



School of Earth sciences and Engineering
Department of Chemical, Materials and Metallurgical Engineering

**FUNCTIONAL PERFORMANCE EVALUATION OF LASER POWDER BED
FUSION FABRICATED AND SUB-TRANSUS ANNEALED Ti-6Al-7Nb ALLOY
FOR ACETABULAR IMPLANTS.**

by

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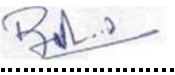
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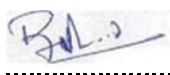
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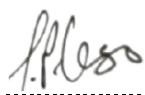
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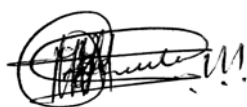
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To Prince, may you be inspired to continue working hard and keep praying that God expands your territory.

To my amazing parents, who never stopped believing in me, I'm blessed to be your child.

To my grandfather, the late Chataika, I dedicate this work to you.

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List of Abbreviations.

THA	Total Hip Arthroplasty
Ti-6Al-4V	Titanium-6wt% Aluminium- 4wt% Vanadium Alloy
Ti-6Al-7Nb	Titanium-6wt% Aluminium- 7wt% Niobium Alloy
UHMWPE	Ultra-High Molecular Weight Polyethylene
THR	Total Hip Replacement
MoM	Metal-on-Metal
MoP	Metal-on-Polyethylene
CoC	Ceramic-on-Ceramic
CoP	Ceramic-on-Polyethylene
CoCrMo	Cobalt-Chromium-Molybdenum Alloy
ISO	International Organisation for Standardisation
ASTM	American Society for Testing and Materials
SEM	Scanning Electron Microscopy
FE	Flexion/Extension
AA	Abduction/Adduction
IER	Internal/External Rotation
EDX	Energy Dispersive X-ray Spectroscopy
XRD	X-ray Diffraction
SBF	Simulated Body Fluid
SLM	Selective Laser Melting
EBM	Electron Beam Melting
DPBSS	Dulbecco's Phosphate Buffered Saline Solution

Abstract.

Additive manufacturing specifically laser powder bed fusion (LPBF) also termed selective laser melting (SLM) has revolutionized the implant production landscape and is a pivotal research topic as it offers the perfect platform for producing complex, personalized implants with precise dimensions. Ti-6Al-4V extra low interstitial (ELI), designated as Grade 23 titanium alloy, is extensively utilized in acetabular implants. However, its corrosion resistance and biocompatibility are compromised by the cytotoxic nature of vanadium, one of its alloying elements. To address these limitations, Ti-6Al-7Nb, a vanadium-free titanium alloy, was developed as a viable alternative. The reduced service life of acetabular implants makes it critical that new biomaterials are explored and microstructural properties are designed to mitigate these failure mechanisms. Despite the potential of Ti-6Al-7Nb, limited studies have investigated how microstructural design through heat treatment of LPBF-fabricated Ti-6Al-7Nb influences its microhardness, wear performance, corrosion resistance, biocompatibility, and compressive behaviour in the context of acetabular implant applications. Thus, this study comprehensively investigated the impact of laser powder bed fusion process and annealing heat treatment (stress-relief annealing at 650°C with a holding temperature of 2 hours followed by air cooling, and full annealing at 850°C with 2 hours holding temperature followed by air cooling) of Ti-6Al-7Nb alloy on the microstructure, compressive behavior, microhardness, wear resistance, corrosion resistance, and invitro apatite-forming capability. The microstructure of as-built LPBF Ti-6Al-7Nb consisted of columnar prior β grains, characterized by fine acicular α' martensite and an intricate basket-weave morphology leading to high compressive yield strength (1230 MPa) and ultimate compressive strength (1634 MPa), enhanced microhardness ($461 \pm 19.69 \text{ HV}_{0.5}$), lower wear rate ($1.33 \times 10^{-3} \frac{\text{mm}^3}{\text{N.m}}$), and higher corrosion rate ($2.285 \times 10^{-4} \frac{\text{mm}}{\text{yr}}$) in simulated body fluid. Stress-relief annealing initiated the precipitation of the β phase while full annealing completely transformed the α' martensite to a lamellar $\alpha + \beta$ microstructure. The lamellar microstructure exhibited a reduced compressive yield strength (973 MPa) and ultimate compressive strength (1183 MPa) critical for minimizing the stress shielding in implants, 19.7% reduction in microhardness ($370 \pm 4.99 \text{ HV}_{0.5}$), and the lowest corrosion rate in simulated body fluid ($1.885 \times 10^{-4} \frac{\text{mm}}{\text{yr}}$). Furthermore, during the 14-day immersion period in simulated body fluid, the fully annealed specimens displayed faster apatite nucleation and proliferation, demonstrating superior biocompatibility and bone tissue integration than the as-built and stress-relief annealed specimens. This study provides new

insight, for acetabular implant manufacturers, on how laser powder bed fusion process and annealing heat treatment can be adopted to design microstructural properties possessing superior biocompatibility, excellent tribological performance and corrosion resistance, as well as good compressive biomechanical properties for improved service life of the implant.

Keywords: Hip joint, Acetabular implant, Laser powder bed fusion, Ti-6Al-7Nb, Apatite, Dulbecco's phosphate buffered saline solution (DPBSS).

List of Publications.

Paper 1:

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Paper 2:

K. Posiyano, A. T. Sello, R. V. S. Prasad, T. C. Dzogbewu, E. O. Olakanmi, T. P. Leso, and K. Setswalo, "Performance evaluation of laser powder bed fusion (LPBF) fabricated and sub-transus annealed Ti-6Al-7Nb alloy for acetabular implant application," (in en), *Journal of Alloys and Compounds*, p. 182421, 2025/07// 2025, doi: 10.1016/j.jallcom.2025.182421.

Paper 3:

A. T. Sello, **K. Posiyano**, M. R. Maina, P. I. Muiruri, R. V. S. Prasad, D. C. Thywill, and E. O. Olakanmi, "Influence of process parameters in selective laser melted Ti6Al7Nb alloy for biomedical applications," *Journal of Manufacturing Processes*, vol. 150, pp. 111-128, 2025/09/30/2025, doi: 10.1016/j.jmapro.2025.06.049.

List of Major Presentations.

Kingsley Posiyano, Amantle T Sello, and RVS Prasad. "Additive Manufacturing of Ti-6Al-7Nb alloy for hip implants." National Science Month NSW-BIUST Satellite, Mahalapye, Botswana, 2024.

Chapter 1: Introduction.

1.1 Chapter Summary.

This introductory chapter presents an overview of the current work's background, motivation and objectives as well as the overall context that the thesis seeks to contribute knowledge to. It discusses the Total Hip Arthroplasty (THA) procedure, emphasizing the critical need to extend the service life of prosthetic components, particularly the acetabular components. The problem statement discusses current challenges supporting the necessity to undertake this study. The main objective and specific objectives were developed to provide solutions to this identified problem. The anticipated benefits from conducting this study are provided, justifying the significance of this study. The next section outlines the organization of this thesis, and finally, a conclusion section summarizes the findings of this chapter.

1.2 Background to the Study.

Total Hip Arthroplasty (THA) is a proven standard procedure, consistently registering positive outcomes in patients with advanced hip joint degeneration or high-impact traumatic injuries (Learmonth et al., 2007). Since its inception by Sir John Charnley, THA has been an unparalleled practice in orthopaedics field. The global demand for hip prostheses is experiencing a sustained upward trajectory, mainly due to factors like the rising incidences of hip-related disorders primarily due to the ageing population and injuries during accidents. Cooper et al. (Cooper C, 1992) projected that by 2050 the prevalence of hip fractures is anticipated to exceed 6.26 million cases. This is coherent with the projections of Kurtz et al. (Kurtz et al., 2007), which predicted the THA demand to rise by 175% between 2005 and 2030. The need for more active and long-lasting hip implants has been fuelled by the more active elderly population and younger patients undergoing THA. Though it is well acknowledged that THA is a very successful and effective procedure, it has been observed that a patient's return to full mobility and absence of hip discomfort lasts for about 15 years, which is significantly less than the orthopaedic community's 30-years goal (National Health Service, 2024). This shows that despite continuous improvements in prosthesis materials, design and manufacturing techniques, the development of hip implants that can satisfy the needs of the more active, longer-living population remains a challenge.

Advancements in prosthetic design, biomaterials, and surgical techniques have led to an improved 10-year femoral component survival rate of 81–95%. In contrast, the survival rate

for acetabular components is slightly lower, ranging 68-84% at 10 years ([Callaghan et al., 1997](#); [Z. Liu et al., 2021](#); [Themes, 2016](#)). The 20th National Joint Registry ([Achakri et al., 2022](#)) identifies Metal-on-Polyethylene (MoP) as the frequently used bearing (articulating) surface, consisting of a Cobalt-Chromium-Molybdenum (CoCrMo) femoral head and a polyethylene insert. This polyethylene liner is backed up by a metallic acetabular shell specifically made from Ti-6Al-4V extra low interstitial (ELI). This titanium alloy predominates in the manufacturing of acetabular cups because of its combination of high specific strength, good biocompatibility, strong corrosion resistance, and reduced elastic modulus, when compared to stainless steel and CoCrMo alloys. Despite all these excellent properties, Ti-6Al-4V ELI acetabular implants do not perform to the expected 30-year service life since they operate in a very aggressive body environment.

The physiological environment, characterized by a highly oxygenated saline electrolyte with a pH of approximately 7.4 and a temperature of 37°C (98.6°F), presents significant challenges to the structural integrity and sustained stability of metallic implants. Additionally, the presence of most aggressive and corrosive chloride ions, and protein concentration in the bodily fluids further increases the hostility of the body system to acetabular implants. A load-bearing implant operating in this severe environment is subjected to different failure modes and mechanisms. Thus, it makes sense that the prevalence of corrosion, wear, and fatigue in acetabular implants have been extensively documented in comprehensive implant retrieval investigations ([Birkett et al., 2015](#); [Sonn & Meneghini, 2020](#)) (See **Figure 1. 1**). Pure titanium, valued for its outstanding resistance to corrosion and excellent biocompatibility, is an ideal material for physiological applications. However, its lower strength may compromise the load-bearing capability of hip implants ([Abd-Elaziem et al., 2024](#); [Sidambe, 2014](#)).



Figure 1. 1. (A). A radiograph of the failed left hip implant showing femoral head migration, wear debris, and bone loss due to stress shielding ([Birkett et al., 2015](#)). (B).

Severe corrosion of the implant at the liner-shell interface ([Sonn & Meneghini, 2020](#)).
(C). Extreme wear of the metal (Ti-6Al-4V) backed-acetabular cup ([Birkett et al., 2015](#)).

There is therefore a serious need to address problems of acetabular implant failure due to wear, and corrosion within the body system. Titanium alloys with excellent chemical composition have to be investigated to replace the mostly used Ti-6Al-4V ELI to improve the service life of acetabular prostheses. However, implant material properties are directly linked to the material's microstructure, which is not only dependent on the elemental composition, but also the manufacturing process (e.g. forging, casting and additive manufacturing) and subsequent heat treatments.

1.3 Problem Statement.

Despite many surgical advantages, premature failure of acetabular components has significantly limited the longevity of a hip joint prosthesis in vivo due to corrosion, and wear failure mechanisms necessitating revision of the prosthesis ([Posiyano et al., 2024](#)). Revision hip arthroplasty procedures present increased surgical complexity, often resulting in less favorable clinical outcomes compared to primary hip replacements, as described by Ong et al. ([Ong et al., 2010](#)) emphasizing that patients are five times more likely to undergo a re-revision after revision of a primary THA. This highlights the need for researchers in materials and metallurgical engineering to collaborate with those in biomedical engineering to bridge the gap between engineering and medicine to improve the acetabular implant survival rate and better people's health.

The limited corrosion resistance and biocompatibility due to the cytotoxic alloying element vanadium, is a major concern against continued use of Ti-6Al-4V alloy in THA prostheses ([Bocchetta et al., 2021](#)). Laing ([Laing, 1979](#)) acknowledged the increasing concerns regarding the cytotoxicity of the β -stabilizer vanadium (V), released from Ti-6Al-4V implants. Toxicity of the vanadium ions released due to this passive dissolution is a significant concern, with adverse effects like tissue discoloration and inflammatory response well documented in the literature ([Choubey et al., 2004](#)). It is also reported that vanadium ions affect implant osseointegration with the surrounding bone tissues by inhibiting osteoblast differentiation which hinders the normal bone deposition process, leading to poor implant-bone fixation and subsequent implant failure ([Thompson & Puleo, 1996](#)). Apart from the material perspective, the current conventional manufacturing methods (casting, forging or machining) do not allow customization of hip implants to match the individual's

skeletal anatomy. There is a need to customize or personalize hip implants to the individual's anatomy for easier osseointegration with the surrounding bone. As shown in **Figure 1. 2**, additive manufacturing preferably laser powder bed fusion (LPBF) provides an opportunity for implants personalization, producing acetabular components with complex and precise structures. Therefore, the LPBF technology coupled with post processing annealing heat treatments of more biocompatible materials like Ti-6Al-7Nb alloy which can reduce the corrosion and wear failure mechanisms as well as facilitate osseointegration through apatite formation on the remodelling bone surface, needs to be explored for acetabular implant applications.

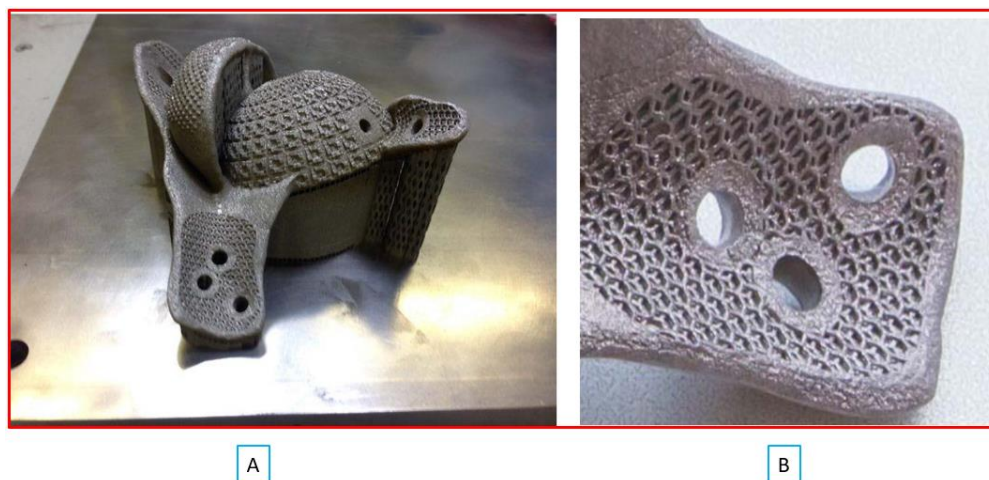


Figure 1. 2. (A). LPBF-fabricated Ti-6Al-4V acetabular implant on a base plate. (B). Magnified view of the intricate structures (texturized surface) that can facilitate osseointegration and reduce stress shielding (Popovich et al., 2024).

1.4 Aims and Objectives.

This section provides a road map outlining the main aim of this project and how this aim will be accomplished through specific objectives.

1.4.1 Aim.

This research aims to investigate the functional performance and microstructural design through laser powder bed fusion and post process annealing heat treatment of Ti-6Al-7Nb specimens on compressive behaviour, microhardness, wear and corrosion resistance in body simulated fluids along with *in-vitro* apatite forming-capability for the targeted application of acetabular hip implant components.

1.4.2 Specific Objectives.

The below specific objectives were designed to maintain focus and steer the research in the right direction toward achieving its intended aim:

- ***Investigate the microstructural features of the as-built, and heat-treated LPBF Ti-6Al-7Nb alloy specimens.***

Microstructure is a critical determinant of material properties (physical, mechanical, and chemical), directly impacting its performance in biological environments. This analysis provides insights into features like grain size, morphology, and the phases within the material which affect characteristics like strength, biocompatibility, and corrosion resistance.

- ***Determine compressive properties and tribological behaviour of the as-built, and heat-treated LPBF Ti-6Al-7Nb alloy specimens.***

Acetabular components are susceptible to structural failure due to significant compressive stresses transmitted across the hip joint, combined with the complex biomechanical interaction between the acetabular cup and the femoral head. In this research work, wear resistance and compression strength tests have been conducted. These tests are crucial to determine if laser powder bed fusion fabricated and heat-treated Ti-6Al-7Nb alloy is appropriate for load-bearing acetabular prosthesis applications.

- ***Evaluate corrosion resistance and biocompatibility of the LPBF Ti-6Al-7Nb specimens through immersion corrosion test in body simulated fluids.***

Immersion test to determine the corrosion rate through weight loss of LPBF Ti-6Al-7Nb parts exposed to simulated body fluid, Dulbecco's Phosphate Buffered Saline (DPBSS) mimicking the body pH of 7.4, body temperature of 37°C, and ionic concentration similar to the bodily extracellular fluid. An extensive surface properties analysis was then performed to examine the potential for biomimetic hydroxyapatite (HAp) formation on the specimen's surface, serving as an indicator of favourable biocompatibility.

1.4.3 Research questions.

The key research questions for this study are presented below;

- What are the mechanisms responsible for the failure of hip implants leading to their low survival rates of 68-84%?

- Is it possible to utilise the ability of additive manufacturing methods, particularly LPBF, to produce intricate structures like acetabular implant with high dimensional accuracy, excellent compressive properties, and good tribological performance?
- What is the corrosion rate of LPBF Ti-6Al-7Nb alloy in body simulated fluids, and is Ti-6Al-7Nb alloy more resistant to corrosion than LPBF Ti-6Al-4V corrosion rate results from previous studies?
- How does annealing heat treatment of LPBF fabricated Ti-6Al-7Nb specimens influence the osseointegration process in the body simulated fluids through the formation of Hydroxyapatite in body simulated fluids?

1.4.4 Hypothesis.

- Laser powder bed fusion and subsequent annealing treatment of Ti-6Al-7Nb alloy will contribute to increased service life of acetabular components due to excellent biomechanical behaviour, improved corrosion resistance, excellent biocompatibility through bone-like apatite formation, and excellent wear performance.

1.5 Delimitation of the Scope of this Study.

The scope of this research encompasses the investigation of laser powder bed fusion with post-process annealing heat treatment of Ti-6Al-7Nb alloy for application in acetabular prosthetic components. This comprised three main theoretical and experimental sections;

1. Identifying the failure mechanisms of acetabular hip implants in service through a thorough review of literature. The susceptibility of the mostly used alloy, Ti-6Al-4V, to the identified failure mechanisms, corrosion, wear, and fatigue failure mechanisms, was researched and superior properties of Ti-6Al-7Nb alloy to overcome these failure mechanisms were explored.
2. Different manufacturing methods, traditional as well as additive manufacturing processes, for acetabular implants regarding Ti-6Al-7Nb alloy were researched. This study utilized the laser powder bed fusion technique based on its proven track record for fabrication of intricate structures with very high precision, high material efficiency, fast production speed, and improved mechanical strength of the printed parts which can play an important role in mitigating acetabular implant failures by optimizing material properties, implant fit, and biomechanical compatibility.
3. Fabricating Ti-6Al-7Nb alloy parts, performing post process annealing heat treatment on some of the produced Ti-6Al-7Nb parts, and investigating their compressive mechanical behaviour, wear performance, corrosion resistance, and

biocompatibility with the aim of producing implants that will help mitigate the currently observed failure mechanisms and have a longer service life.

However, this research does not investigate the antibacterial properties or the fatigue performance of LPBF fabricated Ti-6Al-7Nb alloy specimens.

1.6 Significance of Study.

This research in acetabular prosthesis seeks to improve the quality of life in individuals who have had hip replacement surgery. This research seeks to advance orthopaedics by offering critical insights into the implementation of laser powder bed fusion and annealing heat treatment processes for Ti-6Al-7Nb alloy to enhance wear performance, corrosion resistance, biocompatibility, and compressive biomechanical behaviour for acetabular components of hip implants. It is anticipated that acetabular implant service life will be improved together with the promotion of sustainable manufacturing processes like additive manufacturing that generate zero or minimal waste products (Green manufacturing).

On the socio-economic side, individuals who have undergone THA will return to their work and other activities for a longer period (increased man-hours) without revision surgeries that might jeopardise their confidence with the hip prosthesis. This will enhance their ability and efficiency to contribute to the national development. These benefits are summarized in **Figure 1. 3.**

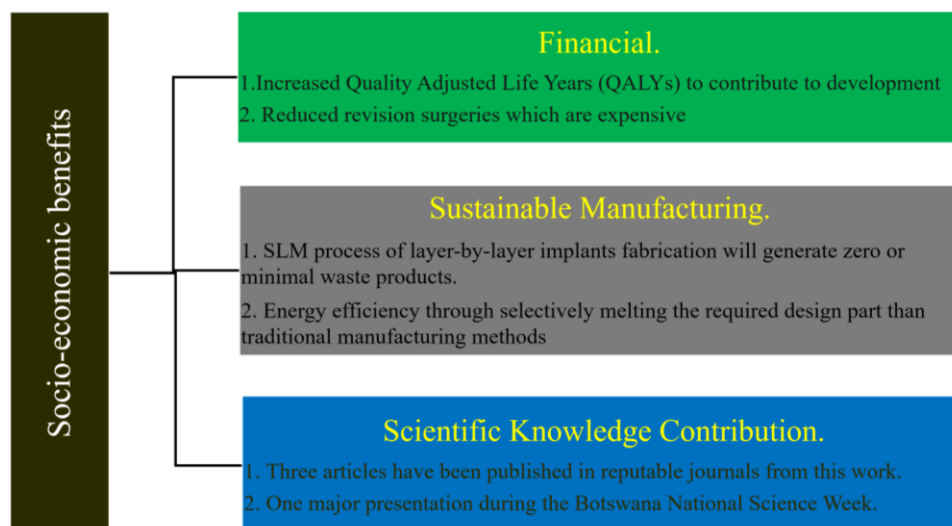


Figure 1. 3. Socio-economic benefits of this research.

1.7 The organisation of the Thesis.

The thesis is divided into eight comprehensive chapters. Chapter One offers a comprehensive overview of the research background, followed by the articulation of the problem statement, description of study's aims and objectives, development of the research questions and hypothesis, and highlighting the study's significance. Literature review (Chapter Two) follows the introduction chapter then methodology/ theoretical framework (Chapter Three), results (Chapter Four), discussion of results (Chapter Five) and conclusion and recommendations (Chapter Six). The thesis concludes with the references/bibliography and the appendices sections respectively.

Chapter Two provides an in-depth literature review that establishes a strong foundation for the subsequent chapters, offering relevant context and insights into key concepts and prior research. This encompasses the structural configuration of the natural hip joint, the biomechanics of hip movement, the human gait cycle, as well as in vitro and in vivo retrieval analyses aimed at assessing the failure mechanisms of acetabular hip implants and concluding with proposed mitigation strategies that can improve prosthesis' longevity. Different additive manufacturing methods that have been applied in acetabular implants fabrication have also been implored and finally, the conclusion section summarizes the literature review chapter.

In Chapter Three, description of the selected materials and the experimental methods used in the current study are systematically outlined. The laser powder bed fusion (LPBF) system, utilized materials, experimental procedures, sample preparation, and analysis techniques are all described in this chapter. All the experiments were conducted as per their respective international standards (ISO standards) and all these standards are described in this section. Chapter Four presents the results while Chapter Five evaluates and interprets findings from compression, microhardness, wear, and corrosion tests. The research thesis' conclusions regarding the behaviour of as-built, and heat-treated LPBF Ti-6Al-7Nb alloys for potential application in acetabular components of hip implants have been presented in Chapter Six. Additionally, this chapter outlines potential areas for future research.

Chapter 2: Literature Review.

In order to tackle the problem of acetabular implant failures, it is imperative to have a thorough grasp of the natural structural configuration of the human hip joint, its biomechanics, and the biomaterials utilized in total hip replacements. A deep metallurgical understanding of titanium and its alloys, which serve as primary biomaterials for acetabular implants, is crucial for addressing material-related concerns that may impact their durability and functional longevity. It is imperative to subsequently determine the failure mechanisms that have limited the lifespan of acetabular implants in the body and explore mitigation measures from the materials perspective, design of the implant, and also manufacturing processes. This literature review chapter is structured to address all these issues and concludes with a summary that highlights the identified research gaps, which aided in the formulation of this research's aim and objectives.

2.1 The Natural Hip.

This section presents an overview of the structural configuration of the hip joint, along with a discussion of its biomechanics and the gait cycle. Fabricating a hip prosthesis of good mechanical integrity, requires a careful consideration of the gait cycle, hip movement planes, and the forces exerted on a natural hip joint.

2.1.1. Structural configuration of the natural hip joint.

Linking the body's lower limbs to its trunk is the hip joint, which is a ball and socket joint (Ramage JL, July 2023). The primary role of the hip joint is to transfer force and load from the axial skeleton to the lower limbs, enabling movement while also providing dynamic support for the body's weight. Thus, it is regarded as the most crucial joint in the body and one of the major weight-bearing joints in both daily life activities as well as sporting activities. To understand the interaction of the hip joint's forces and moments, it must be noted that a hip joint is a synovial or diarthrodial joint due to its four principle features shown in **Figure 2. 1** and described in the next paragraph.

