

FACULDADE DE ENGENHARIA DA UNIVERSIDADE DO PORTO

Characterization of Voronoi Lattice Structures Obtained by Additive Manufacturing for Biodegradable Porous Iron Orthopedic Implants

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MASTER'S DISSERTATION



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Abstract

In the context of generative design within the biomedical sector, advances in additive manufacturing (AM), materials science, and tissue engineering have driven innovation. Bone scaffolds, crucial for bone tissue growth, find applications in spinal fusion, joint repair, and craniofacial reconstruction. Generative design, utilizing algorithms and simulation, has emerged as a powerful tool to create complex and innovative designs. This dissertation explores how generative design can revolutionize the production of bone implants, maximizing patient recovery chances. Selective Laser Melting (SLM) is essential for manufacturing geometrically complex and porous implants, particularly with novel materials. Including biodegradable pure iron, which offers potential benefits such as temporary implants that gradually disappear from the body without needing to be surgically removed.

This dissertation aims to characterize the mechanical behavior of Voronoi lattice structures, varying two geometric parameters: number of points and wall thickness. It also investigates the SLM process's ability to print Voronoi structures in 316L stainless steel to inform future production of pure iron samples. Samples were produced in ABS resin and 316L stainless steel, tested for compression, tensile, and flexural properties, and analyzed across different materials and geometry variations.

The study designs Voronoi lattice structures for two manufacturing processes: SLM in 316L stainless steel and Vat-Photopolymerization in resin. The mechanical properties of these structures are evaluated, including Young's modulus, yield strength, flexural strength, and compressive strength. Scanning electron microscope (SEM) analysis of 316L metallic powder quality and optical microscopic analysis of the sample's microstructure were conducted.

Compression testing of 316L samples was influenced by equipment issues, limiting comparison with literature. However, a comparison between fully dense and Voronoi specimens indicated an 88% compressive strength reduction. In testing of resin samples, varying the number of points in Voronoi structures led to reductions in Young's modulus, yield strength, flexural strength, and compressive strength. Wall thickness adjustments also impacted these properties. Transitioning from fully dense to Voronoi structures resulted in reductions ranging from 57% to 92% in mechanical properties. Despite similar average reduction values, the broad range of results highlights the complexity of Voronoi structures, calling for more testing to understand variations and anisotropy.

This dissertation contributes valuable insights into the mechanical behavior of Voronoi lattice structures, enhancing our understanding of their potential for biodegradable bone implants and paving the way for further research in the GradImp project. The study's comprehensive approach encompassed various fields, including orthopedic implants, generative design, and advanced manufacturing technologies, laying the groundwork for future advancements in biodegradable porous iron Voronoi implants.

Resumo

No contexto do design generativo no setor biomédico, avanços na manufatura aditiva (AM), ciência de materiais e engenharia de tecidos têm impulsionado a inovação. As estruturas de suporte ósseo, cruciais para o crescimento do tecido ósseo, podem ser aplicadas em fusão vertebral, reparação de articulações e reconstrução craniofacial. O design generativo, utilizando algoritmos e inteligência artificial, emergiu como uma ferramenta poderosa para criar designs complexos e inovadores. Esta dissertação tem como objetivo explorar a forma como o design generativo pode revolucionar a produção de implantes ósseos, maximizando a recuperação do paciente. O processo de AM seletivo a laser (SLM) desempenha um papel fundamental na produção de implantes porosos, particularmente com materiais inovadores, como o ferro puro biodegradável, que permite a produção de implantes temporários que se degradam biologicamente sem necessidade de remoção cirúrgica.

A dissertação visa caracterizar o comportamento mecânico de estruturas Voronoi, variando dois parâmetros geométricos: número de pontos e espessura da parede. Investiga, ainda, a capacidade do processo SLM em produzir estruturas Voronoi em aço inoxidável 316L, a fim de permitir a produção futura de amostras em ferro puro. Foram produzidas amostras em resina ABS e aço inoxidável 316L que foram submetidas a testes de tração, flexão, e compressão, e analisadas em diferentes materiais e variações geométricas.

O trabalho desenvolve estruturas Voronoi para dois processos de fabrico: SLM em 316L e vat-photopolimerization em resina. As propriedades mecânicas dessas estruturas são avaliadas, incluindo o módulo de Young, tensão de cedência, resistência à flexão e resistência à compressão. Foram, ainda, realizadas análises de microscopia eletrônica (SEM) relativas à qualidade do pó metálico 316L e análises microscópicas óticas da microestrutura das amostras.

Os testes de compressão das amostras 316L foram influenciados por falhas no equipamento, limitando a comparação com a literatura. No entanto, uma comparação entre amostras totalmente densas e Voronoi indicou uma redução de 88% na resistência à compressão. Nos testes efetuados nas amostras de resina, a variação do número de pontos nas estruturas Voronoi levou a reduções no módulo de Young, tensão de cedência, resistência à flexão e resistência à compressão. Variações na espessura da parede também reduziram estas propriedades. A transição de estruturas densas para estruturas Voronoi resultou em reduções que variam de 57% a 92% nas propriedades mecânicas. Apesar da semelhança dos valores médios de redução, a amplitude dos resultados destaca a complexidade das estruturas Voronoi, exigindo mais testes para compreender as variações entre resultados e possível anisotropia.

Esta dissertação contribui substancialmente para o conhecimento do comportamento mecânico de estruturas Voronoi, aprimorando a compreensão do seu potencial para implantes biodegradáveis e expandindo o futuro do projeto GradImp. O trabalho englobou várias áreas, incluindo implantes ortopédicos, design generativo e tecnologias avançadas de produção, servindo como base para futuros avanços em implantes porosos de ferro puro com estrutura Voronoi.

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“The most exciting phrase to hear in science, the one that heralds new discoveries, is not ‘Eureka!’ but ‘That’s funny...’ ”

Isaac Asimov

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Acronyms and Symbols

2D	Two Dimensions
3D	Three Dimensions
ABS	Acrylonitrile Butadiene Styrene
ALM	Additive Layer Manufacturing
AM	Additive Manufacturing
ASTM	American Society for Testing and Materials
BSE	Backscattered Electron Detector
CAD	Computer-Aided Design
CDLP	Continuous Digital Light Processing
CLIP	Continuous Liquid Interface Production
CV	Coefficient of Variation
DED-L	Directed Energy Deposition - Laser
DIW	Direct-Ink-Writing
DLP	Digital Light Processing
EBM	Electron-Beam Melting
EDM	Electrical Discharge Machining
EPU	Elastomeric polyurethane
FCT	Fundação para a Ciência e a Tecnologia
FDM	Fused Deposition Modelling
FEUP	Faculdade de Engenharia da Universidade do Porto
HDH	Hydride-Dehydride
INEGI	Instituto de Ciência e Inovação em Engenharia Mecânica e Engenharia Industrial
IPA	Isopropyl Alcohol
LBM	Laser Beam Melting
LED	Light-Emitting Diode
LMD	Laser Metal Deposition
PBF	Powder Bed Fusion
PBF-L	Powder Bed Fusion - Laser
PE	Polyethylene
PLA	Polylactic Acid
PP	Polypropylene
PPF	Polypropylene Fumarate
SD	Standard Deviation
SEs	Secondary Electrons
SEM	Scanning Electron Microscopy
SLA	Stereolithography
SLM	Selective Laser Melting
SLS	Selective Laser Sintering
UV	Ultraviolet
UVD	Ultra-Variable-Pressure Detector
VPP	Vat-Photopolymerization

Chapter 1

Introduction

1.1 Contextualization

In recent years, generative design has gained significant importance in the biomedical field, driven by advancements in additive manufacturing, materials science, and tissue engineering. One notable application is in the design of bone scaffolds, which play a crucial role in supporting the growth of new bone tissue. These scaffolds find use in various medical procedures such as spinal fusion, joint repair, and craniofacial reconstruction. In the rapidly evolving landscape of technology, generative design stands out as a powerful tool for adaptation and innovation. It leverages computational techniques, algorithms, and simulations to explore extensive design possibilities and generate optimized solutions. This transformative technology has the potential to reshape the way implants are designed, allowing the medical industry to create customized and optimized structures tailored to individual patient needs. As a result, it maximizes the prospects for patient recovery and overall treatment success.

Additive manufacturing has emerged as an ideal solution for producing generatively designed implants, which often exhibit intricate geometries and high porosity, necessitating precise control over design geometry and mechanical properties. Given these demanding requirements, Selective Laser Melting (SLM) stands out as the primary choice for fabricating such implants using a range of metals commonly used in this field.

In recent advancements within the biomedical industry, innovative materials such as biodegradable pure iron have emerged. These materials are particularly employed in the creation of porous implants, designed to lower the material's Young's modulus, reducing the occurrence of stress-shielding. Notably, iron-based materials have the unique ability to gradually degrade within the body, eventually disappearing completely, leaving behind fully regenerated bone. This advancement eliminates the need for additional surgical interventions, enhancing patient comfort and expediting recovery.

1.2 GradImp Project

This work was supported by FCT (Fundação para a Ciência e Tecnologia), within the project PTDC/CTM-CTM/3354/2021 “Biodegradable implants in porous iron obtained by additive manufacturing” and is a partnership between the Faculdade de Medicina da Universidade do Porto, Instituto Superior Técnico, Instituto de Ciência e Inovação em Engenharia Mecânica e Engenharia Industrial and Instituto Politécnico de Setúbal.

1.3 Motivation

The primary objective of the GradImp project is the development of a novel concept of biodegradable iron implants that offer improved degradation rates, mechanical performance, and biocompatibility. These implants incorporate a unique Voronoi generative design optimized for enhancing bone cell growth and regeneration while reducing stress shielding effects. The advanced additive manufacturing technique, Selective Laser Melting (SLM), is utilized to precisely fabricate these complex Voronoi structures using pure iron.

However, due to limited expertise in manufacturing, along with the high cost and limited availability of pure iron, it is crucial to verify the printability of Voronoi structures using more affordable materials. Additionally, studying the mechanical behavior of Voronoi structures in different materials is essential to gain comprehensive insights into this innovative structural design before proceeding with the production of pure iron Voronoi samples. This endeavor aims to contribute to the development, production, and streamlining of future design efforts for optimized biodegradable iron porous Voronoi structures in the GradImp project.

1.4 Objectives

1.4.1 Main Objective

This master’s dissertation primarily aims to investigate the mechanical behavior of Voronoi lattice structures, focusing on variations in two geometric parameters: the number of points in the structure and wall thickness. The study involves the utilization of ABS-like resin, produced through vat-photopolymerization, and 316L stainless steel, manufactured using SLM. Various sample types were generated, with a larger number in ABS due to cost considerations and the limited availability of the SLM process during the experimental work.

The samples underwent comprehensive mechanical testing, including compression, traction, and flexural tests, to obtain a wide range of mechanical properties composed of the Young’s modulus, yield strength, flexural strength and compressive strength. The characterization of these properties was conducted across different materials and various geometric variations. Additionally, the research delves into the printability and quality assessment of Voronoi structures produced through the SLM manufacturing process. This assessment encompassed visual analysis of the

samples, microscopic examination of the microstructure, and SEM observation of virgin samples of 316L metallic powder to analyze its quality and geometry.

These goals are anticipated to establish a robust basis for designing and manufacturing Voronoi structures in alternative materials, like pure iron, and streamline future efforts in biodegradable iron Voronoi structure design and manufacturing within the GradImp project.

1.4.2 Specific Objectives

The master's dissertation encompasses several key topics and objectives:

- **Voronoi Geometry Parameter Selection:** This phase involves the careful selection of Voronoi geometry parameters for study, taking into account different materials and manufacturing methods;
- **316L Metallic Powder Characterization:** The quality and characteristics of the 316L metallic powder will be thoroughly examined and verified through detailed SEM analysis;
- **Visual Inspection:** The 316L Voronoi samples were visually inspected to assess the printing quality of the SLM process;
- **Microstructure characterization:** The microstructure of the 316L samples will be meticulously examined and characterized using optical microscopic analysis;
- **Compression Test Analysis (316L):** Comprehensive comparisons and analyses will be conducted on the compression test results among the 316L samples;
- **Mechanical Test Analysis (ABS-like Resin):** Mechanical tests, including compression, traction, and flexural tests, will be performed on ABS-like resin samples, and thoroughly analyzed;
- **Data Validation (Resin Samples):** The mechanical properties results obtained for the resin samples will be subjected to data validation to ensure accuracy and reliability of the results;
- **Final Comparative Analysis:** A comprehensive comparison and analysis will be carried out on the mechanical results, taking into account the different tested materials, production processes, and geometry variations;
- **Validation with Existing Literature:** The research findings will be validated by cross-referencing them with existing literature related to each of the approached topics.

Chapter 2

State of the Art

2.1 Preamble

This chapter aims to provide a comprehensive foundation for the study of Voronoi lattice implants, manufactured from various materials and employing different manufacturing technologies.

The chapter commences by delving into the realm of orthopedic implants, highlighting their paramount significance in today's world. It places a particular focus on elucidating the intricacies and prerequisites associated with designing novel implants. Furthermore, it elucidates the spectrum of materials currently in use and emerging materials like biodegradable options such as pure iron.

Next, the burgeoning field of generative design is introduced and categorized into two primary design groups: periodic and stochastic structures. The Voronoi lattice structure is expounded upon in meticulous detail, as this serves as the fundamental design methodology employed in the development outlined in this dissertation.

The application of intricate structures like the Voronoi in orthopedic implant design has only become feasible due to the advent of a relatively recent manufacturing process known as additive manufacturing. This chapter underscores the significance and scope of additive manufacturing. It also offers a comprehensive explanation of two manufacturing processes: Selective Laser Melting (SLM) and vat-photopolymerization. These two processes are pivotal in the creation of various samples within this dissertation.

2.2 Orthopedic Implants

Bone is a component of the human body that has the ability to regenerate itself. When cracks and fractures in the bone are small, they can be repaired on their own, but when they are larger than a critical threshold size, they cannot. Due to this, severe injuries or infections may develop, requiring treatments to restore the bone's former function and shape and to aid in its regeneration [1].

Orthopedic implants serve the critical purpose of restoring the strength and functionality of damaged or fractured joints and bones. To be effective and safe, these implants must possess specific mechanical properties, wear resistance, corrosion resistance, biocompatibility, and the ability to integrate with the surrounding bone tissue, a process known as osseointegration. These qualities ensure the implants provide long-lasting and secure solutions without triggering adverse reactions or rejection by the patient's body [2].

Osteoporosis, one of the major risk factors for accidental bone fractures, is characterized by a decrease in bone density and an increase in bone fragility. As the population ages and lifespans increase, the occurrence of this medical condition increases. According to a study of 2020, by 2030, it is feasible to see and anticipate an increase in the incidence of osteoporotic disorders and, subsequently, an increase in bone fractures in six European countries (Figure 2.1), highlighting the importance of this field [3].

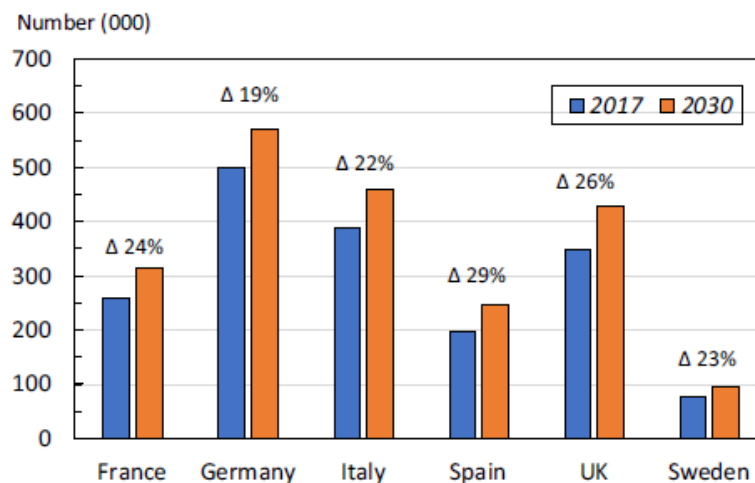


Figure 2.1: Number of fragility fractures by country and the projected numbers in 2030 [3].

In clinical practice, various total joint replacement procedures are used for the hip, knee, ankle, shoulder, elbow, wrist, and finger joints. Metals, ceramics, and polymers are frequently used in these permanent orthopedic implants, which are meant to last the patient's entire lives. A total hip and knee replacements are shown in Figure 2.2 [2]. In Figure 2.3, a plate-type implant is shown, which is usually used to secure severe or even complete fractures of the bone, such as the femur, or the phalanges.

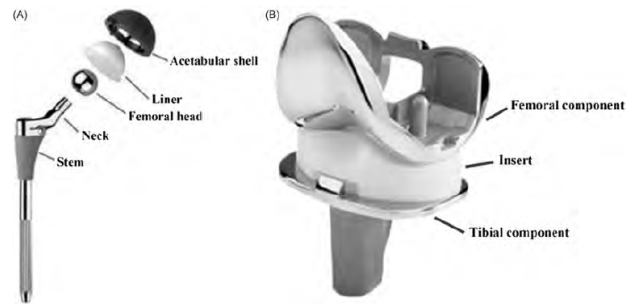


Figure 2.2: (a) Total hip and (b) knee replacements [2].



Figure 2.3: Example of a plate type implant used in a complete bone fracture [4].

2.2.1 Bone Structure and Composition

Bone is a dynamic tissue composed of roughly 60% inorganic material, 10% water and 30% organic material, which is constantly undergoing remodeling. A healthy skeleton must be maintained throughout life, for vital skeletal functions to be carried out properly. This is where remodeling comes into play [5].

Depending on its shape, bones can be classified as long, short, flat or irregular. The bone tissue in long bones is composed of the macroscopically distinct cortical and trabecular bone structures [6]. Cortical or compact bone, which has superior mechanical properties and is 80–90% mineralized, can be found on the exterior surfaces of bones. In this type of bone, collagen fibers may be oriented differently, altering the tissue's mechanical properties and creating anisotropy [7].

Trabecular or cancellous bone, which differs from cortical bone in that it contains bone marrow, has a structure that exhibits porosity and interconnecting trabeculae [8]. Trabecular bone often develops in the extremities of long bones and in vertebrae because it is where compressive stresses build up in the tissue as a result of movement of the body [7]. Figure 2.4 shows the macrostructure composition of bone.

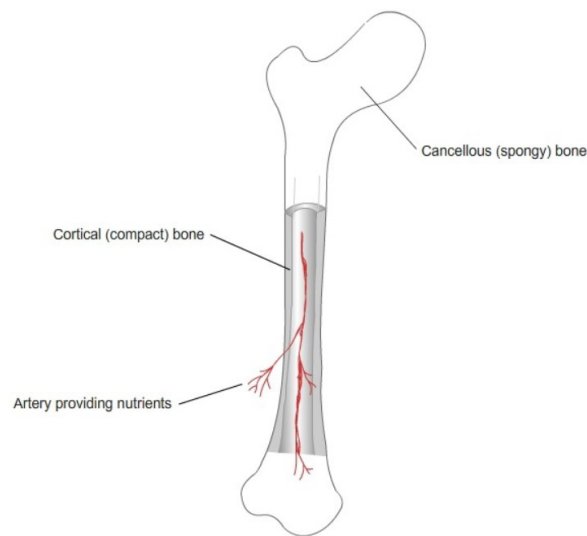


Figure 2.4: Macrostructure of bone [9].

2.2.2 Bone Mechanical Properties

Both the bone's structural organization and composition affect its mechanical properties. Therefore, depending on whether the force is applied longitudinally or transversely, the bone has varied mechanical characteristics [10][11]. Table 2.1 displays the mechanical characteristics of the two types of bone structure: cortical bone and cancellous bone. The mechanical properties of cancellous bone have been reported to be significantly influenced by aging and the consequent changes in bone density [12].

Table 2.1: Mechanical properties of cortical and cancellous bone [12].

Mechanical Property	Cortical Bone	Cancellous Bone
Young's Modulus (GPa)	7-30	0.5-0.05
Compressive Strength (MPa)	100-230	2-12
Flexural Tensile Strength (MPa)	50-150	10-20
Strain to Failure (%)	1-3	5-7

2.2.3 Implant Mechanical Properties

The intended working environments and applications have an impact on the mechanical requirements for orthopedic implant materials. Four crucial mechanical properties include Young's modulus, yield strength, flexural strength, and compressive strength [13]. These four properties can be related to and used to predict other unique mechanical properties, such as resistance to fatigue [2].

To mitigate stress shielding, it's essential for orthopedic implant materials to have a Young's modulus similar to that of human bone, preventing bone re-absorption and implant loosening. Stress shielding occurs when the implant material is significantly stronger than surrounding bone tissue. In addition to Young's modulus, orthopedic implants should exhibit excellent fatigue

strength, as fatigue fractures are a leading cause of early implant failure. Furthermore, ensuring adequate fracture toughness is crucial, particularly for brittle ceramics, to resist crack propagation when subjected to load [2][13]. The Young's modulus values of various materials currently used in the biomedical implant field are shown in Figure 2.5.

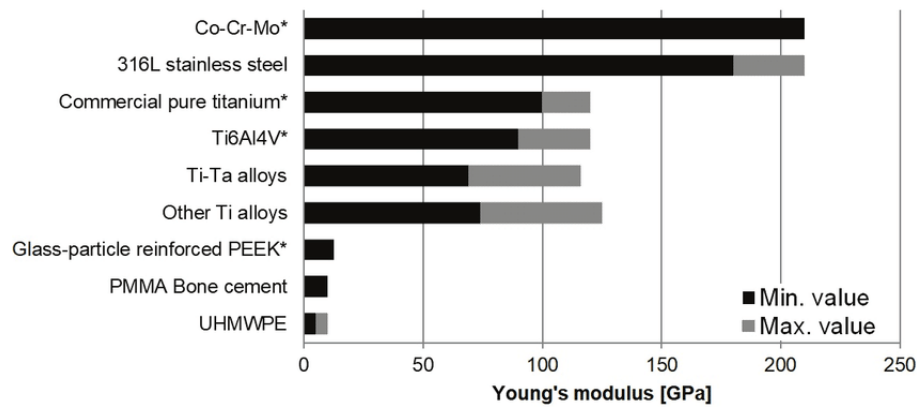


Figure 2.5: Examples of selected materials currently used for medical implants ranked by Young's modulus values [14].

2.2.4 Biocompatibility

The ability of a material to function with an appropriate host response in a particular situation is referred to as biocompatibility, which is regarded as the most crucial component of implant materials [2]. The term can also be further defined as "the capability of a prosthesis implanted in the body to exist in harmony with tissue without causing harmful changes" [15].

Due to the close contact with human tissues, it is crucial that implant materials are nontoxic and do no harm to the host [2]. It is well known that placing a metallic, ceramic, or a combination of both in a bone changes the biomechanical environment and affects the stresses and strains placed on the bone. It is crucial to emphasize that material incompatibility may cause both interface and mechanical failure, resulting in damaging particulate debris [13].

The immune system may be stimulated and a hypersensitive reaction may be brought on by metal ions released from metallic implants into surrounding tissues, which could eventually lead to implant failure. Therefore, when designing and making metallic implants, nontoxic elements should be chosen as alloying elements, and corrosion and wear resistance should be prioritized [2].

Table 2.2, presents the major requirements of biomaterials, which ensure the correct function and compatibility with the human body, and details the severe consequences that the implants can have when these requirements are not followed in the implant design and implant material choice.

Table 2.2: Requirements of biomaterials and problems resulting from inadequate requirements [13].

Significant Requirements	Consequences of not Fulfilling the Requirements
Long Fatigue Life	Implant mechanical failure and revision surgery
Adequate Strength	Implant failure, pain to patient and revision surgery
Young's Modulus Equivalent to Bone's Modulus	Stress shielding, loosening, failure
High Wear Resistance	Implant loosening, severe inflammatory response, destruction of the healthy bone and production of wear debris, which can go to blood, causing adverse local tissue reactions
High Corrosion Resistance	Releasing non compatible metallic ions and allergic reactions
Biocompatibility	Body reaction and adverse effects in the organic system
Osseointegration	Fibrous tissue between the bone and the implant, poor integration of the bone and implant and finally, implant loosening

2.2.5 Osseointegration

Another essential requirement for long-term orthopedic implants is the ability of the implant to bind with the host bone. Osseointegration is the structural and functional union of a load-bearing implant's surface with the living bone [16]. Nevertheless, because temporary devices are removed after healing, bone-bonding is not always necessary or even desired for short-term devices [2].

An implant must be successfully osseointegrated into the host bone in order to establish a structural and functional link between the bone and the implant and enable physiological load bearing [17]. The process of osseointegration is shown in detail in Figure 2.6, wherein immediately after the implantation of an implant or fixture, the development of a hematoma at the bone-implant interface permits the formation of a clot and fibrin matrix on the implant's surface (Figure 2.6 a). The formation of woven bone and subsequent conversion to lamellar bone is caused by the development of a calcified matrix on the device's surface (Figure 2.6 b to e) [18].

Implant design can either encourage or prevent osseointegration, with compositions and surface treatments being linked to successful or unsuccessful osseointegration. Beyond design, successful osseointegration depends on early implant stability and fixation to the host bone. Excessive micro and macro motion can ultimately result in failure [19].

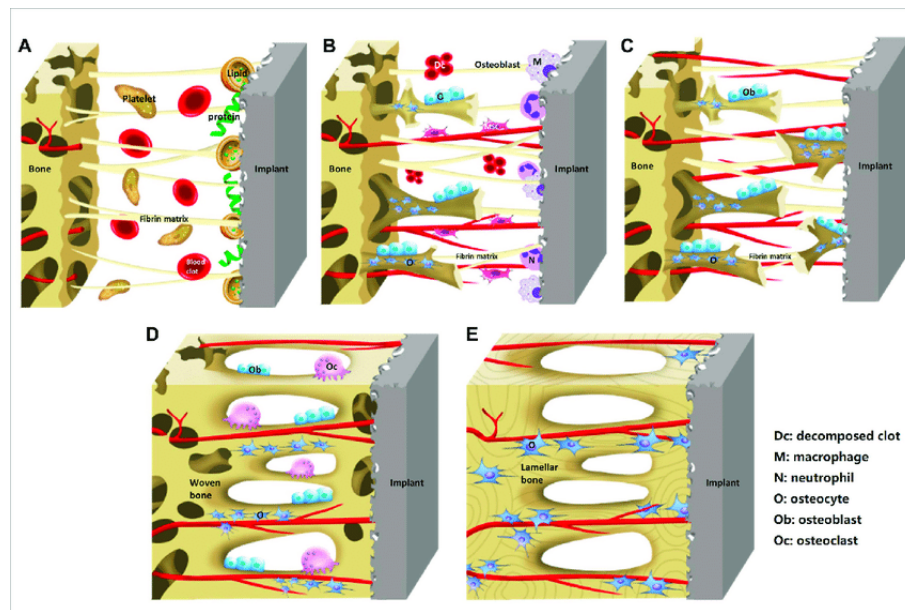


Figure 2.6: Schematic diagram of the process of implants osseointegration. (A) Blood clot and fibrin matrix formation. (B) Woven bone formation. (C) Distance osteogenesis and contact osteogenesis. (D) Newborn woven bones fill up the gap. (E) Woven bone matures into lamellar bone [18].

2.2.6 Tissue Engineering

Tissue engineering (Figure 2.7) is a rapidly growing field in the medical implant industry, that aims to develop different strategies for the regeneration and repair of damaged or diseased tissues. One of the key components in tissue engineering, is the use of scaffolds, which are three-dimensional structures capable of supporting cell attachment and proliferation [20].

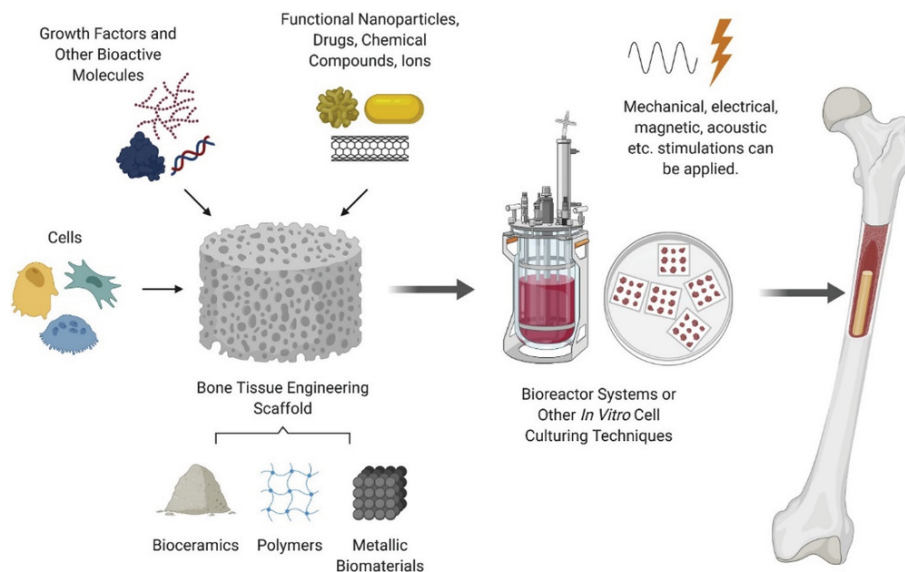


Figure 2.7: Bone tissue engineering strategies [21].

Scaffolds used in bone tissue engineering are made to fill the space left by a fracture and to mechanically support the damaged bone area. The typical porous scaffolds promote bone ingrowth and osseointegration to create new tissue while also encouraging cell adhesion and development. Functional nanoparticles or molecules can be incorporated into the scaffolds for better bone repair and the delivery of therapeutic agents to the patient [22].

2.2.7 Implant Materials

There is a great variety of materials used in the biomedical implant industry, even though these materials must fulfil rigorous criteria, in terms of mechanical properties, biocompatibility, and osseointegration. The many materials available today in the field of medicine have improved as a result of all the advancements and technological developments. The currently used implant materials are mainly metallic, ceramic, and polymeric materials, with new materials being constantly introduced.

No one class of materials is appropriate for all applications, due to the magnitude of different requirements. The majority of orthopaedic devices have been made of metallic materials, which have dominated the field of orthopedic surgery. Even though there are many metals, only a select few are biocompatible, and used as implant materials where they must remain stable in vivo for ten or more years [13].

Pure titanium or titanium alloys are frequently used in many applications due to their mechanical properties, which include good chemical stability and excellent in vivo biocompatibility. These include intramedullary rods and spine surgery applications [23][24].

Stainless steels have the benefits of being relatively inexpensive, readily available, and biocompatible. Because stainless steel's mechanical properties can be extensively controlled, the fabricated products can have the best ductility and strength for medical applications. However, it's important to note that human body fluids are highly corrosive, and this can pose challenges for the long-term use of stainless steel. As a result, they are only used for short-term devices like traction or internal fixation devices [13][23]. Nickel, an alloying element common in stainless steels, such as 316L, helps to increase corrosion resistance, but it should be noted that, in certain quantities, nickel can be extremely toxic to humans [13].

Cobalt alloys are often used in dental and orthopedic implants due to their high strength, temperature resistance, and wear resistance. When a highly polished, extremely wear-resistant material is required, cobalt alloys are normally used [25].

Ceramic's wear resistance and biochemical inertness have led to recent considerations for orthopedic implant applications. Although due to their brittleness, load-bearing applications run the risk of unpredictable catastrophic failure. Since ceramic materials, especially calcium phosphates, are naturally present in the bone mineral phase, they can be used as bone graft replacements in scaffolds for improved bone growth and osseointegration [26].

As scaffolds, polymeric materials can be both bioactive and biodegradable, and are also utilized in tissue regeneration applications. Polypropylene fumarate (PPF) is an example of a polymer with compressive strength comparable to that of cortical bone and with adjustable degradation

rate [27]. The packaging of biomedical devices and implants can be made from a wide range of polymers [28].

Table 2.3, compiles the mechanical properties of the main metallic, ceramic, and polymeric materials currently used in the medical implant industry.

Table 2.3: A comparison of mechanical properties of metallic, ceramic and polymeric implant materials with bone tissue [12][29][30][31][32][33][34][35].

Tissue/Material	Young's Modulus (GPa)	Yield Strength (Mpa)	Compression Strength (MPa)	Tensile Strength (MPa)
Cortical Bone	7-30	-	100-230	164-240
Cancelous Bone	0.5-0.05	-	2-12	-
Ti-6Al-4V	114	827-1103	896-1172	860-965
Stainless Steel 316L	193	170-310	480-620	540-1000
CoCrMo Alloy	240	500-1500	-	900-1540
Mg Alloy	41	100	100-140	180
Zn Alloy	84-210	114-291	273-434	104-399
Fe	210	150-360	-	200-970
Hydroxyapatite	70-120	-	100-900	40-200
PLA	2.86	-	58.6	15.5-150

2.2.8 Biodegradable Implant Materials

Due to their mechanical qualities and biodegradability, metals, such as magnesium, iron, and zinc, are gaining attention as materials for bone implants [36]. Biodegradable implants can be used so that their removal from the body is no longer required once the fractured bone has healed. These implants will temporarily take over the role of the bone until, after a gradual degradation, they are replaced by the newly formed bone tissue [37]. This ensures a significant benefit for the patient's quality of life as well as in terms of costs [38].

Magnesium and its alloys have densities and elastic modulus that are comparable to those of natural bone. However, these materials degrade too quickly in physiological environments and are not currently suitable for use as a biodegradable implant [36][37].

When compared to the rates at which natural bone grows, zinc and its alloys degrade at appropriate rates. However, their applications in bone repair are limited by their inadequate strength and ductility [36][39].

