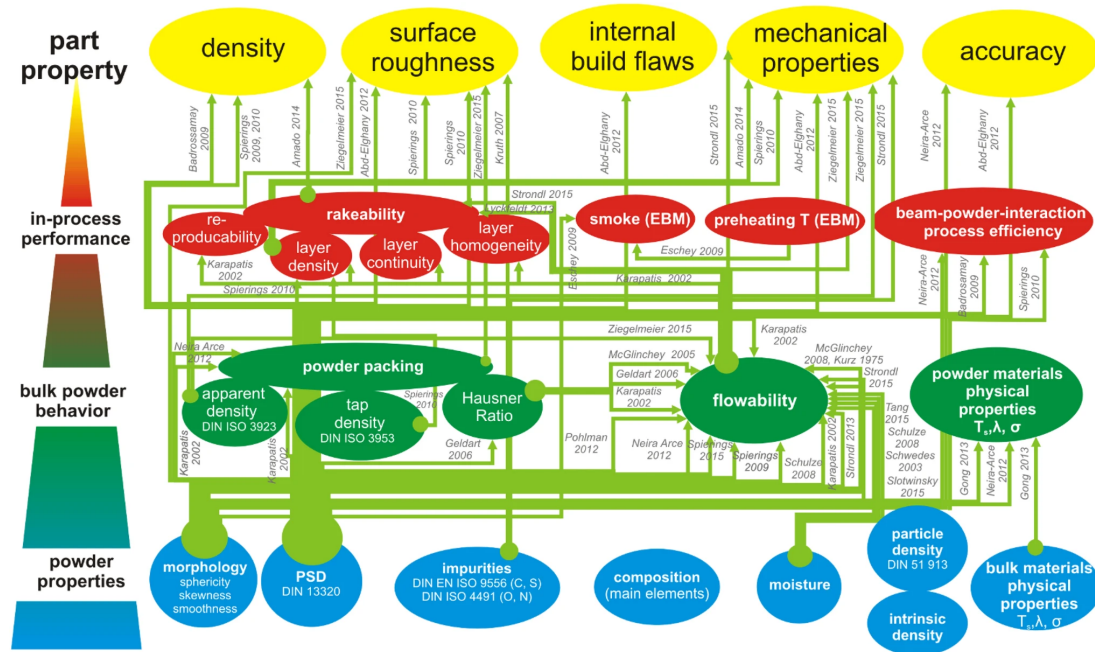


Figure 2.10: Visualization of the relationships between powder properties, bulk powder behavior, powder performance in the process, and finally the manufactured part quality as elaborated by different research groups [82].

### 2.5.3 Powder Production Methods

The choice of atomization process is critical in determining metal powder properties such as particle size, shape, purity, and flowability. Common methods include gas, water, centrifugal, and plasma atomization, each suited to specific materials and applications. Table 2.5 summarizes these techniques, comparing particle size ranges, morphologies, advantages, limitations, and typical uses. Figure 2.11 shows representative particle morphologies from gas and water atomization.

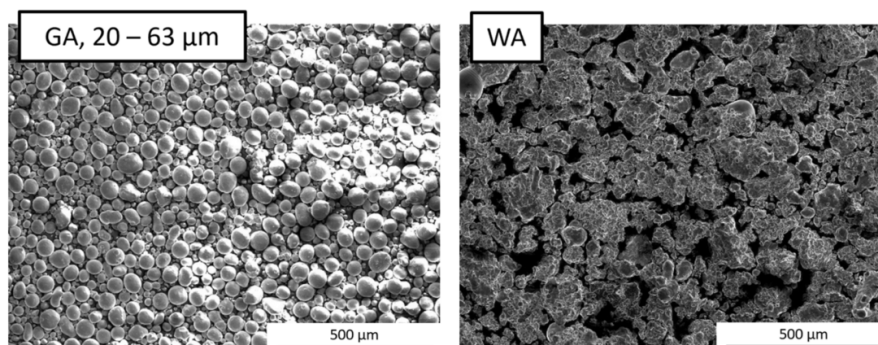


Figure 2.11: Morphology of GA and WA iron powders [89].

Table 2.5: Comparison of Metal Powder Atomization Methods [90–92].

| Method                  | Size Range (μm) | Particle Shape       | Advantages                                                                                                                                  | Limitations                                                                                                                | Applications                                         |
|-------------------------|-----------------|----------------------|---------------------------------------------------------------------------------------------------------------------------------------------|----------------------------------------------------------------------------------------------------------------------------|------------------------------------------------------|
| Gas Atomization         | 0–500           | Spherical            | High production rate; near-spherical morphology; controlled oxygen content; suitable for various alloys, including reactive alloys via EIGA | Higher cost than water atomization; inert gas use; low yield in 20–150 μm range; possible satellites and internal porosity | Ni, Co, Fe, Ti (EIGA), Al alloys                     |
| Water Atomization       | 0–500           | Irregular, dendritic | High production rate; fast cooling; broad feedstock compatibility; low cost; simpler equipment                                              | Requires moisture removal; irregular shapes; unsuitable for reactive alloys; hydrogen contamination; low 20–150 μm yield   | Non-reactive metals                                  |
| Plasma Atomization      | 0–200           | Highly spherical     | Highly spherical particles; high purity; minimal porosity                                                                                   | High cost; needs wire/pre-powder feed; moderate throughput                                                                 | Titanium alloys (mainly Ti-6Al-4V)                   |
| Centrifugal Atomization | 0–600           | Spherical            | Narrow PSDs across size ranges; controlled cooling rates                                                                                    | Expensive; fine powders need very high speed; low throughput                                                               | Zinc for batteries, solder pastes, Ti and steel shot |

#### 2.5.4 Economic and Quality Considerations in Powder Reuse

To ensure economic viability in L-PBF additive manufacturing, metallic powders are commonly reused. Sartin et al. [93] reported that only 6.7% of the powder is consumed per build, with 93.3% recoverable for reuse. Reusing powder up to 15 times can reduce material costs by up to 92%. However, repeated use exposes the powder to high temperatures, spatter, partial melting, and suboptimal atmospheres, causing changes in particle size, morphology, and chemical composition, as well as contamination from oxygen, moisture, and previous builds. Sieving systems are widely used during recycling to control PSD, remove oversized or fused particles, and maintain purity and flowability by eliminating contaminants and agglomerates. Although powder characterization standards exist, no standardized recycling methodology has been established, leading to variability in powder degradation across the industry. Some automated systems help reduce degradation by maintaining an inert atmosphere and minimizing manual handling, while manual recovery increases contamination and degradation risks. Overall, while powder reuse offers significant economic benefits, it must be carefully controlled to preserve material quality [71, 94].



## 2.6 Defects

The manufacturing quality can be evaluated by analyzing the porosity, dimensional accuracy, surface roughness, residual stress-induced distortion, cracking, and delamination of the manufactured parts [71, 74]. Next, the influence of processing parameters on the occurrence and severity of these defects will be discussed.

### 2.6.1 Porosity

Porosity plays a huge role in the final part quality, having an important influence on mechanical properties. Because of that, it's important to select the appropriate process parameters to minimize its occurrence. Table 2.6 presents the types of porosity defects in the L-PBF process and the parameter influence on their appearance. Figure 2.12 illustrates the different types of porosity commonly observed in L-PBF components.

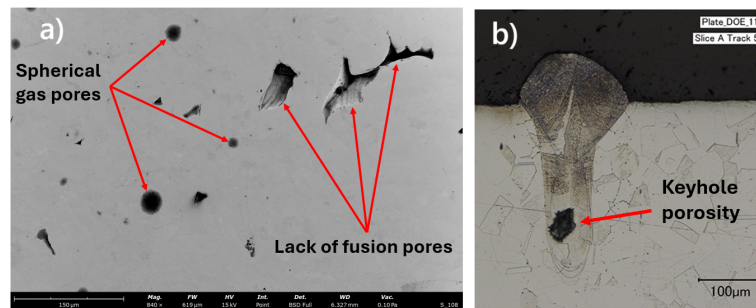


Figure 2.12: (a) Gas and lack of fusion porosity, and (b) keyhole porosity. Adapted from [75].

Table 2.6: Influence of process parameters on porosity defects in L-PBF.

| Process Parameter | Gas pores (spherical) | Keyhole pores | Lack-of-fusion porosity | Unstable melt pool porosity | Intra-layer porosity | References |
|-------------------|-----------------------|---------------|-------------------------|-----------------------------|----------------------|------------|
| Energy Density    | High                  | Excessive     | Low                     | High                        | —                    | [71, 76]   |
| Powder Quality    | Low                   | —             | —                       | —                           | —                    | [71]       |
| Packing Density   | Low                   | —             | Low                     | —                           | —                    | [76, 95]   |
| Hatch Spacing     | —                     | —             | —                       | —                           | Large                | [71]       |
| Layer Thickness   | —                     | —             | —                       | Large                       | —                    | [71]       |

*Note:* Powder quality includes morphology, PSD, gas entrapment, moisture, and composition.

### 2.6.2 Surface Roughness

Surface finish is one of the most important factors when analyzing the quality of parts produced by L-PBF and is caused by oxidation, adhesion of partially melted powders on the part surface, layered nature of the process, balling, and process parameters [75]. Additionally, contour parameters significantly influence surface quality and rugosity as discussed in section 2.4.2. The balling effect occurs when too high scanning speeds are used,

elongating the melt pool and turning it unstable, separating it into small spherical balls to maintain uniform capillary pressure inside the melt pool. These spherical balls accumulate on the edges of the deposit, causing roughness on the surface [75]. Figure 2.13 illustrates this balling phenomenon.

Alongside porosity, surface roughness has a strong influence on the fatigue life of AM-produced parts, due to the increase of stress concentration for higher roughness. The optimization of process parameters to optimize the surface roughness is a topic that continues to be researched [73, 96].

Surface roughness can be minimized by reducing balling, providing a high energy density, a lower layer thickness, and a suitable hatch spacing. The staircase effect, typical of AM processes, is a factor that also influences roughness. Reducing the layer thickness, although beneficial for the surface finish, increases the product build-up time. Although parameter optimization can improve the surface finish to a certain extent, the best way to reduce roughness is through conventional post-processing. Almost all parts produced by L-PBF require post-processing [73].

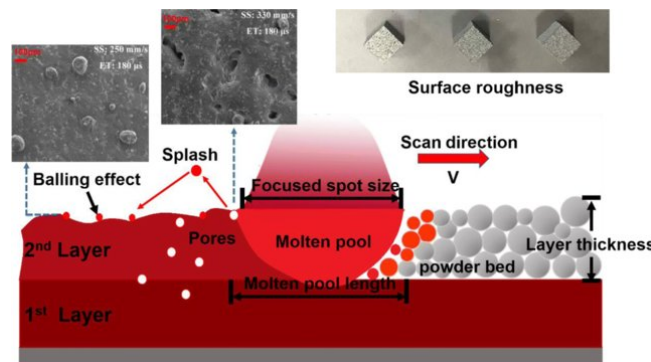


Figure 2.13: Balling effect [97].

### 2.6.3 Residual stress - Distortion, Cracking, and Delamination

In L-PBF, a high-power laser induces extremely high temperature gradients (often exceeding  $10^8$  K/s), which lead to the formation of residual stresses in the manufactured part [98]. As shown in Figure 2.14a, the temperature distribution through the part height can be divided into three regions: the melted zone (Region I), the heat-affected zone (Region II), and the unaffected zone (Region III), with respective temperature ranges from above the melting point ( $T_m$ ), between the ambient temperature ( $T_n$ ) and the plastic point ( $T_p$ ), and near  $T_n$  [99].

When the laser rapidly melts the powder within its beam spot, the underlying solidified material experiences thermal expansion. As the laser moves forward, the previously heated regions cool and shrink. This shrinkage is constrained by the surrounding material, leading to the buildup of residual stresses. Moreover, the previously deposited layers may undergo remelting and resolidification, causing compressive stresses to accumulate layer by layer, which helps balance the tensile stresses concentrated near the surface [98].

During the heating stage (Figure 2.14b, stage 1), Region I enters a fully molten, stress-free state. Region II heats up and expands but is restricted by Region III, leading to compressive stress in Region II and tensile stress in Region III. In the subsequent cooling stage (Figure 2.14b, stages 2 and 3), Region I solidifies and contracts. However, the contraction is constrained by Region II, which results in tensile stress in Region I and increasing compressive stress in Region II. As cooling continues, these compressive stresses extend into Region

III. The repetition of this thermal cycle during layer-by-layer fabrication is the primary mechanism behind the formation and accumulation of residual stresses in L-PBF parts [99].

Residual stresses can result in part distortion, cracking, and delamination of the part [71]. To solve these problems, pre-heating the substrate and improving ambient temperature are recommended [100].

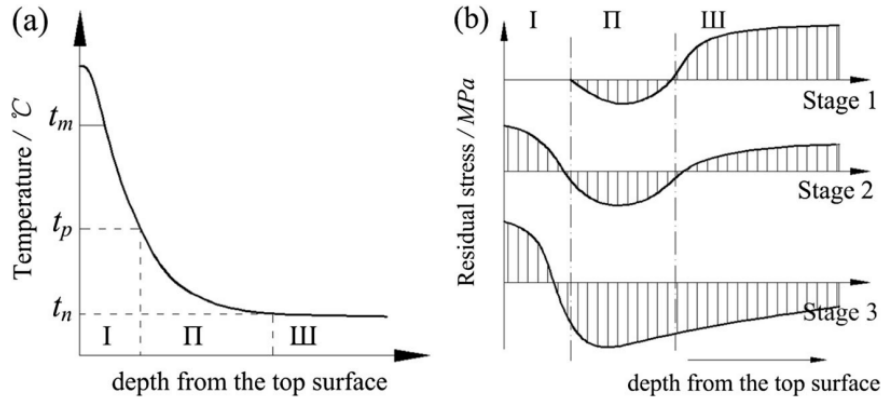


Figure 2.14: Temperature (a) and residual stress (b) distribution in height direction during L-PBF [99].

## 2.7 Additive Manufacturing of Pure Iron

Table 2.7 provides an overview of key studies on producing pure iron parts through AM. It summarizes the focus and results of each study. One of the earliest investigations was conducted by Song et al. [101], who demonstrated the feasibility of fabricating fully dense pure iron parts using a 100 W fiber laser. The process parameters included scanning speed, ranging from  $100 \text{ mm s}^{-1}$  to  $400 \text{ mm s}^{-1}$ , a hatch spacing of  $40 \mu\text{m}$ , and a layer thickness of  $50 \mu\text{m}$ . These settings facilitated the formation of very fine ferritic grains within the material. The resulting parts exhibited favorable mechanical properties, with a yield strength of 305 MPa and an ultimate tensile strength of 412 MPa, while maintaining a high density.

### 2.7.1 Typical Microstructure

The typical microstructure of alloys produced by L-PBF processes is strongly influenced by rapid solidification and the predominantly unidirectional heat flow during fabrication, resulting in columnar structures with pronounced crystallographic texture and non-equilibrium phases, as observed in alloys such as Ti6Al4V, nickel-based superalloys, and aluminum-silicon alloys. These features arise from epitaxial grain growth and directional solidification, producing elongated grains aligned with the build direction, while the high cooling rates, ranging between  $10^3$  and  $10^8 \text{ K/s}$ , contribute to a refined microstructure [71].

The production of pure iron by L-PBF has garnered significant attention due to its ability to generate unique microstructures that directly influence the material's mechanical and magnetic properties. Recent studies by Torrent et al. [89], Lejček et al. [107], and Zanni et al. [110] highlight the distinctive microstructural features of pure iron produced by L-PBF, often comparing them to those obtained through conventional methods such as hot rolling.



Table 2.7: Summary of recent studies on pure and porous iron processing using AM techniques.

| Technique                         | Focus                                                                          | Key results                                                                                                                           | Author(s)               |
|-----------------------------------|--------------------------------------------------------------------------------|---------------------------------------------------------------------------------------------------------------------------------------|-------------------------|
| SLM                               | Effect of scanning speed on density, microstructure, and mechanical properties | Grain size decreased with higher scan speed; UTS 412 MPa, YS 305 MPa; strength via grain refinement and work hardening.               | Bo Song et al. [101]    |
| SLM                               | Optimization for low porosity and high build speed                             | Optimal at 400 W & 750 mm/s; porosity 0.15%; UTS 449 MPa; ductile fracture mechanism.                                                 | Palousek et al. [102]   |
| DMP                               | Porous iron scaffolds for biomedical applications                              | Up to 12× faster degradation vs. cold-rolled iron; properties retained after 28 days; ISO 10993 biocompatible.                        | Li et al. [103]         |
| 3D printing + microwave sintering | Porous iron foams with various unit-cell structures                            | Porosity 45.6–86.9%; compressive modulus and plateau stress matching cancellous bone.                                                 | Sharma and Pandey [104] |
| SLM, LMD, Casting                 | Comparison of microstructure, mechanical, and corrosion properties             | SLM iron had finest grains and 50% higher corrosion rate vs. cast; superior strength and biodegradability.                            | Carluccio et al. [105]  |
| SLM                               | Functionally graded porous scaffolds                                           | Tailored permeability and biodegradation; maintained bone-like mechanical properties; 5–16% weight loss over 4 weeks.                 | Li et al. [106]         |
| SLM                               | Multiscale microstructural analysis                                            | Hierarchical grains via $\alpha \rightarrow \gamma \rightarrow \alpha$ transformations; nanocell/subgrain structures refine strength. | Lejček et al. [107]     |
| SLM                               | Fe–35Mn load-bearing scaffolds                                                 | Predominantly $\gamma$ -austenite; high ductility; accelerated corrosion; good in-vivo osteointegration.                              | Carluccio et al. [108]  |
| Extrusion 3D printing + sintering | Porous scaffolds for enhanced biodegradation                                   | Porosity 67%, 96% connectivity; 7% mass loss in 28 days; reduced cell viability due to high $\text{Fe}^{2+}$ release.                 | Putra et al. [109]      |
| Computational                     | Lattice geometry evaluation for implants                                       | TO and RTCO unit cells are stable across densities; mechanical properties mimic bone.                                                 | Nogueira et al. [54]    |
| Review                            | Iron and porous iron as biodegradable materials                                | AM routes yield bone-like mechanical properties and higher corrosion rates vs. conventional iron.                                     | Salama et al. [7]       |
| Computational                     | Graded lattice structures (TO, RTCO)                                           | Graded specimens match mechanical behavior at lower relative density; potential for stress-shielding reduction.                       | Nogueira et al. [55]    |
| Computational                     | Compression performance of lattice structures                                  | RTCO and TO cells mimic trabecular bone; graded structures reduce stress shielding.                                                   | Nogueira et al. [56]    |

The microstructure of pure iron manufactured via L-PBF is described by Lejček et al. [107] as hierarchical, consisting of fine equiaxed grains with a size of  $12 \pm 6 \mu\text{m}$  in the section parallel to the building direction and a size of  $9 \pm 4 \mu\text{m}$  in the section perpendicular to the building direction, meaning that the grains are only slightly elongated (25%) in the building direction. This contrasts with the columnar grain structures commonly observed in other metallic alloys processed by L-PBF. Complementary results by Carluccio et al. [111], Limón et al. [20], and Song et al. [101] further confirm that pure iron processed by L-PBF exhibits a predominantly equiaxed  $\alpha$ -ferrite grain structure, with only occasional remnants of elongated, columnar grains in the building direction. The formation of this equiaxed structure is largely attributed to the allotropic phase transformations ( $\alpha \leftrightarrow \gamma$ ) that occur during the repeated thermal cycles inherent to the process. Rapid heating and cooling promote recrystallization of initially formed columnar grains, resulting in a refined equiaxed morphology [89,

101, 105, 107, 110, 111]. Furthermore, the absence of alloying element segregation in pure iron prevents the development of dendritic or cellular structures typically seen in more complex alloys [110].