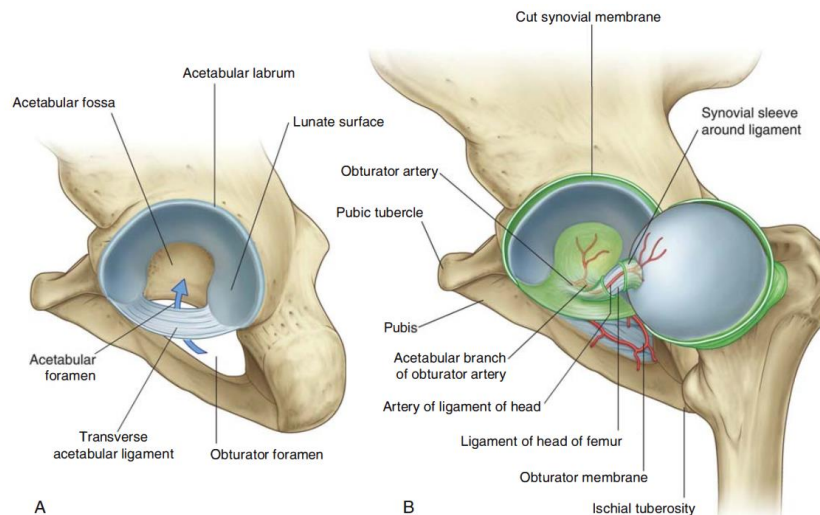


Figure 2. 1: Anatomical features of the hip joint. (A) Transverse view of the acetabular component with the ligament. (B) Lateral view of the head of the femur that has been rotated laterally out of the acetabulum to show the ligament and the cut synovial membrane (Drake et al., 2014).

In contrast to cartilaginous joints or fibrous cartilage, the hip joint has a fluid-filled space known as the joint or synovial cavity. Within this cavity, the acetabulum and the femoral head articulate (Ng et al., 2019). The acetabulum, the deep socket component, gives the femoral head a wide area of articulation thereby providing stability and weight bearing ability to the hip joint. The further deepening of the acetabulum is provided by the acetabular labrum, a fibrocartilage lip connected to the outside margin of the acetabulum forming a ring around the socket of the hip joint. This ring substantially deepens the socket, raising its concavity and giving the femoral head a more stable and secure fit (Harty, 1984). The osseous surfaces of the hip joint are covered with hyaline cartilage from the articular region, a smooth tissue that acts as a teflon coating by cushioning the bones' ends, enabling easy movement (Eschweiler et al., 2021). This hyaline cartilage ensures that friction between the articulating bones is prevented and facilitates load transmission and distribution with a low coefficient of friction. The hip joint is surrounded by a thin layer of synovial membrane that secretes synovial fluid, a thick slimy substance in a healthy joint. This fluid lubricates the joint and further reduces friction between the bones (Liang, 2018). Additionally, the secreted synovial fluid plays an important role of nourishing the blood vessel-free hyaline articular cartilage. Robust fibrous connective tissue bands called ligaments hold bones together and prevent disproportionate or unusual joint motion. These ligaments provide stability and reinforce the joint structure (Chruścik et al., 2021). Ligaments allow full range of motion in

situations where the ball-to-socket configuration of the hip joint provides all the required stability but restricts the range of rotation in situations where the hip is susceptible to impingement, edge loading, or subluxation (Dangin et al., 2016).

In addition to the above features, a complex network of muscles surrounds the hip joint that not only facilitate intended hip motions but also play a crucial role in preventing unwanted movements. Depending on the inertial forces that result from the movements of the masses, this muscle network generates a stabilizing force that leads to a reaction force of the hip joint that sometimes exceeds many times a person's body weight (G. Bergmann et al., 2001; Paul, 1966). In these situations, the muscles function as a "dynamic ligament" resisting forces and providing extra support to the hip joint (Usgu & Usgu, 2023).

2.1.2. Normal Hip Joint Biomechanics.

Understanding the natural hip joint's biomechanics is essential for successful hip prosthetic design. Mechanism of injury is best understood by exploring the interactive forces of the hip joint kinetics and kinematics. The understanding of these interactive forces provides a clear in-depth knowledge of the anatomy and details about the interaction of the forces at the hip provides a better comprehension of some of the past's hip implant failures and gives credence to current and future solutions (Byrne et al., 2010).

2.1.2.1. Hip Joint Kinematics.

Hip joint kinematics evaluates the motion and position of the hip joint without taking into consideration the physical properties or the forces acting on it (Callaghan et al., 2007). A comprehensive understanding of the human hip joint kinematics is crucial for proper design of a prosthesis that will provide the required mobility as the replaced natural hip.

Movement between the femoral head and the acetabulum is almost entirely rotational, with little discernible translation due to the congruency of the hip's surface (Bowman et al., 2010). Hip motion is commonly described as the femur's movement around the hip joint centre with respect to the pelvic and the hip moves as a pivot point in three different planes namely frontal, transverse, and sagittal planes (Galmiche et al., 2020). As detailed by (Jamari et al., 2022; Sebti et al., 2020; Zaghoul & Mohamed, 2018), three degrees of freedom exist at the hip joint, which are flexion-extension rotation (F/E) along the sagittal

axis (**Figure 2. 2A**), adduction-abduction rotations (AD/AB) in the frontal axis (**Figure 2. 2B**), and internal-external rotations (IER) in the vertical axis (**Figure 2. 2C**).

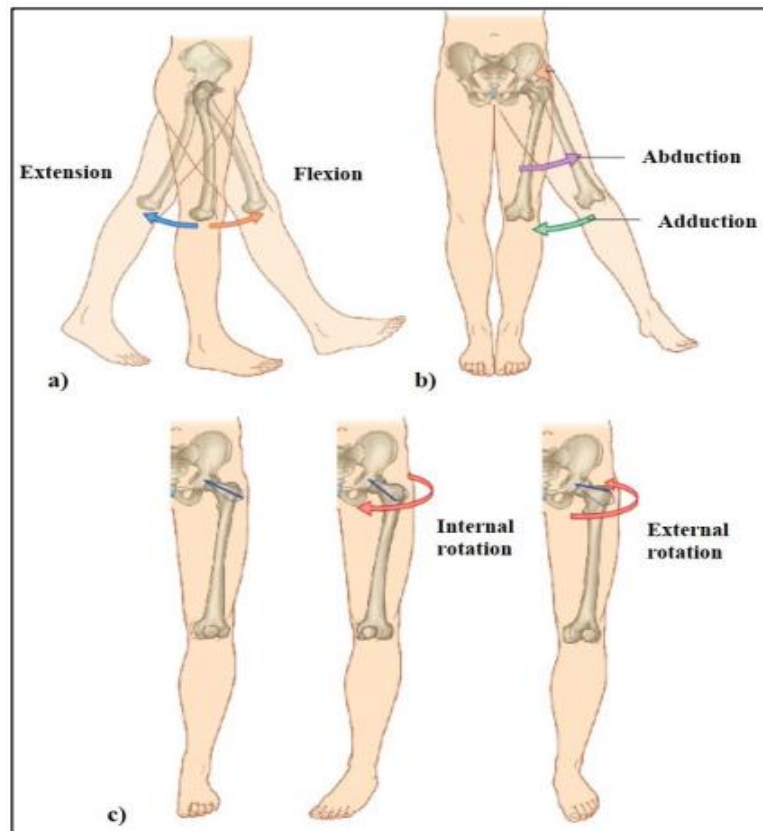


Figure 2. 2: Hip joint movements are (A) flexion-extension (FE) movement, (B) abduction-adduction (AA) movement and (C) internal-external rotation (IER) (Drake et al., 2009).

In the front plane, abduction allows the lower limb to diverge from the midline. In contrast, the lower limb advances towards the midline during adduction. When the hip joint is extended, the frontal plane's abduction range is 0° to 50° and the adduction range is 0° to 30° (*Basic biomechanics of the musculoskeletal system, 2001*). Flexion and extension movements happen in the sagittal plane, where the lower extremity moves forward in flexion and backward in extension. The sagittal plane is the plane of largest movement, with flexion angles ranging from 0° to 140° and extension angles from 0° to 20° (*Basic biomechanics of the musculoskeletal system, 2001*). The hip joint additionally permits the limbs to rotate medially and laterally around their long axis. When one rotates in the direction of the midline, the big toe projects medially, which is known as medial rotation or internal rotation. Lateral rotation also called external rotation is a rotation such that the big toe points laterally and away from the midline (*Galmiche et al., 2020*). With the hip flexed to 90°, the transverse

plane's limitations for internal and external rotation are 40° and 50°, respectively (*Basic biomechanics of the musculoskeletal system*, 2001).

This information is crucial for the design engineers to ensure a hip prosthesis that will restore a patient's mobility without limiting the range of motion. It must be noted however that though the hip joint can reach the described motion ranges in these different planes, it rarely reaches such extreme cases during daily activities.

2.1.2.2. Hip Joint Kinetics.

The kinetics of the hip joint focuses on forces applied to the hip that result in motion. Hip motion arises from the interplay of internal forces (ligaments, muscles, tendons, joints, and joint contact forces) and external forces on the body system (ground reaction force, gravity, and inertia). To generate motion and preserve balance, the hip's soft as well as bony tissues interact in dynamic and static situations that are well described by the gait cycle.

2.1.2.2.1 The Gait cycle.

The gait cycle gives a detailed description of the cyclic pattern of movement that occurs during walking. The terms “gait” and “walking” are commonly used interchangeably but there is a difference that has to be emphasized. According to Michael Whittle (*Whittle, 1998*), Walking is a form of locomotion that involves the alternating use of both legs to provide stability and propel forward movement. Whittle distinguished running from walking by highlighting that during the process of walking, at least one foot must maintain ground contact at all times to ensure stability. Whittle further defines the gait cycle as the duration between two consecutive occurrences of a repetitive walking event. Therefore, the gait cycle is more detailed in that it encompasses all the phases of walking rather than the walking process itself.

The moment at which one foot makes ‘initial contact’ with the ground is typically considered to be the most convenient moment to describe the gait cycle, though any event could be used. Should the decision be made to commence at the right foot's initial touch, as illustrated in **Figure 2. 3** the cycle goes on until the right foot makes contact with the ground once more (*Price et al., 2021*).

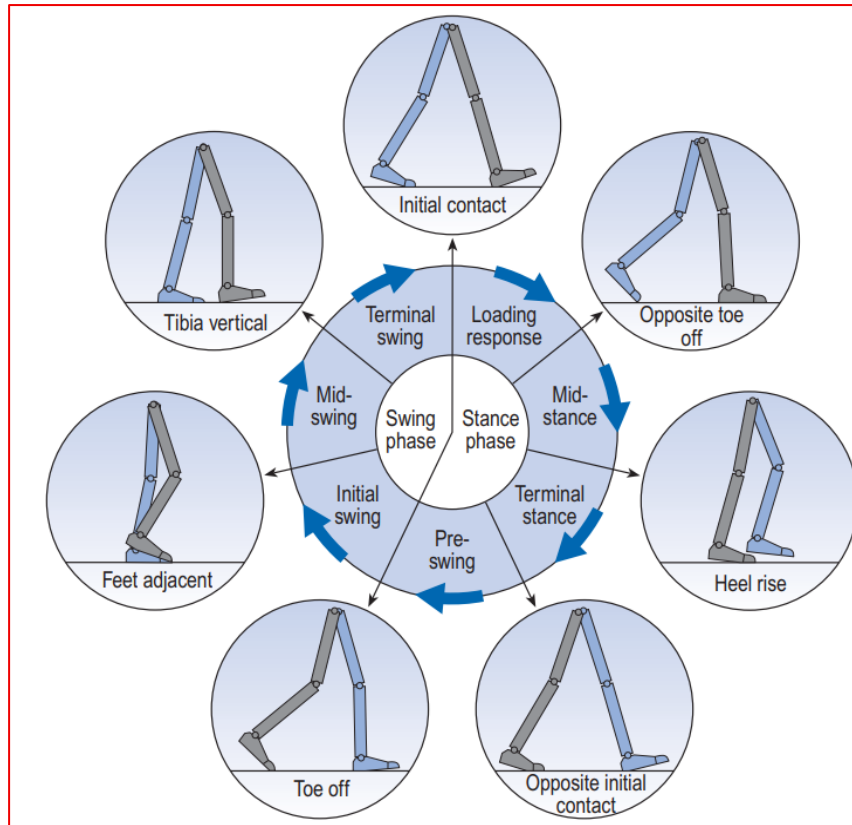


Figure 2. 3: Position and sequence of events in the legs during the right leg's gait cycle. (Gray coloured)(Whittle, 1998).

The sequence of events for the left foot is identical to that of the right foot, however it occurs half a cycle later. As shown in **Figure 2. 3**, there are seven unique events in every gait cycle which are; heel strike (initial contact), opposite toe off, heel rise, opposite initial contact, toe off, feet adjacent, and tibial vertical. These events define two main phases: (1) stance phase (support/contact phase) spanning from heel strike to toe off, where the foot maintains ground contact. (2) Swing phase (oscillation phase) extending from toe off to heel strike, allowing forward progression of the limb (Di Gregorio & Vocenas, 2021). As presented in **Figure 2. 3**, with a detailed illustration in **Figure 2. 4**, the gait cycle comprises seven distinct periods, each marked by a specific event, with the stance phase comprising of loading response, mid-stance, terminal stance, and pre-swing, accounting for 60% of the entire gait cycle. Conversely, the swing phase includes three stages: initial swing, mid-swing, and terminal swing, covering the remaining 40% of the cycle. The cycle time represents the full duration of one gait cycle.

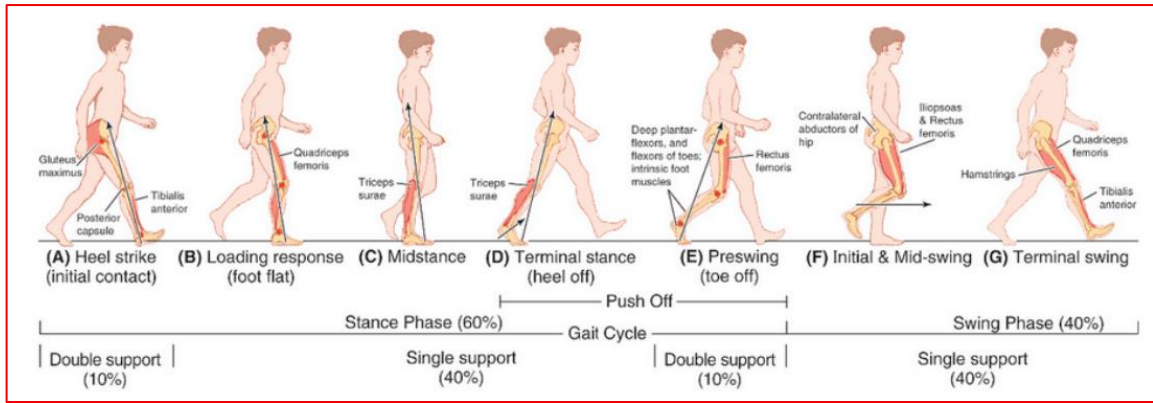


Figure 2. 4: Periods of the gait cycle (Rajčúková et al., 2014).

The hip joint experiences continuous biomechanical loading during routine daily activities in a healthy individual. The magnitude and distribution of forces exerted on the hip joint vary considerably depending on the specific activity, such as walking, stair climbing, or running, due to differences in movement dynamics and mechanical demands. Different techniques are used to understand the loading of the hip joint in both static and dynamic situations. These include the application of static principles, use of smart hip implants that can monitor the load, or displacement on the implant, as well as the application of finite element analysis (FEA) to simulate and analyse the behaviour of hip implants under various conditions. The application of static principles in hip joint kinetics is well described below in static loading phase of the hip joint while smart (telemetric) implants provide detailed information in both static and dynamic phases of hip joint loading. Similarly, FEA provides an overview of hip loading in both static as well as dynamic loading phases.

Finite element analysis provides crucial information like: (1) Equivalent von-Mises Stress. This measures the stress state in the hip implant to predict yielding. Areas of high stresses are prone to material fatigue, and excessive wear resulting in eventual failure of the implant. (2) Equivalent elastic strain. This measures the deformation of the hip implant material under load, relative to its original shape. High equivalent elastic strain indicates that the implant is overstretched or compressed and can fail due to material fatigue over time. (3) Deformation. This describes the change in shape or displacement of the material under load. Excessive deformation in the implant may lead to misalignment, and increased wear making the implant susceptible to failure. The equivalent (von-Mises) stress is determined by the stress components $\delta_{i,j,k}$ acting on the hip implant represented by the stress tensor in the below matrix (1).

$$\delta_{i,j} = \begin{bmatrix} \delta_{11} & \delta_{12} & \delta_{13} \\ \delta_{21} & \delta_{22} & \delta_{23} \\ \delta_{31} & \delta_{32} & \delta_{33} \end{bmatrix} \quad (1)$$

The stresses with repeated numbers i.e. δ_{11} are normal stresses while stresses with mixed numbers are shear stresses. The von-Mises stresses are calculated on principal planes where there are no shear stresses, hence, the matrix for principal stresses is presented as per matrix (2). Equation (3) presents the formula for the calculation of the von-Mises stresses in the hip implant.

$$\delta_{i,j} = \begin{bmatrix} \delta_1 & 0 & 0 \\ 0 & \delta_2 & 0 \\ 0 & 0 & \delta_3 \end{bmatrix} \quad (2)$$

$$\delta_v = \sqrt{\frac{(\delta_1 - \delta_2)^2 + (\delta_2 - \delta_3)^2 + (\delta_3 - \delta_1)^2}{2}} \quad (3)$$

The analysis of these stresses provides detailed information on the loading of the hip implant and lead to the prediction of potential failure areas on the designed prosthesis.

2.1.2.2.2 Static loading.

Typically, during static loading, the sum of forces, measured in Newtons (N) acting on the hip joint is zero in all axes. Thus, for translational static equilibrium;

$$\sum F_{x,y,z} = 0 \quad (4)$$

Where F is the force and x, y, z are the axes of rotation, specifically frontal, transverse, and sagittal axes of the hip joint.

Torque measured in Newton.metre (Nm) is generated in the hip joint due to load acting at a distance. Similarly, for static rotational equilibrium the sum of these moments must be zero;

$$\sum M_{x,y,z} = 0 \quad (5)$$

Where M is the moment and x, y, z are the axes of rotation for the hip joint.

The Pauwels balance, a fundamental concept in hip biomechanics, takes into account the intricate interplay of forces that act on the hip joint during two critical situations, the double-leg stance and the single-leg stance.

2.1.2.2.1 Double-leg stance.

Simply presented in a free-body diagram in **Figure 2. 5A**, for a double-leg stance, there is a functional balance in the normal hip joint with equal loading on both hips and its magnitude being the combination of one-third of the body weight and the muscular force (Pauwels, 1976; Savci & Bilik, 2014). Each of the two hip joints is subjected to equal and opposing magnitude moments.

2.1.2.2.2 Single-leg stance.

The human body can be viewed as a lever system when standing on one leg, with the femoral head serving as the fulcrum. This situation is mostly experienced on the swing phase of the gait cycle and is schematically presented on a free-body diagram **Figure 2. 5B**.

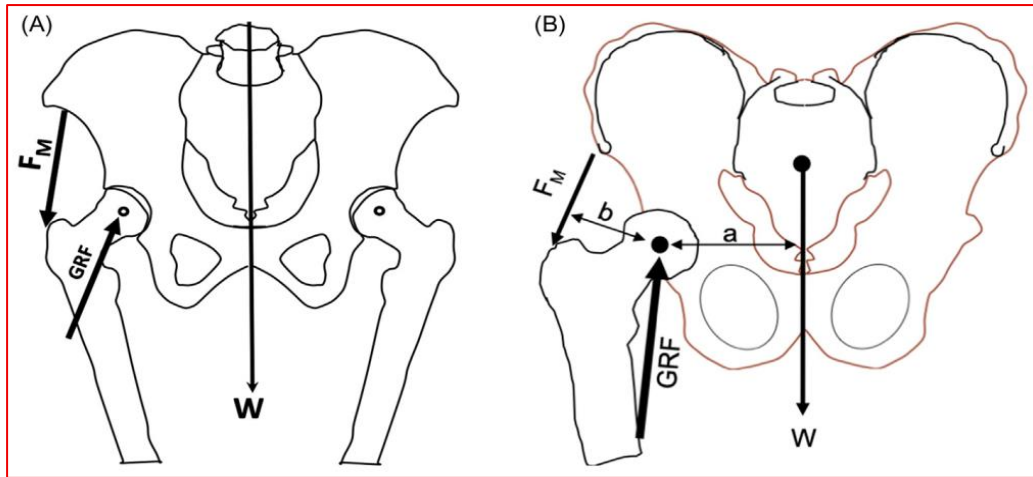


Figure 2. 5: (A) Double-leg stance. The force placed on each hip is equivalent to the combination of force of the muscles surrounding the hip joint and one-third of the body weight. (B) Loading of the hip joint in unilateral stance. The weight of the lower limb without load = $\frac{1}{6}BW$; Body weight across the hip = $\frac{5}{6}BW$; load exerted by muscle (FM) = $2(\frac{5}{6}BW)$; $\Sigma F = GRF$ (ground reaction force) = $(\frac{5}{6}BW) + 2(\frac{5}{6}BW)$ (Eyüboğlu et al., 2020).

A detailed analysis of the swing phase shows that lifting one foot shifts the body weight to one side, and the centre of gravity moves accordingly. The weight of this shifted side is

incorporated into the overall body weight; as a result, the centre of gravity shifts toward the side with more weight. This causes a much higher load at the hip joint than the body weight thereby producing a moment centrally around the femoral head. In the context of a hip bearing a load and achieving a balanced pelvis, the abductor muscle force (F_M) needs to generate an opposing moment that is twice the moment generated by the body weight (Pauwels, 1976). Therefore, when a person stands on one leg unassisted, a load of approximately three times the body weight is placed at the hip joint.

2.1.2.2.3 Dynamic loading.

Hip contact forces can be estimated indirectly using free-body diagrams as reviewed in the static loading case. Apart from indirect measurements, the dynamic hip contact forces can also be measured in vivo. To have a better understanding if the designed hip prosthesis has sufficient strength to withstand the forces and failure mechanisms in the body system, it is vital to know the real-time hip contact forces during everyday dynamic activities like walking, running, and squatting among others. Using instrumented telemetric prostheses, dynamic hip contact forces are evaluated in vivo (Georg Bergmann et al., 2016). These forces summarised in **Table 2. 1**, provide reference data for the accuracy of the musculoskeletal models that are used to determine the joint forces. **Figure 2. 6** shows results of the musculoskeletal model after THA with the resulting force (F_{res}) determined using the force components F_x , F_y and F_z . The obtained forces were within the range of the forces experienced in the joint during walking as measured by the instrumented prostheses indicating that the used model was accurate.

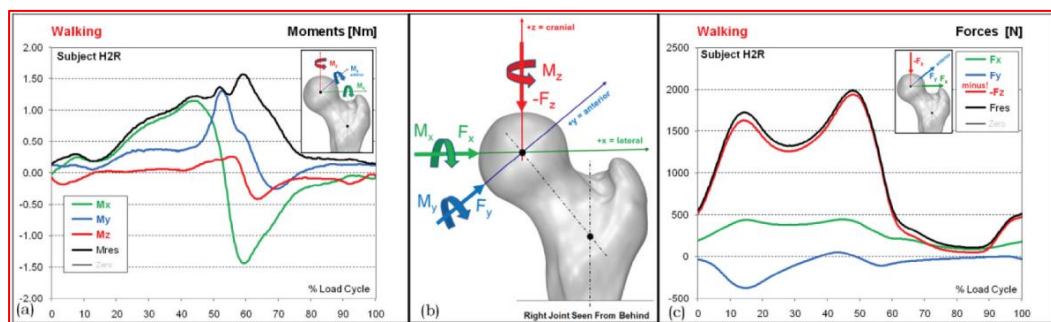


Figure 2. 6: (a) Resultant and directional moments obtained by the musculoskeletal model. (b) The CAD model of a right hip prosthesis and the applied loads and moments. (c) Directional and resultant forces obtained by the simulation (Reginald et al., 2023).

Table 2. 1. Measured hip joint forces aided by instrumented prostheses. Source: Bryne et al. (Byrne et al., 2010).

Activity	Typical peak force applied to the hip × Body Weight (N)	References
Slow walking	1.6 to 4.1	(G. Bergmann et al., 2001; G. Bergmann et al., 1993, 1995; Rydell, 1966)
Normal walking	2.1 to 3.3	(G. Bergmann et al., 2001)
Fast walking	1.8 to 4.3	(G. Bergmann et al., 2001; G. Bergmann et al., 1993, 1995; Rydell, 1966)
Ascending stairs	1.5 to 5.5	(G. Bergmann et al., 2001; G. Bergmann et al., 1995; Rydell, 1966)
Descending stairs	1.6 to 5.1	(G. Bergmann et al., 2001; G. Bergmann et al., 1995; Rydell, 1966)
Jogging, running	4.3 to 5.0	(G. Bergmann et al., 1993, 1995)
Standing up	1.8 to 2.2	(G. Bergmann et al., 2001)
Sitting down	1.5 to 2.0	(G. Bergmann et al., 2001)

Stumbling	7.2 to 8.7	(G. Bergmann et al., 1993, 2004)
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2.2 Total hip arthroplasty (THA).

This section provides a detailed technical overview of total hip arthroplasty (THA) prostheses, including a summary of its historical development and the terminology associated with THA implants. It further explores the classification of THA systems and examines the various biomaterials utilized in the fabrication of prosthetic components. Lastly, the failure modes and mechanisms affecting hip prostheses, are systematically analyzed.