The GradImp project, of which this dissertation is a part, aims to study the potential of pure iron biodegradable implants in Voronoi lattice structures, as discussed in Chapter 1. As a result, in this section, more focus is placed on the present state, applications, and future of biodegradable iron implants.

A true reflection of the current importance given to biodegradable materials and iron in the biomedical industry is the large number of recent publications collected in a 2022 study [40]. Figure 2.8 covers articles in the last 5 years and the rising interest on biodegradable iron. The

steady rise in publications demonstrates how important and current this topic is [40]. To note, that the data was collected in the middle of 2022, and as such it does not cover the entirety of publications in that year.

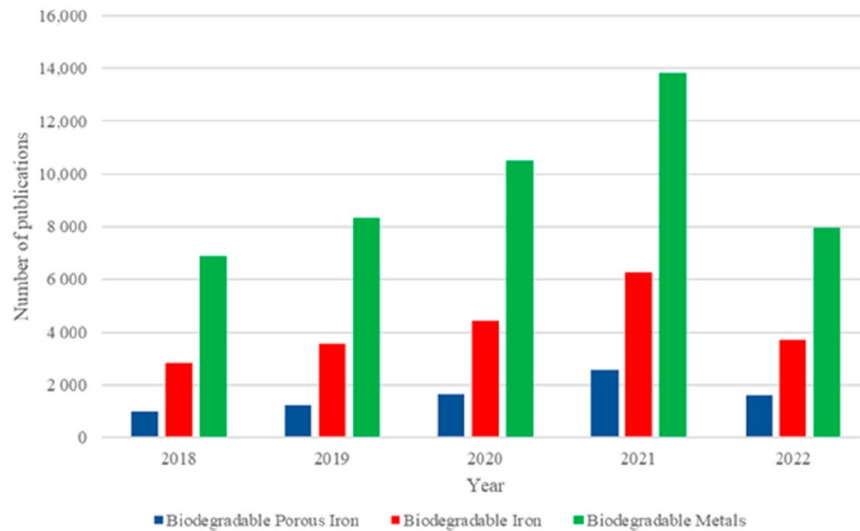


Figure 2.8: Number of publications related to biodegradable metals, iron and porous iron from 2018 until 2022 [40].

Despite having exceptional mechanical qualities, pure iron and its alloys have degradation rates that are too low to support bone repair. [36]. Pure iron is an ideal biodegradable material because it degrades without releasing harmful substances, being completely absorbable after the bone is repaired [41].

The possibility of using iron biodegradable implants is not deterred by the current degradation rate. New strategies for speeding up iron's degradation rate are being researched and developed [36][38]. By generating more active degradation sites, implant porosity greatly accelerates the rate of degradation. Additionally, alloying can speed up the degradation of the implant. Thus, it is clear that many useful tools and approaches are being created for controlling how quickly iron implants degrade, and that soon it will become an optimal biodegradable implant material [38].

2.3 Generative Design

The exponential growth and innovation in the biomedical and additive manufacturing industries have led to a demand for ever-increasing geometric complexity, manifested in the form of freeform geometries, thin walls, and organic shapes. These demands are impractical to satisfy solely through the use of traditional design approaches and are better met by generative design, a developing field of artificial intelligence.

Generative design is a process that employs algorithms to explore design variations beyond the scope of conventional design methods. By utilizing user-defined parameters and objectives, generative design can rapidly examine a multitude of design possibilities, often numbering in the

thousands, to identify the most optimal solution. This approach mirrors the evolutionary process found in nature [42].

Generative design produces optimized parts that are lighter, stronger, and more effective to meet the unique requirements of the applications. The final form's shape is distinctively organic as they are created to meet a particular need [42].

Figure 2.9, displays the design evolution of a chair through the use of increasingly complex generative design tools. Model 1 is a basic chair that serves as an example of traditional design methods. For model 2 a simple generative design algorithm is used to generate a uniform lattice structure. While in model 3, a complex evolutionary tool is used, that harnesses the capabilities of artificial intelligence. With the increase in design method complexity, the chair lost significant weight with only a small decrease in performance, thus reducing material usage and cost, proving to be a great example of the capabilities of generative design.

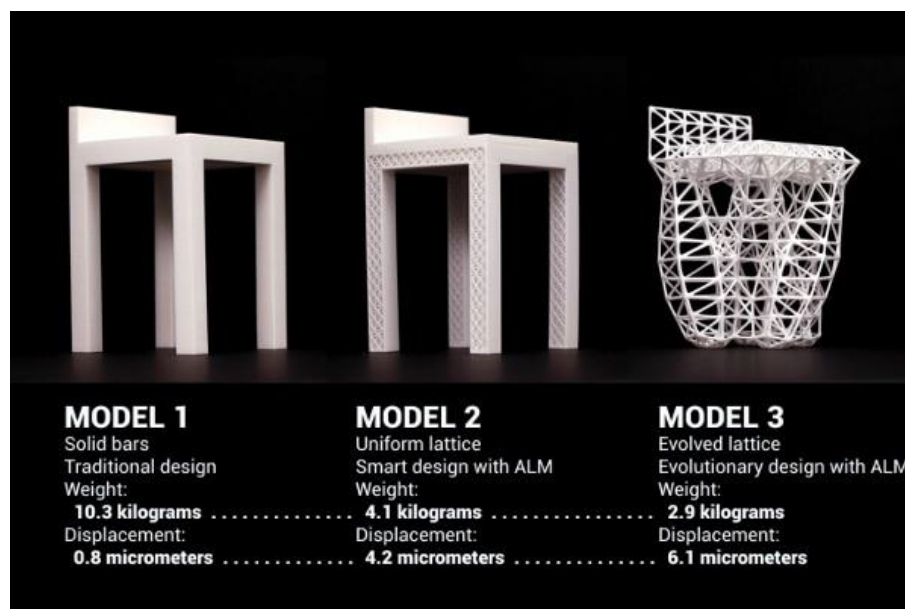


Figure 2.9: Mechanical characteristics of a chair designed using generative design [42].

In conclusion, generative design will play a significant role in the development of novel orthopedic implants because it offers the chance to challenge accepted norms and makes it possible to produce incredibly efficient parts that are impossible to duplicate using conventional design and manufacturing methods.

2.3.1 Lattice Structures

A lattice structure, which is regarded as a type of cellular material, is a space-filling cell with continuously repeating unit cells that connect in three dimensions and can be tessellated along any axis without gaps between cells [43][44].

As shown in Figure 2.10, lattice structures typically consist of either periodic or stochastic topologies. While stochastic structures are created at random and typically have an organic shape, periodic structures have a regular repeating pattern.

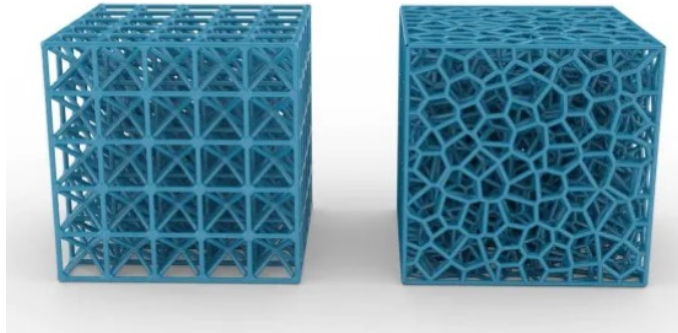


Figure 2.10: Example of lattice structures. (Left) Periodic lattice structure. (Right) Stochastic lattice structure [45].

Utilizing lattice structures can have a number of benefits, including lowering weight, consuming less energy during manufacturing, and improving efficiency in sectors like the aerospace, automotive, and medical industries [43][44]. Lattice structures can be especially helpful in the creation of scaffolds that are optimized for tissue and bone replacement in order to promote osseointegration. They can also be very helpful in minimizing material requirements during implantation, greatly reducing invasiveness and recovery time [43].

2.3.1.1 Periodic Lattice Structures

Literature describes periodic or non-stochastic lattice structures as consisting of continuously repeating, three-dimensionally interconnected unit cells [43][44]. These structures are produced as a result of their inherent geometric characteristics, such as cell size, angle, number, and boundary conditions for the final objects [43].

The design of the unit cells determines the classification of lattice structures used in tissue engineering and bone scaffolds. These unit cell's design can be divided into three major categories (Figure 2.11): CAD-based, implicit surfaces, and topology-optimized unit cells [44].

Unit cells used in CAD-based design (Figure 2.11 a) are derived from Platonic and Archimedean polyhedral solids, and they have simple geometries [44].

Implicit-based unit cells are gaining popularity in bone tissue engineering because they resemble the mean curvature of trabecular bone. Figure 2.11 b) shows several examples of implicit based unit cells [44].

Topology optimization of the base unit cell, a type of generative design, uses numerical techniques to modify the shape of the unit cell to best meet the required specifications (Figure 2.11 c). It works well in load-bearing implants [44].

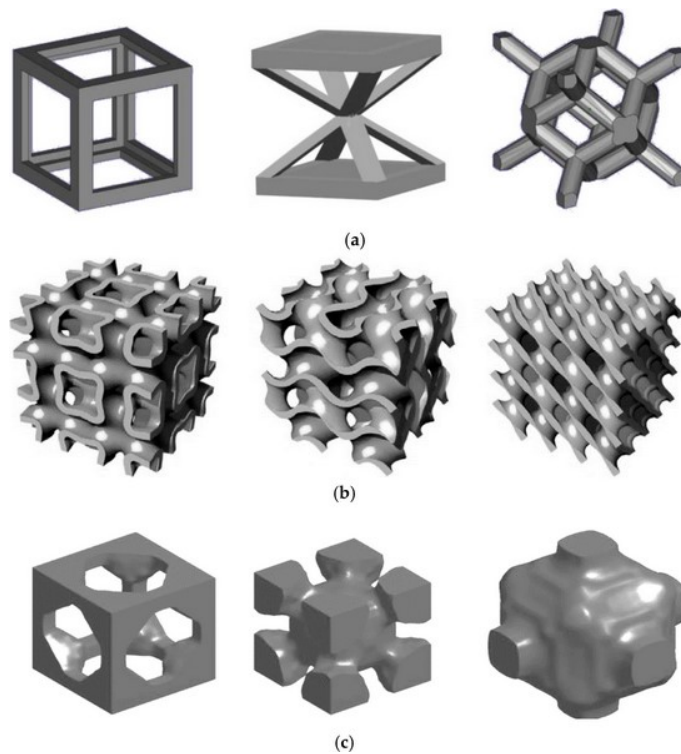


Figure 2.11: Different unit cell designs of lattice structures used in biomedical implants. (a) CAD-based unit cells; (b) Implicit surface based unit cells; (c) Topology optimized unit cells [44].

2.3.1.2 Stochastic Lattice Structures

A random probability distribution is used to define the distribution of cell patterns and shapes in stochastic lattice structures. Although these randomized structures can be statistically approximated and roughly analyzed, they can never be precisely predicted or duplicated. Naturally occurring stochastic structures examples are: bone, cellulose aggregates in wood, and collagen aggregates in cartilage [43].

According to recent findings, stochastic structures, like those produced by Voronoi tessellation, exhibit minor amounts of anisotropy in contrast to periodic structures, which are thought to be isotropic. This is due to flaws and defects brought on by the stochastic structure's random irregularities, which causes higher strains in some cell's walls [46].

Currently in the field of lattice structures and generative design, about 98% of the current research is focused on periodic lattice structures, leaving the field of stochastic structures relatively untapped [43].

2.3.1.3 Voronoi Structures

Voronoi tessellation is an irregular biomimetic pattern based on the Voronoi diagram, which is very similar to a structure that would result from a natural growth process and natural structures, such as dragonfly wings, vegetables, animal fur patterns, and muscle cells [46][47][48].

Due to its ability to mathematically describe natural models, Voronoi tessellation has had an exponential growth in many scientific areas, with applications going from biology and engineering to astronomy [46][47].

A Voronoi diagram is a formula that divides metric space into a number of areas based on distances from a particular collection of discrete elements [46][47].

As shown in Figure 2.12, the definition of a set of points known as seeds initiates the construction of the Voronoi structure. By using Delaunay triangulation and connection of bisector lines, the resulting cells form the basis of the Voronoi structure [46].

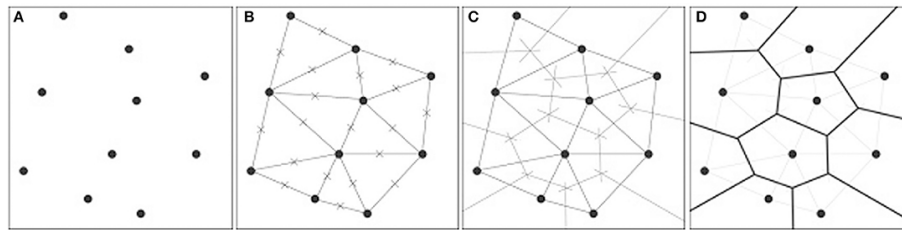


Figure 2.12: Generation process of the Voronoi diagram. (A) Distribution of the seeds within the design domain, (B) Delaunay triangulation, (C) Identification of the bisector lines, (D) Voronoi tessellation [46].

By modifying or adding parameters during the Voronoi tessellation process, the geometry and arrangement of the cells within a given domain may be changed after the Voronoi structure has been obtained to meet the needs of the project. This iterative design will alter the cell geometry, directly affecting the Voronoi structure, leading to Voronoi structures that are optimized [46].

2.4 Additive Manufacturing

Additive manufacturing (AM) is a revolutionary manufacturing process that builds components layer by layer, typically from CAD models. Unlike traditional subtractive manufacturing methods that involve cutting or shaping material to create a final product, AM adds material precisely where it's needed, from material under the form of powder, filament, or liquid. This technology allows for greater design flexibility, reduced waste, and the production of complex geometries that would be challenging or impossible to achieve with conventional methods. AM is used across various industries, including aerospace, medical, and automotive, offering new possibilities for innovation and customization.

In the early days of AM technologies, rapid prototyping was the primary benefit of the technology. However, with the current surge in development of AM, there is a worldwide large-scale implementation at an industrial level, leading to the need to characterize individual AM applications and their scope in a way that reflects the rapid and continuous evolution of AM technologies [49].

Currently, AM of metal components is mainly performed using the selective laser melting (SLM) technology, which melts and fuses layers of metallic powder into near-net shaped parts,

using a high power density laser [50]. In the AM industry, similar technologies can have different denominations, due to different manufacturers and used patented technologies, for example, SLM is a patented technology, and the technology technical name is PBF (Powder Bed Fusion) [51].

As previously mentioned, to better understand the reach of AM technologies, there is a need to identify and discuss the current applications of AM, as can be seen in Figure 2.13, where functional parts, tooling components, and prototypes, are currently being manufactured across a series of sectors, such as industrial machines, aerospace, biomedical, and automotive industries [52].

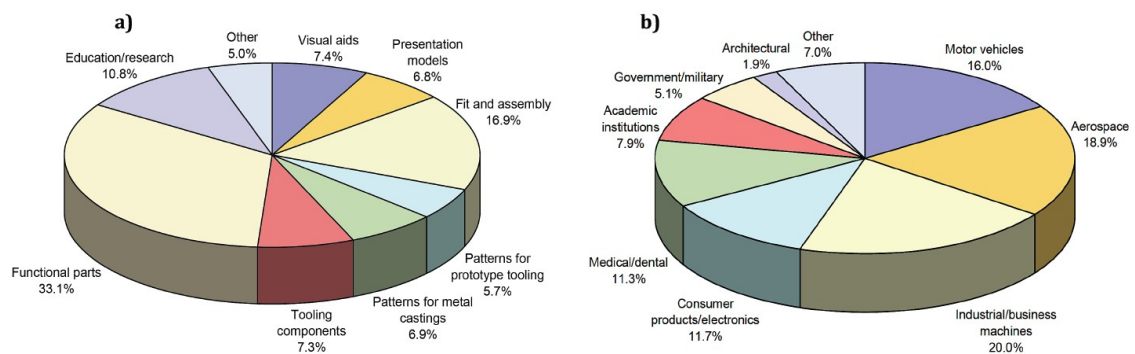


Figure 2.13: a) Graphical representation of AM applications. b) Main industry sectors of AM [52].

Over the past few decades, AM has experienced remarkable and exponential growth in both industrial applications and innovative advancements, with an impressive average annual growth rate of 15% (Figure 2.14). To provide context for this growth, the industry has expanded sevenfold over the last decade alone. In 2022, the global income generated by the AM industry reached a substantial figure of 18 billion dollars (Figure 2.14), highlighting its increasing significance and impact across various sectors [53].

The advantages of AM over more traditional manufacturing techniques include the manufacture of complex and intricate designs that are often impossible or very costly to achieve using traditional manufacturing methods, and made it possible to create components using cutting-edge design methods, such as topological optimization and other generative design techniques. The technology is well-suited for rapid prototyping, allowing designers and engineers to quickly iterate and test their concepts, reducing development time. This flexibility enables the creation of lightweight, optimized, and innovative structures. Traditional manufacturing methods often produce significant material waste, while AM minimizes waste by using only the material necessary to create the component, and reducing the necessity of special tooling [49][51]. It enables customization of products, making it ideal for producing unique, personalized items, such as medical implants. These advantages can result in significant financial savings [53]. AM has become specially important in low volume industries that prioritize performance over cost, such as automotive, biomedical, and aerospace. But due to its constant growth, AM technologies are increasingly used in more and more industries, and are now not only limited to prototyping and niche fields [54].

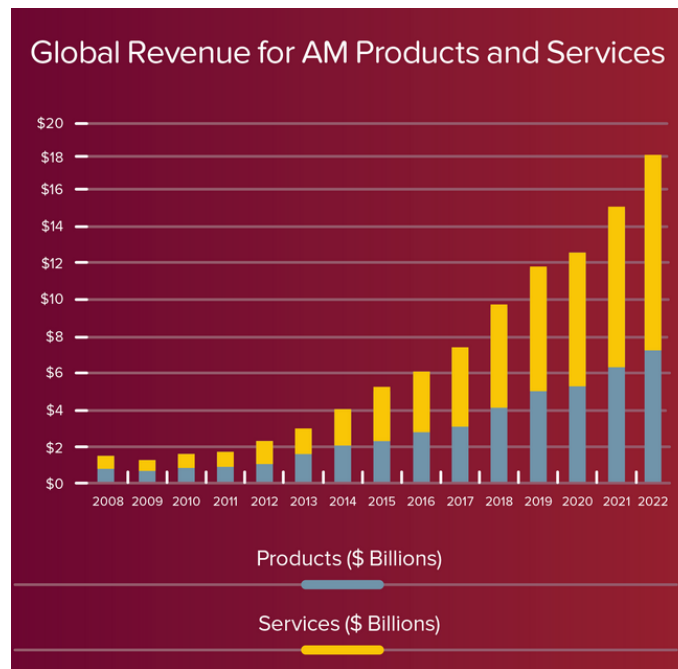


Figure 2.14: Worldwide generated income by AM in services and products in the last decades [53].

AM technologies also have their disadvantages, including the limited selection of materials compared to traditional manufacturing processes, with raw materials being more expensive due to special manufacturing methods necessary to produce them. Parts produced using this technology are usually near-net shaped and need post-processing to meet specific requirements, which adds time and cost. The size of components that can be produced using AM is limited by the size of the machine, which is usually inferior to traditional methods. The production process can be slower and less cost-effective than traditional methods for producing large quantities of parts, making it less suitable for mass production. Consistency and quality control is still a challenge in the current state of development of AM technologies. The use of high-quality AM equipment can be expensive to purchase and maintain, especially for industrial-grade applications [49][51][54].

Given the variety of terms used to describe AM technologies, it was necessary to specify a global scientific meaning in a standardized manner. The ASTM F2792 standard is currently in charge of defining terminologies in the AM industry. According to this standard, AM is described as: "A process of joining materials to make objects from 3D model data, usually layer upon layer, as opposed to subtractive manufacturing methodologies." [55].

2.4.1 AM in the Biomedical Field

As an emerging technology, AM has the potential to significantly contribute to the advancement of the biomedical field.

AM is highly attractive for biomedical applications, particularly in the creation of patient-specific products. It enables the design and development of customized medical devices, anatomi-

cally accurate body parts, implants, porous scaffolds, and even the fabrication of living cells, revolutionizing the possibilities in surgical and dental implants and various other aspects of healthcare [56][57].

Due to the lack of suitable donors and the complexity of the procedure, organ transplantation is still very difficult. In this circumstance, 3D printing of organs is becoming more and more practical. In recent years, kidney and heart organ models have been developed that, when implanted into human bodies, will function as the original organs [56].

Skin tissue fabrication is a new bio-printing technique used to create full-thickness, two-layered, three-dimensional skin that is cell-oriented. Similar to this, the creation of scaffolds is becoming more crucial in tissue engineering for the repair of damaged body parts like bones and nerves. Newly formed tissues are structurally supported by scaffold design, which also permits cell growth in specific directions [56]. Biodegradable scaffolds are one of the most recent and important advances, as they permit degradation of the scaffold and its disappearance from the body, reducing discomfort and recovering time of the patient.

The most frequently used AM technologies in the biomedical field are extrusion printing/bioprinting, stereolithography (SLA), fused deposition modelling (FDM), selective laser sintering (SLS), selective laser melting (SLM), electron beam melting (EBM), and direct ink writing or inkjet bioprinting (DIW) [56].

Thermoset photopolymers are typically used in stereolithography, but there have been a number of applications in the field of ceramic biomedical devices. Additionally, it is employed in the creation of dental implants and cardiovascular therapies [56].

Technologies such as selective laser sintering, electron beam melting and selective laser melting are normally suitable for metals like stainless steel, cobalt-chromium alloys and titanium, which are mainly used in orthopedic implant applications [56][58].

AM is one of the most efficient technologies for the biomedical field, which is used to create patient-specific scaffolds, medical devices, bone and tissue implants, surgical tools, and organ models. Its use results in quick, cost-effective productions while minimizing post-operative complications and surgical problems. The quality of life for many patients is anticipated to increase as AM develops further. Even so, there are still some difficulties in the field of biomedical AM, including material restrictions, high investment costs, and a lack of fully optimized manufacturing techniques. With a view to maintaining its exponential growth and innovation, future efforts in this field will focus on the creation of new and more effective designs and materials [56][57][58].

2.4.2 Hybridization of AM

Currently, enhancing the geometric tolerances of the AM process without significantly increasing costs or sacrificing manufacturing speed remains a challenge. This limitation persists until further innovations and breakthroughs in the field are achieved [59]. Consequently, meeting the requisite precision and tolerances for specific applications, such as those in the automotive, medical, and aerospace sectors, remains an ongoing challenge.

To address these challenges, the majority of components or parts produced through metal AM typically require some form of post-processing [59][60]. The objective of this post-processing is to eliminate AM-specific characteristics, such as the staircase effect depicted in Figure 2.15. This is critical because significant variations in surface roughness and tolerances can have adverse effects on the mechanical performance and accuracy of AM components. These challenges necessitate the use of hybrid processes to enhance the quality of AM parts, as illustrated by the difference in surface quality between an AM as-built part and an AM part subjected to machining post-processing in Figure 2.16 [60].

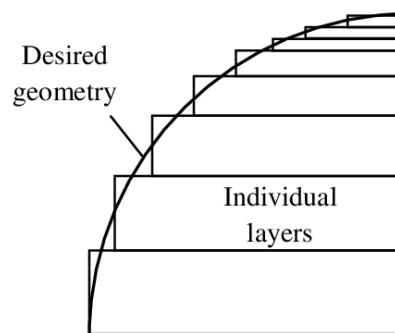


Figure 2.15: Staircase effect, highlighting the need for post processing [61].

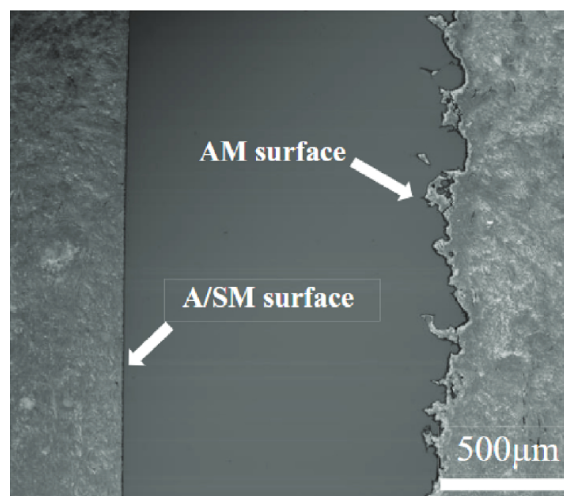


Figure 2.16: Comparison of an as-built AM Surface and hybrid processed machined surface [60].

2.4.2.1 Hybrid SLM

The integration of built-in machining capabilities within AM machines represents a notable advancement in the realm of hybrid AM processes. At present, there are SLM machines equipped with 3-axis milling systems readily available. This milling process is conducted between layers,

directly within the powder bed, allowing for the machining of each component layer, as illustrated in Figure 2.17 [62]. This hybrid approach combines the benefits of additive and subtractive manufacturing techniques, offering improved precision and surface quality in AM-produced parts.

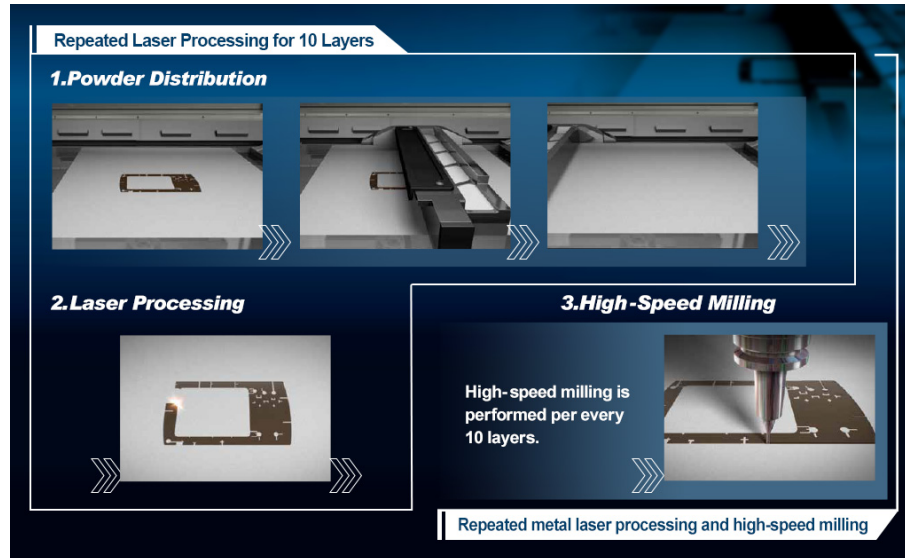


Figure 2.17: Hybrid AM process developed by Matsuura where an in built milling system, performs a machining operation every 10 layers during the AM process [63].

2.5 Selective Laser Melting

Selective Laser Melting (SLM) is an Additive Manufacturing (AM) technique that was originally developed in 1995 under license from EOS GmbH. While EOS GmbH played a pivotal role, it's important to note that SLM technologies were also made available by various other companies. This manufacturing process employs a high-intensity laser to selectively fuse metallic powder, layer by layer, onto a powder bed. Crucially, the entire process unfolds within a closed processing chamber. The fundamental concept of the SLM process is visually depicted in Figure 2.18 [64].

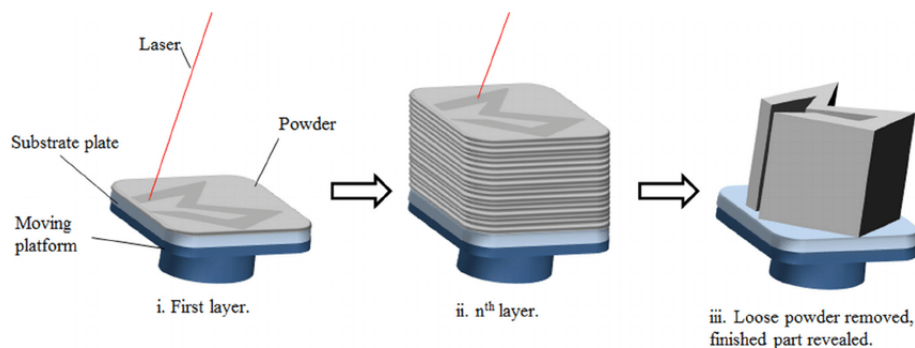


Figure 2.18: Selective Laser Melting Concept [64].

In the SLM process, a base plate, ideally made of the same or a compatible material for improved adhesion, serves as the substrate for the entire construction. Initially, a thin layer of metal powder is evenly spread onto this base plate to initiate the process. Once the powder deposition is complete, a high-intensity laser beam is employed to selectively melt the metal powder, following the cross-section of the object's current layer. As the melted powder cools, it solidifies and fuses together, forming a solid layer. This layer-by-layer approach is iteratively repeated for each subsequent layer, progressively building up the object from the bottom to the top. Once the construction of the part is finished, any loose and unprocessed metal powders are carefully removed. To separate the part from the substrate, a common technique involves using Electrical Discharge Machining (EDM) [64]. A more detailed working principle of the SLM process is visually depicted in Figure 2.19.

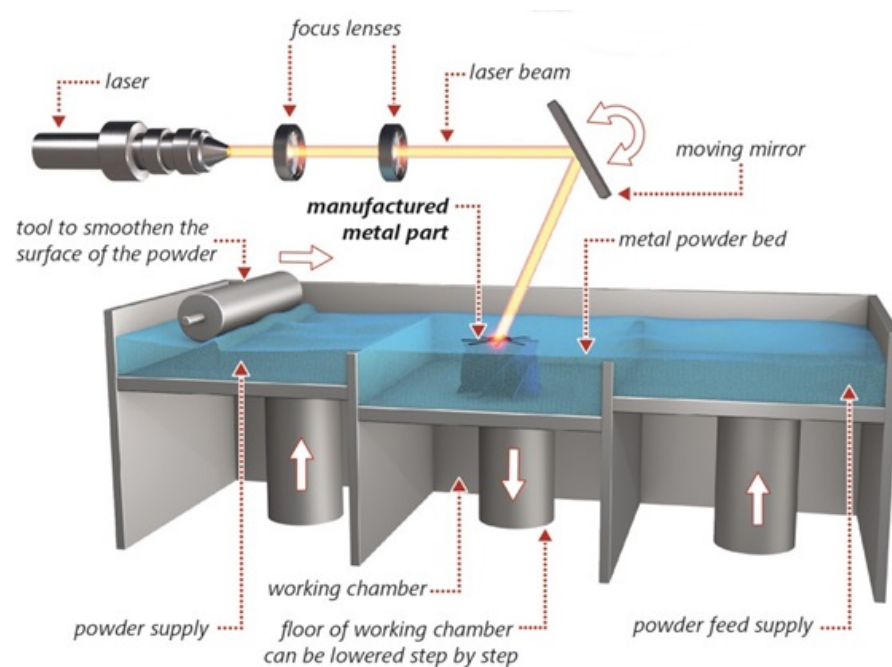


Figure 2.19: SLM working principal [65].

In SLM, the powder is coated on the substrate, with the aid of a special blade, in layers ranging in thickness from $20\ \mu\text{m}$ to $100\ \mu\text{m}$. This variation in thickness is the result of a necessary compromise between the desire for fine resolution and good powder properties. Some SLM machines also have preheating systems for the building chamber or the base plates, which act as the substrate [64].

The build chamber is filled with inert gases (nitrogen or argon) during the SLM process, creating an atmosphere that is continuously regulated. The purpose of using these inert gases is to stop the metal powder from reacting with other gases and to prevent the component from oxidizing at high temperatures [64].

The main process parameters of SLM are: hatch spacing, scanning speed, layer thickness and laser powder, which are represented in Figure 2.20, as well as scanning strategy and spot size [64].

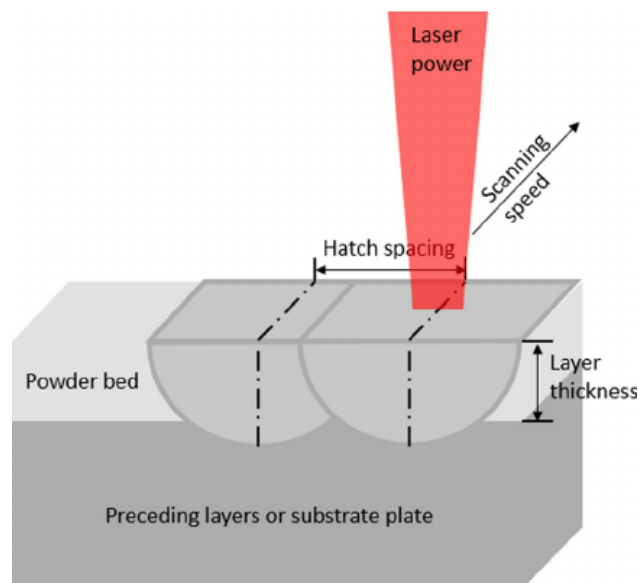


Figure 2.20: SLM processing parameters [64].

Support structures are a crucial element in the SLM process, as well as in all AM processes. Their primary function is to serve as a means of dissipating heat and providing mechanical support for intricate and suspended geometries that might otherwise deform without such support [66].

These support structures typically take on various forms, as illustrated in Figure 2.21, and they must be removed during post-processing because they do not form part of the functional component. Given their temporary nature and role as support during the manufacturing process, optimizing these structures is imperative to reduce production time and minimize material usage. Furthermore, support structures should be designed for easy removal, as any difficulties in this process can potentially lead to damage to the final part [66].