In Figure 2.15, the microstructure of pure iron produced via L-PBF is shown in a section cut parallel to the building direction. The arrow indicates the build direction, and the white dots mark the melt pool boundaries. The top image presents a optical microscopy (OM) view, while the bottom image shows a [001] Inverse Pole Figure (IPF) map of a selected area. A magnified detail on the right highlights grains labeled  $N_a-N_x$ , which form due to the fragmentation of columnar grains during repeated  $\alpha \leftrightarrow \gamma$  phase transformations, as reported by Lejček et al. [107].

At the nanoscale, subgrains delineated by low-angle grain boundaries (LAGBs) and dislocation walls are frequently observed, contributing to the material's hardening behavior [89]. This hierarchical microstructure is visualized in the GND density map shown in Figure 2.15, where high-angle grain boundaries appear in black, low-angle grain boundaries appear in white, corresponding to regions of increased dislocation density, and dislocation cells are indicated by green lines. A schematic overview of this hierarchical microstructure is also provided in Figure 2.15. These substructures form due to the high residual stresses and steep thermal gradients characteristic of the L-PBF process. Lejček et al. [107] also reported the presence of submicron-sized particles enriched with oxygen and trace elements such as Cr and Mn, which may act as pinning sites for dislocations and influence the mechanical strength.

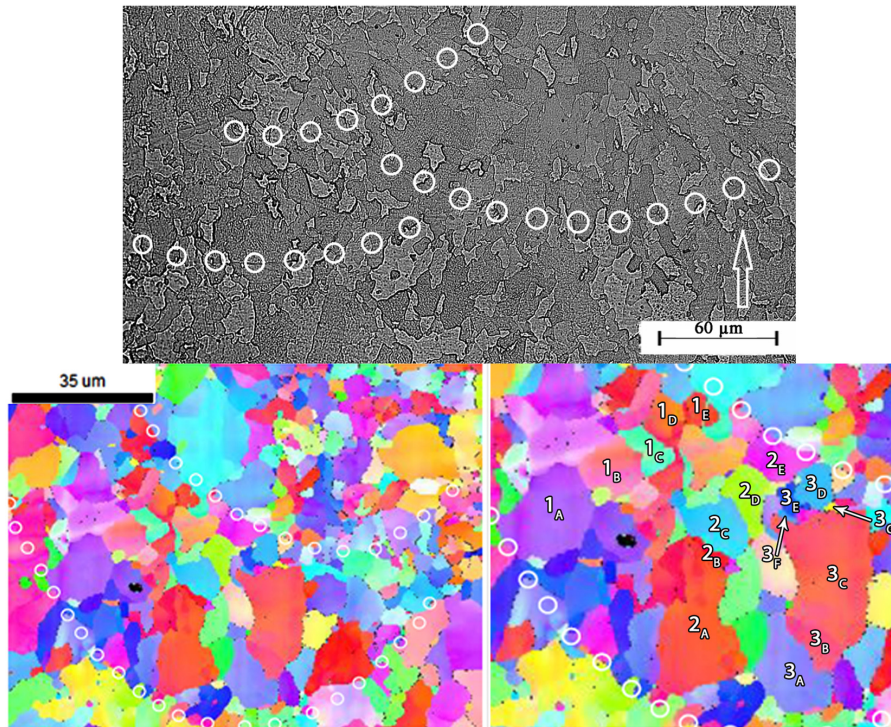


Figure 2.15: Microstructure of the cut parallel to the building direction. The arrow indicates the building direction, and white dots mark the melt pools. On the top: OM image. At the bottom: [001] IPF map of a selected area (left); its detail (right) with marked grains  $N_a-N_x$  which occur by breaking columnar grains during repeated  $\alpha \leftrightarrow \gamma$  phase transformations [107].

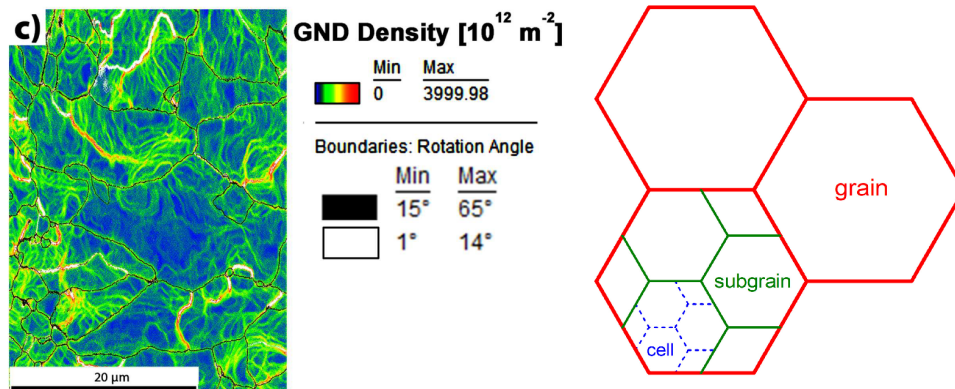


Figure 2.16: GND map of L-PBF produced pure iron microstructure (left) and schematic depiction of the microstructural hierarchy in the L-PBF produced pure iron (right) [107].

### 2.7.2 Mechanical Properties

AM enhances pure iron strength compared to casting [110]. L-PBF shows higher strength but lower elongation, while cast pure iron has higher ductility [112]. LMD/DED has intermediate properties [105]. Table 2.8 compares mechanical properties across processes.

Table 2.8: Mechanical properties of commercially pure iron produced by different processes (all data refer to tension unless otherwise stated).

| Process and Condition Orientation |                        | Yield Strength [MPa] | Ultimate Strength [MPa] | Young's Modulus [GPa] | Elongation [%] | Citation   |
|-----------------------------------|------------------------|----------------------|-------------------------|-----------------------|----------------|------------|
| L-PBF (transverse direction)      | As-built               | 359–554              | 433–599                 | 200                   | 12–23          | [105, 110] |
| L-PBF (build direction)           | As-built               | 356–455              | 435–500                 | 200                   | 10–18          | [105, 110] |
| L-PBF (build direction)           | As-built (compression) | 421                  | 760                     | 200                   | –              | [105]      |
| LMD (build direction)             | As-built (compression) | 242                  | 581                     | 203                   | –              | [105]      |
| Hot rolled                        | Annealed (650–950 °C)  | 186                  | 290                     | 207                   | 38             | [112]      |
| Hot rolled                        | Annealed (1200 °C)     | 138                  | 276                     | 207                   | 40             | [112]      |
| Cast                              | As-cast (compression)  | 157                  | 498                     | 203                   | –              | [105]      |

## Chapter 3

# Materials and Methods

This chapter presents the experimental procedures carried out in this study. An overview of the experimental workflow is illustrated in Figure 3.1.

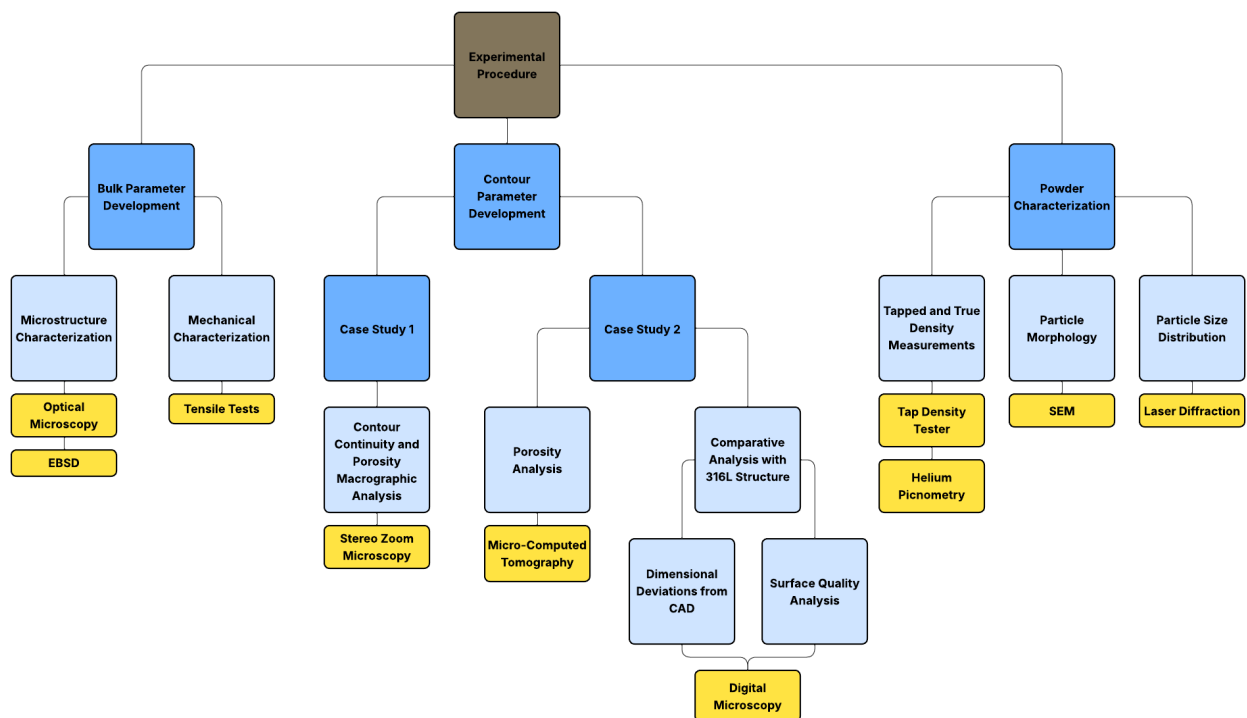


Figure 3.1: Overview of the experimental procedure.

### 3.1 Materials

#### 3.1.1 ATOMET FeAM Powder

The metallic powder used for the fabrication of all components in this study was a water-atomized iron powder (ATOMET FeAM), supplied by Rio Tinto (Canada). According to the manufacturer's specifications [113], this powder has an apparent density of  $3.10 \text{ g cm}^{-3}$  and consists primarily of iron (more than 99.5% by weight). The chemical composition and PSD are summarized in Table 3.1.

Table 3.1: Chemical composition and PSD of Atomet FeAM pure iron powder (manufacturer data) [113].

| Chemical composition (% w) |                  |
|----------------------------|------------------|
| Iron                       | >99.5            |
| Manganese                  | 0.04             |
| Carbon                     | 0.004            |
| Oxygen                     | 0.08             |
| Sulfur                     | 0.007            |
| Nitrogen                   | 0.004            |
| PSD                        |                  |
| $d_{10}$                   | 15 $\mu\text{m}$ |
| $d_{50}$                   | 30 $\mu\text{m}$ |
| $d_{90}$                   | 45 $\mu\text{m}$ |
| $d_{99}$                   | 60 $\mu\text{m}$ |

Further compositional data reported by Paloušek et al. [102] indicates the presence of additional alloying elements, including 0.07% nickel, 0.05% chromium, 0.03% copper, 0.006% tin, and 0.004% phosphorus by weight. In their study, slightly higher concentrations of oxygen (0.14%), sulfur (0.008%), nitrogen (0.007%), and carbon (0.004%) were also observed. The particle size in that analysis ranged from 10  $\mu\text{m}$  to 45  $\mu\text{m}$ . The powder morphology was notably heterogeneous, with particles exhibiting round, oval, and irregular shapes, typical of water-atomized powders [89, 102, 114]. Representative Scanning Electron Microscope (SEM) images provided by the supplier are shown in Figure 3.2.

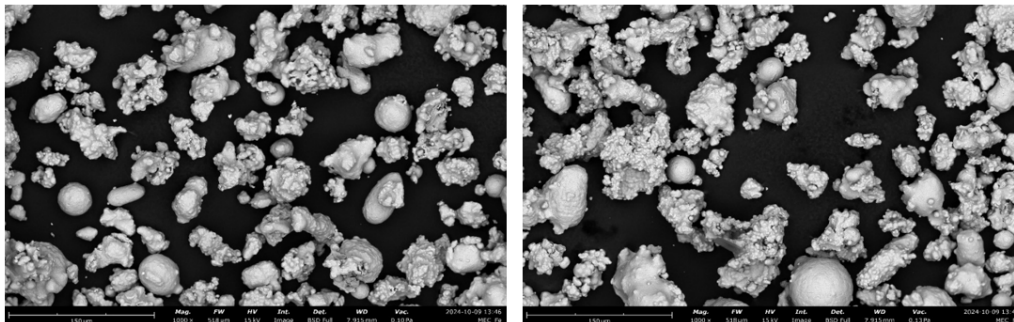


Figure 3.2: SEM images of the ATOMET FeAM powder.



### 3.1.2 ATOMET 195SP Powder

In addition to ATOMET FeAM, a second iron powder, also supplied by Rio Tinto, was included in this study for characterization purposes only. This material was not used in the printing process, but its properties were analyzed to assess its potential for future applications.

ATOMET 195SP is a high-purity iron powder and, according to the supplier's data sheet, it has an apparent density of  $3.0 \text{ g cm}^{-3}$ . The chemical composition and PSD are summarized in Table 3.2 [115]. Representative SEM images are shown in Figure 3.3.

Table 3.2: Chemical composition and PSD of ATOMET 195SP iron powder (manufacturer data) [115].

| <b>Chemical composition (% w)</b>  |       |
|------------------------------------|-------|
| Iron                               | >99.0 |
| Carbon                             | 0.005 |
| Oxygen                             | 0.08  |
| Sulfur                             | 0.006 |
| Phosphorus                         | 0.001 |
| <b>PSD (screen analysis)</b>       |       |
| >75 $\mu\text{m}$ (+200 mesh)      | Trace |
| 45-75 $\mu\text{m}$ (200–325 mesh) | 3     |
| <45 $\mu\text{m}$ (-325 mesh)      | 97    |

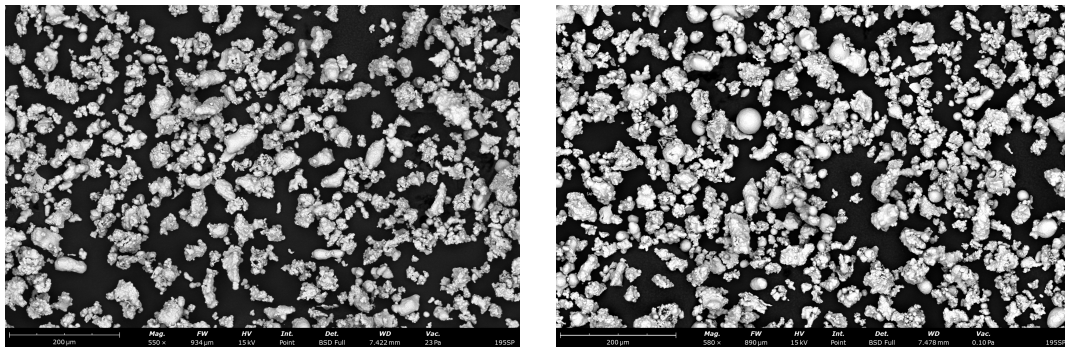


Figure 3.3: SEM images of the ATOMET 195SP powder.

The high purity and small particle size make ATOMET 195SP a potential candidate for future applications requiring fine iron powders with low contamination levels, although further characterization and evaluation under L-PBF conditions are necessary.

### 3.1.3 316L Stainless Steel Powder

Although the 316L stainless steel powder itself was not directly analyzed in this study, a cellular structure manufactured from this material was used as a reference for comparison with an equal structure produced from ATOMET FeAM powder.

The 316L powder used in this work was supplied by Colibrium Additive. It is an austenitic stainless steel composed primarily of iron, chromium, nickel, and molybdenum, with a PSD ranging from 15 to 45  $\mu\text{m}$ , making it suitable for L-PBF. Importantly, the processing parameters for this specific powder have already been developed and optimized by Colibrium Additive, enabling its reliable use in the production of high-quality components.

316L is well known for its excellent corrosion resistance, particularly in environments containing chlorides and other halides, due to the presence of molybdenum. Compared to the more common 304 stainless steel, 316L provides enhanced performance in chemically aggressive environments without significant cost penalties. It also exhibits good weldability, high ductility, and notable creep strength at elevated temperatures.

Typical application areas include chemical and plant engineering, the oil and gas industry, automotive components, medical technology, jewelry, and mold tooling.

The standard chemical composition of 316L powder, according to ASTM F3184 (UNS S31603) and ASTM A276, is provided in Table 3.3.