2.2.1 THA prostheses: An Overview.

Total hip arthroplasty (THA) is recognized as a technically advanced and highly effective surgical intervention in the domain of orthopaedic medicine. This procedure ensures a safe and reliable treatment approach, promoting the restoration of joint functionality, pain relief, and the reintegration of patients into their daily activities (Sheth et al., February 2024). As shown in **Figure 2. 7**, the total hip arthroplasty procedure replaces the compromised hip joint with engineered prosthetic components, including the acetabular cup, polyethylene insert, femoral head, and femoral stem, which are merged to form a THA prosthesis. These prosthetic components have been explained in detail in **Table 2. 2**.

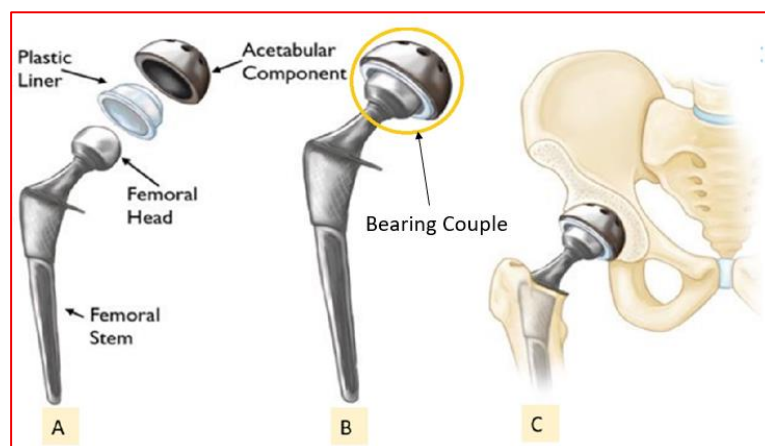


Figure 2. 7: (A) A total hip arthroplasty's individual part, (B) The merged components to make an implant, (C) The prosthesis fixed into the hip (Sheth et al., February 2024).

Table 2. 2. A detailed description of THA prosthesis components.

Term	Definition	References
Acetabular shell	The component of a THA prosthesis implanted within the acetabulum, the concave structure that constitutes the socket of the hip joint.	(Achakri et al., 2022)
Acetabular liner	A plastic, ceramic or metal spacer (liner) featuring a hemispherical socket that interfaces with the femoral head internally and the acetabular shell externally, which might be stabilised in place through a locking mechanism. It serves to create a smooth gliding surface.	(F. Committee; Sheth et al., February 2024)
Femoral head	The femoral component's spherical portion enabling articulation in an artificial hip replacement.	(Achakri et al., 2022)
Femoral stem	The section of the femoral component implanted in the femur bone, providing structural support for the femoral head.	(Achakri et al., 2022)
Bearing couple	A ball-and-socket system designed to replace the natural hip's articulating surfaces, with its contact areas referred to as bearing surfaces.	(F. Committee)

Indeed, nothing lasts forever, and no operation is 100% successful, in February 2019 the medical journey Lancet published extensive research by Evans et al. (Evans et al., 2019) which found that nearly 90% of hip replacements remained operational for 15 years, whereas only 58% sustained structural integrity for 25 years. Hip replacement surgeries are becoming more and more popular due to the increased life expectancy and population growth (Kurtz et al., 2007). A primary THA refers to the initial surgical implantation of a hip prosthesis, while revision hip replacement involves the removal and replacement of one or more prosthetic components from a previous THA procedure.

2.2.2 Hip Arthroplasty prosthesis types.

There are four distinctive categories of hip arthroplasty as reflected in the NJR ([Achakri et al., 2022](#)) classified by the hip replacement type, fixation method, prosthesis bearing surfaces, and the femoral head size or acetabular component's internal diameter.

2.2.2.1 The hip replacement type.

Hip replacement procedures are classified into two techniques: total hip arthroplasty (THA/THR) and hip resurfacing (HR). During THA, the head of the femur and the compromised acetabular socket are excised and substituted with prosthetic components composed of metal, ceramic, or polymeric materials to restore joint function and stability. This procedure consists of removing the natural head of the femur, precise preparation of a canal in the femur for the placement of a femoral prosthetic stem, along with the implantation of a prosthetic acetabular cup into the pelvic bone. In contrast, the hip resurfacing technique retains the femoral head by modifying its anatomical structure through reshaping rather than replacing it entirely. This involves trimming the femoral head and covering it with a smooth metal cap ([Sheth & Foran, 2023](#)). As a result, hip resurfacing is a more bone-preserving procedure compared to THR, as depicted in **Figure 2. 8**.

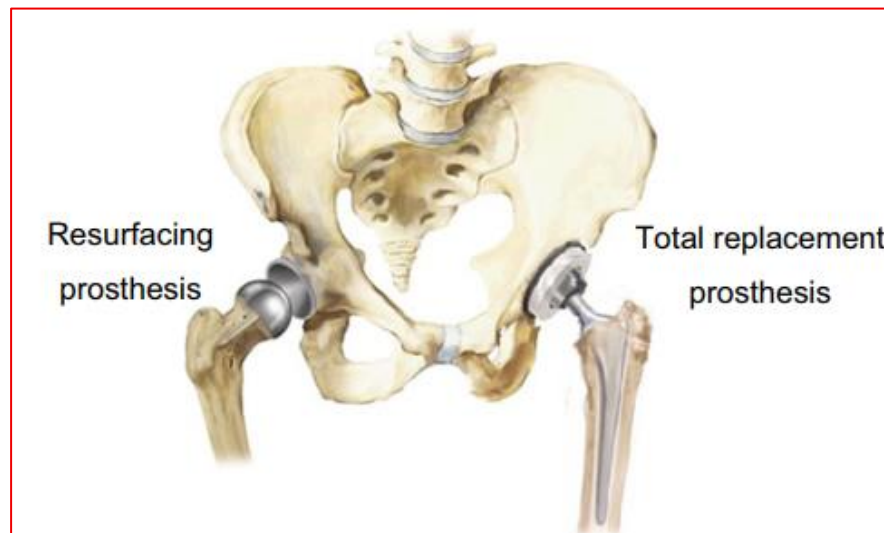


Figure 2. 8: Hip resurfacing arthroplasty on the right hip and total hip arthroplasty of the left hip ([Mattei et al., 2011](#)).

2.2.2.2 The fixation of the replacement.

The following categories apply to hip replacements with regard to the type of fixation utilized; cemented fixation (where polymethylmethacrylate (PMMA) bone cement acting as a ‘grout’ is used to secure the femoral stem and acetabular components to the bone), uncemented fixation (no bone cement is used and the prosthetic components rely on press fit and bone ingrowth or ongrowth within the femur and the acetabulum that on some occasions is supplemented by screw fixation), hybrid fixation (the femoral stem is held in place through cemented fixation and uncemented fixation is utilized on the acetabular component), and reverse hybrid fixation (the opposite of hybrid fixation where constructs contain a cemented acetabular component and uncemented femoral stem). According to the 20th Annual NJR report, uncemented fixation was the most common used fixation type accounting for 37% of all THR performed in 2022-2023.

2.2.2.3 *The prosthesis bearing surfaces.*

Hip replacements can be classified into different primary categories according to the materials deployed for the bearing (articulating) surfaces. For THA, there are seven primary bearing surfaces including; ceramic-on ceramic (CoC), ceramic-on-metal (CoM), ceramic-on-polyethylene (CoP), metal-on-metal (MoM), metal-on-polyethylene (MoP), metal-on-polyethylene-on-metal (MoPoM), and ceramic-on-polyethylene-metal (CoPoM). For the case of hip resurfacing arthroplasty, two bearing surfaces are currently used, which are; metal-on-metal (MoM) and ceramic-on-ceramic (CoC). Conventionally, the femoral head’s material is specified before the acetabular component material, and the inclusion of three bearing materials signifies the utilization of a dual mobility bearing system. In the 20th Annual NJR, for 2022-2023 the mostly used combination was MoP followed by MoM.

2.2.2.4 *The femoral head size or acetabular component’s internal diameter.*

Hip replacement prostheses are also categorized according to the selected femoral head diameter for implant design. Some of these types are; 22, 26, 28, 30, 32, 34, 36, 38, 40, 42, 44, 46, 48, 50, 52, and 54+ (all the dimension in mm) ([Achakri et al., 2022](#)). Consideration of head size is very important as it impacts the likelihood of requiring revision surgery after hip arthroplasty. As shown in **Figure 2. 9**, the 22.25 mm head size had an overall worst revision rate while the 26mm diameter had a better revision rate. The size varies according

to the patient's anatomical structure, with 32 mm being the most frequently implanted dimension ([Achakri et al., 2022](#)).

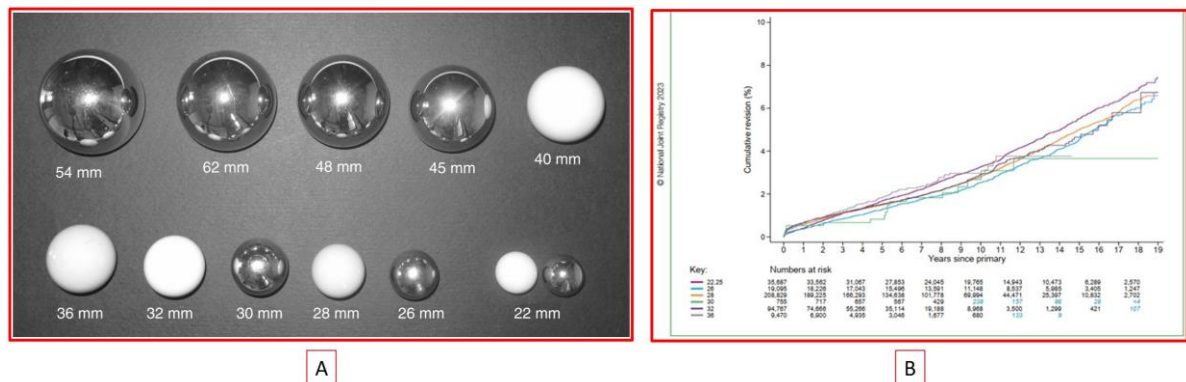


Figure 2. 9: (A) Various femoral head sizes ([Saverio Affatato, 2014](#)) and (B) Revision of primary cemented MoP hip arthroplasty by femoral head size (mm) ([Achakri et al., 2022](#)).

2.2.3 Biomaterials used in THA.

A biomaterial is any non-pharmaceutical substance, either synthetic or natural, that can be applied for a specific duration to support, augment, or replace biological tissues, organs, or physiological functions, thereby enhancing an individual's quality of life ([C. Bergmann & Stumpf, 2013](#)). A critical term demanding careful attention when dealing with biomaterials is biocompatibility, which was defined as “*the ability of a material to perform with an appropriate host response in a specific application*” by the Williams dictionary of biomaterials ([D. F. Williams, 1999](#)). The body’s immune system is vigilant and constantly monitors for foreign invaders and despite being biocompatible, no biomaterial is completely free from the host immune response. Biomaterial quality directly influences prosthesis longevity in total hip arthroplasty (THA), with failures often linked to material properties. Advances in biomaterial quality have given rise to new biomaterials throughout time. Rojas et al. ([Rojas et al., 2023](#)) emphasized that a perfect/ideal biomaterial should be resistant to corrosion, biocompatible, have sufficient mechanical strength and a low friction coefficient. This section explores the two main classifications of biomaterials used in hip implants: chemical composition classification and functional classification. A systematically comprehensive analysis of the scientific literature will be undertaken to assess the advantages and limitations of various biomaterials employed in total hip arthroplasty (THA).

2.2.3.1 Chemical Composition Classification.

Biomaterials are categorized into metals, polymers, ceramics, and composites based on their chemical composition.

2.2.3.1.1 Metals.

Metallic biomaterials have dominated medical implant applications for decades, comprising over 70% of all implanted medical devices attributable to their exceptional mechanical properties, durability, and biocompatibility (Okazaki, 2019). Metallic materials are utilized for both the mobile (femoral head and liner) and stationary (femoral stem and acetabular component) components in THA. Pure metals find applications in THA but their inherent limitation in mechanical properties specifically strength often require alloying to improve their long-term success as body implants. During alloy development, only non-toxic elements should be used as alloying constituents to ensure biocompatibility and material safety. The primary metallic materials utilized in THA include stainless steels, cobalt-chromium (Co-Cr) alloys, and titanium alloys. **Figure 2. 10** provides a comparative analysis of the biocompatibility of selected pure metals and biomedical metallic alloys.

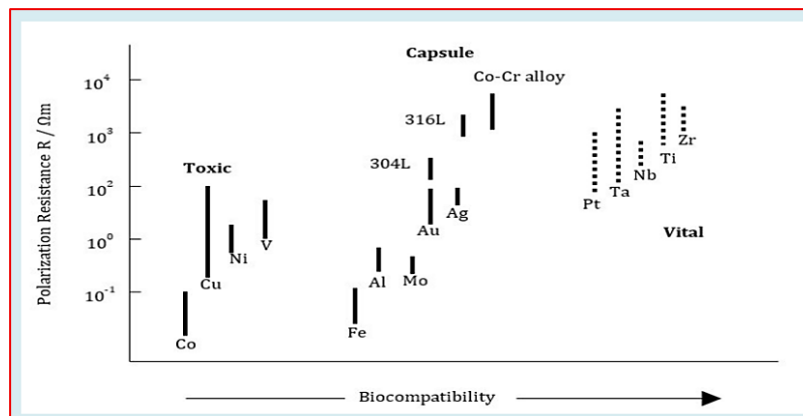


Figure 2. 10: The correlation between electrochemical resistance and the biocompatibility of different biomaterials (Yamanoglu, 2020).

2.2.3.1.1.1 Stainless Steels.

According to Bronzino and Peterson (Bronzino & Peterson, 2018), stainless steel was the first metallic biomaterial to be successfully utilized as an implant material in surgical

applications. They are ferrous alloys containing at least 10.5 wt.% Cr and other elements like Ni, Mo, Mn, and C. A minimum chromium content of 10.5 wt.% is required to initiate the development of a stabilizing chromium oxide barrier on the metal surface, ensuring enhanced corrosion resistance and material durability. Stainless steels are categorized into ferritic, austenitic, and martensitic classes, each defined by distinct crystallographic structures and mechanical properties. However, only austenitic steels are applied for hip implants fabrication. Austenitic stainless steels are extensively utilized due to their cost efficiency and exceptional formability.

Austenitic steels mostly 316L alloy has been predominantly employed for femoral stem in THA for the past years. However, the limited biocompatibility, corrosion resistance, and wear resistance of these materials have led to critical concerns, including corrosion-induced toxicity, implant degradation, and hypersensitivity reactions in adjacent tissues due to wear debris. Consequently, their application is primarily restricted to temporary or temporary orthopaedic implants, including bone screws, fixation plates, and intramedullary nails.

2.2.3.1.1.2 Cobalt Chromium Molybdenum Alloys.

A metallic alloy containing 60-70% cobalt (Co), 25-30% chromium (Cr), and 5-7% molybdenum (Mo) is extensively utilized as a biomaterial for femoral heads because of its remarkable hardness, superior corrosion resistance, and outstanding wear resistance (Khalifa & Bakr, 2021). Cast CoCrMo alloy and Wrought CoCrMo alloy are two most prevalent types of CoCrMo alloys. Two types of wrought CoCrMo alloys are commercially available; (1) low carbon (< 0.14 wt.% carbon) and (2) high carbon (0.15-0.35 wt.% carbon) (F. Committee). Studies show that high-carbon CoCrMo alloys outperform low-carbon variants in corrosion and wear resistance. However, it has been reported that this increase in carbon promotes the formation of carbides. These carbides create localized areas with depleted alloying elements like Cr and these areas might become the initiation sites for corrosion thereby reducing its corrosion resistance (Kinbrum & Unsworth, 2008; Valero-Vidal et al., 2013). Despite the excellent recorded clinical performance of CoCrMo alloys, the discharge of corrosion and wear debris coupled with adverse biological response of surrounding tissues, has emerged as a critical concern. The principal drawback of CoCrMo alloys lies in their potential cytotoxicity and carcinogenicity, as particulate release within the joint elicits an inflammatory response, ultimately resulting in osteolysis.

2.2.3.1.1.3 Titanium alloys.

Titanium and its alloys are widely utilized biomaterials in THA. They are the preferred material for hip implants application due to their advantageous properties, characterized by excellent specific strength, superior corrosion resistance to body fluids, and excellent biocompatibility due to its ability to promote osteointegration. Compared to stainless steels, titanium alloys demonstrate superior corrosion resistance in body fluids resulting from the passivated oxygen protective film on its surface.

Pure titanium exhibits the α -phase, a hexagonal close-packed (hcp) crystal structure, at room temperature. At 882°C, an allotropic phase transformation occurs, resulting in the body-centered cubic (bcc) β -phase formation from the hcp α -phase (Glavicic et al., 2003; Jeong et al., 2022) as illustrated in **Figure 2. 11**, and this temperature of transformation is called the beta transus temperature. This β -phase remains stable between 882°C and approximately 1688°C, which is the melting point of titanium. Suitable mechanical and physical properties for hip prosthesis application can be obtained by selective alloying of titanium with other elements. The α -phase and β -phase stabilizing properties of these alloying elements are determined by their influence on the beta transus temperature. Some alloying elements like Zr and Sn categorized as neutral elements, do not affect the beta transus temperature. The α -stabilizers like C, O, N, Al raise the beta transus temperature to expand the α -phase's stability region. The β -stabilizers which are either isomorphous like Mo, Nb, Ta, and Nb aid the retention of the single β -phase microstructure or eutectoid like Cr, W, Si, Fe, Ni, H, Mn, and Co which facilitate the development of two-phase microstructure, decreases the beta transus temperature thereby widening the region in which the β -phase is stable. As a result, there are four types of Ti alloys: α , near- α , $\alpha+\beta$, and β alloys.

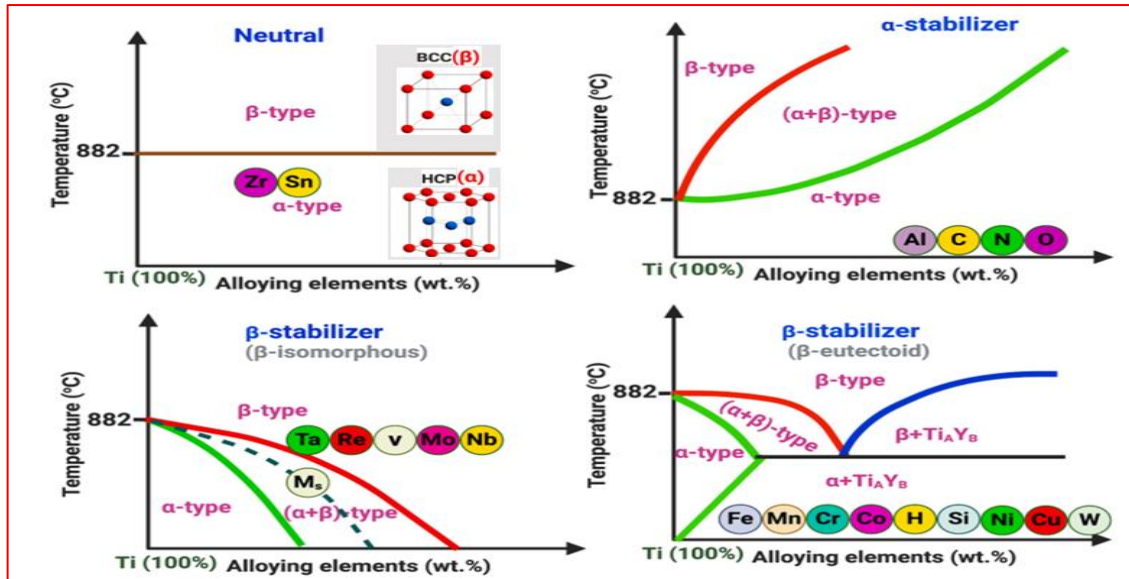


Figure 2. 11: Different categories of alloying elements and how they affect titanium. Presented from the top left corner; neutral, α -stabilizers, β -stabilizers (both isomorphous and eutectoid) (Abd-Elaziem et al., 2024).

The properties of α and near- α alloys are comparable and their low mechanical strength has limited their applicability in load bearing implants like hip prosthesis (L. C. Zhang & Chen, 2019). The ($\alpha+\beta$) Ti alloys exhibit a significant β -phase (5-30 vol%) (Jawed et al., 2022). They demonstrate exceptional mechanical strength, excellent corrosion resistance, and promote rapid osteointegration by forming a TiO_2/OH coating on the surfaces of the bones (Sidambe, 2014). The β alloys (β – phase > 30 vol%) maintain the β -phase fully at room temperatures and express superior biocompatibility, however their application in medical implants is limited since the β -stabilizing elements are costly, rare, and possess high melting temperatures making the processing of these alloys more difficult (Narita et al., 2012).

The transition metal Ti, when properly alloyed, has proven itself to be a game-changing biomaterial. The chemical composition, phase constituents, and crystal structure of titanium alloys have resulted in biomaterials with an exceptional blend of mechanical strength (Elias et al., 2019), corrosion resistance (Tomashov et al., 1961), biocompatibility (Kaur & Singh, 2019), and the capacity to foster integrative tissue connections (Bosshardt et al., 2017). Titanium has excellent specific strength (strength to density ratio), $222.22 \text{ MPa cm}^3/\text{g}$, significantly higher than steels, cobalt alloys, aluminum (Kishawy & Hosseini, 2019). This is a desired property for implant application to minimize weight of the implant while concurrently maintaining its structural integrity. The low density of titanium alloys is a key

factor in reducing the overall load and movement inertia on the human body when titanium implants are used. Apart from its exceptional blend of strength and low modulus than stainless steels, cobalt alloys, and ceramics, titanium alloys have a high affinity towards oxygen thereby spontaneously forming a superficial oxide protective layer when exposed to an oxidizing atmosphere. This protective layer forms a strong barrier between the aggressive physiological fluids and the underneath metal resulting in excellent corrosion resistance. Furthermore, this protective oxide layer is responsible for the exceptional biocompatibility and osseointegration of implants as bone tissues can adhere and grow on the titanium alloy's surfaces (Branemark et al., 2001). This is a very crucial property for permanent replacements in the body system. As a result, titanium alloys have emerged as the most successful material over CoCr alloys, stainless steels, ceramics and others in biomedical applications particularly in the field of orthopaedics.

Up to date, Ti-6Al-4V ELI, an upgraded form of Ti-6Al-4V possessing minimal levels of impurities like C, O, H, and N is frequently utilized for prosthetic hip joints. However, due to low wear and abrasion resistance, these titanium alloys are not employed in bearing surfaces like femoral heads.

2.2.3.1.2 Ceramics.

Ceramic materials were integrated into THA to enhance acetabular insert wear resistance and mitigate peri-implant osteolysis, (Hannouche et al., 2018) and they are predominantly utilized in the fabrication of the prosthesis' mobile components, including the femoral head and acetabular liner. Bioceramics possess a distinctive blend of high intrinsic strength, excellent biocompatibility, and versatility, giving them a competitive advantage over other biomaterials (Vaiani et al., 2023). The predominantly utilized ceramic materials in THA are alumina and zirconia.

2.2.3.1.2.1 Alumina.

High-density, ultra-pure (>99.5%) Al₂O₃ is among the most extensively utilized biomaterials in load-bearing hip prostheses, boasting a 45-year clinical track record. Its widespread adoption is attributed to its enhanced corrosion resistance, superior biocompatibility, excellent wear resistance, and remarkable strength (Ceramics in clinical applications, 1987; Christel et al., 1988). With a commercial name of BioloX in biomedical use, it possesses

beneficial properties, including a low coefficient of friction, exceptional hardness, and superior wear resistance with some studies reporting that wear rate of this CoC bearing is 4000 times lower than the MoP articulation ([Macdonald & Bankes, 2014](#)). Recently Xu et al. ([J. Xu et al., 2022](#)) demonstrated that alumina CoC implants achieve a survival duration exceeding 20 years with minimal post-THA complications. However, the high elastic modulus (400GPa) of BioloX surpassing the cortical bone's 30GPa means stress shielding is inevitable, leading to implant loosening ([Macdonald & Bankes, 2014](#)). The other weak points of alumina are the mechanical fragility and possibility of breakage in addition to the squeaking noise produced when used in CoC bearings ([Zivic et al., 2017](#)).

2.2.3.1.2.2 Zirconia.

To overcome the mechanical limits like low fracture toughness of alumina which resulted in high incidences of fractures of alumina femoral heads, in the mid-1980s zirconia was developed as a biomaterial. Among monolithic ceramics, zirconia exhibits superior toughness and exceptional crack resistance, making it a widely adopted alternative to alumina in THA ([De Aza et al., 2002](#); [Khalifa & Bakr, 2021](#)). However, zirconia is associated with three primary concerns: its progressive decline in mechanical strength upon prolonged exposure to physiological fluids, its wear performance limitations, and its potential radioactivity. Additionally, the documented phase transformation from tetragonal to monoclinic phase in yttria-stabilized zirconia, induced by aging in aqueous and biological environments, significantly deteriorates its mechanical integrity ([Masonis et al., 2004](#)).

2.2.3.1.3 Polymers.

During THA, polymers are employed in the metal acetabular cup's liner as well as a cementing material for prosthesis fixation. Prosthesis stability is affected by acetabular liner's wear performance over time, which is influenced by the type of polymer used.

2.2.3.1.3.1 Polymethyl methacrylate (PMMA).

This was first used by as a bone cement by Charnley in the 1960s to bind the THA components to the native bone achieving favourable long-term clinical outcomes ([Charnley, 1964](#)). Subsequent research, however, has demonstrated that PMMA cement has been

connected to implant loosening and displacement because of exothermic polymerization of the methyl methacrylate monomer, facilitated by a catalyst, which releases a toxic monomer that may trigger bone necrosis ([Abdel-Kader et al., 2001](#)).