Figure 2.21: Example of SLM support structures [67].

2.5.1 Process Parameters

With an emphasis on SLM, this section will go over some of the crucial process parameters used in AM processes. The proper use and optimization of these parameters is essential for the advancement of AM technologies since they directly affect the quality of the parts produced.

Iterative testing and optimization are frequently required to find the ideal combination of these interrelated parameters for a particular material and part design. These parameters are used by manufacturers and researchers to strike a balance between the need for productivity and speed and the need for high-quality, functional parts.

2.5.1.1 Scanning Strategy

Scanning strategy refers to the specific pattern and sequence in which the laser scans over the powder bed to selectively melt and fuse the metal powder particles, layer by layer, in order to build a component. The choice of scanning strategy plays a crucial role in determining the final part's quality, accuracy, microstructure, mechanical properties, and residual stresses. The choice of scanning strategy depends on various factors, including the geometry of the part, material properties, desired mechanical characteristics, and the SLM machine's capabilities [68].

The most common scanning strategies in SLM are the uni-directional (Figure 2.22 a) and bi-directional scanning (Figure 2.22 b), with both having long scanning vectors. The chessboard scanning strategy (Figure 2.22 c) is achieved by dividing the printing zone into square areas. Figure 2.22 (d) and (e), use uni and bi-directional strategies with alternating vector order, in order to improve temperature distribution. The helix scanning strategy (Figure 2.22 f) gradually reduces the vector size. The contour scanning strategy (Figure 2.22 g) continuously scans the contour of the part, and then hatches the area inside the contour. Figure 2.22 (h) represents a re-scanning with multi-passes of the laser. Figure 2.22 (i), (j), (k), (l), and (m) show the rotation of scan vectors between layers. Figure 2.22 (n) displays the point melting exposure scanning strategy, where it selects one point at a time and creates a discontinuous scanning strategy [68].

When it comes to the previously explained scanning strategies, long scanning vectors cause excessive and accumulated stress, which has a negative impact on the quality of the parts. Therefore, scanning strategies with smaller vectors can produce larger and higher quality parts. The biggest difference among the scanning strategies is the level of re-melting which generally improves the densification, surface roughness and defects of the components. More complex scanning strategies with repeating and rotating passes usually lead to better quality parts [68].

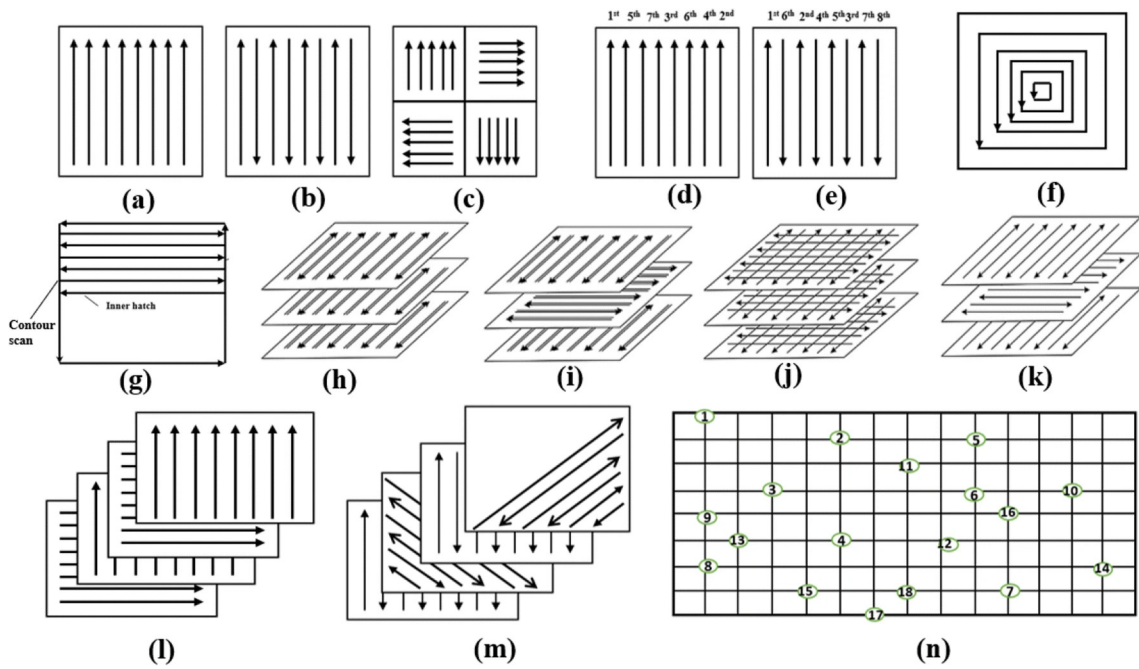


Figure 2.22: Different scanning strategies used in the SLM process [68].

2.5.1.2 Scanning Speed

The laser beam's speed as it scans the powder bed is referred to as scanning speed. It affects how energy is distributed among the layers and has an impact on part quality. The scan speed is one of the main parameters of AM technologies and has a high influence on the final properties. Slower scanning speeds improve control and accuracy but may lengthen build times. Increased productivity may be achieved through the use of faster scanning rates, but the part's quality must not be sacrificed [69].

2.5.1.3 Laser Power

The amount of energy delivered to the powder bed is determined by the laser power, which is a crucial SLM parameter. Laser power is a parameter that may vary during the construction stages. This means that it is possible to produce different areas of the part with different powers. More intense melting and deeper penetration into the powder are produced by higher laser power. To avoid defects like excessive porosity or material spattering, this parameter must be carefully controlled to avoid overheating [70].

2.5.1.4 Spot Size

The spot size refers to the diameter of the laser beam at the point where it interacts with the material. A smaller spot size concentrates the laser energy into a smaller area, which can lead to higher energy density. This increased energy density can be beneficial for precision work or for

achieving finer details. However, as spot size is reduced, the energy density increases, leading to issues such as excessive heat, vaporization, or spattering of the material [69].

2.5.1.5 Hatch Spacing

This parameter defines the distance between successive scans during the process of building a layer (Figure 2.23). It affects the density of the part and the overlap between subsequent scans. By setting hatch spacing correctly, gaps or excessive overlaps that might impair part quality are prevented and uniform material distribution is ensured. In the microstructures of the materials produced by AM, it is common to verify the overlapping of the melting pools in the same layer [69][71].

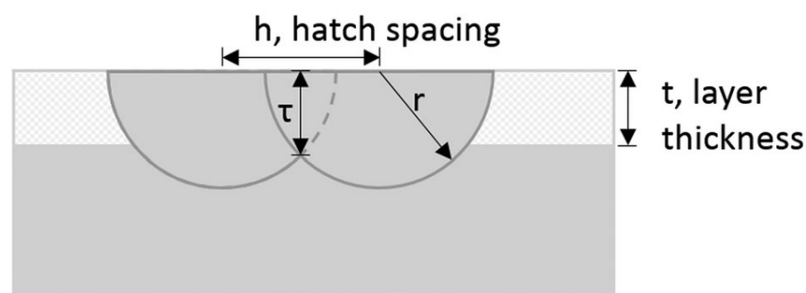


Figure 2.23: Illustration of hatch spacing and layer thickness [71].

2.5.1.6 Layer Thickness

Layer thickness determines the height of each deposited layer, as shown in Figure 2.24. It has an impact on the part's surface finish and resolution. Finer details are produced by thinner layers, but the part may need to be built in more passes, increasing the build time. Although thicker layers may compromise surface quality, they can speed up construction. The physical characteristics of the powder, such as flowability, as well as the material of the spreading blade, the spreading speed, and the atmosphere conditions of the build chamber directly affect how uniformly the new layer deposition occurs [72].

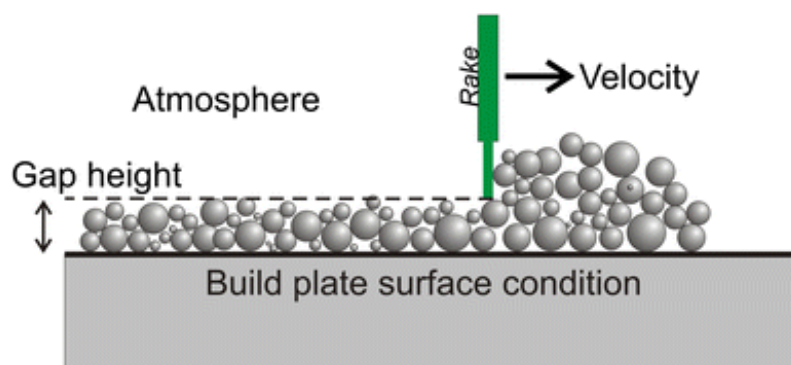


Figure 2.24: Schematic of the deposition of a new layer [72].

2.5.1.7 Melt Pool Geometry

The geometry of the melt pool plays a pivotal role in influencing the mechanical properties of materials and structures during the solidification process. Specific parameters such as scanning speed, scan distance, power, and layer thickness exert significant influence on the geometry of the melt pool [69].

2.5.1.8 Pre-Heating Temperature

The printing process often involves the creation of high thermal gradients, which, in turn, lead to the generation of significant residual stresses and thermal distortions within the components being manufactured. One effective solution to mitigate these issues is to preheat the powder bed. This preheating process serves to reduce the magnitude of thermal gradients, subsequently resulting in a decrease in the level of residual stresses and distortions present in the final components [69].

2.5.1.9 Building Atmosphere

The atmosphere within the SLM process is a pivotal factor that significantly influences the quality and success of metal part production. Precise control of the building atmosphere inside the SLM chamber is essential to create specific conditions that optimize the printing process. One critical aspect of this control is shielding the melting pool from oxidation at high temperatures. By creating an environment that minimizes exposure to oxygen, the risk of oxidation and its adverse effects on the metal being processed are mitigated. Additionally, the flow of gas within the chamber serves the purpose of removing hazardous fumes and particles [73].

2.5.2 Metallic Powder

Controlling the properties of metallic powders, which serve as the raw material for the SLM process, is crucial for maintaining consistent quality across each part and layer. To reduce costs, metallic powder is often recycled and reused in subsequent printing jobs. Although the properties of reused powder may differ from the virgin powder, proper control and limited usage percentages ensure that it doesn't negatively impact the final components [74][75].

In Figure 2.25, a combination of both new and recycled powder was utilized in the manufacturing of the component, showcasing how a balanced approach can be employed for cost-effective production.

According to the flowchart in Figure 2.26, there are three main steps in the production of metal powders for AM. The material must first be extracted from its ores to create the used pure or alloyed metals. Production of the powders takes place in the second stage, and validation of the powders is covered in the third stage. The most common and efficient way to produce powders with the desired properties is through atomization processes [77].



Figure 2.25: Production of metal components by SLM from GE Additive [76].

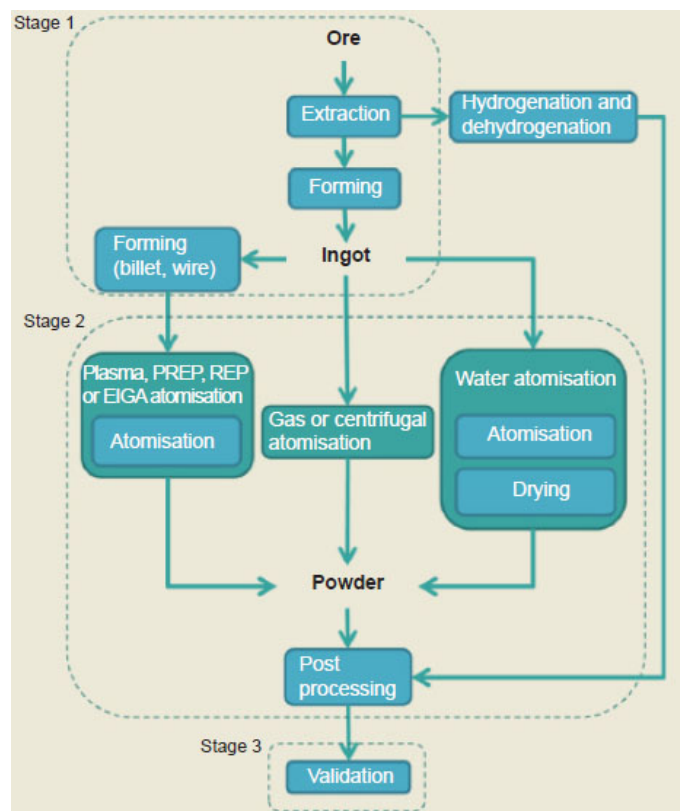


Figure 2.26: Scheme of the steps for the production of metallic powders [77].

2.5.2.1 Powder Properties

The surface morphology of metal powders is a crucial property, and one key aspect to consider is the presence of satellites or aggregates of smaller particles on their surface. These satellites can adversely affect the flowability of the powder, potentially leading to defects in the manufacturing process. It's important to note that the production methods used to create metallic powders play a significant role in determining the quantity of satellites generated. Therefore, the choice of manufacturing method can impact the quality and performance of the powders in various applications [78][79].

During the SLM process, the fusion and remelting of metal powders lead to the merging of satellites with other particles, effectively causing them to disappear. This phenomenon means that the reuse of metal powders can gradually reduce the presence of satellites, as illustrated in Figure 2.27. However, it's important to note that reusing powders can also result in surface wear of the particles, leading to a decrease in their spherical shape. This wear and change in particle morphology should be considered when evaluating the quality and performance of reused powders in the SLM process [80].

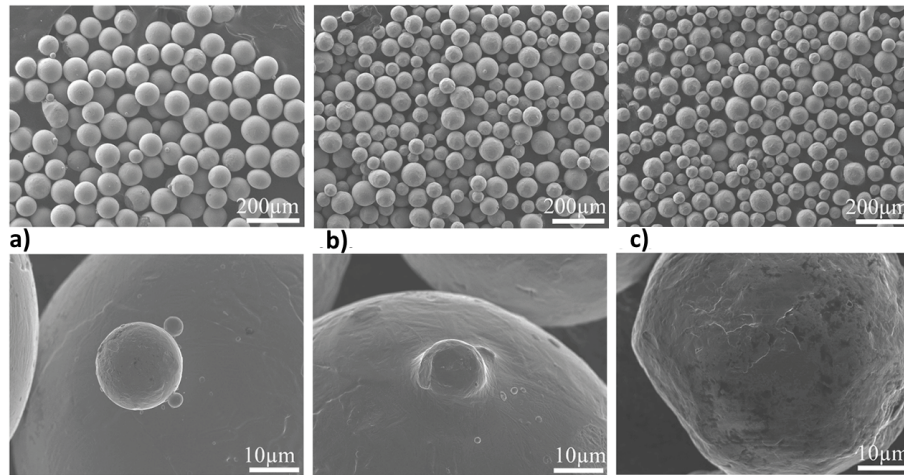


Figure 2.27: Morphology and surface features of reused Ti-6Al-4V alloy powder, with number of times of reuse being 2 in (a), 11 in (b), and 21 in (c) [80].

The manufacturing process of metallic powders can introduce various types of imperfections, including voids, porosities, and inclusions. When these powders are used in the SLM process, the porosities can be released into the melting pool and become trapped within the liquid metal. Consequently, this can result in defects in the final component after the solidification process, in the form of porosities. These porosities can significantly impact the quality of the finished SLM component, affecting both the structural integrity and overall performance of the manufactured part [72]. The effect of powder porosity in the melt pool is depicted in Figure 2.28.

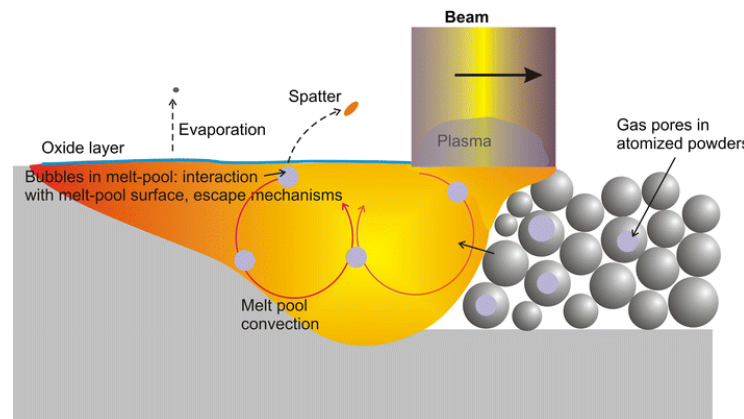


Figure 2.28: Schematic illustration of melt pool defect phenomena during PBF processes [72].

2.5.2.2 Powder Production

The techniques used in metal powder production have a direct impact on the quality of the final components, as previously mentioned. Among these techniques, atomization stands out as the most prevalent, efficient, and cost-effective method of production. Therefore, it is essential to delve further into the details of this method to understand its significance in achieving high-quality metal powders for various applications.

The casting of the desired metal or metal alloy serves as the initial step in all atomization processes. In order for the metal to turn into powder, the process involves pouring of the molten metal. The three most common atomization techniques are plasma, gas, and water atomization [77][79].

In water or gas atomization processes, the liquid metal enters the atomization chamber and falls freely under the influence of gravitational forces, taking on a spherical shape. Jets of gas or water are directed at the liquid metal, causing it to disperse into particles. These particles subsequently cool and solidify into powder [77][79].

In contrast, the plasma atomization process employs jets of ionized gases at high temperatures (plasma) to achieve atomization. This method results in the production of more spherical powder particles compared to other processes. Additionally, in plasma atomization, the plasma itself is responsible for melting the metal, contributing to the unique characteristics of the resulting powder [77][79].

2.5.2.3 Powder Geometry

As previously mentioned, the choice of powder production method directly influences the geometry achieved during the solidification process, which is especially critical in SLM processes.

SLM processes require powder with uniform particle size distributions and a more spherical shape. Figure 2.29 illustrates the significant differences in powder morphology resulting from different atomization methods. Powders produced through gas/water atomization (Figure 2.29 b) exhibit irregular morphology and a high number of aggregated satellites. In contrast, plasma atomization processes (Figure 2.29 c) yield metal powders with improved spherical morphology and a lower number of satellites. These distinctions in powder characteristics are crucial for achieving the desired outcomes in SLM manufacturing [77].

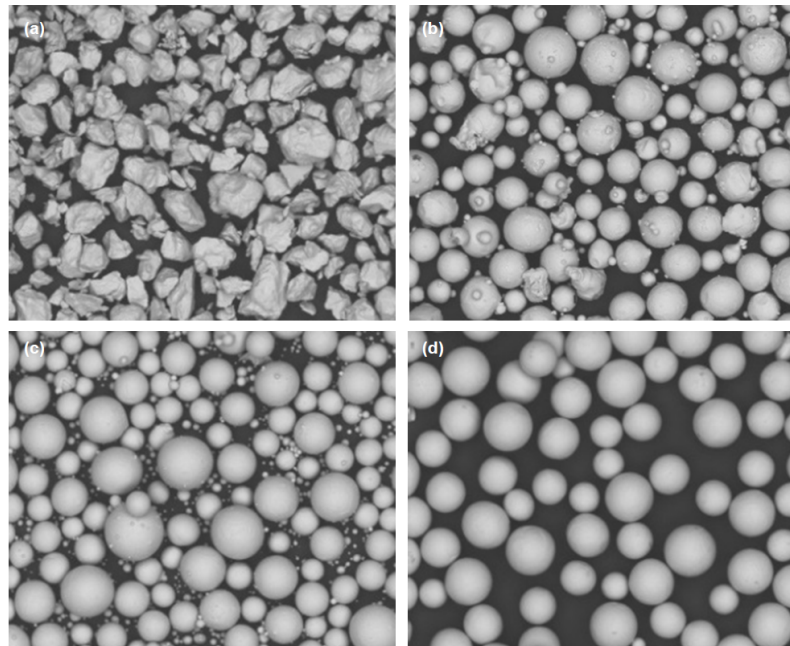


Figure 2.29: Example of SEM micrographs of typical particle morphologies obtained using: (a) HDH process; (b) gas atomization; (c) plasma atomization; and (d) plasma rotating electrode process [77].

2.5.3 Microstructure

When utilizing AM techniques, the material undergoes a complex thermal cycle that is repeated in all layers. During this process, the material is exposed to either an electron or laser beam, causing it to rapidly heat above its melting point and then quickly solidify. Consequently, many AM processes yield phases that can vary between each layer of deposited material. This variability poses a challenge in modeling and predicting the microstructure and composition of AM-fabricated parts [70].

Across different materials, AM has been observed to produce finer-grained structures compared to traditional manufacturing methods. This phenomenon can be attributed to the rapid solidification resulting from the aforementioned thermal cycle. Several process variables, including energy density, layer thickness, and pre-heating temperature, influence the temperature gradients during the process. Additionally, the geometry of an AM-fabricated part plays a role in shaping its microstructure [70].

2.5.3.1 Solidification Structure

The fusion and solidification that takes place in the material during the melting process determine the grain structure. The melt pool can have an oval, drop, curved, or keyhole shape depending on the process parameters of heat source intensity and scanning speed [69].

Through factors like the temperature gradient G and growth rate R , it is possible to analyze the microstructure of different AM materials as they solidify. This is accomplished by creating a

solidification map (Figure 2.30) using the GR and G/R combined forms. While the product GR controls the scale of the solidification microstructure, the ratio G/R determines the morphology of solidification [69].

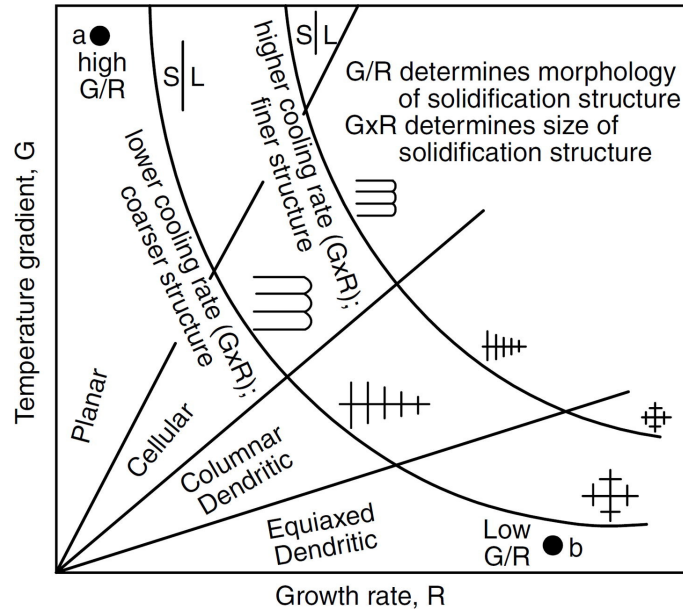


Figure 2.30: Effect of temperature gradient G and growth rate R on the morphology and size of solidification microstructures [69].

With the gradual decrease of the parameter G/R , the solidification microstructure can be planar, cellular, columnar dendritic, or equiaxed dendritic, respectively. With an increase in cooling rate GR , the dimension of all four solidification microstructures decreases. The most prevalent structures in AM processes are columnar and equiaxed [69].

In the SLM process, the remelting of old layers due to new formed layers causes growth of the solidification structures, removing surface impurities such as oxide films. Thereby producing higher quality components [69].

2.5.3.2 Grain Structure

As previously explained, the grain morphologies in most microstructures obtained by AM, are columnar structures with a high degree of grain orientation, with these structures being very common in the SLM process. These columnar structures form due to the melt pool geometry and the direction of the biggest heat transfer [70]. Columnar structures typically have anisotropic mechanical properties and are bulkier than equiaxed grains, which have more consistent mechanical properties in all directions [69].

In the PBF process, grain growth closely follows the construction direction due to fast scanning speed. In Figure 2.31 it is possible to observe the crystallographic grain growth in the construction direction [69].

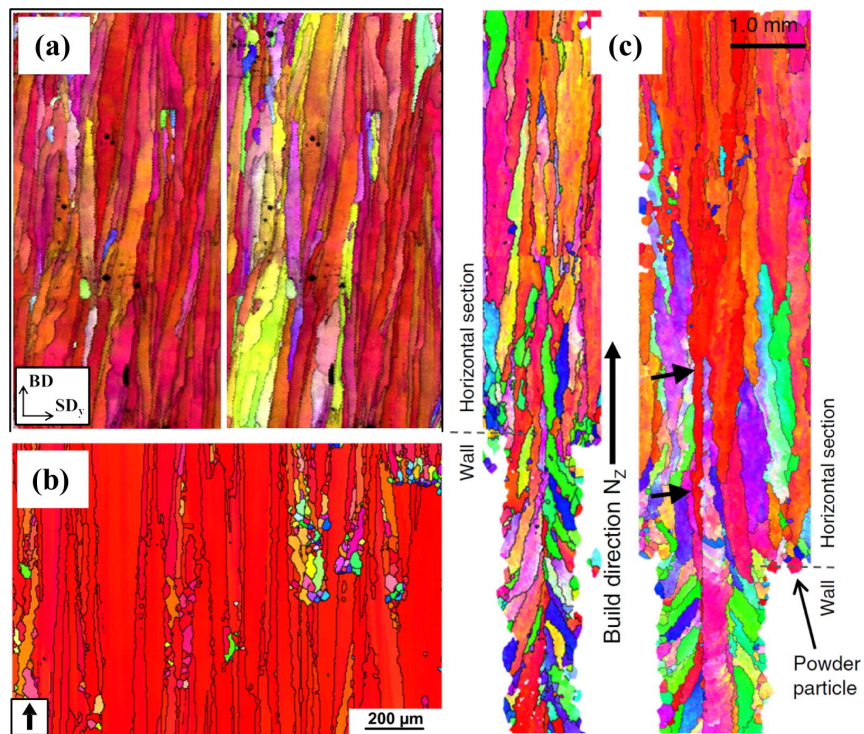


Figure 2.31: Grain orientations closely aligned with the build direction in samples made by PBF. (a) high-silicon steel, (b) IN 718, (c) Ti-6Al-4V [69].

The dimensions and shape of the melt pool have an impact on the grain structure. The process variables, material characteristics, and cooling conditions all play a role in determining the melt pool's geometry. The shapes of the melt pools created by PBF are depicted in Figure 2.32. The melt pool's shape during PBF can be seen to have a thin and elongated shape, although this shape can vary significantly depending on the used parameters [69].

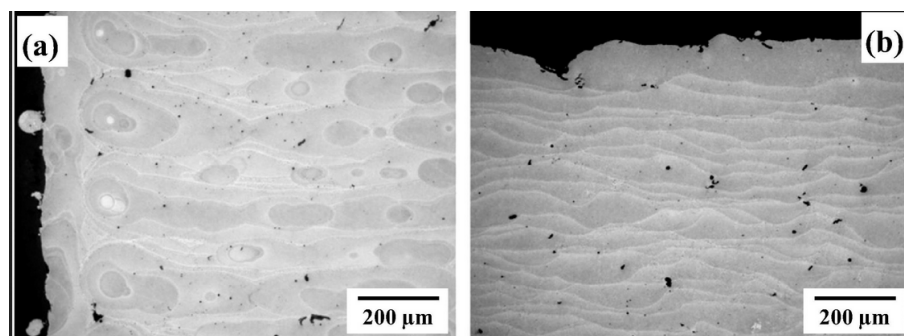


Figure 2.32: Shapes of the melt pools of AlSi10Mg produced by PBF-L. a) Top view. b) Side view [69].

The effect of undercooling, brought on by an increase in scanning speed, can be seen in Figure 2.33, where differences in grain orientation due to scanning speed are evident, with the degree of grain orientation variation increasing with the increase of scanning speed [69].

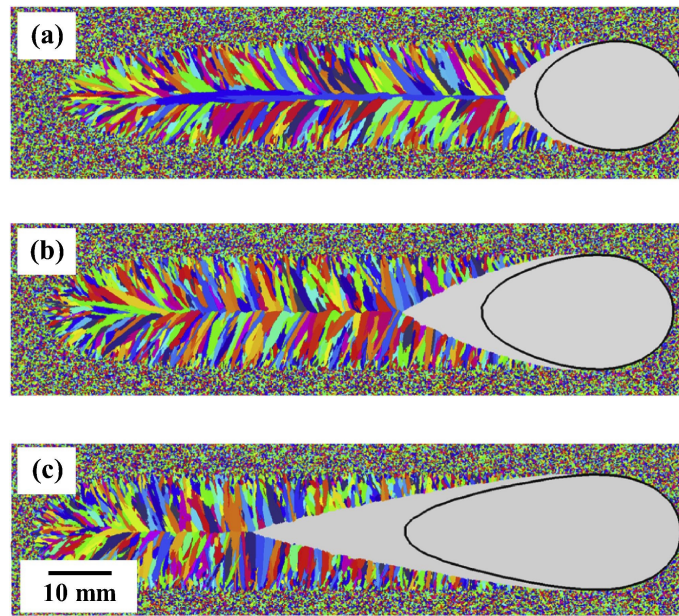


Figure 2.33: Effect of undercooling on the grain structure, with different scanning speed (a) 1 mm/s, (b) 2 mm/s, and (c) 5 mm/s [69].

2.5.3.3 Preferential Granular Growth

Figure 2.34 highlights the directions of the maximum temperature gradients for successively alternating laser scanning directions. Where it is observed that the grain orientation is influenced by the laser scanning direction. However, with certain laser scanning patterns, the actual direction of grain growth may differ substantially from the local maximum temperature gradient [69].

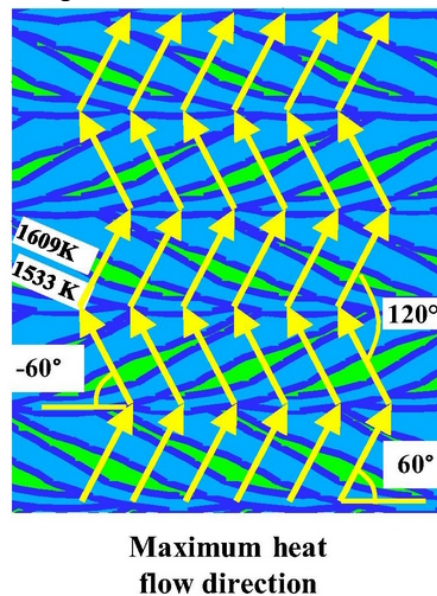


Figure 2.34: DED-L of IN 718 Maximum temperature gradient during bidirectional laser scanning, with DED-L of IN 718 [69].

2.5.3.4 Steel Microstructure

AM has been used to process a variety of steels, including maraging steels, soft magnetic high silicon steel, austenitic stainless steels, and precipitation-hardenable steels [69]. In this section, special attention is given to austenitic stainless steels, such as the 316L, since this is the material used in the experimental work of the dissertation.

Due to rapid cooling, steel components typically have a fine-grained microstructure. At the macrostructure level, these structures have a cellular structure with several smaller grains arranged in different directions, and the borders of the melt pool are visible in the microstructures (Figure 2.35) [70].

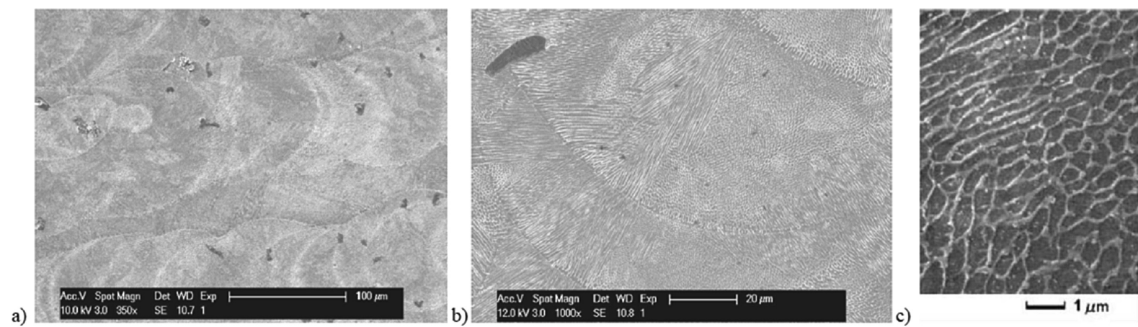


Figure 2.35: Fine-grained microstructure of maraging steel (18Ni-300) in LBM as-fabricated condition, a) top view, b) cross-section, c) magnification of dendritic structure [70].

In figure 2.36, the microstructure of stainless steel 316L produced with LMD (laser metal deposition) is displayed, showing equiaxed grains inside the melting pool, and near the borders columnar grains [70].

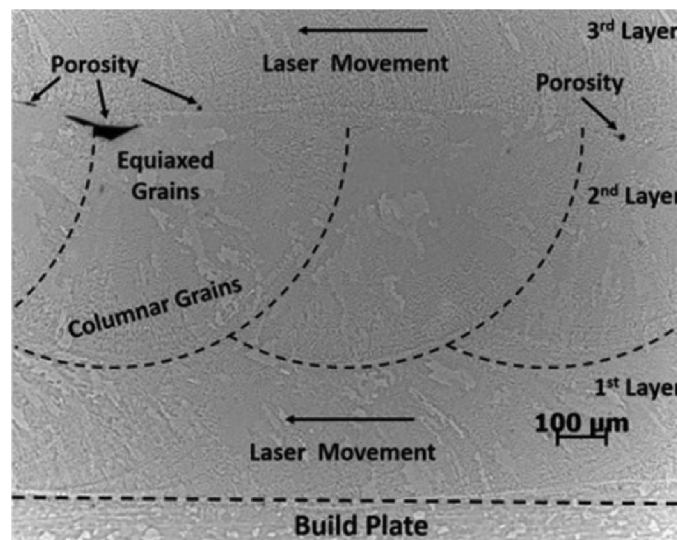


Figure 2.36: Section of three layers of LMD-produced austenitic stainless steel (316L) in as-fabricated condition, revealing the footprint of the laser tracks [70].