Table 3.3: 316L powder chemical composition according to ASTM F3184 UNS S31603 / ASTM A276.

| Chemical composition (% w) |              |
|----------------------------|--------------|
| Iron (Fe)                  | Balance      |
| Chromium (Cr)              | 16.0–18.0    |
| Nickel (Ni)                | 10.0–14.0    |
| Molybdenum (Mo)            | 2.0–3.0      |
| Manganese (Mn)             | $\leq 2.0$   |
| Silicon (Si)               | $\leq 1.0$   |
| Carbon (C)                 | $\leq 0.03$  |
| Phosphorus (P)             | $\leq 0.045$ |
| Sulfur (S)                 | $\leq 0.03$  |

The cellular structure produced using the 316L powder from Colibrium Additive, under previously developed and validated processing conditions, served as a benchmark to evaluate the surface finish and dimensional deviations of the corresponding structure fabricated from ATOMET FeAM powder.

### 3.2 L-PBF Equipment

All specimens in this study were fabricated using a Concept Laser M2 Series 5 L-PBF system at INEGI, manufactured by Colibrium Additive (Ohio, United States), as shown in Figure 3.4a. The manufacturing process was carried out in a controlled nitrogen atmosphere to minimize oxidation and maintain material integrity during printing.

Before each build, the feedstock powder was conditioned using a dedicated powder sieving unit (Figure 3.4b), also provided by Colibrium Additive. This pre-processing step was essential to ensure the consistency and quality of the powder by removing contaminants, breaking up agglomerates, and achieving a more uniform PSD. Such preparation contributes to improved dimensional accuracy and overall process stability in L-PBF manufacturing.

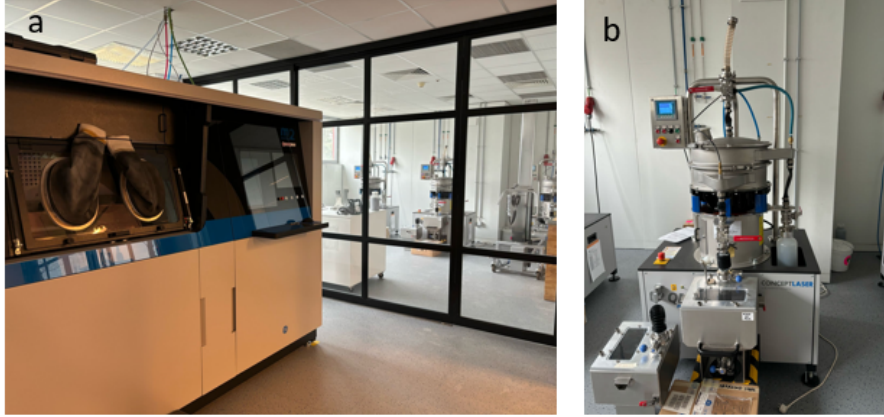


Figure 3.4: a) Concept Laser M2 Series 5; b) Powder sieving system.

### 3.3 Bulk Parameter Development - Previous Study

As part of a previous study, an intricate geometry was designed using SolidWorks (Vélizy-Villacoublay, France), incorporating  $45^\circ$  overhangs and a horizontal hole. This configuration aimed to evaluate how different parameter settings manage critical regions. The specific geometry and its dimensions are illustrated in Figure 3.5a. The choice of a  $45^\circ$  overhang reflects the geometric characteristics of the RTCO lattice structure, which has garnered attention due to its reliable mechanical properties and structural stability, irrespective of the applied relative density, as discussed in subsection 2.2.1. A horizontal hole was deliberately incorporated to test the printer's capability in fabricating unsupported features and to examine the likelihood of powder entrapment.

As illustrated by the workflow shown in Figure 3.5, samples were fabricated using the L-PBF process to perform hardness testing, porosity analysis, and dimensional measurements of the fabricated holes. The process parameters included a constant layer thickness of  $50\ \mu\text{m}$  and a laser power of 300 W. Laser spot size, scanning speed, and hatch spacing were systematically varied, as detailed in Table 3.4. For each combination of scanning speed and spot size, the hatch spacing was initially calculated to achieve a target peak VED of  $75\ \text{J/mm}^3$ . Subsequently, the hatch spacing was decreased in 0.01 mm increments to generate six hatch spacing values for each scanning speed and spot size combination. The data was analysed using contour plotting of VED and energy intensity ( $E_{\text{int}}$ ).

Following fabrication, the samples were sectioned along the Oyz plane using a FANUC EDM sys-115 system (Japan). The cross-sections were then embedded in thermoset resin and mechanically polished using 300 and 800 grit SiC abrasive papers (Streuss Roboplot-21, Denmark). Hole heights were determined with a digital caliper (Limit Tools, 150 mm,  $\pm 0.01\ \text{mm}$  accuracy). Porosity analysis was conducted using an Olympus SZ-ET microscope (Japan), and images were processed with ImageJ software (NIH, USA). Hardness measurements were carried out on a Wolpert Dia Testor 2 RC (Germany) with a 5 kg load, and results were averaged from three separate measurements per sample. The overall procedure is illustrated in Figure 3.5.

$E_{\text{int}}$  was selected alongside VED to characterize the process, as its use for parameter development was recommended by Colibrium Additive, and it provides valuable insight into how laser power and spot size influence the concentration of energy on the powder bed surface. It is calculated as:



$$E_{\text{int}} = \frac{P}{A} = \frac{4P}{\pi d^2}$$

where:

- $P$  is the laser power (W),
- $d$  is the laser spot size ( $\mu\text{m}$ ),
- $A$  is the irradiated area on the powder bed surface.

$E_{\text{int}}$  reflects how energy is concentrated at the surface, influencing melt pool stability, particle fusion, and surface quality.

Table 3.4: Process parameters of the previous study.

| Parameter                         | Range / Value | Step |
|-----------------------------------|---------------|------|
| <b>Variable Parameters</b>        |               |      |
| Scanning Speed (mm/s)             | 500–1000      | 100  |
| Spot Size ( $\mu\text{m}$ )       | 110–170       | 20   |
| Hatch Spacing (mm) <sup>a</sup>   | 0.09–0.21     | 0.01 |
| <b>Constant Parameters</b>        |               |      |
| Laser Power (W)                   | 300           | –    |
| Layer Thickness ( $\mu\text{m}$ ) | 50            | –    |

<sup>a</sup> The hatch spacing values were derived per scanning speed and spot size combination, starting from a value corresponding to a target VED of  $75 \text{ J/mm}^3$  and then reduced in  $0.01 \text{ mm}$  steps to generate six values per set.

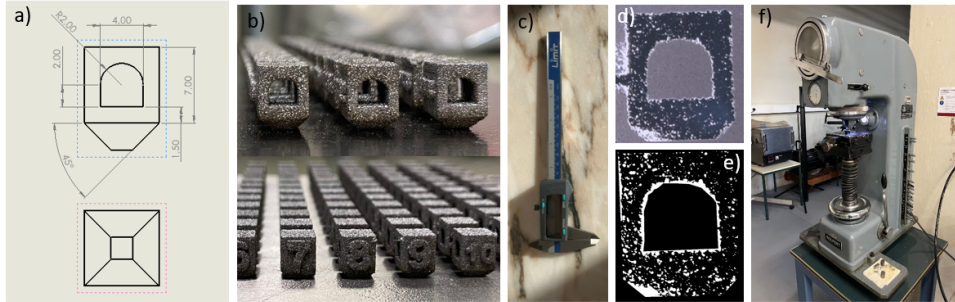


Figure 3.5: Workflow of previous study: a) modeled geometry (mm); b) printed specimens; c) digital caliper used to measure the hole height; d) stereo zoom microscope images taken with 20x magnification; e) porosity measurement after data manipulation on ImageJ (Maryland, United States); f) Wolper Dia Testor 2 RC (Aachen, Germany) hardness tester.

Contour plots of VED and  $E_{\text{int}}$ , in relation to porosity, hardness, and hole size, are presented in Figures 3.6, 3.7, and 3.8, respectively. The study identifies an optimal processing window with VED ( $47\text{--}55 \text{ J/mm}^3$ ) and  $E_{\text{int}}$  (approximately  $1.6 \times 10^4\text{--}2.6 \times 10^4 \text{ J/mm}^2$ ). Within this range, parts consistently exhibit porosity below 1% and

hardness values exceeding 140 HV. Additionally, comparable density and mechanical integrity are observed in narrower windows extending up to  $75 \text{ J/mm}^3$ . These results provide a robust foundation for further work on mechanical and microstructural characterization, optimization of contour parameters, as well as biocompatibility and biodegradation assessments of pure iron components manufactured using L-PBF.

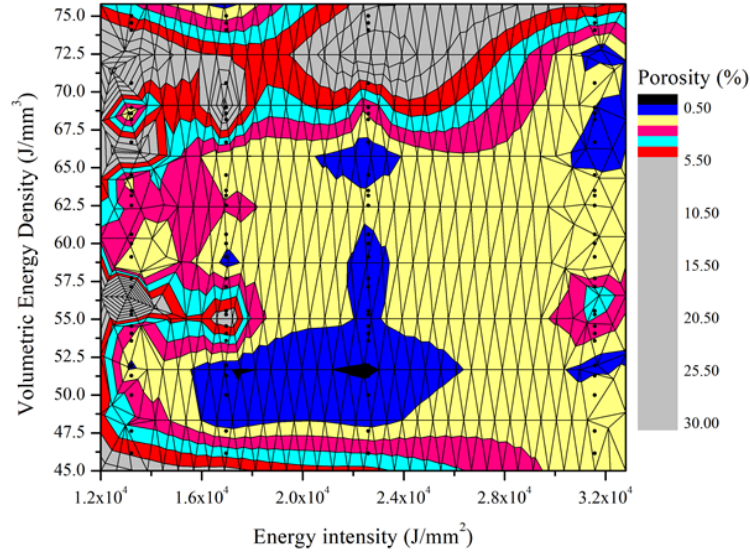


Figure 3.6: Contour plot of porosity as a function of VED and ( $E_{\text{int}}$ ).

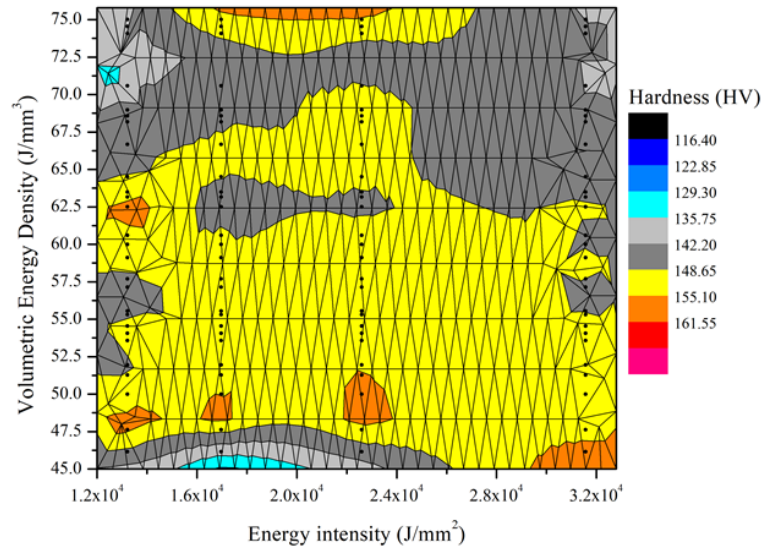


Figure 3.7: Contour plot of hardness as a function of VED and ( $E_{\text{int}}$ ).

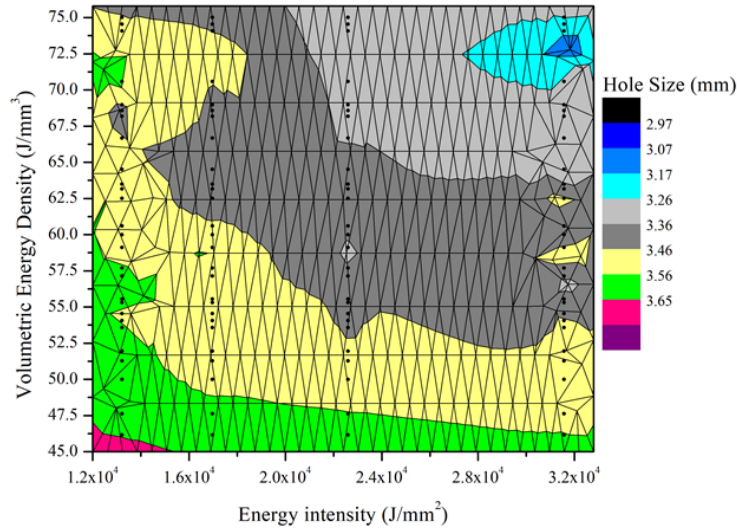


Figure 3.8: Contour plot of hole size as a function of VED and ( $E_{int}$ ).

### 3.4 Bulk Parameter Development - Microstructure Characterization

For microstructure characterization, twelve samples were selected, all produced using a laser spot size of  $130\ \mu\text{m}$ . The samples were grouped into four sets of three, each corresponding to scanning speeds of 600, 700, 800, and 900 mm/s, as detailed in Table 3.5.

Table 3.5: Process parameters of selected samples for microstructure characterization.

| Sample | Laser Power (W) | Layer Thickness ( $\mu\text{m}$ ) | Spot Size ( $\mu\text{m}$ ) | Scanning Speed (mm/s) | Hatch Spacing (mm) | Volumetric Energy Density ( $\text{J}/\text{mm}^3$ ) | Energy Intensity ( $\text{J}/\text{mm}^2$ ) |
|--------|-----------------|-----------------------------------|-----------------------------|-----------------------|--------------------|------------------------------------------------------|---------------------------------------------|
| 56     | 300             | 50                                | 130                         | 600                   | 0.145              | 68.97                                                | $2.26 \times 10^4$                          |
| 58     |                 |                                   |                             | 600                   | 0.165              | 60.61                                                |                                             |
| 60     |                 |                                   |                             | 600                   | 0.185              | 54.05                                                |                                             |
| 80     |                 |                                   |                             | 700                   | 0.125              | 68.57                                                |                                             |
| 82     |                 |                                   |                             | 700                   | 0.145              | 59.11                                                |                                             |
| 84     |                 |                                   |                             | 700                   | 0.165              | 51.95                                                |                                             |
| 104    |                 |                                   |                             | 800                   | 0.110              | 68.18                                                |                                             |
| 106    |                 |                                   |                             | 800                   | 0.130              | 57.69                                                |                                             |
| 108    |                 |                                   |                             | 800                   | 0.150              | 50.00                                                |                                             |
| 128    |                 |                                   |                             | 900                   | 0.100              | 66.67                                                |                                             |
| 130    |                 |                                   |                             | 900                   | 0.120              | 55.56                                                |                                             |
| 132    |                 |                                   |                             | 900                   | 0.140              | 47.62                                                |                                             |

The specimens, initially sectioned perpendicular to the build plate using electrical discharge machining (EDM) and hot-mounted in epoxy resin, were repolished with a sequence of silicon carbide abrasive papers (grit sizes 120, 320, 400, 600, and 1000 mesh). Final polishing was done with diamond suspensions of 6  $\mu\text{m}$  and 1  $\mu\text{m}$  particle sizes.

After polishing, the microstructure was revealed using chemical and electrolytic etching methods. Chemical etching was performed with a 4% Nital solution, while electrolytic etching used a 10% oxalic acid solution at 3 V. A hole was drilled in each mount to insert a copper wire for electrical contact during electrolytic etching. However, unstable contact between the wire and sample caused current fluctuations, making precise control of the etching duration difficult. Consequently, successful electrolytic etching was achieved in only one sample.

### 3.4.1 Optical Microscopy

The samples were analyzed using an optical microscope (Axiophot, ZEISS/Germany) under bright field illumination. Microstructural images were captured for each sample using a digital camera (DP-22, OLYMPUS/Japan), operated with the OLYMPUS Stream Basic software.

### 3.4.2 Electron Backscatter Diffraction (EBSD)

For the SEM-EBSD analysis, sample 108 was selected as it corresponded to one of the best combinations of processing parameters obtained in the previous work described in section 3.3. Microstructural characterization was performed using a SEM coupled with EBSD. The analyses were carried out at CEMUP, using a FEI QUANTA 400 FEG ESEM microscope. Additionally, the EBSD results were obtained using ATEX - software. This approach enabled the characterization of the sample's texture, grain size, grain boundary structure, and dislocation distribution.

## 3.5 Bulk Parameter Development - Tensile Tests

The parameters of sample 108 were selected to produce two specimens, which were subsequently machined into tensile specimens for testing in the transverse direction, following the ISO 6892-1:2009 standard. The tests were conducted at a crosshead speed of 1.5 mm/min, using a 100 kN load cell and a 25 mm extensometer. The transverse direction refers to the orientation perpendicular to the build direction of the additively manufactured part, meaning that the tensile load was applied parallel to the layers, rather than perpendicular to them. The tensile tests were carried out at the Technological Testing Laboratory of DEMec.

## 3.6 Contour Parameter Development - Case Study 1

In the initial stage of contour parameter development for pure iron processed via L-PBF, a systematic parametric study was conducted to evaluate the influence of laser power, scanning speed, and spot size on contour quality. A matrix of process parameters was defined, with scanning speed, laser power, and spot size varying according to Tables 3.6 and A.1. Specimens were fabricated using a post-contour scanning strategy with a 0.09 mm offset from the CAD model. The printed specimens are shown in Figure 3.9a, while the original CAD model is

presented in Figure 3.9b. The geometry of the parts included curved zones, straight sections, and corners to analyze the influence of scanning path and geometry on contour quality. The primary objective was to perform a screening to discard the worst parameter sets characterized by low surface quality, presence of undermelting or overmelting, and contour porosity. Qualitative evaluation under a microscope was performed to assess contour continuity and porosity. The bulk parameters used in this study correspond to those of sample 108, as listed in Table 3.5.