2.2.3.1.3.2 Polytetrafluoroethylene (Teflon) (PTFE).

Dr. Roy Plunkett, a chemist at DuPont, made the accidental discovery of polytetrafluoroethylene in 1938. He accidentally produced a white, waxy substance that was slippery and resistant to most chemicals while working with refrigerant gases (["Overview of PTFE: Composition, Properties, Processing, and Uses - HDC," 2024](#)). It is an extremely high wear coefficient, hydrophobic, and thermally stable tetrafluoroethylene fluoropolymer that was initially used by Charnley et al. ([Charnley, 1970](#)) as a substitute for polymethyl methacrylate. While PTFE's coefficient of friction made it seem like it would function similarly to the hyaline cartilage found in natural joints, very high wear rates (0.5mm/month) and voluminous mass of loose debris caused adverse local tissue reactions that facilitated osteolysis, aseptic loosening, and clinical failure ([Saverio Affatato et al., 2015](#); [Feder et al., 2023](#)).

2.2.3.1.3.3 Ultra-high molecular weight polyethylene (UHMWPE).

The most often utilized bearing material that was introduced in THA to replace the unsuccessful PTFE in the mid-1960s ([Slouf et al., 2023](#)). Most hip implants design use a ceramic or metallic femoral head articulating with a UHMWPE liner. High wear resistance, toughness, durability, and biocompatibility are some of its advantageous properties ([Hasegawa et al., 2023](#)). The alarming frequency of THA failure due to wear of UHMWPE and the discharge of polyethylene particles which would potentially lead to implant loosening and peri-implant osteolysis meant that further improvement had to be done on this material ([Bistolfi et al., 2021](#)).

2.2.3.1.3.4 Highly crosslinked polyethylene (HXLPE).

Originally introduced in the 1990s to enhance the wear resistance of UHMWPE, HXLPE liners have been extensively studied. Recently Fransen et al. ([Fransen et al., 2023](#)) confirmed that their use in combination with 36 mm femoral heads ensures safe mid-term

clinical outcomes. These findings display strong coherence with the work of Devane et al, (Devane et al., 2017) who demonstrated that after ten years following primary THA, HXLPE exhibited reduced wear compared to conventional UHMWPE. As of right now, the CoCr femoral head paired with an HXLPE liner is the most utilized bearing coupling since it has been shown in numerous longitudinal inspections to exhibit increased durability, decreased wear rates, and good safety (Thejeel & Endo, 2023).

2.2.3.2 Functional Classification.

When implanted into the human body, biomaterials elicit distinct tissue responses depending on their material properties. The tissue interaction mechanism is dictated by the biological reaction to the implant surface. Accordingly, biomaterials are classified based on their biological response and interaction with bone, categorizing them as bioactive, bioinert, or bioresorbable.

2.2.3.2.1 Bioinert biomaterials.

Bioinert biomaterials demonstrate limited reactivity with the surrounding body tissues when implanted in the body system. Typical examples include stainless steel, titanium, alumina, zirconia, and UHMWPE. These often become encapsulated by fibrous tissue after implantation, and their functional performance is largely dependent on the close apposition of bone to their surface, facilitating successful tissue integration.

2.2.3.2.2 Bioactive biomaterials.

Bioactive materials exhibit direct interaction with surrounding bone tissue and, sometimes, soft tissue upon implantation. This interaction is governed by an ion-exchange process between the implant and physiological fluids, leading to the development of a biologically active carbonate apatite (CHAp) layer on the surface of the implant. This layer closely resembles the chemical composition and structure of the bone's mineral phase, promoting osseointegration. Representative examples include synthetic hydroxyapatite $[\text{Ca}_{10}(\text{PO}_4)_6(\text{OH})_2]$, glass-ceramic, and bioglass, each designed to enhance biological compatibility and long-term implant stability.

2.2.3.2.3 Bioresorbable biomaterials.

These biomaterials, upon implantation in the human body, undergo gradual dissolution and are progressively supplanted by newly regenerated tissue, such as bone. Typical examples of such biomaterials include tricalcium phosphate $[\text{Ca}_3(\text{PO}_4)_2]$ and polylactic-polyglycolic acid copolymers.

The functional categorization of biomaterials is illustrated schematically in **Figure 2. 12**.

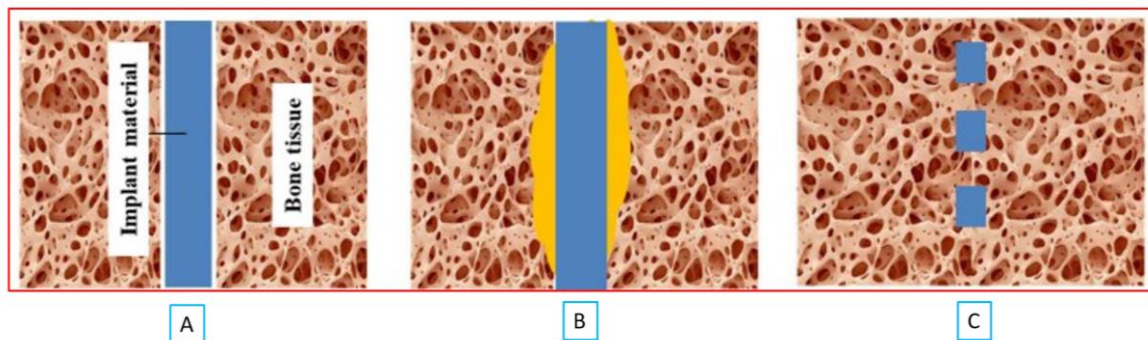


Figure 2. 12: (A) Bioinert materials form a fibrous tissue layer due to minimal interaction. (B) Bioactive materials create a chemical bond at the interface with bone. (C) Bioresorbable materials facilitate tissue regeneration and gradually degrade, allowing bone healing.

2.2.4 Analysis of the major hip prosthesis failure modes and mechanisms in service.

Despite THA being a highly successful operation and described as one of the 20th century's most significant medical breakthroughs ([Eisler et al., 2002](#); [Mahomed et al., 2003](#); [Wroblewski, 1984](#)), problems develop over time that require revision surgery 10-15 years after the first implant to replace the original components ([De Steiger et al., 2018](#); [Varacallo et al., 2025](#)). However, with the increase in life expectancy due to improved health care systems and young people undergoing THA, the current goal is to develop prosthetic hip joints that are mechanically reliable and wear resistant to ensure they can endure the demands of active patients for a minimum of 25 to 30 years regardless of age ([Khanna et al., 2017](#)). For this goal to be achieved, the occurrence of THA failure that limits its durability and useful life must be minimized. This is possible through a thorough analysis

of failure mechanisms to identify the failure's causing factors and then implement measures to reduce the incidence of these failures.

2.2.4.1 Fatigue failure mechanism.

One of the mechanisms of hip implants failure is fatigue failure. The femur experiences load from body work and physical activities which induces torsional and bending stresses in a hip prosthesis' femoral component. These recurrent and fluctuating stresses in the hip implant gives rise to fatigue fractures or deformations (Delikanli & Kayacan, 2019). The fatigue behaviour of metallic materials mostly stainless steels, and titanium alloys used in hip implants has been extensively studied, and its relation to their microstructure, surface, and corrosion properties have been reported. Hip implants are designed to withstand a minimum of 15 million cycles considering 15 years of service life with a million cycles each year (Corda et al., 2023). Despite these numerous studies, the prevalence of fatigue failures still remains a significant problem.

2.2.4.1.1 Fatigue failure mechanism in Stainless steel hip implants.

Stainless steel alloys especially 316L with the “L” designating low carbon content (up to 0.03%), has been widely used for hip implants due to its corrosion resistance, good mechanical properties, ease of fabrication, and cheaper price as compared to titanium alloys (Moghadasi et al., 2022; Singh et al., 2018). The in vivo performance of hip implants fabricated from this biomaterial has heavily suffered from fatigue failure mechanisms as reported in various studies.

The failure investigation of a cemented femoral stem hip prosthesis (SS316L double-tapered), shown in **Figure 2. 13**, by Marques et al. (Marquez et al., 2023) found that fatigue was the primary cause of failure.



Figure 2. 13: (a) Side view presentation of the failed SS316L femoral implant's neck region. (b) Higher magnification diagonal view displaying the initiation point (red

arrow) as well as the “undulating” propagation of beach marks from the failure initiation site to the femoral stem’s central region(Marquez et al., 2023).

This fatigue mechanism was initiated by a manufacturing surface defect taking the shape of an indentation at the femoral stem’s neck surface and propagated transversely down the inside section of the prosthesis stem. The transversal orientation and propagation of the implant’s failure beach markings from the stem’s edge, indicated that the implant failed due to low cycle stresses probably from walking within the 4 years period it survived in the body.

Similarly, Roffey (Roffey, 2012) in his investigation of a forged M30NW high-nitrogen nickel- free stainless steel femoral implant that failed just after 4.5 years of use, identified high cycle fatigue evidenced by closely spaced beach marks resulting from surface defects as a cracking mechanism. As shown in **Figure 2. 14**, the two cracks initiated from diametrically opposite points within introducer slots designed to help in implantation of the prosthesis which subsequently propagated and resulted in the failure of the implant.

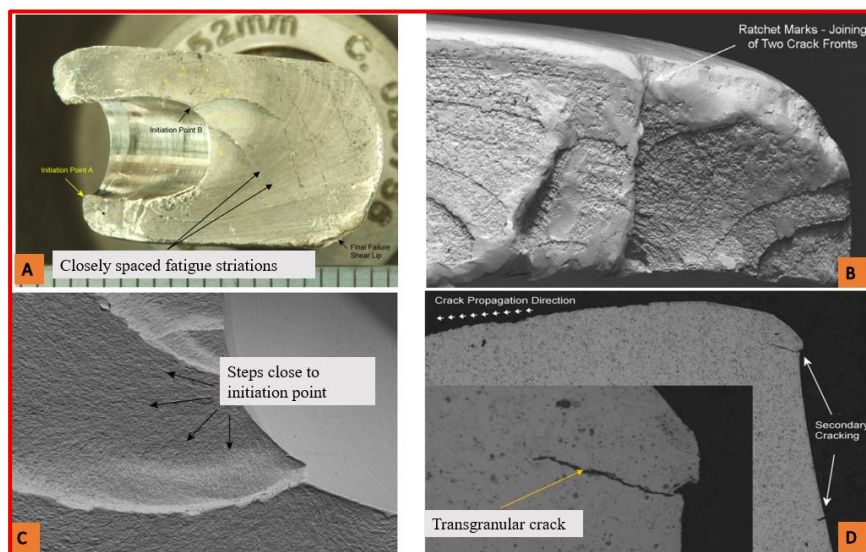


Figure 2. 14. (A) Beach marks and two crack initiation spots visible on the fractured surface. (B). Merging of the second fatigue crack and the failure initiation spot A forming a ratchet mark. (C) The steps around initiation point B provide information about variations in loading and stress amplitude. (D) Cross-sectional view of initiation spot A’s surface: a fatigue-typical transgranular crack with detailed secondary cracking near the fracture surface in the absence of metallurgical defect and any localised form of corrosion (Roffey, 2012).

2.2.4.1.2 *Fatigue failure mechanism in Ti-6Al-4V alloy hip implants.*

Fatigue failure mechanism is also a significant concern for Ti-6Al-4V hip implants. This section discusses some selected cases of Ti-6Al-4V fatigue failure mechanisms in literature.

Chao and Lopez ([Chao & López, 2007](#)) at CENIM laboratory investigated the failure of three Ti-6Al-4V alloy femoral prostheses of the same design that broke at the neck's junction radius with the collared device shortly after two years of use. The study revealed that the fracture of the prostheses was produced by a fatigue process that was initiated at an area indicated by the white arrow in **Figure 2. 15** (A and B). The fracture features in **Figure 2. 15** (C and D) with enlarged inserts in **Figure 2. 15** (E and F) presents striation marks a characteristic figure of fatigue failure. According to this failure analysis, the fracture initiated on the lateral side of the neck, propagated lateromedially through the cross section, and ultimately resulted in overload failure of the remaining cross section along with severe plastic deformation. Fatigue tests conducted using a two-point strategy method on an MTS 810 servo-hydraulic testing machine at 5×10^6 cycles as per ISO 7206 standard ([Subcommittee., 2010](#)), justified the findings of this study.

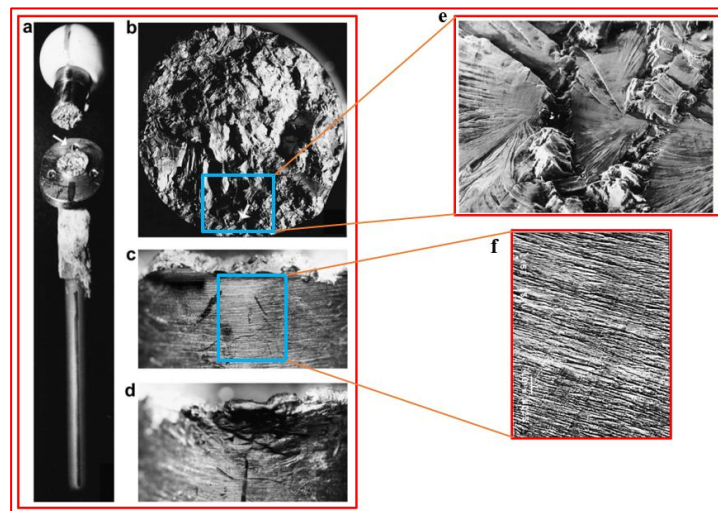


Figure 2. 15. Retrieved Ti-6Al-4V hip implant details. (A) The implant's medial view with the crack initiation point indicated by the white arrow. (B) Normal view of the fractured neck surface with the initiation site indicated by the white arrow. (C & D) Lateral and medial view of the fractured surface respectively. (E) Higher magnification insert displaying the discontinuous nature of the fatigue crack growth. (F) Higher magnification insert of the fracture surface depicting fatigue striations justifying the bottom to up crack growth ([Chao & López, 2007](#)).

All these studies show how prosthesis performance is heavily affected by fatigue failure mechanism. From these case studies, two main points should be highlighted; (1) manufacturing-related flaws can exacerbate damage under dynamic loads, reducing stiffness and fatigue life of the hip prosthesis. (2) poor design choices increase the risks for fatigue failure. Efforts have been made to optimize the hip implant design to prevent premature failure due to fatigue. For example, Corda et al. (Corda et al., 2023) conducted a fatigue failure analysis on different hip profiles and compared the outcome for various combinations of materials. The trapezoidal and circular stem design with a profile having increased arc length and diameter had a 2.7% and 2.6% higher safety factor respectively. This is in agreement with another study which found the trapezoidal cross-shaped femoral stem as an optimum design with equivalent von Mises stress of 258.1MPa, equivalent strain of 0.004, total deformation of 0.24mm and frictional stress of 0.362 MPa which were lower compared to the elliptic and oval cross-section design (Göktaş et al., 2022). Chethan et al. (K.N et al., 2019) had a similar finding which concluded that circular and trapezoidal-shaped implants exhibit a minimum of 27% lesser von Mises stresses compared to the elliptical and oval-shaped stem implants.

2.2.4.2 Corrosion failure mechanism.

The body's fluids are generally made up of different cations (usually Ca^{2+} , K^+ , Mg^{2+} , Na^+), anions (Cl^- , PO_4^{3-} , HCO_3^-), dissolved oxygen, and organic compounds including proteins (David F. Williams & Williams, 2013). This composition makes body fluids highly aggressive and easily influence the onset of corrosion in hip implants. The prevalence of corrosion in hip prosthesis have been well documented by implant retrieval studies due to these highly aggressive body fluids (Gilbert et al., 2012). The types of corrosion that are pertinent to the currently used alloys are: pitting, fretting, stress-corrosion cracking, galvanic, corrosion fatigue, and crevice corrosion.

Pitting corrosion. Pitting is the most common form of corrosion arising from the breakdown of passivating oxide film, which can be enhanced by the presence of proteins in the tissue fluid and serum. A deep-penetrating attack, the pit, with a diameter of less than a few millimetres, is produced by this intense localised corrosion. The pits are often solitary and vary in number from a few to several hundred per square meter (Pedefferri, 2018).

Crevice corrosion. This is a type of localised corrosion brought on by the presence of a geometrical fissure (crevice). It occurs in stagnant areas where fluid diffusion is limited,

often at the modular taper interfaces of the hip prosthesis. The presence of chlorides and reduced pH in the crevice disrupt the passive film leading to significant corrosive attack ([Bundy, 2008](#)). 316L stainless steel is most susceptible to this type of corrosion and the occurrence of corrosion on the bone plate and screws made of 316L at the interface between the screw heads and the countersink holes is a common feature ([Gao et al., 2022](#)).

Galvanic corrosion. Galvanic corrosion in hip implants occur when two different metals with varying electrochemical potentials come into contact within the body, leading to accelerated degradation of the less noble metal. A typical example of galvanic corrosion was observed on the taper junction of the modular hip implant designs which utilized CoCrMo femoral heads coupled with Ti-6Al-4V ELI femoral stem ([Collire et al., 1991](#); [Yerby et al., 1996](#)). Galvanic corrosion also occurs when metals processed through different methods (cast vs Wrought CoCr) are joined together.

Fretting. Similar to crevice corrosion, fretting corrosion occurs at the interface between two different surfaces that brings them into close contact. It is a typical mechanical-assisted corrosion (MAC) behaviour that causes the contact interface to undergo a dual degradation of fretting wear and local corrosion ([Mali et al., 2021](#)). Synergetic effect of local corrosion and wear when interfaces rub against each other from forces applied during the gait cycle will cause severe material damage and loosening or even failure of the implant ([Zhu et al., 2020](#)). An example of fretting corrosion occurs at the screw/plate interface, or the femoral head/ femoral stem taper junction.

Stress corrosion cracking (SCC). This is a form of corrosion that involves both corrosive attack and mechanical static tensile stresses (either applied or residual) ([Khalifeh, 2019](#)). It is a gradual process and can take prolonged periods of time for both crack initiation and propagation. SCC is a significant concern for hip implants, particularly because it can lead to premature failure of the implant.

Corrosion fatigue (CF). Being a load bearing implant, the combination of cyclic loading on the prosthesis and the corrosion phenomenon results in a failure mechanism known as corrosion fatigue. Contrary to simple fatigue where stresses below the endurance limit do not cause any damage to the material, in corrosion fatigue the material's resistance to fatigue is degraded due to corrosion sharply decreasing the endurance limit making even high strength materials more susceptible to corrosion fatigue failure ([Ahmad, 2006](#)).

2.2.4.2.1 Corrosion failure mechanism in stainless steel hip implants.

Different stainless steel hip prostheses have prematurely failed and hence removed from the body system due to corrosion. This section discusses some of the retrieval studies that determined corrosion as the mechanism that caused failure and explores the event or series of events that caused the corrosion failure mechanism to particularly become operative.

Failure investigation of a 316L stainless steel Austin Moore design hip prosthesis (shown in **Figure 2. 16**) that only served for 8 months in a 53-year-old man by Sivakumar et al. (M. Sivakumar, Kamachi Mudali, et al., 1995), revealed the detrimental impact of corrosion on the performance and lifespan of hip implants.

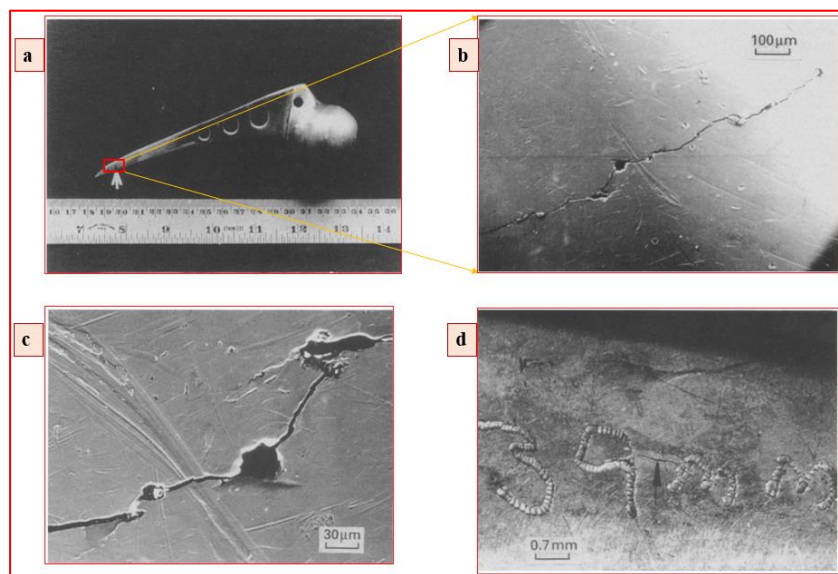


Figure 2. 16. (A). Retrieved Austin Moore design hip prosthesis. The arrow indicates a hairline crack on the proximal end of the implant. (B). A stereomicroscope image of the crack on the femoral stem on the Austin Moore design hip implant. (C). Scanning electron microscope image presenting a higher-magnification view of the crack on the failed implant. It can be clearly seen that the crack was transgranular, unbranched, blunted and also associated with the pits. (D) Stereomicrograph with the black arrow showing microcracks at the imprinted characters “39 mm” on the failed implant (M. Sivakumar, Kamachi Mudali, et al., 1995).

The failure analysis found that the 316L hip implant failed due to pitting corrosion mechanism whereby the developed pits acted as stress raisers resulting in the initiation of microcracks in the material. These cracks mostly developed on the edges of the implant and around the area where the implant number was printed. Elemental composition analysis

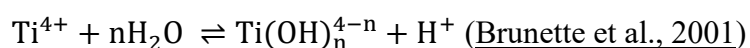
revealed that the 316L stainless steel used for this implant had a molybdenum content of 1.24% which is 0.76% less than the ASTM standard limit of 2% (F04. Committee). A lot of sulphide inclusions were also identified in this 316L stainless steel. Furthermore, potentiodynamic anodic polarization study found that this material had a pitting potential of 240 mV (versus SCE) which is lower than the ASTM recommended pitting potential of +365 mV (versus SCE) for 316L stainless steel (ISO). It was therefore concluded that the implant was susceptible to pitting attack due to the low molybdenum content as well as excess sulphide inclusions which play a critical role in initiating pits in stainless steels.

Similarly, eleven retrieved Charnley hip implants design fabricated from 316L stainless steel that failed prematurely revealed excessive corrosion that occurred in layers (Walczak et al., 1998). Through elemental composition analysis, Nickel, which makes about 13% of this alloy, was consistently absent in the 11 retrieved implants, suggesting that it had been released into the surrounding body tissues through corrosion. Several studies have also reported about Nickel release with regards to 316L stainless steel implants (Guerra-Fuentes et al., 2015; Marcomini et al., 2014; Thapa et al., 2015).

Over 90% implant failures made by the 316L stainless steel are caused by the crevice and attacks of pitting corrosion (M. Sivakumar, Kumar Dhanadurai, et al., 1995). This localised leaching of corrosion and metallic ions attacks from the implants require improvements in the corrosion resistance of 316L stainless steel by modifying the surface or bulk alloying through the addition of elements like titanium, boron, and niobium in the stainless-steel matrix to improve its corrosion resistance (Ali et al., 2022). However, most designs use this alloy for temporary fixation devices like screw plates and prefer titanium alloys for permanent replacements like hip prosthesis.

2.2.4.2.2 Corrosion failure mechanism in Ti-6Al-4V alloy hip implants.

The stresses of cyclic loading erode and fracture the passivated protective layer on the Ti-6Al-4V alloy, the mostly used material for hip prosthesis, resulting in the development of microfractures. The metal's surface potential consequently shifts, becoming extremely negative, and the oxides continue to fracture and repassivate, changing the chemistry of the crevice solution leading to de-aeration and a fall in pH (Brunette et al., 2001). This has two detrimental effects on titanium's ability to withstand corrosion: Initially as per the below reaction, the Ti ions hydrolyse water:



Hydrochloric acid (HCl) is produced when these generated H^+ ions react with chlorides present in the serum. Through the formation of grayish chloro-complexes, HCl obstructs the passivation process resulting in the loss of the protective layer and increased corrosion of the prosthesis metal. Secondly, hydrogen embrittlement due to the elevated H^+ ions concentration causes microcracks to develop in the material (Brunette et al., 2001).

Manish et al. (Paliwal et al., 2010) analysed the mechanism of failure for a Ti-6Al-4V modular femoral hip implant that fractured after 38 months of service. As shown in **Figure 2. 17**, they found that failure was initiated by the fretting corrosion which caused subsurface microcracks. The crack propagated due to corrosion-assisted fatigue process up to the point the implant's material yield strength was reached leading to catastrophic failure.

