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Laser Powder Bed Fusion of Ti-5Al-5Mo- 5V-3Cr; Processability – Microstructure - Properties Analysis

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Turin, July 2026

Abstract

Ti-5Al-5Mo-5V-3Cr (Ti-5553) is a metastable β titanium alloy with potential for high-performance structural and biomedical applications because of its strength, hardenability, toughness, and corrosion resistance. In this thesis, as-built Ti-5553 specimens were fabricated by laser powder bed fusion (LPBF) using a TRUMPF TruPrint 1000 system and characterized to evaluate powder quality, consolidation, microstructure, mechanical behaviour, and corrosion response.

The Ti-5553 powder showed predominantly spherical morphology, limited satellite particles, and a particle size distribution of $D_{10} = 23.0 \mu\text{m}$, $D_{50} = 38.7 \mu\text{m}$, and $D_{90} = 56.3 \mu\text{m}$. The selected LPBF condition produced dense specimens, with a relative Archimedes density of 99.45%, an XCT defect volume ratio of 0.01%, and an ImageJ porosity of $0.301 \pm 0.041\%$. Optical microscopy showed characteristic LPBF melt-pool features, while XRD indicated a predominantly β -phase structure in both the as-built and tensile-tested conditions.

The specimens showed an average ultimate tensile strength of $834.6 \pm 4.7 \text{ MPa}$, fracture strain of $19.45 \pm 2.08\%$, and toughness of $139.96 \pm 15.49 \text{ MJ m}^{-3/2}$. Fracture analysis revealed crystallographic slip, isolated micropores, and mixed-mode fracture with both cleavage and dimpled regions. Nanoindentation showed hardness values of approximately 4.15–4.40 GPa and reduced modulus values of 88–100 GPa. Electrochemical testing in 0.9 wt.% NaCl solution showed passive corrosion behaviour, with $E_{\text{corr}} = -0.2203 \text{ V}$ and $I_{\text{corr}} = 7.45 \times 10^{-8} \text{ A}$.

The results show that the selected LPBF processing condition can produce dense as-built Ti-5553 with limited defects, retained β -phase stability, repeatable tensile response, mixed-mode fracture behaviour, and passive corrosion behaviour.

Keywords

LPBF; Ti-5Al-5V-5Mo-3Cr; as-built microstructure; mechanical properties; corrosion behaviour; powder analysis.

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Abbreviation	Definition
AM	Additive Manufacturing
CAD	Computer-Aided Design
DED	Direct Energy Deposition
L-PBF	Laser Powder Bed Fusion
SLM	Selective Laser Melting
EB-PBF	Electron Beam Powder Bed Fusion
CET	columnar-to-Equiaxed Transition
α	Alpha
HCP	Hexagonal Close-Packed
β	Beta
BCC	Body-Centered Cubic
ω	Omega
CP	commercially Pure
GB	Grain Boundary
BJ	Binder Jetting
UTS	Ultimate Tensile Strength
VED	volumetric energy density
EBM	Electron Beam Melting
LMD	Laser Metal Deposition
DMD	Direct Metal Deposition
P	Laser Power
v / S.S	Scan speed
h	Hatch Distance
t	layer Thickness
LOF	Lack Of Fusion
PSD	Particle Size Distribution
T _m	Melting Temperature
MA	Mechanical Alloying
CVD	Chemical Vapor Deposition
HCS	Hot-Cracking risk
L-DED	Laser-Directed Energy Deposition
EBSD	Electron Backscatter Diffraction
BSE	Backscattered Electron
TPMS	triply periodic minimal surface
XRD	X-ray Diffraction
SEM	Scanning Electron Microscopy
TEM	Transmission Electron Microscopy
I _{corr}	corrosion current density
EDS	Energy-Dispersive X-ray Spectroscopy
Q or GRF	Growth Restriction Factor
Mo _{eq}	Molybdenum equivalent
IPF	Inverse Pole Figure
R _{ct}	Charge-transfer resistance
I _{pp}	primary passivation current density
HAZ	Heat-Affected one

Chapter 1

Introduction

1.1 Background and motivation

Titanium and its alloys are among the most important metallic materials used in advanced engineering applications. Their relatively low density, high specific strength, excellent corrosion resistance, and good fatigue performance make them highly attractive for aerospace, biomedical, automotive, and energy-related components [1–5]. In applications where weight reduction and reliable mechanical performance are essential, titanium alloys offer a strong advantage over many conventional structural metals. This is mainly due to their favorable strength-to-weight ratio, which results from a low density of about 4.5 g cm^{-3} combined with high achievable strength levels [5–7].

A key reason for the versatility of titanium alloys is the allotropic transformation of titanium. At room temperature, titanium has a hexagonal close-packed α structure, while above approximately 882°C it transforms into the body-centered cubic β phase [3,4,8,9]. This transformation makes it possible to control the final microstructure through alloying, thermomechanical processing, and heat treatment. Depending on their chemical composition and phase stability, titanium alloys are usually classified as α , near- α , $\alpha+\beta$, near- β , and β alloys [4,5,8,9].

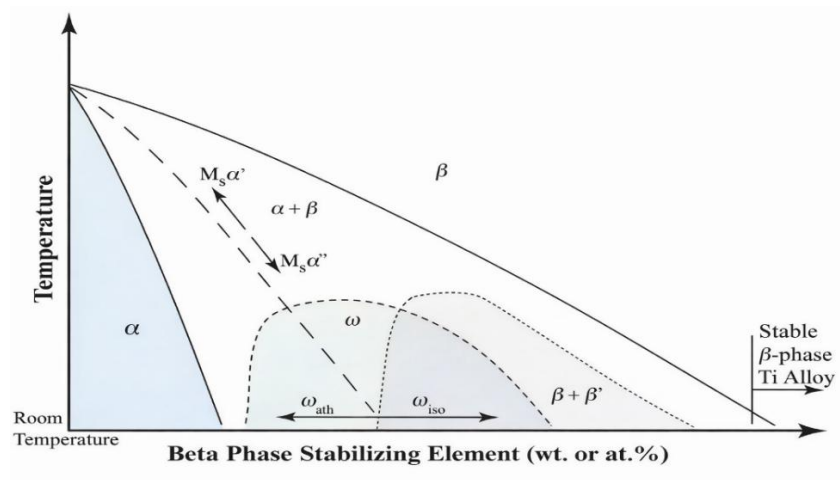


Figure 1. Schematic classification of titanium alloys according to phase stability and the effect of α - and β -stabilizing alloying elements [4].

The role of alloying elements in controlling phase stability is schematically shown in Fig. 1. In general, α -stabilizing elements expand the stability range of the α phase, whereas β -stabilizing elements lower the β -transus temperature and promote the retention of the β phase at lower temperatures. As the amount of β -stabilizing elements increases, the alloy system gradually shifts from α or $\alpha+\beta$ compositions toward near- β and β titanium alloys. This classification is particularly important because phase stability directly determines the transformation pathways, heat-treatment response, and mechanical behavior of titanium alloys[10–14].

Among these alloy families, metastable β titanium alloys are particularly important for high-strength structural applications. Their relatively high content of β -stabilizing elements, such as Mo, V, Cr, Nb, and Fe, allows the β phase to be retained after solution treatment. Subsequent ageing can then promote the controlled precipitation of fine α phases within the β matrix [4,8,9,13]. This combination of retained β phase and precipitation strengthening provides a useful balance of strength, toughness, fatigue resistance, and hardenability, which makes metastable β titanium alloys suitable for highly loaded aerospace components [9,10,15,16].

1.2 Ti-5553 alloy and LPBF processing challenges

Among metastable β titanium alloys, Ti-5Al-5Mo-5V-3Cr, commonly known as Ti-5553, has become an important candidate for high-strength aerospace structures. This alloy was developed to provide a combination of high strength, good fracture toughness, fatigue resistance, and deep hardenability, which are essential for thick and highly loaded components such as landing gears and other critical airframe structures [9,10,17–20]. The alloy chemistry contains Al as an α -stabilizing element, together with strong β -stabilizers such as Mo, V, and Cr. This compositional design allows the β phase to be retained after solution treatment and provides the possibility of further strengthening through controlled α precipitation during subsequent ageing [4,9–11,16].

Compared with conventional $\alpha+\beta$ titanium alloys such as Ti-6Al-4V, Ti-5553 can offer higher achievable strength and superior hardenability, particularly in large cross-sections where uniform mechanical properties are required [6,18,21]. However, the conventional manufacturing of Ti-5553 components remains challenging. Titanium alloys are generally difficult and costly to process because of their high chemical reactivity at elevated temperatures, low thermal conductivity, poor machinability, and significant material waste during subtractive manufacturing [22–25]. These limitations are especially important in aerospace applications, where complex component geometries and high buy-to-fly ratios can substantially increase production cost.

Laser Powder Bed Fusion (LPBF), also referred to as Selective Laser Melting (SLM), offers a promising route for producing Ti-5553 components with complex geometries and reduced material waste. In this process, a focused laser selectively melts thin layers of metallic powder according to a digital model, enabling near-net-shape fabrication with high geometrical flexibility[26–30]. For Ti-5553, LPBF is particularly attractive because the rapid solidification and repeated thermal cycling associated with the process can strongly influence β -phase retention, α precipitation, crystallographic texture, and defect formation [31–34].

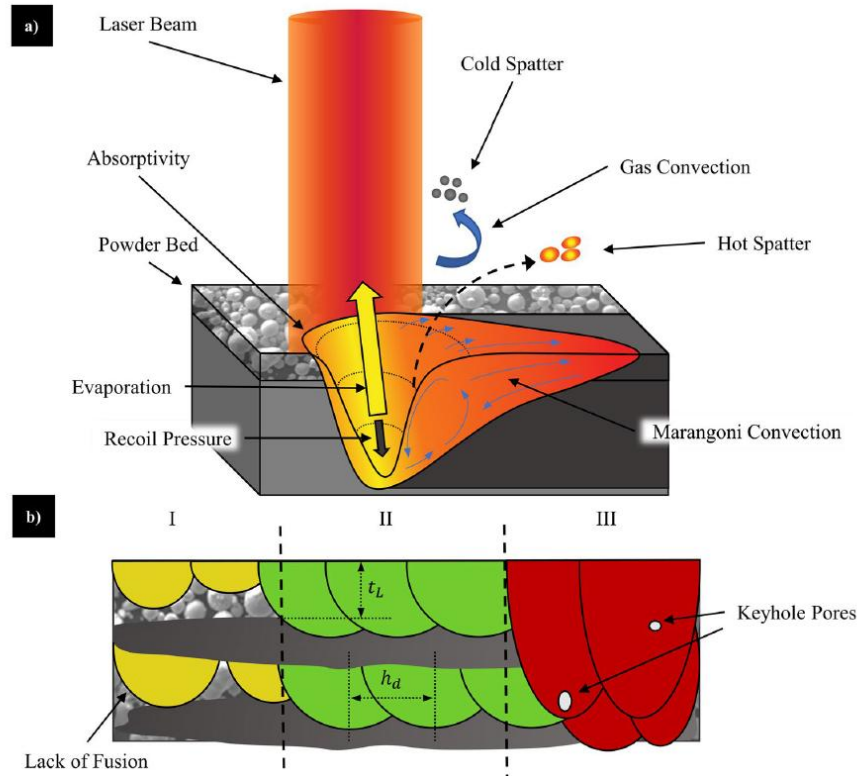


Figure 2. Schematic illustration of the LPBF process and associated melting regimes.(a) Laser–powder interaction, melt-pool formation, evaporation, recoil pressure, Marangoni convection, gas convection, and spatter generation. (b) Cross-section of an LPBF part produced under different processing regimes: lack of fusion at insufficient energy input, stable conduction-mode melting under optimized conditions, and keyhole-mode melting with keyhole pores at excessive energy input [35].

The main physical phenomena involved in LPBF are schematically illustrated in Fig. 2. During laser irradiation, energy absorption by the powder bed produces a localized melt pool, where heat transfer, Marangoni convection, evaporation, recoil pressure, and gas flow collectively control melt-pool stability [35]. Depending on the balance between energy input and heat dissipation, the process may operate in different melting regimes. At insufficient energy input, the melt pool is too shallow or discontinuous, leading to lack-of-fusion defects and incomplete bonding between adjacent tracks or layers[36]. Under stable intermediate conditions, conduction-mode melting promotes sufficient overlap and improved densification.

At excessive energy input, deep melt-pool penetration and vapor depression can produce keyhole instability, which may trap gas and generate near-spherical keyhole pores [35,37–40]. Therefore, although LPBF provides major manufacturing advantages, processing Ti-5553 by this route is scientifically and technologically complex. The final part quality depends on the combined effects of laser power, scan speed, hatch spacing, layer thickness, beam characteristics, and scanning strategy[23]. Insufficient energy input promotes lack-of-fusion porosity, whereas excessive energy input increases the risk of keyhole porosity, spatter formation, and melt-pool instability. Even when high relative density is achieved, LPBF Ti-5553 may still contain surface-connected defects, residual stresses, strong prior- β grain elongation, crystallographic texture, and local microstructural heterogeneity [35,41–43]. Moreover, the as-built microstructure of LPBF Ti-5553 cannot be fully described only by nominal process parameters such as volumetric energy density. It is also controlled by melt-pool geometry, local heat flow, intrinsic thermal cycling, and build orientation. These factors usually promote a predominantly retained β microstructure, but they may also lead to nanoscale ω formation or incipient α precipitation depending on the local thermal history [29,34–36]. As a result, the mechanical behavior of LPBF Ti-5553 is highly sensitive to defect morphology, surface condition, phase constitution, and microstructural anisotropy. Understanding these coupled process–microstructure–property relationships is therefore essential for the reliable application of LPBF Ti-5553 in high-performance structural components.

1.3 Research gap, objectives, and thesis outline

Although the feasibility of fabricating Ti-5553 by LPBF has already been demonstrated, the scientific understanding of this alloy under laser powder bed fusion conditions is still less mature than that of more widely studied titanium alloys such as Ti-6Al-4V. Existing studies have shown that near-full density can be achieved when suitable processing windows are selected; however, high relative density alone does not guarantee reliable mechanical or functional performance. Defect morphology, surface-connected porosity, melt-pool geometry, crystallographic texture, retained β stability, local thermal history, and surface condition can all strongly influence the final response of LPBF Ti-5553 [16,29,34–43].

A key challenge is that the process–microstructure–property relationships in LPBF Ti-5553 are highly coupled. Even when samples are fabricated under optimized processing conditions, the final material response can still be affected by residual porosity, surface roughness, phase constitution, microstructural heterogeneity, and fracture mechanisms. These factors may influence not only tensile behavior and local mechanical response, but also corrosion

performance, since surface condition, defects, and phase constitution can affect electrochemical stability. Therefore, a systematic experimental characterization is required to understand how the as-built condition of LPBF Ti-5553 controls its mechanical and corrosion behavior[44–46].

Another important aspect concerns the relationship between microstructure, defects, and local material response. Ti-5553 is a metastable β titanium alloy whose properties are strongly influenced by β -phase stability and possible secondary-phase formation. In LPBF materials, these features are additionally affected by rapid solidification and repeated intrinsic thermal cycling during layer-by-layer fabrication. As a result, the as-built microstructure of LPBF Ti-5553 must be analyzed together with defect distribution, phase constitution, tensile behavior, local hardness/modulus response, corrosion response, and fracture morphology [47–49].

Based on these considerations, the main aim of this thesis is to experimentally investigate the microstructure, defect state, phase constitution, mechanical behavior, and corrosion performance of Ti-5553 fabricated by Laser Powder Bed Fusion under optimized processing conditions. In this work, the Ti-5553 samples were produced using a TRUMPF TruPrint 1000 LPBF system, and the experimental activity focused on post-fabrication characterization rather than process parameter optimization.

The specific objectives of this thesis are:

- to characterize the Ti-5553 powder feedstock through particle size distribution analysis;
- to examine the surface condition and metallographic features of LPBF-fabricated Ti-5553 using optical microscopy;
- to evaluate internal defects and porosity using X-ray computed tomography;
- to characterize the microstructure and fracture surfaces using scanning electron microscopy;
- to identify the phase constitution of the LPBF Ti-5553 samples using X-ray diffraction;
- to assess the mechanical behavior through tensile testing and nanoindentation;
- to evaluate the corrosion behavior of LPBF Ti-5553 using electrochemical testing;
- to correlate the observed defects, microstructure, phase constitution, corrosion behavior, and fracture morphology with the measured mechanical response.

The thesis is organized as follows. Chapter 2 presents the state of the art on titanium alloys, Ti-5553 metallurgy, LPBF processing, defect formation, as-built microstructure, mechanical properties, and corrosion behavior. Chapter 3 describes the Ti-5553 powder feedstock, LPBF

fabrication using the TRUMPF TruPrint 1000 system, and the experimental methods used in this work, including particle size distribution, metallography, optical microscopy, computed tomography, SEM, XRD, tensile testing, nanoindentation, electrochemical corrosion testing, and fractography. Chapter 4 presents and discusses the experimental results, focusing on powder characteristics, surface and internal defects, microstructure, phase constitution, tensile response, local mechanical properties, corrosion performance, and fracture behavior. Finally, Chapter 5 summarizes the main conclusions and provides recommendations for future work on LPBF Ti-5553.

Chapter 2

State-of-the-art

2.1 Titanium alloys and Ti-5553 metallurgy

Titanium alloys are among the most important engineering materials for applications where a combination of low density, high specific strength, corrosion resistance, and reliable mechanical performance is required. Their use in aerospace, biomedical, automotive, marine, and energy-related sectors is mainly linked to their ability to provide high strength-to-weight ratios while maintaining good environmental resistance [1–5]. Compared with many conventional structural alloys, titanium alloys offer a relatively low density of approximately 4.5 g cm^{-3} , while their strength can vary widely depending on alloy composition, processing route, and heat-treatment condition [5,6,50]. This broad range of attainable properties makes titanium alloys highly adaptable to different engineering requirements.

The versatility of titanium alloys is fundamentally related to the allotropic transformation of pure titanium. At room temperature, titanium has a hexagonal close-packed α structure, while above the β -transus temperature, approximately 882°C for pure titanium, it transforms into a body-centered cubic β phase [3,4,8,9]. This reversible transformation provides the metallurgical basis for controlling microstructure through alloying, cooling rate, thermomechanical processing, and heat treatment. By stabilizing either the α or β phase, alloying elements modify phase stability and transformation behavior, allowing titanium alloys to be designed for different combinations of strength, ductility, fracture toughness, creep resistance, and fatigue performance[51,52].

Alloying elements in titanium are generally classified according to their influence on α and β phase stability. Elements such as Al, O, N, and C act as α stabilizers and increase the β -transus temperature, thereby promoting the stability of the hcp α phase. In contrast, elements such as Mo, V, Cr, Nb, Fe, and Ta act as β stabilizers by lowering the β -transus temperature and expanding the stability range of the bcc β phase [4,8,9]. This distinction is essential because the final phase constitution of a titanium alloy strongly controls its mechanical behavior. Based on the relative stability of the α and β phases, titanium alloys are commonly divided into α , near- α , $\alpha+\beta$, near- β , and β alloys [4,5,8,9].

Each class of titanium alloys offers a different balance of properties. α and near- α alloys are typically preferred for high-temperature applications because of their good creep resistance and

microstructural stability. $\alpha+\beta$ alloys, such as Ti-6Al-4V, are widely used because they offer a balanced combination of strength, ductility, toughness, and fatigue resistance. Near- β and metastable β titanium alloys, however, are particularly attractive for applications requiring high strength, deep hardenability, and strong response to ageing treatments [4,9,10]. These alloys contain sufficient amounts of β -stabilizing elements to retain a β -dominated microstructure after solution treatment or rapid cooling, while subsequent ageing can induce controlled precipitation of fine α phase within the β matrix.

The mechanical properties of metastable β titanium alloys are highly sensitive to the morphology, size, distribution, and orientation of the α precipitates. Fine and uniformly distributed α precipitates can significantly increase strength by impeding dislocation motion, while the retained β matrix can contribute to toughness and ductility. However, excessive α precipitation, coarse α plates, continuous grain-boundary α , or heterogeneous precipitation may reduce ductility and damage tolerance [9–11,15]. Therefore, the performance of metastable β titanium alloys is not governed only by chemical composition, but also by the thermal history that determines phase transformation and precipitation behavior.

Ti-5Al-5Mo-5V-3Cr, commonly referred to as Ti-5553, is a high-strength metastable β titanium alloy developed for advanced structural applications. Its composition combines aluminum as an α -stabilizing element with molybdenum, vanadium, and chromium as strong β -stabilizing elements. This alloy design enables the retention of a β -rich matrix after solution treatment and provides a strong precipitation-hardening response through the formation of fine α phase during ageing [4,9–11]. Because of this microstructural flexibility, Ti-5553 can achieve high strength while maintaining useful levels of toughness and fatigue resistance.

One of the major advantages of Ti-5553 is its deep hardenability. Unlike some conventional titanium alloys, whose properties may vary significantly across large sections, Ti-5553 can maintain relatively uniform mechanical properties in thick components. This makes it particularly suitable for heavily loaded aerospace structures, including landing gear components, wing attachments, and other critical structural fittings [9,10,17–20]. In such applications, the combination of high strength, fracture toughness, fatigue resistance, and hardenability is essential for safe and reliable service.

Compared with Ti-6Al-4V, which is the most widely used $\alpha+\beta$ titanium alloy, Ti-5553 offers higher achievable strength and better hardenability, especially in thick-section components [6,18,53]. However, these advantages are accompanied by strong sensitivity to processing history. Cooling rate, prior deformation, thermal exposure, and ageing conditions can all alter β stability and α precipitation behavior. As a result, Ti-5553 may exhibit significantly different

mechanical responses depending on whether it is conventionally processed, heat treated, or fabricated by additive manufacturing routes[5,13,54,55].

This sensitivity becomes particularly important in Laser Powder Bed Fusion. During LPBF, Ti-5553 experiences rapid melting and solidification, steep thermal gradients, and repeated intrinsic thermal cycling as new layers are deposited. These conditions differ strongly from conventional thermomechanical processing and can lead to non-equilibrium microstructures. In the as-built state, LPBF Ti-5553 is often dominated by retained β phase, but local thermal history may also promote nanoscale ω formation or incipient α precipitation under certain processing conditions [35,41–43]. Therefore, understanding the metallurgy of Ti-5553 is essential for interpreting its LPBF processability, microstructural evolution, mechanical response, and corrosion behavior.

Table 2. Main alloying elements in Ti-5553 and their metallurgical role.

Element	Nominal content	Metallurgical role	Main effect in Ti-5553
Ti	Balance	Matrix element	Base metal and α/β phase transformation
Al	~5 wt.%	α stabilizer	Solid-solution strengthening and α -phase stabilization
Mo	~5 wt.%	β stabilizer	β retention, hardenability, and precipitation response
V	~5 wt.%	β stabilizer	β -phase stability and strength contribution
Cr	~3 wt.%	β stabilizer	Improved hardenability and β stabilization

2.2 Additive manufacturing routes for titanium alloys

Additive manufacturing (AM) has become an important manufacturing approach for titanium alloys because it enables the fabrication of geometrically complex components with reduced material waste compared with conventional subtractive routes. This advantage is particularly relevant for titanium alloys, since their high material cost, poor machinability, low thermal conductivity, and strong chemical reactivity at elevated temperatures make conventional manufacturing expensive and technically demanding [22–24,56]. In aerospace and biomedical applications, where complex geometries, lightweight structures, and near-net-shape production are often required, AM provides a practical route for improving material efficiency and design flexibility.

In metal AM, components are fabricated directly from a digital model by adding material layer by layer. Depending on the feedstock form, heat source, and consolidation mechanism, several AM routes can be used for titanium alloys. As shown in Fig. 3, the leading metal AM processes can be broadly divided into directed-energy-based and powder-bed-based routes. Directed-

energy-based processes deliver powder or wire directly into a melt pool generated by a focused energy source, while powder-bed-based processes selectively melt regions of a pre-deposited powder layer using either an electron beam or a laser. This classification is useful because the way in which material is supplied and melted strongly affects melt-pool size, thermal history, cooling rate, surface quality, and final microstructure[29].

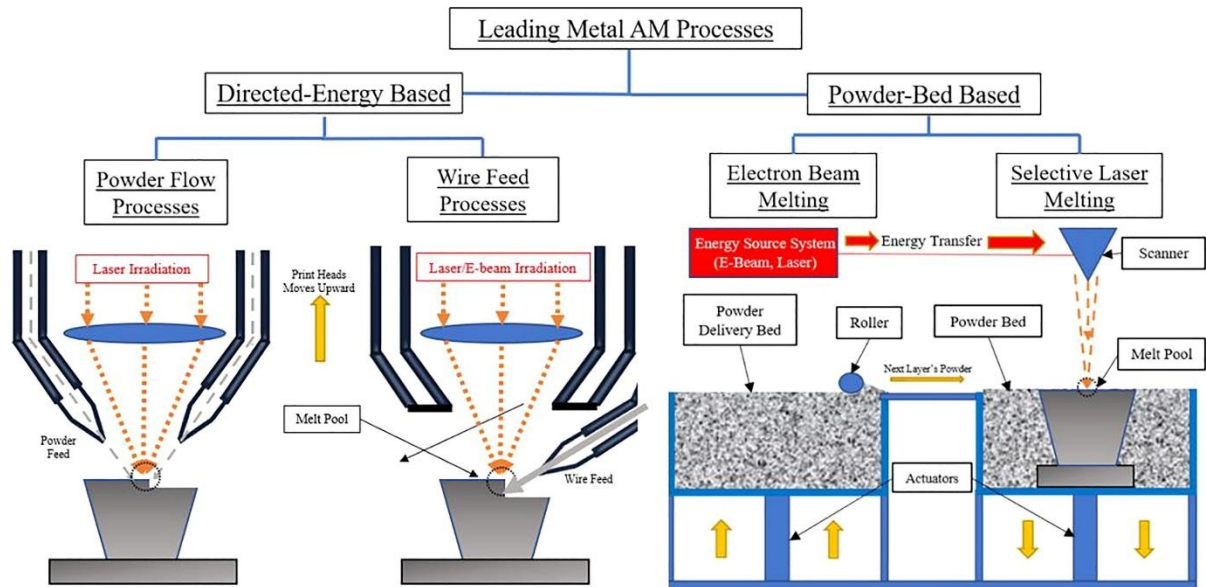


Figure 3. Schematic classification of leading metal additive manufacturing processes, showing directed-energy-based routes, including powder-fed and wire-fed DED, and powder-bed-based routes, including electron beam melting and selective laser melting [29].

The main beam-based processes used for titanium alloys include Directed Energy Deposition (DED), Electron Beam Powder Bed Fusion (EB-PBF), and Laser Powder Bed Fusion (LPBF), also referred to as Selective Laser Melting (SLM). Although these processes are based on localized melting and layer-by-layer fabrication, they operate under different thermal and geometrical conditions. DED typically involves larger melt pools and higher deposition rates, EB-PBF is performed under vacuum with elevated powder-bed temperatures, and LPBF uses a focused laser beam to produce small melt pools and rapid solidification. These differences are especially important for titanium alloys, because their final properties are highly sensitive to thermal history, phase transformation, porosity, and crystallographic texture[48,57].

2.2.1 Directed Energy Deposition (DED)

Directed Energy Deposition is a metal AM process in which powder or wire feedstock is delivered directly into a melt pool generated by a focused energy source, such as a laser, electron beam, or electric arc. As illustrated in the directed-energy-based branch of Fig. 2.1, the feedstock may be supplied either as a powder flow or as a wire feed. In powder-fed DED,

metallic powder particles are carried by a gas stream and injected into the melt pool, whereas in wire-fed DED, a metallic wire is continuously fed into the heat-affected region and melted during deposition. In both cases, the material is deposited along a programmed toolpath and the component is built by successive layers[50,58–60].

Compared with powder bed fusion processes, DED generally uses a larger melt pool, higher heat input, and higher deposition rate. These characteristics make it suitable for large-scale components, repair operations, cladding, and functionally graded materials. For titanium alloys, DED offers several advantages, including the ability to process powder or wire feedstock, manufacture large parts, and locally modify chemical composition during deposition. However, the larger melt pool and slower cooling rate usually lead to coarser microstructures compared with LPBF. In β and near- β titanium alloys, such thermal conditions may promote columnar grain growth, microstructural heterogeneity, and residual stress development depending on the deposition strategy and thermal accumulation[22,29,30].

Another limitation of DED is its lower dimensional accuracy and rougher surface finish compared with LPBF. Because material is delivered directly into the melt pool rather than being spread as a uniform powder layer, the deposited tracks are larger and the surface often requires subsequent machining or finishing. Therefore, although DED is highly valuable for large titanium components and repair applications, it is less suitable when fine geometrical resolution, small features, or highly localized microstructural control are required[61–64].

2.2.2 Electron Beam Powder Bed Fusion (EB-PBF)

Electron Beam Powder Bed Fusion is a powder-bed-based process in which an electron beam selectively melts regions of a metallic powder layer under vacuum. As shown in Fig.3, EB-PBF belongs to the powder-bed-based family, where powder is distributed across a build platform and selectively melted layer by layer. Since electron beams require electrically conductive materials and vacuum operation, EB-PBF is particularly suitable for reactive metallic materials such as titanium alloys[65–68].

A major feature of EB-PBF is the use of elevated build temperatures and powder-bed preheating. This reduces thermal gradients, lowers residual stress accumulation, and can decrease the risk of distortion or cracking during fabrication. For titanium alloys, this thermal environment can be beneficial because it reduces stress-related defects and provides a more stable build condition. However, the sustained thermal exposure also affects microstructural evolution. In Ti-6Al-4V, for example, EB-PBF commonly promotes $\alpha+\beta$ microstructures rather than highly metastable martensitic structures because the elevated temperature allows phase

decomposition during the build. For metastable β titanium alloys, similar thermal exposure may influence β stability, recovery, grain growth, and subsequent phase transformation behavior[29,31,49].

Despite these advantages, EB-PBF has limitations compared with LPBF. The process generally produces rougher surfaces and lower dimensional accuracy, partly because of the powder-bed preheating and beam characteristics. The minimum feature size is also typically larger than in LPBF. In addition, the need for vacuum operation and conductive feedstock limits process flexibility. Therefore, EB-PBF is an important route for titanium alloy fabrication, especially where residual stress reduction is desirable, but it is less suitable than LPBF for applications requiring high precision and fine feature resolution[69–72].

2.2.3 Laser Powder Bed Fusion (LPBF/SLM)

Laser Powder Bed Fusion, also known as Selective Laser Melting, is one of the most widely used metal AM processes for titanium alloys. As represented in the powder-bed-based branch of Fig. 3, LPBF uses a laser beam and a scanning system to selectively melt regions of a pre-deposited powder bed. A thin layer of metallic powder is first spread over the build platform, the laser scans the selected cross-section of the component, and the platform is then lowered before a new layer of powder is deposited. Repetition of this sequence allows the final component to be built directly from a digital model[50–53].

LPBF is characterized by highly localized laser–powder interaction, small melt-pool dimensions, steep thermal gradients, and rapid cooling rates, commonly reported in the range of 10^5 – 10^6 K s⁻¹. These conditions enable the production of fine microstructures and near-net-shape components with high dimensional accuracy. For titanium alloys, LPBF is particularly attractive because it can fabricate lightweight structures, lattice geometries, internal channels, and complex aerospace or biomedical components with reduced material waste[27,73–76].

At the same time, the rapid and localized thermal cycle of LPBF introduces several processing challenges. The final part quality is highly sensitive to laser power, scan speed, hatch spacing, layer thickness, beam size, scanning strategy, powder characteristics, and shielding atmosphere. If the energy input is insufficient, lack-of-fusion defects may form because of incomplete melting or poor overlap between adjacent scan tracks. If the energy input is excessive, deep vapor depressions and keyhole instability can occur, leading to gas entrapment and near-spherical pores. Spatter formation, residual stresses, surface roughness, and anisotropic grain growth are also common concerns in LPBF titanium alloys[45,48,52,77–79].

For Ti-5553, LPBF is especially interesting because the rapid solidification conditions can retain a β -dominated microstructure. However, repeated intrinsic thermal cycling during layer-by-layer fabrication may also promote local phase instabilities, including nanoscale ω formation or early-stage α precipitation, depending on the local thermal history. Therefore, LPBF does not only define the final geometry of Ti-5553 components; it also strongly influences phase constitution, prior- β grain morphology, texture, defect population, mechanical response, and corrosion behavior [19,23,24,33,80,81].

2.2.4 Comparison of AM routes and rationale for LPBF

The comparison shown in Fig. 3 highlights that the main difference between the principal beam-based AM routes lies in how material is supplied to the melt pool. In DED, material is continuously fed as powder or wire into the melt pool, while in powder-bed processes the material is first spread as a thin layer and then selectively melted. This distinction leads to major differences in melt-pool size, thermal exposure, cooling rate, surface quality, and geometrical precision[61–63,82,83].

DED generally provides high deposition rates and is suitable for large components and repair, but it often produces coarser microstructures and lower dimensional accuracy. EB-PBF provides vacuum processing and reduced residual stresses due to powder-bed preheating, but it usually results in rougher surfaces and lower feature resolution. LPBF, in contrast, offers higher geometrical accuracy, finer feature resolution, and rapid solidification conditions, making it particularly suitable for studying process–microstructure–property relationships in titanium alloys[70–72,84,85].

For metastable β titanium alloys such as Ti-5553, the selection of LPBF is both technologically and scientifically relevant. From a manufacturing perspective, LPBF enables the production of near-net-shape Ti-5553 components with complex geometries and reduced material waste. From a metallurgical perspective, its steep thermal gradients and rapid solidification conditions provide a unique environment for investigating β -phase retention, defect formation, crystallographic texture, microstructural heterogeneity, and mechanical response. For these reasons, LPBF is the central manufacturing route considered in this thesis.

2.3 LPBF processing of Ti-5553: parameters, melt-pool stability, and defects

Laser Powder Bed Fusion of Ti-5553 is governed by a strong interaction between processing parameters, powder-bed characteristics, laser–material interaction, and local thermal history.

In this process, the laser does not only consolidate the powder into a dense component; it also defines the solidification conditions that control defect formation, phase stability, grain morphology, and local microstructural heterogeneity. This is particularly important for metastable β titanium alloys such as Ti-5553, because relatively small changes in thermal history can influence β retention, α precipitation, possible ω formation, and the morphology of the prior- β grain structure[86–89].

For this reason, LPBF processing of Ti-5553 should be considered as a coupled process–structure problem. Parameters such as laser power, scan speed, hatch spacing, layer thickness, beam characteristics, and scanning strategy determine melt-pool dimensions, track continuity, interlayer bonding, and heat accumulation. These factors control whether the material is processed under insufficient melting, stable conduction-mode melting, or keyhole-dominated conditions. Therefore, the assessment of LPBF Ti-5553 cannot rely only on relative density; it must also include melt-pool stability, defect morphology, surface condition, and the resulting microstructural state[90–93].

2.3.1 Main LPBF parameters

The main processing parameters in Laser Powder Bed Fusion are laser power, scan speed, hatch spacing, layer thickness, beam diameter, beam profile, baseplate temperature, and scanning strategy. Although these parameters are usually defined independently in the machine setup, their effects during fabrication are strongly coupled. Together, they determine the energy absorbed by the powder bed, the dimensions and stability of the melt pool, the degree of overlap between adjacent scan tracks, the quality of interlayer bonding, and the extent of local heat accumulation. In LPBF Ti-5553, this coupling is particularly important because the same thermal conditions that control densification also affect solidification behavior, retained β stability, phase evolution, crystallographic texture, and defect formation[19,37,92–94].

Laser power and scan speed are widely identified as the dominant variables controlling melt-pool stability in LPBF Ti-5553. Laser power determines the amount of energy delivered to the powder bed, while scan speed controls the interaction time between the laser beam and the material. Increasing laser power generally increases melt-pool temperature, width, and penetration depth. A sufficiently high power is required to fully melt the powder layer and partially remelt the previously solidified material, ensuring proper bonding between layers. However, excessive power can produce strong evaporation, recoil pressure, deep vapor depression, and keyhole-type instability. Conversely, if the laser power is too low, the melt

pool may become shallow or discontinuous, leading to incomplete melting and lack-of-fusion defects[45,48,49,95,96].

Scan speed has the opposite effect on interaction time. At low scan speeds, the laser remains longer over a given location, increasing the local heat input and melt-pool penetration. This can improve consolidation when the initial energy input is insufficient, but it can also increase the risk of vaporization and keyhole formation when combined with high laser power. At high scan speeds, the laser–powder interaction time is reduced, which limits melt-pool penetration and lateral spreading. Under such conditions, discontinuous tracks, insufficient remelting, and lack-of-fusion porosity may develop. Therefore, the transition from lack-of-fusion to conduction-mode melting and then to keyhole instability in LPBF Ti-5553 is mainly governed by the coupled effect of laser power and scan speed [97–102].

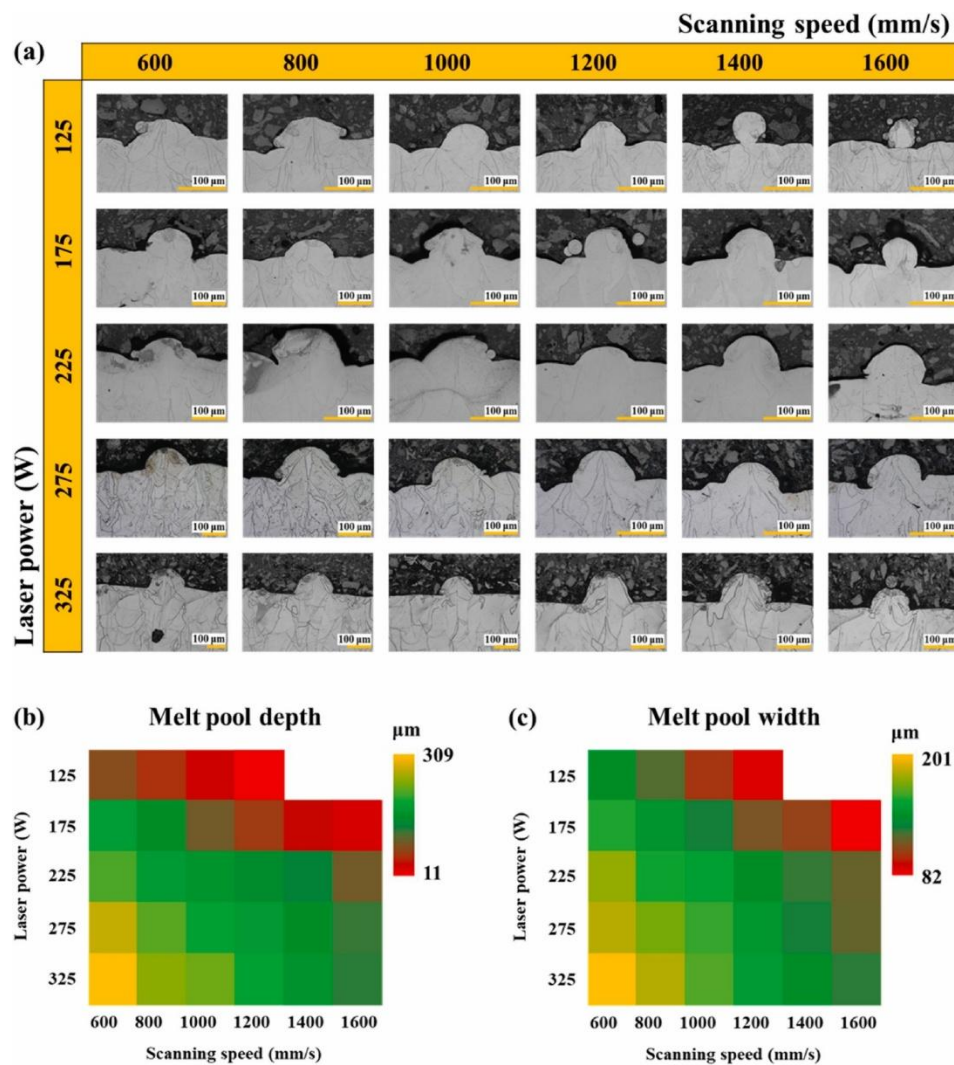


Figure 4. Melt-pool geometry evolution in LPBF Ti-5553 as a function of laser power and scanning speed[99].

The influence of laser power and scan speed on melt-pool geometry is shown in Fig. 4. The single-track cross-sections demonstrate that changes in the power–speed combination directly modify both melt-pool depth and width. In general, higher laser power increases penetration and widens the molten region, whereas higher scan speed reduces the interaction time and limits melt-pool growth. The depth and width maps further show that melt-pool geometry evolves systematically across the investigated processing space. This observation is important because melt-pool depth and width are more directly related to bonding quality and defect formation than nominal energy input alone[99].

A key implication of Fig. 4 is that process stability cannot be interpreted only from a single scalar parameter. Even when different power–speed combinations appear comparable in terms of nominal energy density, they may produce different melt-pool penetration depths, widths, and aspect ratios. This means that laser power and scan speed must be considered as coupled parameters rather than independent variables. In Ti-5553, this coupling is particularly relevant because excessive penetration can promote keyhole porosity, while insufficient penetration can lead to lack-of-fusion defects and weak interlayer bonding.

Hatch spacing introduces an additional level of parameter interaction because it controls the lateral overlap between adjacent melt tracks. If the hatch spacing is too large relative to the melt-pool width, neighboring tracks may not overlap sufficiently, leaving unmelted or poorly bonded regions between scan lines. This promotes lack-of-fusion defects even when laser power and scan speed appear to provide an acceptable energy input. On the other hand, reducing hatch spacing increases overlap and can improve densification, but it also increases heat accumulation, processing time, and the possibility of thermal distortion. In LPBF Ti-5553, near-full density conditions are commonly associated with hatch spacing values close to 95–100 μm when combined with stable laser power and scan speed conditions [98–100].

The importance of hatch spacing has also been confirmed by regression-based process studies. Statistical modelling over a constrained design space showed that hatch spacing exhibits a strong negative correlation with relative density, reflecting the reduction in inter-track overlap at larger hatch distances. Moreover, a strong interaction between laser power and hatch spacing has been reported, indicating that the effect of hatch spacing cannot be separated from the thermal input provided by the laser. These results show that densification in LPBF Ti-5553 is governed not only by the amount of energy delivered, but also by how effectively adjacent melt tracks coalesce during scanning [103].

Layer thickness controls the amount of powder deposited before each laser exposure and therefore affects the penetration depth required for interlayer bonding. A thicker layer increases

build productivity because fewer layers are required to fabricate a component. However, it also requires greater melt-pool penetration to fully melt the powder layer and remelt the underlying solidified material. If the penetration depth is insufficient, interlayer lack-of-fusion defects may form. Conversely, thinner layers require less penetration and can allow stable melting at higher scan speeds, but at the cost of longer build time. Reported LPBF Ti-5553 studies have used layer thicknesses such as 30 and 45 μm , and the stable processing window has been shown to shift with layer thickness [98–100].

This layer-thickness dependence is important when comparing processing windows across studies. For example, under thinner layer conditions of approximately 30 μm , relative densities above 99% have been reported within a VED interval of about 64.3–91.7 J mm^{-3} , with scan speeds approaching 1400 mm s^{-1} under suitable power–speed combinations [100]. In contrast, studies using a 45 μm layer thickness have identified different conduction-mode windows, often bounded by lack-of-fusion defects at insufficient energy input and keyhole instability at excessive energy input [37,98,99]. These differences show that stable processing windows are configuration-dependent and cannot be transferred directly without considering layer thickness, hatch spacing, beam geometry, and melt-pool penetration.

Beam diameter and beam profile further influence melt-pool behavior by controlling the spatial distribution of laser energy. The same laser power can produce different local power densities depending on beam size and shape. A smaller beam diameter produces a more concentrated heat input and can increase penetration depth, while a larger or elliptical beam distributes energy over a wider region and may modify the melt-pool width-to-depth ratio. In LPBF Ti-5553 studies, beam diameters typically range between approximately 70 and 100 μm , while some single-track investigations have used elliptical beam profiles. Such differences in optical configuration affect melt-pool geometry, local heat flux, and interaction time, even when nominal energy-density values are similar [97–102].