Table 3.6: Process parameters for contour parameter development – Case Study 1 (range and step size).

| Parameter                   | Range   | Step Size |
|-----------------------------|---------|-----------|
| Scanning Speed (mm/s)       | 300–900 | 150       |
| Laser Power (W)             | 110–260 | 50        |
| Spot Size ( $\mu\text{m}$ ) | 75–95   | 10        |

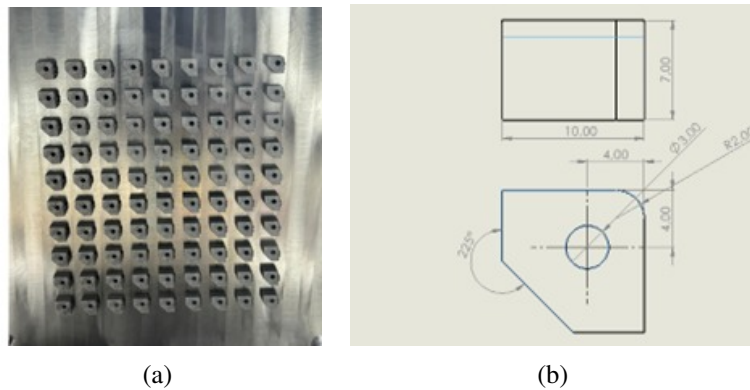


Figure 3.9: (a) Printed specimens for Contour Parameter Development (Case Study 1). (b) Printed specimen geometry.

### 3.6.1 Macrographic Analysis

Following fabrication, the specimens were sectioned parallel to the build plate using a FANUC RoboCut  $\alpha$ -C400iC electrical discharge machining (EDM) system (Oshino, Japan). The sectioned samples were then ground using a sequence of silicon carbide abrasive papers with grit sizes of 320 and 800 mesh.

The continuity and porosity of the contour were analyzed using an OLYMPUS SZ-ET optical microscope. This allowed for the qualitative assessment of surface integrity and the identification of any lack of continuity or pore formation along the contour.

## 3.7 Contour Parameter Development - Case Study 2

In the second stage of contour parameter development, cellular structures with a RTCO geometry and a relative density of 10.28% were fabricated. All specimens were produced using the bulk parameters of sample 108

(see Table 3.5). Contour parameters were varied as shown in Table 3.7. A post-contour scanning strategy was employed for all builds with a 0.09 mm offset from the CAD model. These conditions were selected to investigate the influence of contour parameters on the quality of complex lattice geometries. Produced specimens are shown in Figure 3.10.

Table 3.7: Contour parameters used in Case Study 2 for the fabrication of RTCO cellular structures.

| Specimen | Laser Power (W) | Spot Size ( $\mu\text{m}$ ) | Scanning Speed (mm/s) | Linear Input Energy Density ( $\text{J}/\text{mm}^3$ ) |
|----------|-----------------|-----------------------------|-----------------------|--------------------------------------------------------|
| 1        | 200             | 85                          | 300                   | 117.48                                                 |
| 2        |                 |                             | 450                   | 78.32                                                  |
| 3        |                 |                             | 600                   | 58.74                                                  |

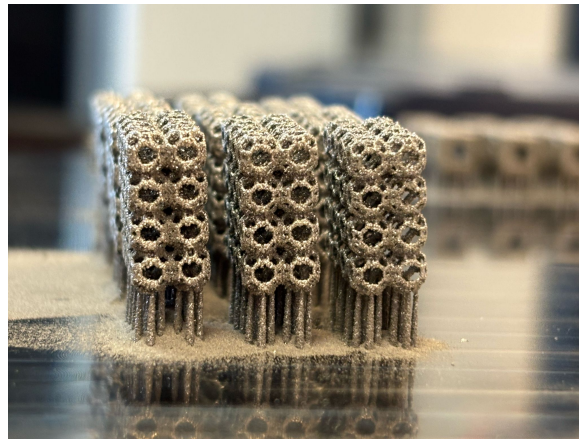


Figure 3.10: Printed Cellular Structures for Contour Parameter Development - Case Study 2.

### 3.7.1 Micro-computed Tomography

Cellular structures 1, 2, and 3 underwent micro-computed tomography (micro-CT) analysis to quantify internal porosity and evaluate its relationship with the applied contour settings. The scans were performed using a *phoenix v|tome|x m* system, at the facilities of Celoplás.

### 3.7.2 SEM Analysis

Afterwards, specimen 3 was selected for SEM visualization. This allowed for a qualitative assessment of surface integrity. The analysis was performed using a Phenom XL G2 Desktop SEM at DEMec.

### 3.7.3 Macrographic Analysis

Using a digital microscope (Emspire 3, LEICA/Germany) operated with the Enersight software, specimen 3 was also compared to a manufactured counterpart in 316L stainless steel, produced using optimized process



parameters, to qualitatively assess defect formation and overall build quality between materials and processing conditions.

### 3.8 Powder Characterization

The powder ATOMET 195SP, intended for future printing processes, was characterized to evaluate its physical and morphological properties. This preliminary characterization is essential to ensure the powder's suitability for L-PBF. The characterization techniques employed included determination of tap density, laser diffraction for PSD, helium pycnometry for true density measurement, and SEM analysis for morphological assessment.

#### 3.8.1 Determination of Tap Density

The tap density was measured using a Copley JV 110i tap density tester following ISO 3953:2011. A powder mass of 50 g was placed in the cylinder, and the machine was set to tap 300 times per minute. The test was stopped after 600 taps and the final volume after tapping was used to calculate the tap density.

#### 3.8.2 Helium Pycnometry

Helium pycnometry was used to determine the real (true) density of the powders by measuring the skeletal volume, which excludes completely closed pores. Helium, with its small atomic size, penetrates even the finest open pores. The sample is placed in a known volume chamber, and the pressure change caused by helium expansion is used to calculate the sample's volume. The real density is then calculated by dividing the sample's mass by the measured volume. This highly precise method is commonly used for quality control and research applications where true density is required [116]. The measurements were conducted at the Department of Chemical Engineering (DEQ).

#### 3.8.3 Laser Diffraction

Laser diffraction was used to measure the particle size distribution (PSD) of the powders. The sample, dispersed in a liquid or gas medium, is passed through a laser beam, and the scattered light is detected at various angles. Smaller particles scatter light at wider angles, and the data is analyzed using models like Mie theory or the Fraunhofer approximation to calculate the PSD. The results are reported as volume-based distributions, showing the range and proportions of particle sizes. The measurements were performed with a *Coulter LS 230* analyzer at DEQ [117].

#### 3.8.4 SEM Analysis

The pure iron powder was morphologically analyzed using a Phenom XL G2 Desktop SEM at DEMec to assess particle shape, surface texture, and the presence of agglomerates or irregularities. This analysis provides some insights into the production, flowability and packing behavior of the powder.

## Chapter 4

# Results and Discussion

### 4.1 Bulk Parameter Development - Microstructure Characterization

#### 4.1.1 Impact of Process Parameters on Microstructure

OM analysis of the twelve samples fabricated under varying L-PBF process parameters, as presented in Table 3.5, revealed no significant differences in their microstructure. Despite variations in scanning speed, hatch spacing, and energy density, all samples exhibited similar grain morphology. Representative micrographs of all twelve samples are provided in Figure 4.1. Some specimens were less effectively etched, which made the grain boundaries less distinct. However, it is still evident some grain elongation. A more detailed quantitative assessment of grain elongation is provided in the following section, based on EBSD results and the grain aspect ratio map.

Variability in grain size was evident within each sample. As shown in Figure 4.2b, regions with finer grains coexist alongside areas of coarser grains, accompanied by porosity defects. Notably, Figure 4.3 highlights substantial porosity in Sample 84. Figure 4.2a also reveals darker regions, likely resulting from oxidation during sample storage and handling. Grain elongation is apparent in this image as well. Furthermore, electrolytic etching exposed the presence of melt pools, as illustrated in Figure 4.4, confirming the characteristic layered deposition of the L-PBF process.



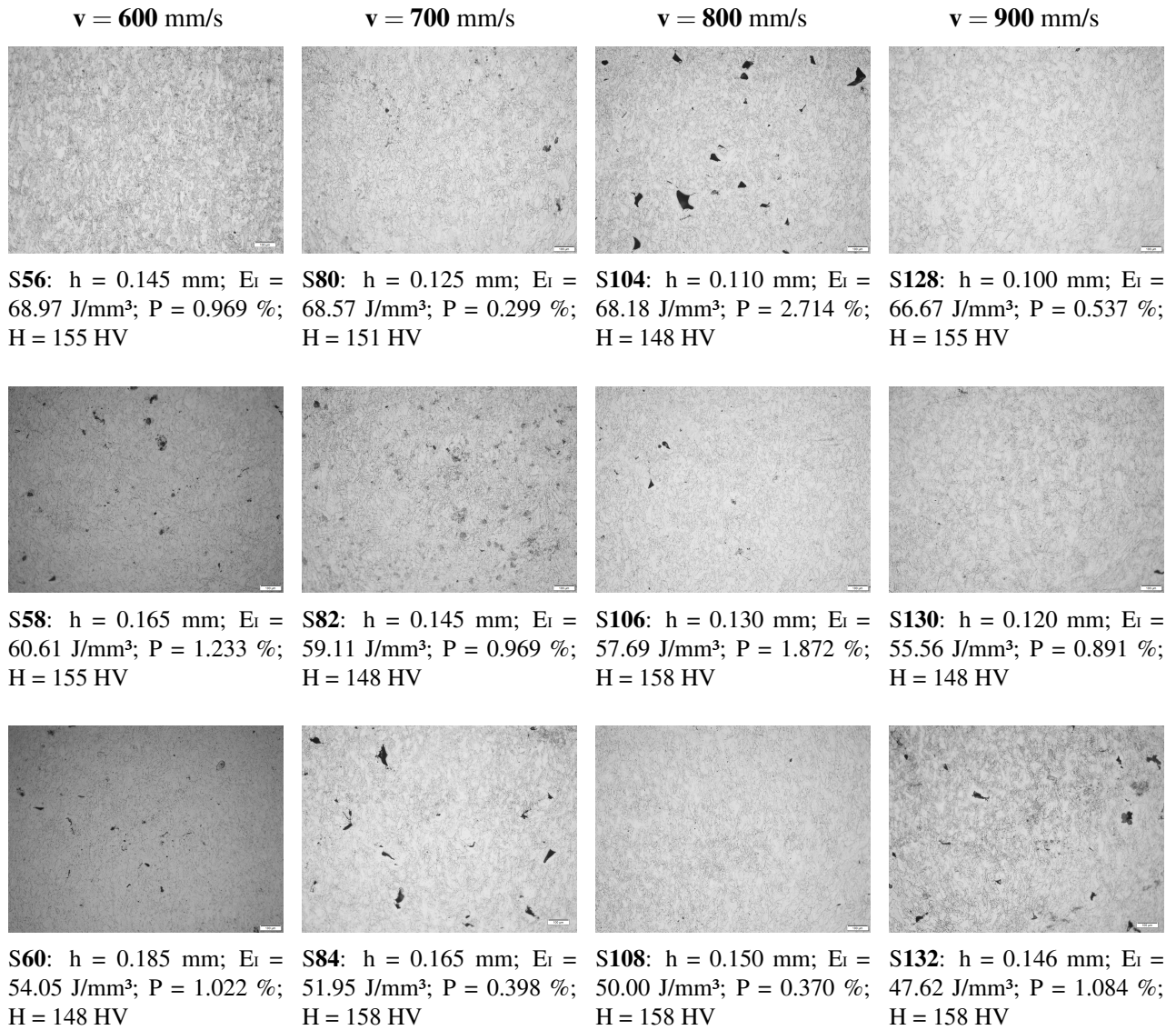


Figure 4.1: Microstructural images of the twelve samples selected for characterization. Each column corresponds to a different scanning speed ( $v$ ) of 600, 700, 800, and 900 mm/s.  $S$  is sample number,  $h$  is hatch spacing,  $E_l$  is linear input energy density,  $P$  is porosity, and  $H$  is hardness.

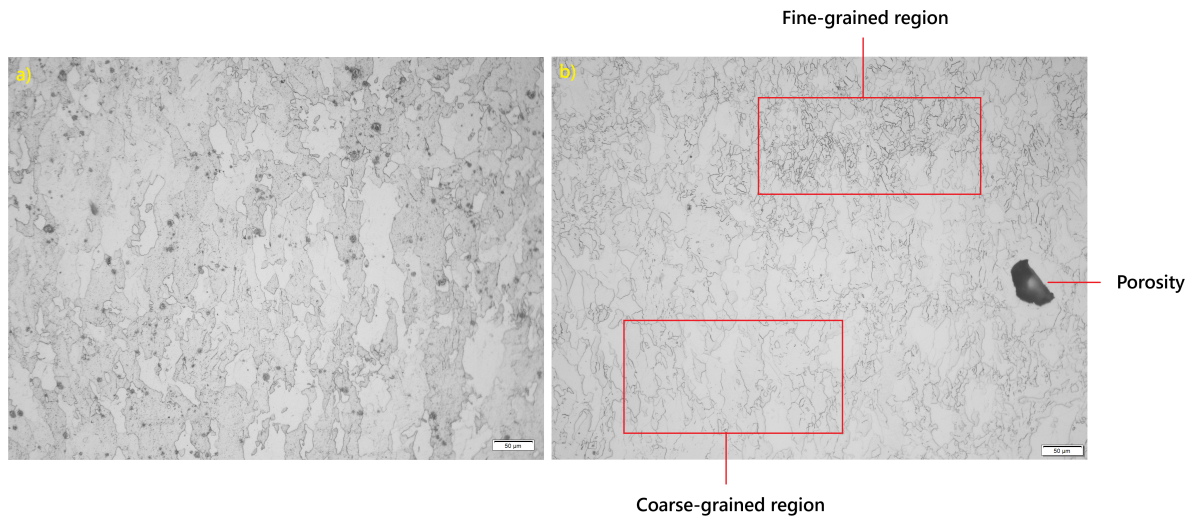


Figure 4.2: Optical micrograph showing (a) sample 56 with darkened spots likely caused by surface oxidation, and (b) sample 108 with visible porosity and regions of fine and coarse grains.

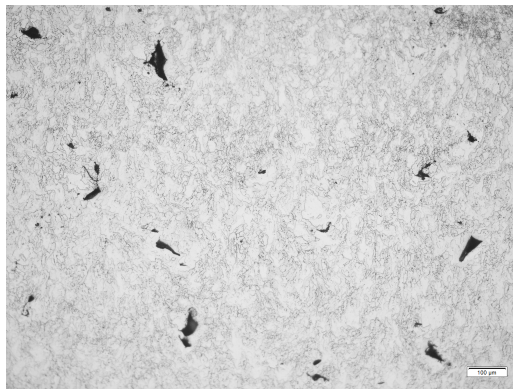


Figure 4.3: Sample 84 with visible porosity.

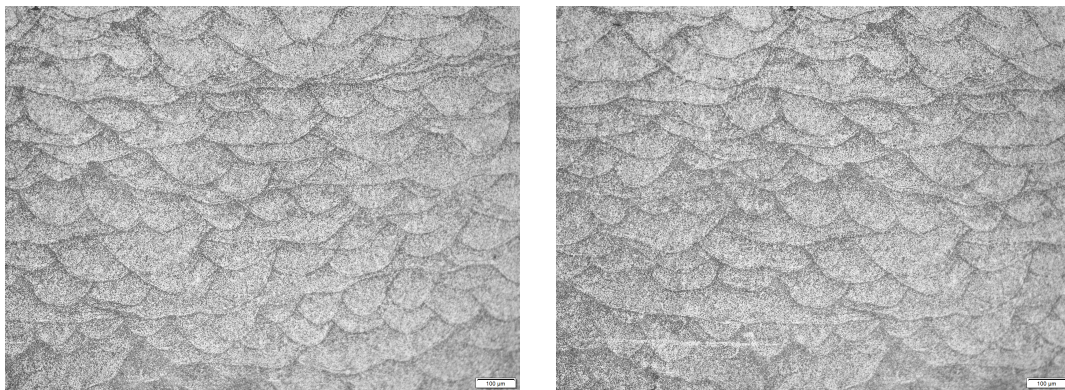


Figure 4.4: Melt pools on sample 108 after electrolytic etching.



#### 4.1.2 Grain Morphology and Size

EBSD analysis of the section parallel to the building direction provided a quantitative assessment of the microstructure observed in the OM characterization. The grains were confirmed to be elongated, with a mean aspect ratio of 2.16, as shown in the aspect ratio map (Figure 4.5a). While the literature typically describes the grain morphology of L-PBF processed pure iron as predominantly equiaxed with only slight elongation [20, 101, 107, 111], the present results indicate a more pronounced elongation and a lower proportion of equiaxed grains. Despite this difference, the results remain consistent with the expected anisotropic grain growth along the build direction, which is characteristic of the L-PBF process. As explained in section 2.7.1, this behavior results from directional solidification and epitaxial grain growth driven by a predominantly unidirectional heat flow, leading to the formation of columnar grains aligned with the build axis [71].

The average grain size was measured to be  $17.95 \pm 11.56 \mu\text{m}$ , which is larger and more variable than the  $12 \pm 6 \mu\text{m}$  reported by Lejček et al. [107]. This spatial variation in grain size is visible in the grain size map (Figure 4.5b).