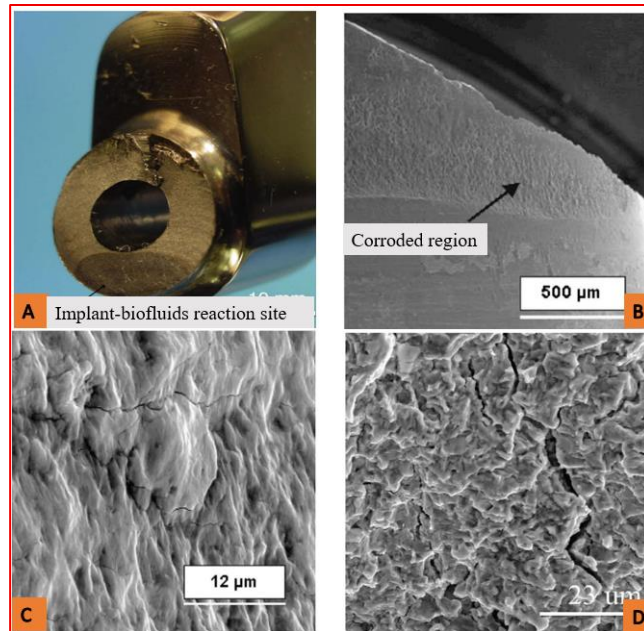


Figure 2. 17. (A) The fractured region of the implant with a funnel-shaped, corrosion product-filled crack in a meniscus-like region (red arrow) that is an indirect sign of corrosion. (B) SEM back-scattered side view of the fractured proximal taper with the corroded area indicated by the black arrow. (C) The SEM back-scattered illustration depicting the initiation point of the corrosion. (D) Latent microcracks close to the fracture surface evidence of the profound hydrogen penetration under stress and the implant's hydrogen embrittlement (Paliwal et al., 2010).

The modular design is associated with metal-to-metal interaction and with the implant functioning in a highly corrosive environment, inclines the implant to fretting and corrosion failure mechanisms.

Pimenta et al. (Pimenta et al., 2021) investigated the cause of failure for a Ti-6Al-4V hip prosthesis that prematurely failed after 2 years in service (see **Figure 2. 18**). This study exposed how an implant suffers from the constituents of the body fluids like plasma, amino acids, sodium, and chlorine through corrosion (Manivasagam et al., 2010). Dense micro-pitting in a region closer to the fracture region indicated significant corrosion in the neck region of the implant which led to its subsequent failure.

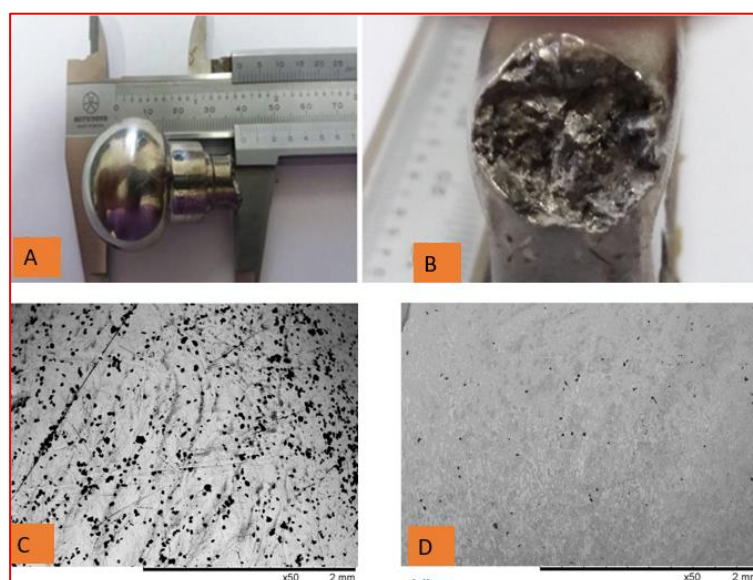


Figure 2. 18. (A) Femoral head detached from the femoral stem prosthesis. (B) Fractured surface of the femoral neck. (C) Several pits on the external implant surface in the vicinity of the fracture, an indication of severe pitting corrosion. (D) Fewer Pits on an external surface for a region far from fracture indicating little corrosion in this area (Pimenta et al., 2021).

ASTM F 136 (F. Committee) standard, emphasizes that for orthopaedic applications, Ti-6Al-4V should have a Widmanstätten microstructure, a fine dispersion of the α -phase surrounded by the β -phase, however, microstructural analysis of this implant revealed the presence of the α -phase at prior β -grain boundaries in some regions. This non-compliance probably due to poor control of the cooling rates during manufacturing greatly reduced the material's resistance to corrosion. Several scholars have also reported corrosion problems with Ti-6Al-4V alloy in implant application (Rodrigues et al., 2009; B. Sivakumar et al., 2011; Smith & Gilbert, 2020) prompting research interest in alternative titanium alloys with improved resistance against corrosion. These studies have revealed that surface treatment to reduce fretting, research on materials with a superior corrosion resistance and

manufacturing methods to ensure metallurgical purity and improvement in the microstructure can help deal with these kinds of failures.

2.2.4.3 Wear failure mechanism in hip implants.

Wear of an implant during service is the primary limiting factor for a THA prosthesis' long-term survival, and it is described as an undesirable progressive loss of material in particulate form as a consequence of surface-to-surface interaction ([Bhushan, 2013](#)). It is the main cause of material waste and loss of mechanical performance of the implant, and in the body environment, wear debris gets washed by moving body fluids, hence it is always accompanied by the tribocorrosion phenomenon ([Hallab, 2016](#); [Muruve et al., 2017](#)).

There are two main areas of concern regarding the wear of a hip implant. The first is the bearing surfaces (articulating surfaces) (labelled C in **Figure 2. 19**) wherein the rotating femoral head and the acetabular components-primarily the polyethylene liner-create friction that wears down the bearing surfaces and expedites corrosion [121]. The second area is the interface of the prosthesis' components with the host bone or between the connective components where due to repetitive forces during movement, there is a subtle relative motion between the components that leads to fretting wear (marked A,B,C and D in **Figure 2. 19**) ([Royhman et al., 2016](#)).

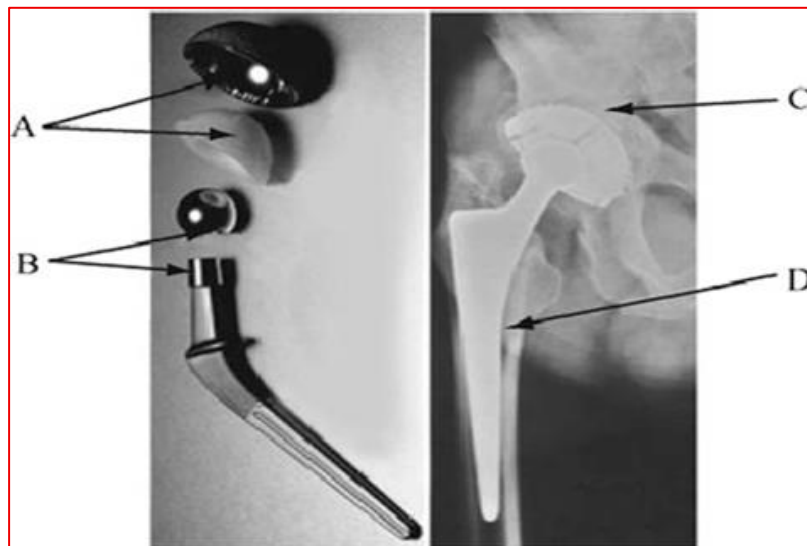


Figure 2. 19. Wear focus areas of a hip implant ([C. T. Wang et al., 2012](#)).

Most hip implants today comprise of a titanium femoral stem, a cobalt chrome prosthetic head, an acetabular cup primarily composed of titanium, and a plastic liner mostly from polyethylene positioned between the acetabular cup and the prosthesis' femoral head to

create a smooth, low friction gliding surface (Sheth et al., February 2024; Tapscott & Wottowa, 2024). Polyethylene wear is the main concern in THA, and a significant number of patients are subjected to this mechanism that leads to prosthetic failure (Boulila et al., 2008). Apostu et al. (Apostu et al., 2018) emphasized that the resulting particle wear debris is linked to local aggressive biological reaction resulting in periprosthetic bone loss and subsequent implant's aseptic loosening. As shown in **Figure 2. 20**, the mechanisms of wear experienced in a hip implant change during movement.

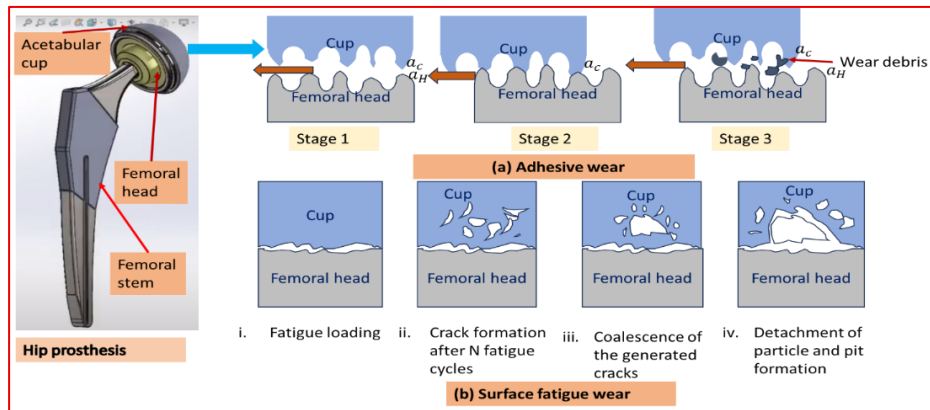


Figure 2. 20. Diagram illustrating hip implant's wear phenomena and different wear mechanisms, adhesion as well as surface fatigue wear where a_c is the asperity of the cup and a_H is the asperity of the head.

Initially, the wear mode is adhesive wear, which then changes to surface fatigue wear and then transforms to abrasive wear. This change in the dominant wear mode occurs due to variations in the material surface's properties and dynamic surface reactions caused by friction and wear itself (Dwivedi, 2018). It must be emphasized that prosthesis wear occurs in multiple modes, hence it is crucial to comprehend the wear process in each mode to avoid implant failure.

In adhesive wear, type (a) in **Figure 2. 21**, wear is generated by sliding action of the femoral head against the ultra-high molecular weight polyethylene (UHMWPE) acetabular liner. Macroscopically, the acetabular liner and the femoral head's surfaces may appear smooth but at the micro scale they have asperities making them rough. When assembled in the implant and normal load applied for example during walking, few asperities contact each other, and local pressure at the asperities becomes high leading to deformation of the asperities. The relative tangential motion disperses the surface films at the asperities points of contacts leading to the formation of a bond or junction sometimes called a cold weld.

Continued sliding causes the junctions to shear and new junctions are formed. When this shearing occurs away from the interface, the small portions of UHMWPE adhere to the metallic or ceramic femoral head's surface. With further sliding, some of the adhered materials on the femoral head's surface are detached to form loose wear particles transferring back to the surface of the UHMWPE. Frequently these loose particles form groups and break away as a single entity. This break-away of materials results in the development of cracks or voids that may not be visible on visual inspection of the joint surface (A. Wang et al., 1995). **Figure 2. 21** shows damage on hip prosthetic components resulting from adhesive wear.

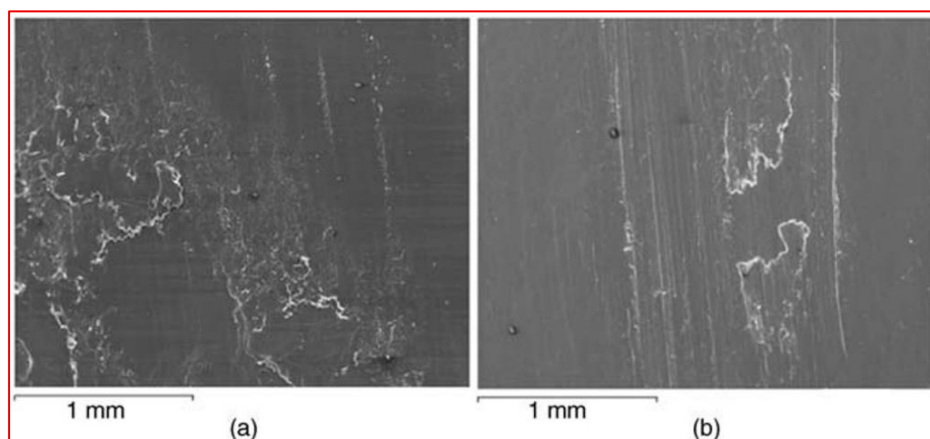


Figure 2. 21. Adhesive wear in some components of a hip prosthesis. (a) Adhesive wear in a metallic femoral head (b) Adhesive wear in a ceramic femoral head (S. Affatato & Brando, 2013).

The second wear mode is surface fatigue wear, (see **Figure 2. 22**). This wear mechanism occurs when the shear stresses or strains on the surface and subsurface exceed the fatigue limit of the soft material in the bearing surface ("14 Fatigue Wear," 1993).

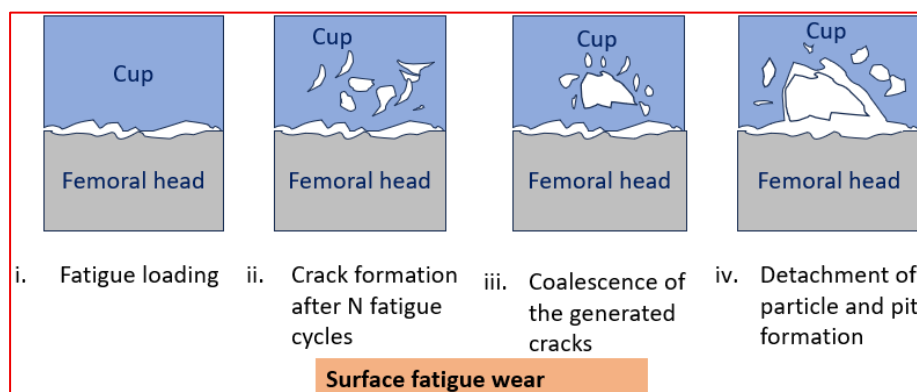


Figure 2. 22. Detailed mechanism of the surface fatigue wear in hip implants.

In fatigue wear, damage dominates on the UHMWPE since it is the weakest of the two articulating materials. Repetitive cycling loading causes subsurface cracking and delamination, which allows UHMWPE particles to be released. The induced damage ranges from microscopic, which involves tiny pits that are invisible to the naked eye, to macroscopic, which involves pits covering large areas.

The final Stage involves abrasive wear of the implant as shown in **Figure 2. 23**.

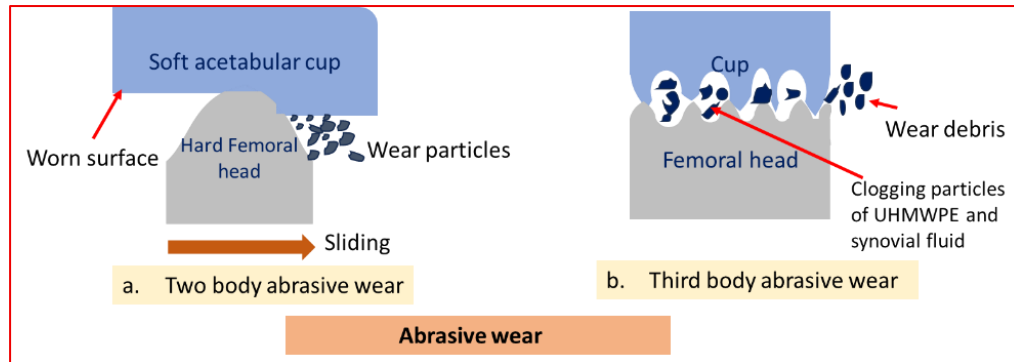


Figure 2. 23. Abrasive wear mechanism in hip implants.

Generally, abrasive wear is described as a solid surface damage that typically entails a motion between a hard and sharp material that contacts a softer material, whereby the harder material gouges into the softer material to form wear debris. Two classifications apply to abrasive wear: two-body and three-body abrasion. In two-body abrasion (**Figure 2. 23A**), debris particles are generated when the counter body's soft matrix is penetrated by the hard surface's asperities (Sarath et al., 2023). Surface roughness plays a central role in material loss and can be controlled by improving the smoothness of the interacting surfaces. However, the third-body abrasion also known as dry sand abrasive wear presented in (**Figure 2. 23B**), occurs when hard particles are embedded in the softer surface (Bheemappa et al., 2023). In this case, hard particles outside the articulating surfaces, such as polymethyl methacrylate (PMMA) debris, bone fragments, or particles from one of the contact components, become trapped between the bearing surface and act as the tougher material's asperity, hence eliminating material from both the UHMWPE and the ceramic or metal femoral head on its movement (Davidson et al., 1994). Abrasive wear damage on some hip implant components is illustrated in **Figure 2. 24**.

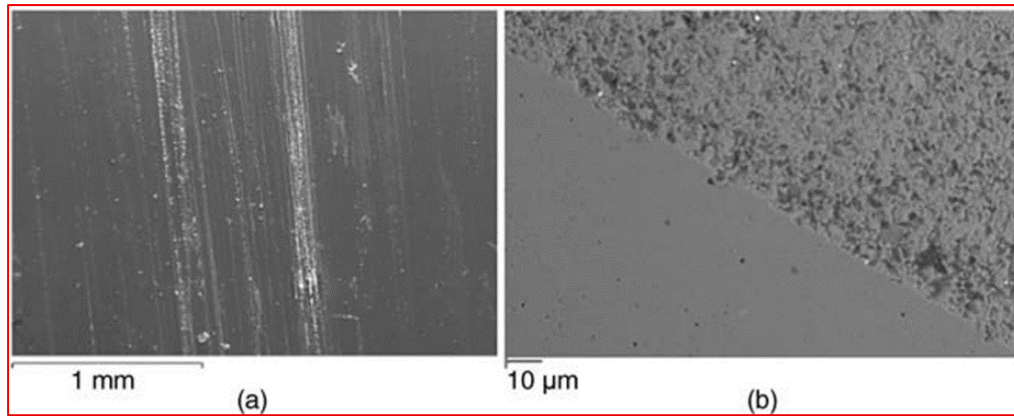


Figure 2. 24. Examples of abrasive wear in retrieved hip prosthesis. (a) SEM image of worn metallic femoral head due to abrasive wear. (b) SEM image of abrasive wear in ceramic femoral head (S. Affatato & Brando, 2013).

Naim et al. (Naim et al., 2023) highlighted that tribocorrosion is the predominant wear mechanism in hip implants because it operates in a biologically hostile environment and is exposed to a variety of dynamic loads within the human body system. This substantial coexistence of tribocorrosion and mechanical wear in hip implants is well documented in literature (Dowson & Neville, 2014). The study of Khan (Khan, 1999) demonstrated that Ti-6Al-4V has poor resistance to tribocorrosion making it susceptible to failure in the body environment.

2.2.5 Mitigation measures for failures of hip implants in service.

The studied failure mechanisms and hip joint biomechanics clearly point out the need to improve hip implants for a better performance and longer service life in the body. This section provides different ways of mitigating these failures taking into consideration the materials, design, and manufacturing approaches.

2.2.5.1 Materials selection considerations (Ti-6Al-7Nb alloy over Ti-6Al-4V alloy).

For hip implants, choosing the right materials is essential for failure prevention. A prosthesis manufactured with a material of unfavorable mechanical properties, microstructure or chemical composition is susceptible to malfunction, so it is critical to ensure that the right material has been utilized in implant's fabrication. Section 2's analysis of hip joint biomechanics makes it evident that high forces cross the hip joint in a variety of scenarios. High tensile strength, fatigue strength, yield strength, compressive strength, adequate ductility, fracture toughness, hardness, and shear strength are therefore required of the implant in addition to its superior biocompatibility (J. Li & Liu, 2015). This means that

metals, specifically metallic alloys, are a preferable choice for hip prostheses, with conventional ceramics and polymers being limited to the articulating surfaces (C. Y. Hu & Yoon, 2018).

Up to date, Ti-6Al-4V ELI, an upgraded form of Ti-6Al-4V possessing minimal levels of impurities like C, O, H, and N is frequently utilized for prosthetic hip joints. Recently Andrews et al. (Andrews et al., 2024) established that material issues contribute to approximately 48% of hip implant failures. From the failure mechanisms studied in the previous section, it is widely acknowledged that poor wear resistance, notch sensitivity, and comparatively poor resistance against corrosion are the main drawbacks of Ti-6Al-4V ELI causing solid particles and metallic ions discharge into the implant's surrounding tissue which is the main cause of the different failure modes observed in hip implants ultimately requiring revision surgery (Hall et al., 2020). According to clinical studies regarding the biotolerance of Ti-6Al-4V ELI alloy prostheses, vanadium causes cytotoxic responses, which in turn lead to neurogenic diseases (Paszenda et al., 2010). Titanium alloys devoid of vanadium as well as improved resistance to corrosion and wear for orthopaedic implant devices have been developed due to issues surrounding the release of cytotoxic vanadium ions from the Ti-6Al-4V ELI alloy. In this section, the superior properties of Ti-6Al-7Nb alloy, an alternative to Ti-6Al-4V ELI, are critically scrutinized as one of the mitigation measures for some of the observed failure modes and mechanisms in hip implants.

2.2.5.1.1 Corrosion Resistance of Ti-6Al-7Nb and Ti-6Al-4V alloys.

A material's ability to withstand corrosion is crucial for prolonging its useful life in the aggressive body system. The technical report for Ti-6Al-7Nb (Protasul 100 (Ti-6AL-7Nb) Vanadium-Free, High Strength Titanium Alloy, 1988) evaluated this alloy's resistance to corrosion relative to Ti-6Al-4V as well as pure Ti and **Figure 2. 25** shows their polarization curves at 37°C in 2 molar hydrochloric (HCl) acid (Semlitsch et al., 1995). 2 M HCl is an exact replication of the body environment to allow prediction of the in vivo corrosion behaviour.

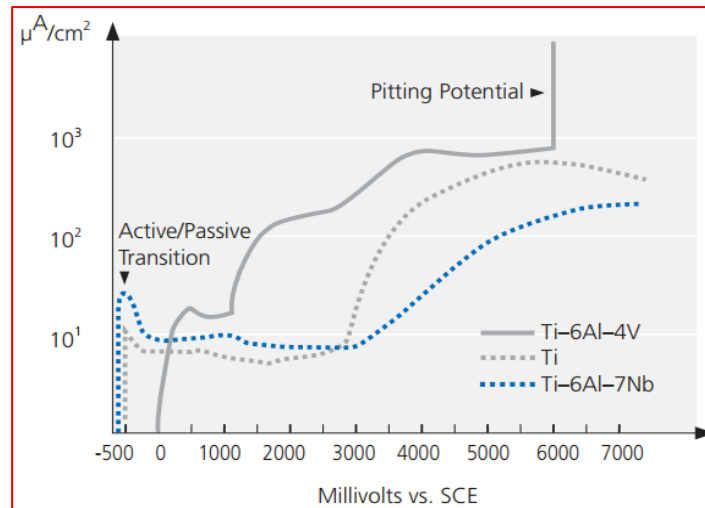


Figure 2. 25. Polarization curves displaying superior resistance to corrosion for pure Ti, and Ti-6Al-7Nb while Ti-6Al-4V experiences a break away pitting potential at 6000 millivolts directly translating to seepage of metal ions in the human body system (*Protasul 100 (Ti-6Al-7Nb) Vanadium-Free, High Strength Titanium Alloy*, 1988).

On the graph, the classic active-passive transition can be observed in unalloyed titanium and Ti-6Al-7Nb at -500mV with steady, passive behaviour up to a minimum of +3000mV. Pure Ti and Ti-6Al-7Nb resist corrosive damage, whereas Ti-6Al-4V displays a significant rise in current at a pitting potential of 6000 mV. These findings affirm Ti-6Al-7Nb's excellent corrosion resistance in reducing acid environments over Ti-6Al-4V.

Simpson (J, 1986), tested the Ti-6Al-7Nb, Ti-6Al-4V, as well as Cp-Ti's pitting potential against a saturated calomel electrode (SCE) at 37°C in a hostile environment using 2M HCl as a reducing acid. The findings are presented in **Figure 2. 26**. The ground-breaking findings reveal that Ti-6Al-7Nb has a pitting potential greater than +10.0 volts, equivalent to that of pure titanium and significantly better than Ti-6Al-4V alloy's + 5.9 volts. According to these results, in contrast to Ti-6Al-4V, the protective passivated film of Ti-6Al-7Nb offers superior resistance to degradation under an aggressive reducing acid environment.

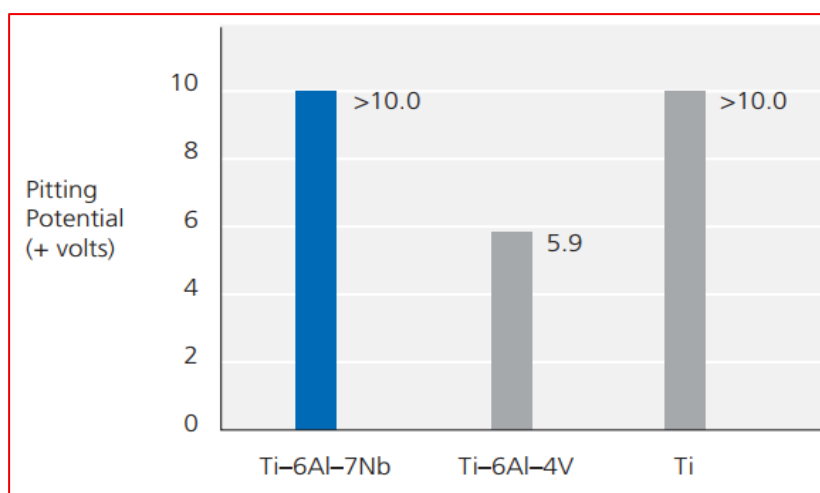


Figure 2. 26. Results of a comparative study on the pitting potential of Ti-6Al-7Nb, Ti-6Al-4V, and pure Ti at 37 °C in 2M HCl (J, 1986).