Baseplate temperature and preheating conditions also influence the thermal boundary conditions during LPBF. Several process-mapping studies on Ti-5553 used baseplate preheating on the order of 200 $^{\circ}\text{C}$. Although preheating was not always varied as an independent parameter, it modifies the initial thermal gradient and heat extraction rate, and therefore it must be considered when comparing processing windows across studies [100,103]. More substantial thermal modification has been observed at higher substrate temperatures. Fabrication of Ti-5553 at a constant substrate temperature of approximately 500 $^{\circ}\text{C}$ produced near-full density of about 99%, while significantly altering the solidification thermal history and promoting in-situ phase transformation during deposition [104]. Under these conditions,

hardness increased from approximately 290 HV in the as-built state to about 463 HV after in-situ thermal exposure, indicating that baseplate temperature can affect not only densification but also microstructural evolution and local mechanical response.

Scanning strategy defines the path followed by the laser within each layer and the relationship between scan vectors in successive layers. It can include contour scans, hatch scans, stripe or island strategies, and rotation of the scan direction between layers. While laser power and scan speed primarily control local melt-pool formation, scanning strategy determines how heat is distributed over larger regions of the component. It therefore influences residual stress, heat accumulation, crystallographic texture, and microstructural anisotropy. In Ti-5553, where directional solidification can generate elongated prior- β grains and build-direction texture, scanning strategy may affect the continuity of epitaxial growth across melt-pool boundaries and layers [97,98,102].

Taken together, these parameters define the LPBF processing regime. Low laser power, high scan speed, excessive hatch spacing, or excessive layer thickness can produce insufficient melting and lack-of-fusion defects. Intermediate combinations can generate stable conduction-mode melting with continuous melt tracks and improved densification. Excessive laser power or very low scan speed can generate deep melt pools, vapor depression, and keyhole porosity. Therefore, the main LPBF parameters determine not only relative density, but also melt-pool geometry, pore morphology, surface condition, microstructure, and mechanical reliability [100,104,105].

For the present thesis, this parameter background is essential because the investigated Ti-5553 samples were fabricated under optimized LPBF conditions rather than through a new parameter-optimization campaign. The aim is therefore not to establish a new process window, but to interpret the resulting as-built material state. The subsequent CT, optical microscopy, SEM, XRD, tensile, nanoindentation, corrosion, and fractographic analyses must be understood in relation to how LPBF parameters influence melt-pool stability, internal defects, surface condition, phase constitution, and microstructural development.

2.3.2 Volumetric energy density and its limitations

Volumetric energy density (VED) is one of the most widely used descriptors for comparing LPBF processing conditions. It is commonly defined as the laser energy delivered per unit volume of powder and is calculated according to:

$$VED = P / (v \cdot h \cdot t)$$

where P is the laser power, v is the scan speed, h is the hatch spacing, and t is the layer thickness. The main advantage of this descriptor is that it combines four major LPBF variables into a single value, making it convenient for comparing parameter sets and identifying approximate processing regimes. For this reason, VED is frequently used in process-mapping studies to distinguish lack-of-fusion, conduction-mode, and keyhole-dominated processing windows. In LPBF Ti-5553, VED has been widely used as a first-order indicator of densification and melt-pool stability. In a broad pulsed-laser parameter sweep using a beam diameter of approximately $70\text{ }\mu\text{m}$ and a layer thickness of $45\text{ }\mu\text{m}$, Bakhshivash et al. reported lack-of-fusion-dominated morphologies at a very low VED of 20.9 J mm^{-3} and keyhole-related porosity at a very high VED of 584 J mm^{-3} . Between these two extremes, a dense processing window was identified between approximately 50 and 175 J mm^{-3} , with a maximum relative density of 99.92% obtained near 112 J mm^{-3} [98]. This result shows that VED can be useful for defining a first approximation of the acceptable energy-input range for LPBF Ti-5553.

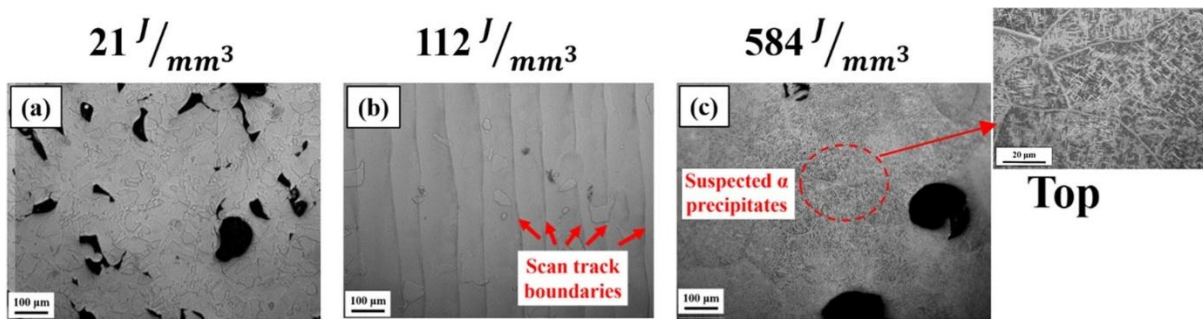


Figure 5. Optical micrographs illustrating defect evolution across different volumetric energy densities in LPBF Ti-5553: (a) irregular lack-of-fusion porosity at low VED, 21 J mm^{-3} ; (b) improved consolidation within the conduction-mode window, 112 J mm^{-3} ; and (c) defect evolution associated with excessive energy input, 584 J mm^{-3} [98].

The effect of changing VED on defect evolution is illustrated in Fig. 5. At approximately 21 J mm^{-3} , irregular lack-of-fusion defects dominate because the melt pool is not sufficiently continuous to ensure complete bonding between adjacent tracks and layers. At approximately 112 J mm^{-3} , melt-track consolidation is improved and the process lies within a more stable conduction-mode window. At approximately 584 J mm^{-3} , the excessive energy input promotes melt-pool instability and defect formation associated with keyhole-type behavior. This sequence clearly demonstrates the usefulness of VED as an initial indicator of the transition from insufficient melting to stable consolidation and finally to excessive energy input[98]. Comparable trends have been reported in independent single-track and multilayer mapping studies. Hasanabadi et al. investigated LPBF Ti-5553 over a VED span of approximately $17.4\text{--}120.4\text{ J mm}^{-3}$ and observed a similar transition sequence, from discontinuous tracks to

stable conduction-mode melting and eventually to keyhole instability [100]. Their analysis further showed that melt-pool geometry provides a more direct measure of process stability than VED alone. In particular, keyhole behavior was reported when the melt-pool depth-to-width ratio (D/W) exceeded approximately 0.8; for example, a D/W ratio of about 1.3 was observed at 325 W and 800 mm s⁻¹ [100]. This indicates that the transition from conduction-mode melting to keyhole instability is more closely related to penetration behavior and melt-pool aspect ratio than to nominal energy density alone.

The importance of melt-pool geometry is further supported by studies using thinner powder layers. At a layer thickness of 30 μm, sustained relative densities above 99% were achieved within a VED interval of approximately 64.3–91.7 J mm⁻³ under controlled power–speed combinations [100]. This shows that the same VED range cannot be universally applied across different processing configurations. The penetration depth required for reliable interlayer bonding depends on layer thickness, while the lateral overlap depends on hatch spacing and melt-pool width. Therefore, VED should be interpreted together with the geometric requirements for track overlap and layer bonding.

Although VED is useful for mapping broad processing regimes, it is not a unique descriptor of melt-pool behavior. The same VED value can be obtained from different combinations of laser power, scan speed, hatch spacing, and layer thickness. However, these combinations may generate different laser–powder interaction times, local heat fluxes, cooling rates, melt-pool geometries, and vaporization tendencies. As a result, nominally identical VED values may lead to different defect populations and microstructures[103].

This limitation has been clearly demonstrated by Ramme et al. Maintaining a constant nominal energy density while proportionally increasing laser power and scan speed reduced melt-pool penetration depth. In their single-track experiments, melt-pool widths were typically on the order of 400–500 μm, while penetration depths mostly clustered between approximately 20 and 70 μm across an energy-density range of 400–700 J mm⁻³ [101]. These observations demonstrate that melt-pool geometry is controlled by the specific power–speed pairing and spatial energy distribution, rather than by VED alone.

The limitation of VED is also linked to beam geometry and beam profile. VED does not include beam diameter, beam shape, absorptivity, or spatial energy distribution, although these parameters strongly affect the local power density and melt-pool shape. In LPBF Ti-5553 studies, beam diameters typically range from approximately 70 to 100 μm, and variations in beam geometry have been shown to modify melt-pool width, local heat flux, and penetration behavior even when nominal VED values are comparable [98–102]. Therefore, VED values

should only be compared when the underlying optical configuration and machine conditions are also considered.

Another limitation is that VED does not fully describe inter-track and interlayer overlap. Hatch spacing and layer thickness appear in the VED equation, but actual bonding depends on the relationship between hatch spacing and melt-pool width, and between layer thickness and penetration depth. A parameter set may have a suitable nominal VED, but if the hatch spacing is too large relative to melt-pool width, lack-of-fusion defects can form between adjacent tracks. Similarly, if the layer thickness is too large relative to penetration depth, interlayer bonding may be incomplete. For this reason, LPBF Ti-5553 processing windows should be interpreted as multidimensional regions controlled by power–speed coupling, hatch spacing, layer thickness, beam geometry, and melt-pool shape[104,106].

The non-unique character of VED becomes particularly important when density is used as the main quality criterion. Once lack-of-fusion defects are suppressed and a stable conduction-mode regime is established, relative density often reaches a plateau. In LPBF Ti-5553, relative density typically increases from approximately 95–96% in lack-of-fusion-dominated regimes to values exceeding 99–99.9% within conduction-mode windows[37,98,99]. Dammulla et al. reported an absolute density of $4.62 \pm 0.04 \text{ g cm}^{-3}$ compared with a nominal reference of approximately 4.65 g cm^{-3} , corresponding to residual porosity on the order of about 1% [106]. However, reaching high density does not necessarily mean that the defect population is harmless or uniform.

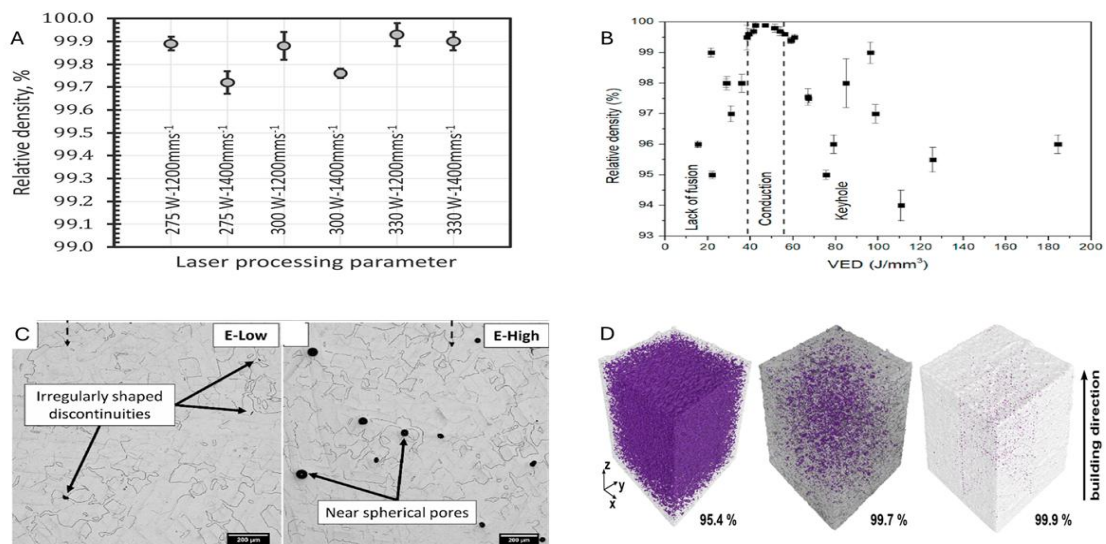


Figure 6. Process stability and defect evolution in LPBF Ti-5553: (a) relative density versus laser processing parameters showing a densification plateau[100]. (b) transition from lack-of-fusion to conduction and keyhole regimes with increasing VED [37]. (c) representative defect morphologies at low and high energy conditions[37]. (d) three-dimensional pore topology showing that comparable relative densities may exhibit distinct defect distributions[102].

This point is illustrated in Fig. 6. The relative density plot shows that, after entering the stable conduction-mode window, further increases in energy input do not necessarily lead to proportional improvements in density. Instead, densification approaches a plateau governed by sufficient melt-pool penetration and inter-track overlap. However, the transition map and defect images show that the nature of the defect population still changes across the processing space. At insufficient energy input, irregular lack-of-fusion defects dominate, whereas excessive energy input promotes near-spherical pores associated with keyhole instability.

The three-dimensional pore topology in Fig. 6d is especially important because it shows that specimens with comparable relative densities may still contain different pore populations and clustering behavior. This means that scalar density values cannot fully describe the internal defect state. A part with high relative density may still contain surface-connected defects, clustered pores, or sharply shaped lack-of-fusion defects that are critical for mechanical performance. This is particularly relevant for Ti-5553, where failure is often initiation-controlled and strongly influenced by defect location and morphology[102].

Advanced defect-characterization studies support this interpretation. Shin et al., using Archimedes measurements together with μ CT at approximately 5 μm voxel resolution and machine-learning-based classification, showed that samples with comparable relative densities, up to about 99.2%, could still contain markedly different pore populations depending on processing conditions [102]. Under insufficient energy input, irregular melt-boundary-connected pores associated with lack of fusion dominated. Under excessive penetration, near-spherical pores characteristic of keyhole instability became more common. Their statistical analysis also identified laser power as the dominant contributor to densification, with an F-value of approximately 4000, further emphasizing that melt-pool penetration and stability are more informative than VED alone [102].

Complementary studies further show that defect size distributions under high scan speed or marginal-overlap conditions commonly fall in the range of approximately 20–60 μm [107]. These defects may be small in terms of volumetric fraction but significant in terms of morphology and location. In particular, surface-connected or sharply irregular defects can act as crack-initiation sites under tensile or fatigue loading. Therefore, defect morphology and spatial distribution must be evaluated together with density and VED[108].

The limitations of VED have led to the use of additional descriptors. Melt-pool depth-to-width ratio is one of the most useful, because it directly captures the penetration behavior associated with conduction-mode and keyhole-mode melting. Energy per layer has also been suggested as a complementary descriptor because it reflects the cumulative thermal input per deposited

layer and helps explain why stable power–speed domains shift when layer thickness changes from 45 μm to 30 μm , even when VED intervals partially overlap [37]. These approaches do not replace VED entirely, but they provide a more physically grounded interpretation of LPBF stability.

Overall, VED should be considered a contextual processing descriptor rather than a transferable design rule for LPBF Ti-5553. It is useful for comparing broad energy-input regimes, but it cannot independently predict melt-pool geometry, defect morphology, surface condition, or mechanical reliability. A reliable interpretation of LPBF Ti-5553 therefore requires simultaneous consideration of laser power, scan speed, hatch spacing, layer thickness, beam geometry, melt-pool dimensions, pore topology, and surface condition. In the present thesis, VED is therefore treated as a supporting descriptor, while the experimental interpretation relies on complementary characterization methods, including CT, optical microscopy, SEM, XRD, tensile testing, nanoindentation, corrosion testing, and fractography.

2.3.3 Lack-of-fusion, keyhole porosity, and surface defects

Defect formation in LPBF Ti-5553 is closely controlled by melt-pool stability and by the balance between energy input, geometrical overlap, and heat dissipation. In general, insufficient energy input leads to incomplete melting and lack-of-fusion defects, whereas excessive energy input promotes vaporization, keyhole instability, and near-spherical pores. Between these two extremes, a stable conduction-mode regime can be achieved, where melt-track continuity, lateral overlap, and interlayer bonding are improved. These regimes are not governed by VED alone, but by the combined influence of laser power, scan speed, hatch spacing, layer thickness, beam geometry, powder-bed condition, and local heat accumulation[97,99,104,109].

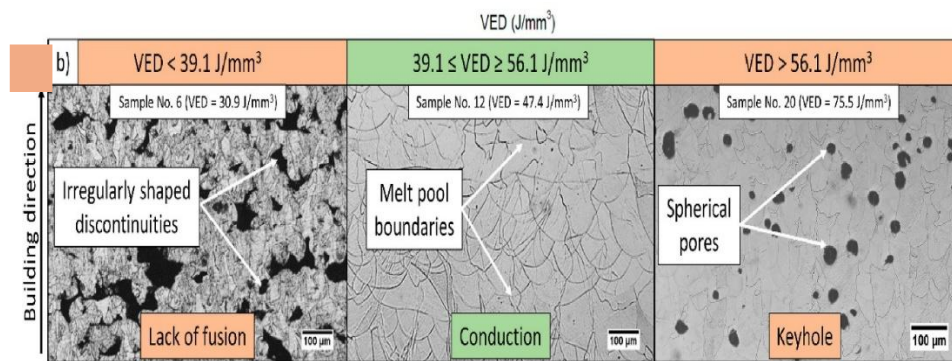


Figure 7. Optical micrographs illustrating characteristic melting modes of LPBF Ti-5553 across different VED regimes: lack-of-fusion defects with irregular discontinuities at low VED, stable conduction-mode melting with visible melt-pool boundaries at intermediate VED, and keyhole-induced spherical porosity at high VED [99].

The three principal melting regimes in LPBF Ti-5553 are summarized in Fig. 7. At low VED, irregular lack-of-fusion discontinuities dominate because the melt pool is too shallow or discontinuous to fully melt the powder layer and remelt the underlying solidified material. In the intermediate conduction-mode regime, continuous melt-pool boundaries and improved bonding between adjacent tracks and layers are observed. At high VED, the melting mode shifts toward keyhole behavior, where excessive energy input produces a deeper melt pool and spherical pores associated with vapor entrapment during rapid solidification[99].

Lack-of-fusion defects are among the most critical defect types in LPBF Ti-5553 because of their irregular and often sharp morphology. They typically form when laser power is too low, scan speed is too high, hatch spacing is too large, or layer thickness is excessive relative to the melt-pool penetration depth. Under these conditions, the melt pool cannot fully bridge neighboring tracks or layers, resulting in planar or elongated discontinuities along scan-track boundaries, layer interfaces, or melt-pool boundaries. Even when the total porosity fraction is low, such defects can be mechanically severe because their sharp edges act as stress concentrators and preferential crack-initiation sites[98,99,108,110].

Keyhole porosity develops under the opposite condition, when energy input is excessive. Strong evaporation and recoil pressure generate a deep vapor depression within the melt pool. If this vapor cavity becomes unstable and collapses during solidification, gas can be trapped inside the material, forming near-spherical pores. Compared with lack-of-fusion defects, keyhole pores are generally more rounded; however, they can still reduce mechanical reliability, particularly when they are large, clustered, or located near the surface. Keyhole-mode processing may also increase spatter formation, surface instability, and local chemical changes caused by selective vaporization[103,106,107].

The distinction between lack-of-fusion and keyhole defects is important because similar density values may correspond to very different defect morphologies. In LPBF Ti-5553, reported relative density values can exceed 99% under stable processing conditions, but the residual pore population may still differ in size, shape, and spatial distribution. μ CT studies have shown that high-density specimens may contain different pore topologies depending on the processing regime. This means that density alone cannot determine whether the remaining defects are mechanically harmless or potentially critical. In particular, irregular lack-of-fusion defects and surface-connected pores are more dangerous than isolated rounded pores because they provide easier paths for crack initiation and propagation[95–97,109].

Advanced defect analyses further show that the size range and detectability of defects depend strongly on the characterization method. Reported μ CT-based investigations of LPBF Ti-5553

have used voxel resolutions on the order of approximately 5–6.5 μm , meaning that very small pores below the resolution limit may not be detected. Complementary studies have reported defect size distributions commonly in the range of approximately 20–60 μm under high scan-speed or marginal-overlap conditions associated with insufficient energy input. Therefore, a complete evaluation of LPBF Ti-5553 requires not only density measurement, but also three-dimensional defect characterization and microscopic examination of surface-connected defects[104].

Surface condition represents another critical aspect of defect formation in LPBF Ti-5553. Even when internal densification is high, the free surface can contain partially melted powder particles, melt-track ripples, surface-connected pores, localized cracks, and protrusions. These features originate from local melt-track instability, insufficient wetting, incomplete lateral remelting, powder adhesion, and vapor-driven material ejection. Surface defects are particularly important because they are directly exposed to the loading and corrosion environment and can therefore control failure initiation more strongly than internal porosity[102].

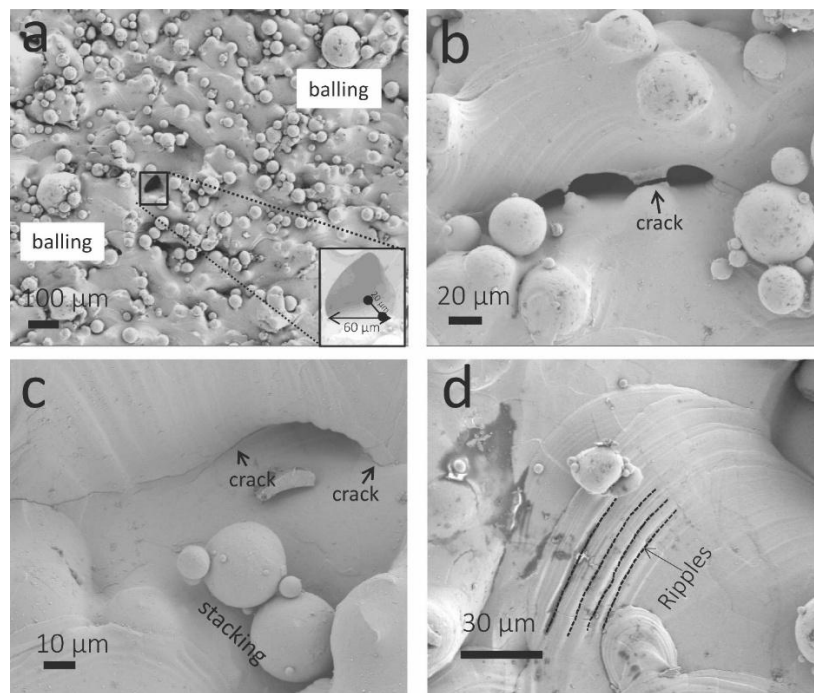


Figure 8. Representative surface defects observed in LPBF Ti-5553: (a) balling caused by unstable melt-track formation and agglomeration of molten material; (b) surface-connected pore and crack initiation along the melt-track boundary; (c) powder stacking and crack formation associated with partially melted particles; and (d) ripple patterns along the melt track indicating periodic melt-pool instability during rapid solidification [102].

Representative surface defects in LPBF Ti-5553 are shown in Fig. 8. Balling, shown in Fig. 8a, is associated with unstable melt-track formation and the agglomeration of molten material into

spherical or bead-like features. Surface-connected pores and cracks, such as those shown in Fig. 8b, can form along melt-track boundaries where bonding is incomplete and local stress concentration is high. Fig. 8c shows powder stacking and crack formation associated with partially melted particles, indicating incomplete incorporation of surrounding powder into the melt pool. Ripple patterns along the melt track, shown in Fig. 8d, reflect periodic melt-pool instability and surface-tension-driven flow during rapid solidification[102].

Quantitative surface measurements reported for LPBF Ti-5553 confirm that surface quality remains sensitive to processing conditions even when high bulk density is achieved. Under dense processing conditions, as-built surface roughness values have commonly been reported in the range of $S_a \approx 15\text{--}20\text{ }\mu\text{m}$. Within a conduction-mode window corresponding to approximately $64.3\text{--}91.7\text{ J mm}^{-3}$, minimum S_a values of about $16.7\text{ }\mu\text{m}$ were reported while maintaining relative density above 99%. However, further increases in scan speed degraded surface quality by promoting melt-track undulations and adhesion of partially fused powder particles, even when bulk density remained nearly unchanged. This indicates that internal densification and surface integrity can become partially decoupled in LPBF Ti-5553[99,102,106].

Other studies have reported as-built surface roughness values of approximately $R_a \approx 20\text{--}23\text{ }\mu\text{m}$, depending on build location and local processing conditions. Surface protrusions with characteristic heights on the order of approximately $80\text{--}130\text{ }\mu\text{m}$ have also been observed under certain parameter combinations. Such protrusions are significant because they can act as local stress concentrators, alter the effective surface area, and influence both mechanical and electrochemical response. Therefore, surface morphology must be considered as part of the process-quality assessment, rather than treated only as a post-processing issue[95,97,100].

Surface-connected defects are especially relevant to mechanical performance. Fracture analyses of LPBF Ti-5553 have shown that crack initiation often occurs at surface or near-surface defects, even when the bulk density exceeds 99%. This behavior reflects the initiation-controlled nature of failure in additively manufactured titanium alloys. Lack-of-fusion defects connected to the surface, partially melted particles, and sharp melt-track irregularities can all reduce the effective load-bearing area and promote localized stress concentration. As a result, tensile ductility and fatigue resistance may vary significantly even among specimens with similar density[104,107,108].

The same surface defects can also influence corrosion behavior. Titanium alloys generally rely on the formation of a stable passive oxide film for corrosion protection. However, rough LPBF

surfaces, surface-connected pores, cracks, and partially melted powder particles can locally disturb passivation, increase the real surface area, and create micro-environments where electrolyte penetration is easier. Therefore, surface roughness and defect topology may affect corrosion potential, current density, passive-film stability, and susceptibility to localized corrosion. This is directly relevant to the present thesis because corrosion testing was performed together with mechanical and microstructural characterization [70,111–113].

The combined influence of internal and surface defects explains why multiple characterization techniques are necessary for LPBF Ti-5553. CT analysis provides three-dimensional information on internal porosity and pore distribution, while optical microscopy and SEM reveal melt-pool boundaries, surface-connected defects, partially melted particles, and fracture origins. Tensile testing and nanoindentation provide the mechanical response, whereas fractography links failure initiation to defect morphology. Corrosion testing adds another functional perspective by showing how surface condition and microstructural state affect electrochemical behavior. These techniques are complementary and are required to build a reliable process–defect–property interpretation [70,108,112,114].

Overall, defect formation in LPBF Ti-5553 must be understood across multiple length scales. At the process scale, lack-of-fusion and keyhole defects reflect the stability of the melt pool and the balance between energy input and heat dissipation. At the surface scale, balling, ripples, powder adhesion, cracks, and protrusions reflect local melt-track instability and incomplete powder incorporation. At the performance scale, defect morphology, location, and connectivity control tensile behavior, fracture initiation, local mechanical response, and corrosion performance. Therefore, robust evaluation of LPBF Ti-5553 requires simultaneous consideration of relative density, pore topology, surface morphology, microstructure, mechanical behavior, corrosion response, and fracture morphology.

2.4 Microstructure evolution in as-built LPBF Ti-5553

The microstructure of as-built LPBF Ti-5553 is governed by the highly non-equilibrium thermal conditions imposed during the layer-by-layer LPBF process. Localized melting, steep thermal gradients, rapid solidification, repeated partial remelting, and intrinsic cyclic reheating collectively generate a microstructure that differs markedly from conventionally processed Ti-5553. In the as-built condition, the microstructure is not determined only by alloy chemistry, but also by melt-pool geometry, heat-flow direction, scan strategy, cooling rate, and cumulative thermal exposure[103,106,111,113].

Owing to the high concentration of β -stabilizing elements in Ti-5553, the as-built LPBF microstructure is generally dominated by the β phase. However, the microstructure is hierarchical and spatially heterogeneous, consisting of elongated prior- β grains, crystallographic texture, cellular or cellular-dendritic solidification substructures, and, under certain thermal histories, nanoscale metastable phase constituents. Therefore, the microstructural evolution of LPBF Ti-5553 must be discussed across multiple length scales, from melt-pool-scale grain morphology to sub-grain solidification structures and nanoscale phase instabilities [95,114].

The following subsections review the main microstructural features reported for as-built LPBF Ti-5553. First, the development of prior- β grain morphology and crystallographic texture is discussed. This is followed by phase constitution, with emphasis on retained β , α , and ω phases. Finally, microstructural heterogeneity associated with build direction, local thermal history, and melt-pool instability is considered.

2.4.1 Prior- β grain morphology and texture

Directional solidification during LPBF/SLM organizes the β -dominated matrix of Ti-5553 into a characteristic mesoscale grain architecture governed by melt-pool geometry and steep thermal gradients [95,108]. A clear anisotropy in grain morphology is commonly observed depending on the sectioning direction. Sections perpendicular to the build direction typically reveal more equiaxed β grains, whereas longitudinal sections parallel to the build direction show elongated columnar prior- β grains aligned approximately with the build direction [16], [95,110,114,115]. This morphology reflects directional heat extraction during solidification and the repeated epitaxial extension of β grains across successive melt pools.

Quantitative EBSD measurements reported in the literature define a relatively broad but consistent range for prior- β grain dimensions in as-built LPBF Ti-5553. Grain widths are typically in the range of approximately 10–40 μm , while grain lengths can extend from about 400–500 μm to several millimeters, giving aspect ratios from approximately 3 to values greater than 20 depending on processing conditions [114–116]. These observations indicate that the as-built grain structure is strongly anisotropic and dominated by elongated prior- β grains produced by directional solidification and epitaxial growth.

A key mechanism responsible for this morphology is repeated partial remelting of previously deposited layers. During the deposition of a new layer, the upper region of the preceding layer is remelted, allowing existing β grains at the fusion boundary to act as crystallographic templates for further growth. This enables individual prior- β grains to traverse melt-pool

boundaries, layer interfaces, and scan-track boundaries, even when scan vector rotation or stripe-based scanning strategies are used [19,110,114]. Melt-pool-resolved observations show that columnar grains initiate at the fusion boundary and grow along the local thermal gradient, while local reductions in thermal gradient near the melt-pool surface can promote columnar-to-equiaxed transitions [60,117].

This multiscale solidification structure is summarized in Fig. 9, which combines EBSD and SEM observations to show how melt-pool geometry controls grain morphology from the mesoscale grain structure to the cellular solidification scale.

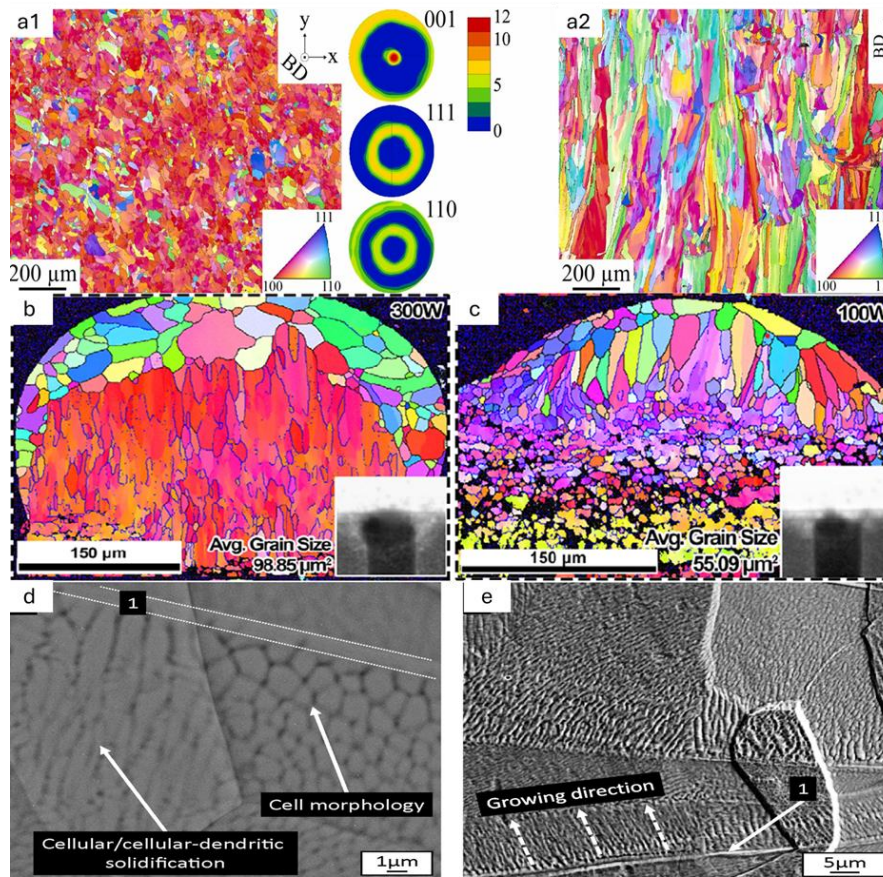


Figure 9. Hierarchical as-built microstructure of LPBF Ti-5553 across grain, melt-pool and sub-grain scales: (a1,a2) orientation-dependent β -grain morphology [110]. (b,c) power-dependent melt-pool grain morphology in microlattice struts [60]. (d,e) cellular/cellular-dendritic solidification substructures [19].

As shown in Fig. 9, the section perpendicular to the build direction contains comparatively equiaxed β grains, whereas the longitudinal section contains elongated columnar prior- β grains aligned with the build direction. This comparison directly illustrates the anisotropic grain morphology produced by directional solidification. The melt-pool cross-sections further demonstrate epitaxial grain growth from the fusion boundary, confirming that previously solidified β grains can continue growing into subsequent melt pools. At smaller length scales,

the SEM images reveal cellular to cellular–dendritic features aligned with the thermal gradient, showing that the as-built microstructure is hierarchical rather than uniform [19,60,110].

The effect of melt-pool regime on grain morphology is significant. Conduction-mode melting tends to produce smaller and shallower melt pools with relatively high thermal gradients, which can favor finer and locally more equiaxed microstructures. In contrast, near-keyhole conditions produce larger and deeper melt pools with reduced thermal gradients, promoting coarser and more columnar grain growth. These observations highlight the sensitivity of microstructure evolution to transient thermal conditions during rapid solidification, where melt-pool lifetimes on the order of approximately 4 ms have been reported [60].

At smaller length scales, as-built LPBF Ti-5553 exhibits cellular or cellular–dendritic solidification substructures within the prior- β grains. Reported characteristic cell dimensions range from sub-micron features below 1 μm to approximately 1–3 μm depending on processing parameters and characterization conditions [19,114]. These features are associated with rapid solidification under cooling rates on the order of 10^4 – 10^5 K s^{-1} and reflect the local relationship between thermal gradient and solidification rate [114,118]. Therefore, the as-built microstructure should be understood as a hierarchical structure, consisting of elongated prior- β grains at the mesoscale and cellular solidification features at the micro- to sub-micrometer scale.

This behavior is consistent with classical solidification theory, where the stability of columnar growth and the tendency for columnar-to-equiaxed transition are governed by the relationship between thermal gradient and solidification rate. In LPBF Ti-5553, the combination of steep thermal gradients, rapid cooling, and repeated remelting creates a narrow processing window in which relatively small variations in melt-pool geometry, scan strategy, or local energy input can produce measurable changes in grain morphology [19,108]. The high fraction of low-angle grain boundaries, reported to be approximately 70% in as-built material, further supports the role of repeated partial remelting and sub-grain inheritance during layer-by-layer deposition [34].

Available studies also suggest that the prior- β grain morphology is controlled more strongly by solidification conditions than by modest differences in feedstock particle size. Across LPBF studies using predominantly spherical gas- or plasma-atomized powders with overlapping particle size distributions of approximately 15–63 μm , elongated build-parallel prior- β grains are consistently observed [19,37,97,105,106]. This indicates that, within commonly used powder-size ranges, powder size alone does not establish a clear correlation with prior- β grain

morphology. Instead, melt-pool geometry, thermal gradient, solidification rate, and scan strategy are the dominant factors controlling mesoscale grain selection.

The elongated and directionally continuous prior- β grain structure frequently produces a $\langle 001 \rangle \beta$ fiber texture aligned with the build direction. This is consistent with preferential growth along $\langle 001 \rangle$ directions in the bcc β phase during sustained epitaxial solidification under strong thermal gradients [19,104,110]. Such texture is important because it can contribute to anisotropic deformation and mechanical response in as-built LPBF Ti-5553, especially when combined with columnar grain morphology and build-direction-dependent defect distributions. The variability of β texture in as-built LPBF Ti-5553 is illustrated in Fig. 10. This figure is important because it shows that grain morphology and crystallographic texture, although related, should not be treated as identical microstructural descriptors.

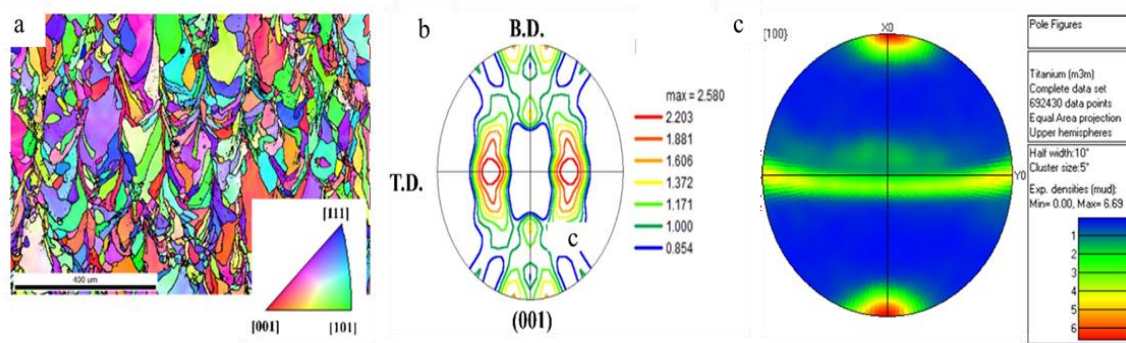


Figure 10. Representative β -texture variability in as-built LPBF Ti-5553: (a) EBSD IPF map showing elongated prior- β grains with weak crystallographic alignment along the build direction[19].(b) corresponding (001) pole figure showing weak $\langle 001 \rangle \beta$ fiber texture; and (c) representative pole figure showing a stronger $\langle 001 \rangle \beta$ fiber aligned with the build direction[95].

As seen in Fig. 10, elongated prior- β grains may coexist with only weak $\langle 001 \rangle \beta$ alignment. The EBSD IPF map shows columnar prior- β grains extending along the build direction, but the corresponding pole figure indicates a relatively weak fiber texture, with reported IPF values of approximately 1.8–2.2 and a maximum intensity of about 2.5 MUD [95]. This distinction is important because it demonstrates that columnar grain morphology does not necessarily imply strong crystallographic alignment.

The coexistence of elongated prior- β grains with weak or near-random texture has also been reported by Karimi et al. and Casias et al. [95,116]. In contrast, stronger $\langle 001 \rangle \beta$ fiber textures with intensities approaching approximately 6 MUD have been observed under comparable columnar grain morphologies [19]. The comparison in Fig. 10 therefore highlights that texture intensity is governed not only by the presence of elongated grains, but also by melt-pool regime, cumulative thermal history, and local heat-flow conditions.

The variability in texture can be rationalized by considering melt-pool geometry and intrinsic thermal exposure. Enlargement of the melt pool, particularly during transitions from stable conduction-mode melting toward near-keyhole conditions, can promote stronger crystallographic alignment, whereas stable conduction-mode conditions with limited intrinsic thermal exposure may produce weaker textures [108,119,120]. Additional heterogeneity may arise from build height and geometry-dependent heat flow, with sharper $\langle 001 \rangle \beta$ alignment reported in regions subjected to greater cumulative thermal exposure [108,121].

Overall, the as-built prior- β grain architecture of LPBF Ti-5553 is governed by directional solidification under steep thermal gradients. It is characterized by elongated prior- β grains, cellular or cellular-dendritic substructures, and variable $\langle 001 \rangle \beta$ texture. Grain widths of approximately 10–40 μm , lengths from several hundred micrometers to several millimeters, aspect ratios from approximately 3 to greater than 20, and sub-grain features below 1 μm to approximately 1–3 μm have been reported depending on processing regime and characterization method [19,104,114,116]. This microstructural framework provides the basis for understanding the anisotropic mechanical behavior and spatial variability often observed in as-built LPBF Ti-5553.

2.4.2 Phase constitution: retained β , α , and ω

The phase constitution of as-built LPBF Ti-5553 is strongly controlled by the metastable β nature of the alloy and the rapid thermal cycles imposed during fabrication. Because Ti-5553 contains a high concentration of β -stabilizing elements, such as Mo, V, and Cr, the β phase is generally retained during rapid solidification. As a result, most studies report a predominantly β microstructure in the as-built condition, with quantified β fractions typically ranging from approximately 96 to 100 wt.% and only minor secondary phase contents where [111–113].

The predominance of retained β is consistent with the alloy design of Ti-5553. Unlike $\alpha+\beta$ titanium alloys, where rapid cooling may promote martensitic transformation, the high β -stabilizer content in Ti-5553 suppresses martensite formation and favors retention of a metastable β matrix [109,114]. This retained β phase forms the baseline microstructure of as-built LPBF Ti-5553. However, the β matrix is not necessarily fully stable. Depending on local thermal history, intrinsic reheating, and cumulative thermal exposure during layer-by-layer fabrication, nanoscale ω phase or limited α precipitation may occur.

Diffraction- and EBSD-based analyses generally confirm the β -dominated nature of the as-built condition, but they may not always capture nanoscale phase instabilities. In selected cases, weak reflections consistent with hexagonal ω have been observed in XRD, with quantitative

analyses reporting ω fractions up to approximately 4 wt.% under specific processing conditions [112]. However, ω identification can be difficult using XRD or SEM alone because of peak overlap, low volume fraction, and nanoscale precipitate size. Therefore, transmission electron microscopy and selected area diffraction are often required for more reliable confirmation of ω phase formation.

Evidence of nanoscale phase evolution in LPBF Ti-5553 is shown in Fig. 11. This figure is particularly important because it demonstrates that the as-built β matrix can undergo local metastable decomposition, even when the overall microstructure appears β -dominated in conventional microscopy or diffraction analysis.

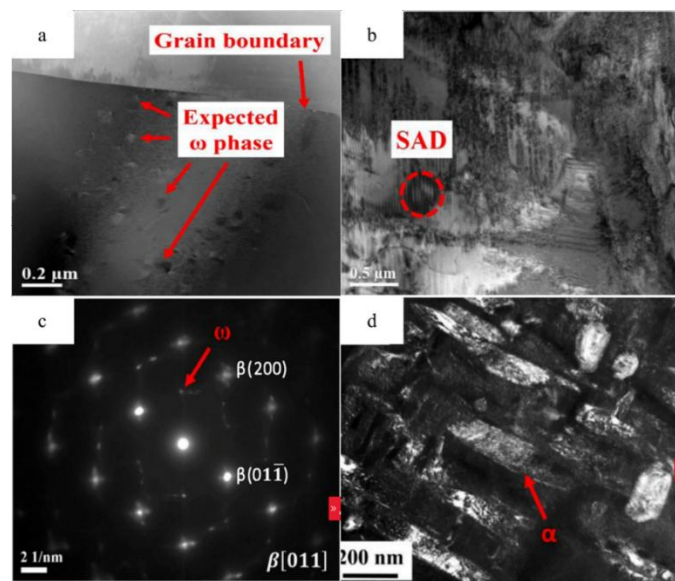


Figure 11. TEM analysis showing the phase transformation pathway in LPBF Ti-5553: (a) nanoscale ω precipitates within the β matrix; (b) region selected for diffraction; (c) SAD pattern confirming ω phase formation; and (d) α needle precipitation within the β matrix. The observations indicate a two-step transformation sequence $\beta \rightarrow \beta + \omega \rightarrow \beta + \alpha$, where ω may act as a precursor for α nucleation[98].

As shown in Fig. 11a, nanoscale ω precipitates can form within the retained β matrix, particularly in regions affected by local thermal exposure. The selected area diffraction pattern in Fig. 11c confirms the presence of ω through characteristic diffraction features that are not easily resolved by lower-resolution techniques. Fig. 11d shows the subsequent formation of fine α needles within the β matrix, indicating that the metastable β phase can evolve toward a more stable $\beta + \alpha$ configuration. Reported α needles are typically in the range of approximately 20–150 nm, while ω precipitates may be smaller than 25 nm [98].

The observations in Fig. 11 support a two-step transformation pathway in LPBF Ti-5553, described as $\beta \rightarrow \beta + \omega \rightarrow \beta + \alpha$. In this sequence, ω may act as an intermediate metastable phase and provide favorable sites for subsequent α nucleation. This pathway is important

because even small fractions of nanoscale ω or α can influence mechanical response by modifying local slip behavior, work hardening, and deformation localization. Therefore, the absence of strong α or ω peaks in conventional XRD does not necessarily imply a fully stable single-phase β microstructure[98].