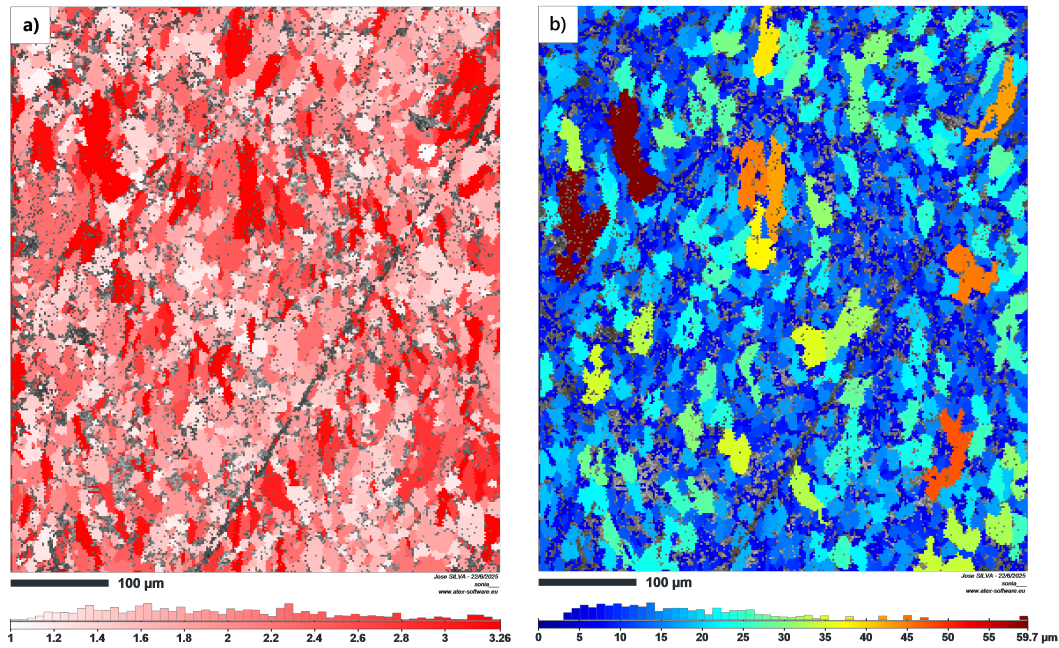


Figure 4.5: EBSD results. (a) grain aspect ratio map and (b) grain size distribution map.

Although the IPF map (Figure 4.6a) does not show a strong preferential orientation, the 001, 101, and 111 pole figures (Figure 4.6b) reveal a moderate to weak crystallographic texture. The 001 pole figure shows the strongest orientation, with a peak along an inclined direction. The 111 and 101 pole figures indicate progressively weaker textures.

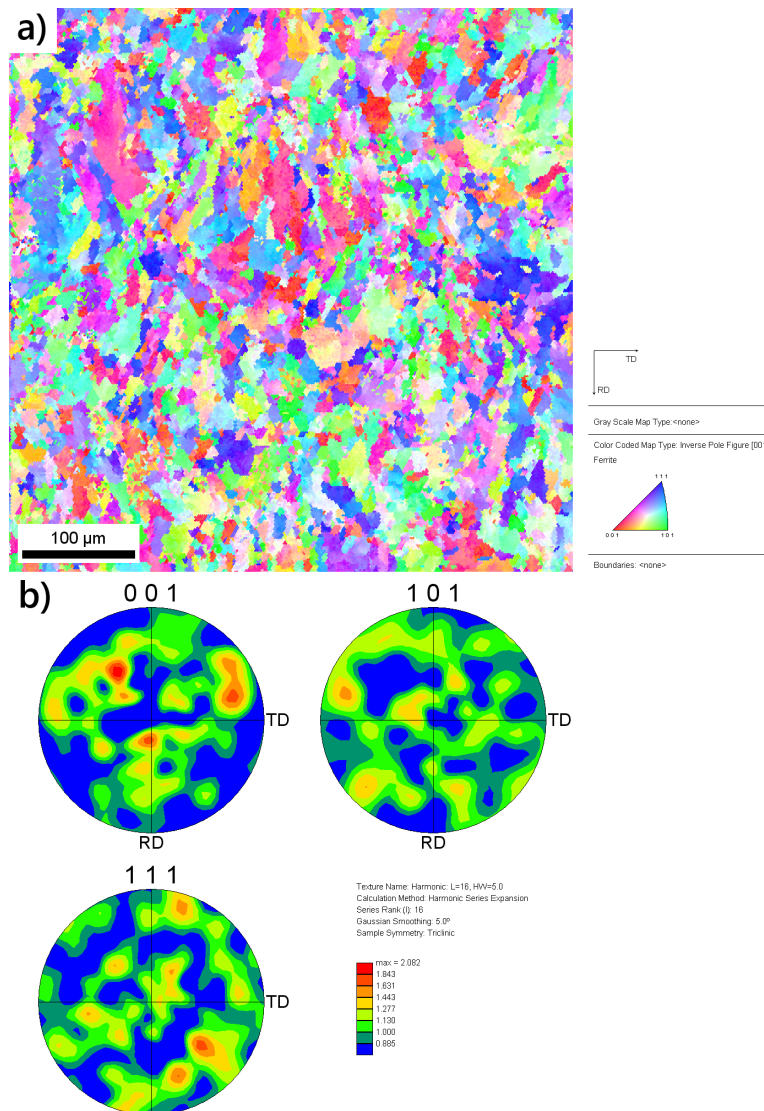


Figure 4.6: EBSD results. (a) Inverse Pole Figure (IPF) map showing the crystallographic orientation of sample 108. (b) Corresponding pole figures for the 001, 101, and 111 planes.

#### 4.1.3 Hierarchical Microstructure

As shown in the grain boundary map (Figure 4.7a), grain boundaries are highlighted in black and low-angle grain boundaries (LAGBs) in red. The presence of LAGBs indicates the formation of subgrains within larger grains.

The corresponding GND (geometrically necessary dislocation) map (Figure 4.7b), taken from the region marked by a square in the boundary map, reveals the distribution of dislocation density. Notably, the LAGBs consistently align with areas of elevated dislocation density. This correlation suggests a hierarchical microstructure composed of grains and subgrains.

According to the literature, such microstructures in L-PBF processed pure iron are typically hierarchical, comprising grains, subgrains, and dislocation cells within the subgrains [89, 107]. However, due to the limited

spatial resolution of the current EBSD dataset, it is not possible to confirm the presence of dislocation cells. The step size used in the scan does not permit direct observation of such fine-scale features, and their existence cannot be determined with confidence.

These observations are partially consistent with the results reported by Lejček et al. [107] and Torrent et al. [89], and support the presence of a hierarchical microstructure in pure iron fabricated via L-PBF. This hierarchy likely consists of primary grains subdivided into subgrains, as evidenced by the LAGBs. While dislocation cells have been reported in the literature, their identification in the present study is limited by the resolution of the available EBSD data.

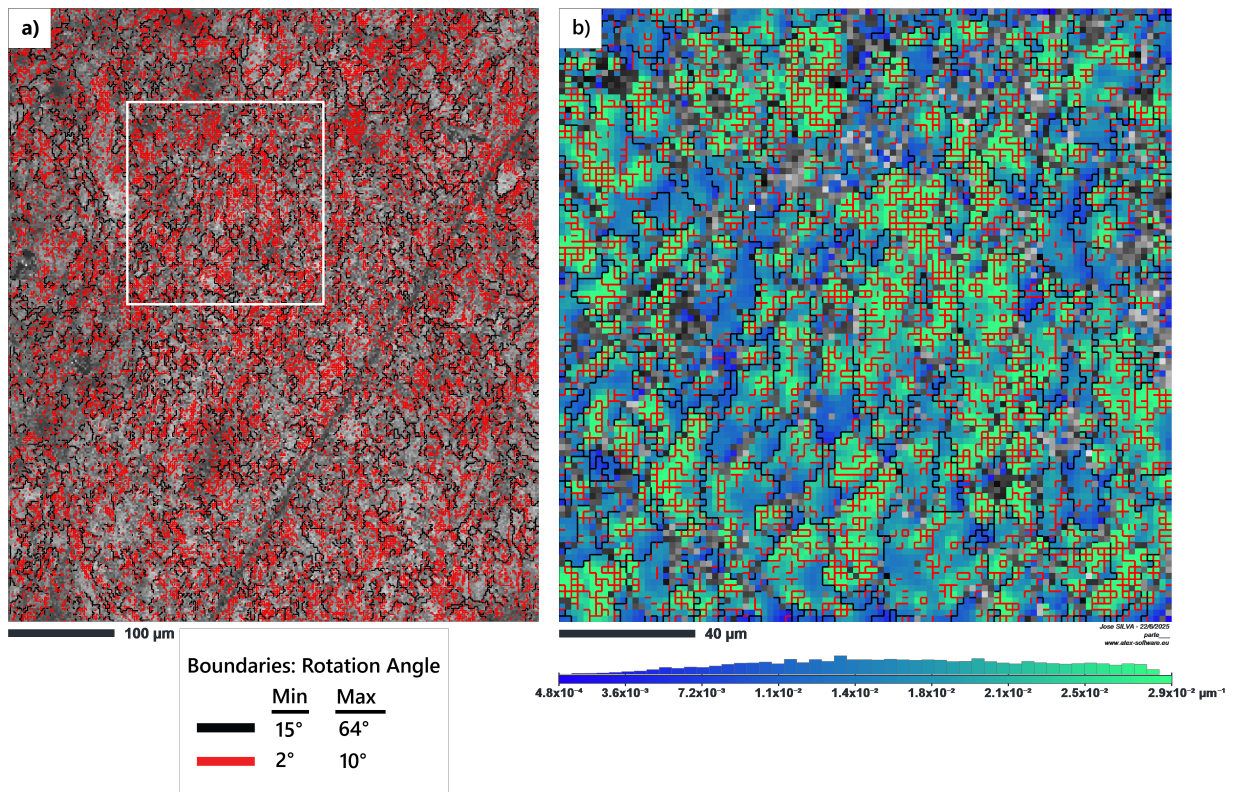


Figure 4.7: EBSD results. (a) Grain boundary map of the scanned area. (b) Geometrically Necessary Dislocation (GND) density map corresponding to the region marked by the white square in (a).

## 4.2 Bulk Parameter Development - Tensile Tests

The tensile tests performed on the bulk parameters of sample 108 in the transverse direction revealed the tensile curves shown in Figure 4.8. The mechanical properties obtained from the tests are presented in Table 4.1. As the fracture occurred outside the extensometer gauge length, the strain values beyond necking are not representative. However, for test 2, the final elongation after fracture was manually measured and is indicated by the larger point on the right side of the graph. For test 1, post-fracture elongation could not be measured because the fracture occurred very close to the end of the specimen, causing one of the measurement marks to be lost.

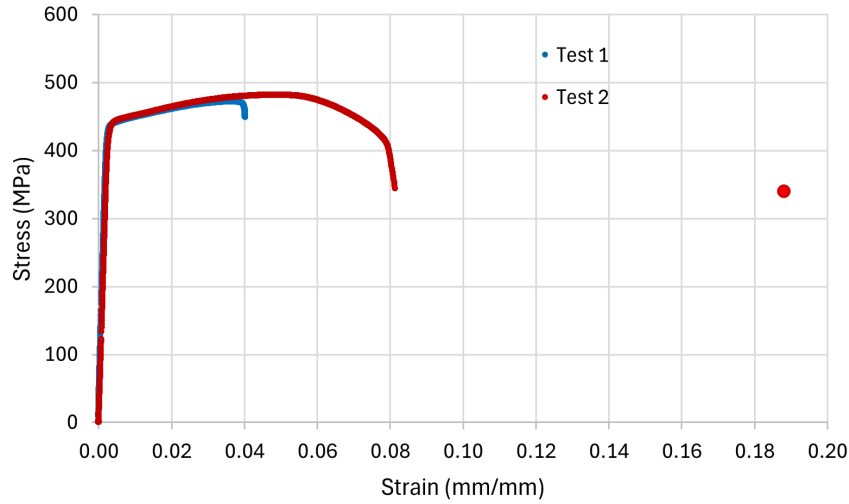


Figure 4.8: Stress-strain curves of the tensile specimens printed with the parameters of sample 108. The data point on the right side of the Test 2 curve indicates the manually measured elongation after fracture.

Table 4.1: Mechanical properties obtained from tensile tests carried out on specimens printed with the parameters of sample 108, compared with literature values for L-PBF pure iron in the transverse direction.

| Test       | E (GPa) | R <sub>m</sub> (MPa) | R <sub>p0.2</sub> (MPa) | A (%) |
|------------|---------|----------------------|-------------------------|-------|
| 1          | 186     | 472                  | 440                     | -     |
| 2          | 174     | 482                  | 443                     | 18.8  |
| Literature | ≈200    | 433–599              | 359–554                 | 12–23 |

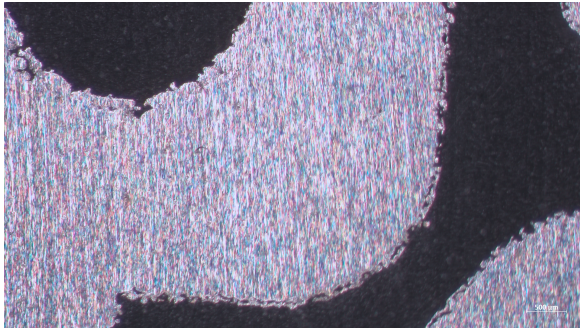
The mechanical properties measured for parameters of sample 108 fall within the ranges reported in the literature for additively manufactured pure iron produced by L-PBF in the transverse direction. Specifically, these parameters exhibited an ultimate tensile strength ( $R_m$ ) of 472–482 MPa and a yield strength ( $R_{p0.2}$ ) of 440–443 MPa, both within the reported ranges of 433–599 MPa and 359–554 MPa, respectively, although they do not reach the highest values found in the literature. The Young's modulus of 174 and 186 GPa is lower than the typical value of 200 GPa reported for L-PBF pure iron. In terms of ductility, the elongation of 18.8% observed in Test 2 falls comfortably within the reported range of 12–23%. Overall, these results demonstrate that the mechanical performance is consistent with the literature values for L-PBF-produced pure iron, reflecting successful processing. However, the chemical composition of the samples may have also influenced these results, as slight variations in purity or the presence of residual elements can significantly affect both mechanical strength and elastic modulus. In addition, variations in process parameters and powder properties can further impact these values.

### 4.3 Contour Parameter Development - Case Study 1

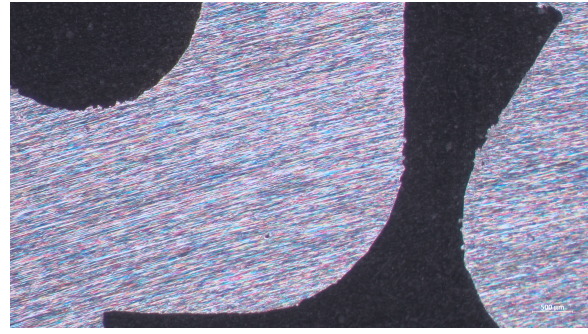
The qualitative analysis of the samples was divided into two distinct evaluations: one focusing on surface continuity and the other on contour porosity.



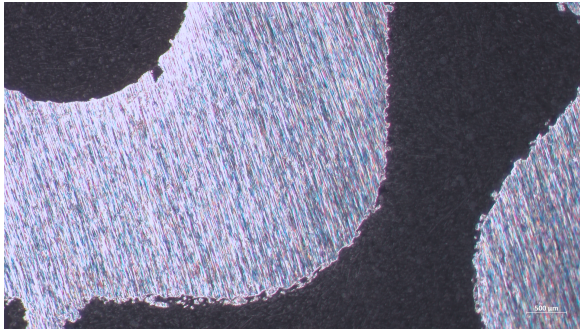
In the context of surface continuity, lower continuity was interpreted as increased surface roughness, as interruptions and irregularities in the surface profile resulted in greater deviations in height along the contour. It was observed that samples with a linear input energy density ( $E_l$ ) lower than  $48,91 \text{ J/mm}^3$  exhibited poor performance in this criterion, as seen in Figure 4.9 (a) and (c), where parts 8 and 24, both below this threshold, show signs of undermelting, resulting in insufficient fusion and reduced surface continuity.



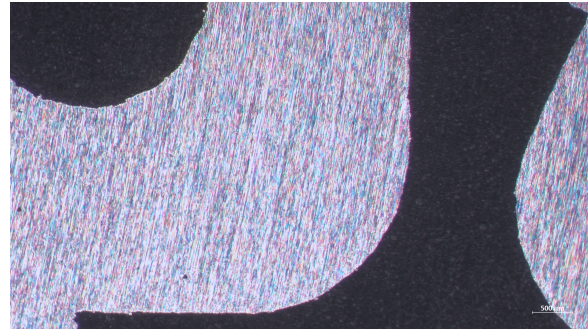
(a) S8:  $P = 110 \text{ W}$ ,  $v = 600 \text{ mm/s}$ ,  $d = 85 \mu\text{m}$ ,  $E = 32.31 \text{ J/mm}^3$



(b) S46:  $P = 260 \text{ W}$ ,  $v = 300 \text{ mm/s}$ ,  $d = 75 \mu\text{m}$ ,  $E = 196.17 \text{ J/mm}^3$



(c) S24:  $P = 160 \text{ W}$ ,  $v = 600 \text{ mm/s}$ ,  $d = 95 \mu\text{m}$ ,  $E = 37.62 \text{ J/mm}^3$



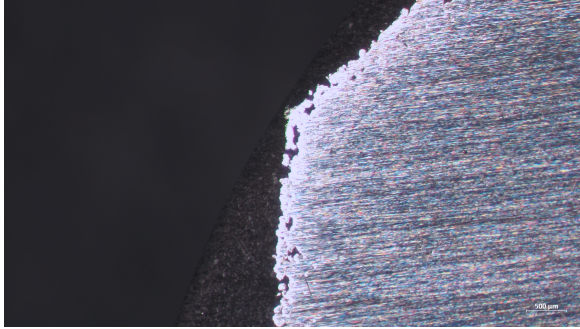
(d) S47:  $P = 260 \text{ W}$ ,  $v = 300 \text{ mm/s}$ ,  $d = 85 \mu\text{m}$ ,  $E = 152.73 \text{ J/mm}^3$

Figure 4.9: Contour continuity of samples 8 (a), 46 (b), 24 (c), and 47 (d) under varying processing parameters.