Ti-6Al-7Nb samples with specimen size $70\text{mm} \times 10\text{mm} \times 2\text{mm}$ were also tested by the four-point bend (FPB) test to evaluate their stress cracking resistance and sulphide stress cracking resistance in an aggressive NACE TMO177 solution for 760 hours as per the ISO 7539-2 standard (J, February 3, 1990). Sodium chloride as well as glacial acetic acid are present in saturation with bubbling hydrogen thereby making this test solution extremely aggressive. Following 760 hours of subjection to the aggressive NACE test solution at 24 °C the samples did not reveal any damage under high bending loads and extreme reducing environment.

Based on these studies, it is evident that Ti-6Al-7Nb is more resistant to corrosion unlike Ti-6Al-4V and retains its structural integrity upon exposure to highly corrosive environments which is linked to a stable, very dense and passive layer of rutile-like structure that offers the underlying alloy more protection (Khan, 1999; S. Y. Yu et al., 2001). Another contributing factor to Ti-6Al-7Nb's enhanced corrosion resistance stems from the strong Ti and Nb covalent bonding forming a stable Nb_2O_5 oxide, which improves the protective layer's stability on the alloy surface (S. Y. Yu et al., 2001).

2.2.5.1.2 Friction and Wear of Ti-6Al-4V and Ti-6Al-7Nb alloys.

Various researchers have closely studied Ti-6Al-4V and Ti-6Al-7Nb's wear and friction properties. Fellah et al. (Fellah et al., 2013) evaluated Ti-6Al-7Nb wear characteristics under a wear test that replicated friction for the use of total hip prosthesis in compliance with ASTM G99-95a, ISO 7148, and ASTM G133-95 standards. They concluded that, the friction coefficient rises with increased sliding distance and that Ti-6Al-7Nb's resistance to

wear is lower relative to Ti-6Al-4V. Miura-Fujiwara et al. (Miura-Fujiwara et al., 2015), investigated wear and friction properties of Ti-6Al-7Nb and pure Ti in synthetic saliva at 310K and deionized water, in order to examine the wear surface's microstructural characteristics and to comprehend the wear mechanism. Adhesive wear was reported as the sample's primary wear mechanism and the increased static load caused the reduction in Ti-6Al-7Nb volume damage.

Tribocorrosion, the broad term for the tandem action of wear and corrosion, is the primary root of many failure mechanisms. Therefore, examining the synergetic effects of corrosion and wear on Ti-6Al-7Nb is crucial. Khan (Khan, 1999), investigated the tribocorrosion resistance of Ti-6Al-4V and Ti-6Al-7Nb, in body simulated fluids (BSF). They found that the total deterioration when wear and corrosion processes coexist is higher for Ti-6Al-4V and lower for Ti-6Al-7Nb.

Figure 2. 27 shows a schematic representation of tribocorrosion in Ti-6Al-4V and Ti-6Al-7Nb alloys.

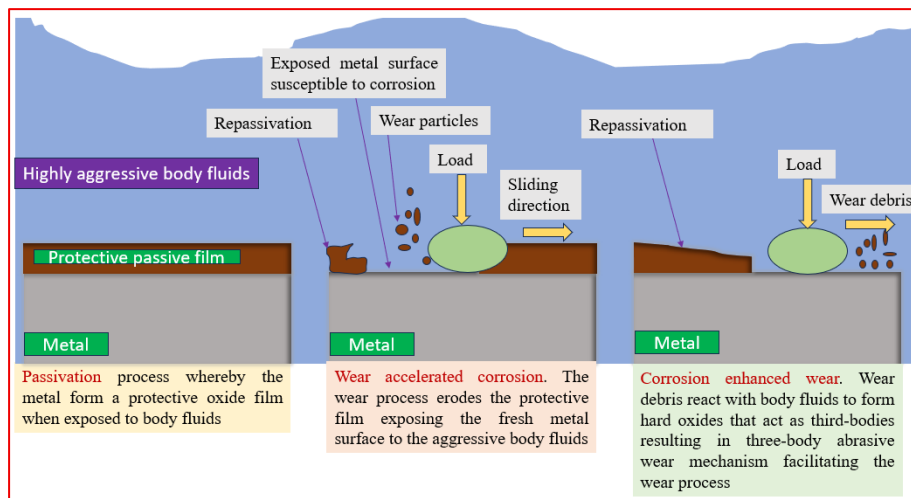


Figure 2. 27. Schematic presentation of the passivation and repassivation processes during tribocorrosion.

Contrary to ordinary wear process, which is primarily determined by the material's hardness, the key elements in tribocorrosion are the passive film's susceptibility to breaching and subsequent repassivation in the surrounding environment. The material composition, properties of the metal's passivated surface protective layer, and the surrounding environment greatly influence the repassivation process. Hence, the chemical makeup and the hardness of the repassivated protective layer will be altered. Thus, the

enhanced Ti-6Al-7Nb's tribocorrosion resistance stems from its harder surface oxides than those of Ti-6Al-4V which makes Ti-6Al-4V less resistant to tribocorrosion.

In summary, the Nb₂O₅ oxide protective layer formed on Ti-6Al-7Nb's surface is extremely hard and together with a minimal friction coefficient contributes massively to the observed improved wear resistance for Ti-6Al-7Nb ([Aniołek et al., 2022](#); [Aniołek et al., 2019](#)). Thus, the observed degradation of Ti-6Al-4V implants as a result of tribocorrosion can be significantly reduced by employing the resistant Ti-6Al-7Nb.

2.2.5.1.3 Biocompatibility of Ti-6Al-4V and Ti-6Al-7Nb.

The main factor that decides the acceptance of the material in the body system is biocompatibility. A material is deemed biocompatible if it does not elicit adverse reactions upon implantation in the biological system.

Comparative fixation study of Bozoglan and Dundar ([Bozoglan & Dundar, 2021](#)) investigated bone-implant connections (BIC) of the alloys Ti-6Al-7Nb and Ti-6Al-4V in rats employing two different surface morphology techniques: sandblasting and acid etching, and resorbable blast material. Both techniques reported a greater bone-implant connections ratio in Ti-6Al-7Nb than Ti-6Al-4V. This shows that Ti-6Al-7Nb confirmed excellent biocompatibility in bone cells, suggesting that it would provide a superior material option for hip replacements.

The human hip joint prosthesis retrieval analyses conducted by Zweymuller et al. ([Zweymuller, 1988](#)), had a striking observation based on osseous ingrowth developed on the prosthesis surface indicating Ti-6Al-7Nb's incredible biocompatibility. This formation of osseous tissue along the length of the stem provides secondary implant stabilization and contributes much to mitigating some failure mechanisms.

The studied literature addressed concerns about vanadium toxicity in the alloy Ti-6Al-4V and contrasted its behaviour to that of Ti-6Al-7Nb which replaced the cytotoxic vanadium with a more biocompatible niobium as a β -stabilizer. Ti-6Al-7Nb alloy's improved cellular response with reference to Ti-6Al-4V suggests that niobium is less toxic. Several in vivo and in vitro investigations that have been undertaken on cell viability, proliferation, morphology, and dissemination all support this assertion ([Challa et al., 2013](#); [S. P. L. Kumar & Avinash, 2019](#); [Lavos-Valereto et al., 2001](#)). The surface oxide investigation by Tamilselvi et al. ([Tamilselvi et al., 2006](#)) determined that the TiO₂ oxide layer generated on

unalloyed titanium is not as chemically stable as the mixed oxide film passivated on the Ti-6Al-7Nb alloy.

Based on this review, Ti-6Al-7Nb, an alloy incorporating niobium, is recommended to be the focus for THA because of its enhanced resistance to tribocorrosion, superior resistance to corrosion, and excellent biocompatibility. Superior osseointegration than Ti-6Al-4V alloy as observed by Zweymuller et al. (Zweymuller, 1988), excellent resistance to tribocorrosion as opposed to Ti-6Al-4V which suffered severe deterioration in the study of Khan (Khan, 1999), and higher resistance to pitting corrosion than Ti-6Al-4V as observed by Simpson (J, 1986) all support this assertion.

2.2.5.2 Design optimization using finite element analysis.

The finite element method (FEM), initially developed to solve challenging issues in structural mechanics, is now widely used in other fields as well including orthopaedics like hip implants. This method subdivides a structure into smaller components known as elements, each of which has a simpler geometry and is therefore much easier to analyze than the original structure. These elements are interconnected at specific points referred to as nodes. Thereafter, a series of enormous simple calculations are used to find solutions for complicated structures through approximation depending on the degree of simplification in the FE-model.

For multiple elements, the formulation of the FEM problem adopts the equation;

$$[K] d = f$$

In this context; $[K]$ represents the stiffness matrix, d is the vector expressing nodal displacements, and f is the vector that describes the nodal forces and external forces. The stiffness matrix $[K]$ defines the relationship between the known forces and the unknown nodal displacements. This matrix contains the elements' geometry and material properties.

Crucial input parameters like boundary conditions, external loads on the structure, material properties of different parts of the component, the mesh's degree of accuracy, and the utilized elements type affect accuracy of the FE-models. Elements are defined in various ways like simple one-dimensional line elements or three-dimensional elements determined by the geometry and loading conditions of the component being analyzed. 3D solid elements involving different types like tetrahedral, brick, and wedge elements are preferred elements for real 3-dimensional problems. While brick and wedge elements are better suited for

manual meshing, tetrahedral elements are more geometrically versatile and suitable for automatic mesh generation ([Zheng et al., 2020](#)). The most basic tetrahedral element is a 4-node tetrahedral element, which contains one node in each corner. However, the 10-node tetrahedral element (4-corner and 6-side nodes) has proven to be the best element for stress calculations ([Zienkiewicz et al., 2014](#)).

Through Finite Element Analysis (FEA), design engineers evaluate and achieve the optimal hip prosthesis design geometry. Simulations by means of finite element analysis evaluate hip prosthesis' durability under various loading conditions like walking and running. The CAD model of the prosthesis closely replicates the healthy femur, and using forces similar to those found in vivo, FEA assesses stress and strain distribution over the implant ([Risse et al., 2022](#)). Some of the failure modes observed arose from the surface notches and imperfections which lead to the initiation and propagation of cracks with subsequent in vivo fatigue-induced failure. Stress analysis during different activities helps identify critical implant areas, the magnitude of the maximum stresses, and necessary dimensional changes that can be made for a safe design of hip implant.

2.2.5.3 *Manufacturing considerations.*

Achieving high quality implants with desired biomechanical properties requires careful consideration of the manufacturing process. The manufactured product should replicate the implant design as much as possible. As emphasized by Andres et al. ([Marquez et al., 2023](#)), it is important to have an optimal design to enhance the performance of implants but it is also critical to use the best manufacturing approaches with high quality control measures to minimize imperfections over the implant's surface which act as stress amplifiers leading to crack initiation and subsequent fatigue failure of the implant.

Semlitsch et al. ([Semlitsch et al., 1992](#)) used Ti-6Al-7Nb alloy to produce hip implants by hot-forging. These forged parts demonstrated high strength, remarkable resistance to corrosion and superior biocompatibility with the body tissues and bony surfaces. However, a 780µm deep wear track was observed in a pin-on-disk wear test indicating its poor wear resistance ([Semlitsch et al., 1992](#)). This can be attributed to the inhomogeneous microstructure whereby stress concentration is likely to take place in coarse regions leading to subsequent fracture ([Jackson & Boyer, 2010](#)). The dimension accuracy of hot forging is also a serious concern as only small components (about 500mm) can be produced with great precision using this procedure, and larger tolerances on longer parts necessitate more

machining, making the whole process expensive (Jackson & Boyer, 2010). This implies that, if no alternative production methods are found for hip implants, there will likely be more inhomogeneous parts with poor wear resistance produced and more material wastage during machining.

Casting-produced Ti-6Al-7Nb parts are cost-effective but they do not meet the ISO 7206:4 fatigue life requirement of 5×10^6 cycles (Subcommittee., 2010). The ability to sustain cyclic loading for a predetermined amount of time is another crucial property that must be demonstrated by hip implants to prevent the observed implant fatigue failures. Casted Ti-6Al-7Nb alloy parts have a relatively coarse microstructure with several metallurgical defects like inclusions and shrinking porosities which probably contribute to their low fatigue life (Saudi et al., 2022). Saudi et al. (Saudi et al., 2022) fabricated Ti-6Al-7Nb's femoral stems using centrifugal investment casting and during the fatigue test, these stems ruptured at 5.2×10^5 cycles highlighting the significant effects of casting defects in the fatigue behavior of Ti-6Al-7Nb femoral stems. Therefore, alternative production routes need to be exploited to meet the homogeneous microstructure and biomechanical requirements of the orthopaedics industry.

Mechanical properties like low density, superior biocompatibility, and exceptional resistance to corrosion make Ti-6Al-7Nb suitable for hip implant application (Yao et al., 2011), however, for improved performance of hip implants, mimicking the graded porous natural bone structure is now a fascinating approach and intricate, near-net shapes that would enhance osseointegration while matching elastic modulus to the surrounding bone are now of great interest. Given the functional needs of near-net shapes of intricate geometries such as lattice structures, additive manufacturing presents itself as a promising method for producing Ti-6Al-7Nb alloy hip implants with precise geometric properties. Due to the drawbacks of utilizing traditional manufacturing techniques to produce hip implants, materials science researchers are exploring additive manufacturing techniques like powder bed fusion as potential solutions.

2.2.5.3.1. Metal additive manufacturing of Ti-6Al-7Nb alloy for hip implants.

Additive manufacturing (AM) is the technique of layer-upon-layer component fabrication from a computer-generated design model. Using laser, electron beam, or the application of ultrasonic vibration as energy sources, AM methods consolidate raw materials like wire, sheets, or powder into dense metallic components in a layerwise process. AM has brought

significant transformation in the realm of hip joint replacements offering several advantages compared to traditional manufacturing methods as summarized in Table 2. 3.

Table 2. 3. Comparison of additive manufacturing and traditional manufacturing techniques with critical insight on how additive manufacturing has impacted hip implant manufacturing through different characteristic properties.

Defining Factor	Conventional Manufacturing	Additive Manufacturing	Impact on the field of hip prosthesis.
Precision, and Accuracy	Limited by the capabilities of machining	High precision with process parameter optimization.	Significantly improved fit and functionality of hip prosthesis
Geometry and customization	- Limitations due to machining possibilities. - Time-consuming and costly	Complex shapes and intricate internal structures achievable due to layer-by-layer manufacturing	Easy to produce patient-specific implants mimicking the bone anatomy for improved functionality and fit.
Mechanical Properties	Depends on bulk material properties	Adjustable through AM process parameters	Tailored strength and stiffness to match the surrounding bone.
Cost	- High for customized and complex parts - Industrial systems require large space	- Relatively lower for complex parts. - Require moderate space	Implants being produced at a low cost

Material utilization (Wastage)	• Significant waste in subtractive machining	• Near-net shape products resulting in minimal waste generation	• Cost-effective and sustainable manufacturing of hip prosthesis
Surface finish	• Long post-processing chain required	• High surface quality directly achievable.	• Reduced need for post-processing
Lead time	• Long lead time when parts are complex	• Rapid prototyping and production	• Faster time-to market and patient treatment
Porosity control	• Limited control over pore shape, size and distribution	• Precise control over pore shape, size, and density	• Implants with better osseointegration and reduced stress shielding

According to ASTM F2792 standard ([ASTM, 2013](#)), AM processes are classified into Powder Bed Fusion (PBF) and Directed Energy Deposition (DED). PBF manufacturing technique comprising of electron beam melting (EBM) and laser powder bed fusion (LPBF) enables the fabrication of extremely complex parts monolithically coupled with good surface finish, granting nearly unrestricted design flexibility without the need for part-specific tooling ([Mphafudi et al., 2019](#); [Yadroitsau, 2008](#)). Producing Ti-6Al-7Nb implants with intricate and near-net shapes mimicking the natural bone using PBF technology can save a significant amount of production time, improved material efficiency due to reduced buy-to-fly ratio, reduce the implant weight, and improve the performance reliability of the hip prosthesis ([Lannunziata et al., 2022](#); [Mirzaali et al., 2021](#)). Like all AM processes, PBF techniques follow 4 major steps; (1) Design; first step which involves development of a model through a computer-aided design (CAD) software serving as a blueprint for the desired object. (2) Preparation; involves slicing of the digital model into cross-sectional layers following a desired thickness as instructed by the machine. (3) Printing process; the

focused laser or electron beam selectively scans a working plane on a powder bed, and then the powder fuses based on the CAD data of the determined slice. (4) Post-processing; the final step which refines the printed part and may involve removing support structures, cleaning, polishing, and surface treatment to achieve the desired final properties.

One of the advancements PBF technique has brought to implant manufacture is the design of predefined and controlled porosity to lower implant stiffness and enhance osseointegration (Jun Li et al., 2020; Sarker et al., 2020). The CAD model for the implant may be stochastically or randomly porous (e.g. honeycombs) as well as controlled porous implants (e.g. lattice structures). Process parameter optimization ensures that the printed part closely resembles the design model and significantly influences part quality (e.g. surface roughness). These parameters include; laser or electron beam power, scanning speed, hatch distance, and layer thickness. Volumetric energy density (VED), and Linear energy density (LED) expressed as J/mm³ and J/mm respectively provide a combined influence due to the interaction of these parameters. VED and LED are presented in equations (6) and (7) below where P is the laser power, v represents scan speed, t is the layer thickness, and s is the hatch distance.

$$LED = \frac{\text{Laser or electron beam power (P)}}{\text{Scan speed (v)}} \quad (6)$$

$$VED = \frac{P}{v \times t \times s} \quad (7)$$

Despite the fact that VED formula is the precursor path that helps to minimize surface flaws and porosity defects on the printed product, the VED formula's parameters lead to a discrepancy between the formulated VED and the actual (efficient) energy density (De Leon Nope et al., 2021; Ferro et al., 2020). According to Ferro et al (Ferro et al., 2020). an effective new VED formula, VED_{eff} (J/mm³), was generated to address this unreliability by including two additional unaccounted parameters: absorbed energy factors and material property. Presented in equation (8), where α is the material thermal diffusivity, β is absorption factor, Φ is the laser or beam spot diameter, while P and v is laser or beam power and scan speed respectively.

$$VED_{\text{Eff}} = \frac{\beta}{s\sqrt{4\alpha\Phi}} \frac{P}{\sqrt{v}} \quad (8)$$

Using these equations to optimize the laser powder bed fusion (LPBF) parameters ensure that pore shape, size, density, and distribution is controlled as per design model and superior surface properties like roughness with minimal defects are attainable.

2.2.5.3.1.1. *Electron beam melting of Ti-6Al-7Nb alloy hip prosthesis.*

Electron beam melting (EBM) is a popular powder bed manufacturing technology that has revolutionized the orthopaedics industry by producing intricate 3D prosthesis components ([Harrysson et al., 2008](#)). Schematically presented in **Figure 2. 28**, it comprises of a thermionic emission electron gun that generates a focused high energy electron beam, and a series of electromagnetic lenses to concentrate the beam on the build plate with the aid of electromagnetic coils. The LPBF process occurs in a vacuum environment to avoid oxidation which is an important consideration for highly reactive metals like titanium leading to the production of robust, high quality titanium structures.

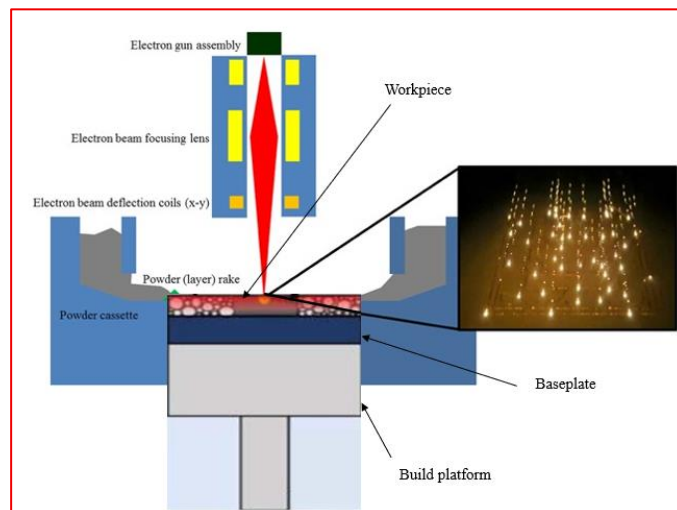


Figure 2. 28. A representation of the EBM process. The system is comprised of mobile powder rake and stationary powder hoppers as a mechanical system and the electron beam system that includes an electron gun assembly, electron beam focusing lens, and deflection coils for regulating the electron beam. Also displayed is a shot of the powder bed being scanned by an electron beam (redrawn with permission from ([Sing et al., 2016](#))).

Different Scholars have experimented the production of titanium-based intricate, near-net shape structures for hip implants using EBM. Though Ti-6Al-4V is the only alloy that has been widely analyzed, its encouraging results from the current literature suggest that Ti-6Al-7Nb implants can be fabricated so long as its processing parameters are optimized to

print a fully dense part with minimum or no defects. Harrysson et al. (Harrysson et al., 2008) used EBM to fabricate complex non-stochastic mesh structures for hip stems with the goal of reducing stress shielding (see **Figure 2. 29**).

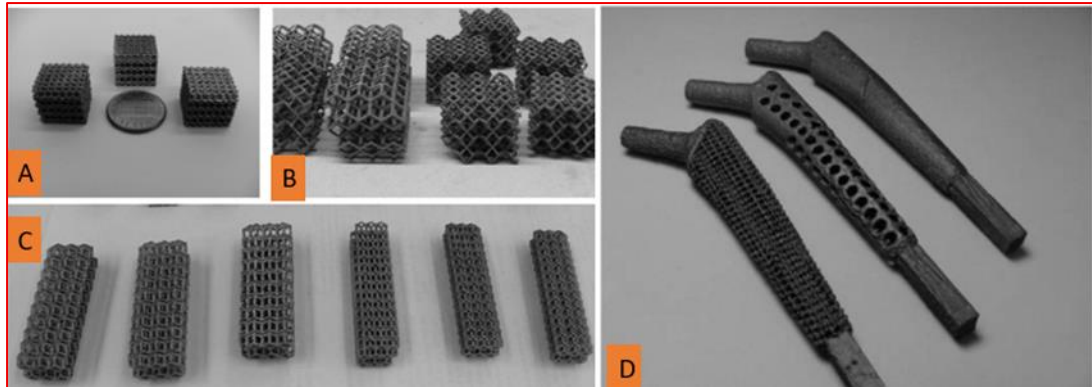


Figure 2. 29. EBM fabricated structures (A) Cubic stochastic structures with 60% porosity, (B) Compression testing specimen with 8%, 5%, and 3.8% relative densities (C) Bending specimen with relative density of 7.3% and 11.9% (D) Femoral stem prosthesis with non-stochastic mesh structures, hole configuration, and bulk solid designs (Harrysson et al., 2008).

The compression, flexural testing and hip stems weight analysis results revealed that EBM-fabricated components are light weight, have higher bending strength and an elastic modulus within the trabeculae bone range. These are promising structures for the reduction of stress shielding and reduced bone remodeling thereby preventing hip implant failure.

Considering the highly corrosive body environment, several researchers have also investigated the corrosion resistance of EBM fabricated components against conventionally produced components. The work of Bai et al. (Bai et al., 2017) determined that EBM products have better corrosion resistance in body simulated fluids (DPBSS) than wrought titanium products. This was ascribed to the microstructure of the EBM products consisting of a higher proportion of the β -phase and the lamellar α/β phase with ultra-fine grains, leading to the homogeneous dispersion of alloying elements limiting the galvanic action between the α and β phases in the material.

Despite all the exciting properties of its products, the EBM manufacturing technique has poor geometrical accuracy because of the melt pool's dimension (larger focus diameter, powder particle size, and layer thickness) (Y. J. Liu et al., 2016; Vo et al., 2018). This necessitates increased machining tolerances thereby complicating the post-processing phase

of manufacturing (Caprio et al., 2020). Moreover, product machining presents material wastage. These inherent drawbacks of the EBM process have prompted researchers to explore other PBF technologies for hip implants manufacturing, such as the LPBF technique which has demonstrated excellent geometrical accuracy when producing components from titanium-based alloys.

2.2.5.3.1.2. LPBF of Ti-6Al-7Nb alloy hip prosthesis.

Laser powder bed fusion (LPBF) utilizes thermal energy supplied by the laser beam to melt metal powders on selected regions of the powder bed and produce homogeneous components. The primary components of the SLM process are presented in **Figure 2. 30**, which includes a fiber laser, producing the laser beam, a galvanometer which controls the beam focus, and the F-theta lens controlling the laser beam's movement on the build platform. Initially, a recoater system spreads very thin layers of metal powders (20-100 μ m thickness) on the build surface (Sing et al., 2016). Then, a laser beam selectively melts this powder layer in accordance with the desired component's provided CAD model (Aboulkhair et al., 2019; E. O. Olakanmi et al., 2015).