The sensitivity of nanoscale phase detection is further highlighted by observations after localized surface laser polishing or remelting, where TEM diffraction showed only β -phase reflections and no detectable secondary-phase spots [100,111]. This does not necessarily contradict reports of ω or α in other as-built conditions; rather, it confirms that nanoscale phase constitution in LPBF Ti-5553 is highly dependent on sampling location, local thermal history, and characterization volume. Therefore, the absence of detectable ω or α in a given TEM or XRD analysis should be interpreted carefully, particularly when the expected precipitates are nanoscale and present at low volume fractions.

Additional thermal-analysis evidence supports the interpretation that the as-built β matrix can contain metastable transformation precursors even when conventional diffraction suggests a nearly single-phase β structure. Differential scanning calorimetry has identified ω -associated thermal events between approximately 220 and 320 °C, transformation signatures related to ω -assisted α nucleation between about 350 and 480 °C, and progressive β decomposition toward α formation between approximately 480 and 650 °C [112]. These observations indicate that the as-built LPBF Ti-5553 condition should not be treated as a fully relaxed β phase, but rather as a metastable starting state containing stored defect energy and possible nanoscale transformation precursors [110,112,116].

The sensitivity of phase constitution to thermal history has also been demonstrated by studies investigating different energy inputs and build locations. Denkena et al. reported no detectable α formation by XRD/EBSD at an energy density of 32.7 J mm⁻³, where the microstructure contained approximately 96 wt.% β and about 4 wt.% ω [120]. At a slightly higher energy density of 37.0 J mm⁻³, α peaks and diffraction spots became evident, particularly toward higher build heights where cumulative thermal exposure reduced the effective cooling rate [81,120]. This indicates that relatively small changes in energy input and heat accumulation can shift the as-built β matrix from a predominantly β/ω state toward incipient α precipitation. EBSD-based phase mapping has also quantified very small α fractions in as-built LPBF Ti-5553. For example, Casias et al. reported a phase constitution of approximately 99.4% β and 0.6% α in as-LPBF material, which is consistent with the broader range of β fractions reported in the literature [112,113,116]. Such low α fractions indicate that α precipitation in the as-built condition is generally limited, but not always absent. The detectability of α depends on

processing conditions, local thermal history, build height, and the characterization method used.

The phase constitution of as-built LPBF Ti-5553 should therefore be interpreted as condition-dependent rather than fixed. A β -dominated microstructure is the baseline state, but nanoscale ω formation or limited α precipitation may occur under specific thermal histories. The local thermal cycles generated during LPBF, including repeated reheating from subsequent layers, can act as an intrinsic thermal treatment. This is especially important in Ti-5553 because the alloy is designed to respond strongly to thermal exposure through precipitation reactions within the β matrix.

The presence or absence of α and ω also has direct implications for mechanical behavior. A retained β matrix may provide lower modulus and improved ductility, while fine α precipitation can increase strength. However, excessive or heterogeneous precipitation may promote localized deformation or reduce ductility. Similarly, nanoscale ω can influence deformation mechanisms and may contribute to embrittlement or reduced work-hardening capacity depending on its distribution and volume fraction. For this reason, phase constitution must be considered together with grain morphology, defect population, and local thermal history when interpreting the properties of as-built LPBF Ti-5553[118,119,121].

In summary, as-built LPBF Ti-5553 is generally characterized by a retained β -dominated microstructure, with reported β fractions of approximately 96–100 wt.% and very limited α formation in most conditions [111–113]. Nevertheless, nanoscale ω precipitates and fine α needles may form locally depending on energy input, build height, and intrinsic thermal cycling [98,112,120]. This phase-stability window is one of the key features distinguishing LPBF Ti-5553 from more conventional titanium alloy systems and provides an essential basis for understanding its mechanical and corrosion behavior.

2.4.3 Microstructural heterogeneity and thermal history

The solidification-controlled grain framework and thermally driven phase instabilities described in the previous sections coexist with an inherent degree of microstructural heterogeneity in as-built LPBF Ti-5553. Even when nominally optimized processing conditions are used, the as-built microstructure is not perfectly uniform at the component scale. Instead, heterogeneity arises from spatial variations in melt-pool stability, energy input, local heat dissipation, intrinsic thermal cycling, and part geometry [97,106,108,120,121]. These factors can locally modify defect population, phase constitution, grain morphology, texture, residual strain, and hardness.

At the melt-pool scale, heterogeneity is closely related to local thermal gradients and solidification conditions. The geometry of each melt pool defines the local heat-flow direction, solidification rate, and remelting depth, while melt-pool boundaries can also act as preferential locations for defect formation or microstructural discontinuities. This relationship is illustrated in Fig. 12, where the melt-pool cross-section is defined by its characteristic width and depth, and partially melted powder particles are observed near the melt-pool boundary.

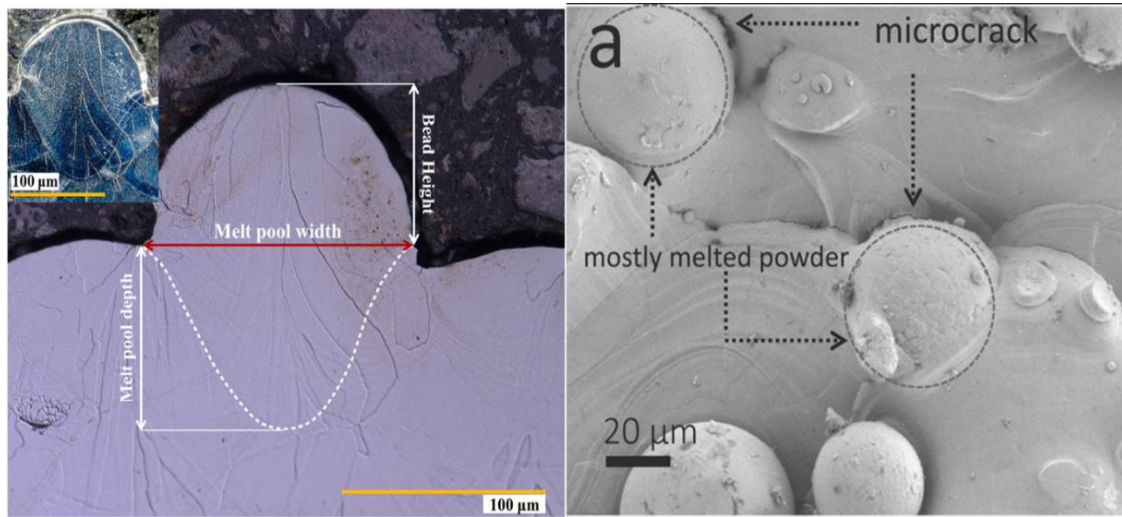


Figure 12. Representative melt-pool geometry and defect nucleation features in as-built LPBF Ti-5553: (a) cross-sectional melt-pool profile showing characteristic width and depth associated with directional solidification [99](b) partially melted powder particles at the melt-pool boundary serving as preferential sites for microcrack initiation under unstable thermal conditions[100].

As shown in Fig. 12a, the melt-pool profile provides a direct indication of the local solidification environment. Variations in melt-pool depth and width change the thermal gradient and the degree of remelting of the previous layer, thereby influencing grain continuity and defect formation. Fig. 12b shows partially melted powder particles at the melt-pool boundary, where incomplete incorporation of powder and local stress concentration can promote microcrack initiation. This confirms that heterogeneity in LPBF Ti-5553 is not only a bulk-scale phenomenon, but also originates locally at melt-pool boundaries and track interfaces [99,100].

Quantitative density measurements reported across the literature define a broad but well-characterized envelope for as-built LPBF Ti-5553. Relative densities as low as approximately 97.8% have been reported under low energy input conditions, where insufficient melt-pool overlap promotes lack-of-fusion porosity. Denkena et al. correlated this low relative density of approximately $97.8 \pm 0.3\%$ with a VED of 20.8 J mm^{-3} , identifying planar and irregular lack-of-fusion defects aligned with melt-pool boundaries as the dominant porosity type [120]. When VED increased to intermediate levels of 29.2 and 32.7 J mm^{-3} , porosity was

reduced and relative density improved to approximately 98.7–99.4%, consistent with improved melt-pool continuity and overlap [120]. Under optimized conduction-mode processing windows, multiple studies have reported high densification, with relative densities exceeding 99% and approaching near-fully dense conditions [115,120,122].

However, defect populations do not decrease monotonically with energy input. At higher energy input, Denkena et al. reported changes in porosity characteristics and increased microstructural variability at 37.0 J mm^{-3} , coincident with deeper melt-pool penetration and the onset of α precipitation under enhanced thermal accumulation [120]. This shows that increasing energy input can reduce lack-of-fusion porosity, but it may also promote keyhole-related instability, deeper penetration, and phase evolution when thermal accumulation becomes significant [99,115,120].

The relationship between melting regime, melt-pool geometry, and microstructural heterogeneity is summarized in Fig. 13. This figure is used here not only to describe defect formation, but also to show how different melting modes create different local thermal histories and therefore different microstructural states.

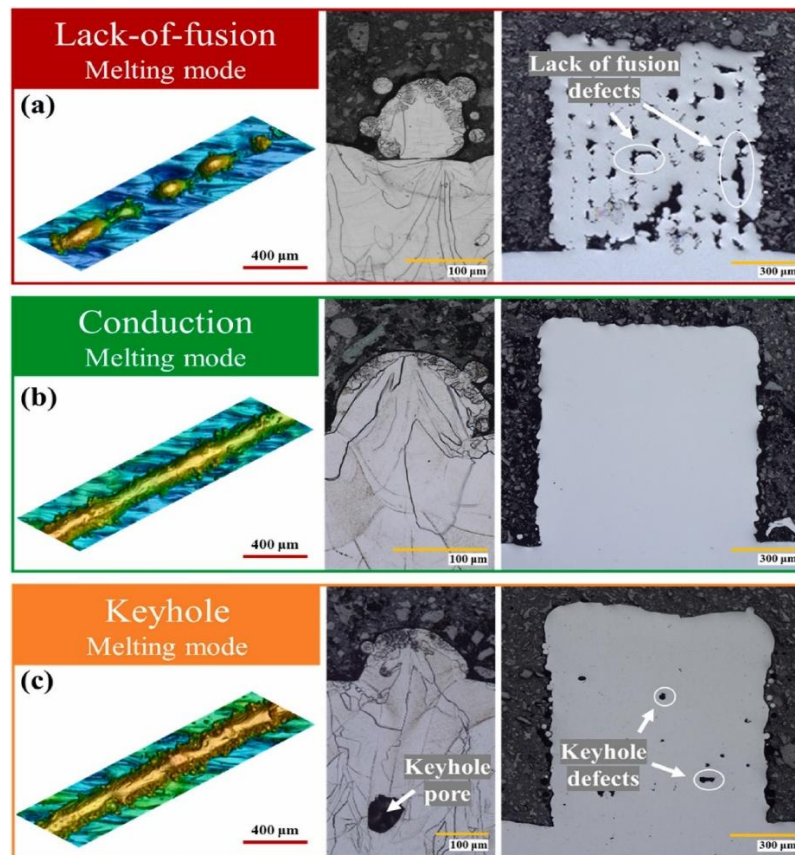


Figure 13. Track surface morphology, melt-pool geometry, and multilayer cross-sectional images for three melting modes in LPBF Ti-5553: (a) lack-of-fusion mode, produced at $P = 125 \text{ W}$ and $v = 1600 \text{ mm s}^{-1}$; (b) conduction mode, produced at $P = 325 \text{ W}$ and $v = 1200 \text{ mm s}^{-1}$; and (c) keyhole mode, produced at $P = 325 \text{ W}$ and $v = 600 \text{ mm s}^{-1}$ [99].

As shown in Fig. 13a, the lack-of-fusion regime is characterized by unstable or discontinuous melt tracks, shallow melt pools, and irregular planar discontinuities aligned with melt-pool boundaries. These features are associated with insufficient melting and poor track continuity, leading to reduced densification and spatially heterogeneous defect distributions [99]. In contrast, the conduction-mode condition shown in Fig. 13b exhibits more continuous melt tracks, bowl-shaped melt pools, and improved interlayer bonding. In this regime, the melt-pool width and depth are more balanced, promoting more uniform solidification and near-full densification [99]. At higher energy input, the keyhole regime shown in Fig. 13c produces deeper and narrower melt pools, vapor-induced cavities, and near-spherical pores located near the bottom of the melt pool. This condition is associated with greater melt-pool instability, deeper penetration, enhanced thermal accumulation, and increased susceptibility to local phase evolution [99,115,120].

Spatial heterogeneity along the build direction is another important feature of as-built LPBF Ti-5553. It originates from cumulative thermal exposure during layer-by-layer fabrication, where lower regions of the component are repeatedly reheated by subsequent layers while upper regions experience a different thermal history. Jingqi Zhang et al. reported location-dependent evolution within a single build, including non-uniform hardness distributions across the xz-plane and corresponding differences in XRD spectra between specimens extracted from distinct build locations [121]. These observations indicate that local thermal history can affect both mechanical response and phase constitution within a single LPBF component.

Denkena et al. reported a similar build-direction dependence at elevated energy density. No significant hardness variation was observed at 20.8, 29.2, and 32.7 J mm⁻³; however, at 37.0 J mm⁻³, hardness increased with build height, consistent with enhanced thermal accumulation and a greater tendency for α precipitation. Importantly, these gradients develop without necessarily changing the overall prior- β grain architecture. Instead, they represent thermal-history-dependent modifications superimposed on the solidification-imposed grain framework [115,116,120,121].

Part geometry can further amplify microstructural heterogeneity by altering local heat flow. Andrews et al. demonstrated that variations in strut orientation and overhang angle modify local thermal gradients and melt-pool behavior, producing spatially heterogeneous microstructures, texture responses, and residual strain distributions within a single build [108]. High-resolution EBSD strain mapping revealed localized strain concentrations near melt-pool/strut interfaces, which varied systematically with geometry-induced thermal asymmetry. This directly links melt-pool stability and local cooling conditions to spatial variability in

mechanical response [108]. Yasin et al. also emphasized that the as-built condition cannot always be represented by bulk averages, since build-height-relevant microstructures and part-location effects across the build plate may differ even under identical nominal processing parameters [97].

At the microscale, grain boundaries also contribute to heterogeneity in as-built LPBF Ti-5553. The elongated prior- β grain architecture creates extensive grain-boundary networks aligned with the build direction [114,115]. When α is present in the as-built condition, it has been reported at low volume fractions, on the order of approximately 0.6% in EBSD-based measurements and may preferentially localize at prior- β grain boundaries in some cases [95,116]. Such interfaces may coincide with regions affected by melt-pool boundary effects, thermal accumulation, or local phase variation, contributing to spatial variability in the microstructural state [97,108,120].

Integrating these observations, the as-built microstructure of LPBF Ti-5553 can be described as a process-dependent and spatially heterogeneous framework. At low energy input, such as 20.8 J mm^{-3} , insufficient melt-pool overlap produces a β -dominated grain framework containing lack-of-fusion porosity and reduced relative density of approximately $97.8 \pm 0.3\%$ [120]. Within an intermediate conduction-mode processing window, for example $29.2\text{--}32.7 \text{ J mm}^{-3}$, porosity is reduced and relative density improves to approximately $98.7\text{--}99.4\%$, while the elongated columnar prior- β grain structure is retained [120]. At higher energy input, such as 37.0 J mm^{-3} , or under strong intrinsic heat accumulation, deeper melt-pool penetration, increased microstructural variability, and the onset of α precipitation have been documented [81,108,121].

Powder size distribution and machine platform may influence melt-pool stability and cumulative thermal exposure; however, across the reviewed studies, comparable β -dominated grain architectures are reported when similar processing regimes are achieved [95,116]. Therefore, the dominant source of as-built microstructural heterogeneity is not simply the feedstock or machine type, but the local thermal environment imposed by melt-pool geometry, energy input, scan strategy, part geometry, and build position.

Taken together, the as-built microstructure of LPBF Ti-5553 is inherently heterogeneous. Reported relative densities span approximately 97.8% to greater than 99.4% across processing windows, defect populations transition from lack-of-fusion-dominated porosity at low energy input to instability-related pore susceptibility at high energy input, and phase stability varies locally with cumulative thermal exposure [95,105,110,114,116,118,122,123]. This intrinsic

heterogeneity provides the process–structure basis for the anisotropic and defect-sensitive mechanical behavior discussed in the following section.

2.5 Mechanical properties of LPBF Ti-5553

The mechanical properties of as-built LPBF Ti-5553 are controlled by the combined influence of defect population, surface condition, crystallographic texture, phase constitution, and local thermal history. Although Ti-5553 is intrinsically a high-strength metastable β titanium alloy, the mechanical response of the LPBF-fabricated material cannot be interpreted only from alloy chemistry or nominal density. Instead, the as-built condition contains processing-induced heterogeneity, including residual porosity, surface-connected defects, elongated prior- β grains, retained metastable β phase, possible ω or α formation, and local variations in thermal exposure. These features collectively determine tensile behavior, hardness, deformation localization, fracture initiation, and fatigue performance.

Across the available literature, tensile strength and hardness of as-built LPBF Ti-5553 are relatively bounded compared with ductility and fatigue response. Ultimate tensile strength generally falls within a moderate-to-high strength range, while elongation and failure behavior show much larger scatter. This indicates that load-bearing capacity and damage tolerance are partially decoupled in the as-built state. The following subsections first discuss tensile behavior and anisotropy, then hardness and local mechanical response, and finally fracture behavior controlled by defects, surface condition, and microstructural anisotropy.

2.5.1 Tensile behavior and anisotropy

In the as-built condition, LPBF Ti-5553 exhibits a tensile response that is relatively constrained in strength but highly variable in ductility. Across the available literature, the ultimate tensile strength (UTS) generally falls within the range of approximately 780–950 MPa, with some reports approaching about 980 MPa. In contrast, elongation to failure shows a much broader scatter, commonly ranging from approximately 2% to 15%, with isolated cases reaching values close to 30% under specific processing conditions [19,37,105,113,121,124]. This difference between a relatively bounded strength range and a much more scattered ductility range indicates that tensile failure in as-built LPBF Ti-5553 is controlled not only by the intrinsic strength of the β -dominated matrix, but also by defect population, surface condition, build orientation, local thermal history, and metastable phase evolution.

The tensile response of LPBF Ti-5553 is fundamentally linked to its as-built microstructure. As discussed in Section 2.4, the material usually contains a predominantly retained β matrix,

elongated columnar prior- β grains, and variable β texture generated by directional solidification [19,110,121]. These features establish the baseline mechanical response of the alloy. However, they do not uniquely determine ductility. Instead, tensile elongation is highly sensitive to the distribution of lack-of-fusion defects, gas pores, surface-connected irregularities, local thermal exposure, and possible nanoscale phase instabilities such as ω formation [29], [35], [60]. Where ω phase has been reported, it has been suggested to influence deformation behavior by reducing work-hardening capacity or promoting localized embrittlement, depending on its distribution and formation pathway [110,121].

Early feasibility studies demonstrated that useful tensile properties can be achieved in as-built LPBF Ti-5553 when densification is well controlled. Schwab et al. reported UTS values of approximately 800 MPa with elongation of about 14% in specimens with high relative densities of approximately 99–99.9% [19]. These results confirmed that LPBF can produce near-dense Ti-5553 with moderate ductility in the as-built state. However, later studies showed that similarly dense specimens can still display very different elongation values. For example, some processing conditions produced elongations approaching 30%, whereas other conditions led to brittle-like behavior with elongation as low as approximately 2–3% despite high tensile strength [113,124]. This demonstrates that high relative density is necessary but not sufficient for ensuring reliable tensile ductility.

A high-strength but low-ductility regime has been specifically reported in as-built LPBF Ti-5553, where tensile strength approached approximately 950–980 MPa while elongation decreased to only about 2–3% [113]. This behavior suggests that increased strength can be accompanied by reduced deformation stability when local defects, metastable phase evolution, or thermal-history-dependent microstructural heterogeneity promote early strain localization. Therefore, a simple strength-based evaluation can be misleading: a higher UTS does not necessarily indicate better structural performance if ductility and damage tolerance are compromised.

The partial decoupling between strength and ductility is further supported by the contrast between quasi-static tensile loading and dynamic compression. Under dynamic compression, as-built LPBF Ti-5553 has been reported to exhibit simultaneous increases in strength and plastic strain with increasing strain rate, together with stable plastic flow and no premature failure [95]. This behavior indicates that the limited ductility often observed under tensile loading is not simply an intrinsic limitation of the alloy matrix. Instead, tensile failure is strongly influenced by defect-mediated and tension-driven crack initiation, whereas

compressive loading suppresses the opening of critical flaws and allows more stable plastic accommodation.

Build orientation is another important factor controlling tensile behavior. Ramachandiran et al. reported that vertically built specimens exhibited a UTS of approximately 780 MPa with about 23% elongation, whereas horizontally built specimens showed a higher UTS of approximately 840 MPa with about 21% elongation [105]. These results suggest that strength can be more sensitive to build orientation than ductility in some processing conditions. The difference is associated with the alignment of elongated prior- β grains, β texture, and defect morphology relative to the tensile loading direction. In vertically built specimens, cracks may interact more directly with columnar grain boundaries aligned along the build direction, whereas horizontally built specimens can promote different slip localization and crack-propagation paths.

The orientation dependence of tensile deformation is summarized in Fig. 14. This figure is important because it combines interrupted tensile testing with stress–strain curves from specimens extracted from different build locations, allowing the relationship between build orientation, deformation localization, and global tensile response to be examined.

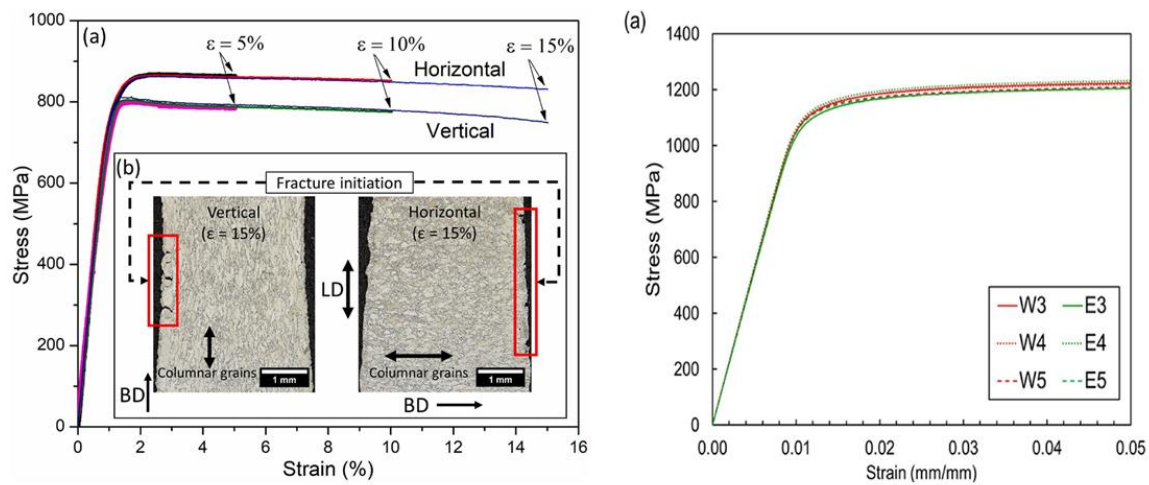


Figure 14. Tensile response of as-built LPBF Ti-5553: (a) representative interrupted tensile response showing orientation-dependent deformation behavior and fracture initiation associated with localized slip activity within columnar β -grain morphology[110]. (b) engineering stress–strain curves of machined specimens extracted from different build locations, demonstrating reproducible elastic–plastic response with limited variation in tensile strength despite location-dependent differences in defect populations[97].

As shown in Fig. 14, interrupted tensile testing reveals that vertically and horizontally built specimens develop different post-yield deformation patterns. Although the overall stress levels remain comparable, strain localization evolves differently depending on build orientation. At increasing strain levels, approximately 5–15%, fracture initiation occurs within localized deformation bands associated with columnar prior- β grains [105]. This indicates that the global

engineering stress–strain curve does not fully capture the local deformation process leading to failure. Instead, the interaction between slip localization, prior- β grain morphology, and build orientation controls where damage first develops.

Fig. 14b shows that specimens extracted from different build locations can exhibit nearly identical elastic–plastic responses and limited variation in ultimate tensile strength, even when local defect populations are not identical [97]. This observation is important because it shows that bulk tensile strength may remain relatively stable across different build locations, whereas ductility, fracture initiation, and fatigue performance may still be sensitive to local microstructural or defect variations. Therefore, tensile strength alone is not a sufficient indicator of mechanical reliability in LPBF Ti-5553.

The larger scatter in elongation compared with strength can be understood by considering the role of defect population and surface condition. In well-processed, near-dense builds, elongation commonly falls within approximately 14–20%, with isolated studies reporting values approaching 30% under specific processing conditions [19,37,103,105]. However, in surface-sensitive or defect-dominated as-built conditions, elongation may decrease to approximately 7–8%, and in more brittle-like regimes it can fall to approximately 2–3% [113,116]. This broad ductility range reflects the strong influence of lack-of-fusion defects, surface roughness, contour flaws, local thermal history, and metastable phase evolution [110,121].

Processing-dependent thermal exposure also contributes to tensile variability. Variations in volumetric energy density, heat accumulation, and cooling rate can modify β -grain morphology, consolidation quality, dislocation density, and the possibility of limited α or ω formation [99,103,120]. Spatial variations in thermal history during LPBF can also lead to local differences in deformation behavior within a single build [121]. Thus, the term “as-built” should not be interpreted as a single uniform condition. Instead, as-built LPBF Ti-5553 represents a spectrum of microstructural states governed by both solidification history and intrinsic cyclic reheating.

Systematic investigations of energy input indicate that tensile strength is generally less sensitive to moderate parameter variations than ductility, although strength increases have been reported under conditions associated with high dislocation density or metastable phase evolution [99,110,113,120]. Within the established strength window, elongation can vary from defect-limited values of approximately 1–8% to more typical values of 2–15%, with isolated cases approaching 30%. This behavior confirms a partial decoupling between load-bearing capacity and deformation capability: the β -dominated matrix sustains a relatively bounded

strength range, while ductility is controlled mainly by defects, surface condition, anisotropy, and local thermal history.

Surface condition is particularly important in tensile testing. Casias et al. showed that removal of the rough as-built surface layer can increase elongation from approximately 7–8% to about 16% and increase apparent tensile strength from approximately 808 MPa to 914 MPa [116]. Because this improvement occurs without a fundamental change in the bulk microstructure, it is mainly attributed to the suppression of surface-initiated failure. This result demonstrates that the as-built surface condition can artificially limit the measured tensile performance by introducing early crack-initiation sites.

A representative intermediate tensile response was reported by Micheletti et al., who measured a Young's modulus of 72 ± 3 GPa, yield strength of 807 ± 7 MPa, UTS of 832 ± 4 MPa, and elongation of $14 \pm 2\%$ for SLM-fabricated Ti-5553 in the as-built condition [125]. These values fall within the broader property envelope reported for LPBF Ti-5553 and are consistent with early SLM datasets [19]. They also represent a useful reference condition in which the material exhibits moderate strength, relatively low modulus, and ductility comparable to well-consolidated as-built specimens.

It is also important to note that high tensile elongation does not necessarily guarantee superior overall structural reliability. Some studies have reported combinations of relatively high elongation with poor impact resistance, suggesting that different mechanical tests probe different damage mechanisms [124]. Tensile elongation reflects the ability of the specimen to accommodate plastic deformation under monotonic loading, whereas impact and fatigue properties are more sensitive to crack-initiation resistance, defect morphology, and local stress concentration. Therefore, the evaluation of LPBF Ti-5553 should not rely on tensile properties alone.

Overall, tensile behavior in as-built LPBF Ti-5553 is characterized by a relatively constrained strength range but a broad ductility range. The available literature shows that UTS values are commonly around 780–950 MPa, while elongation can vary from very low values near 2–3% to values approaching 30% depending on processing condition, surface state, orientation, and defect population [19,37,105,113,121,124]. This variability does not primarily reflect compositional inconsistency, but rather the strong sensitivity of the as-built LPBF condition to process-induced defects, surface roughness, local thermal history, crystallographic texture, and metastable phase constitution. Consequently, tensile data must be interpreted together with microstructural characterization, defect analysis, and fracture surface observations.

2.5.2 Hardness and local mechanical response

Hardness provides a useful complementary measure for evaluating the local mechanical response of as-built LPBF Ti-5553. Unlike tensile testing, which represents the integrated response of the full gauge section, hardness and indentation-based measurements are more sensitive to local variations in phase constitution, solidification substructure, residual defects, surface condition, and thermal history. This is particularly important for LPBF materials because the layer-by-layer process can generate spatially heterogeneous microstructures even when the nominal processing parameters are kept constant. Therefore, hardness should be interpreted not only as a simple mechanical property, but also as an indirect indicator of processing-induced microstructural heterogeneity.

Across independent studies, as-built LPBF Ti-5553 generally exhibits Vickers hardness values within a relatively bounded range compared with the much wider scatter observed in tensile elongation. Reported hardness values typically fall within approximately 260–370 HV, depending on processing conditions, energy input, measurement load, and sample condition [103,106,120]. This range indicates that the local resistance of the β -dominated matrix to indentation is comparatively stable, although it can still be modified by thermal exposure, defect proximity, and local microstructural state.

Under stable processing and well-densified as-built conditions, hardness values commonly cluster within a narrower baseline interval of approximately 290–310 HV. Dammulla et al. reported a hardness value of 292 ± 2 HV for LPBF Ti-5553, which is consistent with the lower part of the as-built hardness range and reflects a predominantly β microstructure under stable processing conditions [106]. Similar values near approximately 290–292 HV have been reported in other dense as-built specimens, suggesting that this range can be considered representative of the conventional as-built β -dominated condition when no additional thermal modification is introduced [106,120].

Process-dependent variations in hardness have also been reported. Denkena et al. observed hardness values of approximately 294 HV at lower energy input, increasing to approximately 311–321 HV at higher energy densities that avoided severe lack-of-fusion defects [120]. This increase was attributed to changes in thermal history and microstructural state rather than to compositional differences. Higher energy input can improve consolidation, alter cooling conditions, increase local thermal exposure, and potentially promote limited phase evolution or changes in defect structure. Therefore, hardness can reflect the combined influence of densification, melt-pool stability, and local thermal accumulation.

Grove et al. reported a surface hardness of approximately 325 HV_{0.025} for as-SLM Ti-5553 fabricated without in-situ substrate heating [118]. This value lies above the typical 290–310 HV baseline and may be related to differences in local solidification substructure, dislocation density, or near-surface thermal history. Such results show that even within the as-built condition, hardness can vary depending on the local processing environment and measurement location. However, these variations remain relatively moderate compared with the much larger changes observed after significant thermal modification.

A distinct upper-bound case has also been reported under thermally modified conditions, where hardness values approached approximately 600 HV_{0.025} [118]. Such values should not be interpreted as representative of the conventional as-built LPBF Ti-5553 state. Instead, they correspond to a substantially altered microstructure, likely involving significant α precipitation or an effectively aged $\alpha+\beta$ condition. When such thermally modified cases are excluded, the conventional as-built hardness envelope is more reasonably described as approximately 260–325 HV, depending on local energy input, melt-pool stability, surface condition, and measurement methodology [103,106,120].

The relationship between hardness and tensile behavior is not straightforward. Although hardness provides information about local resistance to indentation, it does not directly scale with tensile ductility or fracture resistance. As discussed in Section 2.5.1, tensile strength in as-built LPBF Ti-5553 is relatively bounded, while elongation varies much more widely. This means that local matrix hardness may remain within a narrow interval even when tensile ductility changes significantly due to defects, surface flaws, or crack-initiation behavior. Therefore, hardness should not be used as a substitute for tensile testing, but rather as a complementary measurement of local microstructural response.

The sensitivity of hardness to local microstructural state makes it particularly useful for identifying spatial variations caused by LPBF thermal history. Differences in melt-pool overlap, heat accumulation, build height, and energy input can modify solidification substructure, phase stability, dislocation density, and residual defect state. These changes may produce measurable variations in local hardness even when the global tensile response appears similar. In this sense, hardness mapping or indentation-based testing can provide insight into microstructural heterogeneity that may not be fully captured by bulk mechanical testing.

The interpretation of hardness data is also affected by defects and surface condition. Dammulla et al. emphasized that rough as-built surfaces can complicate indentation measurements because surface waviness, partially melted particles, near-surface porosity, and local geometrical irregularities may influence the measured response [106]. This is especially

relevant for LPBF Ti-5553, where surface roughness and contour-related defects are common in the as-built condition. If indentation is performed too close to surface irregularities or pores, the measured value may reflect local defect geometry rather than the intrinsic hardness of the matrix.

In addition to surface condition, the proximity of indentation sites to process-induced defects can influence local hardness measurements. Hasanabadi et al. noted that indents located directly on lack-of-fusion or keyhole defects were excluded from average hardness calculations, but the detrimental influence of such defects on the surrounding material could still affect the measured response [99]. This highlights that indentation-based measurements in LPBF Ti-5553 should be interpreted with respect to local defect state, melt-pool position, and microstructural heterogeneity rather than treated as a purely intrinsic matrix property.

Nanoindentation is particularly valuable in this context because it can probe local mechanical response at much smaller length scales than conventional Vickers hardness testing. In LPBF Ti-5553, this is useful for examining differences between melt-pool interiors, melt-pool boundaries, near-surface regions, and areas affected by local thermal history. Since the as-built microstructure contains elongated prior- β grains, cellular substructures, and possible nanoscale ω or α formation, local indentation measurements may reveal variations in hardness or modulus that are averaged out in bulk tensile testing. Therefore, nanoindentation provides an important bridge between microstructural characterization and macroscopic mechanical behavior.

Nevertheless, nanoindentation data must also be interpreted carefully. The measured response can be influenced by indentation size effect, crystallographic orientation, local surface preparation, residual stress, and the presence of nearby pores or phase heterogeneities. In a β -dominated metastable alloy such as Ti-5553, local modulus and hardness may also vary with crystallographic orientation and phase constitution. Therefore, nanoindentation results should be correlated with SEM, XRD, and, where possible, local microstructural observations to avoid over-interpreting individual indentation values.

Overall, hardness and local indentation response show that as-built LPBF Ti-5553 has a comparatively constrained local mechanical response, but one that remains sensitive to processing-induced heterogeneity. Baseline hardness values around 290–310 HV are commonly associated with dense β -dominated as-built material, while higher values can arise from increased thermal exposure, local microstructural refinement, or phase evolution [106,118,120]. The broader reported range of approximately 260–370 HV reflects differences in processing conditions, measurement methodology, defect proximity, and surface condition [103,106,120]. Therefore, hardness and nanoindentation should be used together with tensile

testing, defect analysis, and microstructural characterization to develop a complete understanding of the mechanical response of LPBF Ti-5553.

2.5.3 Fracture behavior and defect-controlled failure

Fracture behavior in as-built LPBF Ti-5553 is governed by a coupled interaction between defect population, surface condition, build orientation, prior- β grain morphology, crystallographic texture, and local thermal history. Although the alloy can exhibit relatively high tensile strength in the as-built condition, failure is often controlled by crack initiation rather than by the intrinsic strength of the β -dominated matrix. Therefore, process-induced defects such as lack-of-fusion pores, keyhole pores, surface-connected flaws, partially melted particles, and contour irregularities can dominate the effective mechanical performance even when relative density is high [37,97,105,116].

This defect sensitivity explains why tensile strength, elongation, fatigue life, and fracture mode do not always scale directly with bulk density. A specimen may exhibit high relative density and a tensile strength within the expected as-built range, while still failing prematurely because of a critical surface or near-surface defect. In LPBF Ti-5553, the most damaging defects are not necessarily those that contribute the largest total porosity volume, but those that are sharp, connected to the surface, clustered, or unfavorably oriented relative to the loading direction. As a result, fracture behavior must be interpreted using a process–microstructure–defect framework rather than through scalar density or strength values alone.

The relationship between processing condition, microstructure, and fracture behavior is summarized in Fig. 15. This figure connects melting regime, defect formation, grain orientation, and fracture morphology within a single process–structure–fracture framework.

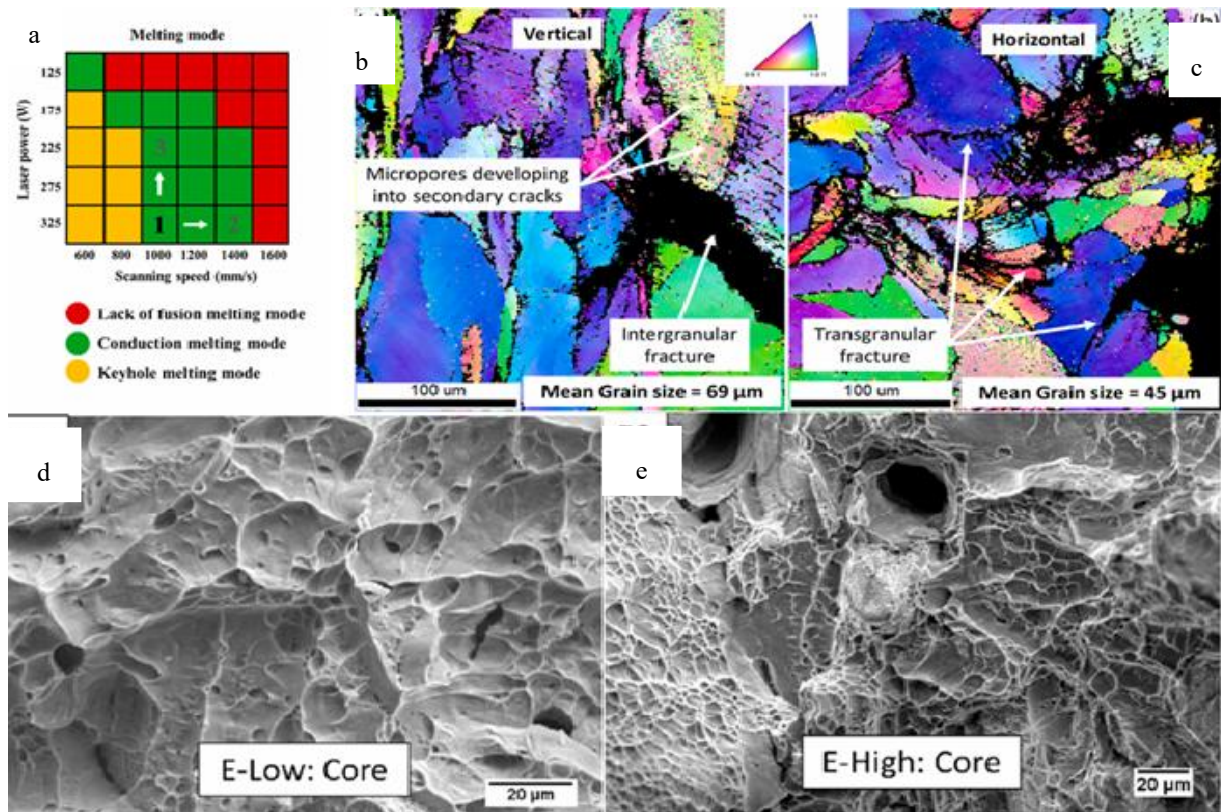


Figure 15. Integrated process–microstructure–fracture relationships in as-built LPBF Ti-5553: (a) process map showing lack-of-fusion, conduction, and keyhole regimes as a function of laser power and scanning speed[37]. (b–c) EBSD orientation maps showing orientation-dependent fracture behavior, with intergranular crack propagation along columnar β grain boundaries in vertically built specimens and more transgranular fracture in horizontally built specimens[105]. (d–e) SEM fractographs comparing low- and high-energy input conditions and highlighting differences in defect morphology, pore distribution, and microvoid coalescence[99].

As shown in Fig. 15a, processing conditions define the melting regime and therefore determine the initial defect population. Lack-of-fusion conditions generate irregular discontinuities that can serve as easy crack-initiation sites, whereas excessive energy input can promote keyhole porosity and modified pore distributions [37,99]. The EBSD maps in Fig. 15b–c show that fracture behavior is also orientation-dependent. Vertically built specimens tend to exhibit intergranular crack propagation along columnar β grain boundaries, whereas horizontally built specimens show more transgranular fracture through the grains [105]. This indicates that crack-path selection is affected by the alignment of elongated prior- β grains relative to the loading direction.

The SEM fractographs in Fig. 15d–e further demonstrate that fracture morphology changes with processing condition. Low-energy conditions are associated with irregular lack-of-fusion defects and discontinuities, while higher-energy conditions modify pore size, pore distribution, and microvoid coalescence behavior [37]. Therefore, fracture behavior in LPBF Ti-5553 cannot be interpreted only from tensile strength or bulk density. Instead, it must be linked to

the specific defect morphology, defect location, and microstructural path available for crack propagation.

At the local scale, crack initiation is closely related to plastic deformation localization. The columnar prior- β grain structure, β texture, and slip localization behavior influence how strain accumulates before final failure. In a β -dominated microstructure, local deformation may concentrate along favorably oriented slip paths or grain-boundary regions, especially when surface defects or micropores act as local stress concentrators. Multi-scale observations of deformation localization and crack initiation are shown in Fig. 16.

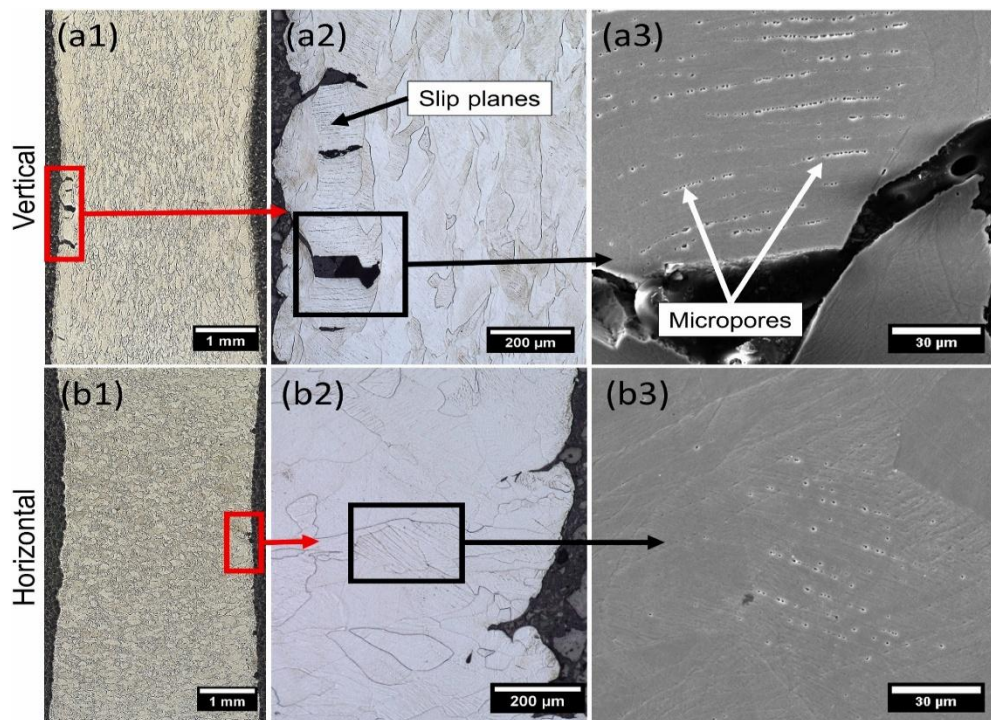


Figure 16. Multi-scale characterization of deformation localization and crack initiation mechanisms in as-built LPBF Ti-5553: vertically built specimens showing macroscopic deformation, slip-band formation within columnar prior- β grains, and aligned micropores along slip planes; and horizontally built specimens showing a similar deformation hierarchy with reduced micropore alignment compared with the vertical condition [105].

Fig. 16 shows that deformation localization develops differently in vertical and horizontal specimens. In vertically built specimens, slip bands form within columnar prior- β grains, and micropores can align along slip planes, indicating that localized plastic deformation contributes directly to damage initiation [105]. Horizontally built specimens show a similar deformation hierarchy, but with less pronounced micropore alignment. This difference suggests that build orientation affects the interaction between slip localization, prior- β grain morphology, and crack nucleation.