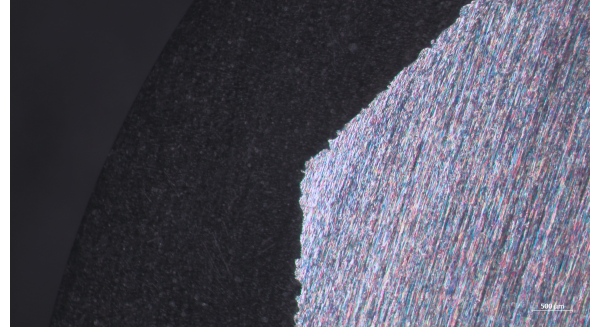
Among all the conditions tested, samples 46, 47, and 48 demonstrated the best results in terms of surface continuity. Processed with a laser power of  $260 \text{ W}$ , a scanning speed of  $300 \text{ mm/s}$ , and spot sizes of  $75 \mu\text{m}$ ,  $85 \mu\text{m}$ , and  $95 \mu\text{m}$ , respectively, these samples exhibited linear input energy densities of  $1.96 \times 10^{-4}$ ,  $1.53 \times 10^{-4}$ , and  $1.22 \times 10^{-4} \text{ J/mm}^3$ , all substantially above the identified threshold for continuity. The combination of high laser power and relatively low scanning speed led to increased energy input, promoting full material melting and minimizing surface irregularities. Samples 46 and 47 are shown in Figure 4.9 (b) and (d).

Lack of continuity may also be partially attributed to pore formation caused by localized vaporization at high energy inputs, which can lead to keyhole instability and partial collapse of the molten material [80]. However, it is important to note that the samples with the highest energy densities in this study did not exhibit such continuity problems, indicating that the energy levels applied were sufficient for effective melting but not excessive enough to trigger keyhole-related defects. Therefore, no signs of overmelting or instability were observed in the best-performing conditions.

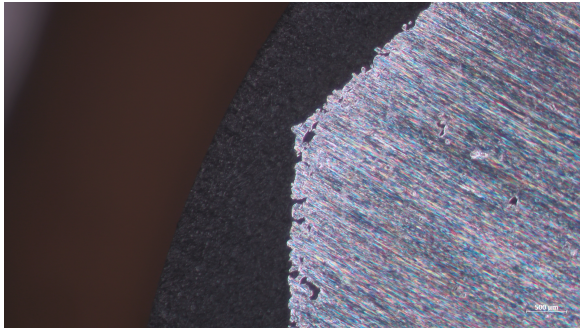
Regarding porosity, the analysis revealed that samples with  $E_t$  below  $30,10 \text{ J/mm}^3$  generally showed inadequate performance, likely due to undermelting conditions that lead to incomplete fusion and pore formation. This indicates a different threshold for acceptable quality in porosity, reinforcing that both factors, while influenced by energy input, respond differently to the processing parameters. Samples 13 and 14, both below this threshold, can be seen in Figure 4.10 (a) and (c), and display noticeably less porosity than samples 25 and 59, shown in Figure 4.10 (b) and (d), which were processed with higher energy densities.



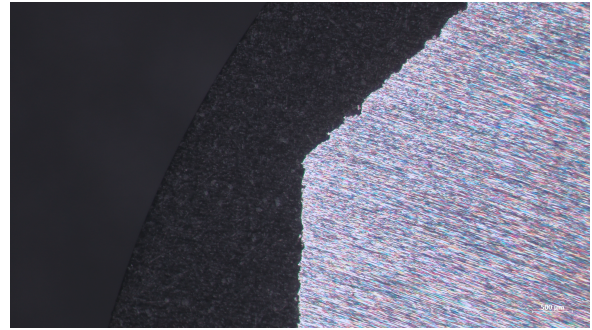
(a) **S13**:  $P = 110 \text{ W}$ ,  $v = 900 \text{ mm/s}$ ,  $d = 75 \text{ }\mu\text{m}$ ,  $E = 27.67 \text{ J/mm}^3$



(b) **S25**:  $P = 160 \text{ W}$ ,  $v = 750 \text{ mm/s}$ ,  $d = 75 \text{ }\mu\text{m}$ ,  $E = 48.29 \text{ J/mm}^3$



(c) **S14**:  $P = 110 \text{ W}$ ,  $v = 900 \text{ mm/s}$ ,  $d = 85 \text{ }\mu\text{m}$ ,  $E = 21.54 \text{ J/mm}^3$



(d) **S59**:  $P = 260 \text{ W}$ ,  $v = 900 \text{ mm/s}$ ,  $d = 85 \text{ }\mu\text{m}$ ,  $E = 50.91 \text{ J/mm}^3$

Figure 4.10: Contour porosity of samples 13 (a), 25 (b), 14 (c), and 59 (d) under varying processing parameters.

It is important to acknowledge that this type of porosity assessment, being based on two-dimensional cross-sectional imaging, may not fully capture the actual extent of contour porosity. The inherent limitations of 2D analysis can lead to a misrepresentation of the true volumetric distribution of pores, particularly in cases where porosity is localized or unevenly distributed along the contour.

In contrast to surface continuity, no specific set of processing parameters was identified as leading to significantly lower contour porosity levels across samples. Although  $E_t$  proved to be a useful indicator, it was not the sole factor influencing final quality. Some samples with  $E_t$  above the thresholds still exhibited low continuity and/or high porosity, suggesting that laser power, scanning speed, and spot size also impact material quality.

In general, curved sections exhibited lower surface continuity compared to straight sections, likely due to more complex scan path dynamics. This suggests that scan path geometry also plays a significant role in the



final surface quality. As illustrated in Figure 4.11, samples 18 and 33 both showed poorer continuity in the curved regions (a) and (b) compared to the corners (c) and (d) and the straight segments (e) and (f).

Overall, samples 46, 47, and 48 stood out as the best in terms of contour quality, exhibiting both high surface continuity and low porosity. This combination indicates that the processing parameters used for these samples were particularly effective in promoting full material melting and superior contour integrity.

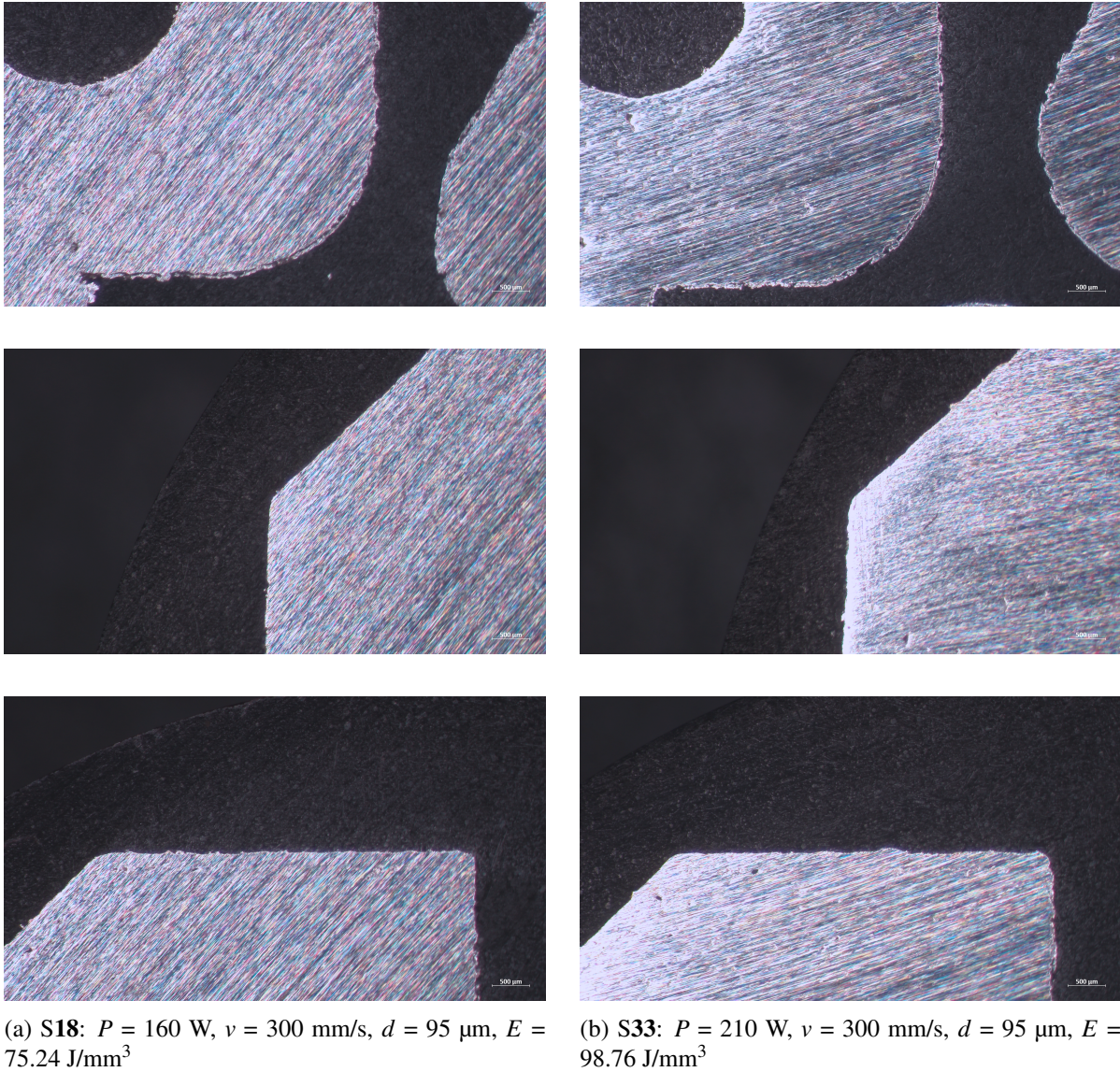


Figure 4.11: Contour continuity of samples 18 (left) and 33 (right) in curved, corner, and straight regions.

Nevertheless, maintaining a sufficiently high  $E_t$  was found to be crucial for achieving good results. It is also important to note that the offset distance, a parameter not optimized in this stage but scheduled for adjustment in future optimization, is expected to significantly influence contour porosity and continuity.

## 4.4 Contour Parameter Development - Case Study 2

### 4.4.1 Cellular Structures – Porosity and Surface Quality

The internal characteristics of the iron cellular structures were captured using micro-CT and subsequently analyzed using MyVGL software to quantify porosity and pore volume distribution. The results, summarized in Table 4.2, show the variation in porosity among the different specimens.

Table 4.2: Contour parameters and porosity values for printed iron RTCO cellular structures in Case Study 2.

| Specimen | Laser Power (W) | Spot Size ( $\mu\text{m}$ ) | Scanning Speed (mm/s) | Linear Input Energy Density ( $\text{J}/\text{mm}^3$ ) | Porosity (%) |
|----------|-----------------|-----------------------------|-----------------------|--------------------------------------------------------|--------------|
| 1        | 200             | 85                          | 300                   | 117.48                                                 | 4.50         |
| 2        |                 |                             | 450                   | 78.32                                                  | 0.19         |
| 3        |                 |                             | 600                   | 58.74                                                  | 0.14         |

Structures 2 and 3 exhibited remarkably low porosity levels of 0.19% and 0.14%, respectively, indicating highly dense structures.

In addition to total porosity, the pore volume distribution provides a more detailed insight into the internal quality of the samples. Specimen 1, which exhibited the highest total porosity (4.50%), also showed a broader pore size distribution. A notably high number of pores with volumes below  $1 \mu\text{m}^3$  was observed, in contrast to the other samples, which exhibited significantly fewer pores in this size range, as illustrated in Figure 4.12.

Moreover, the three largest pores in Specimen 1 had volumes of  $0.110 \text{ mm}^3$ ,  $0.109 \text{ mm}^3$ , and  $0.106 \text{ mm}^3$ , indicating the presence of substantial volumetric defects. In comparison, the largest pores in Specimen 2 were considerably smaller, two at  $0.009 \text{ mm}^3$  and one at  $0.006 \text{ mm}^3$ , while Specimen 3 exhibited larger pores of  $0.023 \text{ mm}^3$ ,  $0.026 \text{ mm}^3$ , and  $0.020 \text{ mm}^3$ . Although Specimen 3 has a slightly lower overall porosity (0.14%) than Specimen 2 (0.19%), it presents a broader pore size distribution.

These results indicate that Specimen 1 has the poorest structural quality, with both a higher pore count and greater pore volumes, pointing to a more porous internal structure compared to samples 2 and 3.

A qualitative analysis of the specimens from Case Study 1 did not reveal significant differences in porosity for parameters similar to those used in Case Study 2, as shown in Figure 4.13. However, it is worth noting that structures 2 and 3 displayed reduced porosity values in Case Study 2, and similar parameters applied in Case Study 1 resulted in good contour quality, both in terms of continuity and porosity. Nevertheless, the parameters employed in Case Study 2 were not identical to those in Case Study 1, differing by 10 W in laser power. Additionally, the geometries of the parts in the two case studies were different, which could also influence the porosity results.



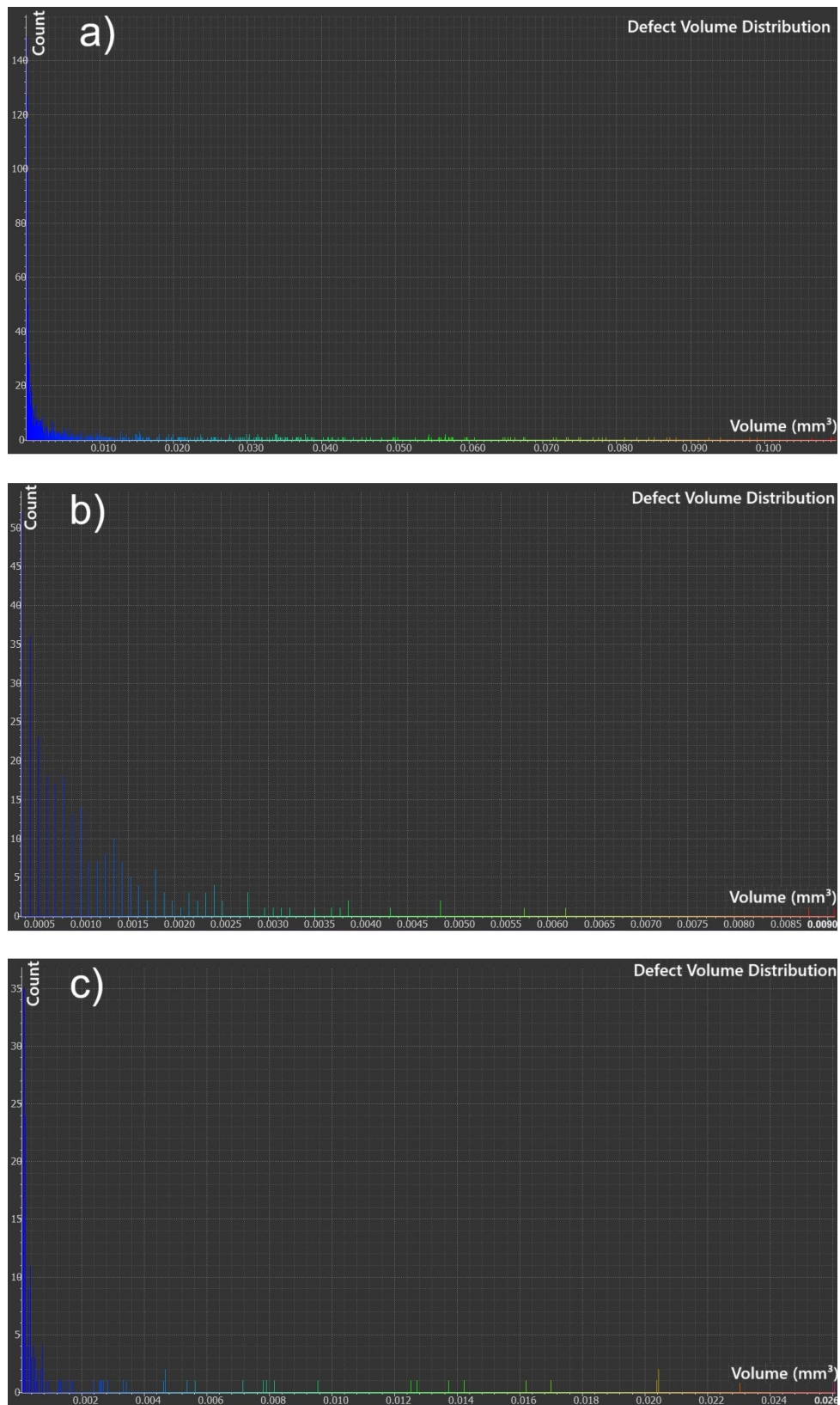
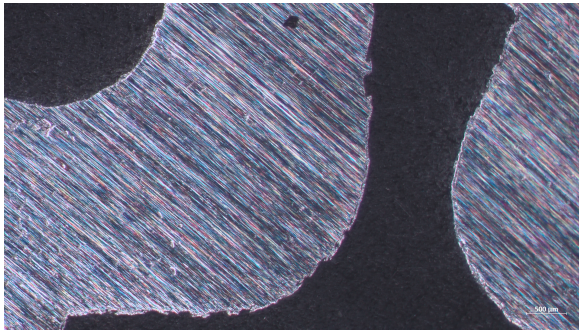
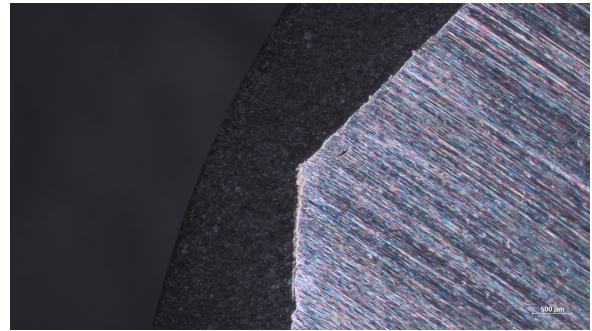


Figure 4.12: Pore size distribution (volume vs. count) for cellular structures: (a) Specimen 1, (b) Specimen 2, and (c) Specimen 3. Axes have different scales, as the graphs were provided by the micro-CT service provider.