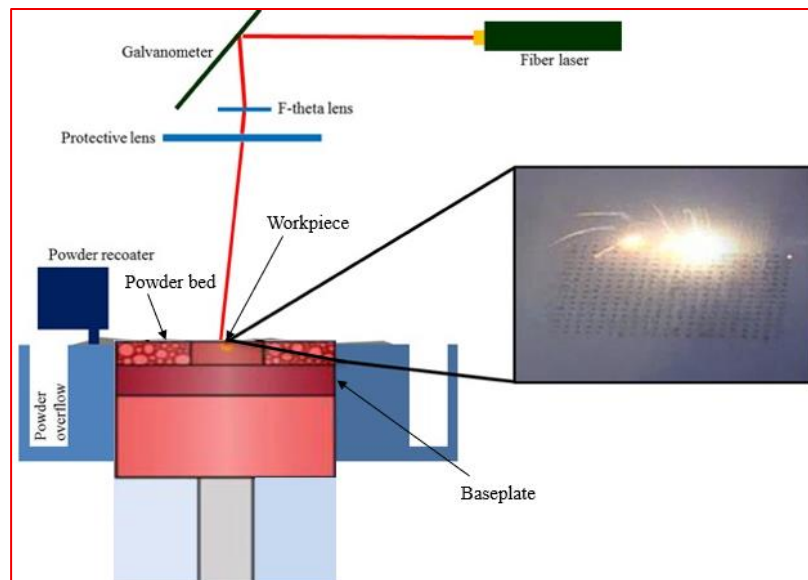


Figure 2. 30. Diagram of the major SLM process machine system. The fiber laser, F-theta lens, and galvanometer lens to regulate the movement of the laser beam make up the laser system with the mobile build platform and powder spreader making up the mechanical system. Also displayed is a snapshot of the laser scanning action on the powder bed (redrawn with permission from (Sing et al., 2016)).

Numerous investigations have been carried out to explore the potential of SLM in creating complex, customized hip prostheses using the more biocompatible $\alpha+\beta$ titanium alloy, Ti-6Al-7Nb. Hein et al. (Hein et al., 2021) produced Ti-6Al-7Nb samples by SLM for biomedical applications. It was reported that Vickers hardness, yield strength, and ultimate tensile strength of the printed parts satisfied the ISO 5832-11 testifying the capability of the SLM products for hip joint prosthesis.

The in vivo comparative studies of Zhao et al. (B. Zhao et al., 2017) found that products from SLM are more resistant to corrosion than EBM-fabricated parts. This is ascribed to the SLM products' microstructure consisting of much finer α' martensite hardened by uniformly distributed dispersive β - phase (β -AlNbTi₂). This microstructure results in lower proportion of phase interfaces compared to the lamellar $\alpha + \beta$ microstructure of the EBM-fabricated components resulting in the reduction of microgalvanic cells hence improved corrosion resistance of the SLM products (Chlebus et al., 2011; Z. Yu et al., 2022). This can play a critical role in mitigating the corrosion failure mechanism of hip implants.

Wang et al. (C. Wang et al., 2022) demonstrated the possibility of producing acetabular cup prosthesis mimicking the natural bone, satisfying the hip joint's mechanical strength requirements during normal walking while simultaneously minimizing the “stress shielding” effect (see **Figure 2. 31**).

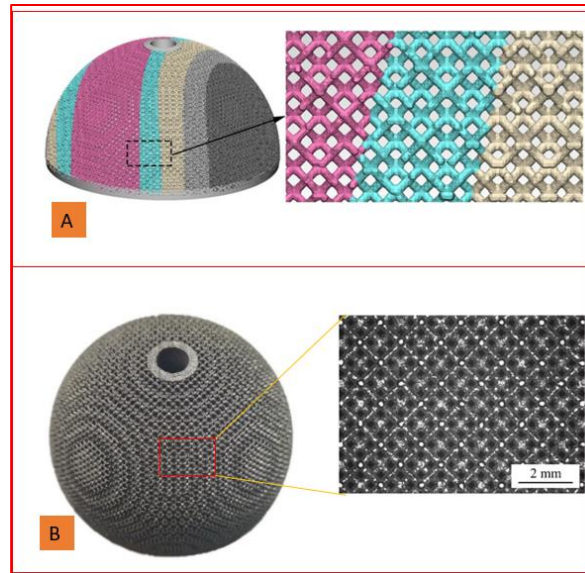


Figure 2. 31. (A) The designed acetabular cup model of rhombic dodecahedron porous structure with 1.5mm unit cell size. (B) The SLM-fabricated acetabular cup from the designed model (C. Wang et al., 2022).

As shown in **Figure 2. 31**, the printed parts had good surface morphology and replicated the designed model with high precision. The specimen's elastic modulus as well as compressive strength matched those of the human cortical bone suggesting reduced stress shielding and improved performance of the hip implant.

LPBF has proven to be a transformative technology in the fabrication of hip implants. Contrary to conventional manufacturing methods, SLM's ability to fabricate interconnected porous structures provide good conditions for cell adhesion, diffusion, differentiation, and the flow of oxygen and nutrients thus accelerating vascularization and implant osseointegration (Nune et al., 2015). The advantages of SLM including fabrication of intricate structures with very high precision, high material efficiency, fast production speed, and improved mechanical strength of the printed parts can play an important role in mitigating hip implant failures by optimizing material properties, fit, and biomechanical compatibility (Ge et al., 2020; J. Liu et al., 2019).

2.3 Research Gaps.

Significant research efforts have been dedicated to enhancing the survival rate of femoral components in hip implants while the acetabular components currently having the lowest survival rate of 68-84% at 10 years' service life have been neglected. The growing demand for high-performance hip prostheses with extended survival rates has become a global research focus. Significant research efforts are required to enhance both the materials and manufacturing techniques to improve the longevity of acetabular implants within the body.

Based on this literature review, the widely utilized Ti-6Al-4V alloy requires replacement due to the adverse effects of its alloying elements, particularly the cytotoxic vanadium component. While commercially pure titanium exhibits excellent biocompatibility and favourable tissue adsorption properties (Shah et al., 2016), its inferior mechanical performance restricts its viability as a replacement for Ti-6Al-4V in acetabular implants (Y. Li et al., 2014). Additionally, the relatively low wear resistance, limited corrosion resistance and biocompatibility contribute to bone demineralization around the Ti-6Al-4V acetabular implant, prompting further exploration of alternative titanium alloys. In this regard, the more biocompatible Ti-6Al-7Nb alloy has emerged as a promising candidate (L.-Y. Chen et al., 2020).

Traditional manufacturing methods have successfully fabricated hip implants, however, the manufacturing defects, inhomogeneous microstructure, and their inherent limitations to

produce intricate near-net shapes mimicking the natural bone have greatly affected the performance of conventionally fabricated hip implants. Ti-6Al-7Nb hip implants with complex shapes can be manufactured using metal additive manufacturing techniques like electron beam melting technology, however, geometrical accuracy is compromised. LPBF technology has demonstrated the capabilities to fabricate intricate near-net shaped hip implants mimicking the natural bone with high geometrical precision.

Therefore, extensive investigation in the utilization of laser powder bed fusion process for the more biocompatible Ti-6Al-7Nb alloy in acetabular cup implants is essential to improve the service life of prostheses. In-depth analysis of the compressive properties, tribological performance, and biocompatibility study in simulated body fluids is required to facilitate the application of this alloy coupled with additive manufacturing to mitigate hip implants failure in the body system.

Chapter 3: Materials and Methodology.

3.1.Chapter Summary.

This chapter outlines the experimental approach, materials, and techniques utilized to fulfill the research objectives presented in chapter 1, section 1.4 of this thesis. The methodology, schematically summarized in **Figure 3. 1** was designed to address the underscored acetabular implants failure mechanisms of corrosion and wear highlighted in chapter two of this thesis. It provides a detailed description of materials (Ti-6Al-7Nb powder as well as the Ti-6Al-4V build plate), equipment, and processes, including the laser powder bed fusion of Ti-6Al-7Nb powder and annealing treatment process utilized in the experiments. The microstructural characterization using advanced equipment; like scanning electron microscope, X-ray diffractometer, tribology tests, compression tests, and biocompatibility through immersion corrosion tests in simulated body fluids are clearly explained.

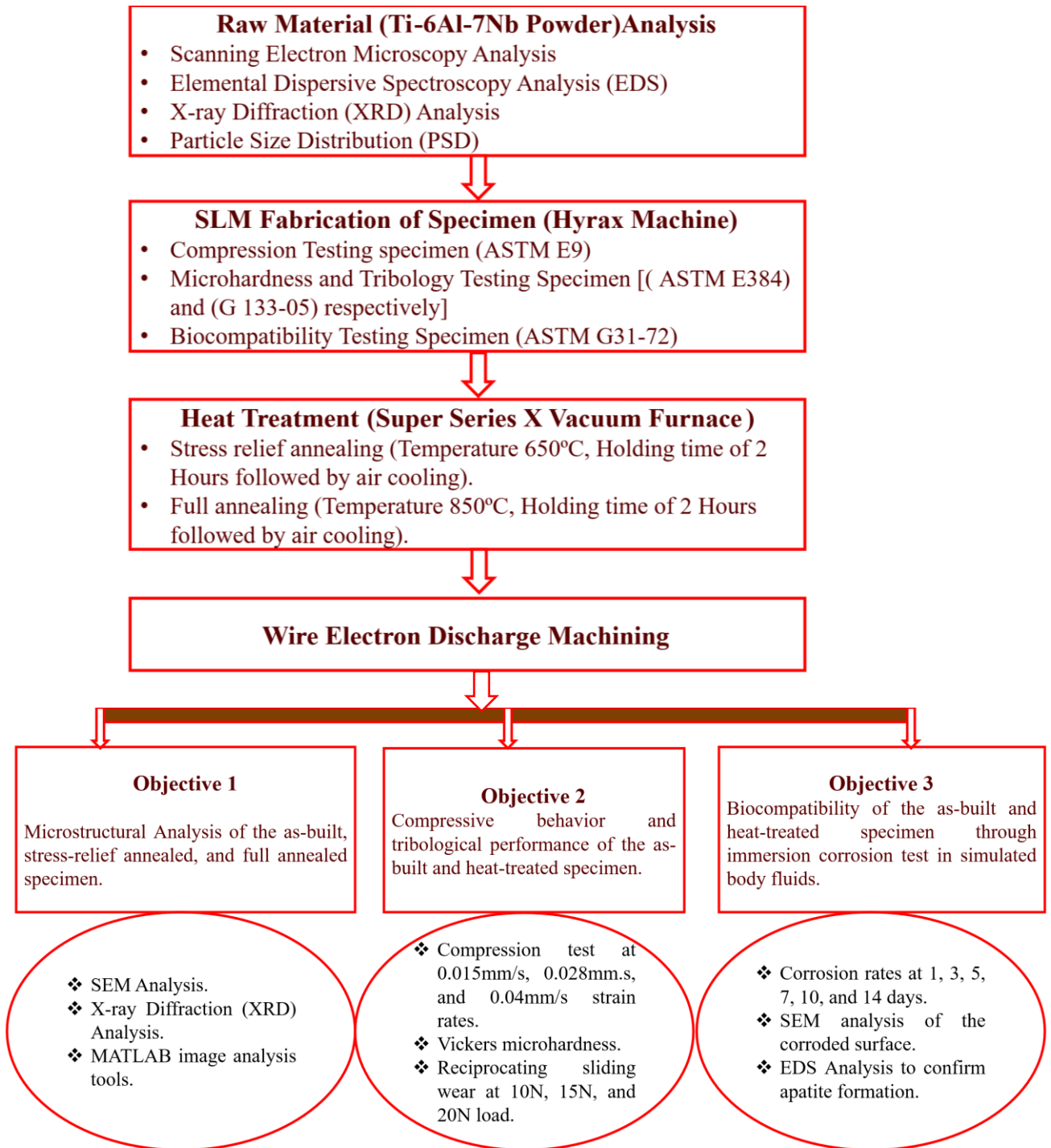


Figure 3. 1: A summary of the research methodology employed in this study.

3.2.Materials.

The experimental work concentrated on using LPBF to build Ti-6Al-7Nb specimens on a wrought Ti-6Al-4V base plate. This was a material choice for the build plate, to provide a strong and stable base for the new biocompatible Ti-6Al-7Nb alloy, since it has well-known mechanical properties and its broad application in biomedical field.

3.2.1. Build plate Material (Ti-6Al-4V).

Ti-6Al-4V was chosen as the build plate material to leverage its proven properties, such as better biocompatibility and good corrosion resistance. The build plate had a square geometry with a side length of 49.95mm as depicted in **Figure 3. 2**.

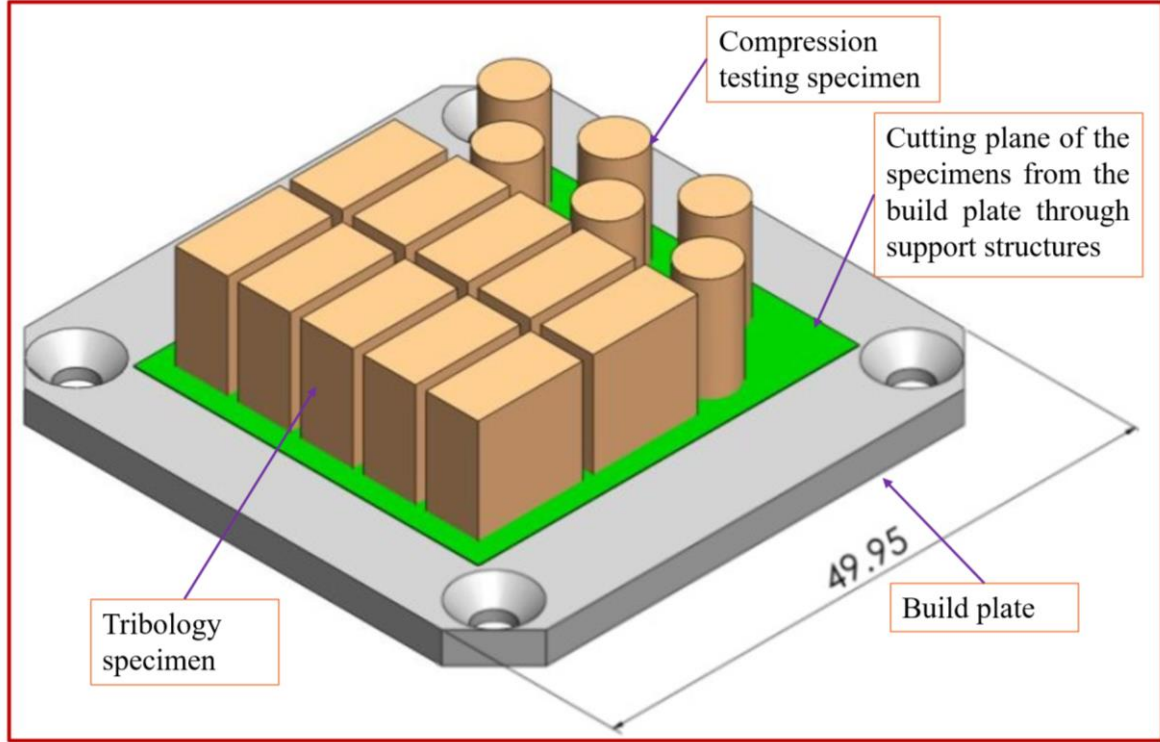


Figure 3. 2: The dimensions of a Ti-6Al-4V build plate having a Ti-6Al-7Nb specimen printed on it.

Lattice support structures, 2 mm thick, were printed on the build plate for each specimen to function as anchors, enhance heat dissipation, and minimize thermal deformation of the components. In orthopaedic applications, Ti-6Al-4V is widely used in the fabrication of acetabular prostheses for its well-documented performance and reliability. However, Ti-6Al-4V alloy has relatively poor wear and corrosion resistance, along with limited biocompatibility leading to the formation of degradation byproducts, which contribute to aseptic loosening and eventual prosthesis failure ([Ganesh et al., 2012](#)). To address these challenges, Ti-6Al-7Nb alloy is being investigated for potential application in acetabular implants.

3.2.2. Ti-6Al-7Nb Alloy Powder (Feedstock).

Gas atomized Ti-6Al-7Nb alloy powders (20-45 μm) sourced from Suzhou Joroyce Products Co. Ltd; China was utilized in this work. Pre-alloyed powders were preferred to

avoid issues of inhomogeneous melt distribution and powder particle segregation during deposition of the powders in the build chamber, which are encountered in coated and mixed powders, respectively. The beta transus temperature of this alloy, marking the complete transition of alpha phase to the beta phase, is 1015°C ("TIMET TIMETAL® 367 (Ti-6Al-7Nb) Titanium Alloy datasheet,").

3.2.2.1. Morphological and elemental characterization of Ti-6Al-7Nb powder.

The morphological features of Ti-6Al-7Nb powders were analyzed using a JEOL JSM-6610 scanning electron microscope. The elemental composition of Ti-6Al-7Nb powders was characterized through Energy Dispersive Spectroscopy (EDS) integrated into the JEOL JSM-6610 scanning electron microscope, employing point scanning for precise elemental analysis. The main objective of point scanning is to simultaneously scan the selected area and perform quantitative analysis. In this study, five points were scanned.

3.2.2.2. X-ray diffraction (XRD) analysis.

X-ray diffraction (XRD) characterization was conducted using a Bruker D8 Advance powder diffractometer to analyse the phase composition of raw powder, as-built, and heat-treated LPBF Ti-6Al-7Nb specimens. This diffractometer consists of a LynxEye XE energy-dispersive strip detector and a Cu tube X-ray source. The analysis employed Cu-K α radiation at a wavelength of $\lambda=1.5418 \text{ \AA}$, utilizing a working current of 40 mA and a voltage of 40 kV. The scan was conducted at a rate of 0.01°s^{-1} within a 2θ range of 30° to 70° , with a 0.01° step size. The XRD results were used to study the phase and crystal structure changes in response to different annealing treatment processes. The longitudinal section oriented along the build direction (BD) was examined for both as-built and heat-treated LPBF Ti-6Al-7Nb specimens.

3.2.2.3. Particle size distribution (PSD) analysis.

The particle size distribution (PSD) of Ti-6Al-7Nb powders was measured using the Mastersizer 3000E (Malvern Panalytical, UK), see **Figure 3. 3**, utilizing laser diffraction to analyze particle size and distribution. The laser diffraction technique, popular for its reproducibility and outstanding broad measuring range, utilizes a laser beam to penetrate a particulate sample, measuring the angular dispersion in scattered light intensity for precise particle characterization (Keck & Müller, 2008; Malvern, 2025; "Malvern Mastersizer 3000,"). Fine particles exhibit wide-angle light scattering, whereas larger particles scatter light at narrower angles relative to the incident laser beam (refer **Figure 3. 3A**). The

Mastersizer-v3.88 software then analyzes the scattering intensity data to determine the particle size producing the observed scattering pattern, employing Mie scattering theory (Mie, 1908). This size is presented as a volume-equivalent sphere diameter.

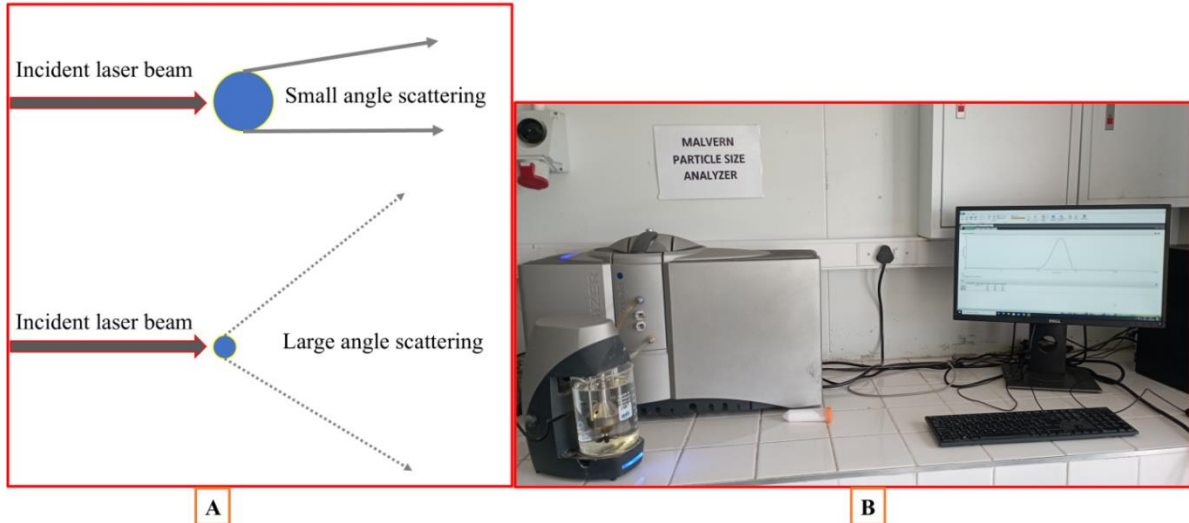


Figure 3. 3: (A) The laser diffraction particle size measurement principle. (B) The Mastersizer 3000E Malvern Particle Size Analyzer used in this study at BIUST.

This study utilized the Hydro LV wet sample dispersion with 600mL dispersant volume. Sodium hexametaphosphate (SHMP) was used as a dispersing agent, ensuring that individual Ti-6Al-7Nb powder particles are evenly suspended in the solution. The Ti-6Al-7Nb powder sample was added drop wise until the obscuration range of 10-15% was reached to observe a stable scattering pattern.

3.2.2.4. Powder Storage and Use.

A total powder quantity of 10kgs were supplied by the supplier using self-scaling containers of 2kgs capacity each. These very same containers were used to store the powders. After specimen fabrication, recycled powders were sieved using a 39 μm sieve to remove metal debris and stored in separate containers.

3.2.2.5. Powder Handling.

To ensure powder quality, prevent contamination, and maintain result reproducibility, the powders were carefully handled under controlled conditions. To minimize powder aeration, the self-scaling powder container lids were only being taken off when the powder is ready for use, and the height at which the powder was poured from the container to the feeder hoppers was maintained to a minimum.

3.3. Laser Powder bed Fusion (LPBF) Process Set Up.

Ti-6Al-7Nb specimens were fabricated using the HYRAX, a laser-based powder bed fusion system developed by Aditiv Solutions, a Pretoria-based technology company (See **Figure 3. 4**). This machine is under the Centre for Rapid Prototyping and Manufacturing (CRPM) department at Central University of Technology (CUT), South Africa. The HYRAX system employs LPBF technology to process various materials including titanium, steel, aluminum, and Inconel parts from powder feedstock. This Hyrax SLM machine is equipped with a single Ytterbium (Yb) fiber laser (single mode) having a maximum power of 400W, beam spot size range of 90-500 μm , and a Gaussian intensity profile. The outstanding features of this powder bed fusion system are presented in **Figure 3. 5**.

Prior to the SLM process, the build plate plates were cleaned with acetone to eliminate contaminants (impurities) and improve their laser absorptivity. Ti-6Al-7Nb powders were supplied to the build plate from multi-hoppers using a gravity-fed powder handling system. The Hyrax system has an optimized configuration through bi-directional powder scraping which helps to achieve a uniform layer of powder across the build area ensuring consistent melting and solidification during the SLM process. Additionally, no fastening holes are present on the build plate allowing full utilization of the build plate. To provide a localized inert atmosphere with oxygen levels below 0.08%, argon was employed as the shielding gas.



Figure 3. 4: The HYRAX SLM machine at CUT, Bloemfontein, South Africa.

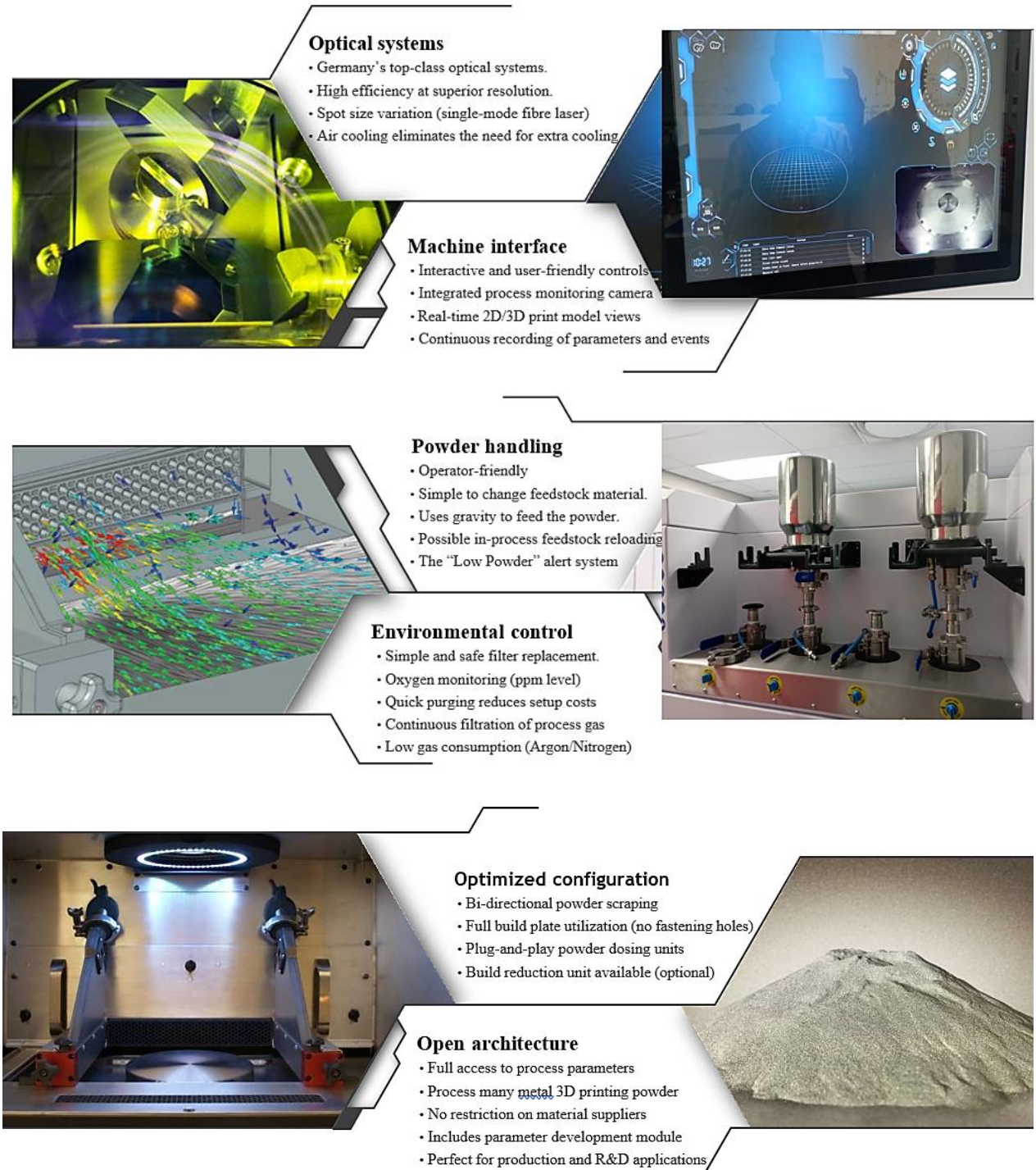


Figure 3. 5: Characteristic features of the HYRAX SLM powder-bed fusion system.