These observations support the interpretation that crack initiation in as-built LPBF Ti-5553 is not controlled solely by pre-existing defects. Instead, localized plastic deformation can generate, enlarge, or connect micropores near slip bands, particularly in regions where the grain structure promotes strain concentration [105]. This helps explain why specimens with comparable global tensile strength may exhibit different ductility and fracture behavior. The local deformation path, grain orientation, surface condition, and defect distribution together determine whether damage accumulates gradually or leads to premature fracture.

Surface condition is one of the most important extrinsic factors controlling fracture behavior. Direct comparisons between as-built and machined specimens show that surface roughness and contour-related defects act as preferential crack-initiation sites [116]. In the native as-built surface condition, elongation is typically limited to approximately 7–8% despite strength levels near 800 MPa. Removal of the rough surface layer has been reported to increase elongation to approximately 16% and raise tensile strength from about 808 MPa to 914 MPa, indicating that the improvement is mainly due to suppression of surface-initiated failure rather than a fundamental change in bulk microstructure [116].

This surface effect is especially important because the as-built LPBF surface contains multiple types of local stress concentrators, including adhered particles, contour irregularities, partially fused powder, melt-track boundaries, and surface-connected pores. These features can reduce the effective load-bearing area and intensify local stress during tensile or cyclic loading. Therefore, the surface state can control the apparent ductility and fracture behavior even when the internal material is relatively dense.

The role of surface morphology and near-surface crack initiation is illustrated in Fig. 17. This figure provides a direct link between powder characteristics, surface topography, local stress concentration, and crack initiation.

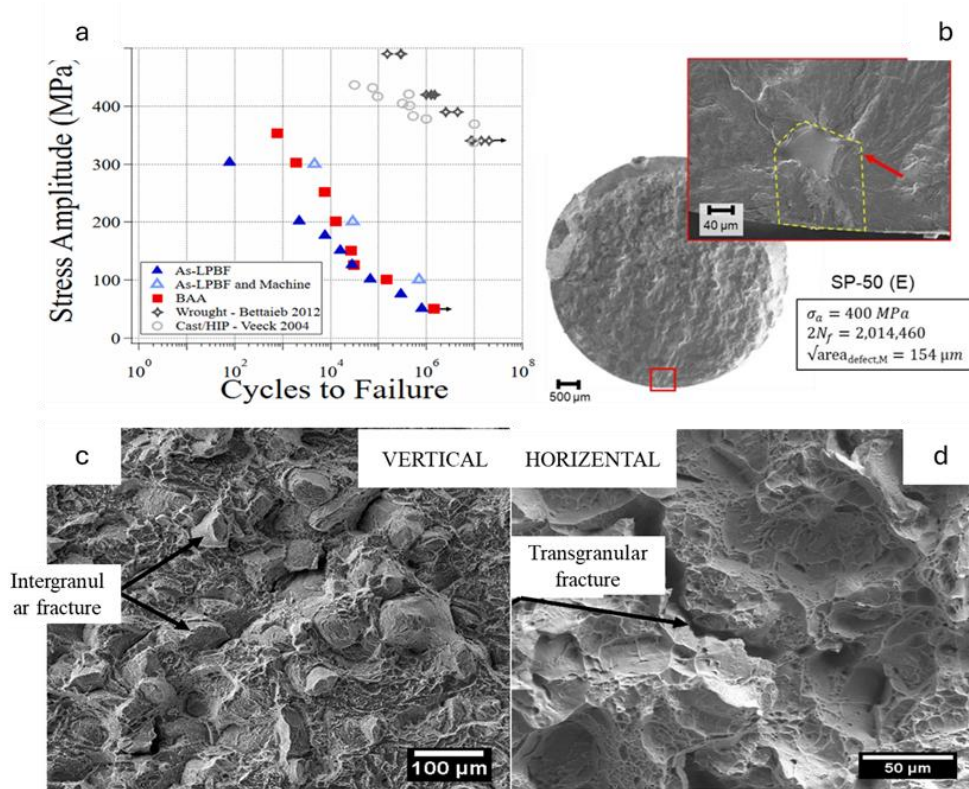


Figure 17. Processing-dependent surface morphology and surface-controlled crack initiation in as-built LPBF Ti-5553: (a) peak-to-valley surface roughness as a function of powder particle size distribution[20]. (b) surface topography maps showing contour irregularities and adhered particles acting as local stress concentrators[105]. (c) fatigue fracture surfaces showing crack initiation associated with surface or near-surface defects in the as-built condition; and (d) near-surface optical micrograph highlighting melt-pool boundaries and contour-induced heterogeneity contributing to localized strain accumulation[116].

As shown in Fig. 17a, surface roughness is influenced by powder particle size distribution, with coarser powder fractions producing higher peak-to-valley roughness despite comparable densification [20]. Reported peak-to-valley roughness values for different powder conditions fall approximately within the range of $R_z \approx 70\text{--}110 \mu\text{m}$, showing that powder morphology and surface formation can strongly influence near-surface defect severity. This means that high bulk density does not necessarily imply a mechanically benign surface state.

Fig. 17b shows surface topography features such as contour irregularities and adhered particles, which act as local stress concentrators [105]. These surface features increase the local stress intensity and can trigger surface-dominated crack initiation during tensile or cyclic loading. The fracture surfaces in Fig. 17c show crack initiation from surface or near-surface defects, confirming that the as-built surface state can dominate failure. The near-surface optical micrograph in Fig. 17d further shows melt-pool boundaries and contour-induced heterogeneity close to the surface, indicating that surface geometry and subsurface microstructure are coupled [105,116]. Therefore, surface roughness in LPBF Ti-5553 should not be treated only as a

dimensional or finishing issue; it is directly linked to mechanical reliability, fatigue resistance, and fracture initiation.

Fatigue performance is even more sensitive to surface condition and defect morphology than monotonic tensile strength. In as-built LPBF Ti-5553, fatigue life is governed largely by the largest critical flaw, especially when defects are surface-connected or located near the surface [97,116]. As-built surfaces with roughness values around $R_a \approx 20\text{--}23\text{ }\mu\text{m}$ have been associated with very low fatigue resistance, with endurance limits around 50 MPa. Machining or surface removal can improve the endurance limit to approximately 100 MPa, although this remains substantially below the endurance limit of conventionally processed Ti-5553, which has been reported around 350 MPa [116]. This large gap indicates that surface finishing improves fatigue performance but does not fully eliminate the limitations imposed by residual defects and AM-specific microstructural features. The defect-controlled nature of fatigue and fracture is shown in Fig. 18.

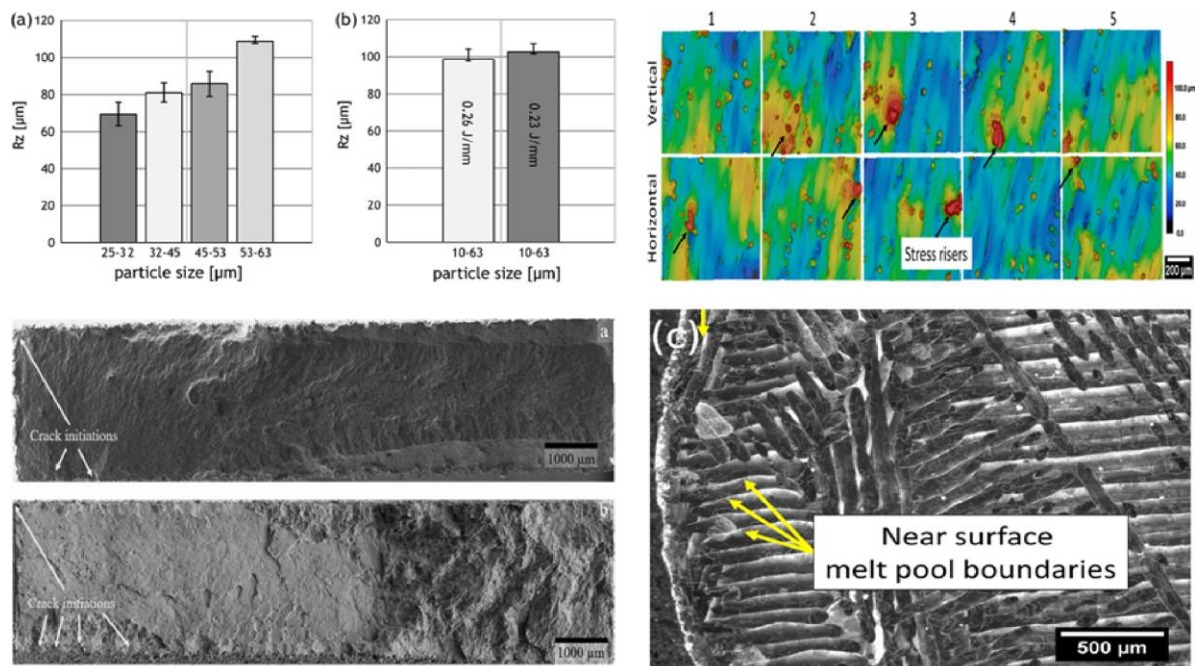


Figure 18. Defect-governed fatigue behavior and fracture characteristics of as-built LPBF Ti-5553: (a) representative S–N data showing pronounced scatter and reduced fatigue resistance compared with conventionally processed counterparts[105]. (b) fatigue fracture surface showing crack initiation from a critical near-surface defect with localized stress concentration and early-stage crack formation[97]. (c) intergranular fracture features in vertically built specimens, indicating crack propagation along prior- β grain boundaries.(d) transgranular fracture morphology in horizontally built specimens, characterized by crack propagation through grains and ductile features associated with microvoid coalescence[116].

Fig. 18a shows that the S–N response of as-built LPBF Ti-5553 exhibits pronounced scatter and reduced fatigue resistance compared with wrought or conventionally processed Ti-5553

[116]. This scatter is consistent with a defect-controlled fatigue mechanism, where fatigue life depends strongly on the size, morphology, and location of the most critical defect rather than on average porosity or nominal density alone. Fig. 18b shows crack initiation from a near-surface defect, confirming the dominant role of surface-connected flaws in fatigue failure [97,116].

The fracture morphologies in Fig. 18c–d also confirm the role of build orientation. Vertically built specimens show intergranular fracture features, indicating crack propagation along prior- β grain boundaries. Horizontally built specimens show more transgranular fracture with ductile features and microvoid coalescence [105]. This difference is consistent with the anisotropic prior- β grain structure discussed in Section 2.4 and confirms that fracture mode is influenced by the alignment of columnar grains relative to the loading direction.

Extreme value statistics further show that fatigue life in LPBF Ti-5553 scales with the largest surface-connected flaw rather than with average defect content [97,116]. This explains why specimens with similar relative density may show substantially different fatigue lives. A small number of critical defects, especially when located at or near the surface, can dominate crack initiation and overwhelm the beneficial effect of the surrounding dense matrix. Therefore, surface finishing, defect control, and pore-topology assessment are essential for improving fatigue performance.

Yasin et al. further showed that machining changes not only the fatigue life of LPBF Ti-5553, but also the statistical nature of crack initiation [97]. In the as-built surface condition, failure was associated with multiple surface-connected initiation sites distributed along the rough contour, producing pronounced life scatter. After machining, crack initiation shifted toward a single dominant volumetric defect, indicating a change in the governing extreme-value distribution despite comparable bulk mechanical properties. This confirms that surface modification changes the crack-initiation statistics without eliminating the fundamentally defect-governed nature of fatigue failure.

Surface modification and environmental exposure further support the surface-dominated interpretation of mechanical performance. Although shot peening can improve endurance strength by introducing compressive residual stresses, fatigue scatter remains strongly influenced by localized defect severity rather than by a single bulk microstructural parameter [108]. Under Hank's balanced salt solution, a clear degradation trend in fatigue resistance was not reported under the investigated conditions, suggesting that crack initiation remained strongly controlled by mechanical surface defects and roughness [108]. Nevertheless, electrochemical measurements show that surface abrasion and polishing can increase

polarization resistance and reduce anodic current density, confirming that the same surface features controlling fatigue initiation may also affect corrosion response [106].

This connection between surface condition, fatigue, and corrosion is particularly important for the present thesis because both mechanical and corrosion behavior are experimentally assessed. Surface-connected defects, rough melt-track features, and near-surface pores can act as stress concentrators during mechanical loading and as preferential sites for electrolyte access during corrosion testing. Therefore, the same processing-induced surface morphology that controls crack initiation may also influence passive-film stability and electrochemical response[123,125].

Overall, fracture and fatigue behavior in as-built LPBF Ti-5553 are controlled by a hierarchy of processing-induced and microstructural features. At the processing scale, laser parameters determine melt-pool stability and defect type. At the microstructural scale, columnar prior- β grains and texture influence crack path and deformation localization. At the surface scale, roughness, adhered particles, contour defects, and near-surface pores act as preferential initiation sites. Consequently, mechanical reliability cannot be assessed only from UTS, elongation, or relative density. A reliable evaluation requires simultaneous consideration of defect morphology, surface condition, build orientation, local microstructure, fracture surface evidence, fatigue sensitivity, and environmental response.

2.6 Heat treatment response of LPBF Ti-5553

Heat treatment plays a central role in defining the microstructure–property relationships of LPBF-fabricated Ti-5Al-5Mo-5V-3Cr, even when the material is initially studied in the as-built condition. Ti-5553 is a metastable β titanium alloy, and its mechanical response is strongly controlled by the stability of the retained β matrix and by the precipitation of secondary phases, particularly ω and α , during subsequent thermal exposure. In LPBF processing, the as-built condition is not equivalent to a conventional solution-treated state. Instead, it is produced under rapid solidification, steep thermal gradients, repeated partial remelting, and intrinsic cyclic reheating. Therefore, the response of LPBF Ti-5553 to post-processing heat treatment depends not only on the nominal heat-treatment temperature and time, but also on the metastable starting condition created during fabrication.

The heat-treatment response of LPBF Ti-5553 must be understood as a sequence of thermally activated transformations. The as-built β -dominated matrix may contain ω -related features, high dislocation density, and stored defect energy, even when conventional XRD or EBSD suggests a nearly single-phase β structure. During aging, these metastable features can

accelerate or modify α precipitation, producing a wide range of microstructures and mechanical responses. Depending on temperature, time, surface condition, and prior LPBF thermal history, heat treatment can improve ductility, increase strength through fine α precipitation, or promote embrittlement through excessive precipitation, α -case formation, or unfavorable fracture-path development. The following subsections discuss the metastable as-built condition, the aging response and α precipitation behavior, high-temperature coarsening and strength–ductility balance, surface damage and α -case formation, and post-processing routes such as HIP and cryogenic treatment.

2.6.1 Metastable as-built condition and thermal transformation pathway

The interpretation of heat-treatment effects in LPBF-fabricated Ti-5553 must begin with the as-built condition. Although the as-built microstructure is commonly described as β -dominant, the available literature shows that it does not represent a unique or fully relaxed β state. EBSD phase analysis has reported approximately 99.4% β in the as-built condition, with only about 0.6% α detected in some cases [116,123]. X-ray diffraction measurements similarly indicate a predominantly β -phase structure with no clearly resolvable secondary phases in certain as-built specimens [126]. However, the absence of strong secondary-phase peaks does not necessarily mean that the β matrix is thermodynamically relaxed or free from nanoscale transformation precursors.

At the mesoscale, LPBF Ti-5553 commonly exhibits columnar β grains aligned along the build direction, reflecting directional solidification under steep thermal gradients. Similar columnar β morphologies have been reported across several LPBF studies, indicating that the solidification-imposed grain framework is broadly reproducible when comparable processing conditions are used [56,114,116,123,127]. However, this morphological similarity should not be interpreted as evidence that the internal substructure of the β matrix is identical across studies. The same β -dominated grain architecture can contain different levels of stored defect energy, dislocation density, and metastable nanoscale features depending on processing history and intrinsic thermal cycling.

The multiscale character of the as-built and heat-treated microstructures is summarized in Fig. 19. This figure is useful because it connects the apparently β -dominant as-built state with the transformation products that develop during post-processing. The as-built EBSD phase map shows a near-single-phase β microstructure, while the heat-treated phase map shows the development of α within the β matrix. The accompanying orientation and TEM images further

demonstrate that phase evolution in LPBF Ti-5553 occurs across multiple length scales, from prior- β grain morphology to nanoscale ω -related features and α precipitation.

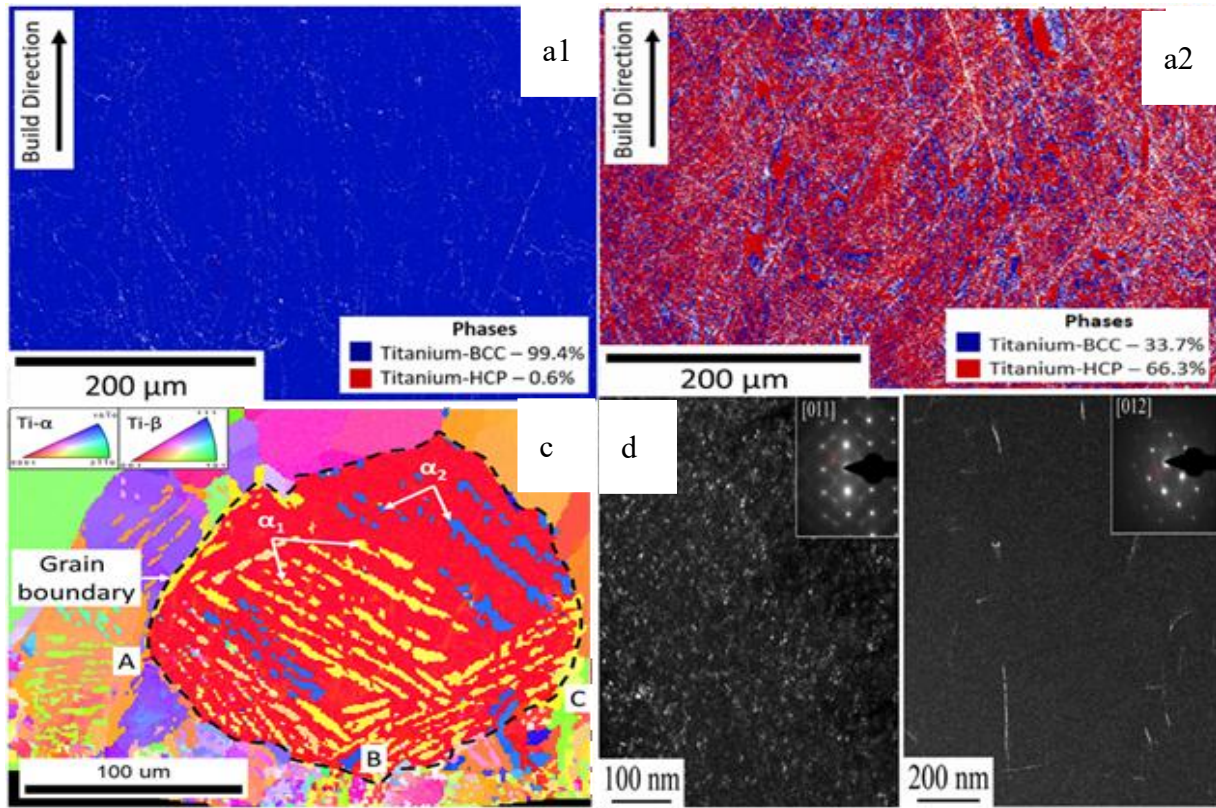


Figure 19. a1 and a2 Representative multiscale microstructural features of LPBF-fabricated Ti-5553 [110]. b is EBSD phase map of the as-built condition showing a near-single-phase β microstructure, EBSD phase distribution after heat treatment illustrating α formation within the β matrix, crystallographic orientation map highlighting prior- β grain morphology and internal α features [123]. TEM images showing nanoscale transformation products including ω -related features and α precipitation within the β matrix [124].

As shown in Fig. 19, the as-built state may appear nearly single-phase β at the EBSD scale, but higher-resolution observations reveal that the β matrix can contain nanoscale transformation-related features. Several studies have reported ω -related features together with high dislocation densities in the as-built condition, indicating that the β matrix is highly non-equilibrium [110]. This is consistent with TEM observations showing transformation-related contrast inside the β matrix. Therefore, the as-built β phase should be interpreted as a metastable starting state rather than as a stable single-phase reference condition.

Differential scanning calorimetry provides further evidence for this metastability. DSC measurements have identified ω -associated thermal events between approximately 220 and 320 $^{\circ}\text{C}$, even in material that appears nominally single-phase β by conventional diffraction [123]. Additional DSC signatures indicate transformation processes associated with ω -assisted α nucleation between approximately 350 and 480 $^{\circ}\text{C}$, followed by progressive β

decomposition toward α formation between approximately 480 and 650 °C [123]. These thermal events show that the as-built β matrix contains transformation precursors that can respond rapidly during subsequent aging.

This transformation sequence is central to understanding heat treatment in LPBF Ti-5553. In a simplified form, the metastable pathway can be described as $\beta \rightarrow \beta + \omega \rightarrow \beta + \alpha$, where ω may act as an intermediate metastable phase that assists α nucleation [98,123]. Because ω -related features and dislocation networks can both influence α precipitation kinetics, nominally identical aging schedules may produce different α distributions if the starting as-built states are different. This helps explain why similar heat-treatment temperatures can lead to different combinations of strength, ductility, and hardness across LPBF Ti-5553 studies.

Fig. 20 further illustrates the thermal response of LPBF Ti-5553 by showing thermal strain, thermal diffusivity, and stress–strain behavior under different thermal and surface conditions. This figure is important because it links phase-transformation signatures with mechanical response. The thermal strain and diffusivity curves show deviations associated with transformation events, while the stress–strain curves demonstrate that heat treatment and surface condition can strongly modify tensile behavior.

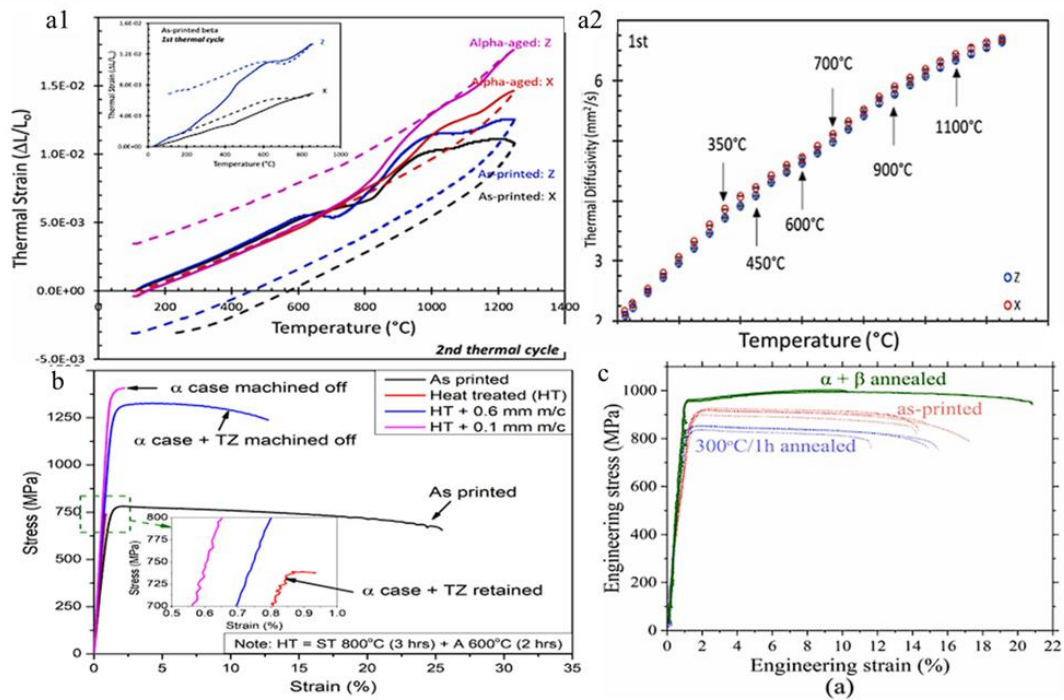


Figure 20. a1 and a2 Representative thermal and mechanical responses of LPBF-fabricated Ti-5553: thermal strain as a function of temperature during heating cycles for as-printed and aged conditions[110]. b is temperature-dependent thermal diffusivity showing deviations associated with phase transformations; engineering stress–strain curves after different levels of surface machining[123]. c is illustrating the influence of α -case removal on tensile ductility; and tensile responses for as-printed, low-temperature aged, and $\alpha + \beta$ annealed conditions [124].

As shown in Fig. 20, thermal analysis does not simply reflect expansion behavior; it also reveals transformation-related changes in the metastable β matrix. Deviations in thermal strain and thermal diffusivity are consistent with the DSC evidence for ω -associated reactions, ω -assisted α nucleation, and β decomposition during heating [123]. These observations support the interpretation that the as-built LPBF Ti-5553 condition contains stored transformation potential, even when the initial phase map appears predominantly β .

The metastable starting condition is also reflected in mechanical behavior. Reported as-built tensile strengths vary across LPBF Ti-5553 datasets, including values of 727 ± 27 MPa [1], 780 ± 10 MPa, and approximately 808 MPa in other studies [116,124]. Other β -dominant conditions show higher strength, including yield strength of 903 ± 8 MPa, UTS of 915 ± 10 MPa, and elongation of $15 \pm 1\%$ [124]. These differences indicate that the as-built condition is not a single mechanical state. Instead, the response depends on local thermal history, retained β stability, defect structure, and nanoscale transformation precursors.

The same point is supported by modulus data. In β -dominant or β -rich conditions, Young's modulus has been reported over a broad range, from approximately 55 GPa to 96.8 GPa, whereas microstructures containing high α fractions, for example about 66% α , can show modulus values up to approximately 120.3 GPa [105,123]. This large modulus variation confirms that phase constitution and α fraction strongly affect stiffness. Therefore, heat treatment can modify not only strength and ductility, but also elastic response through changes in β/α phase balance.

Overall, the as-built LPBF Ti-5553 condition should be understood as a metastable β -dominated starting state with variable substructural content. EBSD and XRD may indicate an apparently single-phase β structure, but TEM and thermal analysis reveal stored defect energy, ω -related features, and transformation pathways that strongly influence subsequent α precipitation [110,116,123]. This metastable baseline is essential for interpreting the aging response, because the same nominal heat treatment can produce different microstructures and properties depending on the initial LPBF thermal history.

2.6.2 Aging response, α precipitation, and strength–ductility balance

The aging response of LPBF-fabricated Ti-5553 is governed by the decomposition of the metastable β matrix and the precipitation of α phase. Because the as-built state already contains a non-equilibrium β -dominated microstructure with stored defect energy, dislocation structures, and possible ω -related transformation precursors, aging does not begin from a fully relaxed β condition. Instead, post-build thermal exposure activates a sequence of phase

transformations whose kinetics and resulting morphology depend on temperature, holding time, prior LPBF thermal history, and surface condition [110,116,123].

Low-temperature aging produces a relatively moderate change in mechanical response. Aging at 300 °C for 1 h has been reported to reduce yield strength from 903 ± 8 MPa to 848 ± 11 MPa while increasing elongation from $15 \pm 1\%$ to $19 \pm 2\%$ [124]. This response suggests that low-temperature exposure can relax part of the as-built defect structure or modify the metastable β state without producing the strong strengthening normally associated with dense α precipitation. In a separate dataset, aging at 300 °C produced UTS of 824 ± 13 MPa and elongation of $17.2 \pm 1.5\%$, again indicating a relatively ductile response compared with peak-aged conditions [110].

The moderate behavior at 300 °C is consistent with the transformation sequence discussed in Section 2.6.1. In this temperature range, ω -associated processes may occur, but extensive α precipitation is limited compared with higher aging temperatures [123]. Therefore, the mechanical response is controlled by a balance between recovery-like effects, changes in metastable β stability, and early-stage precipitation processes. This is why 300 °C aging does not produce the very high strength observed at intermediate aging temperatures, but it can improve or preserve ductility.

Aging in the 400–500 °C range produces a much stronger precipitation response. In this regime, ω -assisted α nucleation and progressive β decomposition become more active, leading to a fine dispersion of α precipitates within the β matrix [123]. This microstructural condition can produce peak-strength behavior, with tensile strengths approaching approximately 1400 MPa and very limited ductility of around 2% under certain aging conditions. Hardness also increases substantially in this temperature range, with reported values of 432 ± 26 HV after aging at 400 °C and 475 ± 20 HV after aging at 500 °C [110,123]. These results indicate that fine α precipitation strongly increases matrix strength, but at the cost of plastic deformation capacity. The strength increase at 400–500 °C should therefore be interpreted as precipitation hardening rather than simple thermal recovery. Fine α precipitates restrict dislocation motion and increase resistance to plastic flow. However, when α precipitation becomes dense, fine, or heterogeneously distributed, deformation can become highly localized. The resulting microstructure may show high tensile strength and hardness but poor ductility. This explains why peak-aged LPBF Ti-5553 can achieve very high strength while simultaneously exhibiting brittle-like tensile response.

The microstructural basis for this behavior is illustrated by Fig. 19, where heat treatment produces extensive α formation within the β matrix. The contrast between the near-single-phase

β as-built phase map and the heat-treated α -containing phase map demonstrates the strong sensitivity of Ti-5553 to thermal exposure. At the nanoscale, the TEM observations in Fig. 19 further support the $\beta \rightarrow \beta + \omega \rightarrow \beta + \alpha$ transformation route, showing that α precipitation may be preceded or assisted by ω -related features [110,123,124].

At higher aging temperatures, particularly in the 700–800 °C range, the transformation behavior shifts from peak-strength precipitation toward morphology coarsening. Instead of producing a very fine and dense α dispersion, higher-temperature exposure promotes coarser α morphologies and modifies the α/β phase balance. This can reduce strength compared with peak-aged conditions, but it may improve ductility by decreasing the severity of localized deformation. Reported tensile data show this transition clearly: aging at 700 °C produced UTS of approximately 1088 MPa with elongation of about 14.2%, whereas aging at 800 °C produced UTS of approximately 951 MPa with elongation of about 15.6% [110,124].

These values show that higher-temperature aging sacrifices some strength compared with peak-strength aging but restores a more useful strength–ductility balance. The change is not controlled only by the total amount of α phase; it is also governed by α morphology, spacing, thickness, and distribution. Coarser or more continuous α morphologies can reduce strength relative to fine α precipitates, but they may allow more stable plastic deformation if they reduce strain localization. Therefore, α morphology is a central parameter in the heat-treatment response of LPBF Ti-5553.

Aging time also has a major influence. At 700 °C, increasing the aging duration from 1 h to 4 h increased elongation from 0.9% to 5.7% [124]. This result demonstrates that heat-treatment temperature alone does not define mechanical behavior. Holding time controls the coarsening, redistribution, and possible spheroidization of α features, which in turn affects strain localization and fracture resistance. Therefore, both temperature and time must be considered when designing aging treatments for LPBF Ti-5553.

The stress–strain data shown in Fig. 2.18 reinforce this point. The comparison among as-printed, low-temperature aged, and $\alpha+\beta$ annealed conditions shows that thermal exposure can either preserve ductility, increase strength, or change the deformation mode depending on the transformation path [110]. The same figure also shows that surface machining and α -case removal can strongly influence measured tensile response [124]. Therefore, the apparent mechanical effect of aging cannot be separated from surface condition, especially when oxygen-enriched surface layers or transition zones are present after heat treatment.

Overall, the aging response of LPBF Ti-5553 is characterized by a strong temperature-dependent strength–ductility trade-off. Low-temperature aging near 300 °C

produces moderate strength and relatively good ductility, while aging in the 400–500 °C range promotes strong α precipitation, high hardness, and peak-strength behavior with limited ductility [110,123,124]. Higher-temperature aging in the 700–800 °C range promotes coarsening and partial ductility recovery, reducing strength but improving deformation stability [110,124]. This behavior confirms that the key variable is not simply the presence of α phase, but the morphology, distribution, and thermal history of α precipitation within the metastable β matrix.

2.6.3 STA, BASCA, and sub-/near-transus heat-treatment routes

Beyond direct aging treatments, post-processing of LPBF Ti-5553 can be interpreted through three broader thermal routes. The first route corresponds to low-temperature aging within the ω -dominated or early α -precipitation regime, typically around 300–500 °C, where strengthening is governed by metastability-driven transformation processes. The second route involves sub-transus solution and aging schedules that promote controlled α precipitation within the metastable β matrix, commonly producing mixed primary and secondary α morphologies. The third route consists of near- or super-transus solution treatments that partially or fully reset the prior β microstructure before subsequent aging, thereby modifying the topology, spacing, and distribution of α precipitates. These different thermal trajectories are important because they do not produce equivalent microstructures, even when the final phase constitution may be broadly described as $\alpha+\beta$ [128–130].

A representative sub-transus two-step aging route consists of treatment at 750 °C for 2 h followed by aging at 600 °C for 2 h. This route transforms the as-built β -dominant microstructure into an $\alpha+\beta$ structure containing approximately 76.9% α [128]. The resulting mechanical properties are balanced rather than peak-strength dominated, with reported ultimate tensile strengths of approximately 1133–1166 MPa and elongations of approximately 13–16% [128]. This response shows that controlled α precipitation can produce useful strength–ductility combinations in LPBF Ti-5553 even without a prior β -solution treatment step. Similar sub-transus heat-treatment routes have also been reported to promote intragranular α precipitation within the β matrix [128,131].

The distinction between STA-type and BASCA-type treatments is especially important. STA treatments generally involve solution treatment within or near the β -transus followed by short aging. This route produces fine secondary α precipitates within the β matrix, which strongly restrict dislocation motion and maximize strength. In additively manufactured Ti-5553, STA processing has generated yield strengths of approximately 1360 MPa and ultimate tensile

strengths of approximately 1460 MPa, but with limited elongation of about 4.2% [131]. This behavior is consistent with a fine α s-dominated microstructure that provides strong precipitation hardening but limited plastic accommodation.

In contrast, BASCA or β -anneal-slow-cool-and-age treatments promote coarser α morphologies, including primary α (α_p) together with secondary α precipitates. These microstructures typically exhibit lower strength but substantially higher ductility. For BASCA-treated additively manufactured Ti-5553, reported properties include yield strength of approximately 888 MPa, ultimate tensile strength of approximately 971 MPa, and elongation exceeding 22% [131]. The comparison between STA and BASCA therefore demonstrates that the strength–ductility balance is governed strongly by α morphology and distribution, not only by the total amount of α phase.

This morphology-controlled behavior is consistent with broader reports showing that fine α s-dominated microstructures maximize strength, whereas coarser α_p -containing architectures improve plastic accommodation and damage tolerance [70,116]. In practical terms, very fine α precipitation increases hardness, modulus, and tensile strength, but can promote localized deformation and early fracture if the α distribution is dense or highly continuous. Coarser α morphologies reduce the strengthening effect but allow more stable deformation, thereby improving ductility and fracture resistance. Therefore, heat treatment of LPBF Ti-5553 should be designed around α morphology, α spacing, and phase topology rather than only target phase fraction.

This interpretation also explains why the sharp strength peak near 1400 MPa obtained after aging in the 400–500 °C range should not be treated as the optimum heat-treatment condition for all applications. That regime represents a localized strengthening maximum associated with ω -controlled transformation and fine α precipitation, but it is accompanied by very limited ductility [119,128]. In contrast, multi-step sub-transus routes generally produce more moderate strengthening with improved ductility retention [119,123,132]. Consequently, the strengthening pathway in LPBF Ti-5553 is not unique; it depends on the selected thermal trajectory, the sequence of $\beta \rightarrow \omega \rightarrow \alpha$ transformation, and the final α morphology [128,131,132].

The mechanical trends are also reflected in modulus and hardness evolution. In systematic isothermal aging experiments, Young's modulus increased from 48 ± 1 GPa at 300 °C to 98 ± 4 GPa at 500 °C, while hardness increased from 291 ± 4 HV at 300 °C to 432 ± 26 HV at 400 °C and 475 ± 20 HV at 500 °C [119]. Within the same temperature interval, ultimate tensile strength approached 1397 ± 36 MPa [119]. The simultaneous increase in elastic stiffness,

hardness, and tensile strength confirms that the mechanical response is controlled by bulk microstructural transformation rather than by isolated surface effects alone.

The coupled thermo-physical and mechanical response shown in Fig. 20 supports this interpretation. The thermal strain and diffusivity curves indicate transformation-related deviations during heating, consistent with ω -associated reactions and subsequent α formation [123]. Meanwhile, the stress–strain curves demonstrate that the final tensile response depends on both heat-treatment route and surface condition [105,110]. This means that even when a heat treatment generates a desirable $\alpha+\beta$ microstructure in the bulk, the measured ductility can still be strongly affected by surface α -case, transition-zone effects, and the extent of surface machining.

Near- or super-transus treatments introduce another level of complexity because they can modify or partially reset the prior β architecture before aging. Such treatments may alter the topology of α precipitation, change the relationship between α colonies and prior β grain boundaries, and influence subsequent fracture behavior. However, the available LPBF Ti-5553 literature indicates that heat treatment does not erase all effects of additive manufacturing. The prior solidification structure, defect population, surface condition, and local thermal history can continue to influence the post-treated mechanical response, particularly when defects or oxygen-enriched surface layers remain present.

Overall, STA, BASCA, and sub-/near-transus heat treatments demonstrate that LPBF Ti-5553 does not follow a single monotonic precipitation-hardening pathway. STA-type treatments maximize strength through fine secondary α precipitation but reduce ductility. BASCA-type treatments promote coarser α morphologies and provide substantially better ductility at lower strength. Sub-transus two-step routes can achieve intermediate and often more useful strength–ductility balances, such as UTS values of 1133–1166 MPa with elongations of 13–16% after 750 °C/2 h + 600 °C/2 h treatment [132]. Therefore, the design of heat treatment for LPBF Ti-5553 must consider the complete thermal route, α morphology, surface condition, and inherited AM defect structure rather than relying only on nominal aging temperature or total α fraction.

An additional feature of near- and super-transus routes is that the concept of a complete β reset does not automatically guarantee improved mechanical performance. Super-transus annealing at 1000 °C for 0.5–10 min followed by iced-water quenching can convert the as-built columnar β morphology into equiaxed β grains while retaining a β -only phase constitution, without martensite formation or α precipitation [114]. However, subsequent cooling and aging

conditions strongly determine the final α topology. For example, furnace cooling at approximately $3\text{ }^{\circ}\text{C min}^{-1}$ followed by aging at approximately $575\text{ }^{\circ}\text{C}$ for 6 h can produce high α fractions of about 66%, but with limited tensile ductility of approximately 2.7% [116,123]. This shows that a high α fraction alone does not ensure a favorable strength–ductility balance. Cooling path also affects impact resistance. Dual heat-treatment schedules using sub-transus solution temperatures of approximately $750\text{--}800\text{ }^{\circ}\text{C}$ with air cooling after both solution and aging steps generate mixed $\alpha\text{p}/\alpha\text{s}$ architectures and improved impact toughness of approximately $10.8\text{--}15.2\text{ J cm}^{-2}$. In contrast, super-transus solution routes suppress primary α formation and produce predominantly secondary α , approximately 92% αs , reducing impact toughness to about 6.5 J cm^{-2} [110]. Therefore, the relative balance between grain-boundary primary α and intragranular acicular secondary α is important for deformation compatibility and crack-initiation resistance.

Localized precipitation heterogeneity can further contribute to premature damage initiation. A recurring feature is the formation of precipitation-free zones. In Zhang et al., aging produced an $\alpha\text{GB}\text{--PZ}\text{--PFZ}$ architecture, where the PFZ corresponds to a softer β region that can act as a preferential crack-initiation site [128]. Similar PFZs have been reported adjacent to grain-boundary α and primary α in β -anneal-plus-aging microstructures, where they were directly associated with crack nucleation and reduced tensile ductility [116]. These observations reinforce that the strength–ductility balance in heat-treated LPBF Ti-5553 is governed less by nominal α fraction than by α morphology, α continuity, PFZ formation, and local deformation compatibility.

2.6.4 Surface damage, α -case, transition zone, and fracture after heat treatment

In addition to bulk phase evolution, heat treatment of LPBF Ti-5553 can produce strong near-surface microstructural gradients that significantly affect mechanical performance. This is particularly important for additively manufactured titanium alloys because the as-built surface already contains roughness, partially melted particles, contour irregularities, and near-surface defects. During post-build thermal exposure, oxygen uptake at the surface can lead to the formation of oxygen-enriched α -case layers and associated transition zones. These surface-modified regions may dominate fracture behavior even when the bulk microstructure has been optimized by aging or solution treatment [124,128].

The presence of α -case is especially detrimental because oxygen-stabilized α is harder and more brittle than the β -rich matrix. As a result, heat-treated specimens retaining oxygen-enriched surface layers can exhibit severe embrittlement regardless of the refined core

microstructure [124]. This means that a heat treatment designed to improve the α/β phase balance in the bulk may still produce poor ductility if the surface layer is not removed or controlled. Therefore, surface condition must be treated as an integral part of the heat-treatment response, rather than as a secondary post-processing issue.

Surface-related damage and fracture features after heat treatment are summarized in Fig. 21. This figure is important because it connects three levels of evidence: fracture morphology, cross-sectional α -case/transition-zone formation, and the transition from ductile to brittle fracture modes.

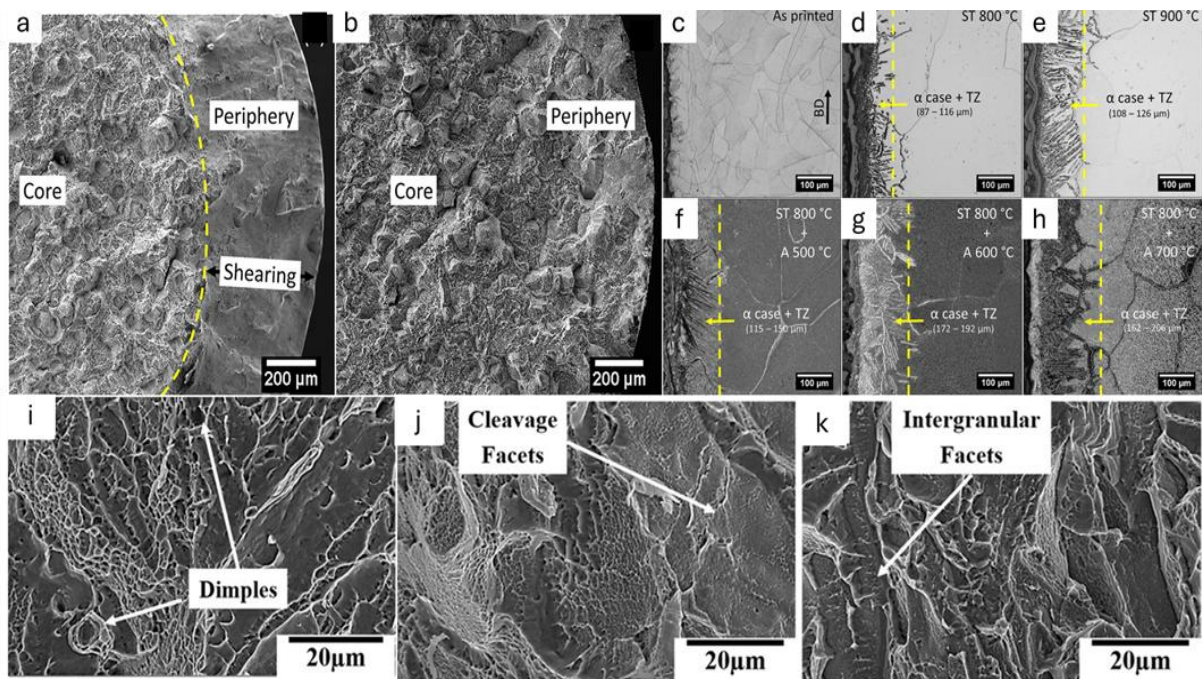


Figure 21. (a-h) Representative surface damage features and fracture morphologies in LPBF-fabricated Ti-5553 after post-processing: fracture surfaces showing the distinction between core and periphery regions with shear-dominated deformation in surface-modified zones[124]. (i-k) cross-sectional microstructures showing oxygen-enriched α -case layers and associated transition zones under different heat-treatment conditions; and fracture modes including ductile dimples, cleavage facets, and intergranular features, demonstrating the transition from ductile to brittle fracture behavior governed by surface-induced microstructural heterogeneity[128].