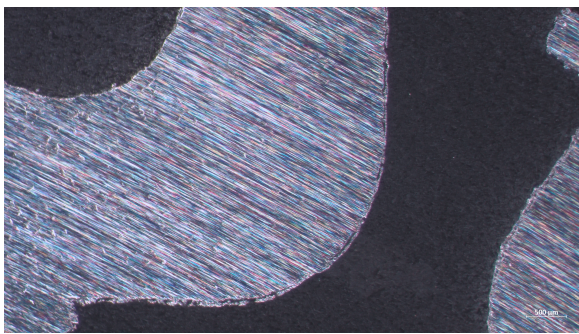




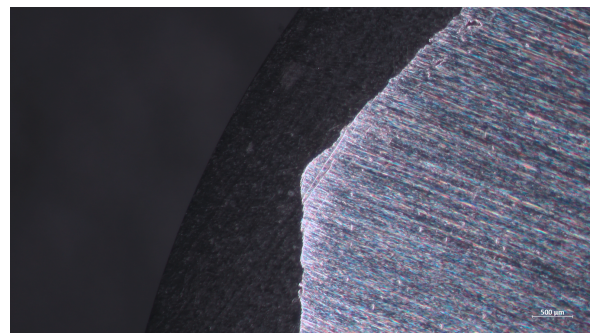
(a) S32:  $P = 210$  W,  $v = 300$  mm/s,  $d = 85$   $\mu$ m,  $E = 123.36$  J/mm<sup>3</sup>



(b) S32:  $P = 210$  W,  $v = 300$  mm/s,  $d = 85$   $\mu$ m,  $E = 123.36$  J/mm<sup>3</sup>



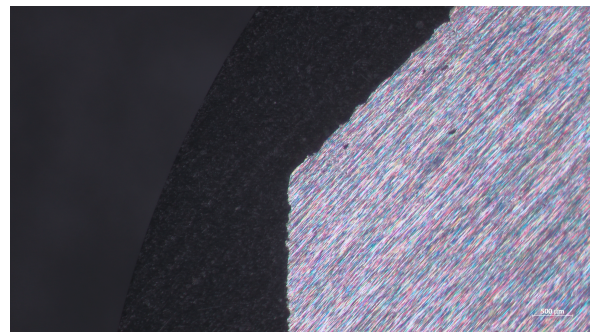
(c) S35:  $P = 210$  W,  $v = 450$  mm/s,  $d = 85$   $\mu$ m,  $E = 82.24$  J/mm<sup>3</sup>



(d) S35:  $P = 210$  W,  $v = 450$  mm/s,  $d = 85$   $\mu$ m,  $E = 82.24$  J/mm<sup>3</sup>



(e) S38:  $P = 210$  W,  $v = 600$  mm/s,  $d = 85$   $\mu$ m,  $E = 61.68$  J/mm<sup>3</sup>



(f) S38:  $P = 210$  W,  $v = 600$  mm/s,  $d = 85$   $\mu$ m,  $E = 61.68$  J/mm<sup>3</sup>

Figure 4.13: Contour quality of samples 32, 35, and 38, respectively, in curved and corner regions.

It is important to mention that the company performing the micro-CT analysis had no prior experience analyzing dense materials such as iron. As a result, the optimal scanning parameters were determined through trial and error, meaning the results may not be fully optimized, although they provide a useful indication of the internal structure.

Another consideration is that the cellular structures underwent some deformation during removal from the build plate. This deformation occurred primarily at the bottom of the structures, where the supports were located, potentially affecting the micro-CT analysis results.

SEM analysis provided further insight into the surface morphology and quality of the fabricated cellular

structures. Several surface features were consistently observed in specimen 3, including powder accumulation, oxidation, and the characteristic surface roughness typically associated with L-PBF.

Powder accumulation was observed in several regions, forming irregular clusters that significantly contributed to increased surface roughness. Oxidation was identified by the presence of darker regions on the surface, indicative of oxide layer formation. These features are illustrated in Figure 4.14. Although the manufacturing process was carried out in an inert gas atmosphere, some oxidation may have occurred during production. However, the majority likely formed post-production due to exposure to ambient air during handling or storage.

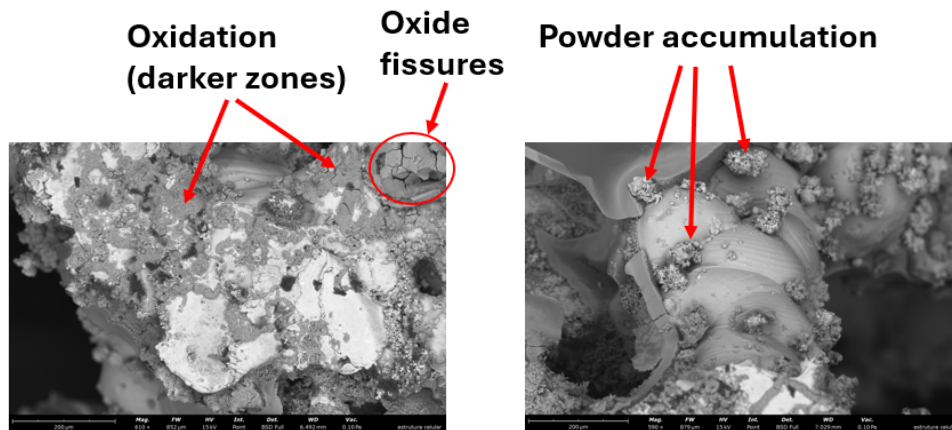


Figure 4.14: SEM images of specimen 9, highlighting powder accumulation, oxidation, and oxide fissures.

#### 4.4.2 Pure Iron vs. Stainless Steel Cellular Structures

Figure 4.15 present a comparison between iron specimen 3 and a 316L stainless steel counterpart, fabricated using pre-established parameters developed by Colibrium Additive, revealing notable differences in surface quality and dimensional accuracy. Despite the presence of a consistent defect on the top surface of the holes, likely caused by excessive laser penetration, the 316L stainless steel Specimen demonstrated a superior surface finish and exhibited less deformation. In contrast, the pure iron structures presented a generally rougher and more irregular surface texture. Such surface imperfections may adversely affect the mechanical performance and reliability of the final components, highlighting the critical importance of precise control over processing parameters during fabrication. It is also worth noting that the 316L powder used in this study was gas atomized, which likely contributed to the improved surface quality due to its typically more spherical particle morphology [82].

Achieving dimensional accuracy in metallic porous components manufactured by L-PBF remains a persistent challenge due to the complex interplay of process parameters, thermal gradients, and material behavior during solidification. Deviations between the CAD model and the printed part are expected and must be taken into account during the design process.

Figure 4.16 shows the CAD model of the cellular structure used in this study, highlighting the three dimensions evaluated: hole height (Hh), hole width (Hw), and strut thickness (t). These features were selected due to their geometric relevance to the mechanical performance and porosity of the structure.



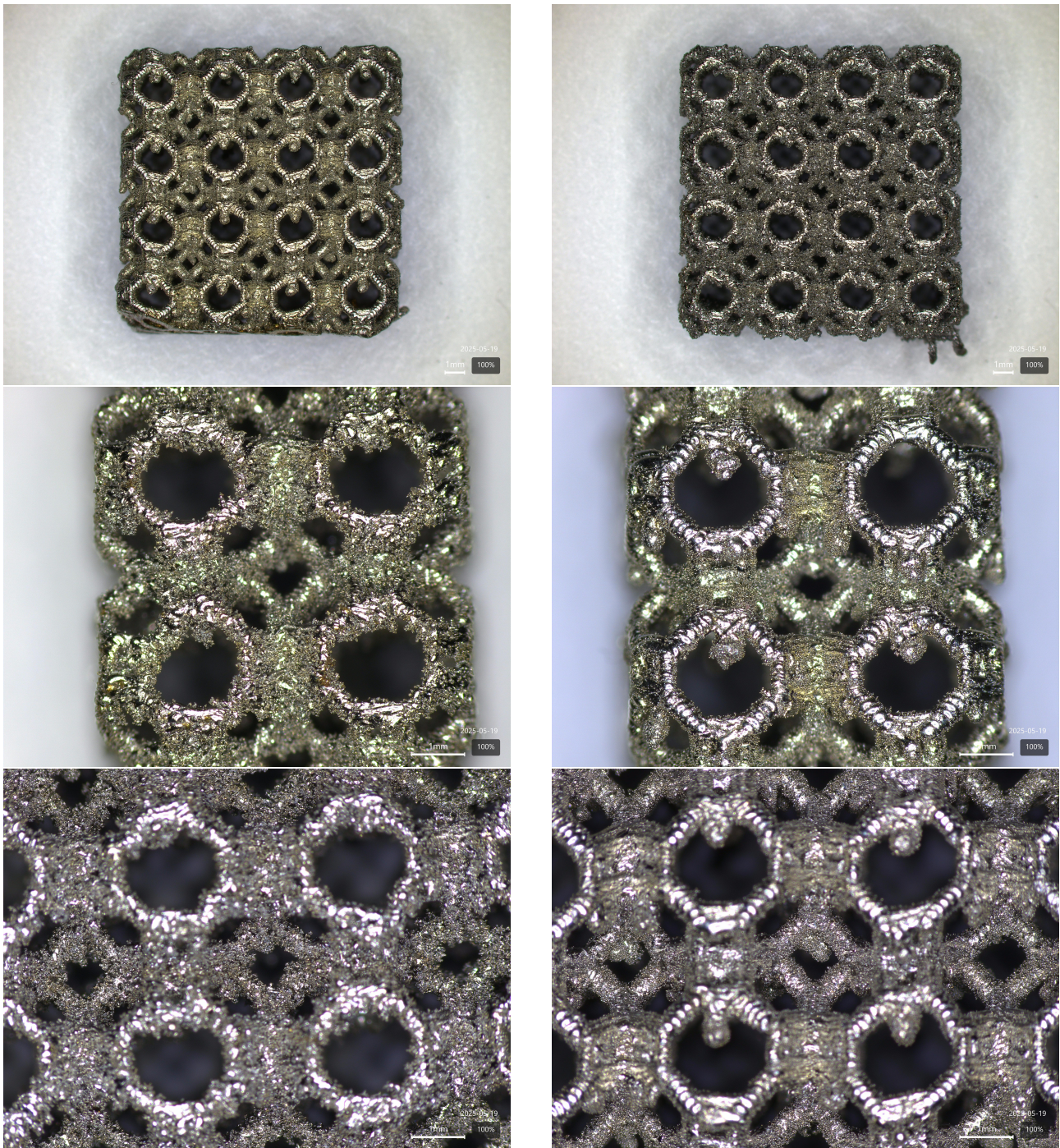


Figure 4.15: Optical macrographs comparing pure iron (left) and 316L stainless steel (right) cellular structures front and side views.

Dimensional measurements were extracted from macrographic images of the fabricated samples, as illustrated in Figure 4.17. Hole height and width were measured directly. However, the strut thickness could not be measured directly due to the inclination of the struts relative to the image plane. Instead, the actual strut



thickness was calculated by dividing the measurement illustrated in Figure 4.16 by 1.41.

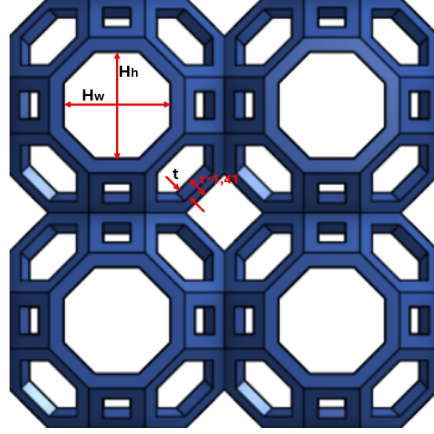


Figure 4.16: RTCO CAD model showing the measured dimensions: hole height (Hh), hole width (Hw), and strut thickness (t).

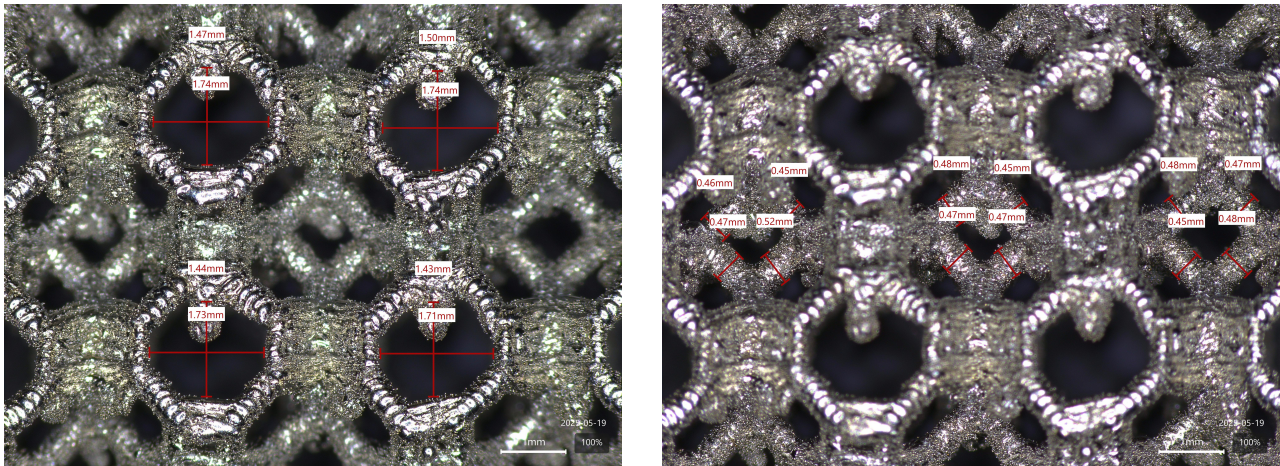


Figure 4.17: Measurements performed on 316L cellular structures: (left) hole height and width; (right) indirect strut thickness measurement (to be divided by 1.41 to obtain actual strut thickness).

The iron cellular structure presents greater dimensional deviation from the CAD model, with differences of 25%, 4% and 61% for hole height, hole width, and strut thickness, respectively. In contrast, the 316L structure shows smaller deviations of 17%, 2% and 52% for the same dimensions. This is quantitatively summarized in Tables 4.3 and 4.4.

In addition to larger dimensional deviations, the iron structure also exhibits greater variability, as evidenced by standard deviations of 0.10 mm, 0.03 mm, and 0.06 mm, compared to 0.05 mm, 0.02 mm, and 0.03 mm in the 316L structure. In both specimens, the variation in hole height is particularly significant when compared to the relatively small variation in hole width. This behavior can be explained by the nature of the L-PBF process,



where vertical features are more prone to shrinkage and overmelting during fabrication, leading to reduced height and geometric inaccuracies [71].

Strut thickness also deviated from the nominal CAD value of 0.23 mm, with measured averages of 0.37 mm and 0.35 mm for the iron and 316L structures, respectively. This overbuilding effect is attributed to laser-induced fusion extending beyond the intended melt path, leading to struts that are thicker than originally designed, a phenomenon commonly observed in L-PBF-fabricated lattices.

These dimensional deviations are also consistent with those reported by Bartolomeu et al. [118], who observed systematic differences between CAD models and L-PBF-fabricated structures across multiple geometries. Several studies in the literature report that the fabrication of sub-millimetric porous structures using L-PBF results in open cells approximately  $100\mu\text{m}$  smaller and walls about  $100\mu\text{m}$  thicker. This behavior is largely attributed to the partial melting of surrounding powder particles and the adherence of loose powder to the melt pool boundaries. This not only increases the effective wall thickness but also reduces the intended pore size, which is especially critical in applications such as orthopedic implants where precise morphological and mechanical properties are required for optimal biological performance [119–122].

Overall, dimensional discrepancies observed here reaffirm the difficulty of reproducing fine structural details in porous metallic parts using L-PBF. Despite identical CAD designs, differences in thermal and metallurgical behavior, and process parameters, between iron and 316L result in variations in dimensional accuracy.

Table 4.3: Comparison between the real measured dimensions of the iron cellular structure (specimen 3) and the corresponding CAD model.

| Dimension             | Measured Values (mm)                                                                                                                                                                                          | Average (mm) | Standard Deviation (mm) | CAD Nominal (mm) | Deviation from CAD (mm) |
|-----------------------|---------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|--------------|-------------------------|------------------|-------------------------|
| Hole Height ( $H_h$ ) | 1.40; 1.47; 1.27; 1.39; 1.23; 1.39;<br>1.24; 1.51; 1.28; 1.14; 1.22; 1.27;<br>1.40; 1.30; 1.23                                                                                                                | 1.32         | 0.10                    | 1.75             | 0.43<br>(25%)           |
| Hole Width ( $H_w$ )  | 1.66; 1.64; 1.71; 1.63; 1.68; 1.72;<br>1.67; 1.68; 1.74; 1.70; 1.64; 1.69;<br>1.68; 1.65; 1.69                                                                                                                | 1.68         | 0.03                    | 1.75             | 0.07 (4%)               |
| Strut Thickness (t)   | 0.42; 0.34; 0.45; 0.29; 0.42; 0.30;<br>0.49; 0.33; 0.41; 0.34; 0.40; 0.28;<br>0.35; 0.36; 0.32; 0.39; 0.31; 0.42;<br>0.28; 0.38; 0.32; 0.46; 0.35; 0.35;<br>0.34; 0.45; 0.33; 0.45; 0.40; 0.45;<br>0.42; 0.30 | 0.37         | 0.06                    | 0.23             | 0.14<br>(61%)           |

Table 4.4: Comparison between the real measured dimensions of the 316L cellular structure and the corresponding CAD model.

| Dimension             | Measured Values (mm)                                                                                                                                                                                          | Average (mm) | Standard Deviation (mm) | CAD Nominal (mm) | Deviation from CAD (mm) |
|-----------------------|---------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|--------------|-------------------------|------------------|-------------------------|
| Hole Height ( $H_h$ ) | 1.47; 1.50; 1.44; 1.43; 1.54; 1.43;<br>1.46; 1.35; 1.45; 1.51; 1.52; 1.51;<br>1.46; 1.45; 1.37; 1.50                                                                                                          | 1.46         | 0.05                    | 1.75             | 0.29<br>(17%)           |
| Hole Width ( $H_w$ )  | 1.74; 1.74; 1.73; 1.71; 1.76; 1.73;<br>1.74; 1.74; 1.70; 1.74; 1.70; 1.71;<br>1.70; 1.69; 1.69; 1.73                                                                                                          | 1.72         | 0.02                    | 1.75             | 0.03 (2%)               |
| Strut Thickness (t)   | 0.32; 0.33; 0.33; 0.37; 0.32; 0.34;<br>0.33; 0.33; 0.33; 0.34; 0.32; 0.34;<br>0.38; 0.36; 0.35; 0.38; 0.33; 0.32;<br>0.34; 0.37; 0.34; 0.31; 0.30; 0.36;<br>0.42; 0.42; 0.35; 0.35; 0.34; 0.35;<br>0.36; 0.35 | 0.35         | 0.03                    | 0.23             | 0.12<br>(52%)           |

## 4.5 ATOMET 195SP Powder Characterization

### 4.5.1 Powder Density

To evaluate the density of the pure iron samples, two measurement techniques were employed. The tapped density was determined to be  $4.00 \text{ g/cm}^3$  and the helium pycnometry yielded a density of  $7.75 \text{ g/cm}^3$ , which is close to the theoretical density of pure iron, reported as  $7.87 \text{ g/cm}^3$  [123]. This difference may indicate the presence of some internal closed porosity.