Figure 2.37 shows the microstructures of 316L stainless steel produced by SLM, where variations in the process parameters have an obvious impact on the melt pool's macrostructure. It demonstrates that using a stronger laser power in conjunction with remelting each layer (a technique known as the "crisscrossed strategy") leads to homogenization of the microstructure and a decrease in the number of inclusions [81].

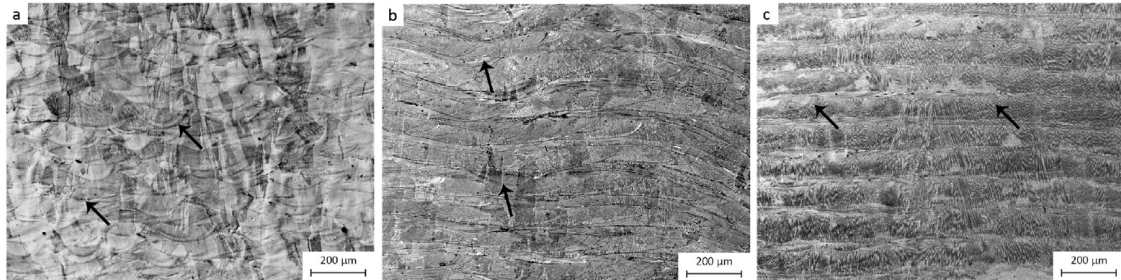


Figure 2.37: Microstructure of 316L by SLM, with three sets of parameters. (a) Alternating scanning, 100W. (b) Alternating scanning, 200W. (c) Crisscrossed xy scanning, 200W [81].

Optical microscope images of 316L by SLM using different building orientations of 45° and 90° are shown in Figure 2.38. The melt pools produced by the laser beam have a layered structure typical of SLM processes. The melt pool's orientation and overlap are significantly impacted by the building's orientation. The horizontally deposited layers in case a) with a building orientation of 90° exhibit melt pool overlap with the typical curved melt pool shape. However, when the build is oriented at a 45-degree angle, the melt pools are diagonally overlapping, due to deformation in the direction of gravity [82].

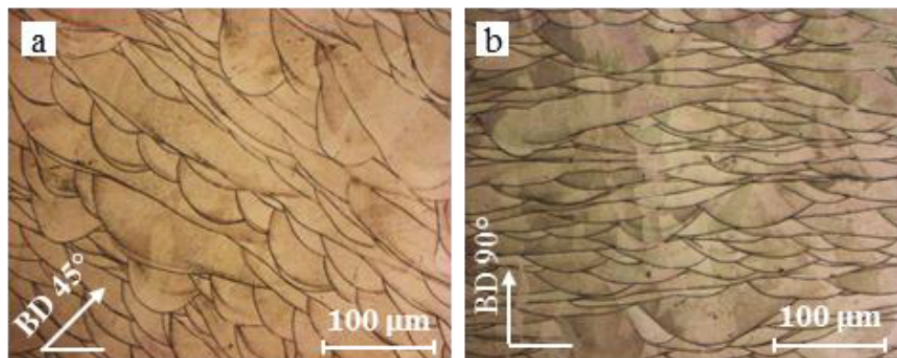


Figure 2.38: Representative optical images of 316L-SLM samples processed with building directions of (a) 45° and (b) 90° [82].

2.5.4 Defects

The next section places its focus on potential defects within the SLM process and their connection to process parameters and characteristics. Gaining insight into these defects enhances the reliability of the technology, reduces variability in both parts and machines, and facilitates the production of higher-quality components.

2.5.4.1 Surface Roughness

Surface roughness is one of the most extensively studied parameters in both AM and SLM. As mentioned earlier, physical process characteristics such as the staircase effect can significantly contribute to surface roughness in components. Additionally, processing parameters, such as scanning strategies and laser energy intensity, play a crucial role in determining surface roughness [69] [83].

Surface roughness is of paramount importance because it affects the ability to meet a part's geometric requirements and has a significant impact on the fatigue behavior of components [69][83]. In SLM, typical surface roughness values can vary depending on the processing parameters and materials used, but they can reach as low as 3 μm . In contrast, other processes like EBM may result in surface roughness values ranging between 11 and 15 μm [83].

One of the major contributors to surface roughness is the accumulation of small particle satellites that adhere to the boundaries of layers. These satellites form due to the partial fusion of metallic powder near melt pools during the SLM process, leading to an increase in surface roughness, as illustrated in Figure 2.39. This phenomenon underscores the importance of understanding and controlling surface roughness in SLM manufacturing [83].

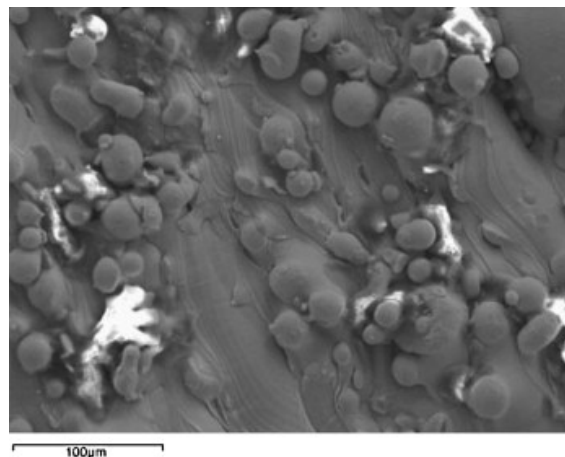


Figure 2.39: Presence of partially fused particles in a 90° surface [83].

The building direction and inclination have an impact on surface roughness as well. In perpendicular orientations to the direction of the building direction, low values of surface roughness are typically achievable, whereas with higher slopes, the roughness value quickly increases. The staircase effect is responsible for this phenomenon, and Figure 2.40 shows how the staircase effect and particle adherence are related. Figure 2.41 clearly illustrates this effect: Step edges with adhered particles are closer to one another as the sloping angle increases, increasing the concentration of particles on the surface area [83].

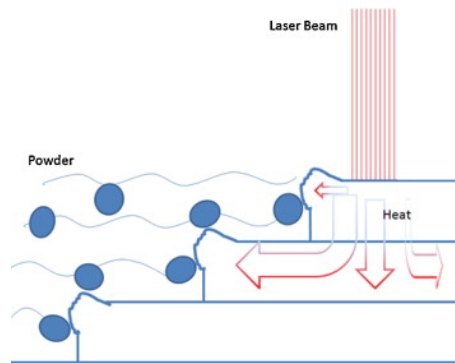


Figure 2.40: Schematic representation of particles at the layer borders, related to the staircase effect [83].

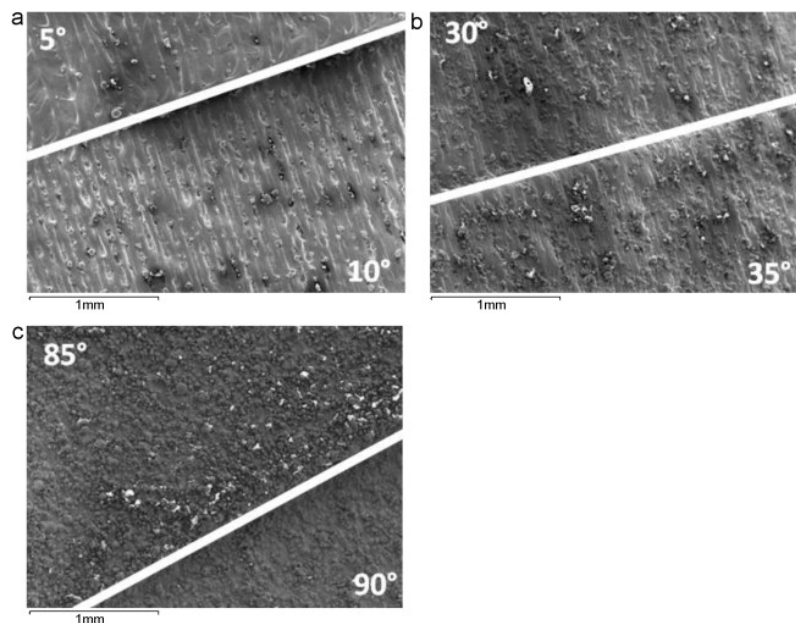


Figure 2.41: Transition between sample steps at (a) 5°–10°, (b) 30°–35° and (c) 85°–90° sloped surfaces [83].

2.5.4.2 Porosity

In some biomedical applications, such as the Voronoi lattice structures created in the GradImp project, geometric porosity is a desirable trait. However, material porosity resulting from the SLM process is an undesirable defect that promotes crack growth and degrades mechanical properties [69].

Porosity in SLM-produced parts can arise from various factors, including the raw material, process parameters, and the solidification process itself. When it comes to the powder's inherent porosity, it can be transferred to the finished part during the SLM process. Using higher-quality powder can reduce porosity levels, but this often comes at a higher cost, necessitating a balance between cost-effectiveness and porosity reduction [70].

Porosity related to process parameters typically results from inadequate laser energy intensity,

where insufficient power is applied to completely melt the material, leading to irregularly shaped porosities. Conversely, excessive laser energy intensity can also generate irregular and regular shaped porosity. Irregularly shaped porosities are caused by material spatter ejection, which expels liquid material outside of the melt pool, resulting in keyhole-induced porosities (Figure 2.42 a). Regularly shaped porosities, on the other hand, result from gas entrapment in the melt pool (Figure 2.42 b). Understanding these factors is crucial for mitigating porosity and ensuring the quality of SLM-manufactured parts [69][70].

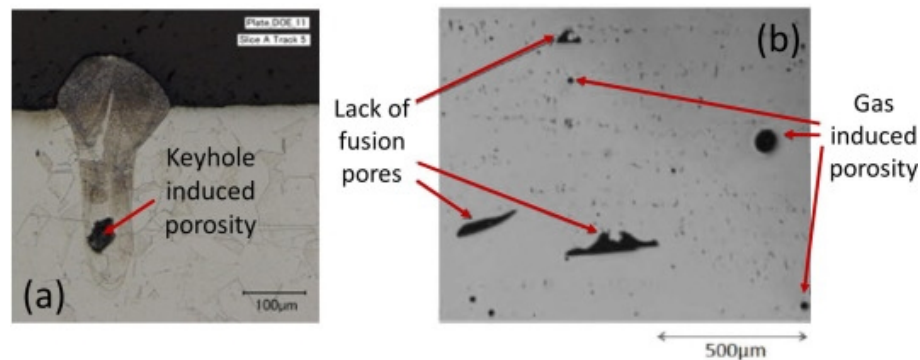


Figure 2.42: Example of different types of porosities. (a) Keyhole porosity (irregular shaped). (b) lack of fusion pores (irregular shaped), and gas induced porosity (regular shaped) [69].

Irregularly shaped porosities are more detrimental to mechanical properties than the more spherical, regular-shaped porosities. This is because irregular porosities create higher stress concentration points, which can weaken the material. However, the presence of porosity can be significantly reduced through the optimization of process parameters. By fine-tuning these parameters, it's possible to minimize porosity and enhance the mechanical properties of SLM-manufactured parts [69].

2.6 Vat-Photopolymerization

In the vat-photopolymerization (VPP) process, a light-curable resin, is placed in a vat and exposed to either visible or UV light. The polymerization reaction is triggered and manifested by the curing light, which then creates chains of polymers or crosslinks them to create solid resin [84]. Three components make up the photopolymer mixture: monomers, oligomers, and photoinitiators, as shown in Figure 2.43. Photoinitiators release reactive species when they are exposed to curing light, acting as catalysts to help monomers and oligomers form chains. The chemical-thermal chain-formation process is irreversible, and the resin that has been solidified cannot be converted back to liquid form [84][85].

Although it can be more expensive than other polymer AM systems and the components are prone to increasing brittleness over time, this rapid manufacturing technology is frequently used to produce parts with extremely high resolution and excellent surface finish [84].

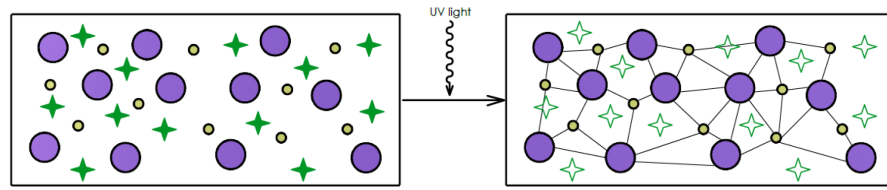


Figure 2.43: Liquid photopolymer (on the left), induced polymerization by light (small circle—monomer, large circle—oligomer, star—photoinitiator) [84].

According to the curing approach (Figure 2.44), photopolymerization is divided into different categories. These methods can use a laser such as stereolithography (SLA), use a projector such as digital light processing (DLP), and make use of LEDs and oxygen as is the case with continuous digital light processing (CDLP)/continuous liquid interface production (CLIP) [84].

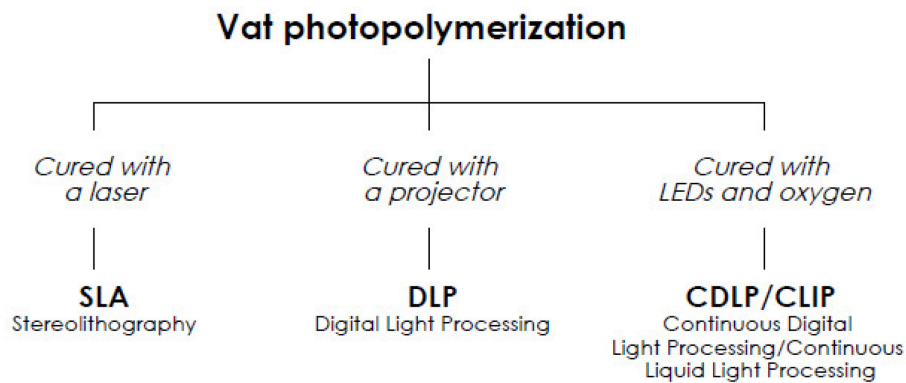


Figure 2.44: Different curing processes for vat-photopolymerization [84].

In laser-based VPP systems such as stereolithography (SLA), manufacturing begins with the deposition of a single layer of photopolymer on the build platform using a recoating mechanism, similar to SLM. A UV laser scans the required pattern on the resin surface, solidifying the liquid photopolymer. The process is repeated on subsequent layers to form a final component. Figure 2.45 a) illustrates the SLA procedure [86].

Another VPP embodiment is digital light processing (DLP), which is the process used in this dissertation. Due to its capacity to project patterns with a feature resolution as small as $30\ \mu\text{m}$, DLP has grown in popularity. In this instance, the resin is cured by projecting a 2D image of the layer that will be manufactured into the resin surface. The DLP process is presented in Figure 2.45 b) [86].

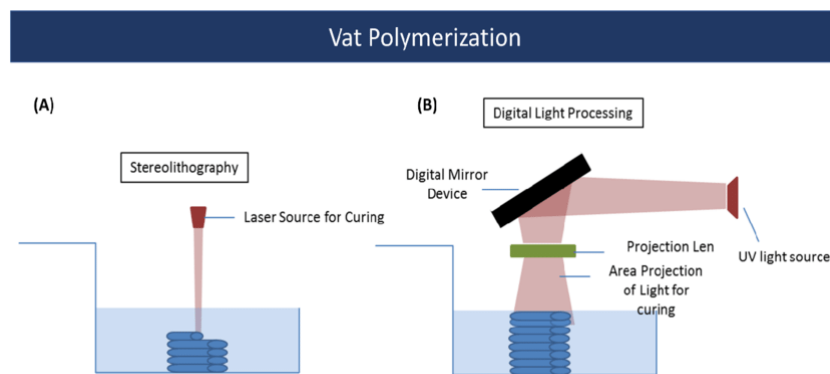


Figure 2.45: Vat polymerization: (A) Stereolithography. (B) Digital Light Processing [87].

2.6.1 Digital Light Processing

In DLP, a projector serves as the source for curing the photo-resin in a single layer through the use of 2D projection. In contrast to the SLA process, which uses UV light and cures the material point by point [85][88].

Through a single exposure of the light source through a patterned mask, the liquid resin surface forms a layer with a complete cross-section image of the component. Because each layer is completely exposed to the curing light projected from the digital screen at once rather than point by point, DLP technology is faster than SLA technology. Each layer appears pixelized when using a digital light projector, and while the accuracy of the printed part can have very good resolution, it is heavily reliant on the projector's resolution [88]. A typical DLP machine and its major components are illustrated in Figure 2.46. DLP has also been used with ceramics, hydrogels, and bioinks in a number of biomedical applications [85].

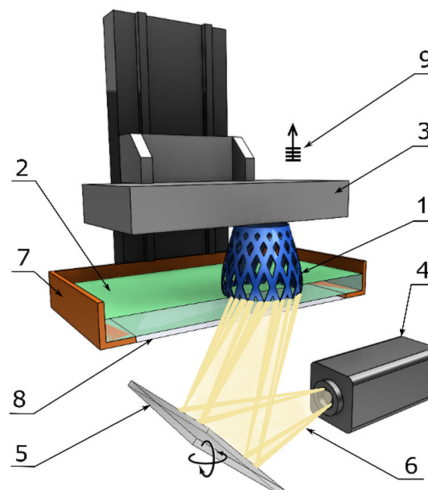


Figure 2.46: Components of a typical DLP machine: 1—printed part, 2—liquid resin, 3—building platform, 4—light source, 5—digital projector, 6—light beam, 7—resin tank, 8—window, and 9—layer-by-layer elevation [84].

2.6.2 Materials

The available materials for VPP technology have grown swiftly in recent years. Today, every resin has a unique set of mechanical and chemical characteristics as well as applications. Standard, structural, durable and tough, elastic and flexible, castable wax and ceramic, multimaterials, and biocompatible materials are the main categories of photopolymer materials [84]. These materials are currently used in VPP processes for a variety of purposes, including mechanical, biomedical, filtration, and electrical applications [89]. Figure 2.47 displays an example of a complex component obtainable through the VPP technology.

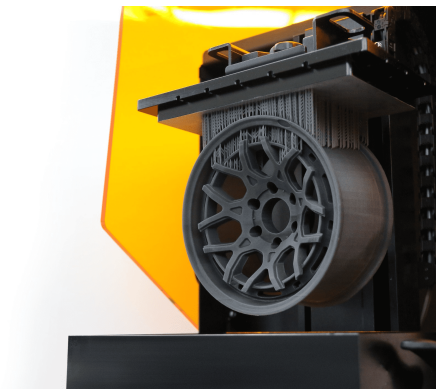


Figure 2.47: Example of detailed ABS component, manufactured using VPP technology [90].

The range of uses for standard resin is the greatest among photopolymer materials. Being the ideal choice for quick prototyping and use in applications that don't call for specific requirements. Additionally, these resins are less expensive than other types of photopolymers [84].

Structural resins can produce printed parts with remarkable rigidity as well as high accuracy. These kinds of resins can also be used to print thin walls and small details in components like turbine blades. Additionally, they can withstand temperatures of up to 289 °C [84].

Polypropylene (PP) or polyethylene (PE), which have high ductility, deformation resistance, and impact resistance, are used to make durable resin. Additionally, strong resins with high tensile strength and elastic modulus, like ABS, are available, which are ideal for functional prototypes [84].

Elastomeric polyurethane (EPU) is a class of extremely flexible and elastic polymers with low stiffness and high ductility. EPU has been widely used in orthotic and prosthetic devices because of its high elasticity [84].

In recent years, there has been a lot of interest in the fabrication of ceramic parts using the vat-photopolymerization process, with two main methods for their manufacture. In one, preceramic polymers are used for printing, and in the other, ceramic-resin composites are printed [84][91].

With the help of VPP technology, multimaterial production has advanced significantly in recent years. Multimaterials are frequently produced by mixing nanoparticles or other nanomaterials with conventional resin. When these nanomaterials are incorporated into the resin, some of the

mechanical, electrical, magnetic, and biological characteristics are partially transferred to the resin matrix [91].

Orthopedic and prosthetic uses for these materials are very common. The customization offered by 3D printing in medicine, where a product can be made specifically for a patient, is its main benefit [84]. Currently, the applications of vat-photopolymerization bioprinting encompass nervous system, cardiovascular system, musculoskeletal system, and respiratory system [91][92]. It has also been used in the production of functional human organs and tissues that are used in tissue engineering and regenerative medicine [84].

2.6.3 Process Parameters

Every AM technology has its own intrinsic process parameters, and VPP processes are no different. These parameters are essential for efficient and trouble-free production, as well as for the highest possible quality and performance of manufactured components made of any material in a variety of applications. The primary process variables of VPP technology, such as layer height, printing orientation, exposure time, and inclusion of supports, are therefore presented and explained in this section.

2.6.3.1 Layer Height

Layer height in VPP refers to the thickness of a single cured resin layer, typically ranging between 12 and 150 μm [84].

Thicker layers tend to enhance a component's mechanical properties by improving interlayer adhesion. However, they can also lead to increased porosity and internal stress, ultimately causing failure [93].

Layer height in VPP affects a component's precision and surface finish. Lower layer heights result in smoother, more detailed surfaces but require longer processing times due to the increased number of layers. This phenomenon is often referred to as the "stair-step effect," as illustrated in Figure 2.48 [84][93][94].

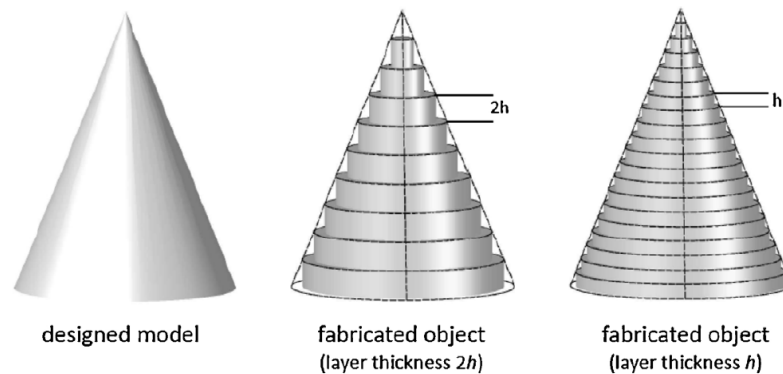


Figure 2.48: Schematic of stair-step effect resulting from layer thickness variation [94].

2.6.3.2 Printing Orientation

Printing orientation is one of the most important parameters in both VPP processes, but also in all AM processes. Printing orientation has major impact in the component's performance due to anisotropic behaviour caused by the layers predisposition [95]. Figure 2.49 represents different possible building orientations, in both the CAD file, and the final printed parts.

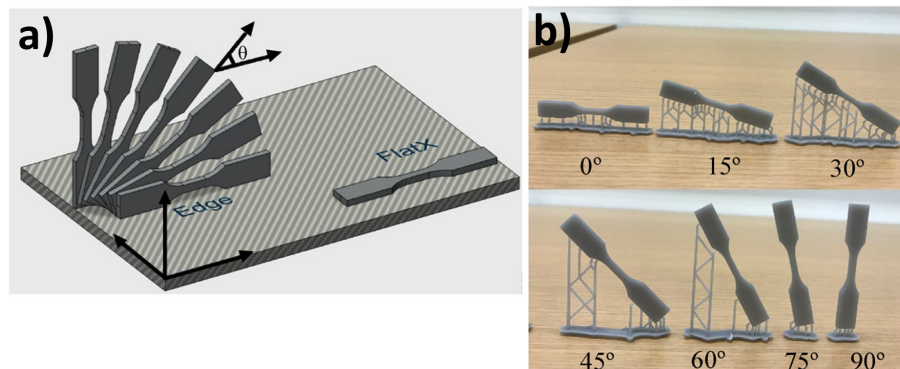


Figure 2.49: a) Orientation of specimens on build platform (CAD representation), b) final fabricated specimens [95].

Even though there are some guidelines and accepted practices for building orientation, it is crucial to emphasize that each application is unique and that each one requires a building orientation optimization in order to achieve the best outcomes.

2.6.3.3 Exposure Time

In the VPP process, the exposure time of the resin determines how long a single layer will be exposed to light. Greater polymerization on each layer is made possible by longer exposure times, which raise mechanical properties [89]. Although longer exposure times are typically preferable, it should be noted that too much exposure to light can cause the component to become excessively brittle.

2.6.3.4 Support Structures

Support structures are needed to enable manufacturing of complex components that have overhanging geometries that can collapse under gravity if not supported. The support structures always need to be removed from the final part, which combined with their use increases the process time and material consumption, raising the process cost [96]. Consequently, it is crucial to optimize the use of support structures, particularly their geometry and positioning. An excellent illustration of the effect that support structure optimization can have on the amount of material used, is provided in Figure 2.50.



Figure 2.50: Example of the application of support structures, and subsequent optimization [97].

2.6.4 Post Processing

The vat-photopolymerization process requires significant post-processing, due to both the nature of the process and type of material used (resin), namely cleaning of the resin and post-curing are necessary. These post processing procedures are further explained in the following sections.

2.6.4.1 Cleaning

In VPP processes, the printing happens inside a vat filled with resin, and as such, upon the printing job completion, the part is covered in uncured resin, both on the outside and possibly inside in any small crevices or geometry. The removal of the unused resin is empirical, both to provide a final streamlined part, as well as to prevent that any uncured resin still remaining, is cured in the post curing process [98].

There isn't currently a universal way to clean VPP components, and both industry and researchers are actively using a variety of methods and products. Various cleaning agents, including ethanol, phosphate buffered saline, plasma treatment, acetone, and hydrogel, are used as cleaning techniques [98].

The parts are first placed in a solvent bath to begin the cleaning process. Depending on the material needs, the solvent bath may then be shaken, spun, or left to soak for a predetermined period of time. An ultrasonic bath can also be used for smaller more complex parts [99]. Over-washing with alcohol-based solutions and agents that dissolve resin may cause the resin's mechanical properties to degrade [100].

Concluding, even though there is multiple evidence to the benefits of a correctly performed cleaning operation, and the consequences that can come from its absence, multiple studies confirm that there is still a significant lack of knowledge and investigation into a standard optimized cleaning strategy for VPP resin parts [98][99][100].

2.6.4.2 UV Post-Curing

The degree of polymerization in VPP-fabricated parts has a big impact on mechanical properties, surface quality, and dimensional accuracy. UV post-curing technique is frequently used as an efficient way to change the level of solidification of the components [101]. This post-processing is required because parts do not achieve optimal polymerization during the printing process, which can significantly affect the performance of the finished part [99].

In the post cure process, parts are typically rotated under UV light, and cycle times can vary from less than a minute to more than a hour, depending on the material. Overcuring can cause the parts to age and fade, which will affect its mechanical performance [99]. With parts that have complex geometries and varying wall thicknesses, it can be challenging to achieve the best material properties, which can result in parts that are fully cured in thinner areas but not in thicker zones [99].

UV post-curing is essential in the manufacture of biophotopolymers. A 2016 study found that, compared to fully cured polymers, polymers not subject to UV post-curing demonstrated significantly higher cytotoxicity [102].

Chapter 3

Materials and Methods

3.1 Scope of the Experimental Work

The experimental work aimed to understand Voronoi lattice structures for potential use in orthopedic bone implants. This understanding is crucial for tailoring implant geometry and mechanical properties to different materials and patient needs, reducing stress shielding, and improving patient outcomes.

316L stainless steel was used for sample production in SLM, due to the material's availability, optimized printing parameters, and well-studied mechanical properties. Even though the GradImp project's main goal is pure iron implants, it was not possible to use iron in the current work, as mentioned in Chapter 1. Optical microscopic analysis of the microstructure and SEM analysis of metallic powder were conducted, along with a visual inspection of various Voronoi structures, to assess their compatibility and quality with the SLM manufacturing process, offering valuable insights for future pure iron Voronoi lattice structure production.

To comprehensively study the Voronoi mechanical behavior in different materials and manufacturing methods, 316L stainless samples and resin samples were produced in SLM and vat-photopolymerization, respectively. Essential mechanical properties, described in the literature as the most relevant to orthopedic implants, were studied: Young's modulus, yield strength, flexural strength, and compressive strength. These properties were obtained through traction, flexural, and compression testing.

The mechanical characterization aimed to investigate the impact of Voronoi geometrical parameters, such as the number of points and wall thickness (mm), on mechanical properties. The study varied the number of points from 6000 to 2000 in 1000 points increments. The wall thickness study had specimens with 0.6, 0.5, and 0.4 mm that varied in 0.1 mm increments. Figure 3.1 visually represents the geometrical parameters of number of points and wall thickness.

To ensure each study was only affected by one variable, the number of points study used a fixed value of 0.4 mm for wall thickness. While in the wall thickness study, a fixed value of 4000 points was used. Control specimens were used, that consisted of fully dense samples of both 316L and resin.

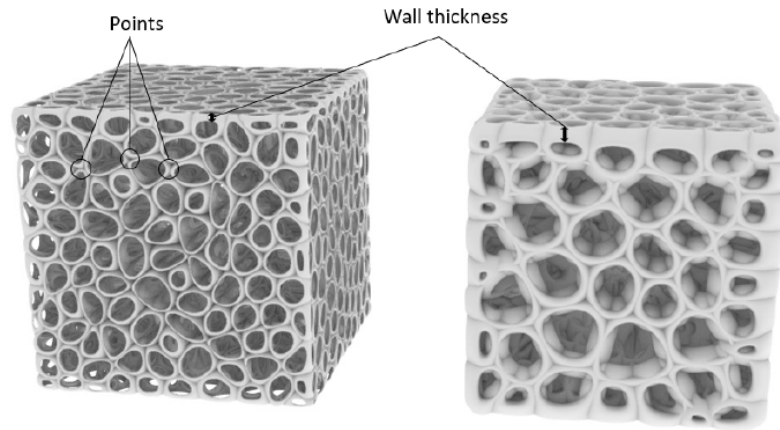


Figure 3.1: Design of Voronoi structures; Left) Higher number of points, lower wall thickness; Right) Lower number of points, higher wall thickness [103].

The 316L stainless steel samples were produced using SLM technology, albeit with some production challenges, resulting in a limited sample set, only for compression testing and microscopic analysis. Resin samples, being more cost-effective and efficient to produce, allowing the manufacture of sample for all mechanical tests.

3.2 Overview

The experimental work began with designing Voronoi lattice structures, which were then manufactured using two different methods and materials. The aim was to explore how Voronoi geometric parameters affect mechanical properties, including Young's modulus, yield strength, flexural strength, and compressive strength.

One part involved creating these structures with 316L stainless steel using SLM technology, focusing on compressive properties, due to a lower number of samples as explained in Chapter 1. The other part involved making specimens from resin using vat-photopolymerization. The samples had varying numbers of points and wall thicknesses and were tested for tensile, flexural, and compression properties.

Additionally, SEM analysis was conducted on the 316L metallic powder, microstructure was observed under the optical microscope, and visual analysis of printed Voronoi samples was performed.

The entire experimental procedure is described in the flowchart of Figure 3.2.

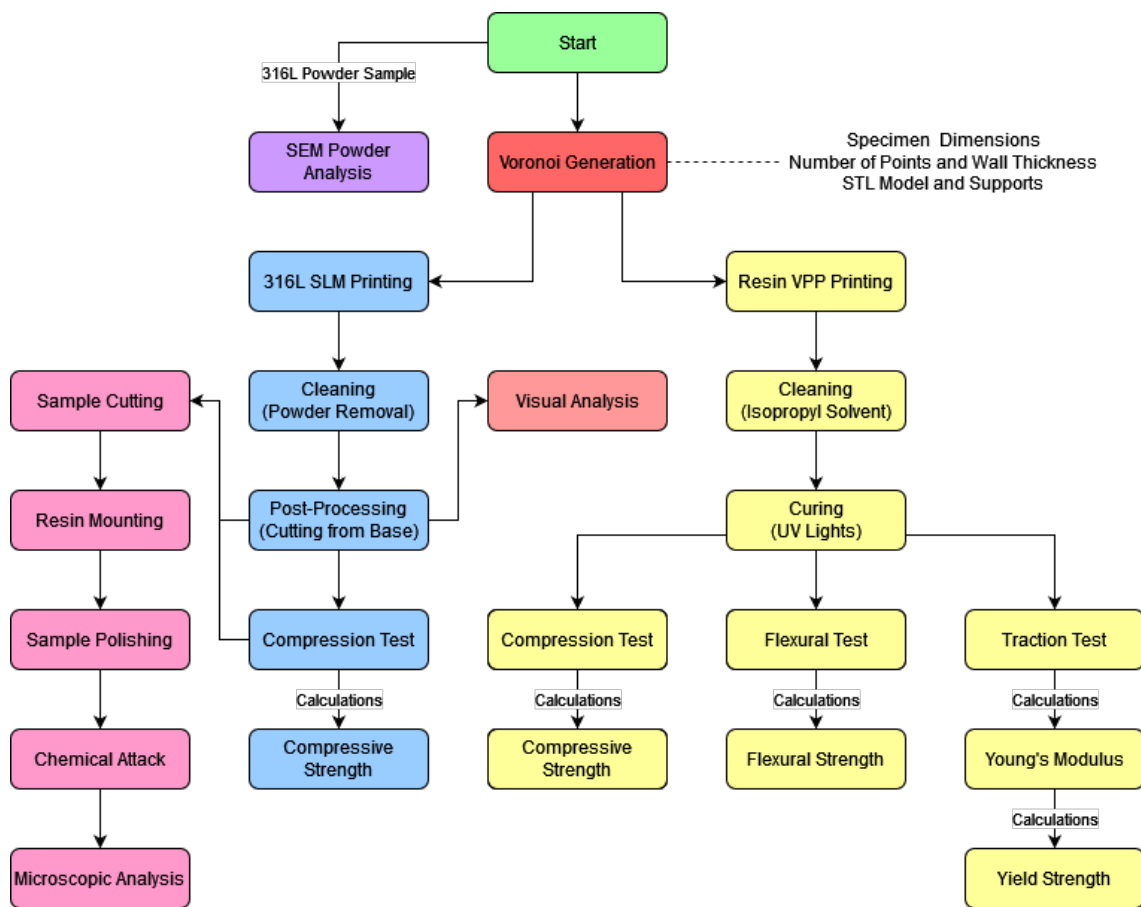


Figure 3.2: Experimental procedure flowchart.