As shown in Fig. 21a–b, fracture surfaces can exhibit a clear distinction between the core and periphery regions. The periphery may show shear-dominated deformation associated with surface-modified zones, indicating that fracture is not governed only by the bulk α/β microstructure. Instead, the oxygen-enriched surface layer and transition zone can locally alter deformation behavior and provide preferential paths for crack initiation or crack propagation [124].

The cross-sectional observations in Fig. 21c–h show the development and thickening of oxygen-enriched α -case layers and associated transition zones under different heat-treatment

conditions. These gradients extend from the surface toward the bulk and produce a local microstructural transition from oxygen-stabilized α -rich surface regions to the underlying heat-treated core. Because the α -case is harder, more brittle, and less deformation-compatible with the β -rich or $\alpha+\beta$ core, it can act as a mechanically weak interface during tensile or cyclic loading [124].

The fracture morphologies in Fig. 21i–k further demonstrate how surface-induced microstructural heterogeneity changes fracture behavior. Ductile dimples indicate microvoid nucleation and coalescence, while cleavage facets and intergranular features indicate a transition toward more brittle fracture modes [128]. The coexistence of these fracture modes shows that heat-treated LPBF Ti-5553 may fail through mixed mechanisms, where the bulk may retain some ductility but the surface-modified layer promotes premature crack initiation and brittle propagation.

This surface-controlled fracture behavior is consistent with the broader defect-controlled mechanical response of LPBF Ti-5553. Fatigue investigations show that crack initiation is frequently governed by near-surface defects, with fracture surfaces indicating nucleation at surface or subsurface flaws [123]. Therefore, even after heat treatment, the improvement in mechanical performance remains limited if surface defects, α -case, or transition-zone heterogeneity are retained. This is particularly important in fatigue, where the largest surface-connected flaw can dominate life even when the internal microstructure is strengthened or homogenized.

The influence of surface condition is also reflected in fatigue performance after post-processing. Endurance limits of approximately 50 MPa in as-built or β -annealed conditions have been reported to increase to approximately 100 MPa after machining, highlighting the dominant role of surface defect removal [116]. However, even the machined additively manufactured condition remains far below wrought Ti-5553, for which an endurance limit of approximately 350 MPa has been reported [116]. This difference indicates that surface machining improves fatigue resistance by removing the most severe surface flaws, but it does not completely eliminate the influence of residual porosity, subsurface defects, or AM-specific microstructural heterogeneity.

The role of surface condition after heat treatment must therefore be interpreted together with α morphology and defect population. A refined $\alpha+\beta$ core microstructure may provide high strength or improved ductility, but if an oxygen-enriched α -case remains at the surface, the measured tensile or fatigue response can be controlled by the surface layer rather than by the bulk. Similarly, if heat treatment produces precipitation-free zones, grain-boundary α

networks, or brittle α s morphologies, these features can interact with surface cracks and accelerate failure [116,128,131].

Overall, heat treatment of LPBF Ti-5553 involves both bulk and surface transformations. In the bulk, aging and solution treatments control β decomposition, α precipitation, and the strength–ductility balance. At the surface, oxygen uptake can form α -case layers and transition zones that introduce local embrittlement and crack-initiation sites. Fig. 2.19 demonstrates that these surface gradients are directly linked to fracture morphology, including ductile dimples, cleavage facets, and intergranular fracture features [124,128]. Therefore, successful heat treatment of LPBF Ti-5553 requires simultaneous control of α morphology, defect population, and surface integrity.

2.6.5 HIP, cryogenic treatment, and post-processing routes

In addition to conventional aging, STA, BASCA, and surface-related heat-treatment effects, other post-processing routes such as hot isostatic pressing and deep cryogenic treatment have been investigated for LPBF Ti-5553. These treatments are important because they modify the material through different mechanisms. HIP primarily targets internal porosity and defect morphology through simultaneous application of high temperature and pressure, while DCT mainly modifies the internal defect structure, residual stress state, sub-grain boundaries, and subsequent precipitation response. Therefore, these treatments provide further evidence that the post-processing response of LPBF Ti-5553 is governed not only by phase fraction, but also by defect state, residual stress, and the sequence of thermal and non-thermal treatments.

Hot isostatic pressing provides a direct example of defect-controlled post-processing behavior in LPBF Ti-5553. By applying elevated temperature and pressure simultaneously, HIP reduces internal porosity, modifies pore morphology, and lowers local stress concentrations within the microstructure. In one reported case, HIP performed at approximately 815 °C increased yield strength from 123.15 ± 1.42 ksi to 141.8 ± 0.81 ksi and increased ultimate tensile strength from 127.633 ± 0.52 ksi to 147.45 ± 0.45 ksi. At the same time, elongation improved from $14.97 \pm 2.89\%$ to $17.99 \pm 1.35\%$, accompanied by approximately 20% α precipitation [133]. This simultaneous increase in strength and ductility is significant because it shows that porosity reduction and controlled phase evolution can partially overcome the conventional strength–ductility trade-off.

However, the effect of HIP is not limited to pore closure. The same study reported an increase in dislocation density after HIP, which was attributed to transformation-related volumetric expansion during $\beta \rightarrow \alpha$ precipitation [133]. This indicates that HIP simultaneously modifies

both the defect structure and the phase constitution. In addition, EDS mapping revealed chemical partitioning during the HIP cycle, with α enriched in Ti and Al and depleted in V, Mo, and Cr [133]. This partitioning is consistent with the thermodynamic roles of Al as an α stabilizer and Mo, V, and Cr as β stabilizers. Therefore, HIP should be interpreted as a coupled densification–transformation treatment rather than a purely defect-closing process.

The improvement in tensile properties after HIP also supports a broader interpretation of LPBF Ti-5553 mechanical behavior. Since HIP reduces the severity of internal pores while promoting α precipitation, the resulting response reflects both lower defect sensitivity and increased precipitation strengthening. This is especially relevant for fatigue and fracture behavior, where internal and near-surface defects can act as crack-initiation sites. Nevertheless, HIP alone may not fully resolve surface-related limitations, because α -case, roughness, contour defects, and surface-connected flaws may still remain unless surface finishing or machining is also applied. Therefore, HIP is most effective when considered as part of an integrated post-processing route that also addresses surface integrity.

Deep cryogenic treatment introduces a different post-processing pathway. DCT does not significantly alter the overall phase constitution of LPBF Ti-5553, and XRD measurements have shown that the β phase remains dominant after DCT without clear α -phase formation [128]. However, SEM and EBSD observations reveal that DCT can substantially modify the microstructure at the sub-grain and defect-structure level. Specifically, cryogenic treatment reduced the average grain width from approximately $131 \pm 21 \mu\text{m}$ to $89 \pm 12 \mu\text{m}$ and increased the fraction of low-angle grain boundaries from about 33.7% to 58.6%. These changes were attributed to cryogenic contraction, increased internal stress, dislocation accumulation, sub-grain formation, and defect rearrangement within the β matrix [128].

The mechanism of DCT is therefore different from conventional aging. Instead of directly producing a new $\alpha+\beta$ phase constitution, DCT modifies the internal defect structure and residual stress state. During cooling to liquid-nitrogen temperature, lattice contraction and high supercooling generate internal stresses that promote dislocation slip, pile-up, and rearrangement into low-energy structures. On reheating, microplasticity associated with these defects may introduce compressive residual stresses, which can suppress microcrack development and contribute to improved mechanical properties [128]. This mechanism explains why DCT can improve strength and ductility without producing large changes in XRD-detectable phase constitution.

Mechanical data support this interpretation. In the reported PBF–LB Ti-5553 study, the as-built material showed UTS of approximately $727 \pm 27 \text{ MPa}$ and ductility of $10.2 \pm 1.0\%$. DCT for

24 h increased UTS to 795 ± 15 MPa and ductility to $11.9 \pm 1.5\%$, while DCT for 48 h further increased UTS to 815 ± 18 MPa and ductility to $12.9 \pm 1.2\%$. The improvement is therefore moderate but consistent. The same study also concluded that the effect tends to saturate with extended cryogenic treatment time, because extending DCT from 24 h to 48 h did not produce a proportional improvement in mechanical properties [128].

The sequence of DCT and aging is particularly important. Since DCT introduces defects, sub-grain boundaries, and internal stress fields, it can change the nucleation behavior of α during subsequent aging. When DCT is performed before aging, the introduced defects provide additional heterogeneous nucleation sites for α precipitation. This can increase the amount of finer intragranular α and produce a stronger but less ductile microstructure. In contrast, when aging is performed before DCT, the major precipitation reaction has already occurred, and the subsequent cryogenic step mainly refines the β -phase defect structure and modifies residual stresses without promoting the same degree of additional α precipitation [128,134].

This sequence dependence is clear in the reported tensile data. DCT followed by aging produced UTS of 1309 ± 63 MPa with very low elongation of $1.8 \pm 0.1\%$, whereas aging followed by DCT maintained comparable strength, UTS of 1254 ± 54 MPa, while increasing elongation to $7.35 \pm 0.33\%$, commonly rounded to about 7.4% [128]. These results show that the order of post-processing strongly controls the final strength–ductility balance. DCT before aging favors stronger precipitation hardening but can severely reduce ductility, while DCT after aging provides a more favorable ductility improvement at comparable strength.

Microstructural observations explain this difference. Aging and aging–DCT conditions show relatively similar microstructures, characterized by a coarse columnar β matrix, lamellar α phase, and thicker continuous grain-boundary α . In contrast, the DCT–aging condition produces finer α near grain boundaries together with non-uniform intragranular α distributions, including flake-like and dot-like morphologies [128]. EBSD analysis further shows that the α fraction in aging and aging–DCT samples is relatively close, approximately 25.3% and 27.8%, respectively, whereas the DCT–aging condition increases α content markedly to approximately 60.1% [128]. Therefore, DCT before aging strongly enhances α precipitation, but the resulting heterogeneous α distribution can reduce ductility.

This behavior highlights a broader point: post-processing routes that introduce defects or stored energy before aging can accelerate precipitation, but they may also promote heterogeneity. In LPBF Ti-5553, additional nucleation sites are not always beneficial. If they produce excessive, fine, or non-uniform α precipitation, the material may become stronger but less deformation-compatible. Conversely, if DCT is applied after a controlled aging treatment, it can refine the

β matrix and modify residual stresses while preserving a more favorable α morphology. Thus, the post-processing sequence must be designed based on the desired balance between strength, ductility, residual stress, and precipitation uniformity.

Dynamic loading data further show that heat-treated dual-phase microstructures may not always outperform the as-built β -dominant state under all loading conditions. Under high-strain-rate loading, heat-treated dual-phase microstructures containing approximately 30% α exhibited a lower Hugoniot elastic limit and reduced spall strength compared with the as-built β -dominant condition, despite showing similar equation-of-state behavior [122]. This indicates that precipitation strengthening under quasi-static conditions does not necessarily translate into improved resistance to dynamic failure. In other words, a microstructure optimized for tensile strength may be less resistant to shock-induced damage or spallation if α precipitation reduces dynamic deformation compatibility.

Taken together, HIP and DCT demonstrate that post-processing of LPBF Ti-5553 is governed by a hierarchy of interacting mechanisms. HIP combines defect closure, pore-shape modification, α precipitation, dislocation-density changes, and elemental partitioning. DCT modifies the internal defect structure, low-angle boundary density, residual stress state, and subsequent aging response without necessarily changing the primary phase constitution. The sequence of DCT and aging is especially important, because it determines whether cryogenic treatment acts mainly as a precipitation accelerator or as a post-aging residual-stress/substructure modifier.

Overall, LPBF Ti-5553 does not follow a single monotonic post-processing pathway. Heat treatment and post-processing response depend on the metastable β starting state, defect population, surface condition, α precipitation pathway, residual stress state, and treatment sequence. HIP can improve strength and ductility by reducing internal porosity while promoting controlled α precipitation [133]. DCT can improve properties moderately by refining substructure and increasing low-angle grain-boundary content, but when combined with aging its effect becomes strongly sequence-dependent [128,134]. Therefore, successful post-processing of LPBF Ti-5553 requires simultaneous control of internal defects, surface condition, β -phase metastability, α morphology, and treatment order.

2.6.6 Summary of heat-treatment–microstructure–property relationships

The heat-treatment response of LPBF-fabricated Ti-5553 is governed by the metastable nature of the as-built β matrix and by the complex thermal history inherited from the LPBF process. Although the as-built material is commonly described as β -dominant, it should not be

considered a fully stable or relaxed β condition. EBSD and XRD may indicate a predominantly β microstructure, with reported β fractions close to 99.4% and minor α contents of approximately 0.6% in some cases [116,123], but TEM and thermal-analysis studies show that the matrix may contain ω -related features, stored defect energy, and transformation precursors [110,123]. Therefore, heat treatment begins from a metastable, process-dependent starting condition rather than from a conventional solution-treated β state.

The transformation pathway during thermal exposure can be broadly described as $\beta \rightarrow \beta + \omega \rightarrow \beta + \alpha$, although the exact sequence depends on local thermal history, energy input, and post-processing schedule [98,123]. DSC evidence indicates ω -associated events between approximately 220 and 320 °C, ω -assisted α nucleation between about 350 and 480 °C, and progressive β decomposition toward α formation between approximately 480 and 650 °C [52]. These transformation ranges explain why low-temperature aging, peak aging, and high-temperature coarsening regimes produce very different combinations of strength, ductility, hardness, and modulus.

Low-temperature aging near 300 °C generally produces moderate mechanical changes, sometimes improving ductility while slightly reducing strength. For example, aging at 300 °C for 1 h reduced yield strength from 903 ± 8 MPa to 848 ± 11 MPa while increasing elongation from $15 \pm 1\%$ to $19 \pm 2\%$ [124]. In contrast, aging in the 400–500 °C range promotes strong precipitation hardening through fine α formation, increasing hardness to values such as 432 ± 26 HV at 400 °C and 475 ± 20 HV at 500 °C, and producing tensile strengths approaching approximately 1400 MPa, but often at the cost of very limited ductility [110,119,123]. At higher temperatures, particularly 700–800 °C, α coarsening and redistribution reduce strength but improve deformation stability, with reported values such as UTS ≈ 1088 MPa and elongation $\approx 14.2\%$ at 700 °C, and UTS ≈ 951 MPa and elongation $\approx 15.6\%$ at 800 °C [110,124].

The comparison between STA, BASCA, and sub-transus heat-treatment routes confirms that mechanical performance is controlled not only by α fraction, but also by α morphology, α spacing, continuity, and distribution. STA-type treatments can produce very high strength, with reported YS ≈ 1360 MPa and UTS ≈ 1460 MPa, but limited elongation of approximately 4.2% [131]. BASCA-type treatments, in contrast, produce coarser α morphologies and lower strength, but much higher ductility, with reported YS ≈ 888 MPa, UTS ≈ 971 MPa, and elongation above 22% [131]. Similarly, sub-transus two-step treatments such as 750 °C/2 h followed by 600 °C/2 h can generate $\alpha+\beta$ structures with approximately 76.9% α and balanced tensile

properties of approximately 1133–1166 MPa UTS and 13–16% elongation [132]. These results demonstrate that a maximum-strength heat treatment is not necessarily the most useful route for structural performance.

Surface condition is equally important in heat-treated LPBF Ti-5553. Oxygen uptake during post-build thermal exposure can produce α -case layers and transition zones that locally embrittle the surface and control fracture behavior [124,128]. Even when the bulk $\alpha+\beta$ microstructure is optimized, retained α -case, surface roughness, near-surface defects, or contour irregularities can promote premature crack initiation. Therefore, the measured ductility and fatigue response after heat treatment may be governed by the surface-modified region rather than the bulk microstructure alone. This is consistent with observations showing that machining or removal of surface-affected layers improves mechanical response by suppressing surface-initiated failure [116,124].

HIP and cryogenic treatment further show that post-processing response is not controlled only by phase transformation. HIP at approximately 815 °C increased YS from 123.15 ± 1.42 ksi to 141.8 ± 0.81 ksi, UTS from 127.633 ± 0.52 ksi to 147.45 ± 0.45 ksi, and elongation from $14.97 \pm 2.89\%$ to $17.99 \pm 1.35\%$, while promoting approximately 20% α precipitation [133]. This simultaneous improvement reflects a combined effect of pore closure, defect modification, and precipitation strengthening. DCT, on the other hand, modifies substructure and defect state without necessarily changing the dominant phase constitution. It increased the low-angle grain-boundary fraction from 33.7% to 58.6% and reduced average grain width from 131 ± 21 μm to 89 ± 12 μm [128]. When combined with aging, the sequence becomes critical: DCT + aging produced UTS = 1309 MPa with elongation = 1.8%, whereas aging + DCT produced UTS = 1254 MPa with elongation = 7.4% [128].

Overall, heat treatment of LPBF Ti-5553 must be understood as a coupled response involving metastable β decomposition, ω -assisted α nucleation, α precipitation and coarsening, defect modification, residual stress evolution, and surface transformation. The strongest conditions are generally obtained through fine α precipitation, but the best strength–ductility balance requires controlled α morphology and avoidance of excessive precipitation heterogeneity, α -case embrittlement, and defect-assisted fracture. Therefore, successful post-processing of LPBF Ti-5553 requires simultaneous control of the inherited LPBF defect state, surface condition, β -phase metastability, α morphology, and treatment sequence. This heat-treatment background is essential for interpreting as-built Ti-5553 as a metastable starting material and for understanding how microstructure, defects, and surface condition influence the mechanical and corrosion behavior discussed in the following sections.

2.7 Corrosion behavior, corrosion–fatigue, and bio-interfacial response of LPBF Ti-5553

The corrosion and biological response of LPBF-fabricated Ti-5553 is governed by a coupled interaction between passive film stability, surface morphology, defect population, phase constitution, and environmental exposure. Like other titanium alloys, Ti-5553 derives its corrosion resistance primarily from the spontaneous formation of a TiO₂-based passive oxide film. However, in LPBF material, the effectiveness of this passive film is not controlled by alloy chemistry alone. The as-built surface contains microscale roughness, partially fused powder particles, balling features, surface-connected pores, and lack-of-fusion defects, all of which increase the effective surface area and create localized electrochemical heterogeneities. Therefore, corrosion behavior in LPBF Ti-5553 must be interpreted as an interfacial process rather than as a purely bulk alloy property. Electrochemical stability depends on the continuity and compactness of the passive oxide, while localized corrosion susceptibility depends on surface-connected defects, pore morphology, α/β phase distribution, and electrolyte access pathways. In physiological or chloride-containing environments, LPBF Ti-5553 generally shows stable passivation, but the measured electrochemical response changes markedly with surface preparation. Surface polishing, abrasion, anodization, hydrogen charging, and cyclic loading can each activate different degradation or interfacial response mechanisms. The following subsections discuss passive film stability, surface-condition effects, defect-mediated corrosion, corrosion–fatigue, hydrogen-assisted degradation, and bio-interfacial behavior.

2.7.1 Passive film stability and electrochemical response

Corrosion resistance in Ti-5Al-5Mo-5V-3Cr alloys is primarily governed by the formation of a passive TiO₂-based oxide film, which acts as a barrier to ionic transport in aqueous environments. Electrochemical measurements on LPBF-fabricated Ti-5553 indicate that this passive film remains stable over a wide potential range, with no clear evidence of passive-film breakdown within the tested anodic regime in chloride-containing solutions [106]. This stable passivation behavior provides the fundamental basis for the corrosion resistance of LPBF Ti-5553.

The protective oxide layer forms spontaneously when titanium surfaces are exposed to air, moisture, or aqueous electrolytes. Although TiO₂ remains the dominant oxide on both α and β titanium phases, the oxide morphology, thickness, and local chemistry may vary because of crystallographic structure and alloying-element partitioning. In Ti-5553, alloying elements

such as Al and V may also contribute to stable oxide species, including Al_2O_3 and V_2O_5 , within the surface film. However, oxide continuity can be locally disturbed at secondary-phase interfaces, defect sites, and chemically heterogeneous regions, creating possible pathways for ionic penetration through the passive layer [106]. Thus, the corrosion resistance of LPBF Ti-5553 depends not only on the presence of TiO_2 , but also on how compact, continuous, and defect-free the oxide film is.

Despite intrinsic passivation, the electrochemical response of LPBF Ti-5553 is highly sensitive to surface condition. Dammulla et al. investigated SLM-fabricated Ti-5553 in naturally aerated 3.5 wt.% NaCl and compared as-processed surfaces with abraded and polished surfaces [46]. The as-processed specimens exhibited significant surface roughness, with $S_q = 16.7 \pm 5.3 \mu\text{m}$ and $S_z = 111 \pm 29 \mu\text{m}$, together with balling features, micropores, and fusion pore defects. After abrading and polishing, S_q decreased to $1.8 \pm 0.1 \mu\text{m}$ and S_z decreased to $6.4 \pm 0.4 \mu\text{m}$ [106]. This surface smoothing strongly modified the electrochemical response.

The open-circuit potential shifted from $-118 \pm 7 \text{ mV}$ versus Ag/AgCl in the as-processed condition to $+107 \pm 26 \text{ mV}$ after abrading and polishing, corresponding to a noble shift of approximately 225 mV [46]. This shift indicates that the surface-pretreated specimens reached a more electrochemically stable state. Importantly, this change occurred without altering the bulk composition, confirming that the metal–electrolyte interface and surface oxide condition dominate the measured corrosion response.

A similar dependence was observed in the polarization response. In the as-processed condition, the anodic current at 0.8 V versus Ag/AgCl was $2.10 \pm 0.77 \times 10^{-4} \text{ A cm}^{-2}$. After surface pretreatment, this current decreased by approximately 72 times to $2.92 \pm 0.19 \times 10^{-6} \text{ A cm}^{-2}$ [106]. The cathodic current at -0.8 V also decreased from $1.76 \pm 0.77 \times 10^{-4} \text{ A cm}^{-2}$ to $2.26 \pm 0.38 \times 10^{-5} \text{ A cm}^{-2}$, corresponding to an approximately eightfold reduction. These decreases show that surface smoothing reduces both oxidation and reduction reaction rates, mainly by decreasing real surface area, removing defective native oxide, and promoting a more compact passive film.

Linear polarization resistance measurements further confirm this improvement. The nominal polarization resistance increased from $4.67 \pm 1.78 \times 10^3 \Omega \text{ cm}^2$ in the as-processed state to $1.99 \pm 0.77 \times 10^6 \Omega \text{ cm}^2$ after surface pretreatment, corresponding to a 426-fold increase [106]. This large increase in polarization resistance indicates a substantial reduction in corrosion activity after abrasion and polishing. Therefore, the corrosion resistance of LPBF Ti-5553 is not simply a function of alloy composition; it is strongly controlled by surface roughness, oxide integrity, and defect exposure.

Electrochemical impedance spectroscopy provides additional evidence for the effect of surface condition. In Bode plots, both as-processed and surface-pretreated specimens show high-frequency impedance values of approximately $20 \Omega \text{ cm}^2$ with phase angles near 0° , corresponding to the series resistance of the electrolyte, electrode, and electrical contact [106]. At intermediate frequencies, the impedance increases with decreasing frequency and the phase angle approaches approximately 70° for as-processed specimens and approximately -80° for surface-pretreated specimens [106]. The higher phase angle and its extension to lower frequencies in the surface-pretreated condition indicate more ideal capacitive behavior and a more compact, less defective oxide film.

At low frequency, the impedance modulus at 0.01 Hz was approximately 35 times larger for the surface-pretreated specimens than for the as-processed specimens, increasing from $5.19 \pm 1.29 \times 10^3 \Omega \text{ cm}^2$ to $1.84 \pm 0.49 \times 10^5 \Omega \text{ cm}^2$ [106]. Equivalent-circuit fitting using a Randles-type circuit also showed a 318-fold increase in fitted polarization resistance, from approximately $5.2 \times 10^3 \Omega \text{ cm}^2$ to $1.7 \times 10^6 \Omega \text{ cm}^2$. These impedance results are consistent with the OCP, polarization, and linear polarization resistance data, all showing that polishing improves corrosion resistance by producing a smoother, less defective, and more passivating surface oxide.

The electrochemical behavior is summarized in Fig. 22. This figure combines EIS, anodic and cathodic polarization, hydrogen charging response, and bioactive surface precipitation, showing that LPBF Ti-5553 corrosion behavior is controlled by the condition of the surface oxide and the surrounding environment.

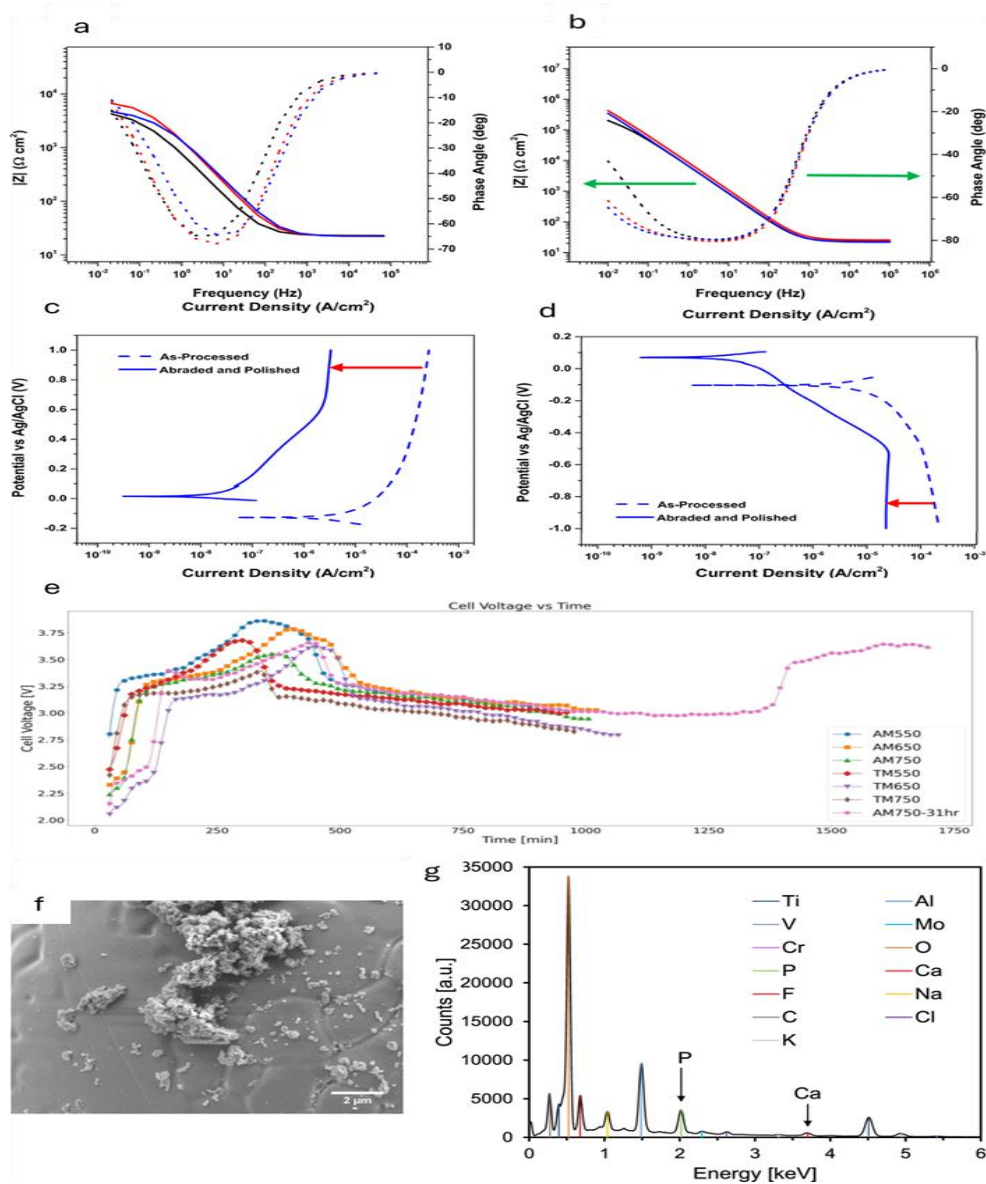


Figure 22. Electrochemical and bioactive surface responses of LPBF Ti-5553: (a,b) Bode plots from electrochemical impedance spectroscopy showing impedance magnitude and phase angle as a function of frequency for different surface conditions[106]. (c,d) potentiodynamic polarization curves in anodic and cathodic regions comparing as-processed and abraded/polished surfaces[106]. (e) cell potential evolution during electrolytic hydrogen charging for additively manufactured and thermomechanically processed samples[106]. (f) SEM image showing surface precipitate formation after immersion in simulated body fluid; and (g) EDS spectrum confirming Ca- and P-containing deposits associated with calcium-phosphate nucleation [108].

As shown in Fig. 22a–b, the surface-pretreated condition exhibits a stronger capacitive response and higher impedance than the as-processed surface, indicating a more protective passive film. The polarization curves in Fig. 22c–d show the same trend: abrasion and polishing shift the electrochemical response toward lower anodic and cathodic currents. The improvement is not caused by a change in Ti-5553 bulk chemistry, but by the removal of

defective surface features and the formation of a more compact oxide on the refreshed and smoothed surface [106].

The electrochemical data also show that the surface response is reproducible and strongly surface-state dependent. Dammulla et al. reported that preliminary deoxidation using a commercial Smut-Go treatment had only a minor effect on the electrochemical behavior of the smoothed alloy under the investigated conditions. The deoxidized specimens showed a noble OCP shift, a twofold increase in nominal polarization resistance, and approximately twofold lower anodic and cathodic polarization currents compared with smoothed specimens without the same deoxidation step [106]. This indicates that mechanical smoothing dominates the corrosion response, while the tested wet-chemical deoxidation step provides only secondary modification.

The hydrogen-charging response shown in Fig. 22e indicates that Ti-5553 can also respond differently under hydrogen-active conditions. Although chloride corrosion behavior is dominated by stable passivation, hydrogen charging introduces another degradation pathway involving hydrogen ingress and trapping, which is discussed in Section 2.7.4. The surface precipitates and EDS spectrum in Fig. 22f–g further show that bioactive deposits containing Ca and P can form after immersion in simulated body fluid, linking the corrosion response to bio-interfacial behavior[108].

Overall, LPBF Ti-5553 exhibits strong passive-film stability in chloride-containing solutions, but the effectiveness of this passivity depends strongly on surface condition. Abrading and polishing reduce roughness from $S_q = 16.7 \pm 5.3 \text{ } \mu\text{m}$ to $1.8 \pm 0.1 \text{ } \mu\text{m}$, shift OCP by approximately 225 mV in the noble direction, reduce anodic current by 72 times, reduce cathodic current by eight times, and increase polarization resistance by several hundred times [106]. These results demonstrate that corrosion resistance in LPBF Ti-5553 is governed by a coupled surface-oxide–roughness–defect framework rather than by alloy chemistry alone.

2.7.2 Effect of surface condition, polishing, and LPBF surface morphology

The electrochemical response discussed in Section 2.7.1 shows that corrosion behavior in LPBF Ti-5553 is strongly surface-state dependent. This dependence originates from the characteristic morphology of the as-built LPBF surface, which contains microscale roughness, melt-track ridges, adhered and partially fused powder particles, balling features, micropores, contour irregularities, and surface-connected lack-of-fusion defects [20,106,108,111,116].

These features increase the true surface area exposed to the electrolyte and introduce local variations in oxide continuity, electrolyte access, and electrochemical activity. Therefore, the surface of LPBF Ti-5553 should be treated as an active microstructural and electrochemical interface, not simply as an external geometric boundary.

As-built LPBF Ti-5553 surfaces can remain highly reactive even when the bulk density is high. This is because surface morphology evolves partly independently of internal densification. Parameter sets optimized for relative density do not necessarily minimize surface flaw severity, and high bulk density values approaching 99.9–99.99% do not guarantee a smooth or electrochemically uniform surface [97,106,116]. Consequently, density metrics alone are insufficient for evaluating corrosion or fatigue susceptibility in the as-built state. The accessibility, connectivity, and morphology of surface-connected defects are more important than the average porosity fraction when assessing interfacial degradation.

The characteristic surface morphology and surface-connected defect state of LPBF Ti-5553 are illustrated in Fig. 23. This figure links topographic roughness, partially fused particles, localized irregularities, and lack-of-fusion defects to the interfacial electrochemical response.

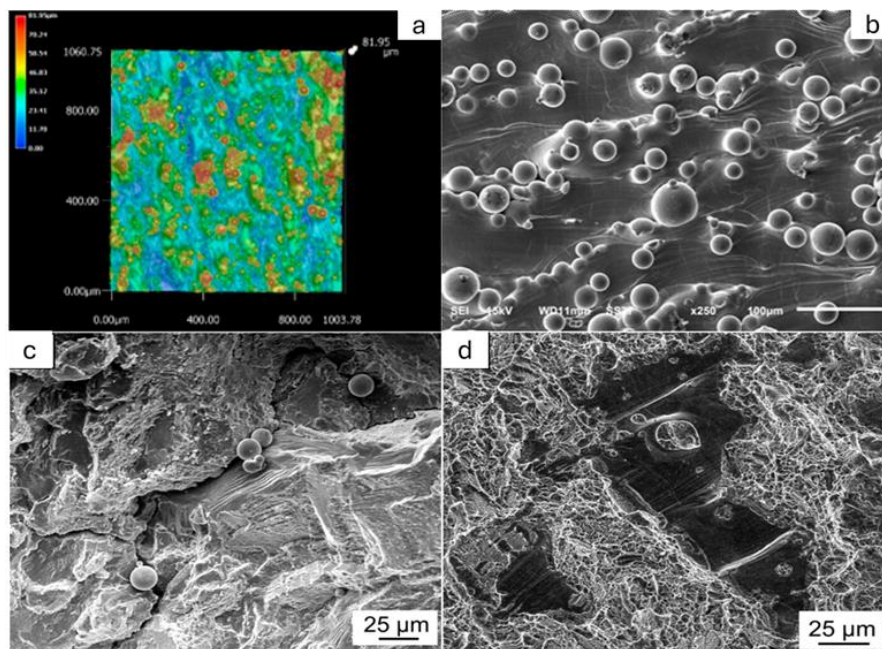


Figure 23. a and b surface morphology and defect characteristics of LPBF-fabricated Ti-5553: surface topography map showing pronounced microscale roughness and spatial heterogeneity across the as-built surface[106]. C and d SEM image showing partially fused spherical particles and irregular surface features characteristic of LPBF processing; higher-magnification SEM image showing localized irregularities and partially fused regions contributing to interfacial heterogeneity; and representative lack-of-fusion defect showing a surface-connected planar feature that can provide a preferential pathway for electrolyte ingress and localized electrochemical activity [108].

As shown in Fig. 23a, the as-built LPBF surface contains pronounced microscale roughness and spatial heterogeneity. Such topographic variation increases the effective electrochemical area and locally modifies the current distribution during polarization or impedance testing. Fig. 23b shows partially fused spherical particles and irregular surface features characteristic of LPBF processing. These particles increase surface area and create local crevice-like regions where electrolyte can be retained. Fig. 23c further shows localized irregularities and partially fused regions that contribute to interfacial heterogeneity. The lack-of-fusion feature shown in Fig. 23d is particularly important because it demonstrates a surface-connected planar defect that can act as a preferential pathway for electrolyte ingress and localized electrochemical activity [106,108].

The surface morphology shown in Fig. 23 explains why mechanical polishing produces such a strong improvement in electrochemical response. Dammulla et al. reported that polishing reduced the root-mean-square roughness from $S_q = 16.7 \pm 5.3 \mu\text{m}$ to $1.8 \pm 0.1 \mu\text{m}$ and reduced the maximum peak-to-valley height from $S_z = 111 \pm 29 \mu\text{m}$ to $6.4 \pm 0.4 \mu\text{m}$ [106]. This smoothing reduces the true surface area, removes defective native oxide, eliminates or blunts surface-connected irregularities, and promotes formation of a more compact passive film. The resulting electrochemical improvement includes a noble OCP shift of approximately 225 mV, a reduction in anodic current from the $10^{-4} \text{ A cm}^{-2}$ range to the $10^{-6} \text{ A cm}^{-2}$ range, and an increase in polarization resistance from $4.67 \times 10^3 \Omega \text{ cm}^2$ to $1.99 \times 10^6 \Omega \text{ cm}^2$ [106].

These results demonstrate that the corrosion response of LPBF Ti-5553 is not controlled only by bulk alloy chemistry or nominal phase constitution. Mechanical surface removal, which does not fundamentally change the bulk microstructure, substantially improves corrosion resistance by reducing true interfacial area and limiting electrolyte access to surface-connected defects [46]. This agrees with mechanical observations in which machining improved elongation from approximately 7–8% to about 16% and increased endurance strength from approximately 50 MPa to about 100 MPa without significantly altering the bulk microstructure [116]. Together, these mechanical and electrochemical results show that the native as-built surface governs both crack initiation and corrosion response.

Laser polishing provides additional evidence for surface-state control. Ahmed et al. showed that laser polishing can significantly reduce surface roughness through localized remelting while retaining a metastable β -dominated phase constitution at the surface. A slight reduction in near-surface hardness after laser polishing was interpreted as localized thermal modification rather than intrinsic strengthening. Therefore, potential improvements in fatigue or corrosion

response following laser polishing should be attributed primarily to geometric smoothing and defect-state modification, with only a limited contribution from bulk phase changes [111].

Surface condition also introduces a trade-off between electrochemical stability and biological interaction. Smoother surfaces improve corrosion resistance by supporting formation of a compact, less defective passive oxide layer [46]. In contrast, rougher LPBF surfaces with partially fused particles increase effective surface area and may promote interfacial interactions with biological environments [125,135]. Thus, the same roughness that enhances biological interaction can also increase electrochemical activity by increasing the effective interfacial area and introducing localized heterogeneities. This coupling creates an inherent design trade-off between corrosion resistance and functional bioactivity.

The term “surface condition” in LPBF Ti-5553 should therefore include both geometric morphology and near-surface microstructural state. Surface integrity studies show that near-surface mechanical properties may differ from the bulk. Grove et al. reported surface microhardness values of approximately 325 HV_{0.025} in conventionally processed as-SLM material, compared with approximately 600 HV_{0.025} in thermally modified conditions [118]. This indicates that near-surface layers can carry the combined imprint of surface geometry, localized thermal history, and process-induced microstructural state. As a result, surface response under corrosion, fatigue, or bio-interfacial loading cannot be interpreted using bulk properties alone.

The available evidence therefore indicates that surface and near-surface conditions exert a dominant influence on LPBF Ti-5553 performance [106,108,116]. The native as-built surface contains adhered particles, melt-pool ridges, contour irregularities, and near-surface porosity, which together define the local stress concentration field and the local electrochemical environment under service conditions. High roughness and surface-connected defects can dominate behavior even when bulk density and tensile strength fall within acceptable ranges. This provides the mechanistic bridge between surface-controlled corrosion, surface-controlled fatigue crack initiation, and the need for surface engineering strategies in LPBF Ti-5553[20,111,116].

Overall, surface morphology is one of the most important controlling factors in the corrosion response of LPBF Ti-5553. Polishing improves electrochemical stability by reducing roughness and defect accessibility, while rough as-built surfaces increase biological interaction but also intensify electrochemical heterogeneity. The ideal surface condition therefore depends on the intended application. For structural applications, minimizing surface-connected defects and roughness is critical for corrosion and fatigue resistance. For bio-interfacial applications,

controlled hierarchical roughness may be beneficial, but it must be engineered so that improved wettability and bioactivity do not compromise passive-film stability.

2.7.3 Defect-mediated localized corrosion and pore/electrolyte interactions

Defect-mediated corrosion in LPBF Ti-5553 is governed by the morphology, connectivity, and spatial distribution of process-induced defects. Although titanium alloys generally exhibit strong passivation in chloride-containing environments, localized electrochemical activity can still develop when defects provide preferential pathways for electrolyte ingress or disrupt the continuity of the passive film. In LPBF Ti-5553, the most relevant defects include lack-of-fusion discontinuities, surface-connected pores, keyhole pores, partially fused particles, melt-pool boundary defects, and contour-related surface irregularities [97,102,106,108]. These defects do not influence corrosion only through their total volume fraction; their shape, connectivity, and accessibility to the electrolyte are more important for localized corrosion behavior.

Lack-of-fusion defects are particularly important because they are often irregular, sharp, and planar. When such defects intersect the surface, they can form interconnected pathways that allow electrolyte penetration into the near-surface region. This behavior is illustrated in Fig. 23d, where a surface-connected lack-of-fusion feature forms a planar discontinuity. Such defects can create confined local environments where oxygen transport, ion accumulation, and pH variation may differ from the external electrolyte. As a result, these regions may promote localized electrochemical activity even when the surrounding titanium surface remains passivated [106,108].

In contrast, keyhole pores are usually more rounded and isolated. Their effect on corrosion is therefore different from that of lack-of-fusion defects. Near-spherical keyhole pores may act as discrete localized reactivity sites, particularly if they are connected to the surface or located in the near-surface region. However, they are less likely than irregular lack-of-fusion defects to form extended interconnected electrolyte pathways. This distinction is important because two specimens with similar porosity levels may exhibit different corrosion responses depending on whether the dominant pore population is irregular and connected or spherical and isolated [102,106].

The importance of defect morphology is consistent with μ CT-based studies showing that high relative density does not guarantee a benign defect population. Specimens with relative densities approaching approximately 99–99.2% may still contain markedly different pore populations depending on processing conditions [102]. Residual porosity on the order of

approximately 1% has also been reported in LPBF Ti-5553, with absolute density values of $4.62 \pm 0.04 \text{ g cm}^{-3}$ compared with a nominal reference of approximately 4.65 g cm^{-3} [106]. Such residual porosity may appear small in a bulk density measurement, but it can be electrochemically significant when pores are surface-connected, clustered, or associated with partially fused particles and rough surface features.

The role of defects is further amplified by the characteristic roughness of LPBF surfaces. Surface-connected pores, melt-track depressions, balling features, and partially melted particles increase the effective surface area and create local regions where electrolyte can be retained [106,108]. These local geometries may behave similarly to micro-crevice environments, especially when narrow openings connect to deeper subsurface defects. Therefore, the corrosion response of as-built LPBF Ti-5553 is controlled by a coupled roughness–porosity–passive-film interaction rather than by passive film chemistry alone.

The passive film can remain globally stable while local electrochemical heterogeneity develops around defects. This explains why LPBF Ti-5553 can show stable passivation in polarization tests while still being sensitive to surface preparation and defect exposure. Abrading and polishing improve corrosion resistance not because they change the alloy chemistry, but because they remove or blunt surface-connected defects, reduce true surface area, and promote formation of a more compact passive film. The strong increase in polarization resistance after polishing, from $4.67 \times 10^3 \Omega \text{ cm}^2$ to $1.99 \times 10^6 \Omega \text{ cm}^2$, directly supports this interpretation [106].

Microstructural heterogeneity can also contribute to localized corrosion. Heat treatment or intrinsic thermal cycling may produce α/β phase coexistence, grain-boundary α , secondary α needles, and precipitation-free zones. These features can introduce local variations in electrochemical potential, passive-film composition, and interfacial reactivity. In $\alpha+\beta$ titanium microstructures, α/β interfaces are often considered electrochemically heterogeneous regions because the local chemistry and crystal structure differ across the interface [106,116,123]. In LPBF Ti-5553, such heterogeneity may be particularly relevant after aging or thermal exposure, where α precipitates form within the retained β matrix.

The morphology of α also matters. Fine secondary α needles, continuous grain-boundary α , and α -rich surface or transition layers can create different electrochemical and mechanical responses. Grain-boundary α may introduce continuous interfacial pathways, while precipitation-free zones adjacent to α can provide local soft regions and compositional gradients [116,128]. Although these features are often discussed in relation to mechanical fracture, they can also influence corrosion by modifying local passive-film stability and

interfacial chemistry. Therefore, localized corrosion in LPBF Ti-5553 should be interpreted using both defect morphology and phase-distribution information.

Surface oxygen enrichment and α -case formation after heat treatment may further change the near-surface electrochemical state. Oxygen-stabilized α -case layers are harder and more brittle than the bulk β -rich or $\alpha+\beta$ microstructure, and they may alter the composition and continuity of the surface oxide [124,128]. If α -case coexists with surface roughness or residual defects, localized stress and localized electrochemical activity may occur in the same near-surface regions. This is important because corrosion, fatigue, and fracture are not independent in LPBF Ti-5553; they are all strongly influenced by surface and near-surface microstructural heterogeneity.