### 4.5.2 Particle Morphology

As shown in the SEM analysis (Figure 4.18), the powder particles exhibit predominantly irregular morphologies, with only a small number displaying near-spherical shapes. This characteristic strongly suggests that the powder was produced via water atomisation, a process in which a high-pressure jet of water disintegrates molten metal, causing rapid cooling. As a result of this rapid solidification, the particles typically lack sufficient time to form smooth, spherical shapes, resulting in their irregular morphology [89]. Additionally, satellite particles were observed attached to larger primary particles, a feature commonly associated with powders produced by water atomisation. These morphological characteristics may negatively impact powder flowability, as discussed in section 2.5.2. For future work, it is important to assess the powder's apparent density in order to calculate flowability indices such as the Hausner Ratio and Carr Index, as outlined in section 2.5.1.

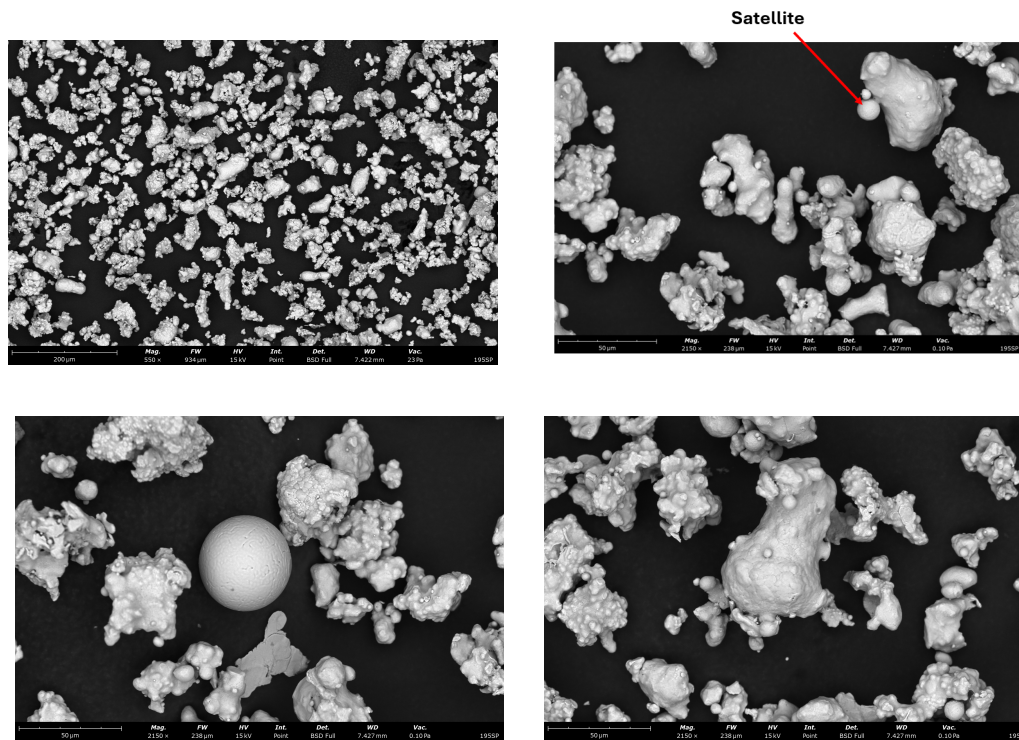


Figure 4.18: SEM images showing general and shape-related features of ATOMET 195SP powder particles: (a) general overview of powder morphology; (b) main particle with attached satellite; (c) spherical particle; (d) larger irregular particle.

### 4.5.3 Particle Size Distribution

The laser diffraction analysis of ATOMET 195SP powder indicates a PSD ranging from  $d_{10} = 15.44 \mu\text{m}$  to  $d_{90} = 53.72 \mu\text{m}$ , which is consistent with SEM observations (Figure 4.18). The small difference between the mean ( $33.89 \mu\text{m}$ ) and the median ( $32.79 \mu\text{m}$ ), along with a positive skewness of 0.426, suggests that the distribution is slightly right-skewed but not strongly asymmetric. The mode, at  $37.96 \mu\text{m}$ , indicates that a larger number of particles are slightly bigger than the average. The low kurtosis value (0.168) suggests the distribution has light tails, with few extreme values. This interpretation is supported by the PSD curve (Figure 4.19), which shows a main peak near the mode and a slightly stretched right tail. Table 4.5 summarizes all relevant PSD statistics.

Table 4.5: PSD Statistics from Laser Diffraction.

| Parameter                         | Value |
|-----------------------------------|-------|
| Mean Diameter ( $\mu\text{m}$ )   | 33.89 |
| Median Diameter ( $\mu\text{m}$ ) | 32.79 |
| Mode ( $\mu\text{m}$ )            | 37.96 |
| Skewness                          | 0.426 |
| Kurtosis                          | 0.168 |

According to the supplier, ATOMET 195SP is expected to contain 97% of particles smaller than  $45\ \mu\text{m}$ , with the remaining 3% in the  $45\text{--}75\ \mu\text{m}$  range (Table 4.6b). However, current measurements (Table 4.6a) indicate a noticeably coarser distribution: the reported  $d_{90} = 53.72\ \mu\text{m}$  suggests that more than 10% of the particles exceed  $45\ \mu\text{m}$ , contradicting the supplier's specification. This is further confirmed by the cumulative volume fraction curve (Figure 4.19).

In addition, the span between  $d_{10} = 15.44\ \mu\text{m}$  and  $d_{90} = 53.72\ \mu\text{m}$  suggests a broader PSD than what might be expected from the supplier's specification. These discrepancies may result from differences in measurement techniques (sieving versus laser diffraction) or batch-to-batch variability. Nonetheless, the measured particle size and distribution fall within a range typically considered suitable for L-PBF.

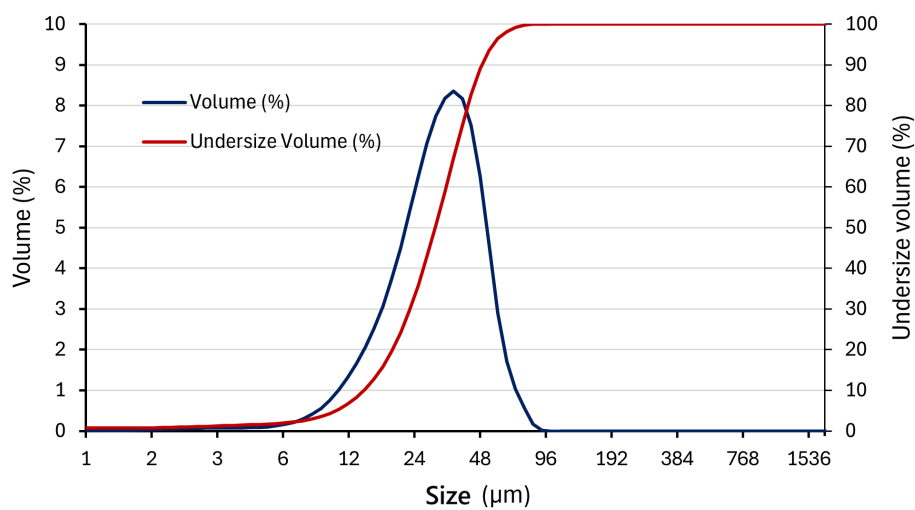


Figure 4.19: PSD and cumulative volume fraction of ATOMET 195SP iron powder.

Table 4.6: Measured vs. Supplier PSD for ATOMET 195SP: (a) Measured percentiles; (b) Supplier PSD (mesh ranges) [115].

| (a)          |                        | (b)                                         |          |
|--------------|------------------------|---------------------------------------------|----------|
| Percentile   | Size ( $\mu\text{m}$ ) | Size Range                                  | Mass (%) |
| d10          | 15.44                  | $>75\ \mu\text{m}$ (+200 mesh)              | Trace    |
| d25          | 23.09                  | $45\text{--}75\ \mu\text{m}$ (200–325 mesh) | 3        |
| d50 (Median) | 32.79                  | $<45\ \mu\text{m}$ (–325 mesh)              | 97       |
| d75          | 43.54                  |                                             |          |
| d90          | 53.72                  |                                             |          |

## Chapter 5

# Conclusions and Future Work

### 5.1 Conclusion

This study aimed to develop and evaluate L-PBF processing parameters for pure iron. The work was structured around microstructural characterization, mechanical testing, contour optimization, and powder evaluation. The main results are summarized below:

- **Microstructural Analysis:**

- OM showed that all pure iron samples produced by L-PBF exhibited similar grain morphology and size across varying bulk processing parameters, indicating a stable microstructure within the selected processing window.
- From EBSD results, the aspect ratio map showed that the grains were elongated, with an average aspect ratio of 2.16, which does not align with the nearly equiaxed grain structure commonly reported in the literature for pure iron processed via L-PBF.
- OM images and the grain size map revealed significant variability in grain size ( $17.95 \pm 11.56 \mu\text{m}$ ), with the presence of both coarse-grained and fine-grained regions.
- The GND and boundary maps showed that the microstructure was hierarchical, with grains and subgrains delineated by boundaries associated with regions of high dislocation density.

- **Mechanical Characterization:**

- Tensile testing of the developed bulk parameters (sample 108) revealed a mean ultimate tensile strength of approximately 477 MPa, yield strength of 442 MPa, a Young's modulus of 180 GPa, and an elongation at break of 19%. These values are within the range reported for additively manufactured pure iron, although below the highest values found in the literature. This difference can be attributed to variations in chemical composition and purity, as well as other factors such as differences in processing parameters and powder properties.

- **Contour Case Study 1:**

- Surface continuity and contour porosity were strongly influenced by linear input energy density. All parameter sets below  $48.91 \text{ J/mm}^3$  resulted in poor continuity, and porosity performance was consistently inadequate below  $\sim 30.10 \text{ J/mm}^3$ , both caused by undermelting.
- Curved regions showed lower continuity, likely due to the more complex scan path required to follow the geometry.
- Laser power, scanning speed, and spot size affected contour quality independently of energy density, as higher energy densities did not always result in improved continuity or reduced porosity.

- **Contour Case Study 2:**

- Micro-CT revealed differences in porosity among samples processed at the same laser power (200 W) and spot size ( $85 \mu\text{m}$ ), but with different scanning speeds (300, 450, and 600 mm/s). Sample 1 (300 mm/s) exhibited higher porosity and a broader pore size distribution, while Samples 2 and 3 (450 and 600 mm/s) showed lower porosity and more uniform pore distribution.
- SEM analysis of the pure iron cellular structures fabricated by L-PBF revealed surface features such as powder accumulation and oxidation.
- Compared to 316L stainless steel cellular structures produced using parameters developed by Colibrium Additive, the pure iron parts exhibited rougher surfaces, larger dimensional deviations, and greater variability.
- Although the pure iron cellular structures still lag behind 316L in terms of surface quality and dimensional accuracy, micro-CT results demonstrated that it is possible to produce low-porosity cellular structures in pure iron.

- **Powder Characterization:**

- Density values aligned with those expected for pure iron.
- SEM revealed irregular, water-atomized particles with some satellites.
- Particle size analysis of ATOMET 195SP powder indicated a PSD ranging from  $d_{10} = 15.44 \mu\text{m}$  to  $d_{90} = 53.72 \mu\text{m}$ . Over 10% of the particles were larger than  $45 \mu\text{m}$ , in contrast to the 3% stated by the supplier.

These results improve the understanding of pure iron behavior during L-PBF processing, highlighting the sensitivity to parameter variations and the challenges of achieving optimal properties in complex geometries. Overall, the insights gained support the development of biodegradable metallic components with tailored mechanical and degradation properties.

## 5.2 Future Work

Future work will continue the development of pure iron for use in L-PBF, focusing on several important areas. Since a new powder (SP150) from a different supplier will be used, the process parameter development will



need to be repeated. Each powder has different characteristics, so it is important to find the best settings for this new material. The full characterization of SP150 powder is still in progress. In particular, the apparent density needs to be measured to calculate flowability indexes. These values will help to understand how well the powder can spread and perform during printing.

In addition to previous simulation studies, it will be essential to experimentally assess the mechanical properties of the printed cellular structures. Measuring properties such as the Young's modulus will enable a comparison with the mechanical behavior of bone, allowing the evaluation of the structures' potential for mitigating stress shielding effects in biomedical applications.

Furthermore, future work should explore and compare the mechanical performance of both graded and non-graded cellular structures. Graded lattices, which feature a gradual variation in cell size or density, may offer a better match to the mechanical properties of bone across different regions, potentially improving load transfer and minimizing localized stress concentrations. Therefore, mechanical testing of these graded configurations, under compression, tension, and fatigue, will be critical to understanding their behavior and suitability for biomedical implants.

Finally, biocompatibility and degradation tests must be done in future work. These tests will show how the material behaves in the body over time and if it is safe and effective for use as a biodegradable implant.

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## Appendix A

# Contour Parameter Development – Case Study 1

Table A.1: Process parameters for contour parameter development – Case Study 1.

| Sample | Laser Power (W) | Scanning Speed (mm/s) | Spot Size (μm) | Linear Input Energy Density (J/mm) |
|--------|-----------------|-----------------------|----------------|------------------------------------|
| 1      | 110             | 300                   | 75             | 83.00                              |
| 2      | 110             | 300                   | 85             | 64.62                              |
| 3      | 110             | 300                   | 95             | 51.73                              |
| 4      | 110             | 450                   | 75             | 55.33                              |
| 5      | 110             | 450                   | 85             | 43.08                              |
| 6      | 110             | 450                   | 95             | 34.49                              |
| 7      | 110             | 600                   | 75             | 41.50                              |
| 8      | 110             | 600                   | 85             | 32.31                              |
| 9      | 110             | 600                   | 95             | 25.86                              |
| 10     | 110             | 750                   | 75             | 33.20                              |
| 11     | 110             | 750                   | 85             | 25.85                              |
| 12     | 110             | 750                   | 95             | 20.69                              |
| 13     | 110             | 900                   | 75             | 27.67                              |
| 14     | 110             | 900                   | 85             | 21.54                              |
| 15     | 110             | 900                   | 95             | 17.24                              |
| 16     | 160             | 300                   | 75             | 120.72                             |
| 17     | 160             | 300                   | 85             | 93.99                              |
| 18     | 160             | 300                   | 95             | 75.24                              |
| 19     | 160             | 450                   | 75             | 80.48                              |
| 20     | 160             | 450                   | 85             | 62.66                              |
| 21     | 160             | 450                   | 95             | 50.16                              |
| 22     | 160             | 600                   | 75             | 60.36                              |

| Sample | Laser Power (W) | Scanning Speed (mm/s) | Spot Size (μm) | Linear Input Energy Density (J/mm) |
|--------|-----------------|-----------------------|----------------|------------------------------------|
| 23     | 160             | 600                   | 85             | 46.99                              |
| 24     | 160             | 600                   | 95             | 37.62                              |
| 25     | 160             | 750                   | 75             | 48.29                              |
| 26     | 160             | 750                   | 85             | 37.60                              |
| 27     | 160             | 750                   | 95             | 30.10                              |
| 28     | 160             | 900                   | 75             | 40.24                              |
| 29     | 160             | 900                   | 85             | 31.33                              |
| 30     | 160             | 900                   | 95             | 25.08                              |
| 31     | 210             | 300                   | 75             | 158.45                             |
| 32     | 210             | 300                   | 85             | 123.36                             |
| 33     | 210             | 300                   | 95             | 98.76                              |
| 34     | 210             | 450                   | 75             | 105.63                             |
| 35     | 210             | 450                   | 85             | 82.24                              |
| 36     | 210             | 450                   | 95             | 65.84                              |
| 37     | 210             | 600                   | 75             | 79.22                              |
| 38     | 210             | 600                   | 85             | 61.68                              |
| 39     | 210             | 600                   | 95             | 49.38                              |
| 40     | 210             | 750                   | 75             | 63.38                              |
| 41     | 210             | 750                   | 85             | 49.34                              |
| 42     | 210             | 750                   | 95             | 39.50                              |
| 43     | 210             | 900                   | 75             | 52.82                              |
| 44     | 210             | 900                   | 85             | 41.12                              |
| 45     | 210             | 900                   | 95             | 32.92                              |
| 46     | 260             | 300                   | 75             | 196.17                             |
| 47     | 260             | 300                   | 85             | 152.73                             |
| 48     | 260             | 300                   | 95             | 122.27                             |
| 49     | 260             | 450                   | 75             | 130.78                             |
| 50     | 260             | 450                   | 85             | 101.82                             |
| 51     | 260             | 450                   | 95             | 81.51                              |
| 52     | 260             | 600                   | 75             | 98.09                              |
| 53     | 260             | 600                   | 85             | 76.37                              |
| 54     | 260             | 600                   | 95             | 61.13                              |
| 55     | 260             | 750                   | 75             | 78.47                              |
| 56     | 260             | 750                   | 85             | 61.09                              |
| 57     | 260             | 750                   | 95             | 48.91                              |
| 58     | 260             | 900                   | 75             | 65.39                              |
| 59     | 260             | 900                   | 85             | 50.91                              |
| 60     | 260             | 900                   | 95             | 40.76                              |