3.3 Voronoi Lattice Structure Design

In the creation of the Voronoi structures developed in this dissertation, Rhinoceros 3D software is employed. This software is utilized to define the geometrical boundaries of the desired component. Through the software's Populate 3D function, seeds are generated and distributed within the part's boundaries, based on the specified number of points. These points are then connected using Delaunay triangulation, facilitating the formation of connection lines and Voronoi tessellation, resulting in the characteristic cells of Voronoi structures. The final step involves defining the wall thickness, ultimately producing the Voronoi structure used in this dissertation.

3.4 Experimental Procedure 316L - SLM

In this section, the steps, materials, and methodologies used in the elaboration of the manufacturing, mechanical testing, and analysis of the 316L samples produced by SLM will be explained in detail. Namely the production steps of the SLM process, the performed compression testing, as well as the microscopic analysis of the 316L sample's microstructure and finally the SEM analysis of the metallic 316L powder.

3.4.1 Sample Production

The experimental work began with the production of the necessary 316L samples, these being the compression testing, microstructure microscopic analysis, and visual analysis samples. The production parameters of the SLM process will not be studied, as they are confidential to INEGI.

The used SLM machine is a ConceptLaser M2 Cusing Series 5 from GE Additive (Figure 3.3), which uses nitrogen as the building chamber inert gas and a 400 W dual laser system with a building capacity of 245x245x350 mm.



Figure 3.3: ConceptLaser M2 Cusing Series 5 SLM machine at INEGI.

The removal of used powder is the first step in the printing job procedure. This powder is transferred into a specialized screener (Figure 3.4), which separates recycled powder from unusable debris through vibration and mesh screens.



Figure 3.4: Metallic powder recycling process. (Left) Used powder container at the machine. (Middle) Vibrating screener. (Right) Recycling process in action, with used powder canister at the top of screener, and the recycled powder canister at the bottom of screener.

The machine is filled with a mixture of both virgin and recycled 316L powder. For compatibility with the parts, the building platform (Figure 3.5) is made of 316L steel. The components are removed from the platform following each printing job using either EDM or a saw. The surface is then milled and sandblasted to repair the damaged areas and enhance sample adhesion.

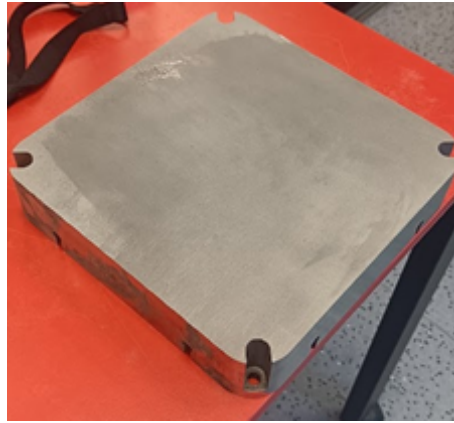


Figure 3.5: Building platform.

The metallic powder needs to be recoated after printing each layer, and its thickness is managed by a recoater, which is a metal component with a rubber edge (Figure 3.6). As a result, a master recoater must be used to calibrate it to the desired layer thickness prior to each job (Figure 3.7).



Figure 3.6: SLM machine printing chamber, with mounted recoater.

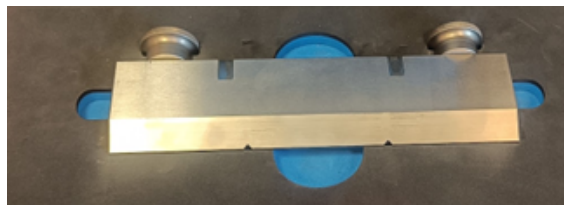


Figure 3.7: Master recoater used for calibration purposes.

The SLM chamber must be purged of oxygen to levels below 0.2% before production can start. Nitrogen is continuously pumped at a rate of 14 L/min during the entire process.

Following printing, the chamber and printed samples must cool to room temperature (18 to 25 °C) [104]. The building platform is then removed and the chamber is cleaned of all unused metallic powder (Figure 3.8). To reduce the operator's exposure to the metallic powder particles, which can be extremely dangerous if inhaled, it is imperative to clean all powder after a printing job [105].

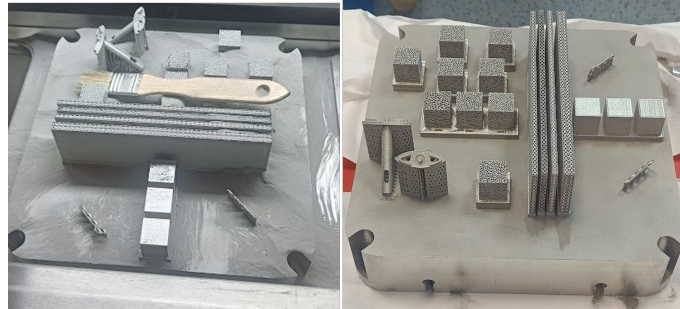


Figure 3.8: Samples after printing. (a) Before cleaning of the powder. (b) After the cleaning of the metallic powder.

The standard procedure for removing samples from the building platform typically involves the use of EDM, which is chosen for its ability to precisely remove components. However, in the case of the Voronoi lattice structures, a metal saw was employed for their removal. The final printed samples are showcased in Figure 3.9.

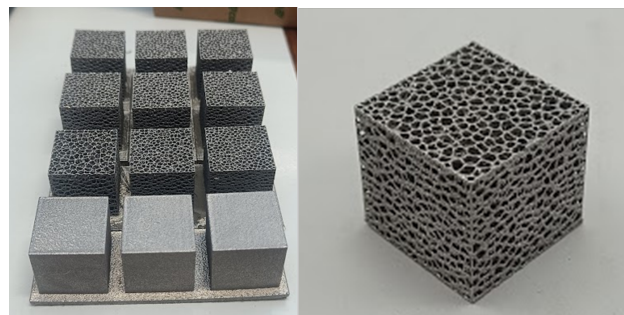


Figure 3.9: Post processing of the samples. (a) Samples after being cut from the building platform, still with the building bases on. (b) Final sample, after removal of base and polishing of the edges.

Table 3.1 provides a list of the printed samples, along with their respective characteristics, intended purpose, and quantities.

Table 3.1: List of the printed samples in the SLM job, describing purpose and number of samples printed.

Type of Sample	Sample Purpose	Number of Samples
Control 316L	Compression Test	3
4000 Points - 0.4 mm	Compression Test	3
4000 Points - 0.4 mm	Optical Microscopy	3
2000, 4000, and 6000 - 0.4 mm	Visual Inspection	1

3.4.2 Compression Test

As previously discussed, two distinct types of samples were manufactured for compression testing. These samples are comprised of fully dense 316L stainless steel control specimens and Voronoi structure specimens, featuring 4000 points and a wall thickness of 0.4 mm.

All the samples are 20 mm-sided cubes, a selection influenced by previous work conducted within the GradImp project. This choice not only ensures consistency in testing methodology but also simplifies the process of comparing results [103].

The Mitutoyo ABSOLUTE Digimatic Caliper series 500 (Figure 3.10), which has a resolution of ± 0.01 mm, was used to measure the specimens. Five measurements were taken along each of the specimen's axis, and their average values were calculated.



Figure 3.10: Mitutoyo ABSOLUTE Digimatic Caliper series 500.

Table 3.2 presents the average dimensions of the compression specimens. The sides of the control specimens align precisely with the specified 20 mm dimension. Conversely, the Voronoi specimen exhibits slightly larger dimensions, a result of geometric variations inherent to the SLM process. The reduction in height observed in both specimen types is attributed to the milling process employed when detaching the samples from the base plate.

Table 3.2: List of dimensions for the SLM control and Voronoi samples, that were subjected to compression tests.

Control	Average
Sides (mm)	20.00
Height (mm)	19.40
4000 Points - 0.4 mm	Average
Sides (mm)	20.35
Height (mm)	19.35

The compression tests were performed at the INEGI facilities using an INSTRON 5900R test machine (Figure 3.11) equipped with a 1000 KN load cell. The tests were conducted at a speed of 1 mm/min, following the guidelines outlined in the ASTM E9 standard [106].

Data pertaining to the applied load on the specimen and the displacement of the testing machine were collected. Subsequently, the compressive strength was calculated based on the applied load and specimen dimensions, while strain was determined from the displacement and specimen dimensions.



Figure 3.11: INSTRON 5900R test machine in INEGI, used for the compression tests.

The testing procedure involves the utilization of a set of tungsten plates (Figure 3.12) as the contact surface between the specimens and the machine. This choice is made due to tungsten's characteristics as an exceptionally hard material. To minimize friction and permit lateral expansion of the specimen during compression, graphite paste was applied on the contact surfaces.

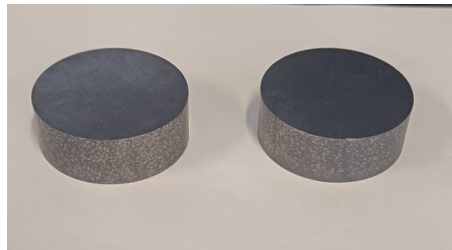


Figure 3.12: Set of tungsten plates used in the compression test.

Figure 3.13 depicts the prepared specimen, ready for compression testing.

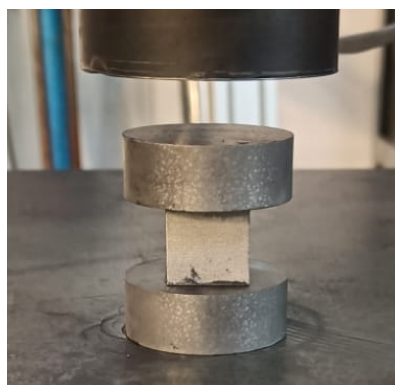


Figure 3.13: Control sample with applied graphite paste and between tungsten plates, ready to be subjected to the compression test.

3.4.3 Microscopic Analysis

As previously mentioned, Voronoi structure samples underwent microscopic analysis of their microstructure. Each sample underwent analysis from two distinct orientations: a horizontal section and a vertical section. This approach allows for comprehensive observation of all the microstructure. The samples were prepared following standard practices, which included cutting, resin mounting, polishing, and a chemical attack.

The cutting process for the samples was executed using the Struers Accutom-5 machine (Figure 3.14). This machine employs a diamond cut-off wheel with a diameter of 127 mm and a thickness of 4 mm. The cutting parameters utilized included a rotation speed of 3000 rpm and a cutting speed of 0.015 m/min. The resulting samples were cut to a thickness of approximately 5 mm (Figure 3.15).



Figure 3.14: Struers Accutom-5 machine, used for cutting of the optical microscope samples.

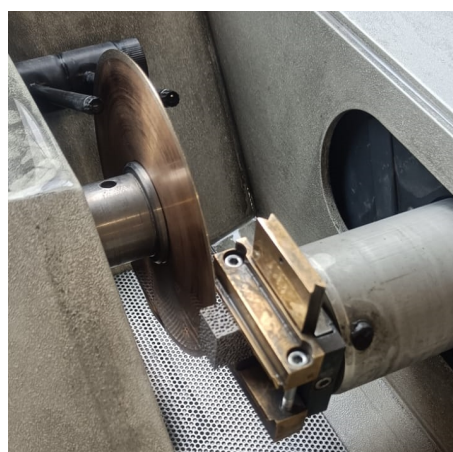


Figure 3.15: Process of cutting the optical microscope samples, using a diamond cut-off wheel.

The sectioned samples were mounted in resin (Figure 3.16). The chosen resin was an acrylic semi-transparent variety, which underwent curing for a duration of 24 hours.

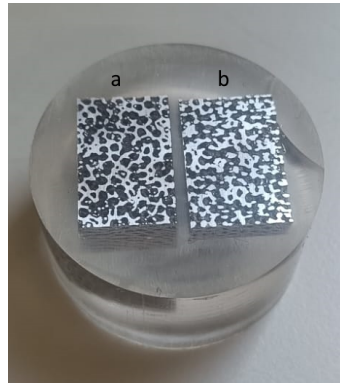


Figure 3.16: Two sections of a 4000 point sample mounted in resin. a) Vertical section of the sample. b) Horizontal section of the sample

The initial polishing stage was conducted utilizing the Struers RotoPol-21 machine (Figure 3.17). During this phase, abrasive discs with progressively finer grits (80, 180, 320, and 800) were employed, with the machine operating at a speed of 300 rpm.



Figure 3.17: Struers RotoPol-21 machine used for the polishing and finishing of the optical microscope specimens.

The final polishing step was carried out at a lower speed of 150 rpm, employing two liquid diamond suspensions with particle sizes of 3 and 1 μm .

To unveil the metal's microstructure through selective chemical exposure, the samples underwent etching. The chosen solution for this purpose was a hydrochloric acid dilution.

The completed samples, prepared and ready for microscopic analysis, are presented in Figure 3.18.

Microscopic analysis was carried out using the ZEISS Axiophot microscope (Figure 3.19), with the utilization of various objective lenses, including 5x, 10x, and 20x lenses.

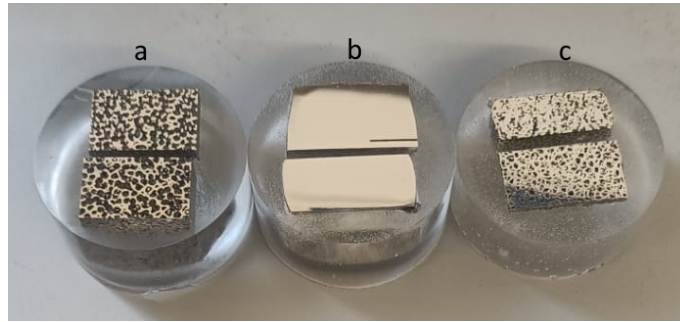


Figure 3.18: Optical microscopy samples, after the preparation phases of cutting, polishing, finishing, and etching. (a) 4000 points Voronoi sample; (b) Control samples after compression test; (c) 4000 points Voronoi sample after compression test.



Figure 3.19: ZEISS Axiophot machine, used for the optical microscopic analysis

3.4.4 Metallic Powder Analysis

The analysis of the virgin 316L powder sample was conducted using a Hitachi FlexSEM 1000 SEM equipment (Figure 3.20). Unlike some other SEM machines, the necessity for sputter coating with conductive metals like gold is eliminated by this equipment. This simplification of the working process, however, can result in a slightly lower image quality [107].

The SEM analysis was carried out with an electron acceleration voltage set at 10 KV, employing a combination of a highly sensitive 4-quadrant backscattered electron detector (BSE) and an ultra-variable-pressure detector (UVD). The UVD is specially designed for the detection of secondary electrons (SEs) under low-vacuum conditions [108].



Figure 3.20: Hitachi FlexSEM 1000 scanning electron microscope.

3.5 Experimental Procedure Resin - VPP

This section provides a comprehensive description of the experimental procedure for producing and conducting traction, flexural, and compression testing on resin Voronoi lattice structure samples produced using vat-photopolymerization. It includes details about the materials, equipment, standards, and steps taken.

3.5.1 Sample Production

The production of resin samples was performed using the DLP equipment, Phrozen Sonic Mighty 4K (Figure 3.21), which possesses the characteristics listed in Table 3.3.



Figure 3.21: DLP machine Phrozen Sonic Mighty 4K.

Table 3.3: Specifications of the DLP machine, Phrozen Sonic Mighty 4K [109].

Specifications	Phrozen Sonic Mighty 4K
XY Resolution	52 μm
Layer Thickness	10-300 μm
Print Volume	200 x 125 x 220 mm
Light Source	405 nm ParaLED Matrix 2.0

The material utilized was the "Phrozen ABS-like resin matte grey," a resin recommended by the DLP machine manufacturer, Phrozen3D. Detailed information about the chemical composition of the resin is presented in Table 3.4, while Table 3.5 provides an overview of the resin's properties.

Table 3.4: Phrozen ABS-like matte grey resin's chemical composition [109].

Substance	Percentage by weight
Acrylate Monomers	Proprietary
Diphenyl Phosphine Oxide (photoinitiator)	<5%
Titanium Dioxide (pigment)	<2%
Carbon Black	<1%

Table 3.5: Phrozen ABS-like matte grey resin's properties [109].

Viscosity (mPa.s)	67
Density (g/cm ³)	1.10
Surface Hardness (Shore D)	76
Ultimate Tensile Stress (MPa)	10
Young's Modulus (MPa)	116
Elongation at Break (%)	3
Notched IZOD Impact (J/m)	1.44
Heat Deflection temperature (°C)	46

The samples were designed in accordance with test standards and specific parameters. These CAD models were subsequently imported into slicing software, Chitubox, to configure factors such as support structures and printing parameters. It's important to note that this dissertation does not encompass the optimization of printing parameters. Therefore, the manufacturer's recommended printing parameters, as outlined in Table 3.6, were employed.

The determination of support structures and printing angles was influenced by prior research within the GradImp project [103]. An illustration of a printing job utilizing these specified parameters is provided in Figure 3.22.

The printer vat is carefully filled with resin to ensure it does not exceed the maximum allowable level, as depicted in Figure 3.23. Following this, the printing process is initiated.

As seen in Figure 3.24, the printing process is performed from bottom to top, as is the standard. Upon completion of the printing job, the printing platform is detached, and the samples are meticulously removed from both the platform and support structures using a thin chisel.

Table 3.6: Phrozen ABS-like matte grey recommended printing parameters [109].

Basic info	Layer Height (mm)	0.05
	Retract Speed (mm/min)	150
Bottom Layers	Layer Count	6
	Exposure Time (s)	35
	Lifting Distance (mm)	8
	Lifting Speed (mm/min)	60
	Light-off Delay (s)	13
Transition Layers	Layer Count	6
Normal Layers	Exposure Time (s)	2.5
	Lifting Distance (mm)	8
	Lifting Speed (mm/min)	60
	Light-off Delay (s)	13

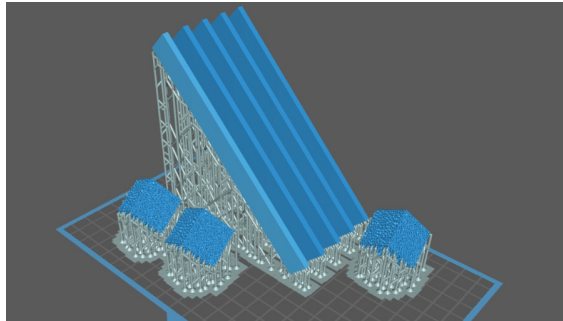


Figure 3.22: Printing job of compression and flexural specimens in Chitubox.



Figure 3.23: Phrozen Sonic Mighty 4K resin vat, displaying the maximum permitted resin level.

Following the printing process, the removal of excess resin is accomplished using a multi-purpose machine, the Creality UW-01 (Figure 3.25). The samples are positioned within a metal basket and submerged in a container filled with a 95% IPA solution (Figure 3.26). These samples are then subjected to agitation at the quick setting within the cleaning solution for a duration of 45 minutes, as established through prior optimization efforts within the GradImp project [103].



Figure 3.24: Example of a finished printing job, with traction, flexural, and compression specimens.



Figure 3.25: Creality UW-01 equipment, used for cleaning and curing procedures.



Figure 3.26: Cleaning procedure. To the right, the placement of samples in the cleaning basket. To the left, the immersion of the samples in the cleaning solution.

Following the cleaning process, post-curing is required using the same equipment as before (Figure 3.25). This machine is equipped with 18 built-in LEDs and a 360° rotating curing tray, designed to maximize UV exposure across the samples. The samples are rotated at the maximum speed for a duration of 60 minutes, a duration determined previously in the GradImp project [103]. To optimize UV exposure of the samples, specially designed supports were 3D printed in PLA using a filament extrusion printer. The utilization of these supports is illustrated in Figure 3.27.



Figure 3.27: Post-cure procedure, with samples placed on different supports (in the back it is possible to see the UV lights used in the curing process). (Left) Traction specimens. (Right) Flexural specimens.

Figure 3.28 showcases the completed samples that were subsequently utilized in the mechanical tests.



Figure 3.28: Resin flexural samples fully post-processed, ready to be tested.

3.5.2 Mechanical Tests

The resin samples underwent three distinct mechanical tests: traction, three-point flexural, and compression tests, with the aim of determining specific mechanical properties including Young's modulus, yield strength, flexural strength, and compressive strength.

The following sections provide a comprehensive account of the procedures, materials, and standards employed in the mechanical testing of the resin samples. Tables 3.7 and 3.8 outline the various specimen types that were subjected to testing. It's important to note that three specimens were produced for each sample type.

Table 3.7: Types of resin specimens used in the mechanical testing of number of points.

Number of Points	Wall Thickness (mm)
2000	0.4
3000	0.4
4000	0.4
5000	0.4
6000	0.4
Control	Control

Table 3.8: Types of resin specimens used in the mechanical testing of wall thickness.

Number of Points	Wall Thickness (mm)
4000	0.4
4000	0.5
4000	0.6
Control	Control

In all testing procedures, the measurement of specimens was performed using the Mitutoyo AOS ABSOLUTE Digimatic Caliper series 500, with a resolution of ± 0.01 mm. In each axis of the specimens, 5 measurements were conducted, and their average determined. The retrieved data from all the tests was applied load (N) and displacement (mm), which were then converted, using the specimens dimensions, into stress (MPa) and strain.

3.5.2.1 Traction Test

Traction testing was carried out following the ISO 527-4 standard [110]. Accordingly, the samples were manufactured with the dimensions delineated in Figure 3.29 and listed in Table 3.9. For visual reference, Figure 3.30 illustrates an example of a traction specimen with 4000 points and a thickness of 0.4 mm.

The traction test was conducted at FEUP facilities, utilizing the Mecmezin Multitest 2.5-dV testing equipment (Figure 3.31). It's worth noting that the Mecmezin equipment was employed for all mechanical tests, albeit with varying load cells and fixtures depending on the specific test type.

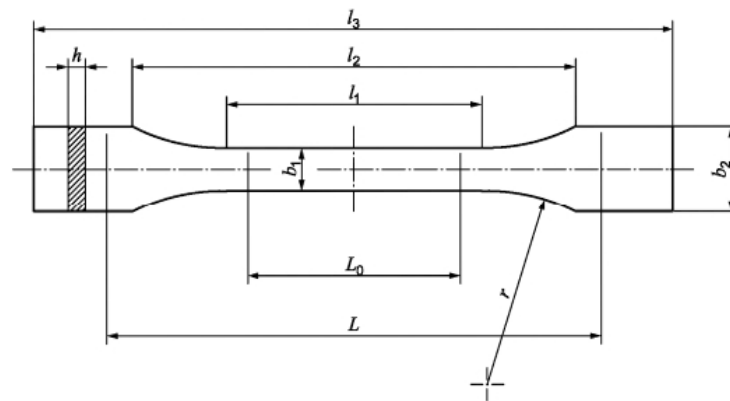


Figure 3.29: Traction specimen dimensions description [110].

Table 3.9: Used ISO 527-4 standard traction specimen dimensions [110].

Symbol	Name	Dimensions (mm)
l_3	Overall length	150
l_1	Length of narrow parallel-sided portion	60
r	Radius	60
b_2	Width at ends	20
b_1	Width of narrow portion	10
h	Thickness	4
L_0	Gauge length	50
L	Initial distance between grips	115



Figure 3.30: Traction specimen with 4000 points and 0.4 mm of thickness.

A Mecmezin 2500 N load cell was employed for the traction test, in conjunction with specialized jaw fixtures designed to securely grip the specimens. Both the load cell and the fixtures can be seen in Figure 3.32.

The testing was executed at a speed of 2 mm/min, which aligns with the specifications outlined in the relevant standard. Figure 3.33 illustrates an example of the testing procedure conducted on a specimen with 4000 points.



Figure 3.31: (Left) Mecmesin Multitest 2.5-dV testing equipment. (Right) Testing equipment with traction test fixtures and load cell.



Figure 3.32: (Left) 2500 N load cell). (Right) Traction jaw fixture.

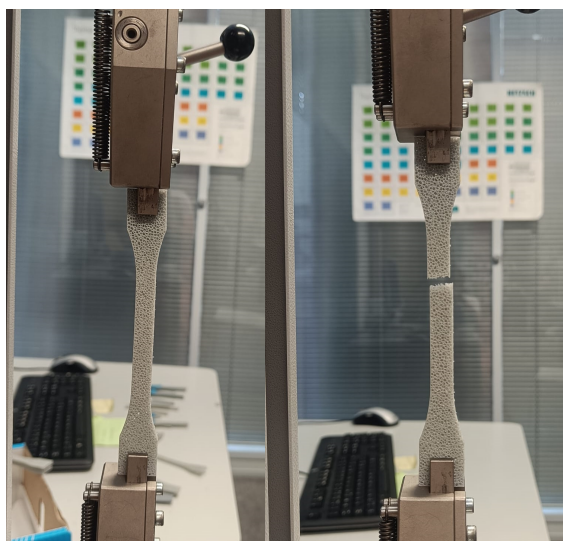


Figure 3.33: Traction testing procedure.

3.5.2.2 Flexural Test

The flexural tests were carried out in accordance with the ASTM D790 standard [111]. Consequently, the flexural specimens were manufactured following the dimensions illustrated in Figure 3.34. To provide visual clarity, Figure 3.35 presents an example of a flexural specimen featuring 3000 points and a wall thickness of 0.4 mm.

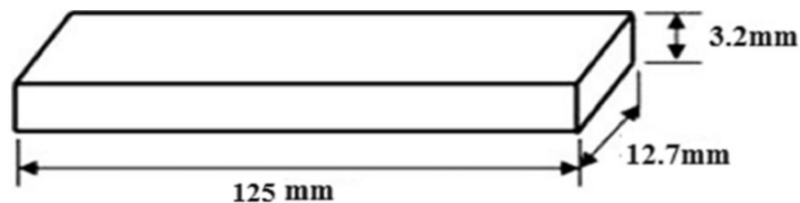


Figure 3.34: Flexural specimen dimensions under the ASTM D790 standard [112].

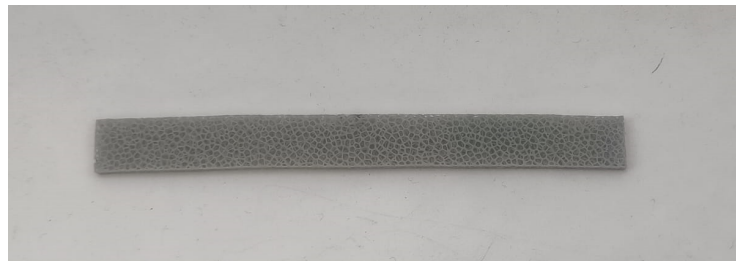


Figure 3.35: Flexural specimen with the used ASTM D790 standard dimensions.

The flexural test was performed using the same Mecmezin 2.5-dV testing equipment, which had been used for previous mechanical tests. The equipment was fitted with the required fixtures and load cell, as illustrated in Figure 3.36.

The three-point flexural test necessitates a fixture consisting of a bottom load support equipped with two cylindrical support points, as well as a top load nose featuring one cylindrical support point. In this test, a Mecmezin 100 N load cell was utilized. Both the fixtures and the load cell can be observed in Figure 3.37.

In this work, the distance between the two support points on the bottom load support, referred to as the support span (Figure 3.38), was intentionally established at 60 mm (Figure 3.39). This specific configuration of the support span was chosen to ensure test repeatability and facilitate accurate calculations.



Figure 3.36: Mecmesin Multitest 2.5-dV testing equipment with the necessary flexural test fixtures and load cell.

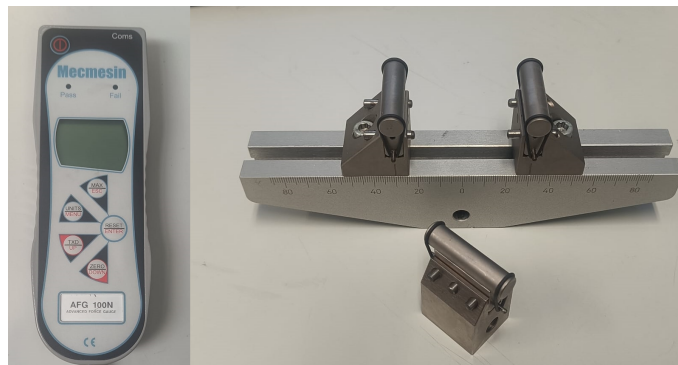


Figure 3.37: (Left) 100 N load cell. (Right) Three-point Flexural fixtures.

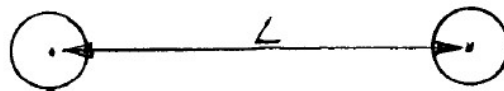


Figure 3.38: Representation of the support span measurement used in the flexural strength calculation [111].

The testing procedure involves positioning the specimen so that it rests at its ends on the bottom support, while applying a load from above at its center point, as depicted in Figure 3.40.

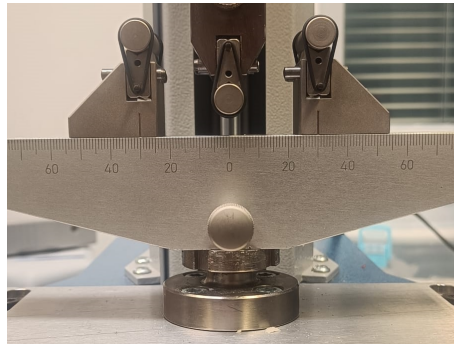


Figure 3.39: Used 60 mm flexural test support span on the bottom load support.

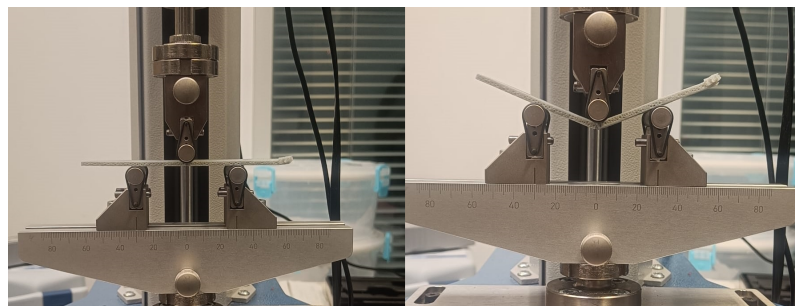


Figure 3.40: (Left) Specimen placement in flexural testing. (Right) Tested specimen.

The test calculates the maximum flexural stress (MPa) of the specimens using Equation 3.1, with the maximum value extracted from each individual specimen.

$$\sigma_f = 3PL/2bd^2 \quad (3.1)$$

Where:

σ = stress in the outer fibers at midpoint (MPa);

P = load at a given point (N);

L = support span (mm);

b = width of specimen (mm);

d = depth of specimen (mm).

3.5.2.3 Compression Test

The compression tests adhered to the ASTM E9 standard [106], predominantly following the standard testing speeds and calculation procedures. The dimensions of the specimens were arbitrarily chosen to be cubes with sides measuring 20 mm, as elaborated upon in section 3.4.2. For visual reference, Figure 3.41 presents an example of a compression specimen featuring 4000 points and a wall thickness of 0.6 mm.

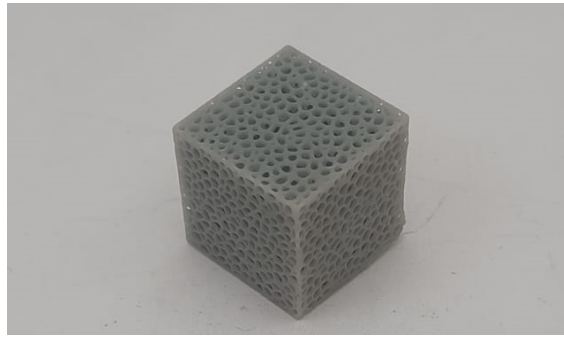


Figure 3.41: Compression specimen.

The compression testing was conducted using the Mecmesin 2.5-dV testing equipment, which was equipped with the necessary compression fixtures, as illustrated in Figure 3.42.



Figure 3.42: Mecmesin Multitest 2.5-dV testing equipment with the necessary compression test fixtures and load cell.

To perform the compression test, a set of aluminum bases, consisting of both a top and bottom base, were employed. These bases feature wide, flat, and durable surfaces, as depicted in Figure 3.43 (Left). To measure the applied loads during the compression test, the Mecmesin 2500 N load cell was utilized (Figure 3.43 Right).

The specimen is centered between the two plates, and the compression testing is carried out at a speed of 2 mm/min. Figure 3.44 provides a visual representation of a specimen both before and after undergoing a compression test.



Figure 3.43: (Left) Compression fixtures. (Right) 2500 N load cell.