From a mechanistic perspective, defect-mediated corrosion can be described as a sequence of electrolyte access, local oxide disruption, localized electrochemical response, and repassivation. In smooth regions, the TiO_2 -rich passive film limits ionic transport and maintains low current density. At surface-connected defects, electrolyte penetration increases the local reaction area and may expose less stable oxide or freshly disrupted metal surfaces. If the passive film reforms quickly, localized corrosion remains limited. If transport is restricted inside a defect or if mechanical loading repeatedly damages the passive layer, local degradation may become more severe. This mechanism links static corrosion behavior to corrosion–fatigue behavior, which is discussed in the following subsection[130,136].

Overall, defect-mediated corrosion in LPBF Ti-5553 is controlled by defect accessibility rather than defect volume fraction alone. Irregular lack-of-fusion defects are especially critical when surface-connected because they can form electrolyte pathways and localized electrochemical sites. Spherical pores are generally less severe unless they are connected to the surface or clustered. Phase heterogeneity, α/β interfaces, grain-boundary α , precipitation-free zones, α -case layers, and surface roughness further contribute to localized electrochemical variability. Therefore, reliable corrosion assessment of LPBF Ti-5553 requires combined analysis of surface morphology, pore topology, phase constitution, passive-film behavior, and electrochemical response [106,108,116,128].

2.7.4 Corrosion–fatigue behavior and environmental degradation

Environmental effects in LPBF Ti-5553 do not operate through a single degradation mechanism. Instead, the available literature suggests two distinct but interconnected pathways. The first pathway involves corrosion–fatigue interactions in aqueous physiological environments, where cyclic loading repeatedly disrupts and restores the passive film. The

second pathway involves hydrogen-assisted degradation, where hydrogen enters through the surface and is transported and trapped within the microstructure [108,130,136]. These two mechanisms are related because both depend on surface condition, defect population, and microstructural heterogeneity; however, their governing processes are fundamentally different. In simulated physiological environments, LPBF Ti-5553 appears to maintain relatively stable fatigue performance under the investigated conditions. Fatigue and corrosion–fatigue tests performed in Hank’s balanced salt solution (HBSS, pH 7.4, 37 °C) showed negligible differences compared with tests conducted in air, with axial fatigue endurance values remaining in the range of approximately 125–135 MPa [108]. This indicates that exposure to HBSS did not significantly reduce the fatigue threshold in the tested condition. The result is consistent with the strong passivation and repassivation ability of titanium-based passive films in chloride-containing physiological environments.

However, the similarity between fatigue performance in air and HBSS does not mean that the passive film remains unchanged during cyclic loading. Electrochemical monitoring during axial constant-amplitude corrosion–fatigue testing shows clear transient responses. During cycling, the free corrosion potential gradually increases but is interrupted by sudden drops, while the corrosion current remains nearly steady but shows distinct current spikes associated with damage events [108]. These coupled potential drops and current transients indicate repeated local depassivation followed by rapid repassivation during cyclic deformation.

The corrosion–fatigue response and corresponding crack evolution are shown in Fig. 24. This figure is important because it links electrochemical transients directly to surface crack initiation and propagation.

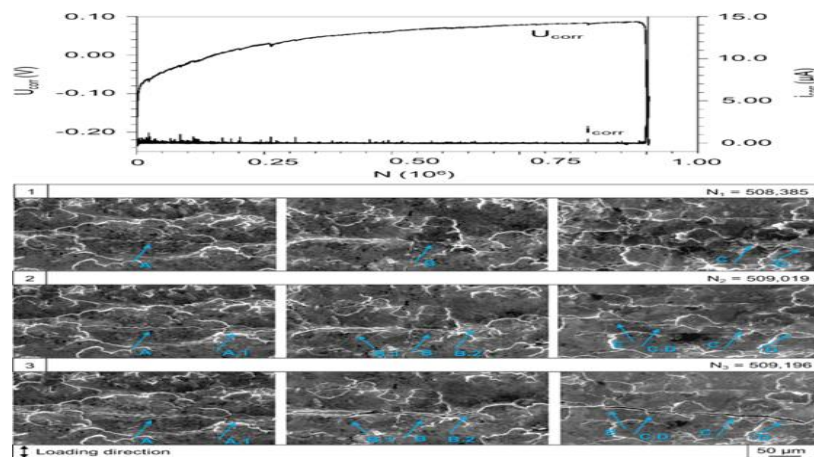


Figure 24. Corrosion–fatigue response of LPBF Ti-5553 during axial constant-amplitude testing: evolution of free corrosion potential and corrosion current density with increasing cycle number, showing progressive changes in electrochemical response with intermittent transients; and SEM micrographs at selected interruption points showing initiation, propagation, and coalescence of surface cracks followed by pronounced crack opening associated with inward crack growth [108].

As shown in Fig. 24, the electrochemical response evolves with the number of cycles during corrosion–fatigue testing. The gradual increase in corrosion potential suggests progressive stabilization or repassivation of the surface, whereas intermittent drops in potential correspond to local surface damage events. The nearly steady corrosion current, interrupted by distinct spikes, indicates that these damage events temporarily increase the active surface area before the passive film reforms [108]. Thus, the passive film behaves as a dynamic self-restoring interface rather than as a static protective layer during cyclic loading.

The SEM interruption sequence in Fig. 24 further confirms that the electrochemical transients are associated with crack evolution. At early interruption points, surface cracks initiate approximately perpendicular to the loading direction. With further cycling, individual cracks propagate, branch, and coalesce. Later stages show pronounced crack opening and inward crack growth. This sequence demonstrates that corrosion–fatigue in LPBF Ti-5553 involves simultaneous mechanical crack development and local passive-film disruption, even when the overall fatigue endurance is not significantly degraded by the physiological environment [108]. Fracture and crack-initiation evidence indicates that the dominant limitation remains defect-controlled. Fatigue life can scatter by up to approximately 10^6 cycles, and this scatter is primarily associated with the distribution of internal defects rather than with direct environmental acceleration [108]. Internal pores and lack-of-fusion features act as dominant crack-initiation sites, indicating that fatigue crack nucleation is governed by defect architecture. Therefore, under the investigated HBSS conditions, corrosion–fatigue behavior is controlled predominantly by defect-mediated initiation, with the environment playing a secondary role. This interpretation is consistent with the static electrochemical behavior discussed in Section 2.7.1. In chloride-containing environments, LPBF Ti-5553 forms a stable passive film and does not show obvious passive-film breakdown over the tested anodic range [106]. Under cyclic loading, the same passivity extends into a dynamic condition through repeated repassivation. In other words, the passive film can be locally disrupted by cyclic strain and crack initiation, but it reforms rapidly enough that the net environmental effect on fatigue endurance remains limited under the investigated testing conditions [108].

Nevertheless, corrosion–fatigue should not be dismissed as irrelevant. The electrochemical transients show that the surface is repeatedly activated during cyclic loading, and the severity of this activation is likely to depend on roughness, surface-connected pores, residual stress, and the density of crack-initiation sites. In rough as-built surfaces, microcracks and surface-connected lack-of-fusion defects can increase the number of local depassivation events. In smoother or polished surfaces, fewer surface-connected defects are available to trigger repeated

film rupture. Therefore, the limited environmental degradation reported in HBSS should be interpreted within the specific context of surface condition, defect population, and test environment [106,108,116].

Hydrogen-assisted degradation represents a second environmental pathway that differs from corrosion–fatigue. Instead of being governed mainly by repeated passive-film rupture and repassivation, hydrogen-assisted degradation involves hydrogen ingress, transport, trapping, and possible hydride formation [136]. In Ti-5553, hydrogen ingress is promoted at surface asperities and irregular features, particularly regions associated with partially fused particles or surface defects. Once hydrogen enters the material, transport is influenced by the β -rich matrix, while trapping can occur at α regions and α/β interfaces [136].

This mechanism has been described as a “ β -transport/ α -trapping” process. The β phase provides a relatively rapid diffusion pathway for hydrogen, while α regions, α/β interfaces, and microstructural heterogeneities act as trapping sites [136]. In heat-treated or α -containing LPBF Ti-5553, the distribution of α phase, grain-boundary α , α/β interface density, and precipitation-free zones may therefore influence susceptibility to hydrogen-related degradation. This links the environmental response directly to the heat-treatment and phase-constitution effects discussed in Section 2.6.

The hydrogen-related features shown later in Fig. 2.23 support the interpretation that hydrogen-related damage is controlled by surface entry followed by microstructure-guided transport and trapping. Unlike corrosion–fatigue in HBSS, where effective repassivation limits net environmental degradation, hydrogen-assisted degradation can produce delayed damage through internal accumulation and hydride formation. Therefore, hydrogen effects may become more significant under hydrogen-generating electrochemical conditions or in environments where hydrogen uptake is enhanced [136].

Taken together, corrosion–fatigue and hydrogen-assisted degradation define a dual-mechanism environmental framework for LPBF Ti-5553. In chloride-containing physiological environments, degradation is dominated by dynamic passivation and defect-mediated crack initiation; the environment plays a secondary role because repassivation is effective [108]. In hydrogen-active conditions, degradation is governed by surface-mediated hydrogen entry, β -phase transport, trapping at α/β interfaces, and possible hydride formation [136]. The common link between both mechanisms is surface and microstructural hierarchy: roughness and defects govern environmental access, while phase distribution and interfacial density control local degradation pathways.

2.7.5 Bio-interfacial response, anodization, TiO₂ nanotubes, and hydrogen effects

The bio-interfacial response of LPBF Ti-5553 is closely connected to the same surface characteristics that control corrosion and fatigue behavior. The as-built LPBF surface contains microscale roughness, partially fused powder particles, melt-track ridges, and localized defects. From a mechanical and corrosion perspective, these features are often detrimental because they increase stress concentration, electrolyte access, and electrochemical heterogeneity. However, from a biological-interface perspective, controlled surface roughness can be beneficial because it increases effective surface area, improves mechanical interlocking with surrounding tissue, and may promote bioactive precipitation in physiological environments [106,108,135].

This dual role creates a fundamental trade-off for LPBF Ti-5553. A smoother polished surface generally improves electrochemical stability by reducing roughness and promoting a compact passive oxide film, as discussed in Sections 2.7.1 and 2.7.2 [106]. In contrast, a rougher LPBF surface may enhance biological interaction by providing hierarchical topography for cell attachment or calcium-phosphate nucleation [125,135]. Therefore, surface engineering strategies for biomedical or bio-interfacial applications must balance corrosion resistance, bioactivity, and mechanical reliability rather than optimizing only one surface property.

One important surface engineering route is electrochemical anodization, which can produce TiO₂ nanotube arrays on titanium alloy surfaces. For LPBF Ti-5553, anodization is particularly attractive because it can superimpose nanoscale oxide features onto the existing microscale LPBF topography. In this way, the surface can develop a hierarchical micro/nano structure: the LPBF process provides microscale roughness, while anodization produces nanotubular TiO₂ features that can improve wettability and bio-interfacial response [125,130].

The surface modification and hydrogen-related features associated with Ti-5553 are shown in Fig. 25. This figure combines anodized TiO₂ nanotube structures with hydrogen-induced features, illustrating how electrochemical surface processes can modify both the external oxide morphology and the internal degradation pathways.

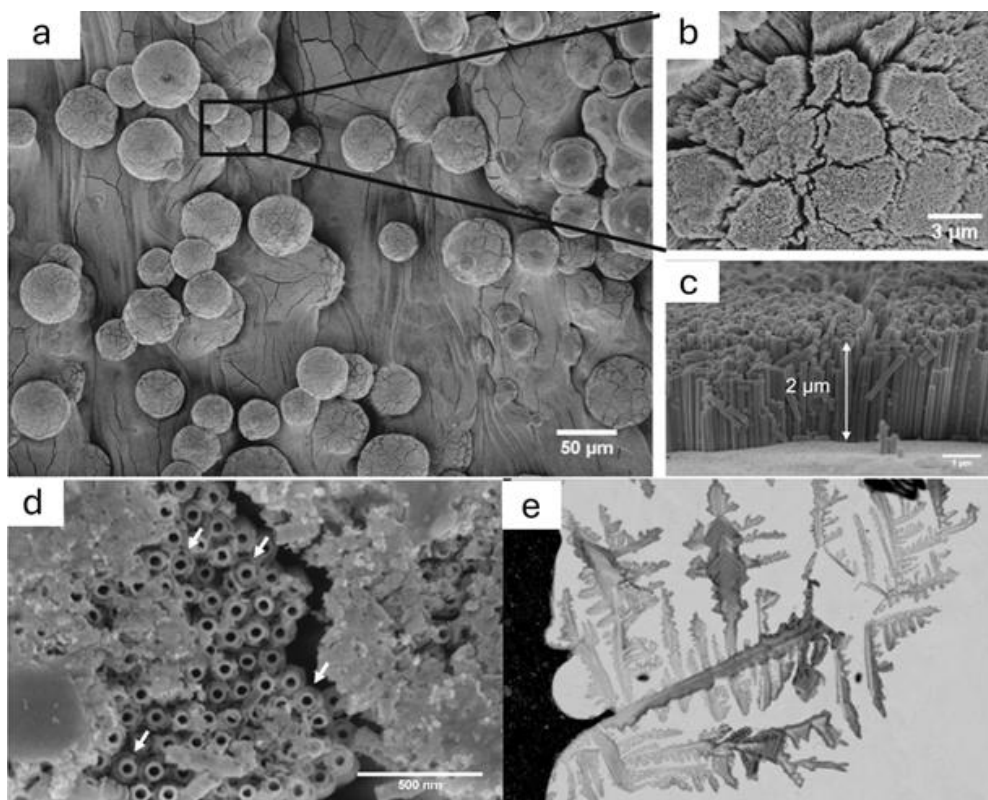


Figure 25. (a-c) surface modification and hydrogen-related features in Ti-5553: SEM images of Ti-5553 anodized at 80 V for 30 min showing crack formation, partial separation of TiO_2 nanotubes into distinct arrays, nanotube formation on spherical particles, and nanotube length of approximately 2 μm ; nanotube arrays obtained after electrochemical anodization [130]. d and e hydride dendritic structures formed due to hydrogen penetration during electrochemical charging, extending along grain boundaries into the Ti-5553 microstructure [136].

As shown in Fig. 25, anodization at 80 V for 30 min produces well-developed TiO_2 nanotube arrays on Ti-5553. The nanotubes form not only on relatively flat regions, but also on spherical or partially fused surface particles, confirming that electrochemical anodization can conform to the complex LPBF surface morphology [130]. The nanotube length is reported to be approximately 2 μm , indicating substantial oxide growth under the applied anodization conditions [130]. However, the figure also shows crack formation and partial separation of nanotube arrays, demonstrating that anodized oxide layers may themselves contain mechanical or morphological discontinuities.

The transition from nanoporous oxide to nanotubular TiO_2 is important for bio-interfacial behavior. Nanotube arrays increase surface area, improve surface wettability, and provide nanoscale features that may promote protein adsorption and cell-surface interactions [125,130]. In LPBF Ti-5553, this nanoscale modification is superimposed on the original microscale LPBF roughness, producing a hierarchical surface that may be advantageous for biological integration. However, the same hierarchical surface can also complicate electrochemical behavior, because cracks, tube openings, and partially separated nanotube

regions may provide pathways for electrolyte penetration if the oxide is not sufficiently stable or adherent.

A major effect of anodization is the strong increase in wettability. Reported contact angles decrease from approximately 78–79° for the as-built or non-anodized surface to about 10° after anodization [130]. This change indicates a transition from a moderately wettable surface to a highly hydrophilic surface. Such a hydrophilic TiO₂ nanotube surface can improve interaction with aqueous physiological fluids and may support early-stage bioactive response. Therefore, anodization modifies not only the surface morphology, but also the surface energy and fluid–surface interaction.

Ti-5553 has also been reported to form relatively larger nanotubes than Ti-6Al-4V under comparable anodization conditions [130]. This difference is attributed to alloy chemistry and the role of β -stabilizing elements in oxide growth and dissolution behavior during anodization. The presence of Mo, V, and Cr can influence local oxide formation, field-assisted dissolution, and nanotube development. Therefore, anodization behavior in Ti-5553 should not be assumed to be identical to that in conventional Ti-6Al-4V, even though both alloys form TiO₂-based surface oxides.

Bioactive response after surface modification has also been observed through the formation of Ca- and P-containing deposits after immersion in simulated body fluid or Hank's solution. As shown earlier in Fig. 2.20f–g, surface precipitates form after immersion, and EDS confirms the presence of Ca and P, consistent with calcium-phosphate nucleation [108,125]. These observations suggest that LPBF Ti-5553 surfaces, particularly when modified by oxide-based surface engineering, may support apatite-like precipitation in physiological environments. Such precipitation is relevant for biomedical interface applications because calcium-phosphate formation is often associated with improved bioactivity.

Post-anodization heat treatment can further modify the oxide structure. Annealing anodized Ti-5553 at 500 °C for 2 h has been reported to promote anatase-containing TiO₂ formation [130]. Anatase is commonly considered beneficial for bioactivity because it can enhance surface reactivity and calcium-phosphate nucleation compared with an amorphous oxide. Therefore, the combination of anodization and low-temperature oxide crystallization provides a route for tailoring bio-interfacial response without fundamentally changing the bulk microstructure of LPBF Ti-5553.

Nevertheless, surface engineering must be evaluated together with corrosion and mechanical reliability. TiO₂ nanotubes can improve wettability and bioactivity, but cracks in the nanotube layer, partial separation of oxide arrays, and underlying LPBF defects may compromise long-

term stability [130]. In load-bearing environments, these discontinuities may act as local sites for film damage or electrolyte penetration. Therefore, anodized LPBF Ti-5553 surfaces require careful optimization of nanotube morphology, oxide adhesion, and substrate roughness to avoid replacing one type of surface defect with another.

Hydrogen-assisted degradation represents another critical environmental issue for Ti-5553. Unlike corrosion-fatigue in HBSS, where passivation and repassivation can limit net environmental degradation, hydrogen charging can introduce hydrogen into the alloy and generate internal damage pathways [108,136]. As shown in Fig. 2.23, hydrogen charging can produce hydride dendritic structures that extend along grain boundaries into the Ti-5553 microstructure [136]. This morphology indicates that hydrogen-related damage is guided by microstructural features rather than being uniformly distributed.

The hydrogen response of Ti-5553 has been described through a β -transport/ α -trapping framework [136]. The β phase provides a relatively rapid diffusion pathway for hydrogen, whereas α regions, α/β interfaces, and grain-boundary features act as trapping sites. In β -dominated as-built LPBF Ti-5553, hydrogen may diffuse through the retained β matrix, but its distribution can be strongly affected by any α precipitates, grain-boundary α , α -case, or heat-treatment-induced α/β interfaces. Therefore, the susceptibility to hydrogen-assisted degradation is linked directly to the phase constitution and heat-treatment state discussed in Section 2.6.

Hydrogen ingress is also surface controlled. Surface asperities, partially fused particles, surface-connected defects, and cracks in oxide layers can all facilitate hydrogen entry under electrochemical charging or hydrogen-generating conditions [136]. Once hydrogen enters the material, internal trapping at α/β interfaces or grain boundaries can promote hydride formation and local embrittlement. This means that surface condition and microstructural state jointly determine hydrogen susceptibility. A smooth, compact passive film may reduce hydrogen entry, whereas rough or cracked oxide surfaces may increase access pathways.

The hydrogen-related features in Fig. 25 therefore extend the corrosion discussion beyond conventional passive-film stability. In chloride-containing environments, the TiO_2 film can remain stable and repassivate effectively [106,108]. Under hydrogen-active conditions, however, degradation can occur through subsurface accumulation, trapping, and hydride formation even if the external surface appears passivated. This distinction is important because electrochemical corrosion resistance does not necessarily guarantee immunity to hydrogen-assisted damage.

Overall, the bio-interfacial and hydrogen-related behavior of LPBF Ti-5553 is governed by the same hierarchical surface–microstructure framework that controls corrosion and fatigue. Anodization can produce hydrophilic TiO₂ nanotube arrays, reduce contact angle from approximately 78–79° to about 10°, and promote Ca/P-containing bioactive precipitation after immersion in physiological media [108,125,130]. However, nanotube cracks, oxide discontinuities, surface roughness, and underlying LPBF defects must be controlled to preserve corrosion resistance and mechanical reliability. Under hydrogen-generating conditions, the β matrix can transport hydrogen while α regions and α/β interfaces act as trapping sites, leading to hydride formation and possible embrittlement [136]. Thus, surface engineering of LPBF Ti-5553 must be designed with simultaneous attention to bioactivity, passive-film stability, defect accessibility, and hydrogen-assisted degradation.

2.7.6 Summary of corrosion, surface, and environmental response

The corrosion response of LPBF-fabricated Ti-5553 is governed by the interaction between passive-film stability, surface condition, defect topology, and microstructural heterogeneity. Although Ti-5553 forms a stable TiO₂-based passive film in chloride-containing and physiological environments, the effectiveness of this film depends strongly on the condition of the LPBF surface. As-built surfaces contain partially fused particles, rough melt-track features, surface-connected pores, and lack-of-fusion defects, which increase the true surface area and create localized electrochemical heterogeneities [106,108].

Surface preparation has a particularly strong effect on corrosion behavior. Abrading and polishing reduce surface roughness from $S_q = 16.7 \pm 5.3 \mu\text{m}$ to $1.8 \pm 0.1 \mu\text{m}$ and shift the open-circuit potential by approximately 225 mV in the noble direction. The same surface treatment decreases the anodic current from the $10^{-4} \text{ A cm}^{-2}$ range to the $10^{-6} \text{ A cm}^{-2}$ range and increases polarization resistance from $4.67 \times 10^3 \Omega \text{ cm}^2$ to $1.99 \times 10^6 \Omega \text{ cm}^2$ [106]. These changes demonstrate that corrosion resistance in LPBF Ti-5553 is controlled not only by alloy chemistry, but also by roughness, oxide compactness, and defect accessibility.

Localized corrosion susceptibility is closely linked to defect morphology. Irregular lack-of-fusion defects are especially critical when surface-connected because they can provide interconnected pathways for electrolyte ingress. In contrast, spherical pores are generally more isolated and act as localized reactivity sites only when they intersect the surface or occur near the surface [102,106,108]. Phase heterogeneity may also contribute to localized electrochemical variability, particularly when α/β interfaces, grain-boundary α , precipitation-

free zones, or α -case layers are present after intrinsic thermal cycling or post-build heat treatment [116,123,124,128].

Corrosion–fatigue studies indicate that LPBF Ti-5553 can maintain stable fatigue performance in Hank’s balanced salt solution under the investigated conditions, with axial fatigue endurance values around 125–135 MPa and no strong degradation relative to air [108]. However, electrochemical monitoring shows repeated depassivation–repassivation events during cyclic loading, with corrosion-potential drops and current spikes associated with crack initiation and propagation [108]. Thus, the passive film remains dynamically active during fatigue, even when the net environmental degradation is limited.

Bio-interfacial behavior introduces an additional surface-design trade-off. Rough LPBF surfaces and anodized TiO₂ nanotube arrays can improve wettability and promote Ca/P-containing surface precipitation, while polishing improves electrochemical stability by producing a smoother and more compact passive film [106,108,125,130]. Anodization at 80 V for 30 min produces TiO₂ nanotubes with lengths of approximately 2 μ m and reduces contact angle from approximately 78–79° to about 10°, indicating a strong increase in hydrophilicity [130]. However, nanotube cracks, oxide discontinuities, and underlying LPBF defects must be controlled to preserve long-term corrosion and mechanical reliability.

Hydrogen-assisted degradation represents a separate environmental concern. In hydrogen-active conditions, hydrogen ingress can occur through surface asperities, oxide cracks, or surface-connected defects. The β matrix can provide a relatively rapid transport path, while α regions and α/β interfaces can act as trapping sites, promoting hydride formation and possible embrittlement [136]. Therefore, environmental performance in LPBF Ti-5553 should be understood through a hierarchical framework in which surface morphology controls environmental access, passive-film stability controls corrosion response, and microstructural phase distribution controls localized degradation and hydrogen trapping.

Overall, corrosion and environmental response in LPBF Ti-5553 cannot be evaluated from bulk density, alloy composition, or passive-film formation alone. A complete assessment requires simultaneous consideration of surface roughness, surface-connected defects, pore topology, passive oxide compactness, phase heterogeneity, corrosion–fatigue crack initiation, and bio-interfacial surface modification. This framework is directly relevant to the present thesis, where corrosion behavior is evaluated together with microstructural, mechanical, nanoindentation, CT, and fractographic characterization of optimized as-built LPBF Ti-5553.

2.8 Summary of research gaps

The literature reviewed in this chapter shows that LPBF-fabricated Ti-5553 has attracted increasing attention as a high-strength metastable β titanium alloy for advanced structural applications. Existing studies have established the fundamental relationships between LPBF parameters, melt-pool stability, defect formation, microstructure, mechanical response, heat-treatment behavior, and corrosion performance. However, the current understanding of as-built LPBF Ti-5553 remains incomplete because many reported studies focus on isolated aspects of the material, such as process optimization, tensile behavior, heat treatment, fatigue, or electrochemical response, rather than providing an integrated experimental assessment of the as-built condition.

One major gap concerns the relationship between optimized LPBF processing and the resulting defect population. Previous studies have shown that stable conduction-mode processing can produce relative densities above 99%, while insufficient energy input promotes lack-of-fusion defects and excessive energy input promotes keyhole porosity [37,98–100,102]. However, high relative density alone does not fully describe the internal defect state. Specimens with comparable density may contain different pore morphologies, pore distributions, and surface-connected defects [97,102,106]. Therefore, there remains a need to evaluate optimized as-built LPBF Ti-5553 using three-dimensional defect characterization, such as X-ray computed tomography, together with surface and microstructural observations.

A second gap is related to the link between surface condition and functional performance. The as-built LPBF surface of Ti-5553 commonly contains partially fused particles, melt-track irregularities, roughness, surface-connected pores, and lack-of-fusion defects [100,105,106,108]. These features can control tensile fracture initiation, fatigue life, and corrosion response even when the bulk material is highly dense [97,106,116]. Existing studies clearly show that polishing or machining can improve both electrochemical response and mechanical performance [106,116]. However, the combined role of as-built surface morphology, internal defects, tensile response, corrosion behavior, and fracture mechanisms remains insufficiently understood for optimized LPBF Ti-5553.

A third gap concerns the microstructure–property relationship in the as-built state. LPBF Ti-5553 is generally characterized by a retained β -dominated microstructure, elongated prior- β grains, variable $\langle 001 \rangle \beta$ texture, and possible nanoscale ω or limited α precipitation depending on local thermal history [19,95,98,110,116,120,123]. These microstructural features can influence tensile behavior, local hardness, deformation localization, and fracture path.

Nevertheless, many studies report global tensile or hardness values without fully correlating them with local phase constitution, melt-pool-scale heterogeneity, and defect distribution. This highlights the need for combined OM, SEM, XRD, nanoindentation, tensile testing, and fractography in a single experimental framework.

A fourth gap is associated with the interpretation of mechanical properties. The literature shows that as-built LPBF Ti-5553 often exhibits a relatively bounded UTS range of approximately 780–950 MPa, whereas elongation is much more scattered, ranging from very low values near 2–3% to values approaching 30% under specific conditions [19,37,105,113,121,124]. This indicates that tensile strength alone is not sufficient to evaluate mechanical reliability. Ductility and fracture behavior are strongly controlled by defect morphology, surface condition, build orientation, local thermal history, and metastable phase evolution [105,110,116,121]. Therefore, a complete experimental assessment should combine tensile data with fracture-surface analysis and local mechanical measurements.

A fifth gap relates to corrosion behavior in the as-built LPBF condition. Ti-5553 forms a stable TiO_2 -based passive film, and polishing can strongly improve electrochemical response by reducing roughness and producing a more compact passive layer [106]. However, the as-built LPBF surface contains roughness, partially fused powder particles, and surface-connected defects that can increase electrolyte access and local electrochemical heterogeneity [106,108]. Although previous work has investigated electrochemical response and corrosion–fatigue behavior, the direct connection between as-built surface state, defect population, phase constitution, and corrosion performance remains limited. This gap is especially relevant for the present thesis because corrosion testing is performed together with microstructural and mechanical characterization.

Heat-treatment studies further demonstrate that LPBF Ti-5553 is a metastable and thermally sensitive alloy. The as-built β matrix can contain stored defect energy and transformation precursors, and subsequent heat treatment can produce a wide range of α morphologies, strength–ductility balances, surface α -case effects, and post-processing responses [110,123,124,128,133]. However, the present thesis focuses on the as-built condition without post-build heat treatment. Therefore, the heat-treatment literature is used here primarily to establish that as-built LPBF Ti-5553 is a metastable starting state whose properties depend strongly on retained β stability, local thermal history, surface condition, and defect structure.

Based on these gaps, the present experimental work focuses on the integrated characterization of optimized as-built LPBF Ti-5553. The aim is not to develop a new LPBF parameter window or to study post-build heat treatment. Instead, the objective is to evaluate the material state

produced under optimized LPBF conditions by combining powder characterization, surface observation, CT-based defect analysis, optical microscopy, SEM, XRD, tensile testing, nanoindentation, corrosion testing, and fractography. This approach enables the relationships among surface morphology, internal defects, phase constitution, local mechanical response, corrosion behavior, and fracture mechanisms to be assessed within a single experimental framework.

Consequently, the central research gap addressed in this thesis can be stated as follows: although optimized LPBF processing of Ti-5553 can produce highly dense β -dominated material, the coupled influence of residual defects, as-built surface condition, local microstructure, mechanical response, corrosion behavior, and fracture initiation has not been sufficiently resolved in an integrated experimental study. Addressing this gap is essential for understanding the reliability of as-built LPBF Ti-5553 and for establishing a basis for future surface modification, post-processing, or application-specific qualification strategies.

Chapter 3

Materials and Methods

3.1 Sample modeling and build layout

The Ti-5Al-5Mo-5V-3Cr (Ti-5553) specimens investigated in this study were designed as cubic samples with nominal dimensions of $9 \times 9 \times 9 \text{ mm}^3$. The computer-aided design (CAD) model was prepared using Materialise Magics software and subsequently exported in STL format for slicing and build preparation. The cubic geometry was selected to provide sufficient material for the subsequent characterization techniques, including density measurement, X-ray computed tomography (XCT), metallographic preparation, optical microscopy (OM), scanning electron microscopy (SEM), X-ray diffraction (XRD), nanoindentation, and corrosion testing. Six specimens were arranged on the build platform with an inter-specimen spacing of approximately 3 mm to minimize thermal interaction between adjacent parts while maintaining stable powder recoating during fabrication. All specimens were built with the same orientation, with the build direction (BD) aligned parallel to the vertical Z-axis, whereas the powder spreading plane corresponded to the XY plane. The build layout used throughout this study is illustrated in Figure 26.

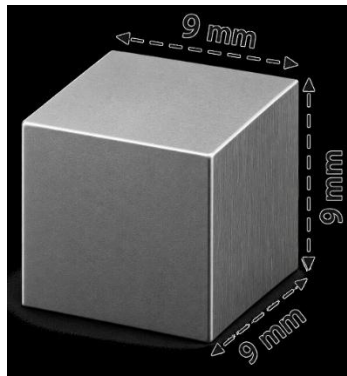


Figure 26. Nominal geometry and build layout of the $9 \times 9 \times 9 \text{ mm}^3$ Ti-5553 cubic specimens fabricated by LPBF on the TRUMPF TruPrint 1000 platform.

Each specimen was assigned a unique identification number to ensure traceability throughout specimen preparation and characterization. Unlike process optimization studies, this work did not investigate multiple processing conditions. Instead, all specimens were fabricated using a single pre-selected LPBF parameter set, and the experimental work focused on evaluating the

resulting as-built microstructure, defect state, phase constitution, mechanical behaviour, and corrosion performance.

3.2 LPBF fabrication procedure

The Ti-5553 cubic specimens were fabricated using a TRUMPF TruPrint 1000 laser powder bed fusion system, shown in Fig. 27. The system was equipped with a Yb-fiber laser with a maximum laser power of 200 W at the workpiece and a nominal laser beam diameter of approximately 80 μm . The use of the same LPBF platform for all specimens ensured that the samples were produced under consistent optical, recoating, and thermal boundary conditions.



Figure 27. TRUMPF TruPrint 1000 LPBF system used for fabrication of the Ti-5553 specimens.

Before printing, the build chamber was purged with high-purity argon to minimize oxidation during powder spreading and laser melting. Argon with a purity of 99.999% was used during fabrication, and the residual oxygen concentration inside the chamber was maintained below 100 ppm. These conditions were selected to limit oxygen pickup, which is particularly important for titanium alloys because oxygen can affect phase stability, surface condition, and mechanical response.

The build plate was preheated to approximately 200 °C prior to fabrication. Preheating was used to reduce thermal gradients during the initial stages of building and to improve the stability of the first deposited layers. Powder layers were deposited using the recoating system and selectively melted according to the sliced CAD model. A constant layer thickness of 30 μm was used throughout the build.

The specimens were fabricated using a fixed LPBF parameter set selected as the optimized processing condition for this work. The laser power was 200 W, the scan speed was 800 mm

s⁻¹, the hatch spacing was 0.115 mm, and the layer thickness was 0.03 mm. Based on these parameters, the volumetric energy density was calculated as 72.46 J mm⁻³. A bidirectional stripe scanning strategy with a 67° rotation between successive layers was applied during fabrication. The build direction was defined as the Z direction, while the layer plane was defined as the XY plane.

Therefore, the purpose of the fabrication step was to produce representative as-built Ti-5553 specimens for subsequent defect, microstructural, mechanical, corrosion, and fracture analyses. After completion of the build, the specimens were allowed to cool to room temperature inside the argon-protected chamber. The samples were then removed from the build plate and cleaned to remove loose powder particles before subsequent cutting, mounting, and characterization.

3.3 Specimen removal and sectioning

After completion of the LPBF fabrication process, the Ti-5553 cubic specimens were separated from the build plate using wire electrical discharge machining (W-EDM). This cutting method was selected because it enables accurate removal of the printed samples while minimizing mechanical deformation, excessive material loss, and thermally induced damage during detachment.

The cutting path was selected to preserve the as-built condition of the specimens as much as possible. Each specimen was cut in a manner that avoided damage to the external surfaces required for subsequent characterization. Where required, additional sectioning was performed through the central region of the cubes to obtain representative cross-sections for metallographic preparation, optical microscopy, SEM observation, XRD analysis, nanoindentation, corrosion-related sample preparation, and fracture-surface examination.

For microstructural analysis, the sectioning orientation was selected so that both the build direction and the layer plane could be identified during observation. The vertical direction of the printed cube was defined as the build direction, while the horizontal plane corresponded to the layer-by-layer deposition plane. This orientation definition was maintained throughout the experimental analysis to allow the observed defect morphology, melt-pool structure, phase distribution, and fracture features to be interpreted consistently.

3.4 Experimental characterization methods

Density and defect characterization were performed to evaluate the consolidation quality of the LPBF-fabricated Ti-5553 specimens. Since process-induced porosity can strongly influence mechanical response, corrosion behavior, and fracture initiation, both bulk density

measurement and three-dimensional defect analysis were used in this work. The Archimedes method was applied to estimate the overall relative density of the as-built cubes, while X-ray computed tomography was used to characterize the internal pore population and defect morphology.

3.4.1 Archimedes density measurement

Archimedes density measurements were performed on the as-built Ti-5553 cubic specimens before any surface modification. This ensured that the original surface texture, near-surface porosity, and LPBF-induced surface condition were preserved during density evaluation. The density of each sample was measured using a precision analytical balance equipped with a density determination kit, following the ASTM B962-17 standard. The Archimedes measurement setup used in this work is shown in Fig. 28.



Figure 28. Archimedes density-measurement setup used for the as-built LPBF Ti-5553 cubic specimens before surface modification.

Before measurement, the samples were cleaned to remove loose powder particles and surface contaminants. Each specimen was first weighed in air to determine the dry mass, w_{dry} . The sample was then fully immersed in room-temperature distilled water and weighed again to obtain the immersed mass, $w_{\text{immersion}}$. The use of distilled water as the immersion liquid allowed the apparent immersed mass to be measured under controlled and repeatable conditions.

The apparent density obtained by the Archimedes method was calculated using Eq. (3.1):

$$\rho_{\text{Archimedes}} = \rho_{\text{liquid}} \times \frac{w_{\text{dry}}}{w_{\text{dry}} - w_{\text{immersion}}} \quad (3.1)$$

where $\rho_{\text{Archimedes}}$ is the apparent density of the specimen, ρ_{liquid} is the density of the immersion liquid, w_{dry} is the mass measured in air, and $w_{\text{immersion}}$ is the apparent mass measured during immersion.

When the wet mass was required, the geometrical or bulk density was calculated using Eq. (3.2):

$$\rho_{\text{Geometrical}} = \rho_{\text{liquid}} \times \frac{w_{\text{dry}}}{w_{\text{wet}} - w_{\text{immersion}}} \quad (3.2)$$

where $\rho_{\text{geometrical}}$ is the bulk density and w_{wet} is the wet mass of the specimen.

The total porosity percentage was calculated using Eq. (3.3):

$$\text{Total porosity percentage} = \frac{\rho_{\text{teoretical}} - \rho_{\text{bulk}}}{\rho_{\text{teoretical}}} \times 100\% \quad (3.3)$$

The relative density was calculated using Eq. (3.4):

$$\text{Relative Density percentage} = \frac{\rho_{\text{Archimedes}}}{\rho_{\text{teoretical}}} \times 100\% \quad (3.4)$$

where $\rho_{\text{theoretical}}$ is the theoretical density of Ti-5553. In this work, the theoretical density was taken as approximately 4.65 g/cm³ based on references.

The Archimedes method provides an average density value for the entire specimen volume and is useful for evaluating the global consolidation quality of the printed samples. However, it does not provide information about pore morphology, pore size distribution, pore connectivity, or the spatial location of defects. Therefore, X-ray computed tomography was also performed to complement the Archimedes measurements and to provide three-dimensional defect characterization. The measured density values were subsequently compared with XCT and ImageJ porosity analyses to provide complementary assessment of the internal defect state.

3.4.2 X-ray computed tomography

In addition to the Archimedes density measurements, X-ray computed tomography (XCT) was performed on selected LPBF-fabricated Ti-5553 specimens to evaluate internal porosity and defect morphology in three dimensions. Unlike the Archimedes method, which provides only an average density value, XCT enables non-destructive visualization of the internal pore population, including pore size, morphology, spatial distribution, and possible connectivity within the printed samples.

The XCT scans were performed using a Phoenix micro-computed tomography system. The specimens were scanned with a voxel size of approximately 2–3 μm , providing sufficient spatial resolution for quantitative pore analysis and three-dimensional reconstruction of the internal defect structure. The reconstructed data were used to examine the distribution of defects with respect to the LPBF build direction and to support interpretation of the density,

mechanical, corrosion, and fracture results. The XCT system and specimen holder used for the analysis are shown in Fig. 29.



Figure 29. X-ray computed tomography system and sample holder used for three-dimensional defect characterization of LPBF Ti-5553 specimens.

After scanning, the reconstructed XCT volumes were processed using threshold-based porosity analysis to identify and segment internal voids from the surrounding Ti-5553 matrix. The defect analysis focused on pore volume fraction, pore size distribution, sphericity, spatial distribution, and the presence of critical internal defects that could influence mechanical response or fracture initiation. Since XCT resolution is limited by the selected voxel size and by the segmentation threshold, defects smaller than the effective detection limit may not be fully resolved. Therefore, XCT results were interpreted together with Archimedes density measurements, optical microscopy, and SEM observations.

The XCT analysis was particularly important for distinguishing between global density and local defect topology. Two specimens may show similar Archimedes density values while containing different pore morphologies or spatial defect distributions. For this reason, XCT was used as a complementary technique to provide three-dimensional information on the internal defect population of the as-built Ti-5553 specimens. However, the classification of defects as gas pores, lack-of-fusion defects, or surface-connected defects was not based solely on XCT data, but was interpreted together with metallographic and SEM observations.

3.4.3 Metallography: grinding, polishing, and etching

Metallographic preparation was performed to obtain representative cross-sections of the LPBF-fabricated Ti-5553 specimens for optical microscopy, SEM observation, and ImageJ-based porosity analysis. The specimens were sectioned according to the required observation plane and mounted in acrylic resin using KMU resin and methyl methacrylate hardener. The mounting procedure was used to facilitate handling during grinding and polishing and to preserve the sample edges during preparation. The mounting stage and materials used for metallographic preparation are shown in Fig. 30.



Figure 30. Mounting stage for metallographic preparation of LPBF Ti-5553 specimens, acrylic resin KMU and methyl methacrylate hardener used for mounting.

After mounting, the specimens were ground using SiC abrasive papers with progressively finer grit sizes. The grinding sequence included P600, P800, P1200, and P2400 papers. During grinding, the samples were rotated between successive steps to remove scratches from the previous stage and to obtain a flat and uniform surface. Water was used as a lubricant and coolant during grinding in order to reduce heating and remove debris from the sample surface. The grinding equipment and abrasive papers used during preparation are shown in Fig. 31.

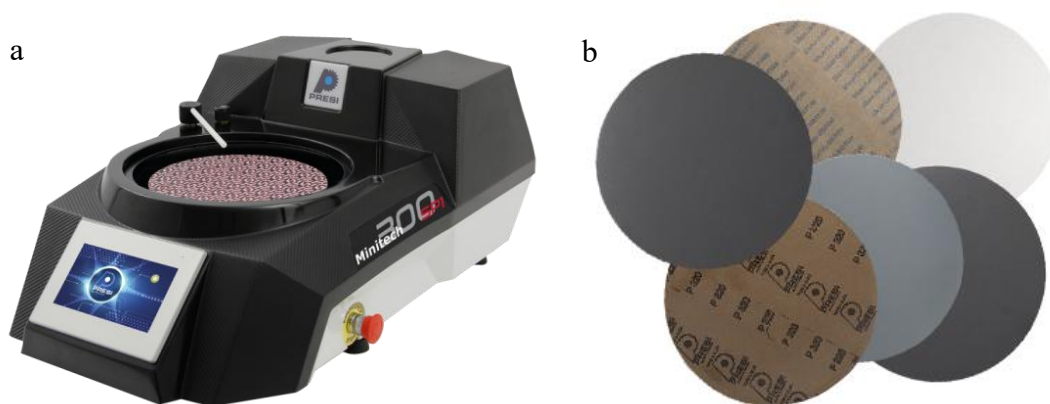


Figure 31. Grinding equipment and consumables used for metallographic preparation: (a) Presi grinding-polishing machine; and (b) SiC abrasive papers with grit sizes of P600, P800, P1200, and P2400.

Following grinding, polishing was performed to obtain a mirror-like surface suitable for microstructural and porosity analysis. The final polishing step was continued until grinding scratches were minimized and a smooth reflective surface was obtained. The polished surface condition was particularly important for distinguishing pores from the metallic matrix during unetched optical microscopy and ImageJ thresholding. The final polished appearance of representative specimens is shown in Fig. 32.

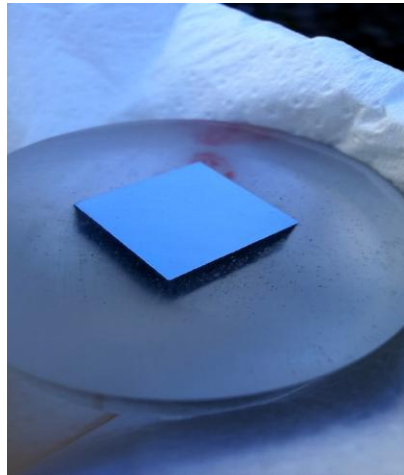


Figure 32. Final appearance of polished LPBF Ti-5553 specimens showing a smooth mirror-like surface ready for microstructural characterization.

Both unetched and etched conditions were used in this work. Unetched polished specimens were used for porosity quantification because pores appear with strong contrast against the metallic matrix without interference from etched microstructural features. Etched specimens were used to reveal melt-pool boundaries, solidification-related features, and the direction-dependent microstructural appearance of the LPBF-fabricated Ti-5553 alloy.