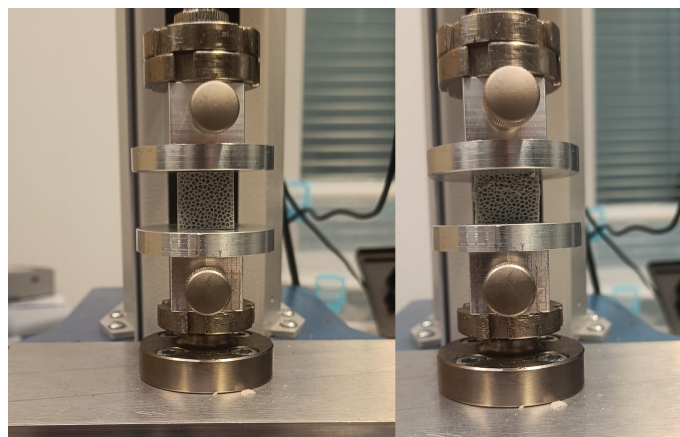


Figure 3.44: (Left) Specimen before compression testing. (Right) Specimen after compression testing.

Chapter 4

Analysis and Discussion

4.1 SLM - 316L Analysis

This section delves into the microscopic analysis of the 316L microstructure, the SEM analysis of the metallic 316L powder, as well as a visual analysis of printed samples. It also explores how the geometry of the Voronoi lattice structure affects mechanical properties, with a particular focus on compressive strength.

4.1.1 Powder Analysis

The metallic powder samples were subjected to SEM, with the goal of analysing its morphology, particle size, and possible presence of defects and porosities. In Figure 4.1 it is possible to observe the results of the SEM procedure.

Significant aggregations of powder particles can indeed hinder powder flowability, resulting in defects and subpar components. However, upon scrutiny of Figure 4.1, it becomes evident that there are no substantial aggregations in the powder sample. Instead, there is only the presence of minor quantities of satellites, a phenomenon that is typically encountered and difficult to entirely eliminate, as corroborated by existing literature.

Porosity is a prevalent defect in metallic powder, capable of adversely impacting the quality of components, often originating from gas absorption during the atomization process. Nevertheless, the observation of Figure 4.1 yielded no evidence of porosity in the 316L powder sample.

In Figure 4.1, the predominant morphology of the particles appears to be spherical, which aligns with the desired shape recommended in the literature. Spherical particles are known to enhance powder flowability. Additionally, the minimal presence of irregularly shaped particles is indicative of high powder quality.

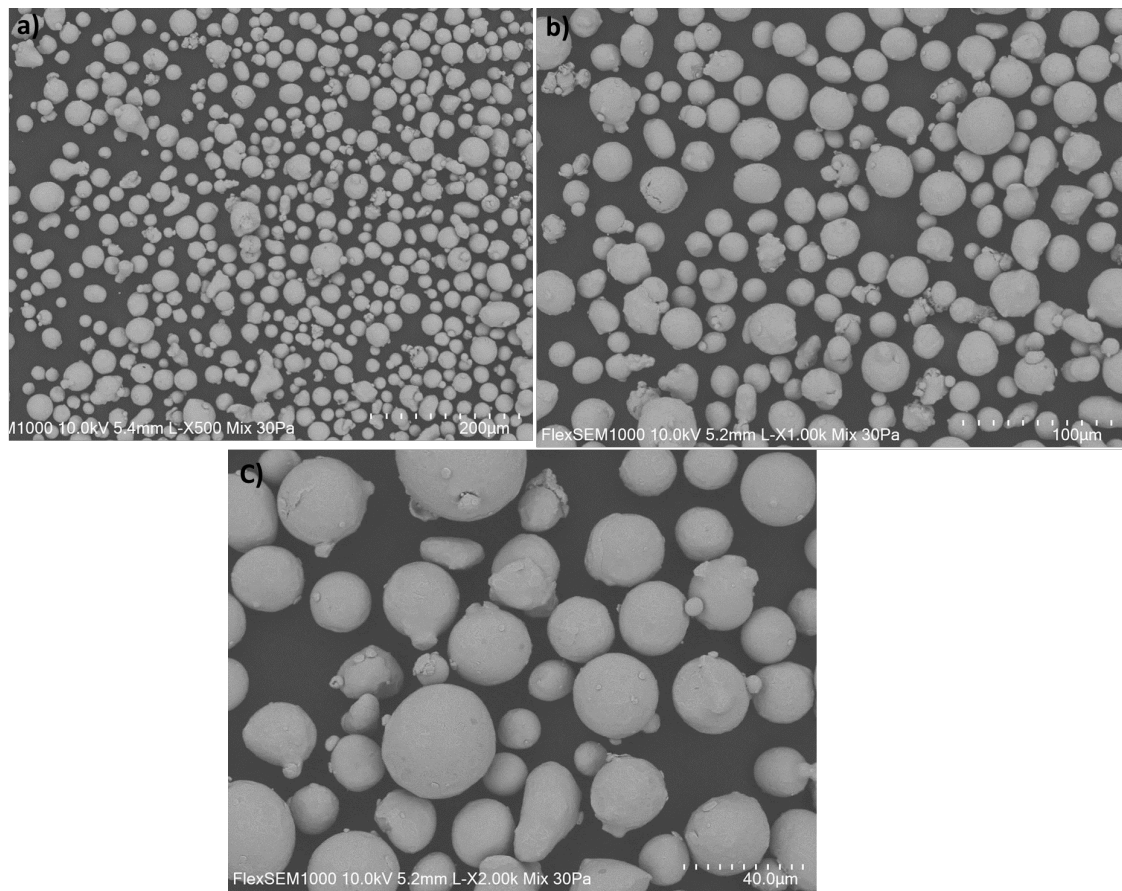


Figure 4.1: SEM images of 316L virgin powder under a magnification of: a)500X; b)1000X; c)2000X.

The SEM analysis of the 316L metallic powder corroborates the characteristics described in the literature as essential for producing high-quality components. Apart from the anticipated presence of satellites, the powder sample appears to be devoid of any significant defects that might compromise its quality. Consequently, the utilized powder is expected to be of good quality, and it should not pose any adverse effects on the production of Voronoi lattice structures in 316L stainless steel.

4.1.2 Visual Inspection

As mentioned in Chapter 3, a set of three Voronoi structures (Figure 4.2) with varying numbers of points (6000, 4000, and 2000) was created to assess the SLM process's capability and quality in producing Voronoi lattice structures. The objective was to visually inspect these specimens for major defects, ensuring they meet quality standards and serve as a foundation for optimizing the SLM process in the production of pure iron Voronoi lattice structures.

Regarding surface roughness, there were no discernible variations across the entire area, with no excessively high surface roughness present in the sample. The observed surface quality falls within the expected range for these structures.



Figure 4.2: Set of 3 Voronoi structures with different number of points (2000, 4000, 6000).

Figure 4.3 shows a slight deformation in the base plate, which is a common occurrence due to thermal gradients in the SLM process, as is mentioned in the literature. Such thermal deformations result from the repeated heating and cooling of a large area like the base plate. Despite the base plate's deformation, the sample set remains unaffected, preserving its geometrical and dimensional integrity.

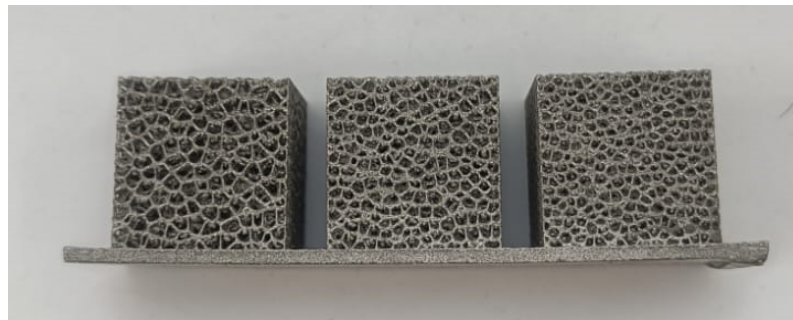


Figure 4.3: Visual deformation of the base plate, in the SLM sample set.

From a geometric perspective, significant distinctions can be observed between the upper and lateral portions of the samples. Examination of the sample tops (as shown in Figure 4.4 Left) reveals a lack of discernible defects, with precise Voronoi cell shapes and accurate geometry. In stark contrast, the lateral sides of the samples (Figure 4.4 Right) display diminished geometric quality, featuring less-rounded characteristics and the presence of small circular defects attributed to excess fused material clinging to the sample's features.

In summary, the sample set displays anticipated levels of surface roughness, normal base plate deformation, and impeccable geometry on the top side of the specimens. However, there is room for improvement in terms of geometric precision on the lateral sides. This disparity underscores the necessity for additional testing of Voronoi lattice structure manufacturing parameters using SLM technology, as outlined in the literature, where process parameters can significantly impact the final part's quality. Ultimately, the optimization of process parameters is pivotal for the prospective implementation of innovative pure iron Voronoi implant production as part of the GradImp project.

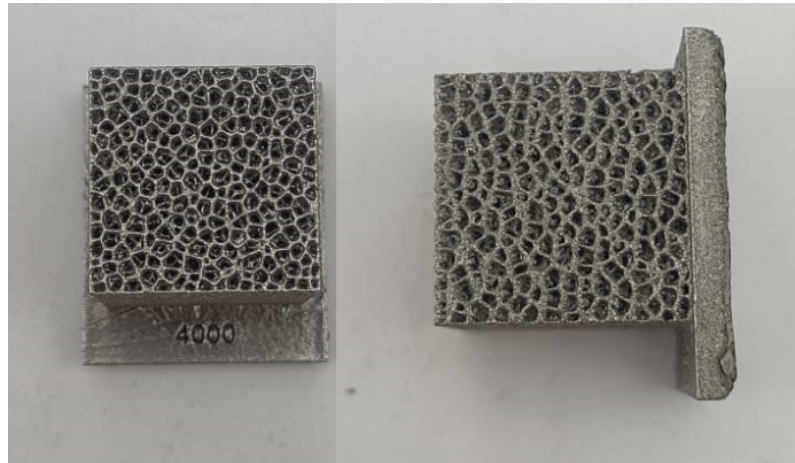


Figure 4.4: Visual inspection of Voronoi lattice structure geometrical accuracy. (Left) Top view of the sample. (Right) Side view of the sample.

4.1.3 Microstructure Analysis

This section delves deeper into the analysis of optical microscopy images to examine the microstructure and defects present in SLM 316L specimens. As previously mentioned in Chapter 3, a set of three specimens was employed for microscopic analysis. This set comprises an as-built 4000-point Voronoi structure, which serves as the primary focus of the analysis. Additionally, two other samples are included: a fully dense 316L specimen and another 4000-point Voronoi specimen, both of which underwent a compression test. It is anticipated that these latter two samples have undergone alterations in microstructure and morphology as a consequence of the applied forces. It's important to note that in Figures 4.5 and 4.6, the samples subjected to compression tests are distinguished by a "C" prefix. Furthermore, in Figures 4.5, 4.6, 4.7, and 4.8, the magnification values of the objective lens are explicitly provided in each caption.

Figure 4.5 provides microscopy images of a vertical section of the three samples, offering insights into the layer-by-layer build process. In Figures 4.5 a), b), and c), the images clearly depict the curved-shaped melt pool contours formed by the SLM process, which closely resemble the shapes described in the literature. These melt pools are the result of the successive deposition of layers, where each deposited layer causes remelting and reshaping of the previous ones. This process creates a characteristic scaled effect, as detailed in the literature, which is readily observable in all the samples, spanning from Figures 4.5 a) to f).

Figure 4.5 c) reveals the presence of columnar grains that traverse the boundaries of the melt pools, seemingly aligning with the directions of the solidification structures. This observation aligns with established findings in the existing literature.

Despite the noticeable compression and fractures in the structure of the samples seen in Figure 4.5 from d) to f), it remains possible to identify the characteristic melt pools and their overlapping patterns. These features persist even under the influence of the applied forces, providing valuable insights into the microstructure of the specimens.

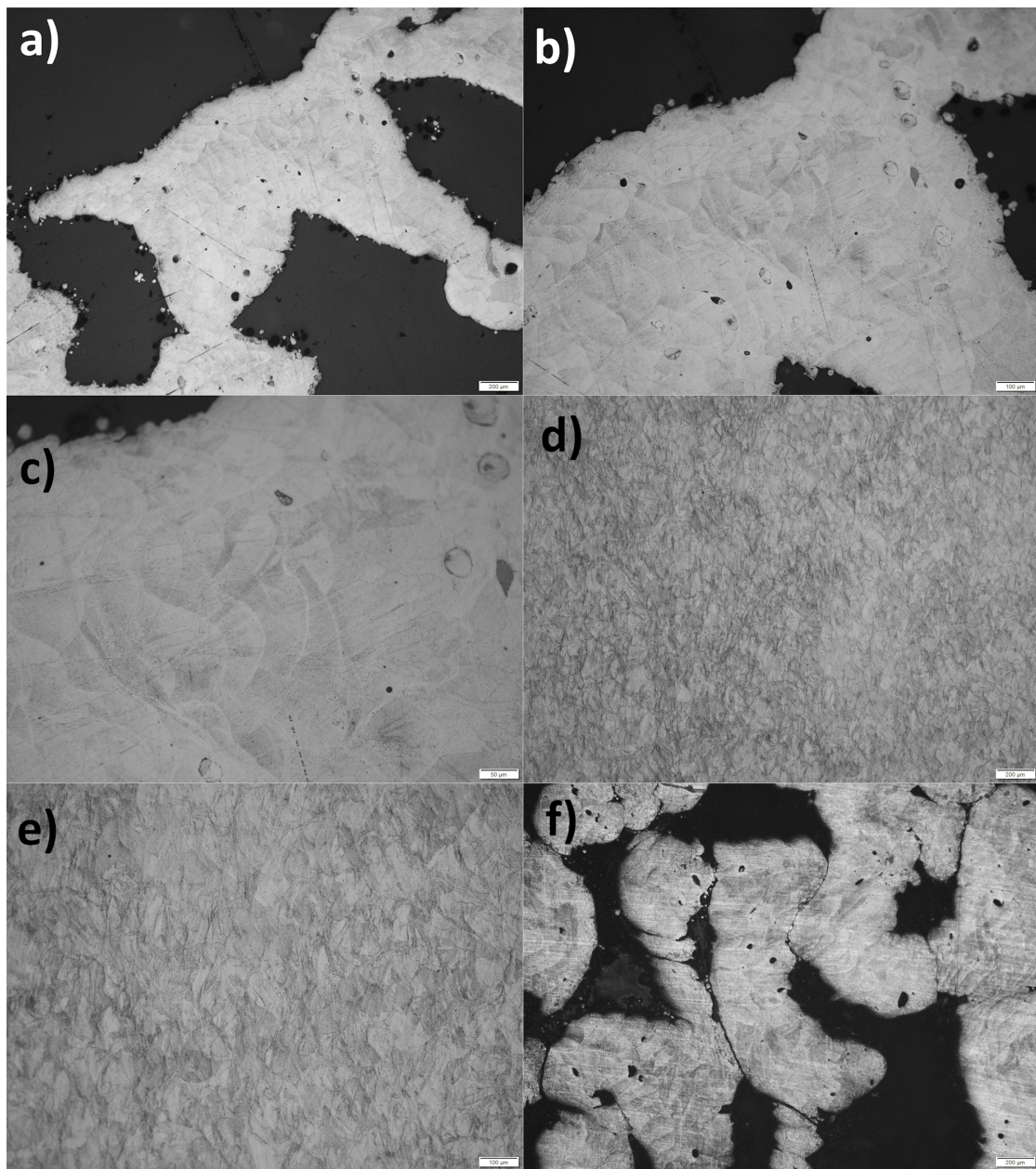


Figure 4.5: Optical microscopy of 316L SLM (vertical section). a) 4000 under 5x. b) 4000 under 10x. c) 4000 under 20x. d) C-Dense under 5x e) C-Dense under 10x f) C-4000 under 5x.

Figure 4.6 presents a horizontal section of the three samples. Providing a view of the SLM building process at a single layer level.

In Figures 4.6 a) to c), the scanning strategy employed in the SLM process is prominently visible. As the laser moves during scanning, it creates distinct melt paths, providing a clear visualization of the precise movement of the scanning laser strategy during the SLM process. Upon closer examination, it becomes apparent that there is a continuous linear melting of the specimen's contours, followed by hatching melting on the interior of these contours. This scanning strategy aligns with what is described in the literature as a commonly used approach in SLM.

Figures 4.6 d) and e) have been notably influenced by the compression test, to the extent that it is no longer possible to identify any discernible scanning strategy. However, Figure 4.6 f) still allows to visualize the scanning strategy, as the contour melting and laser paths are still clearly discernible.

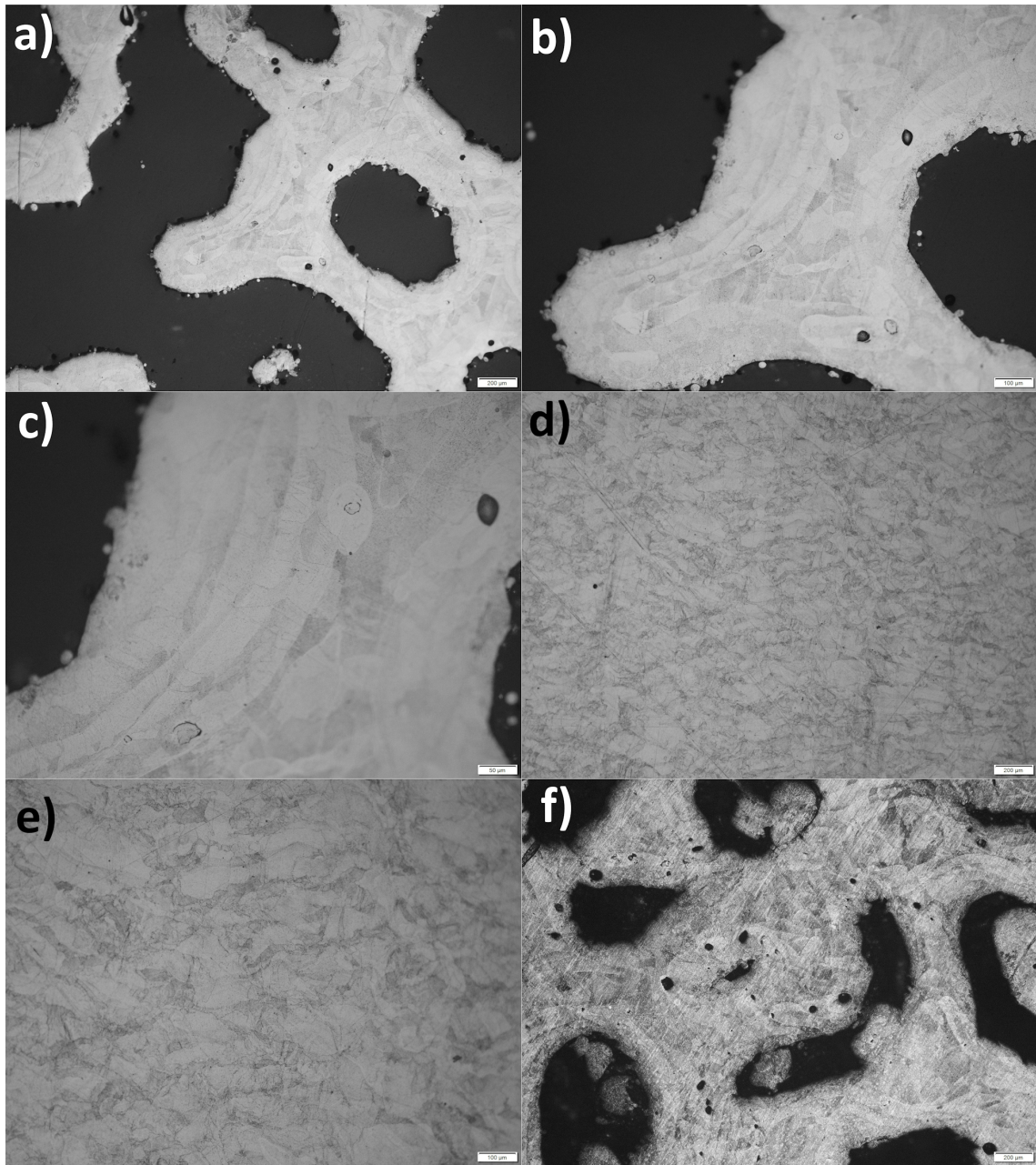


Figure 4.6: Optical microscopy 316L SLM (horizontal section). a) 4000 under 5x. b) 4000 under 10x. c) 4000 under 20x. d) C-Dense under 5x e) C-Dense under 10x f) C-4000 under 5x.

Regarding defects, only the as-built 4000-point Voronoi structure were considered because the other two samples have undergone compression, and any present defects cannot be directly linked to the SLM process, as they could have occurred or worsened during compression testing.

In Figure 4.7 a) and b), showcasing a vertical section, instances of porosity are evident. These porosities, which are spherical in shape and recognized as gas porosities, are caused by trapped gas bubbles resulting from the vaporization of metal during the SLM process. It's noteworthy that no larger, irregular porosities were detected in the specimens, which is significant as such irregularities can often lead to crack propagation and exacerbate mechanical property issues, as outlined in the literature.

Figure 4.7 c) prominently displays spherical defects at the periphery of the sample. These round particles are defects that can be attributed to high laser power and large layer sizes. They result from droplet spatters being scattered across the sample borders, ultimately contributing to increased surface roughness and reduced geometrical tolerances [113].

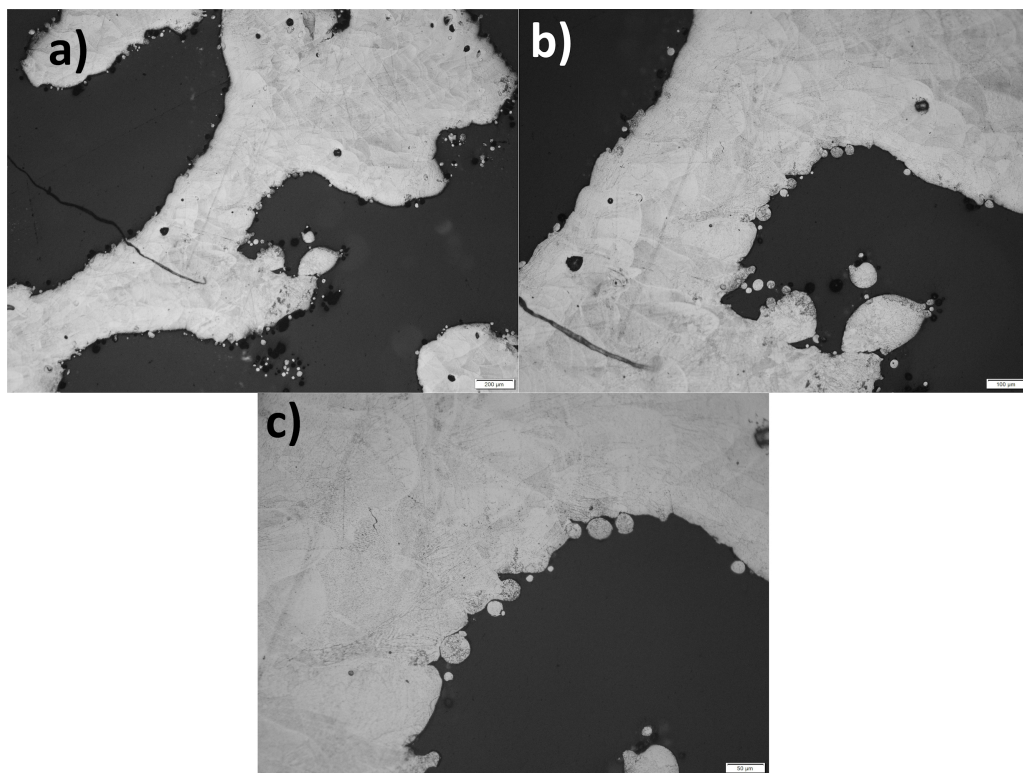


Figure 4.7: Optical microscopy 316L SLM (vertical section), showcasing defects. a) 4000 under 5x. b) 4000 under 10x. c) 4000 under 20x.

Figure 4.8 depicts a horizontal section of the sample, where similar defects to those seen in the previous example in Figure 4.7 are evident. These defects encompass regular porosity and spatter defects. However, it's worth noting that in Figure 4.8 b), at the bottom left corner, an irregularly shaped porosity is also visible, adding to the diversity of porosities present in the sample.

Additionally, in the center of Figure 4.8 b), a novel type of defect is evident—a series of three similar porosities arranged in a line. As per the literature, these patterned porosities are a consequence of the laser beam's movement during the scanning strategy. This phenomenon results in the alignment of similarly shaped porosities, and such patterned porosities are closely tied to process parameters, particularly the laser scanning strategy [114].

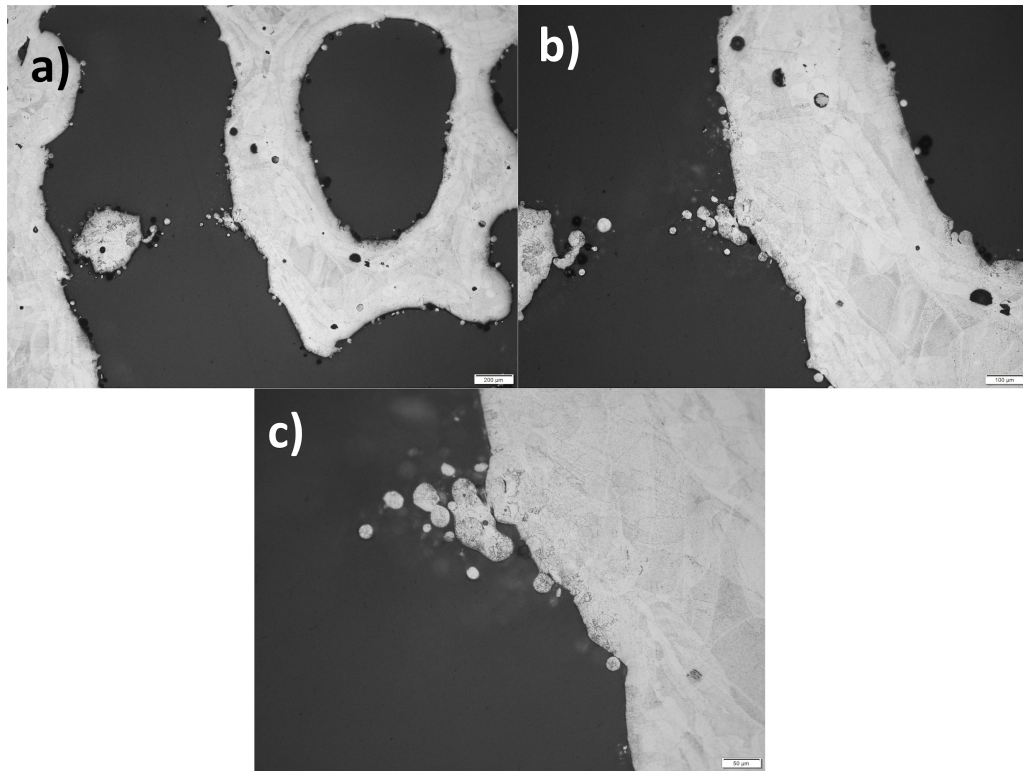


Figure 4.8: Optical microscopy 316L SLM (horizontal section), showcasing defects. a) 4000 under 5x. b) 4000 under 10x. c) 4000 under 20x.

In conclusion, the optical microscopic analysis has yielded valuable insights into the SLM printing of Voronoi lattice structures. It provided a clear visualization of the scanning strategy, the formation of curved melt pools during layer deposition, and the presence of typical columnar grains that grow in the solidification direction, all consistent with findings in the literature. Additionally, various defects were identified, including regular spherical porosities resulting from metal vaporization and patterned porosities resulting from the scanning strategy. Spatter defects at the structure's edges were also observed. This analysis significantly enhances the understanding of the microstructures of 316L Voronoi structures produced by SLM, laying the groundwork for future process improvements in the production of Voronoi lattice structures, not only in 316L but also in other materials, such as pure iron.

4.1.4 Compression Test Analysis

The compression test results for the 316L samples were affected by a problem with the testing equipment. The compression machine lacked full rigidity, leading to unfavorable effects on the results. Therefore, these results cannot be directly compared to existing literature. However, because both the fully dense specimens and the 4000-point Voronoi specimens underwent testing under the same conditions, some level of comparison between the two sample types is still feasible.

In Figure 4.9, it can be observed the stress-strain curve of a fully dense control specimen. It's noteworthy that the curve experiences an unusual and sudden flattening at approximately 450

MPa. This occurs when the testing equipment loses its rigidity and flexes, resulting in a gap in the data. This behavior was repeated in all tests at the same load value, even when the machine was tested without any specimens. All the test data, including data related to the testing equipment, is in Appendix A.

Regrettably, the point at which the control specimen reaches its maximum compressive strength coincides with the data gap. This makes it impossible to accurately determine the compressive strength value. Nevertheless, for the sake of comparison with the Voronoi sample, an average value of 500 MPa was used as the compressive strength of the control specimen.

Compressive Strength - Control Sample

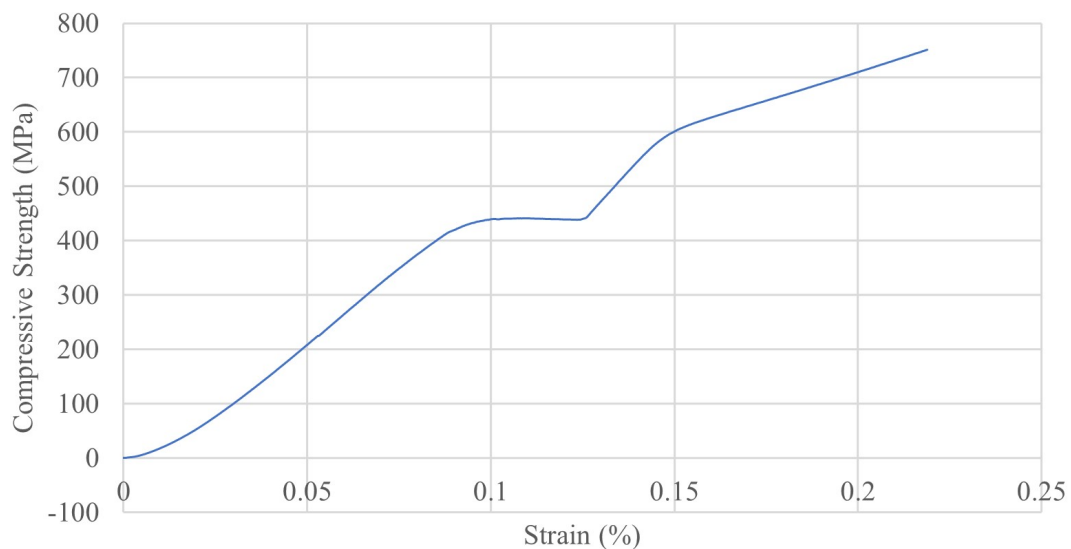


Figure 4.9: Stress-Strain curve of a fully dense 316L control specimen.

The Voronoi samples with 4000 points and 0.4 mm wall thickness experienced the same rigidity issue. However, since the stress levels in these samples are much lower, the stress-strain curve of the Voronoi specimens in Figure 4.10 is clearly visible. The compressive strength was determined by calculating the compressive Young's modulus and using it to find the compressive strength at the yield point. The compressive strength was calculated by dividing the applied force by the specimen cross-section.

In Figure 4.11, a comparison is drawn between the compressive strength of the control 316L samples and the Voronoi lattice structures with 4000 points and a wall thickness of 0.4 mm.

The main takeaway from this comparison is that there's an 88% decrease in compressive strength when comparing a fully dense specimen to a Voronoi specimen with 4000 points and a 0.4 mm wall thickness.

Compressive Strength - Voronoi 4000 Points and 0.4 mm Wall Thickness

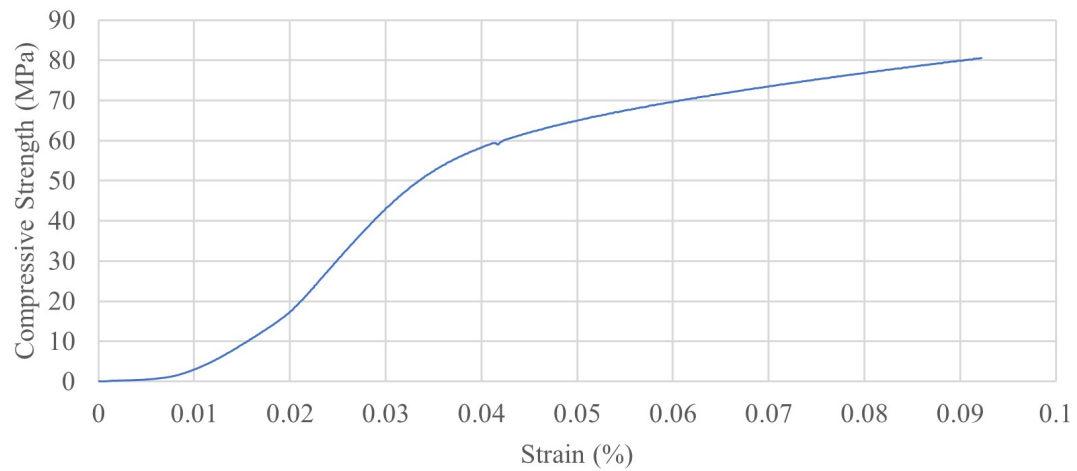


Figure 4.10: Stress-Strain curve of a 316L 4000 point Voronoi lattice structure specimen.

SLM 316L - Compressive Strength

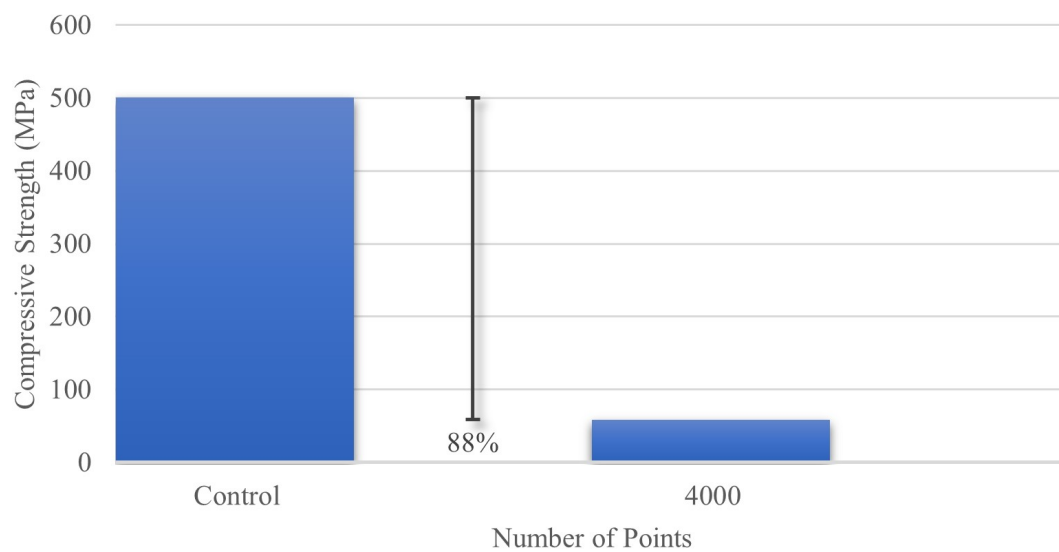


Figure 4.11: Variation of compressive strength in a 316L Voronoi lattice structure, manufactured by SLM.

4.2 Resin Mechanical Properties Analysis

In this section, the impact of the Voronoi lattice structure's geometry on mechanical properties is examined. Young's modulus, yield strength, flexural strength, and compressive strength will be

analyzed. These properties are influenced by variations in the number of points and wall thickness (mm) within the Voronoi lattice structures.

As mentioned earlier, traction tests yield insights into Young's modulus and yield strength. Young's modulus is determined from the slope of the linear elastic section on the stress-strain curve, while yield strength is calculated based on the intersection of the stress-strain curve and the Young's modulus 0.2% offset line.

Flexural tests provide data on the specimen's flexural strength. The maximum applied load during the test helps calculate this property, following the method outlined in Chapter 3.

Compression tests reveal compressive strength, with the compressive young's modulus used to determine the yield point. Due to the specimens' dimensions, there is no catastrophic failure, so compressive strength is determined at the yield point, following standard procedures.

4.2.1 Number of points

In this subsection, the effect of number of points on Voronoi lattice structure's mechanical properties is explored. Three mechanical testing procedures were carried out: traction, flexural, and compression testing. The evolution of Young's modulus, yield strength, flexural strength, and compressive strength is analyzed as the number of points in the Voronoi structures is varied. These results are compared to a fully dense control specimen. The Voronoi structures under study were subjected to variations in the number of points, ranging from 6000 to 2000 in 1000-point intervals.

4.2.1.1 Tensile Properties

Figure 4.12 shows Young's modulus values for both the fully dense control specimen and Voronoi structures with points ranging from 2000 to 6000. Young's modulus reduction, compared to the fully dense control, is evident, ranging from 76% in the 6000-point specimen to 96% in the 2000-point specimen.

Figure 4.13 focuses on the Voronoi structures, excluding the control specimen. It demonstrates the gradual reduction of Young's modulus as the number of points decreases. Notably, the reduction is 71% when transitioning from 4000 points to 3000 points, while other 1000-point reductions tend to be closer to 20%.

Figure 4.14 presents yield strength values for the fully dense specimen and Voronoi structures with varying points (2000 to 6000). There is a gradual reduction in yield strength, ranging from 87% in the 6000-point specimen to 97% in the 2000-point specimen.

Figure 4.15, focusing on the Voronoi structures, illustrates the gradual reduction of yield strength with decreasing points. Reductions of around 32% are observed with each 1000-point reduction, ranging from 25% to 37%. Unlike Young's modulus, yield strength shows a more linear reduction with decreasing points

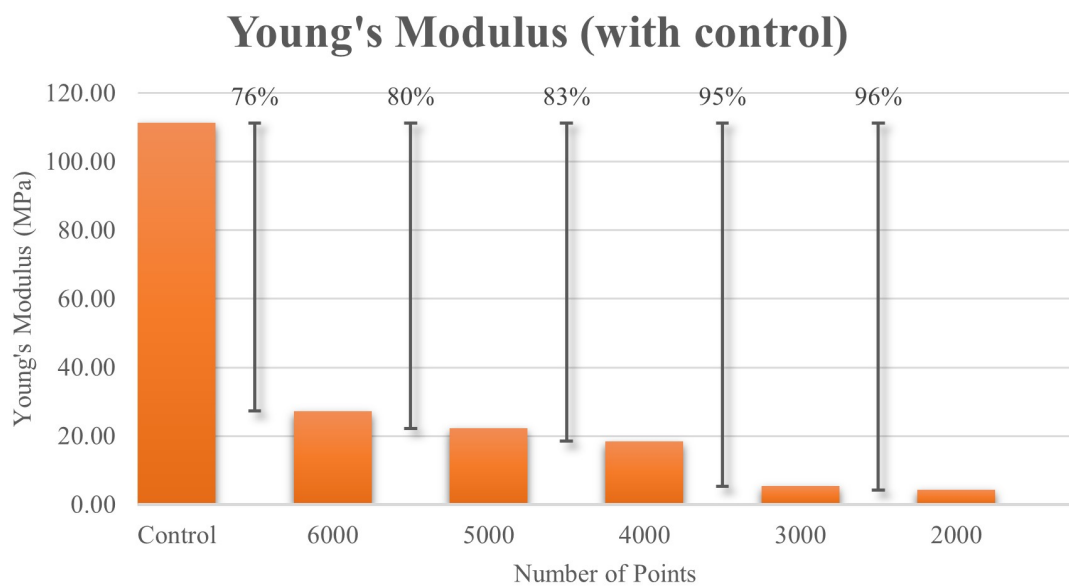


Figure 4.12: Young's modulus (MPa) values for the fully dense control specimen, and Voronoi samples with varying number of points.

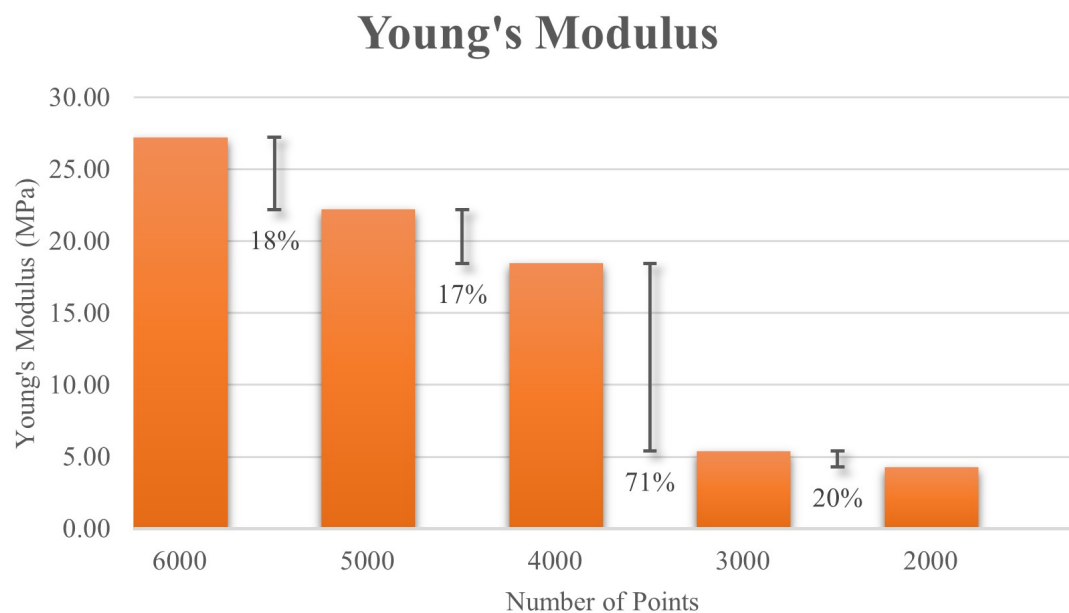


Figure 4.13: Young's modulus (MPa) values for Voronoi samples with varying number of points.

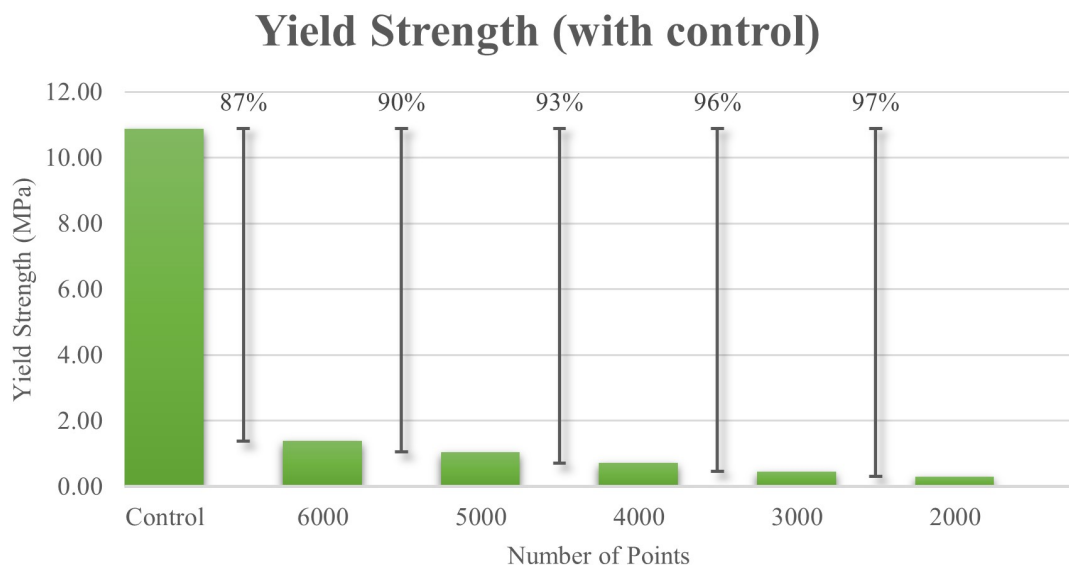


Figure 4.14: Yield strength (MPa) values for the fully dense control specimen, and the Voronoi samples with varying number of points.

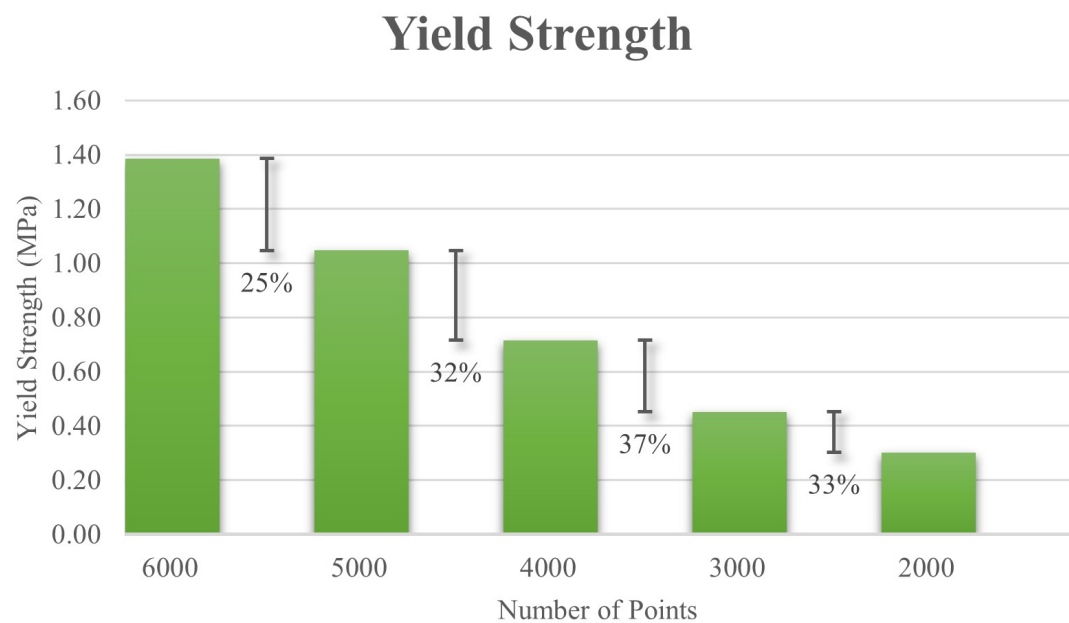


Figure 4.15: Yield Strength (MPa) values for Voronoi samples with varying number of points.

In summary, Young's modulus generally experiences an average reduction of 20% with the decrease in points, except for the transition from 4000 to 3000 points, where the reduction is 71%. In contrast, yield strength shows a linear decrease of approximately 32% with each 1000-point reduction.

4.2.1.2 Flexural Strength

Figure 4.16 displays flexural strength values for the fully dense specimen and Voronoi structures with points ranging from 2000 to 6000. The reduction in flexural strength compared to the fully dense control specimen ranges from 88% to 99%, with the 2000-point specimen experiencing the largest reduction.

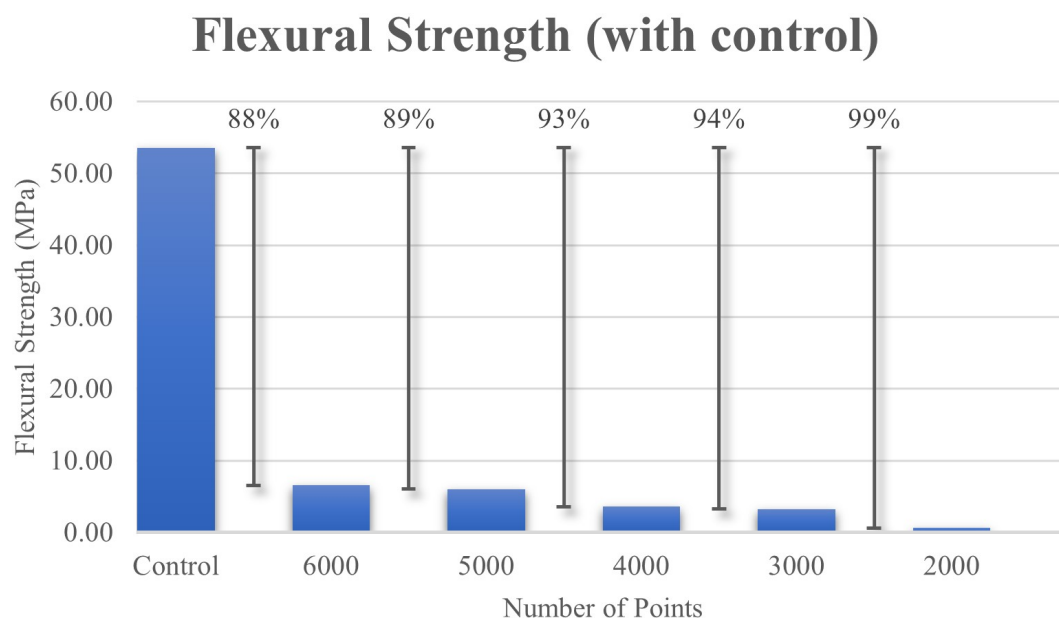


Figure 4.16: Flexural Strength (MPa) values for the fully dense control specimen, and Voronoi samples with varying number of points.

Figure 4.17 excludes the control specimen and illustrates the flexural strength of the Voronoi structures. It shows that the reduction in strength does not follow a consistent pattern, ranging from low values of 8% to 9%, to a notably higher 81% reduction from the 3000-point specimen to the 2000-point specimen.

The variation in flexural strength with the number of points in the Voronoi structure is complex and does not follow a linear pattern. These results suggest that the Voronoi structure's complexity and geometrical unpredictability play a significant role in the results.

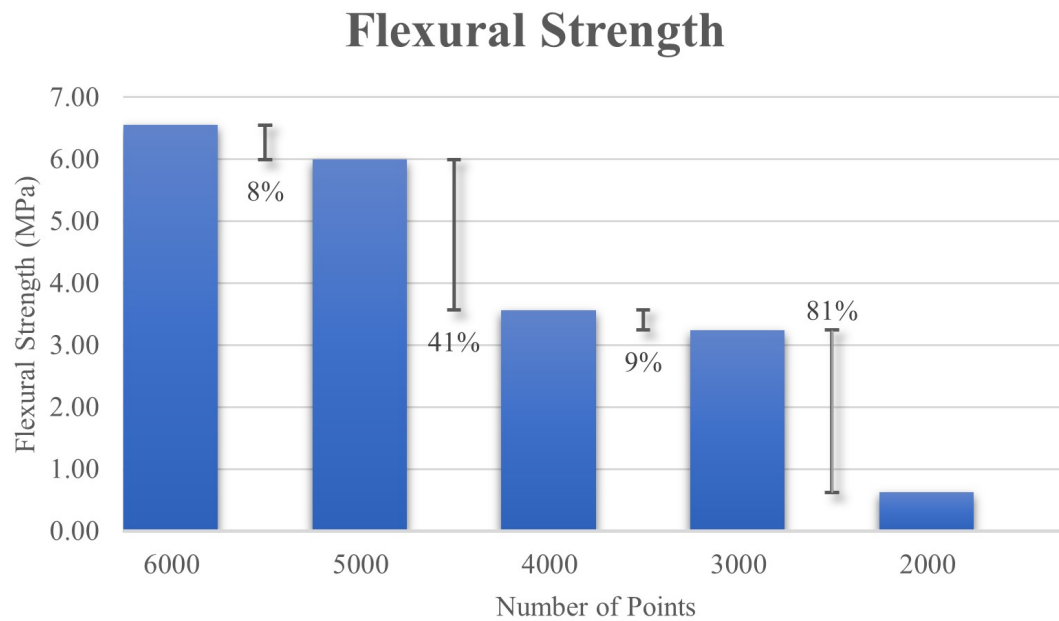


Figure 4.17: Flexural Strength (MPa) values for Voronoi samples with varying number of points.

4.2.1.3 Compressive Strength

Figure 4.18 displays compressive strength values for the fully dense specimen and Voronoi structures with points ranging from 2000 to 6000 in 1000-point intervals. Compressive strength reduction, compared to the control specimen, ranges from 92% to 99%, with the 3000 and 2000-point specimens experiencing the largest reductions.

Figure 4.19 focuses on the Voronoi structures and shows the reduction in compressive strength with decreasing points. The reduction is not linear, with variations as low as 11% and as high as 78% between samples. Notably, there is a 78% reduction in compressive strength from the 4000 to 3000-point specimens.

The variation in compressive strength with the number of points in the Voronoi structure displays varying reductions, with values ranging from 11% to 78% between samples. These differences are likely due to the geometrical complexity of the Voronoi lattice structure.

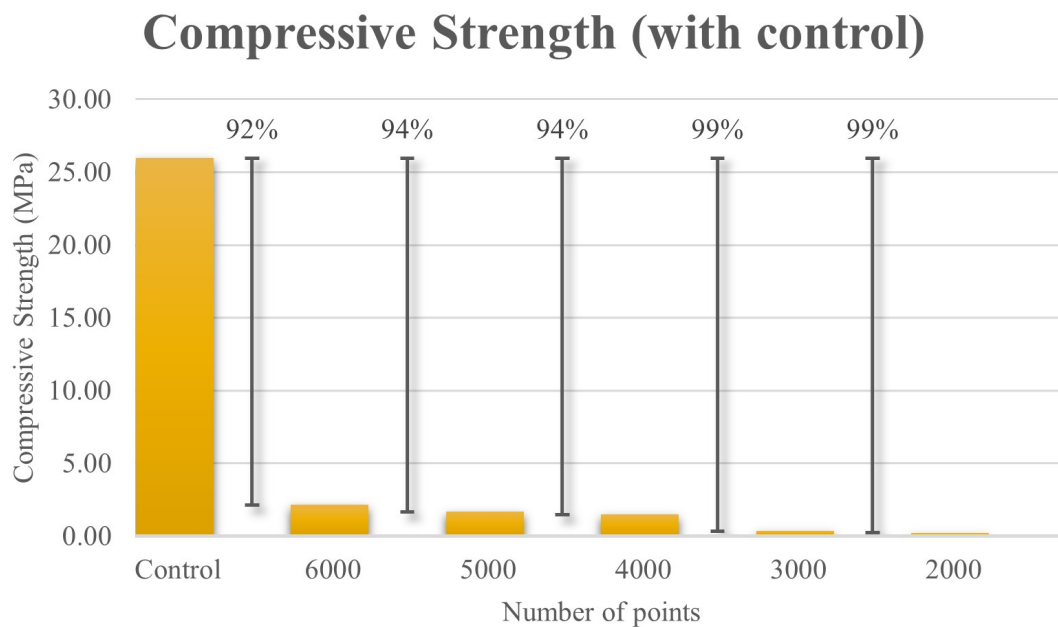


Figure 4.18: Compressive Strength (MPa) values for the fully dense control specimen, and Voronoi samples with varying number of points.

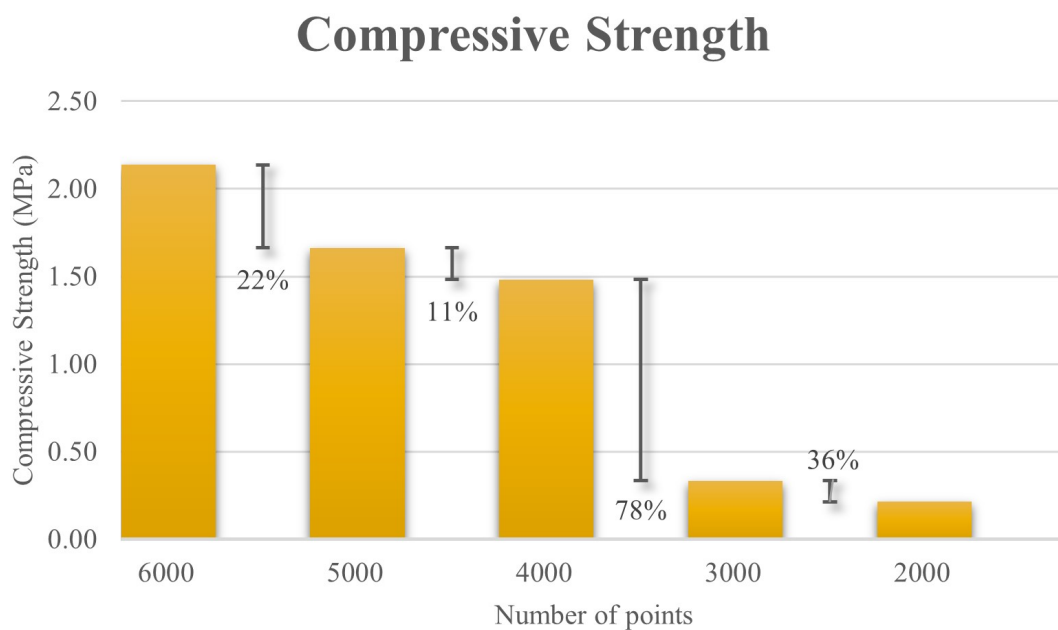


Figure 4.19: Compressive Strength (MPa) values for the fully dense control specimen, and Voronoi samples with varying number of points.

4.2.2 Wall Thickness

In this subsection, the impact of wall thickness (mm) on the Voronoi lattice structure's mechanical properties is examined. Three mechanical testing procedures were performed: traction, flexural, and compression testing. The evolution of Young's modulus, yield strength, flexural strength, and compressive strength is analyzed as the wall thickness is varied. These results are compared to those of a fully dense control specimen. The Voronoi structures have wall thicknesses of 0.6, 0.5, and 0.4 mm, varying in 0.1 mm increments.

4.2.2.1 Tensile Properties

Figure 4.20 presents Young's modulus values for the control specimen and Voronoi structures with wall thicknesses of 0.6, 0.5, and 0.4 mm. Young's modulus decreases gradually with decreasing wall thickness, with reductions of 57%, 62%, and 83% compared to the control.

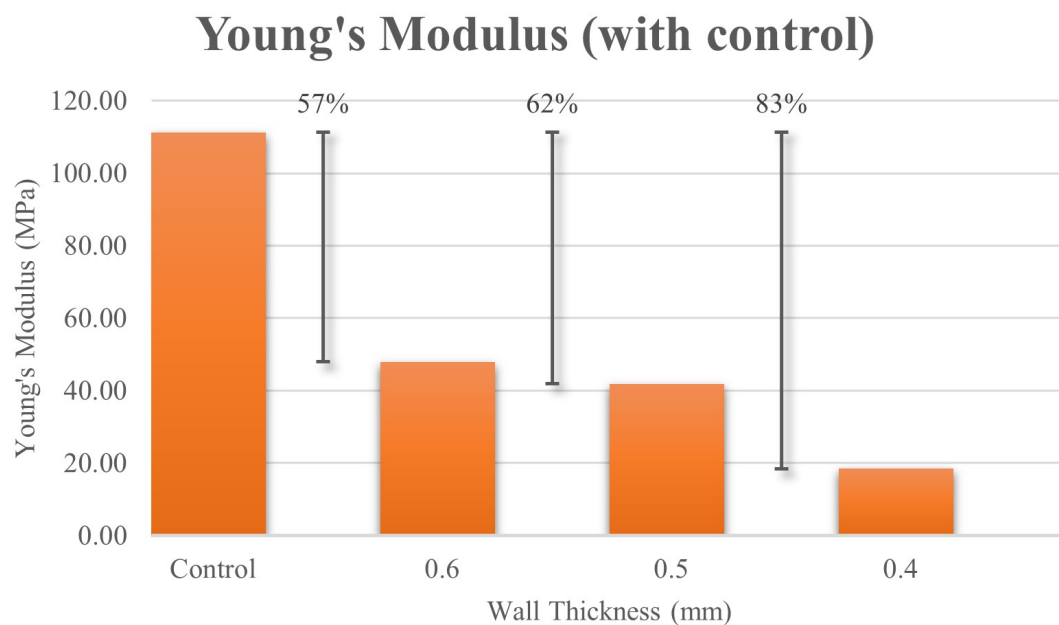


Figure 4.20: Young's modulus (MPa) values for the fully dense control specimen, and Voronoi samples with varying wall thickness (mm).

Figure 4.21 focuses on the Voronoi structures and shows the Young's modulus values with different wall thicknesses. It demonstrates a 13% reduction from 0.6 mm to 0.5 mm and a more significant 56% reduction from 0.5 mm to 0.4 mm.

Figure 4.22 displays yield strength values for the control specimen and Voronoi structures with varying wall thicknesses. There is a gradual reduction in yield strength, with reductions of 80%, 83%, and 93% compared to the control.

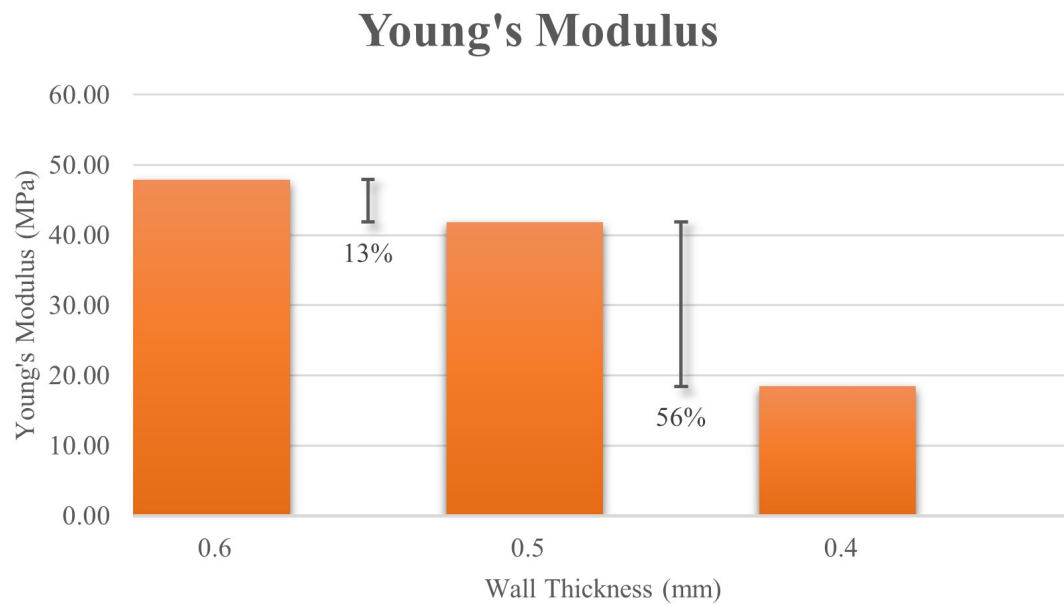


Figure 4.21: Young's modulus (MPa) values for Voronoi samples with varying wall thickness (mm).

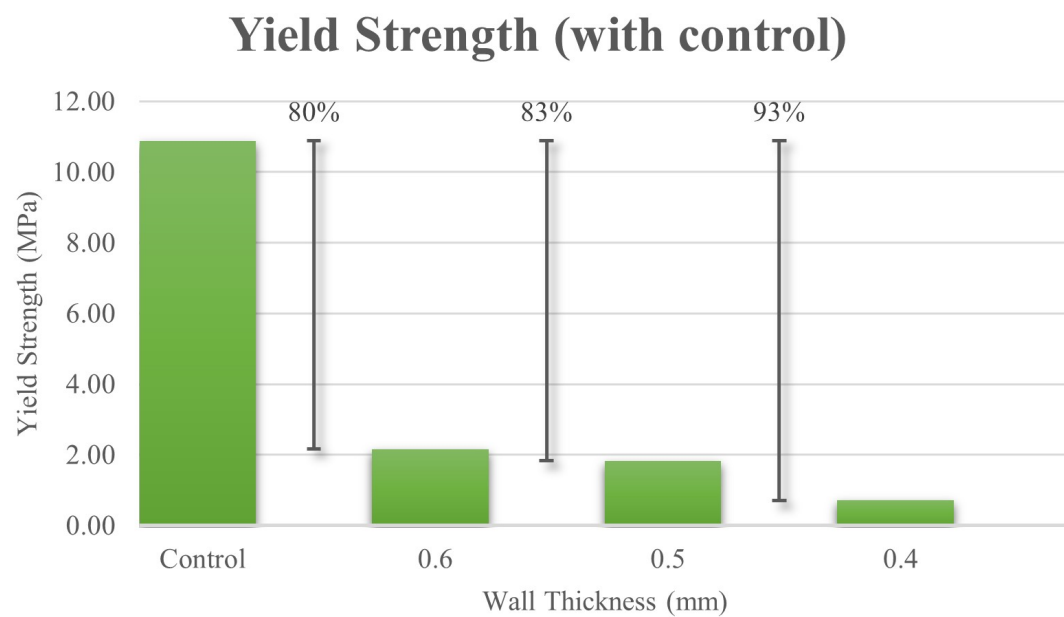


Figure 4.22: yield strength (MPa) values for the fully dense control specimen, and the Voronoi samples with varying wall thickness (mm).

Figure 4.23, concentrating on the Voronoi structures, illustrates yield strength values with different wall thicknesses. Similar to Young's modulus, there is a 16% reduction from 0.6 mm to 0.5 mm and a more substantial 61% reduction from 0.5 mm to 0.4 mm.

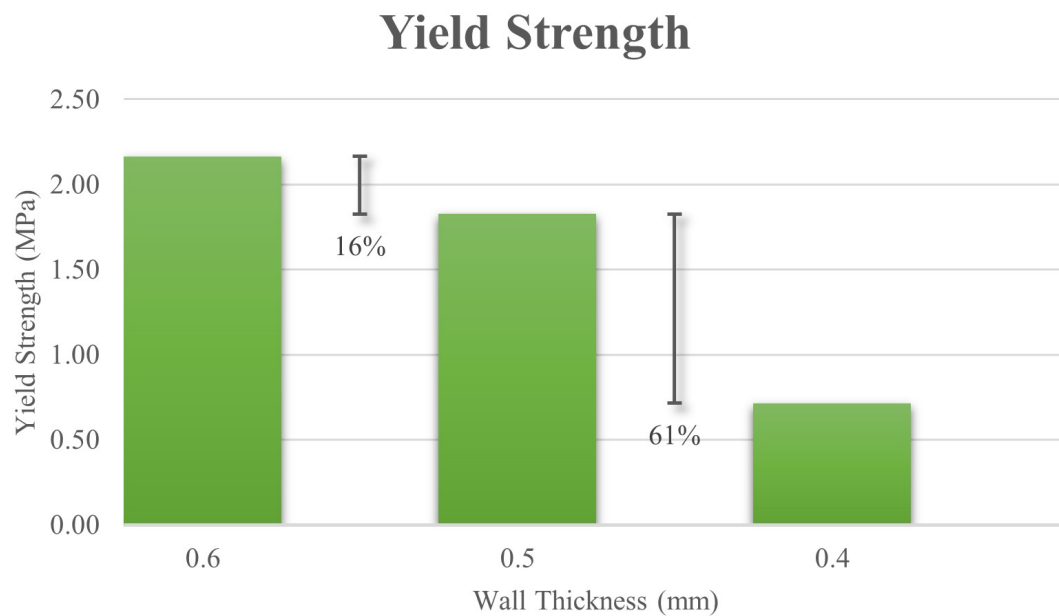


Figure 4.23: Yield Strength (MPa) values for Voronoi samples with varying wall thickness (mm).