Chemical etching was performed using Kroll's reagent to reveal the microstructural features of the Ti-5553 specimens. After etching, the specimens were rinsed thoroughly with water and ethanol and dried before optical microscopy and SEM observation. The etching time was controlled carefully to avoid over-etching, which could obscure fine microstructural details or artificially enlarge surface features.

3.4.4 Optical microscopy

Optical microscopy was used as the first step of cross-sectional examination of the LPBF-fabricated Ti-5553 specimens. The prepared samples were examined both before and after chemical etching. Before etching, the polished surfaces were observed to evaluate pores, polishing quality, and surface-connected defects. After etching, the same cross-sections were

examined to reveal melt-pool boundaries, grain-related features, and α/β phase-related microstructural contrast.

The optical microscopy observations were performed using a LEICA optical microscope equipped with a manual X–Y stage, as shown in Fig. 33. The microscope was equipped with objective lenses providing magnifications from 5 $\times$ to 100 $\times$. Imaging was carried out using a calibrated black-and-white camera connected to image-acquisition and processing software.

For general surface and cross-sectional inspection, magnifications of 5 $\times$ and 10 $\times$ were mainly used. These magnifications allowed relatively large regions of the sample surface to be examined while still providing sufficient resolution to observe pores, melt-pool boundaries, and etching-related microstructural features. The sample position was adjusted using the X–Y stage controls, and multiple images were collected across the examined surface to obtain representative coverage of each section.

During image acquisition, the microscope focus was carefully adjusted for each field of view to obtain sharp images of pores and microstructural features. In the unetched polished condition, pores and defects appeared as dark regions against the brighter metallic matrix. In the etched condition, melt-pool traces and microstructural contrast became more visible. Images were collected in a matrix format over different regions of the section to support systematic observation of the surface and cross-sectional microstructure. The optical microscope used for the examination and the sample positioning arrangement are shown in Fig. 33.

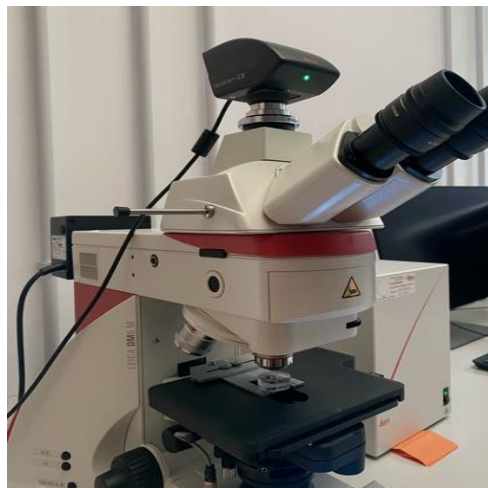


Figure 33. Optical microscopy setup used for examination of the LPBF Ti-5553 specimens, LEICA optical microscope equipped with a manual X–Y stage.

3.4.5 Scanning electron microscopy

Scanning electron microscopy (SEM) was used to examine the microstructure and defect features of the LPBF-fabricated Ti-5553 specimens at higher magnifications than optical microscopy. SEM provides high-resolution surface and cross-sectional images by scanning the specimen with a focused electron beam and collecting signals generated from electron–matter interactions. In this work, SEM observations were used to investigate melt-pool-related microstructural features, pore morphology, surface or near-surface defects, fracture-related features, and local microstructural details that could not be fully resolved by optical microscopy.

Energy-dispersive X-ray spectroscopy (EDS) was employed where chemical information was required, particularly for evaluating the elemental composition of the Ti-5553 powder feedstock and for supporting the interpretation of selected microstructural features. During EDS analysis, interaction between the incident electron beam and the specimen generates characteristic X-rays, which are detected and used to identify the elements present in the analyzed region. This technique was used as a complementary method to evaluate local chemical composition and to support interpretation of microstructural features observed in SEM.

The SEM analysis was performed using a JEOL JCM-6000 Plus scanning electron microscope operated at an accelerating voltage of 5 kV. Images were acquired at different magnifications depending on the feature of interest, including pores, melt-pool boundaries, microstructural contrast, and defect morphology. SEM observations were interpreted together with optical microscopy to provide complementary information on pore morphology, melt-pool characteristics, fracture features, and local microstructural details. The SEM equipment used in this work is shown in Fig. 34.



Figure 34. JEOL JCM-6000 Plus scanning electron microscope used for microstructural and defect characterization of the LPBF Ti-5553 specimens.

3.4.6 Image analysis for porosity quantification

Digital image analysis was performed to quantify the two-dimensional cross-sectional porosity observed on polished cross-sections of the LPBF-fabricated Ti-5553 specimens. The analysis was carried out using ImageJ software, which is widely used for quantitative image processing and microstructural measurements in materials science. This method was used as a complementary approach to Archimedes density measurements and XCT analysis, providing two-dimensional information on the pore area fraction visible on metallographic sections.

Optical micrographs obtained from polished, unetched cross-sections were used for porosity quantification. The use of polished and unetched images was preferred for this analysis because pores and voids appear as dark features against the brighter metallic matrix, while etching-related contrast can complicate threshold selection. The selected images were first converted to 8-bit grayscale format, where each pixel intensity corresponds to a local gray-level value. A suitable threshold range was then applied to separate dark pore regions from the Ti-5553 matrix.

During thresholding, care was taken to minimize the influence of polishing scratches, surface contamination, and image artifacts. The thresholded images were checked visually to ensure that the selected dark regions corresponded mainly to pores or voids rather than preparation-related defects. After segmentation, ImageJ was used to calculate the percentage area of the dark regions relative to the total analyzed image area. This value was taken as the two-dimensional porosity area fraction for each analyzed field. The same thresholding procedure was applied to all analyzed images to ensure consistency during pore segmentation and quantitative image analysis.

The porosity percentage was calculated according to Eq. (3.5):

$$\text{Porosity area fraction (\%)} = (A_{\text{pores}} / A_{\text{total}}) \times 100 \quad (3.5)$$

where A_{pores} is the total thresholded pore area and A_{total} is the total analyzed image area. Multiple images were analyzed for each selected specimen to improve the representativeness of the measurement. The obtained results were then compared with Archimedes density and XCT data to evaluate the consistency between two-dimensional and three-dimensional porosity assessment methods.

A representative thresholded image used for porosity quantification is shown in Fig. 35. In this image, the dark segmented regions correspond to pores or void-like features detected by the thresholding procedure. The ImageJ results were subsequently compared with Archimedes

density measurements and XCT analysis to provide complementary information on the porosity and internal defect state of the LPBF-fabricated Ti-5553 specimens.



Figure 35. Representative threshold image

3.4.7 Nanoindentation

Nanoindentation was performed on the polished cross-sections of representative LPBF-fabricated Ti-5553 specimens to evaluate the local mechanical response of the as-built material. The test was carried out using a Hysitron TI 950 TriboIndenter equipped with a Berkovich diamond indenter, as shown in Fig. 36. This technique enables localized measurement of hardness and elastic response at the micro- and nanoscale by continuously recording the applied load and penetration depth during each indentation cycle.

Before testing, the instrument was calibrated using a fused-quartz reference sample to verify the accuracy of the load and displacement measurements. The polished Ti-5553 surfaces were used for indentation to minimize the influence of surface roughness and preparation artifacts on the measured response.

Nanoindentation measurements were performed using a fixed displacement-depth range of 1000–2000 nm. This indentation depth range was selected to obtain a stable load–displacement response while avoiding excessive interaction with the surrounding mounted material or sample geometry. A minimum spacing of 7 μm was maintained between adjacent indents to reduce overlap between plastic deformation zones.

Load–displacement data were recorded automatically for each indentation. The hardness and reduced elastic modulus were calculated from the unloading portion of the indentation curve using the Oliver–Pharr method. Hardness was calculated using Eq. (3.6):

$$H = \frac{P_{max}}{A_c} \quad (3.6)$$

where H is the indentation hardness, Pmax is the maximum applied load, and Ac is the projected contact area.

For a Berkovich indenter, the projected contact area was calculated using Eq. (3.7):

$$A_c = 24.5Ah_{c2} \quad (3.7)$$

where hc is the contact depth.

The reduced modulus was calculated using Eq. (3.8):

$$E_r = \frac{\sqrt{\pi}}{2\beta} \times \frac{S}{\sqrt{A_c}} \quad (3.8)$$

where Er is the reduced elastic modulus, S = dP/dh is the contact stiffness obtained from the initial unloading slope, and $\beta = 1.034$ is a correction factor for the Berkovich indenter geometry.

The elastic modulus of the specimen was then related to the reduced modulus using Eq. (3.9):

$$\frac{1}{E_r} = \frac{1-\nu^2}{E} \times \frac{1-\nu_i^2}{E_i} \quad (3.9)$$

where E and ν are the elastic modulus and Poisson's ratio of the specimen, respectively, while $E_i = 1140$ GPa and $\nu_i = 0.07$ are the elastic modulus and Poisson's ratio of the diamond indenter.

The hardness-to-modulus ratio, H/Er, and the plastic deformation resistance parameter, H^3/Er^2 , were also calculated from the indentation results. The H/Er ratio was used as an indicator of elastic strain accommodation, while H^3/Er^2 was used as a comparative index related to resistance against plastic deformation and wear. The obtained nanoindentation data were used to assess local mechanical variability in the as-built LPBF Ti-5553 specimens and are presented in the Results and Discussion chapter.



Figure 36. Hysitron TI 950 TriboIndenter used for nanoindentation testing of polished LPBF Ti-5553 specimens.

3.4.8 Tensile testing and fracture-surface analysis

Mechanical testing was performed to evaluate the tensile response of the as-built LPBF-fabricated Ti-5553 specimens. Unlike process-optimization studies in which tensile specimens are selected from different porosity regimes, the specimens in this work were fabricated under a pre-optimized LPBF condition. Therefore, the purpose of tensile testing was to characterize the mechanical behavior of the optimized as-built Ti-5553 material and to relate the tensile response to microstructure, defect population, and fracture morphology.

Tensile specimens were prepared from the LPBF-fabricated material after printing and sectioning. The gauge section of each tensile specimen had nominal dimensions of 10 mm in length, 2.5 mm in width, and 3 mm in thickness. Tensile testing was performed at room temperature using an Instron 5982 universal testing system. The tests were conducted under a strain rate of $1 \times 10^{-3} \text{ s}^{-1}$. Multiple specimens were tested to improve repeatability and to evaluate the consistency of the tensile response.

After tensile testing, the fracture surfaces of the failed specimens were examined using SEM in secondary electron imaging mode. Fractographic analysis was performed to identify the dominant fracture features, including ductile dimples, brittle facets, pore-assisted crack initiation, and defect-controlled failure. The fracture observations were then correlated with the tensile response, XCT defect analysis, optical microscopy, and SEM microstructural observations to understand how the as-built LPBF microstructure and residual defects influenced mechanical performance.

This combined tensile and fractographic approach was used to establish the relationship between the optimized LPBF condition, internal defect population, local microstructure, and fracture mechanism of Ti-5553. Fractographic examination focused on identifying crack initiation sites, crack propagation paths, cleavage-like regions, ductile dimples, and other fracture-related features associated with the LPBF microstructure.

3.4.9 X-ray diffraction (XRD)

X-ray diffraction (XRD) was employed to characterize the phase constitution of the LPBF-fabricated Ti-5553 specimens in both the as-built and tensile-tested conditions. XRD is a well-established technique for identifying crystalline phases and providing information on the crystallographic structure of metallic materials. In addition to phase identification, diffraction analysis provides valuable information regarding lattice distortion, peak broadening, crystallite characteristics, and preferred crystallographic orientation, making it a useful complementary technique for microstructural characterization.

The diffraction patterns obtained in this study were analyzed to identify the dominant phases present in the LPBF-fabricated Ti-5553 alloy and to examine possible microstructural changes associated with plastic deformation. Particular attention was given to the characteristic β -phase reflections and to the possible presence of secondary phases, including α , α' , α'' , and ω . Furthermore, changes in diffraction peak broadening were evaluated to support the interpretation of lattice strain and dislocation accumulation following tensile deformation.

Although XRD provides valuable crystallographic information, its ability to detect minor phases is influenced by factors such as phase fraction, peak overlap, crystallite size, and instrumental resolution. Therefore, the XRD results were interpreted together with optical microscopy, SEM observations, and tensile testing to establish the relationship between phase constitution, microstructural evolution, and the mechanical behaviour of the LPBF-fabricated Ti-5553 alloy.

3.4.10 Electrochemical corrosion testing

Electrochemical corrosion testing was performed to evaluate the corrosion behaviour of the LPBF-fabricated Ti-5553 specimens in the as-built condition. The experiments were conducted in a 0.9 wt.% NaCl solution at room temperature under controlled laboratory conditions. The corrosion tests were designed to assess the electrochemical response of the alloy and the stability of the passive oxide film formed on the specimen surface.

Prior to testing, the specimen surfaces were cleaned to remove contaminants that could influence the electrochemical measurements. During the experiments, the electrochemical

response of the specimens was monitored under controlled conditions, and the obtained data were used to evaluate the corrosion behaviour of the LPBF-fabricated Ti-5553 alloy.

The corrosion performance was assessed using open-circuit potential (OCP), potentiodynamic polarization, and electrochemical impedance spectroscopy (EIS). The polarization measurements were used to determine the corrosion potential (E_{corr}) and corrosion current (I_{corr}), while the impedance measurements provided additional information regarding the protective characteristics of the passive surface film.

The electrochemical results were interpreted together with the density, porosity, microstructural observations, and mechanical characterization in order to establish the relationship between the LPBF processing condition, the resulting microstructure, and the corrosion behaviour of the as-built Ti-5553 specimens.

Chapter 4

Results and Discussion

4.1 Overview

This chapter presents the experimental results obtained from the characterization of laser powder bed fusion fabricated Ti-5Al-5Mo-5V-3Cr specimens. The results are presented in the same general order as the experimental methods described in Chapter 3. The chapter begins with powder-related analysis, followed by bulk characterization, including density, porosity, internal defects, microstructural observations, phase identification, mechanical response, local mechanical properties, and corrosion behaviour.

The main focus of the present stage of analysis is the evaluation of the as-built LPBF Ti-5553 specimens fabricated under the selected optimized processing condition. Density and porosity are first assessed using Archimedes measurements, X-ray computed tomography, and ImageJ analysis of unetched optical micrographs. These methods provide complementary information: Archimedes measurements describe the overall bulk density, XCT provides three-dimensional information about internal defects, and ImageJ analysis quantifies two-dimensional porosity on polished unetched cross-sections.

Optical microscopy is then used to examine the cross-sectional features of the printed specimens. In this work, unetched optical micrographs were used for porosity quantification because they provide a clearer contrast between pores and the metallic matrix without interference from etched microstructural features. Etched optical micrographs were used separately to reveal melt-pool boundaries and solidification-related features associated with LPBF processing.

The mechanical response of the as-built Ti-5553 specimens is evaluated through tensile testing and nanoindentation. Tensile testing provides the global mechanical behaviour, including stiffness, strength, and ductility, whereas nanoindentation provides information about local hardness and reduced elastic modulus. These results are interpreted together with the observed density, porosity, and optical microscopy results. The results are finally extended to corrosion testing in order to evaluate the electrochemical response and passive-film stability of the as-built LPBF Ti-5553 alloy in NaCl solution.

4.2 Powder Analysis

4.2.1 Powder Morphology

The morphology of the Ti-5553 powder feedstock was examined using scanning electron microscopy (SEM) at different magnifications, as presented in Fig. 37. The low-magnification image (Fig. 37a) shows that the powder consists predominantly of spherical and near-spherical particles with a relatively broad size distribution. Such morphology is characteristic of gas-atomized titanium powders and is generally considered beneficial for LPBF because the high degree of particle sphericity improves powder flowability, promotes uniform powder spreading, and contributes to consistent powder-bed packing during recoating.

At a higher magnification (Fig. 37b), the powder exhibits a relatively clean surface with no evidence of severe particle agglomeration. A limited number of fine particles are attached to the surfaces of larger particles, forming typical satellite particles generated during the atomization process. These satellites are commonly reported in Ti-5553 powders and, when present in limited amounts, have little influence on powder processability, although excessive satellite formation may increase interparticle friction and reduce flowability, although excessive satellite formation can increase interparticle friction and reduce powder flowability.. The high-magnification image (Fig. 37c) further confirms that most powder particles exhibit relatively smooth surfaces, although a limited number of satellite particles and a few irregular particles are also observed. Only a few irregularly shaped particles are observed, indicating that the atomization process produced powder with a high overall degree of sphericity. Similar powder morphologies have been reported by Bakhshivash et al., Hasanabadi et al., Ahmed et al., and Yasin et al., who described predominantly spherical Ti-5553 powders with minor satellite particles suitable for LPBF processing. Overall, the observed powder morphology indicates good powder quality and is consistent with the requirements for stable LPBF processing.

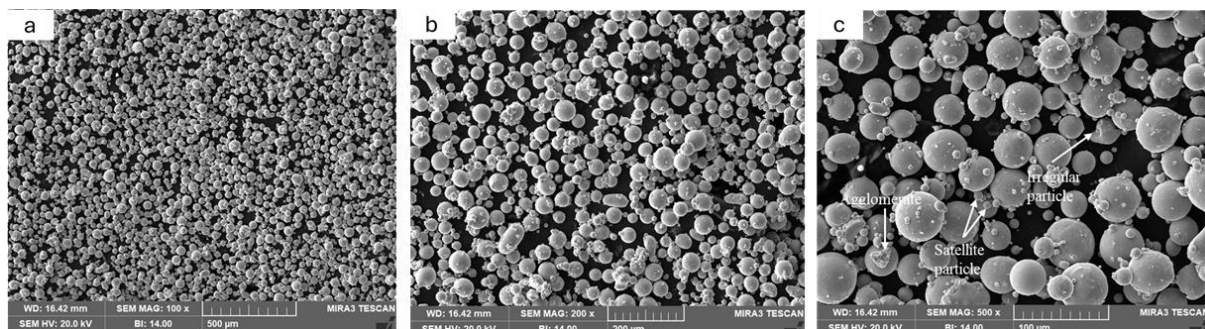


Figure 37. SEM images of the Ti-5553 powder feedstock at different magnifications.

4.2.2 Chemical Composition and Elemental Mapping

The chemical homogeneity and particle size distribution of the Ti-5553 powder feedstock were evaluated to assess its suitability for LPBF processing. The EDS elemental maps, shown in Fig. 38, indicate that Ti, Al, V, Mo, and Cr are uniformly distributed within the powder particles. No obvious elemental enrichment, segregation, or local clustering is observed within the spatial resolution of EDS. This confirms that the powder is chemically homogeneous at the particle scale and is consistent with a pre-alloyed Ti-5553 feedstock.

The homogeneous distribution of Mo, V, and Cr is particularly important because these elements are the main β -stabilizers in Ti-5553. Local enrichment or depletion of these elements could modify β -phase stability and influence melting, solidification, and phase evolution during LPBF. In the present powder, however, the absence of visible elemental clustering suggests that the alloying elements were sufficiently homogenized before processing.

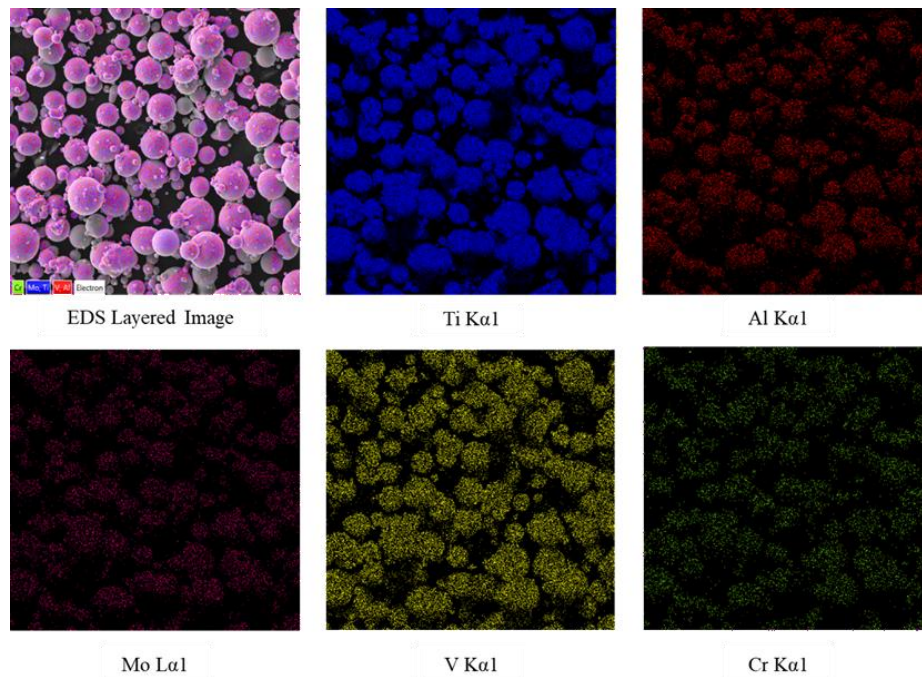


Figure 38. EDS elemental maps of the Ti-5553 powder feedstock

The quantitative EDS result is reported in Table 2. The minor Fe signal was excluded and the remaining elements were normalized to 100 wt.%. The measured composition was Ti = 83.80 wt.%, Al = 4.77 wt.%, V = 5.27 wt.%, Mo = 3.84 wt.%, and Cr = 2.32 wt.%. This composition is close to the nominal Ti-5553 chemistry and supports the use of this feedstock for LPBF fabrication.

Table 3. Normalized EDS chemical composition of the Ti-5553 powder feedstock

Element	Ti	Al	V	Mo	Cr	Total
Wt.%	83.80	4.77	5.27	3.84	2.32	100.00

4.2.3 Particle Size Distribution

The particle size distribution of the same powder is presented in Fig. 39 as relative and cumulative frequency curves. The measured percentile values were D10 = 23.0 μm , D50 = 38.7 μm , and D90 = 56.3 μm . The histogram shows a mainly unimodal distribution, with most particles concentrated approximately between 25 and 45 μm . Only a limited fraction of particles is present below 20 μm or above 60 μm , and the smooth cumulative curve indicates the absence of a clear secondary particle population.

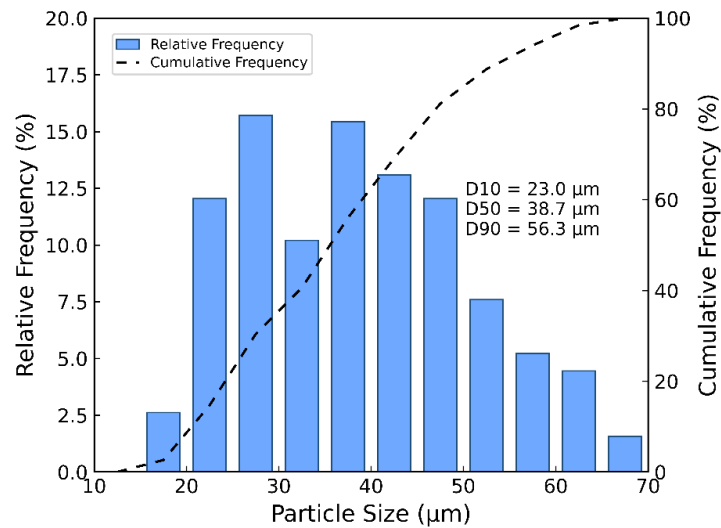


Figure 39. Particle size distribution of the Ti-5553 powder feedstock

From a processing perspective, this particle size distribution is suitable for LPBF. The median particle size of 38.7 μm is not excessively fine, which helps reduce the risk of strong interparticle cohesion and poor powder flowability. At the same time, the presence of smaller particles can contribute to powder-bed packing by filling interstitial spaces between larger particles. The limited fraction of coarse particles is also beneficial because very large particles may disturb layer spreading and increase the risk of local lack-of-fusion during laser exposure. Compared with reported LPBF Ti-5553 powders, the present feedstock is slightly coarser than the plasma-atomized powder used by Yasin et al., which had D10 = 21 μm , D50 = 33 μm , and D90 = 44 μm . However, the present powder still falls within the particle-size range commonly used for LPBF Ti-5553 processing[97]. Therefore, the measured PSD suggests a reasonable balance between powder spreading, packing density, and melt-pool stability.

Overall, the EDS mapping and particle size analysis show that the feedstock has suitable chemical and physical characteristics for LPBF. The uniform elemental distribution supports chemical consistency during melting and solidification, while the particle size distribution provides an appropriate balance between flowability and packing behavior. Together with the predominantly spherical morphology described in the previous section, these results indicate that the powder quality is appropriate for producing dense as-built LPBF Ti-5553 specimens.

4.3 Bulk Analysis

The bulk characterization of the LPBF-fabricated Ti-5553 specimens was performed to evaluate the consolidation quality, internal defect state, microstructural features, and mechanical response of the material produced under the selected processing condition. All specimens discussed in this section were fabricated using a fixed LPBF parameter set consisting of a laser power of 200 W, scan speed of 800 mm/s, hatch spacing of 0.115 mm, and layer thickness of 0.03 mm, corresponding to a volumetric energy density of 72.46 J/mm³. A stripe scanning strategy with a 67° rotation between successive layers was applied during fabrication.

The bulk analysis begins with density and porosity evaluation using Archimedes measurements, X-ray computed tomography, and optical image analysis. These methods were used in a complementary manner because each technique provides different information about the defect state of the material. Archimedes measurement provides an overall estimate of the apparent density and total porosity, XCT enables three-dimensional visualization and quantification of internal defects, and ImageJ analysis of unetched optical micrographs provides two-dimensional porosity information from polished cross-sections.

After density and defect evaluation, the microstructural features of the LPBF Ti-5553 specimens are examined. Optical microscopy is used to observe the polished and etched cross-sections, with unetched images used for porosity quantification and etched images used to reveal melt-pool boundaries and solidification-related features. The microstructural observations are then correlated with the density and defect results in order to evaluate the consolidation quality of the as-built LPBF Ti-5553 alloy.

The mechanical response of the as-built material is evaluated through tensile testing and nanoindentation. Tensile testing provides the global mechanical properties of the LPBF Ti-5553 specimens, whereas nanoindentation is used to assess the local hardness and reduced elastic modulus. Finally, the electrochemical behaviour of the as-built alloy is evaluated

through corrosion testing and discussed in relation to the density, porosity, and microstructural characteristics.

The density of the LPBF-fabricated Ti-5553 specimen was first evaluated using the Archimedes method in order to assess the overall consolidation quality of the printed material. The measurement was performed on the as-built cubic sample before any surface modification, so that the measured density could represent the original LPBF condition, including the contribution of both internal and surface-connected defects.

The specimen was fabricated using the selected LPBF processing condition, consisting of a laser power of 200 W, scan speed of 800 mm/s, hatch spacing of 0.115 mm, and layer thickness of 0.03 mm. These parameters correspond to a volumetric energy density of 72.46 J/mm³. The theoretical density used for Ti-5553 in this work was 4.65 g/cm³.

Based on the Archimedes measurement, the Ti-5553 specimen exhibited an apparent density of 4.624 g/cm³, corresponding to a relative Archimedes density of 99.45%. The calculated total porosity was 1.716%, consisting of 1.173% open porosity and 0.543% closed porosity. The geometrical density was 4.570 g/cm³, corresponding to a relative geometrical density of 98.28%. The density and porosity values obtained from the Archimedes measurement are summarized in Table 3.

Table 4. Archimedes density and porosity results of the LPBF-fabricated Ti-5553 specimen.

Sample	Theoretical density (g/cm ³)	Archimedes density (g/cm ³)	Geometrical density (g/cm ³)	Open porosity (%)	Closed porosity (%)	Total porosity (%)	Relative Archimedes density (%)
Ti-5553	4.65	4.624	4.570	1.173	0.543	1.716	99.45

The measured relative Archimedes density of 99.45% indicates that the selected LPBF processing condition achieved a high level of densification through effective melting and bonding between adjacent scan tracks and successive layers.

However, the presence of measurable open and closed porosity shows that residual defects were not completely eliminated. The open porosity contribution may be associated with surface-connected pores, near-surface defects, or roughness-related features that are typical of the as-built LPBF condition. In contrast, the closed porosity contribution may be related to isolated internal pores trapped within the bulk material during solidification.

The difference between the Archimedes density and the geometrical density can be attributed to the different nature of the two measurements. The Archimedes method estimates the

apparent density through buoyancy, while the geometrical density depends on the external dimensions and mass of the specimen. For as-built LPBF samples, surface roughness, adhered particles, and surface-connected pores can influence geometrical measurements and may lead to differences between these two density values.

Overall, the Archimedes results confirm that the as-built LPBF Ti-5553 specimen reached a high level of densification under the selected processing condition. Nevertheless, because the Archimedes method provides only an average bulk density value and does not reveal the morphology, size, or spatial distribution of defects, the porosity state was further examined using X-ray computed tomography and optical image analysis.

4.3.1 XCT Validation and Defect Morphology

X-ray computed tomography (XCT) was used as a complementary non-destructive technique to evaluate the internal defect population of the LPBF-fabricated Ti-5553 specimen. While the Archimedes method provides an average bulk density value, XCT enables three-dimensional visualization and quantification of the internal porosity distribution. Therefore, XCT analysis was performed to complement the Archimedes measurements by providing three-dimensional information on the morphology and spatial distribution of internal defects.

The XCT analysis was carried out using threshold-based porosity/inclusion analysis. In the report, the analyzed region is identified as Volume 1. The detected discontinuities were classified by the software as voids. The voxel count was 18,979, the material volume was 871.29 mm³, and the total defect volume was 0.10 mm³, corresponding to a defect volume ratio of 0.01%. These values indicate a very low internal defect content within the scanned volume.

Table 5. XCT parameters and porosity results of the LPBF-fabricated Ti-5553 specimen.

Parameter	Value
Scanned volume name	Volume 1
Voxel count	18,979
Material volume	871.29 mm ³
Defect volume	0.10 mm ³
Defect volume ratio	0.01%
Defect type considered by the software	Voids
Approximate defect diameter range	0.10–0.25 mm
Approximate sphericity range	0.42–0.64

The XCT-based defect characterization is shown in Fig. 40. The figure includes the defect diameter distribution, the sphericity–diameter relationship, the three-dimensional spatial distribution of defects, and magnified views of representative void morphologies.

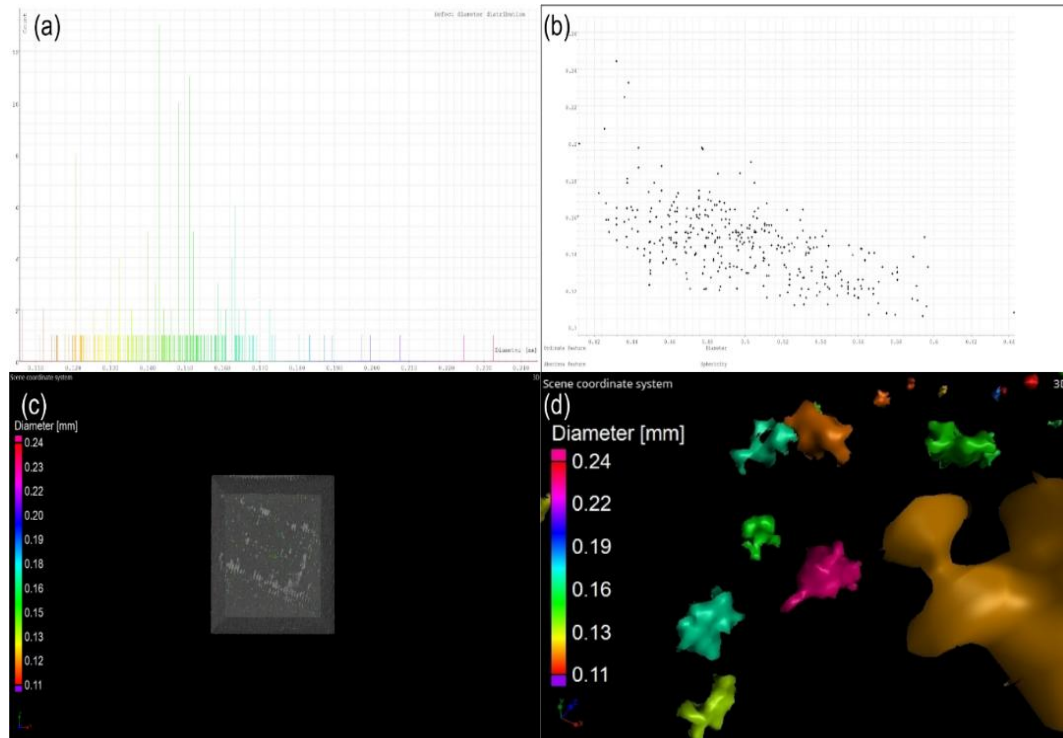


Figure 40. XCT analysis of the LPBF-fabricated Ti-5553 specimen showing (a) defect size distribution, (b) sphericity–diameter relationship, (c) three-dimensional defect distribution, and (d) reconstructed defect morphology.

The defect diameter distribution in Fig. 40(a) indicates that the detected voids were mainly within a relatively small size range, ranged from approximately 0.10 to 0.25 mm. Most of the detected defects were concentrated below approximately 0.17 mm, while only a limited number of larger defects approached the upper end of the measured diameter range. These results indicate that the internal defect population was dominated by small voids rather than large volumetric discontinuities.

The sphericity–diameter map in Fig. 40(b) shows that the detected defects were distributed over a sphericity range of approximately 0.42 to 0.64. Since a perfectly spherical pore would have a sphericity close to 1, these values indicate that the pores were not fully spherical. Instead, the detected defects showed moderately rounded to irregular morphologies. This suggests that the residual defect population cannot be attributed only to spherical gas porosity, and may also include irregular defects associated with local lack of fusion or incomplete track/layer consolidation.

The three-dimensional reconstruction in Fig. 40(c) shows that the residual defects were sparsely distributed throughout the scanned volume. No large interconnected porosity network was observed. This distribution is consistent with the very low defect volume ratio of 0.01% and supports the interpretation that the selected LPBF condition produced a highly consolidated Ti-5553 specimen.

The magnified defect morphology shown in Fig. 40(d) further confirms that several defects were irregular and non-spherical. These morphologies may be associated with local melt-pool instability, gas entrapment, or small residual discontinuities remaining after track and layer consolidation. However, because the analysis was performed using threshold-based segmentation, a definitive distinction between gas pores, lack-of-fusion defects, and surface-connected defects cannot be made only from the XCT report.

Although the XCT result indicates a lower porosity value than the Archimedes measurement, this difference is expected because the two techniques assess porosity in different ways. Archimedes measurement estimates the apparent density of the whole specimen and can be affected by open porosity, surface-connected defects, and surface roughness, whereas XCT detects segmented internal voids within the scanned volume according to the selected threshold and scan resolution. As a result, XCT is particularly effective for visualizing and quantifying internal defects, while Archimedes reflects the overall bulk densification behavior of the specimen. Accordingly, the two techniques provide complementary information regarding the overall densification and the internal defect state of the LPBF-fabricated Ti-5553 specimen.

4.3.2 ImageJ Porosity Analysis from Unetched Optical Micrographs

ImageJ analysis was performed on polished unetched optical micrographs to provide a two-dimensional estimation of the porosity fraction in the LPBF-fabricated Ti-5553 specimen. The unetched condition was selected for this analysis because it provides a clearer contrast between pores and the metallic matrix, while avoiding the additional contrast produced by etched melt-pool boundaries or microstructural features. This is important because etching may introduce contrast variations that can interfere with threshold-based pore segmentation and lead to inaccurate porosity quantification.

A representative unetched optical micrograph after ImageJ thresholding is shown in Fig. 41. The red regions correspond to the segmented pores used for calculating the two-dimensional pore area fraction.

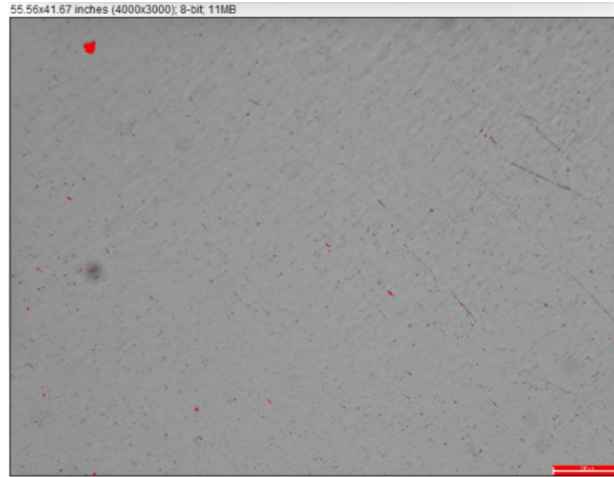


Figure 40. Representative unetched optical micrograph of the LPBF-fabricated Ti-5553 specimen after ImageJ thresholding. The red regions indicate segmented pores used for two-dimensional pore area-fraction quantification.

Five representative optical micrographs were analyzed, and the measured porosity values are summarized in Table 5.

Table 6. ImageJ-based porosity results obtained from unetched optical micrographs of the LPBF-fabricated Ti-5553 specimen.

Image	Region / file label	Porosity area fraction (%)
1	Region 1	0.311
2	Region 2	0.332
3	Region 3	0.228
4	Region 4	0.312
5	Region 5	0.320
Mean $\pm$ SD	—	0.301 ± 0.041

As shown in Fig. 41, the segmented pores are limited in size and are sparsely distributed within the analyzed field of view. No large continuous defect or extended lack-of-fusion region is visible in this representative image. This observation is consistent with the low ImageJ porosity values measured from the unetched optical micrographs.

The ImageJ results show that the measured two-dimensional porosity fraction was low for all analyzed regions, ranging from 0.228% to 0.332%. The average porosity was $0.301 \pm 0.041\%$, indicating a limited visible pore area on the polished cross-sections. The small scatter among the analyzed images suggests that the visible porosity was relatively uniform within the selected fields of view, although local variations were still present.

The ImageJ porosity value is higher than the XCT defect volume ratio but lower than the total porosity estimated from the Archimedes-based calculation. This difference is expected because the three techniques measure porosity using different principles. ImageJ analysis provides a local two-dimensional area fraction from selected polished cross-sections, XCT provides a three-dimensional internal defect volume based on threshold segmentation, and Archimedes measurement reflects the overall apparent density of the specimen, including possible effects of surface-connected porosity and surface roughness.

Therefore, the ImageJ results should not be interpreted as a direct replacement for either the Archimedes or XCT measurements. Instead, they provide complementary evidence that the LPBF-fabricated Ti-5553 specimen contains only a limited amount of visible porosity within the analyzed cross-sections, supporting the overall assessment of a highly consolidated as-built material.

4.3.3 Microstructure Analysis

4.3.3.1 OM & SEM

Representative optical and SEM micrographs of the LPBF-fabricated Ti-5553 specimen are presented in Fig. 42. The central image shows the cubic specimen used for metallographic characterization, while Figs. 42a and 42b show etched optical micrographs acquired from two different observation sections. Figures 42c and 42d present SEM micrographs used to examine the melt-pool-related features, layer-boundary features, grain boundaries, and local defect-like regions at higher magnification.

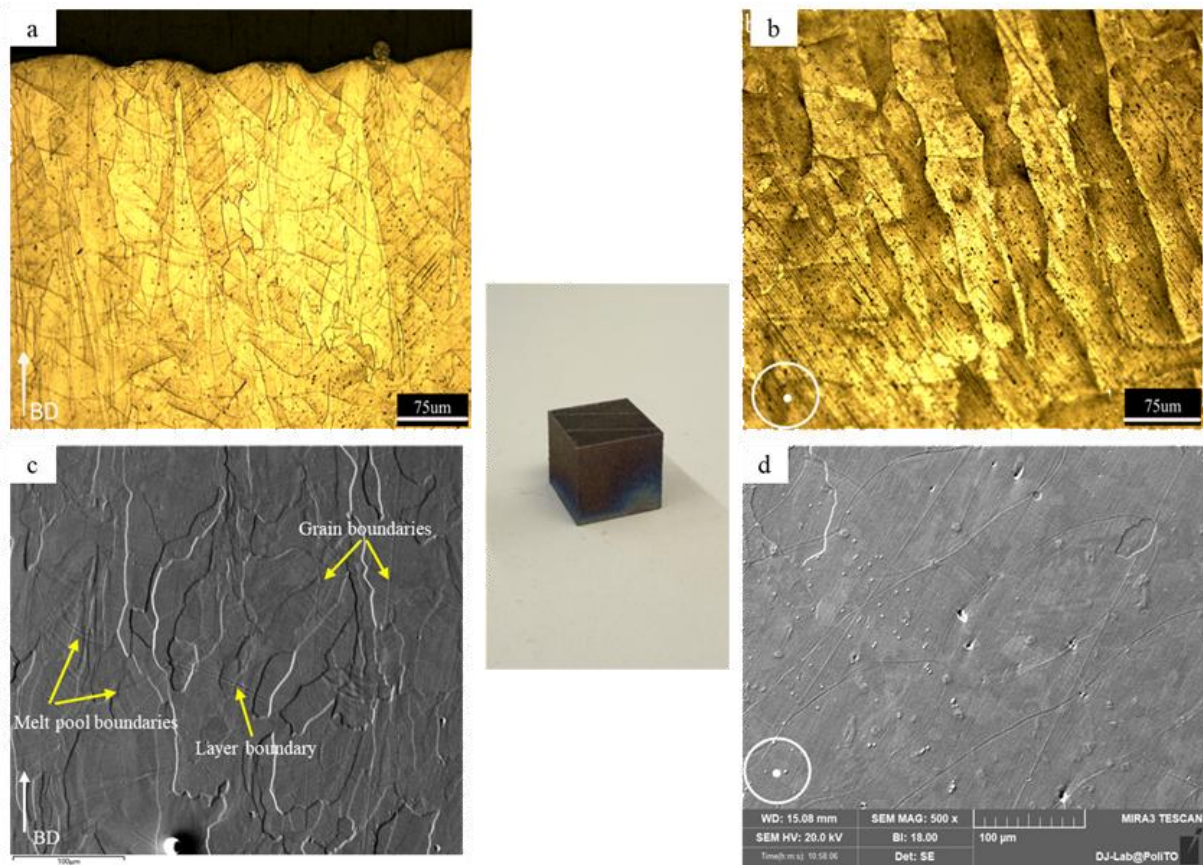


Figure 42. Optical and SEM micrographs of the LPBF-fabricated Ti-5553 specimen. The central image shows the cubic sample used for metallographic characterization. (a,b) Etched optical micrographs from the BD-parallel section and top section, respectively. (c,d) SEM micrographs showing melt-pool boundaries, layer-boundary features, grain-boundary traces, and selected local defect-like regions. BD denotes the build direction.

The etched cross-section parallel to the build direction, Fig. 42a, reveals a direction-dependent microstructure characteristic of LPBF processing. The microstructure contains elongated etched features extending preferentially along the BD, together with curved traces that are consistent with overlapping melt-pool boundaries generated by successive layer-wise melting and solidification. Within the examined field of view, no large lack-of-fusion defect or coarse pore is observed. However, small dark etched regions are present locally, which may correspond to etching response, small surface pits, or small defect-like features; therefore, they are not assigned to a specific defect type without further confirmation.

The top-section optical micrograph, Fig. 42b, exhibits a different etched appearance. In this observation plane, the melt-pool morphology is not expressed as continuous arc-shaped bands as clearly as in the BD-parallel section. Instead, elongated regions with different etching contrast are observed. This contrast variation is attributed to the sectioning direction relative to the three-dimensional solidification structure and to orientation-dependent etching of the prior-

β microstructure. Similar build-direction-dependent features, including columnar β grains, melt-pool boundaries, and layer boundaries, have been reported for SLM-fabricated Ti-5553 by Zafari et al[114].

The SEM micrograph in Fig. 42c further confirms the presence of characteristic LPBF solidification features. Melt-pool boundaries, a layer-boundary feature, and grain-boundary traces are visible after etching. These features are consistent with the repeated remelting and thermal cycling inherent to the LPBF process. The morphology is comparable to previous SEM observations of as-SLM Ti-5553, where grain boundaries, melt-pool boundaries, layer boundaries, and fine internal features were identified in the as-built microstructure.

Figure 42d shows a second SEM image from a different region. The image contains etched linear features and several small local discontinuities or pit-like regions. The white circles in Figs. 42b and 42d highlight representative local defect-like or strongly etched regions. Since these features are identified only from two-dimensional optical/SEM observations, they are not classified here as a specific defect type, such as lack-of-fusion or gas porosity. More definitive identification would require complementary evidence, such as higher-magnification SEM, cross-sectional serial imaging, or X-ray computed tomography.