In summary, reducing wall thickness leads to a decrease in Young's modulus ranging from 57% to 83%. Yield strength follows a similar trend, with reductions from 80% to 93%. The reduction values differ significantly between transitions, indicating non-linearity.

4.2.2.2 Flexural Strength

Figure 4.24 displays flexural strength values for the fully dense specimen and Voronoi structures with wall thickness values of 0.6 mm, 0.5 mm, and 0.4 mm. The reduction in flexural strength, compared to the fully dense control, ranges from 68% to 93%.

Figure 4.25 eliminates the control specimen and shows the flexural strength of the Voronoi structures with different wall thicknesses. It illustrates that the reduction in strength follows an almost linear pattern, with reductions of 56% and 53%.

The variation of flexural strength with wall thickness in the Voronoi structure follows linear evolution and has an average reduction value of 54%.

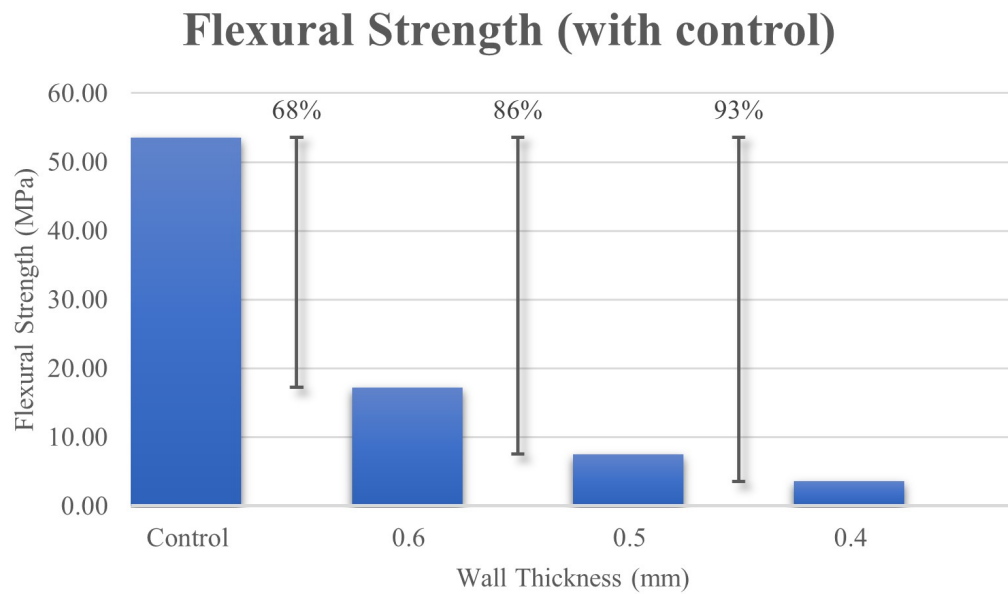


Figure 4.24: Flexural Strength (MPa) values for the fully dense control specimen, and Voronoi samples with varying wall thickness.

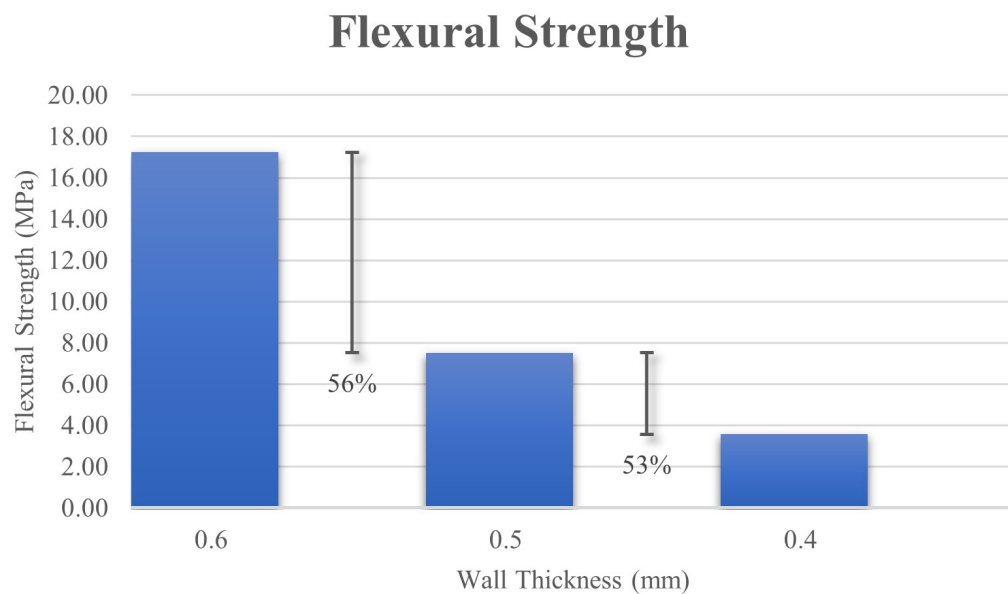


Figure 4.25: Flexural Strength (MPa) values for Voronoi samples with varying wall thickness.

4.2.2.3 Compressive Strength

Figure 4.26 displays compressive strength values for the fully dense specimen and Voronoi structures with different wall thicknesses (0.6, 0.5, and 0.4 mm). The reduction in compressive strength, compared to the control specimen, ranges from 76% to 94%.

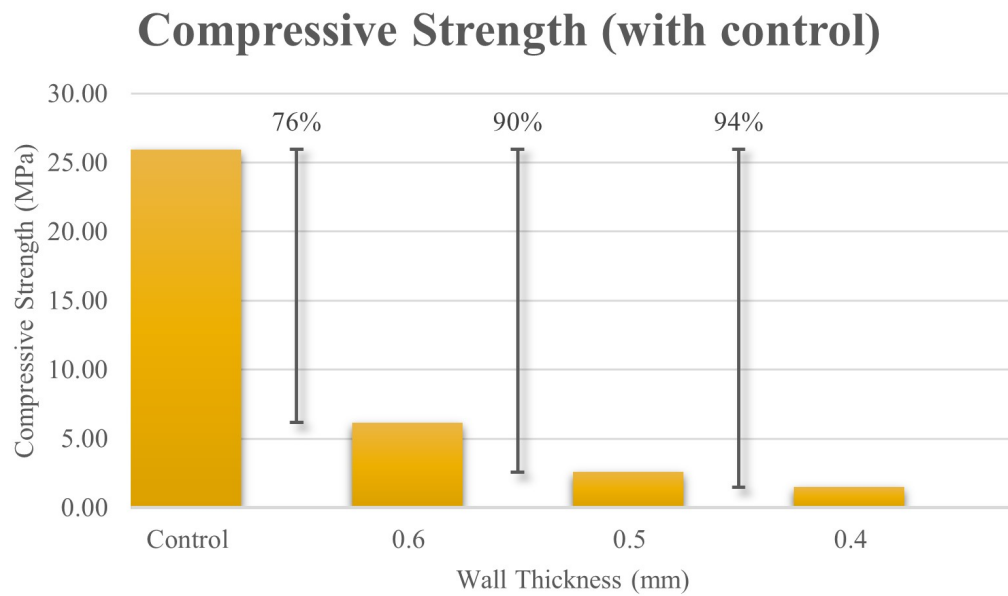


Figure 4.26: Compressive Strength (MPa) values for the fully dense control specimen, and Voronoi samples with varying wall thickness.

Figure 4.27 removes the control specimen and presents the compressive strength of the Voronoi structures with varying wall thicknesses. The reduction is similar in both cases, with a 58% reduction from 0.6 to 0.5 mm and a 42% reduction from 0.5 to 0.4 mm.

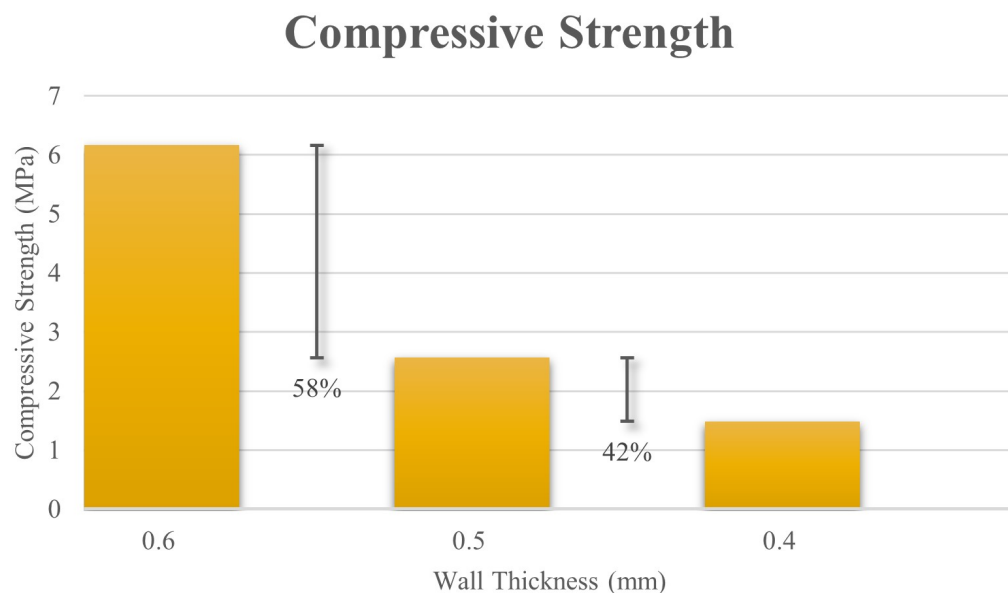


Figure 4.27: Compressive Strength (MPa) values for the fully dense control specimen, and Voronoi samples with varying wall thickness.

The variation in compressive strength with wall thickness in the Voronoi structure exhibits somewhat linear trends. This type of variation is what would be expected when altering geometrical parameters in Voronoi lattice structures.

4.2.3 Data Validation

To ensure the quality and accuracy of the obtained results, a data validation process was conducted. This process involved the use of the coefficient of variation (CV), which is a normalized measure of data variability. It is calculated as the ratio of the standard deviation (SD) to the result's average and is employed to assess the relative variability of different datasets. A low CV typically indicates that a process is stable and consistent, while a high CV may suggest a high variation in data that is usually due to errors, and improvement of the process is recommended [115][116]. The standard deviation quantifies the extent to which individual data points deviate from the average within a dataset, and it plays a fundamental role in the calculation of the coefficient of variation [117].

Tables 4.1, 4.2, and 4.3 present the CV values obtained for each mechanical property dataset across all sample types. These values serve as indicators of data reliability and consistency.

Table 4.1: Coefficient of variation across all sample types for the Young's modulus and yield strength.

Sample Type	CV - Young's Modulus	CV - Yield Strength
Control	0.05	0.05
6000	0.06	0.15
5000	0.07	0.08
4000	0.08	0.02
3000	0.08	0.10
2000	0.34	0.07
0.6	0.06	0.10
0.5	0.01	0.04
0.4	0.08	0.02

Table 4.2: Coefficient of variation across all sample types for the flexural strength.

Sample Type	CV - Flexural Strength
Control	0.07
6000	0.05
5000	0.10
4000	0.05
3000	0.09
2000	0.23
0.6	0.05
0.5	0.05
0.4	0.05

Table 4.3: Coefficient of variation across all sample types for the compressive strength.

Sample Type	CV - Compressive Strength
Control	0.05
6000	0.15
5000	0.02
4000	0.08
3000	0.04
2000	0.05
0.6	0.03
0.5	0.06
0.4	0.10

By examining the CV values provided in the tables, it becomes feasible to pinpoint significantly elevated values that stand out from the remainder. As mentioned earlier, an unusually high CV value is indicative of a dataset with reduced confidence. With that in mind, it is worth noting that the CV values for Young's modulus in the case of the 2000-point sample and for flexural strength in the case of the 2000-point sample exhibit higher values compared to the other datasets. As a result, additional testing should be conducted for these specific cases to further assess their reliability.

4.3 Mechanical Properties Results Interpretation

To better understand the results of the mechanical properties concerning the variation in the number of points and wall thickness (mm) in the Voronoi lattice structures, the results from both the 316L SLM tests and the resin tests were further analyzed.

Comparing the reduction in compressive strength between the tested SLM 316L samples and the resin samples, a similar reduction value of 88% and 94% was observed, respectively, for Voronoi structures with 4000 points and a wall thickness of 0.4 mm. This similarity suggests that the variations in Voronoi geometrical parameters have comparable effects on mechanical properties across different materials and manufacturing processes.

The mechanical properties of the control specimens in resin were similar to those provided by the resin manufacturer, as discussed in Chapter 3. This information enhances the understanding of the testing and production methods of the resin samples and the obtained results.

Table 4.4 presents reduction percentage values in resin for the transition from control samples to both the 6000-point samples and the samples with 0.6 mm wall thickness. For the number of points, the average reduction in mechanical properties from the initial control specimen is approximately 86%, while for the 0.6 mm wall thickness specimens, the average reductions are closer to 70%. These are the reductions that should be expected when transitioning from fully dense material designs into Voronoi lattice structure designs.

Table 4.4: Mechanical properties reductions, in resin, from the control specimens, to both the 6000 points specimen and the 0.6 mm wall thickness specimen.

Transition	Young's Modulus (MPa)	Yield Strength (MPa)	Flexural Strength (MPa)	Compressive Strength (MPa)
Control to 6000 Points	76%	87%	88%	92%
Control to 0.6 mm Wall Thickness	57%	80%	68%	76%

Tables 4.5 and 4.6 display results for all properties in resin for the control specimen, number of points specimens, and wall thickness specimens. These tables illustrate the reduction values of each mechanical property with each increment in both the number of points and wall thickness (mm). They provide an overview of how the variation of Voronoi structure geometrical parameters affects mechanical properties. The tables also include the average reduction per parameter increment and the reduction range (difference between the maximum and minimum reduction) observed in each mechanical property.

Table 4.5 showcases the results for the variation in the number of points in the Voronoi lattice structure. The average reduction per increment for each mechanical property is relatively close, ranging from 31.5% to 36.8%. This consistency indicates that a 1000-point increment in the Voronoi structure has a similar impact on all tested mechanical properties. However, the range of reduction at each property is notably high for Young's modulus, flexural strength, and compressive strength, with values of 54%, 73%, and 67%, respectively.

Table 4.5: Average mechanical properties, in resin, with number of points variation.

Number of Points	Young's Modulus (MPa)	Yield Strength (MPa)	Flexural Strength (MPa)	Compressive Strength (MPa)
Control	111.24	10.88	53.56	25.95
6000	27.2	1.39	6.55	2.14
6000 to 5000	18%	25%	8%	22%
5000 to 4000	17%	32%	41%	11%
4000 to 3000	71%	37%	9%	78%
3000 to 2000	20%	33%	81%	36%
Average	31.5%	31.8%	34.8%	36.8%
Range	54%	12%	73%	67%

Table 4.6 presents results for the variation in wall thickness (mm) in the Voronoi lattice structure. The average reduction per 0.1 mm increment can be divided into two groups. Young's modulus and yield strength have average reductions of 34.5% and 38.5%, respectively, which align with their interdependent nature. Flexural strength and compressive strength form the second group, with average reductions of 54.5% and 50%, respectively. These values represent the expected vari-

ation in mechanical properties with a 0.1 mm increment in wall thickness of the Voronoi lattice structure. The range of reductions is also variable, with values as low as 3% and as high as 45% for flexural strength and yield strength, respectively.

Table 4.6: Average mechanical properties, in resin, with wall thickness (mm) variation.

Wall Thickness (mm)	Young's Modulus (MPa)	Yield Strength (MPa)	Flexural Strength (MPa)	Compressive Strength (MPa)
Control	111.24	10.88	53.56	25.95
0.6	47.92	2.16	17.23	6.16
0.6 to 0.5	13%	16%	56%	58%
0.5 to 0.4	56%	61%	53%	42%
Average	34.5%	38.5%	54.5%	50%
Range	43%	45%	3%	16%

In conclusion, when varying the number of points and wall thickness (mm) in Voronoi lattice structures, the average values of mechanical property variation demonstrate that with each parameter increment (1000 points or 0.1 mm wall thickness), a similar reduction in mechanical properties can be expected. However, the range of reductions in most properties is high, indicating that predicting the exact reduction value of a specific mechanical property in Voronoi structure design is challenging. This phenomenon is likely due to imperfections and defects resulting from the geometrical complexity and random nature of stochastic Voronoi structures, leading to increased strains in certain cell walls and potential anisotropy, as described in the literature. To mitigate these variations, further testing and exploration across different design iterations of the same type of structure are recommended. Since each Voronoi structure is randomly generated, structures of the same type may exhibit slight differences in properties across different generations, contributing to the current high range of results in mechanical tests.

Chapter 5

Conclusions and Future Works

5.1 Conclusions

The experimental work conducted in this dissertation played a pivotal role in advancing the development and production of biodegradable porous iron Voronoi implants using SLM technology, within the context of the GradImp project. This research effort encompassed a complex and wide-ranging array of topics, including biomedical orthopedic implants, innovative generative design techniques, and the emerging AM technologies of SLM and vat-photopolymerization. This comprehensive approach facilitated a rigorous and extensive assessment of the methodology employed and the resulting outcomes.

A thorough investigation and meticulous analysis were undertaken to examine the production of 316L Voronoi structures utilizing Selective Laser Melting (SLM) technology. This involved a multi-faceted approach, incorporating SEM observation of metallic powder, visual assessment of printed samples, and microscopic examination of the microstructure of these samples. Additionally, compression testing was performed on both Voronoi samples and fully dense control samples to elucidate on the mechanical characteristics of 316L Voronoi structures.

Similarly, a parallel analytical methodology was applied to investigate the mechanical behavior of Voronoi lattice structures produced in resin. This analysis incorporated the manipulation of geometric parameters, including the number of points and wall thickness. The study placed particular emphasis on a wide spectrum of mechanical properties, which encompassed traction, flexural, and compression testing. The validity of their findings was further reinforced through a rigorous data analysis method.

Lastly, a comprehensive comparative analysis of the mechanical behavior of Voronoi structures was conducted, incorporating the results obtained from both the 316L SLM samples and the resin VPP samples.

The primary conclusions of this dissertation are summarized below, categorized into the major topics discussed earlier.

316L - SLM

- The SEM analysis of metallic 316L powder unveiled the presence of a minimal yet acceptable number of satellites, while significant particle aggregations were notably absent. Furthermore, there was no discernible porosity detected in the powder samples, and the predominant morphology of the powder particles was consistently regular and spherical. These observations collectively affirm that the virgin powder used in this study exhibits exceptional quality, devoid of significant defects that might adversely impact SLM production;
- Upon visual inspection of various Voronoi samples, noticeable defects and geometric inaccuracies were identified along the sample sides. In contrast, the top portions of the samples exhibited no such issues. This observation underscores the imperative need for further optimization of SLM parameters when producing Voronoi structures;
- The microscopic analysis yielded valuable insights into crucial aspects of the SLM process. These insights included observations of the scanning strategy employed and the presence of distinctive curved-shaped melt pools. Additionally, characteristic columnar grains were discerned, extending across the melt pools in alignment with the direction of the solidification structures;
- The microscopic analysis also revealed the presence of several defects. These included gas-induced porosities within the samples, as well as pattern porosities arising due to the scanning strategy. Additionally, an excess of spatter defects was observed at the edges of the Voronoi structures. These findings underscore the imperative requirement for optimizing the SLM process when producing Voronoi lattice structures;
- Despite encountering issues with the testing equipment during the compression testing of both fully dense control samples and Voronoi samples, a comparison between the two sets of specimens was still feasible. This comparison revealed a substantial reduction in compressive strength, amounting to 88%, in the Voronoi samples when compared to the control specimens.

Resin - VPP

- When assessing the impact of the number of points on Young's modulus, a consistent average reduction of approximately 20% was observed in the majority of cases. However, it's noteworthy that in some instances, there was a significantly higher reduction, reaching up to 71%;
- The influence of the number of points on yield strength displayed a consistent linear trend, indicating a reduction of approximately 32% with each incremental decrease of 1000 points;
- The influence of the number of points on flexural strength did not exhibit a linear progression. Instead, each increment led to reductions that varied, spanning from as low as 8% to as high as 81%;

- The impact of the number of points on compressive strength likewise demonstrated disparate outcomes, with each increment of 1000 points resulting in reductions ranging from as low as 11% to as high as 78%;
- The influence of wall thickness on Young's modulus exhibited notably distinct outcomes with each 0.1 mm increment. In one case, a reduction of 13% was observed, while in the other, the reduction was substantially higher, reaching up to 56%;
- The effect of wall thickness on yield strength produced results similar to those observed for Young's modulus, with reductions of 16% and 61% recorded. This similarity is likely attributed to the interdependence between these two properties;
- The influence of wall thickness on flexural strength yielded more consistent and promising results, with nearly equal reductions of 56% and 53% observed with each increment. These findings align more closely with the expected behavior for this property;
- The effect of wall thickness on compressive strength exhibited similar reductions with each increment in wall thickness, registering values of 58% and 52%;
- Data validation was conducted utilizing the coefficient of variation to uphold the quality and precision of the results. The analysis revealed that the results for the 2000-point samples in both the Young's modulus and flexural strength datasets exhibited low reliability and should be subjected to further testing.

Voronoi Mechanical Behaviour

- The transition from a fully dense specimen to a Voronoi structure had a significant impact on mechanical properties, resulting in reductions ranging from 57% to 92%. The extent of reduction varied depending on the specific property and the geometric parameters of the Voronoi lattice, illustrating the substantial level of reduction and customization achievable through the use of Voronoi lattice structures;
- The consistent reduction patterns observed in all mechanical properties, whether through increments in the number of points or wall thickness, provided valuable insights for the design and evaluation of Voronoi structures. These patterns facilitated a more comprehensive understanding of their behavior and performance characteristics;
- While the average values of property reductions displayed similarities, the wide range of results observed in each reduction is cause for concern. This variability can be directly attributed to imperfections and defects arising from the intricate geometrical complexity and the inherently irregular nature of stochastic Voronoi structures. These factors may lead to increased strains in a select few cell walls and potentially introduce minor levels of anisotropy. Addressing this significant range of results requires further testing involving diverse samples, particularly across multiple iterations of the same sample type.

5.2 Future Works

In contemplating the future work that must be undertaken, it should be emphasized that the dissertation encompasses numerous complex topics. There is a desire to delve deeper into certain of these subjects, even though a comprehensive exploration was not undertaken in the current experimental work. These additional investigations are consistent with the objectives of the dissertation and the GradImp project. Furthermore, it is imperative to make improvements and undertake additional experiments to enhance the results and support the conclusions. Below, a list of the specific future tasks that are essential is presented:

- **Expansion of Specimen Testing:** Expanding the scope of the research involves conducting tests on a more diverse range of specimens. This entails examining different Voronoi types and various generations of the same sample type. By doing so, the aim is to establish a more comprehensive database, thereby mitigating the observed variation in results;
- **Diversification of Mechanical Testing:** To gain a holistic understanding of Voronoi lattice structures, it is essential to undertake additional mechanical testing. This includes subjecting these structures to fatigue testing, as well as conducting torsional tests to assess their performance under different loading conditions;
- **Reassessment of 316L SLM Samples:** The need for comprehensive evaluation calls for a reexamination of the 316L SLM samples. This reevaluation should encompass compression testing, as well as further exploration of previously mentioned tests such as traction, flexural, torsional, and fatigue tests;
- **SEM Analysis of 316L Microstructures:** To gain deeper insights into the microstructural aspects of 316L, it is essential to perform SEM analysis. This will provide valuable information regarding the morphology and characteristics of the microstructures;
- **Printing Parameter Optimization:** Optimizing the printing parameters for Voronoi structures in SLM is a critical endeavor. This optimization study should encompass both 316L and pure iron materials to enhance the quality and consistency of printed Voronoi structures;
- **Pure Iron Voronoi Structures:** The exploration of pure iron Voronoi lattice structures presents an intriguing avenue for investigation. This includes the production of such structures and a comprehensive examination of their mechanical properties. Additionally, SEM analysis of microstructures and powder analysis should be conducted to unravel key insights;
- **Biocompatibility Assessment:** To assess the potential application of pure iron Voronoi lattice structures in biomedical contexts, an investigation into their biocompatibility with the human body is warranted. This study holds promise for diverse applications in medical implants.

These proposed avenues for future work not only extend the horizons of the research but also highlight the dynamic and evolving nature of the field. Through these endeavors, the aim is to deepen the understanding of Voronoi lattice structures and make contributions to the broader body of knowledge in materials science and engineering.

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Appendix A

316L Compression Tests

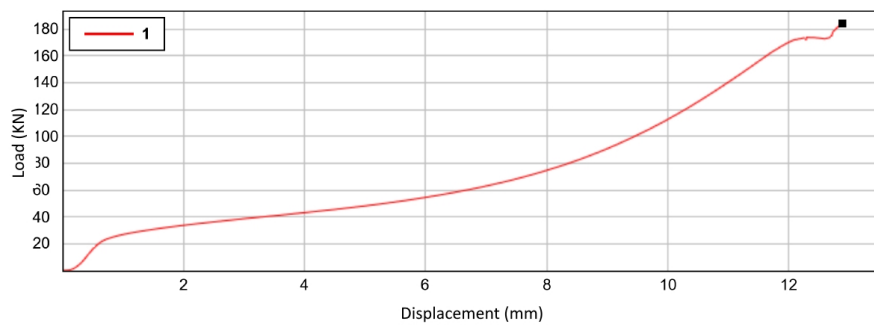


Figure A.1: Raw data of the SLM 316L compression testing, Voronoi sample number 1.

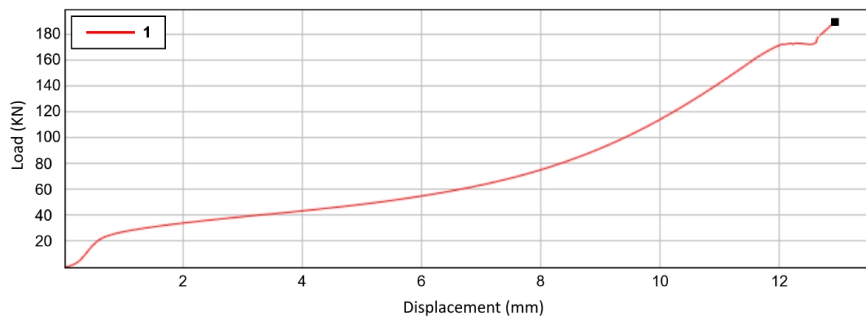


Figure A.2: Raw data of the SLM 316L compression testing, Voronoi sample number 2.

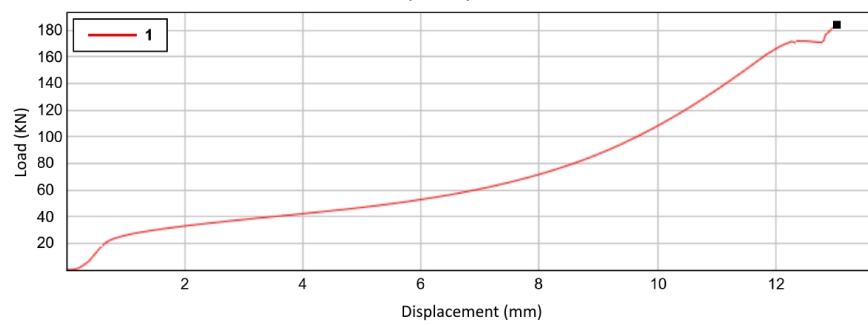


Figure A.3: Raw data of the SLM 316L compression testing, Voronoi sample number 3.

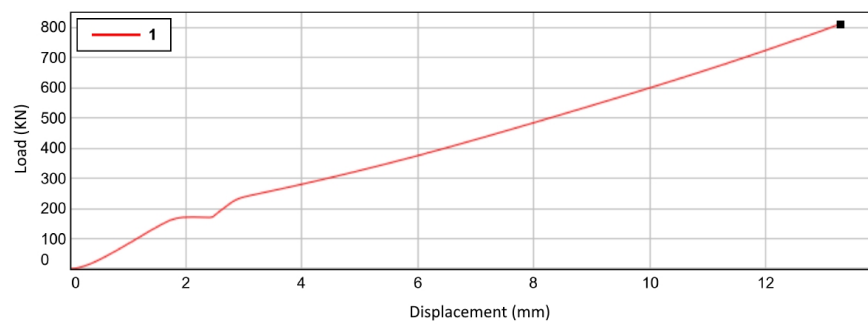


Figure A.4: Raw data of the SLM 316L compression testing, Control sample number 1.

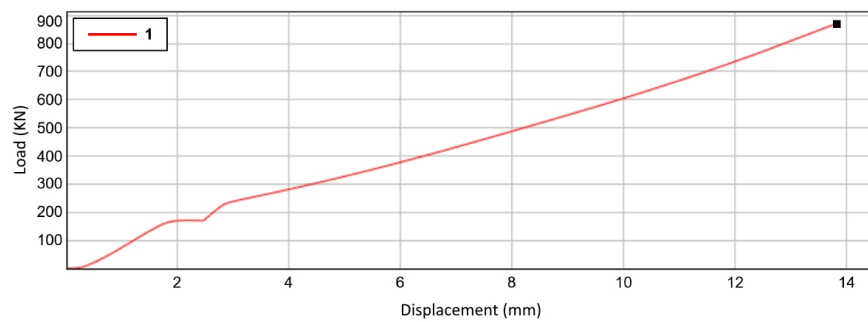


Figure A.5: Raw data of the SLM 316L compression testing, Control sample number 2.

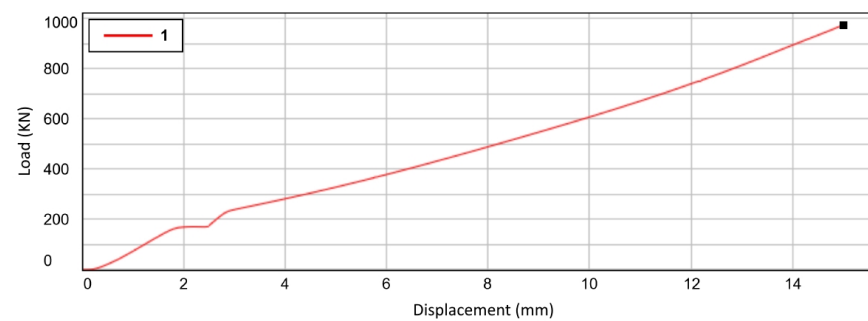


Figure A.6: Raw data of the SLM 316L compression testing, Control sample number 3.

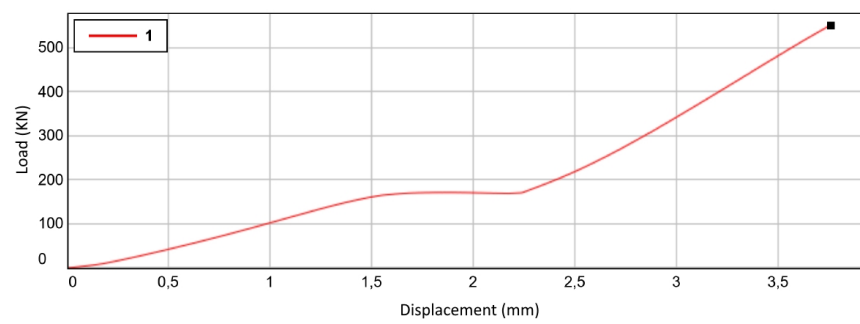


Figure A.7: Raw data of the compression testing, showcasing the rigidity fault on the machine.

Appendix B

Resin Mechanical Tests

B.1 Traction Tests

Table B.1: Results of the traction testing of resin sample's Young's modulus.

Young's Modulus (MPa)			
Sample Type	Sample 1	Sample 2	Sample 3
Control	113.66	115.19	104.88
6000	25.55	28.65	27.41
5000	22.75	20.49	23.35
4000	17.41	17.85	20.11
3000	5.24	5.86	5.05
2000	3.30	3.59	5.95
0.6	51.06	46.30	46.42
0.5	41.21	41.89	42.41
0.4	17.41	17.85	20.11

Table B.2: Results of the traction testing of resin sample's yield strength.

Yield Strength (MPa)			
Sample Type	Sample 1	Sample 2	Sample 3
Control	10.26	11.10	11.29
6000	1.18	1.60	1.38
5000	1.05	0.96	1.12
4000	0.73	0.70	0.71
3000	0.46	0.50	0.40
2000	0.31	0.32	0.28
0.6	2.40	2.03	2.06
0.5	1.74	1.90	1.84
0.4	0.73	0.70	0.71

B.2 Flexural Tests

Table B.3: Results of the flexural testing of resin sample's flexural strength.

Flexural Strength (MPa)			
Sample Type	Sample 1	Sample 2	Sample 3
Control	48.99	55.53	56.15
6000	6.36	6.33	6.95
5000	5.34	6.20	6.44
4000	3.48	3.79	3.43
3000	2.97	3.54	3.21
2000	0.70	0.46	0.70
0.6	18.21	16.77	16.70
0.5	7.41	7.16	7.97
0.4	3.48	3.79	3.43

B.3 Compression Tests

Table B.4: Results of the compression testing of resin sample's compressive strength.

Compressive Strength (MPa)	
Sample Type	Average
Control	25.95
6000	2.14
5000	1.66
4000	1.48
3000	0.33
2000	0.21
0.6	6.16
0.5	2.56
0.4	1.48