Overall, the optical and SEM observations indicate that the as-built LPBF Ti-5553 specimen exhibits a direction-dependent microstructural appearance. Melt-pool-related and layer-boundary features are more clearly resolved in the BD-parallel section, whereas the top section shows a more orientation-dependent etched contrast. These observations provide the microstructural basis for discussing the anisotropic nature of the as-built material and for interpreting subsequent mechanical or post-processing results.

4.3.4 Tensile Response

4.3.4.1 Engineering stress–strain behavior

Uniaxial tensile testing was performed on three LPBF-fabricated Ti-5553 specimens in order to evaluate the global mechanical response of the material produced under the selected processing condition. The engineering stress–strain curves of the three repeated tests are shown in Fig. 43.

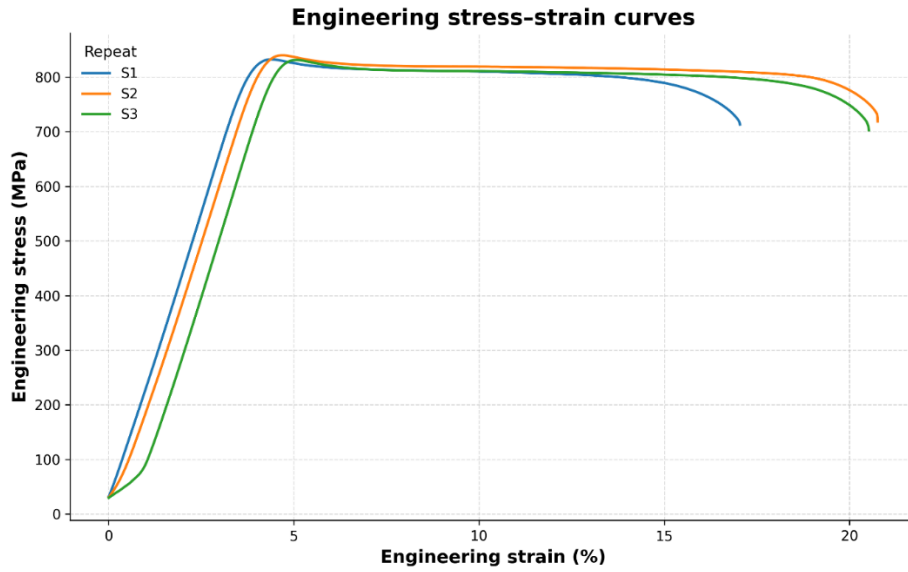


Figure 41. Engineering stress–strain curves of the LPBF-fabricated Ti-5553 specimens obtained from three repeated tensile tests.

The three stress–strain curves show a consistent mechanical response, indicating good repeatability among the tested specimens. All specimens exhibited a rapid increase in engineering stress during the elastic region, followed by yielding and plastic deformation. After reaching the maximum stress, the curves showed a gradual decrease in engineering stress with increasing strain until final fracture. This post-yield softening behavior is consistent with the tensile response commonly reported for metastable β titanium alloys and LPBF-fabricated Ti-5553.

The tensile properties extracted from the stress–strain curves are summarized in Table 6. In addition, the individual and average values of ultimate tensile strength, fracture strain, and toughness are shown in Fig. 44.

Table 7. Tensile properties of the LPBF-fabricated Ti-5553 specimens.

Specimen	UTS (MPa)	Fracture strain (%)	Toughness (MJ/m ³)
S1	832.5	17.1	122.5
S2	840.1	20.8	152.2
S3	831.3	20.5	145.1
Mean $\pm$ SD	834.6 $\pm$ 4.7	19.45 $\pm$ 2.08	139.96 $\pm$ 15.49

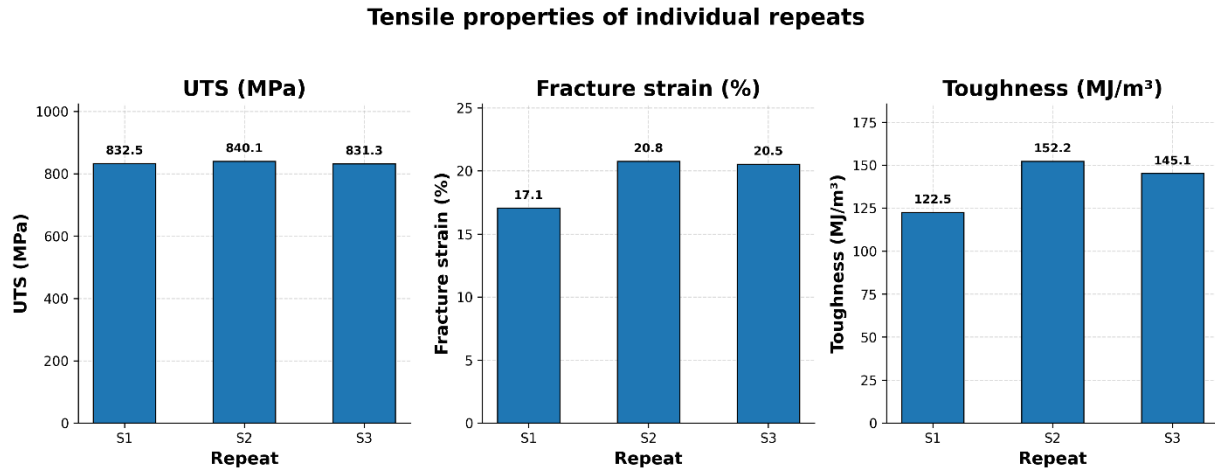


Figure 42. Tensile properties of the LPBF-fabricated Ti-5553 specimens: (a) ultimate tensile strength, (b) fracture strain, and (c) toughness obtained from three repeated tensile tests.

The UTS values were 832.5, 840.1, and 831.3 MPa for S1, S2, and S3, respectively, giving an average UTS of 834.6 ± 4.7 MPa. This suggests a relatively uniform load-bearing response among the tested specimens, consistent with the high relative density and low internal defect content. The fracture strain values were 17.1%, 20.8%, and 20.5% for S1, S2, and S3, respectively, giving an average fracture strain of $19.45 \pm 2.08\%$. Compared with UTS, the fracture strain showed greater scatter, mainly due to the lower ductility of S1. This behavior is reasonable for LPBF materials, where fracture strain is usually more sensitive than tensile strength to local defects, surface condition, and fracture-initiation sites. Since fractographic analysis is not yet available, the reason for the lower fracture strain of S1 cannot be confirmed at this stage.

The calculated toughness values, obtained from the area under the engineering stress–strain curves, were 122.5, 152.2, and 145.1 MJ/m³ for S1, S2, and S3, respectively. The average toughness was 139.96 ± 15.49 MJ/m³. The lower toughness of S1 is primarily related to its lower fracture strain, since the UTS values were very similar among the three specimens.

The average UTS obtained in this work is comparable with values reported for LPBF-fabricated Ti-5553 in the literature. Ramachandiran et al. reported UTS values of 780 ± 10 MPa for vertically built specimens and 846 ± 6 MPa for horizontally built specimens, with fracture strains of $23 \pm 3\%$ and $21 \pm 2\%$, respectively. The present UTS value of 834.6 ± 4.7 MPa is therefore within the expected range for as-built LPBF Ti-5553 and is close to the higher-strength values reported for horizontally oriented specimens. The average fracture strain of $19.45 \pm 2.08\%$ is slightly lower than some reported values but remains within a reasonable ductility range for as-built LPBF Ti-5553.

Overall, the tensile results show that the LPBF-fabricated Ti-5553 specimens exhibit a good combination of strength and ductility in the as-built condition. The high repeatability of UTS, together with the low porosity measured by XCT and ImageJ, indicates that the selected processing condition produced a repeatable tensile response under the tested condition. However, the scatter in fracture strain suggests that failure may still be influenced by local defect distribution, surface condition, or microstructural heterogeneity. This interpretation is further examined through the SEM fractography analysis presented in the following section.

4.3.4.2 Microstructural observations of the tensile fracture region

To clarify the deformation mechanisms responsible for the tensile response, the region adjacent to the fracture surface was examined using optical microscopy and SEM. Figure 45 presents the microstructural characteristics of the LPBF-fabricated Ti-5553 tensile specimen in the vicinity of the fracture region after tensile testing. The macroscopic image identifies the location selected for microstructural examination, while the corresponding optical and SEM images provide detailed observations of the deformation features developed during tensile loading.

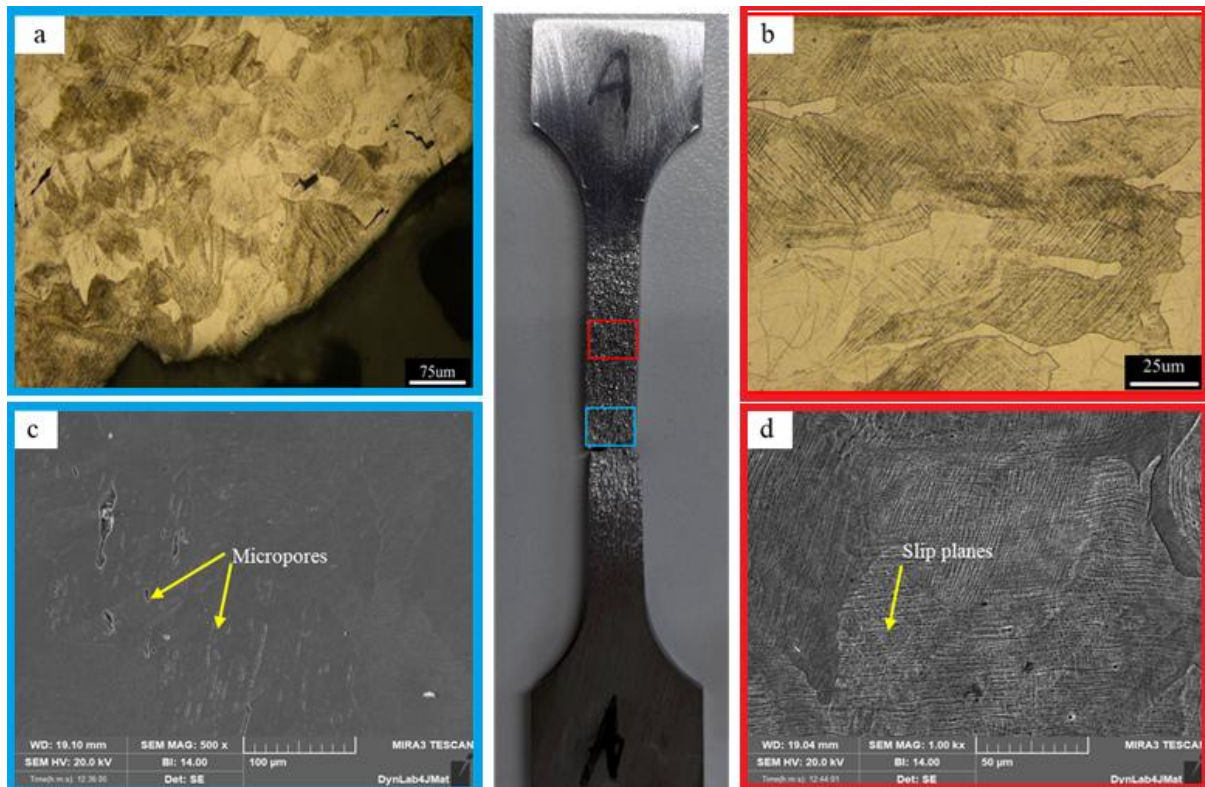


Figure 45. Microstructural observations adjacent to the tensile fracture region of the LPBF Ti-5553 specimen after tensile testing: macroscopic view of the fractured specimen indicating the examined region, (a,b) optical micrographs, and (c,d) SEM images showing micropores and slip planes.

The optical micrographs shown in Figures 45a and 45b reveal that the prior β grain morphology remains clearly distinguishable after tensile deformation. Numerous parallel slip traces are observed within individual grains, whereas their orientation varies from grain to grain according to the crystallographic orientation. The grain boundaries remain well defined, and no pronounced microcracks or severe microstructural discontinuities are observed within the examined region. These observations indicate that plastic deformation was primarily accommodated within the β grains prior to final fracture.

The SEM image presented in Figure 45c reveals the presence of several isolated micropores distributed within the deformed region. These micropores are relatively small and remain spatially separated without forming an interconnected damage network. Their appearance may be associated with localized strain accumulation during tensile deformation and early-stage microstructural damage development.

A higher-magnification SEM image of the same region is shown in Figure 45d. Numerous parallel slip planes are clearly visible within the β grains. These slip planes are confined within individual grains and terminate at grain boundaries, indicating that plastic deformation was mainly accommodated through crystallographic slip. The high density and regular distribution of these features further support the role of crystallographic slip during tensile loading. Although localized deformation is evident, no continuous crack propagation is observed within the examined area.

The deformation characteristics observed in the present study are in good agreement with previous investigations on LPBF-fabricated Ti-5553 alloys. Ramachandiran et al. reported that tensile deformation is initially accommodated through the formation of slip planes, followed by the appearance of aligned micropores that contribute to crack initiation. Similarly, Karimi et al. demonstrated that increasing deformation promotes the development of dense parallel slip bands within the β matrix, reflecting progressive strain localization prior to fracture. Although interrupted tensile testing was not performed in the present work, the simultaneous observation of slip planes and isolated micropores suggests that a similar deformation mechanism may have contributed to the early stages of damage development in the present LPBF Ti-5553 specimen.

Collectively, these observations indicate that tensile deformation in the LPBF Ti-5553 specimen was primarily accommodated through crystallographic slip within the β grains. The presence of isolated micropores further suggests localized damage development during deformation. These microstructural features establish a clear link between the deformation

mechanisms operating within the material and the mixed-mode fracture morphology observed on the final fracture surface, which is discussed in the following section.

4.3.4.3 Fractography and failure mechanism

Figure 46 presents the fracture morphology of the LPBF-fabricated Ti-5553 tensile specimen at different magnifications. The optical micrographs shown in Figures 46a and 46b illustrate the region adjacent to the fracture surface. The examined cross-section suggests that crack propagation likely originated near the specimen surface and subsequently progressed toward the interior of the material. Within the examined region, no dominant fracture-initiating defect, such as a large lack-of-fusion defect or coarse pore, was observed, suggesting that crack propagation primarily occurred through the existing microstructure.

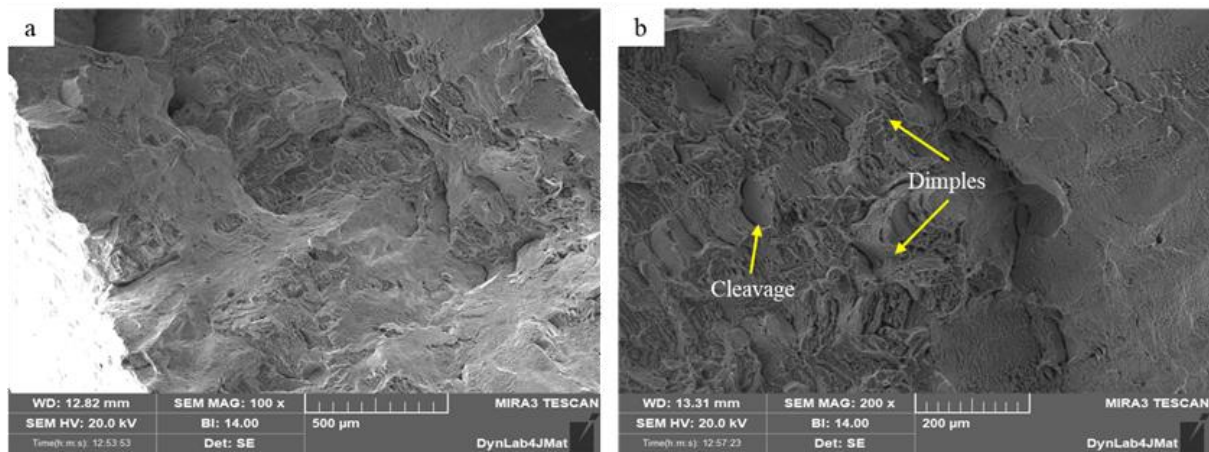


Figure 46. Fractographic characterization of the tensile-fractured LPBF Ti-5553 specimen: (a,b) optical micrographs of the fracture initiation region and (c,d) SEM images of the fracture surface revealing cleavage and dimpled fracture features.

The overall fracture morphology is shown in Figure 46c. At low magnification, the fracture surface exhibits a heterogeneous topography composed of regions with different surface characteristics. Relatively smooth areas coexist with rougher regions, while local fracture steps and noticeable height variations are evident across the fracture surface. These features indicate that crack propagation did not proceed through a single fracture mechanism but involved different fracture processes during tensile loading.

A higher-magnification SEM image of the selected fracture region is presented in Figure 46d. The fracture surface consists of relatively flat cleavage regions together with rougher areas containing numerous dimples. In addition, transition regions between cleavage and dimpled areas are clearly visible, where local ridges and surface steps are observed between adjacent fracture features. The simultaneous presence of these fracture characteristics suggests that

crack propagation alternated between localized brittle fracture and ductile deformation during tensile loading.

Based on these observations, the fracture surface can be classified as a mixed-mode fracture, characterized by the coexistence of cleavage and dimpled regions. The cleavage regions are indicative of localized brittle crack propagation, whereas the dimples reflect localized plastic deformation associated with microvoid coalescence prior to final failure. Therefore, the fracture surface exhibits neither a completely brittle nor a fully ductile morphology, but rather a combination of both fracture mechanisms.

The fracture characteristics observed in the present study are consistent with previous investigations on LPBF-fabricated Ti-5553 alloys. Yasin et al. reported the coexistence of cleavage and dimples on the tensile fracture surface and described the fracture behavior as a mixed fracture mode. Similarly, Ramachandiran et al. identified ductile microvoid coalescence together with brittle fracture regions and referred to the fracture behavior as a bi-modal fracture. Benni et al. also observed fracture surfaces predominantly composed of shallow cleavage accompanied by limited dimpled areas, concluding that tensile failure occurred through a mixed failure mode dominated by brittle fracture. The fracture morphology observed in the present work closely resembles these previously reported features, particularly the simultaneous presence of cleavage and dimples, supporting the interpretation that mixed-mode tensile fracture is a characteristic fracture behavior of LPBF-produced Ti-5553 alloys.

Overall, the fractographic observations demonstrate that tensile failure of the present LPBF Ti-5553 specimen resulted from the combined contribution of brittle and ductile fracture mechanisms. When considered together with the slip traces and isolated micropores observed near the tensile fracture region, the fracture morphology suggests progressive damage evolution involving localized crystallographic slip, localized micropore formation, and final mixed-mode fracture characterized by cleavage and dimpled regions.

4.3.5 X-ray diffraction analysis

X-ray diffraction (XRD) was performed to examine the phase constitution of the LPBF-fabricated Ti-5553 alloy before and after tensile deformation. The diffraction patterns obtained from the as-built and tensile-tested specimens are presented in Fig. 47. In both conditions, the diffraction patterns are dominated by the characteristic β -phase reflections corresponding to the $\beta(110)$, $\beta(200)$, and $\beta(211)$ crystallographic planes. No additional diffraction peaks that could be confidently assigned to α , α' , α'' , or ω phases were observed within the detection capability of the present laboratory XRD system. Therefore, the results indicate that the

investigated material retained a predominantly β -phase crystal structure after tensile deformation.

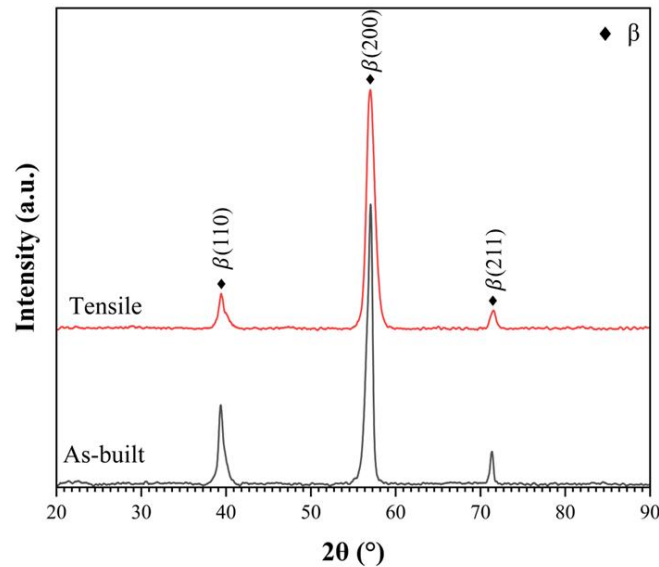


Figure 47. XRD patterns of the LPBF-fabricated Ti-5553 alloy in the as-built and tensile-tested conditions.

Although the phase constitution remained essentially unchanged, a comparison of the diffraction profiles reveals a noticeable broadening of the β reflections after tensile testing, particularly for the $\beta(110)$, $\beta(200)$, and $\beta(211)$ peaks. In contrast, no significant peak splitting or measurable peak shift was observed. This indicates that the applied tensile loading did not induce a detectable phase transformation but instead altered the defect state within the retained β matrix.

The absence of deformation-induced diffraction peaks is consistent with several recent investigations on LPBF Ti-5553. Ahmed et al. reported a single β -phase diffraction pattern for as-built LPBF Ti-5553 produced under different processing parameters, while Karimi et al. similarly observed no evidence of stress-induced phase transformation after dynamic deformation within the resolution limits of XRD, SEM, and EBSD, suggesting that plastic deformation was accommodated while maintaining β -phase stability. Although small amounts of α or α'' have been reported in some LPBF Ti-5553 studies under different thermal histories or processing conditions, these phases were not detected in the present material, indicating that the selected processing condition produced a predominantly metastable β microstructure.

To further evaluate the influence of tensile deformation on the crystal lattice, the average peak broadening parameters were quantified, and the apparent crystallite size, microstrain, and

dislocation density were estimated from the XRD peak broadening analysis. The results are summarized in Table 7.

Table 7. Average XRD peak broadening parameters of the LPBF-fabricated Ti-5553 alloy before and after tensile deformation.

Sample	Avg. FWHM (°)	Apparent crystallite size (nm)	Microstrain	Dislocation density (m^{-2})
As-built	0.634	15.54	5.57×10^{-3}	5.38×10^{15}
Tensile	0.854	10.97	7.20×10^{-3}	9.06×10^{15}

Following tensile deformation, the average FWHM increased from 0.634° to 0.854° , while the apparent crystallite size decreased from 15.54 nm to 10.97 nm. At the same time, the estimated microstrain increased from 5.57×10^{-3} to 7.20×10^{-3} , accompanied by an increase in the calculated dislocation density from 5.38×10^{15} to $9.06 \times 10^{15} \text{ m}^{-2}$. These changes are consistent with an increase in lattice distortion following plastic deformation. It should be noted that the crystallite size, microstrain, and dislocation density were estimated from XRD line broadening and therefore represent apparent values rather than direct measurements of the physical grain size or absolute dislocation density.

The XRD observations are also consistent with the mechanical response presented in Section 4.3.4. The tensile-tested specimens exhibited stable elastic-plastic deformation together with an average ultimate tensile strength of $834.6 \pm 4.7 \text{ MPa}$ and an elongation of $19.45 \pm 2.08\%$, indicating that considerable plastic strain was accommodated before final fracture. The increase in XRD peak broadening after tensile testing provides additional evidence that this deformation was primarily associated with the accumulation of crystallographic defects and lattice distortion within the retained β matrix rather than with a detectable transformation to a new crystalline phase. This interpretation is further supported by the fractographic observations presented in the previous section, where mixed-mode fracture together with localized slip traces indicated progressive plastic deformation prior to final failure.

Overall, the XRD analysis demonstrates that the optimized LPBF Ti-5553 alloy maintained a predominantly β -phase crystal structure after tensile loading. The principal effect of deformation was the development of additional lattice distortion, reflected by increased diffraction peak broadening, higher apparent microstrain, and higher calculated dislocation density, while no detectable phase transformation was identified under the present experimental conditions.

4.3.6 Nanoindentation Response

Nanoindentation was performed to evaluate the local mechanical response of the LPBF-fabricated Ti-5553 specimens. Three different conditions were examined: the top surface of the printed specimen, the cross-section along the building direction (BD), and a tensile-tested specimen after fracture, mounting, grinding, and polishing. Therefore, the tensile-tested condition represents a mechanically deformed and fractured specimen rather than the original as-built state.

Representative load–displacement curves obtained from the three conditions are shown in Fig. 48. All curves reached a maximum load of approximately 7000 μN . The loading portions of the curves show similar overall behavior, indicating comparable resistance to indentation among the examined conditions. However, differences in penetration depth and unloading slope can be observed. These differences may reflect variations in local hardness, elastic recovery, indentation position, and specimen condition.

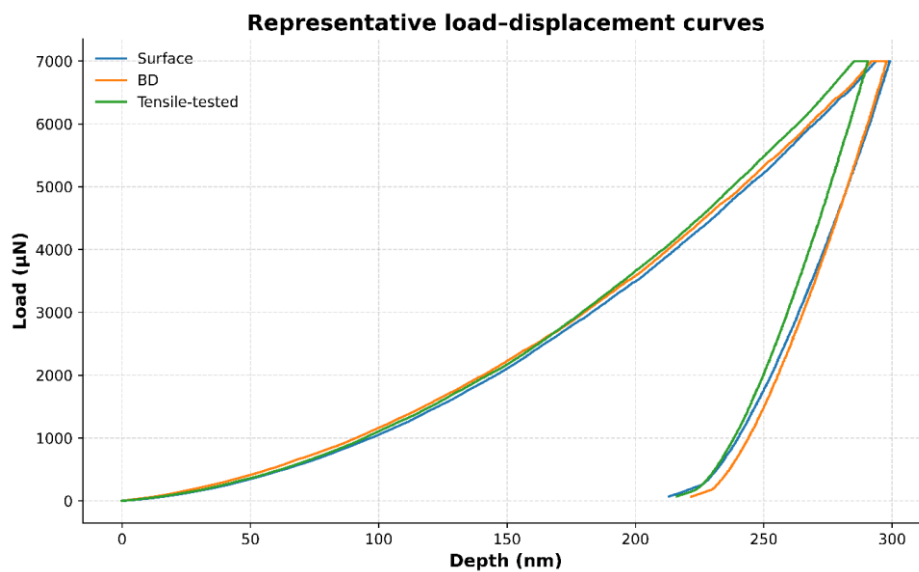


Figure 43. Representative nanoindentation load–displacement curves obtained from the surface, BD, and tensile-tested conditions of LPBF-fabricated Ti-5553.

The mean hardness and reduced modulus values are shown in Fig. 49 and summarized in Table 8. The surface condition showed the highest hardness, with an average value of 4.40 ± 0.16 GPa, while the BD condition showed a slightly lower hardness of 4.22 ± 0.11 GPa. The tensile-tested specimen exhibited the lowest average hardness, 4.15 ± 0.58 GPa, and the highest scatter. The larger scatter in the tensile-tested condition is reasonable because this specimen had already undergone plastic deformation and fracture before metallographic preparation and nanoindentation testing.

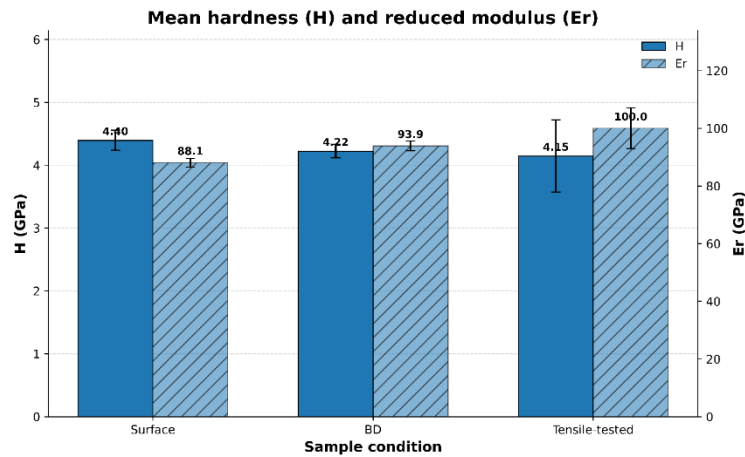


Figure 44. Mean hardness and reduced modulus values obtained from nanoindentation measurements of the surface, BD, and tensile-tested conditions.

Table 8. Nanoindentation results of LPBF-fabricated Ti-5553 under different sample conditions.

Condition	Number of indents	H (GPa)	Er (GPa)	H/Er	H ³ /Er ²
Surface	9	4.40 ± 0.16	88.1 ± 1.5	0.0499 ± 0.0016	0.0110 ± 0.0011
BD	9	4.22 ± 0.11	93.9 ± 1.7	0.0450 ± 0.0014	0.0086 ± 0.0007
Tensile-tested	5	4.15 ± 0.58	100.0 ± 7.0	0.0413 ± 0.0032	0.0072 ± 0.0020

The reduced modulus followed an opposite trend compared with hardness. The surface condition showed the lowest reduced modulus, 88.1 ± 1.5 GPa, while the BD condition showed a higher value of 93.9 ± 1.7 GPa. The tensile-tested specimen showed the highest reduced modulus, 100.0 ± 7.0 GPa, together with the largest scatter. This variation may be associated with differences in local microstructural orientation, indentation position, residual deformation, and the heterogeneous state of the tensile-tested specimen after fracture.

The ratios H/Er and H^3/Er^2 were also calculated as nanoindentation-based surrogate indicators related to elastic strain tolerance and resistance to plastic deformation, as shown in Fig. 50. The surface condition showed the highest H/Er value of 0.0499 ± 0.0016 and the highest H^3/Er^2 value of 0.0110 ± 0.0011 . The BD condition showed intermediate values, while the tensile-tested specimen showed the lowest H/Er and H^3/Er^2 values. This trend indicates that the surface condition exhibited the highest indentation-based surrogate indices, whereas the tensile-tested specimen showed a reduced local resistance to plastic deformation. However, these ratios should not be interpreted as direct wear resistance values without complementary tribological testing.

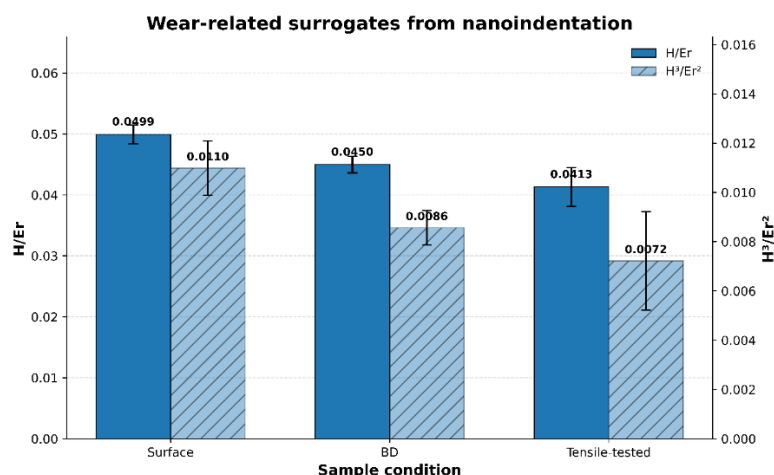


Figure 50. Nanoindentation-based H/Er and H³/Er² ratios for the surface, BD, and tensile-tested conditions.

The lower H/Er and H³/Er² values in the tensile-tested condition should be interpreted carefully. Since this specimen was tested after tensile deformation and fracture, the measured response may be influenced by local plastic deformation, damage accumulation, deformation-induced heterogeneity, and preparation-related position effects. Therefore, these values should not be directly compared with the undeformed as-built surface and BD conditions as a simple anisotropy measurement. Instead, they provide local information about the mechanically affected region after tensile testing.

Overall, the nanoindentation results show that the LPBF-fabricated Ti-5553 specimen exhibits local hardness values in the range of approximately 4.1–4.4 GPa, with reduced modulus values between approximately 88 and 100 GPa. The surface and BD conditions show relatively consistent responses, while the tensile-tested specimen shows greater scatter due to its prior deformation and fracture history. These results complement the tensile testing data by providing local mechanical information and should be interpreted together with the SEM, XRD, and fractography results presented in the previous sections.

4.3.7 Corrosion test

The corrosion behaviour of the as-built LPBF Ti-5553 alloy was evaluated by potentiodynamic polarization and electrochemical impedance spectroscopy (EIS) in 0.9 wt.% NaCl solution (pH = 5) at room temperature. The exposed surface area was 28 mm². The corresponding polarization curve together with the Nyquist and Bode plots are presented in Figure 51. The measured corrosion potential (E_{corr}) and corrosion current (I_{corr}) were -0.2203V and $7.45 \times 10^{-8}\text{ A}$, respectively, indicating a relatively low corrosion rate under the present testing conditions.

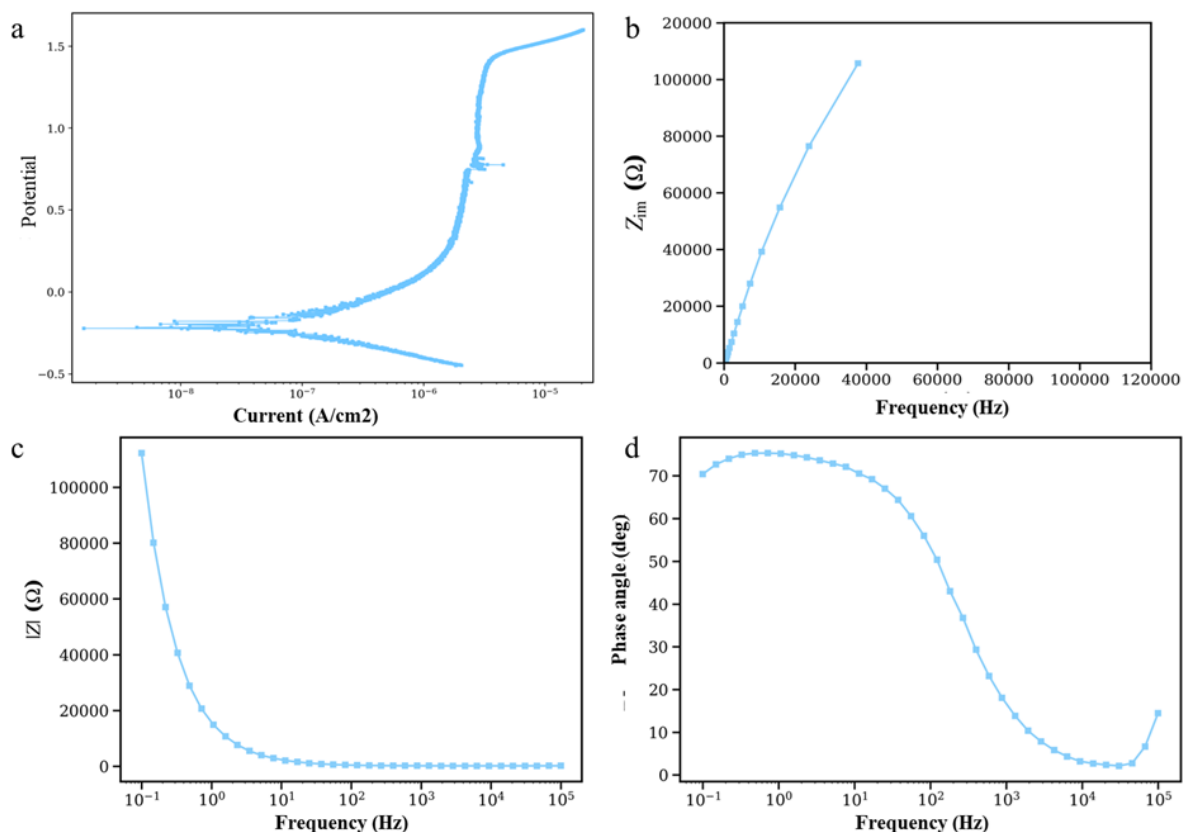


Figure 51. Electrochemical response of the as-built LPBF Ti-5553 alloy in 0.9 wt.% NaCl solution (pH = 5) at room temperature: (a) potentiodynamic polarization curve, (b) Nyquist plot, (c) Bode impedance modulus, and (d) Bode phase-angle plot.

As shown in Figure 51(a), the polarization curve exhibits the characteristic passive behaviour of titanium alloys. After reaching the corrosion potential, the anodic branch increases gradually before entering a passive region, indicating the rapid formation of a protective oxide film on the alloy surface. The relatively low corrosion current suggests that this passive layer effectively suppresses anodic dissolution despite the specimen being tested in the as-built condition. At higher anodic potentials, the current density increases more rapidly, which is most likely associated with the onset of passive-film instability or transpassive oxidation rather than continuous active dissolution. A comparable electrochemical response has been reported for LPBF Ti-5553, where the corrosion behaviour was shown to be primarily controlled by the integrity and compactness of the naturally formed TiO_2 passive layer.

The impedance response presented in Figure 51(b–d) is consistent with the polarization measurements. The impedance modulus increases markedly with decreasing frequency and reaches the order of $10^5 \Omega$ in the low-frequency region, indicating a high resistance of the oxide-covered surface to electrochemical charge transfer. Moreover, the Bode phase-angle plot exhibits a broad maximum of approximately 70–75°, reflecting a predominantly capacitive

electrochemical response that is characteristic of a stable passive oxide film. The presence of a single broad phase-angle maximum further suggests that the corrosion behaviour is mainly governed by one dominant electrochemical process associated with the oxide film/electrolyte interface.

Although the present specimen was examined in the as-built condition, its electrochemical response demonstrates that the passive film remained sufficiently protective throughout the measurement. Nevertheless, the as-built surface produced by LPBF generally contains partially melted particles, surface roughness and process-induced irregularities, which may locally reduce the uniformity of the passive layer. Dammulla et al. reported that mechanical surface finishing significantly improves the corrosion performance of LPBF Ti-5553 by reducing surface roughness and promoting the formation of a denser and less defective TiO_2 film, resulting in a more noble corrosion potential and substantially higher polarization resistance. Therefore, the corrosion resistance observed in the present work is considered to originate primarily from the spontaneous formation of the passive oxide film, while its overall effectiveness is likely limited by the intrinsic surface condition associated with the as-built LPBF process.

The present findings are also in agreement with the observations of Alves Alcântara et al., who demonstrated that the corrosion behaviour of LPBF Ti-5553 is strongly influenced by the local stability of the passive film. Their study showed that process-induced surface defects can serve as preferential sites for passive-film rupture, followed by rapid repassivation through the reformation of the TiO_2 layer. This continuous depassivation–repassivation process explains why LPBF titanium alloys generally maintain good overall corrosion resistance despite the presence of manufacturing-related surface heterogeneities.

Taken together, the electrochemical results indicate that the as-built LPBF Ti-5553 alloy developed a protective passive film in 0.9 wt.% NaCl solution under the present testing conditions. The low corrosion current, high low-frequency impedance, and predominantly capacitive phase-angle response confirm the passive character of the alloy. However, the corrosion response should be interpreted in relation to the as-built surface condition, since surface roughness and process-induced irregularities may locally affect passive-film uniformity and stability.

Conclusion and Future Perspective

- This study investigated the as-built condition of Ti-5Al-5Mo-5V-3Cr fabricated by Laser Powder Bed Fusion using a TRUMPF TruPrint 1000 system. The experimental work combined powder characterization, density and defect analysis, microstructural observation, phase identification, tensile testing, fractography, nanoindentation, and electrochemical corrosion testing to clarify the relationship between feedstock quality, LPBF consolidation, microstructure, mechanical response, and corrosion behaviour.
- The gas-atomized Ti-5553 powder exhibited suitable characteristics for LPBF processing, including predominantly spherical morphology, limited satellite formation, chemical composition close to the nominal alloy, and an appropriate particle size distribution. Under the selected processing condition of 200 W laser power, 800 mm s⁻¹ scan speed, 0.115 mm hatch spacing, and 30 µm layer thickness, the fabricated specimens reached a relative density of approximately 99.45%. XCT and ImageJ analyses further confirmed that the internal defect content and visible cross-sectional porosity were limited, indicating effective consolidation and the absence of dominant lack-of-fusion defects.
- Optical and SEM observations showed characteristic LPBF melt-pool features, continuous metallurgical bonding, and a direction-dependent microstructural appearance. The as-built material was mainly β -phase dominated, and laboratory XRD did not reveal detectable α , α' , α'' , or ω peaks. After tensile deformation, the broadening of β peaks, increased microstrain, and higher estimated dislocation density indicated that plastic deformation was mainly accommodated by lattice distortion and dislocation accumulation rather than by a detectable deformation-induced phase transformation.
- The selected LPBF condition produced a repeatable tensile response, with an average ultimate tensile strength of 834.6 ± 4.7 MPa, fracture strain of $19.45 \pm 2.08\%$, and toughness of 139.96 ± 15.49 MJ m⁻³. The low scatter in strength confirms a stable load-bearing response, whereas the larger variation in fracture strain suggests that failure remained sensitive to local defects, surface condition, and microstructural heterogeneity. Fractographic observations confirmed a mixed-mode fracture mechanism, involving both cleavage-like regions and ductile dimpled areas, with crack propagation starting near the specimen surface.

- Nanoindentation showed a relatively consistent local mechanical response, with hardness values of approximately 4.1–4.4 GPa and reduced modulus values of approximately 88–100 GPa. The larger scatter observed in the tensile-tested condition was consistent with prior plastic deformation and localized damage. Electrochemical testing in 0.9 wt.% NaCl solution demonstrated passive corrosion behaviour, with a corrosion potential of -0.2203 V and a corrosion current of 7.45×10^{-8} A. This response was attributed to the spontaneous formation of a protective oxide film, although the uniformity of this passive layer may still be affected by the intrinsic surface roughness and process-induced surface features of the as-built LPBF condition.
- Overall, the results demonstrate that the selected LPBF processing condition can produce dense as-built Ti-5553 specimens with limited defects, stable β -dominated phase constitution, repeatable tensile strength, mixed-mode fracture behaviour, relatively consistent local mechanical properties, and passive corrosion behaviour. The main contribution of this work is the integrated experimental correlation between powder quality, LPBF consolidation, defect population, β -phase stability, mechanical performance, fracture behaviour, and electrochemical response in as-built LPBF Ti-5553.

Future Work

- Future research on LPBF Ti-5553 should move beyond the isolated optimization of density, volumetric energy density, tensile strength, or hardness and progress toward an integrated qualification framework. Such a framework should connect processing conditions, melt-pool physics, defect topology, surface condition, metastable phase evolution, post-processing response, and hydrogen embrittlement susceptibility within a single processing–structure–property–performance approach.
- Although near-full-density Ti-5553 components have already been demonstrated, the current literature remains fragmented in terms of process windows, powder characteristics, scan strategies, surface states, post-processing routes, and testing protocols. Future studies should therefore place stronger emphasis on standardizing the relationships between LPBF parameters, defect populations, surface-connected porosity, and mechanical reliability, rather than evaluating each response independently.
- Physics-informed machine learning, digital twins, and in-situ monitoring represent promising tools for moving from trial-and-error parameter selection toward real-time

process control. For Ti-5553, these approaches should not be limited to predicting relative density alone. They should also capture melt-pool stability, pore morphology, surface quality, α/ω precipitation tendency, mechanical response, and fatigue-critical defect populations.

- In-situ microstructure control is another important direction for LPBF Ti-5553. Interlayer time engineering, laser post-exposure treatment, substrate preheating, thermal-cycle design, and controlled scan strategies could be used to tailor β texture, prior- β grain continuity, α/ω precipitation, and possibly columnar-to-equiaxed transition behavior. However, these strategies must be validated carefully, since changes in thermal history may also modify residual stress, defect morphology, anisotropy, and local mechanical response.
- Future work should also extend beyond simple bulk specimens toward lattice, microlattice, hybrid, and compositionally graded Ti-5553 systems. These architectures introduce strut-scale thermal histories, high surface-to-volume ratios, powder-quality sensitivity, and recyclability challenges that cannot be directly inferred from bulk specimens. Their qualification will therefore require dedicated process–defect–microstructure–property studies.
- From a qualification perspective, future progress in LPBF Ti-5553 will depend on integrating process control, microstructure engineering, defect-informed assessment, advanced post-processing, and hydrogen-embrittlement-aware service evaluation into a unified framework for reliable aerospace and high-performance structural applications.

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