The optimized LPBF process parameters established by Sello et al. (Sello et al., 2025), were utilized in this study for sample fabrication. A summary of these parameters is presented in **Table 3. 1**. A continuous laser mode was used to scan the layers in a zigzag pattern, rotating each layer by 90° as schematically detailed in **Figure 3. 6**. This scanning strategy

was employed to achieve thermal uniformity, ensuring a more even heat distribution throughout the process, reducing thermal gradients as well as residual stresses and improving layer bonding to attain a maximized relative density ($> 99.3\%$) (Monu et al., 2023).. An inert protective atmosphere was established using argon gas in the LPBF process, reducing the oxygen level to 0.08%.

Table 3. 1: LPBF process parameters for specimen builds.

Process Parameters	
Laser Power (W)	350
Laser spot size (μm)	158
Scanning Speed (mm/sec)	1900
Layer Thickness (μm)	40
Hatch Spacing (μm)	72

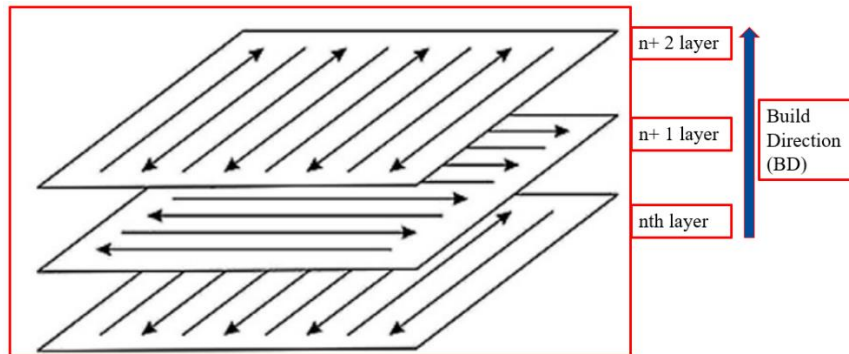


Figure 3. 6: 90° rotation scanning strategy showing laser scanning paths on layer n and subsequent layers $(n + 1)$, $(n + 2) \dots (n + j)$.

3.4.Design and Fabrication of the Compression, Corrosion (Compatibility), and Wear Specimen.

This section outlines the design considerations and fabrication steps followed for producing the specimens used in compression, corrosion (biocompatibility), and wear testing. The specimen geometries were selected in accordance with relevant testing standards to ensure consistency and reliability of results. All specimens were fabricated using the LPBF process under the parameters detailed in Table 3.1.

3.4.1. Laser Powder Bed Fusion of Corrosion Testing Samples.

The dimensions and geometry of immersion corrosion specimens vary based on the test objective, material type, and the experimental apparatus used in the corrosion assessment. The fabrication and experimental set-up of immersion corrosion test carried out in this study adhered to the ASTM G31-72 standard (G-1, 2022). It is desirable that the specimens should have a high surface-to-mass ratio and a minimal edge-to-total area ratio. This minimizes the influence of localized effects and edge areas, which can lead to crevice corrosion or other localized forms of corrosion. Essentially, it helps in achieving a more representative assessment of the material's corrosion behaviour under test conditions. These requirements can be accomplished by using rectangular, square, or circular specimen with minimum thickness (G-1, 2022). In this study, square specimen of side length 10mm were selectively laser melted as shown in **Figure 3. 7**. This were then halved into two to have rectangular specimen of dimensions 10mm × 5mm through wire electric discharge machining.

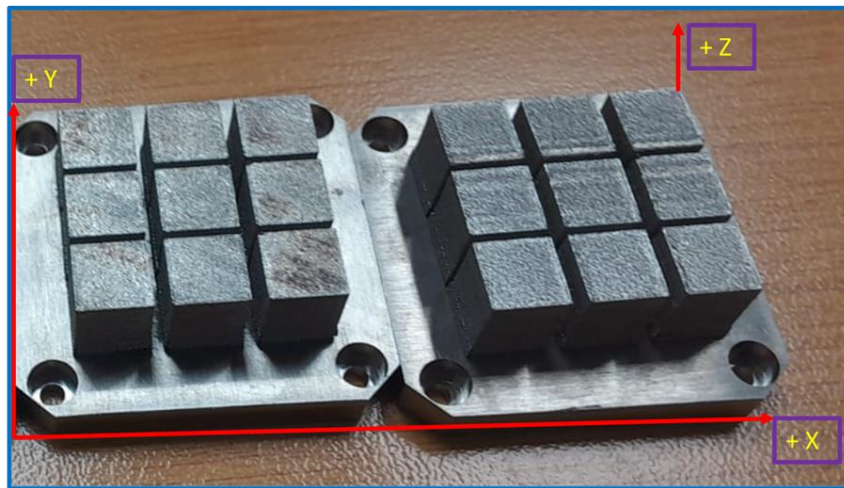


Figure 3. 7: SLM 10mm × 10mm Ti-6Al-7Nb specimens on a Ti-6Al-4V build plate.

3.4.2. Laser Powder Bed Fusion of Compression Samples.

The specimens' dimensions, and axial-load compression testing followed the ASTM E9-09 standard (E. Committee). This standard recommends solid circular cylindrical geometry specimen for compression test. There are three different types of compression test specimens for metallic materials: short, medium-length, and long. This classification depends on the length-to-diameter ratios. Short specimens have approximate length-to-diameter ratios of 0.8 and 2.0. Medium-length specimens have a length-to-diameter ratio of 3.0 while long compression specimens have length-to-diameter ratios of 8.0 and 10.0.

Compression testing of materials like bearing metals, utilized as thin plates to support perpendicular loads, generally employs short specimens for precise mechanical evaluation. Medium-length specimens are primarily utilized to ascertain the general compressive strength properties of metallic materials while long specimens are used when the metallic material's elastic modulus is the main focus of the compression test. More importantly, ASTM E9-09 standard (E. Committee) emphasizes that specimens with a length-to-diameter ratio of 1.5 or 2.0 most suitable for evaluating the compressive strength of high-strength materials like titanium, steel, tungsten, and Inconel. Therefore, in this study the compression specimen had dimensions of length = 9mm and diameter = 6mm (See **Figure 3. 8B**) giving a length-to-diameter ratio of 1.5 to comply with the ASTM E9-09 standard.

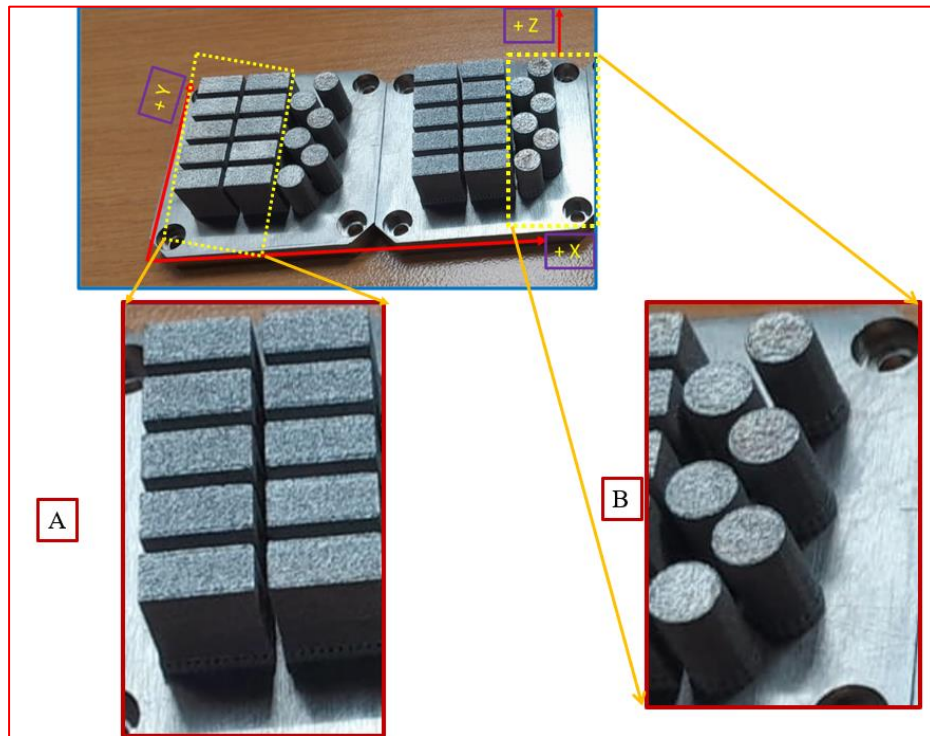


Figure 3. 8: SLM Ti-6Al-7Nb specimen on a Ti-6Al-4V build plate. (A) Ti-6Al-7Nb tribology testing specimen. (B) Ti-6Al-7Nb Compression testing specimen. NB: Tiny hollow support structures can be seen closer to the build plate.

3.4.3. Laser Powder Bed Fusion of Tribology (Wear) Samples.

Common shapes for tribological specimen are pins, disks, or blocks. Considering the apparatus to be used for tribology test in our study, rectangular (block) specimens were recommended. As shown in **Figure 3. 8A**, these specimens had a rectangular geometry of dimension 10mm × 5mm × 5mm which were later machined through EDM to square geometry of 5mm × 5mm × 5mm.

3.5.Heat treatment.

Selectively laser melted titanium components often exhibit residual stresses, metastable phases, and intrinsic defects. These issues lead to poor mechanical, tribological, and biological properties of selectively laser melted parts ([Karimi et al., 2022](#)). These issues are addressed by tailoring the microstructure through post-process treatments like hot isostatic pressing (HIP), stress-relief annealing, full annealing, and solution treatments.

To investigate how annealing heat treatment affects the microstructure, compressive behavior, wear performance, biocompatibility, and corrosion resistance of LPB-fabricated Ti-6Al-7Nb parts, the specimens were grouped into three batches. The first batch was left as-built, the second batch was stress-relief annealed, and the third batch was full-annealed. The heat treatment parameters in **Table 3. 2** which are schematically presented **Figure 3. 9**, (the annealing heat treatment cycles) were used. These heat treatment procedures were done at Central University of Technology (CUT), Bloemfontein, Free State, South Africa, using Super Series X Vacuum Furnace (T-M Vacuum Products Inc., USA) maintaining a high vacuum level of approximately 10^{-5} Torr to ensure a contaminant-free environment (see **Figure 3. 10**).

Table 3. 2: Heat treatment parameters for LPBF-fabricated Ti-6Al-7Nb alloy specimens in this research.

Parameter	Stress-relief annealing (HT650)	Full annealing (HT850)
Heating temperature	650°C (923.15 K)	850°C (1123.15 K)
Heating rate (°C/min)	10	10
Dwell time (hours)	2	2
Cooling rate	Air cooling	Air cooling

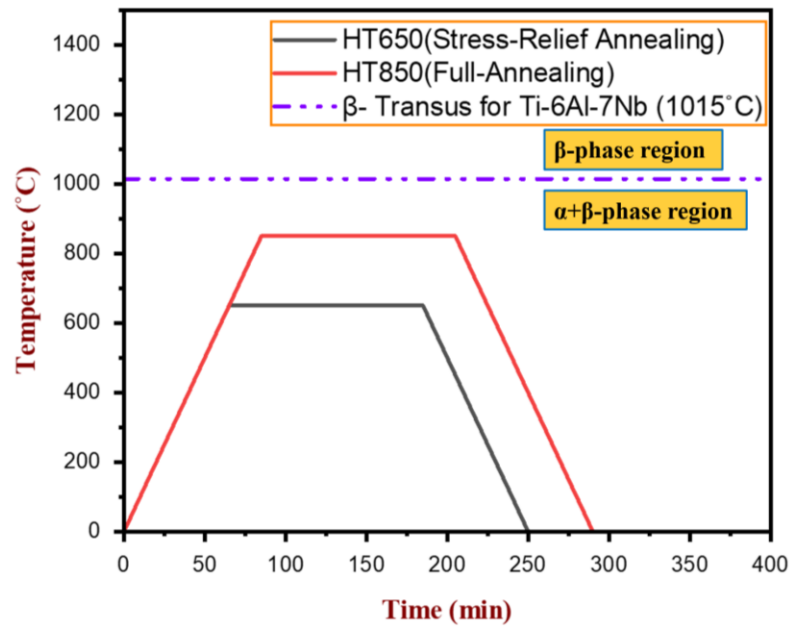


Figure 3. 9: Annealing heat treatment parameters of laser of LPBF Ti-6Al-7Nb samples applied in the experiments.



Figure 3. 10: Super Series X Vacuum Furnace used in this study at CUT, Bloemfontein, South Africa.

3.6. Wire Electric Discharge Machining (WEDM).

After the LPBF process, a numerical control wire electrical discharge cutter; DK7732 (Kokoele Trading, 35 Nicol Road, Bedfordview, Johannesburg, South Africa.) was utilized to extract the specimens from the build plate and prepare the specimens' final geometries.

WEDM process is the useful technique for cutting selectively laser melted samples due to its ability to make precise cuts with minimal mechanical stress as well as a very high-quality surface finish. The samples were cut through the 2mm support structures that were built from the base of the build plate clearly exhibited in **Figure 3. 11**.

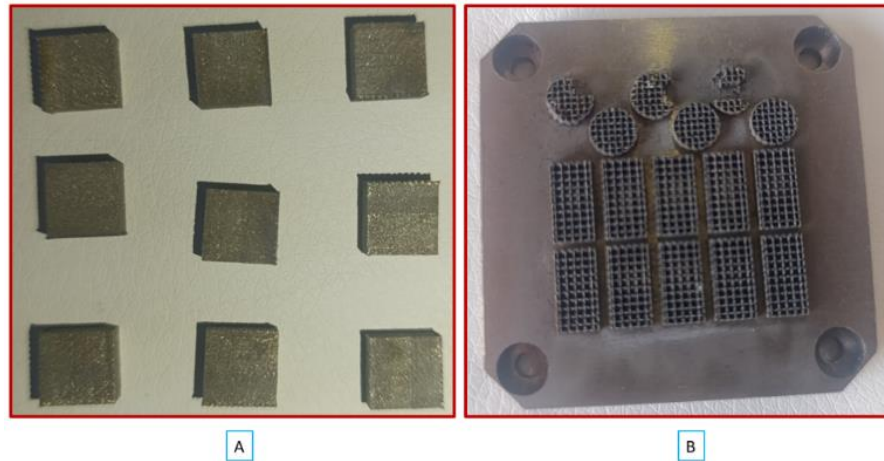


Figure 3. 11: (A) WEDM specimens ready for mounting. (B) The remaining build plate with support structures after WEDM process.

3.7.Metallographic LPBF Ti-6AL-7Nb Sample Preparation.

Metallographic sample preparation procedure was conducted prior to the microstructural analysis. The steps followed in the metallographic sample preparation procedure include; hot mounting, grinding, polishing, and etching before examining the samples under a scanning electron microscope.

3.7.1. Specimens Mounting.

This is the first step after removing the samples from the baseplate and sectioning the samples to the required dimensions using WEDM. The specimens were hot mounted using a hot mounting press Opal 410 machine for a total processing time of 15 minutes with approximately 25g conductive black bakelite resin to create conductive mounts suitable for SEM analysis. (See **Figure 3. 12**). Conductive black phenolic resin is the perfect mounting material for SEM analysis due to its low shrinkage and high conductivity provided by the carbon filler ensuring high-quality, stable mounts with excellent edge retention and conductivity giving a precise high-resolution SEM image (["Metallographic products, knowledge and service | Struers.com," 2024](https://www.struers.com/en-us/knowledge-and-service/metallographic-products)). In this process, a clean and dry specimen facing downwards is placed in a mounting cylinder, and then approximately 25g PolyFast

resin is added. Machine parameters summarized in **Table 3. 3** were used and water cooling was utilized to achieve the shortest possible mounting time.

Table 3. 3: Machine parameter settings for the hot mounting process.

Hot Mounting Parameters		
Pressure (Bars)	400	
Temperature (°C)	180	
Total Processing Time (Min)	15 minutes	
	Heating Time	Cooling Time
	10 minutes	5 minutes



Figure 3. 12: (A) PolyFast resin, a conductive resin used for hot mounting of Ti-6Al-7Nb specimen. (B) An Opal 410 hot mounting press at BIUST’s Materials Laboratory used in this study.

The mounted samples were carefully labelled using a Dremel electric engraver for identification and traceability as shown in **Figure 3. 13**.

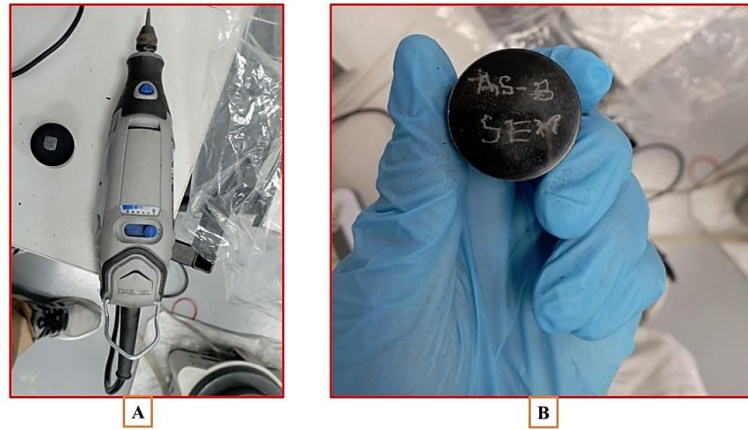


Figure 3. 13: (A) Dremel Electric Engraver used for sample labelling. (B) A properly labelled as-built (AS-B) sample ready for grinding and polishing.

Proper labelling ensures that each sample can be accurately identified throughout the testing process. This is essential for maintaining research integrity and preventing potential errors or misidentifications.

3.7.2. Specimens Grinding and Polishing.

Grinding and polishing were performed with a Qualitest Qualipol VTD SA machine. Silicon carbide abrasive papers (P120, P220, P320, P600, P1000, and P1200) were used following standard procedures to achieve a scratch free surface (refer to **Figure 3. 14**).



Figure 3. 14: (A) Qualitest Qualipol VTD SA grinding and polishing machine at the BIUST Materials Testing Laboratory. (B) Ground Ti-6Al-7Nb specimens.

To avoid overheating and damaging the metallographic structure, this machine has a water-cooling system that cools the specimen as it is being ground. After grinding, the specimens

underwent polishing to achieve a smooth, mirror-like surface free of deformations and scratches. Coarse polishing was initially conducted by rotating the specimens on a polishing machine, utilizing a 6 μ m diamond suspension applied on an Aka-Moran polishing cloth. Additional polishing was conducted using 3 μ m and 1 μ m diamond suspension using the magnetic Mag-Plural polishing cloth. Specifications for the grinding and polishing stage have been summarized in **Table 3. 4**.

Table 3. 4: Grinding and Polishing Procedure for the as-built and heat-treated LPBF Ti-6Al-7Nb Samples.

Parameters	Grinding Steps						Polishing Steps		
	1	2	3	4	5	6	1	2	3
Abrasive discs. (SiC P grade). Polishing Cloth	P120	P220	P320	P600	P1000	P1200	Aka-Moran	Mag-Plural	Mag-Plural
Lubricant	Water	Water	Water	Water	Water	Water	6 μ m diamond suspension	3 μ m diamond suspension	1 μ m diamond suspension
Platen Speed	300 rpm	300 rpm	300 rpm	150 rpm	150 rpm	150 rpm	150 rpm	150 rpm	150 rpm

3.7.3. Ultrasonic Cleaning.

Ultrasonic cleaning is a highly effective method for preparing samples for scanning electron microscopy (SEM). It employs high-frequency sound waves to generate cavitation bubbles in a liquid medium, which implode with great force, effectively removing dirt and grime from surfaces thereby providing a powerful cleaning action ([Awad & Nagarajan, 2010](#)). This process removes contaminants and debris from samples, ensuring they are clean and ready for SEM analysis ([Regi, 2007](#)).

In this study, the mounted and polished specimens were sonicated using acetone, ethanol, and deionized water, sequentially, for 15 minutes per solvent, using a Lab Companion UC-02 ultrasonic cleaner to ensure effective removal of surface contaminants (**Figure 3. 15**).

After each stage of cleaning, the samples were dried using a blower ready for SEM analysis. During this process and afterwards, the samples were handled very carefully using clean gloves.

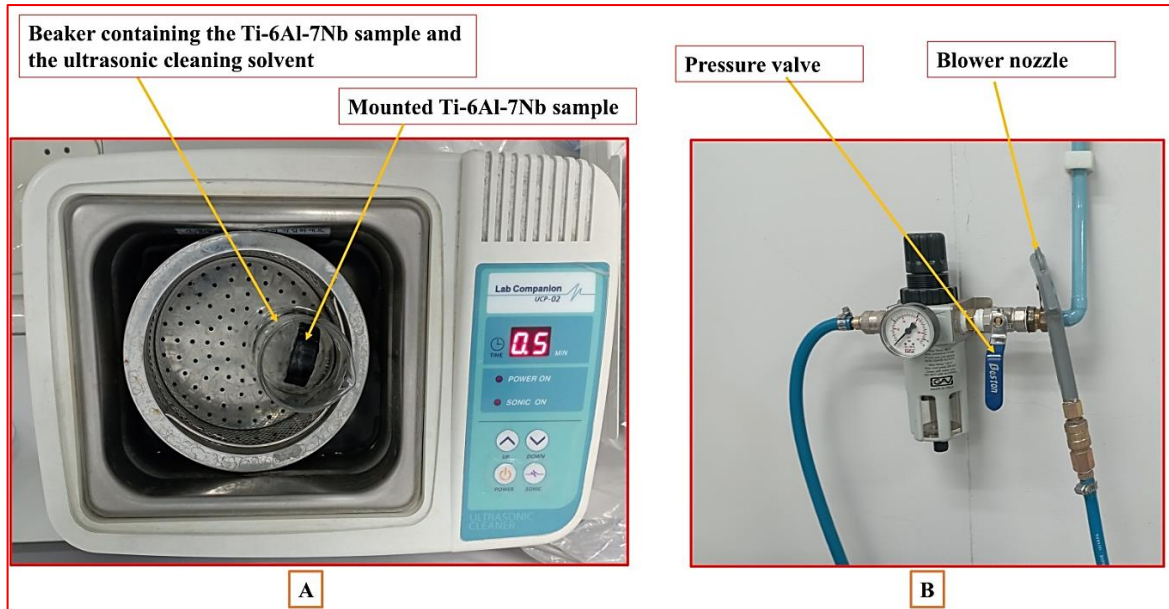


Figure 3. 15: Lab Companion UC-02 Ultrasonic Cleaner. (B) A blower used for drying the samples after ultrasonic cleaning in this study at CUT, Bloemfontein, South Africa.

3.7.4. Etching.

A polished metal specimen typically does not reveal its microstructure, as light is directly reflected off the surface, making subtle variations in reflection imperceptible to the naked eye. To reveal the metal's microstructure and create optical contrast between the constituents of the material, the metal surface is metallographically etched. Etchants are made up of organic or inorganic acids, or alkali in a solvent like distilled water, glycerin, and alcohol. Grain boundaries exhibit enhanced chemical reactivity compared to interior grains, making them more prone to corrosion when exposed to the acidic constituents of etchants (Hammond & Evennett, 2005). This differential etching response between interior grains and grain boundaries induces contrast, effectively delineating grain boundaries, phases, and inclusions that remain undetectable on a polished surface.

The metallographic etchant used in our study is a Kroll's reagent (refer to **Table 3. 5**) as per ASTM E407-187,192 Krolls Etchant(s) standard ("[SMS ASTM 187 192 Krolls | PDF | Toxicity | Dangerous Goods,](#)" 2015). To observe the microstructure of as-built and heat-treated LPBF Ti-6Al-7Nb specimens, the samples were etched by gently swabbing with

Kroll's reagent for about 2–3 seconds. Upon successful etching, the samples were cleaned with ethanol to stop the reaction. The etching process was conducted with extreme care since Kroll's reagent contains the hazardous hydrofluoric acid, HF. Gloves, and protective lab coats were used to prevent direct contact with the acid. The procedure was conducted under a certified fume hood to contain and exhaust the released toxic fumes, reducing the risk of inhalation and exposure (Figure 3. 16).

Table 3. 5: Chemical composition of the Kroll's reagent used in this study.

Ingredient	Volume (ml)
Nitric Acid (HNO ₃)	5 ml (Concentrated Acid)
Hydrofluoric Acid (HF)	10 ml (48% concentration)
Water (H ₂ O)	85 ml

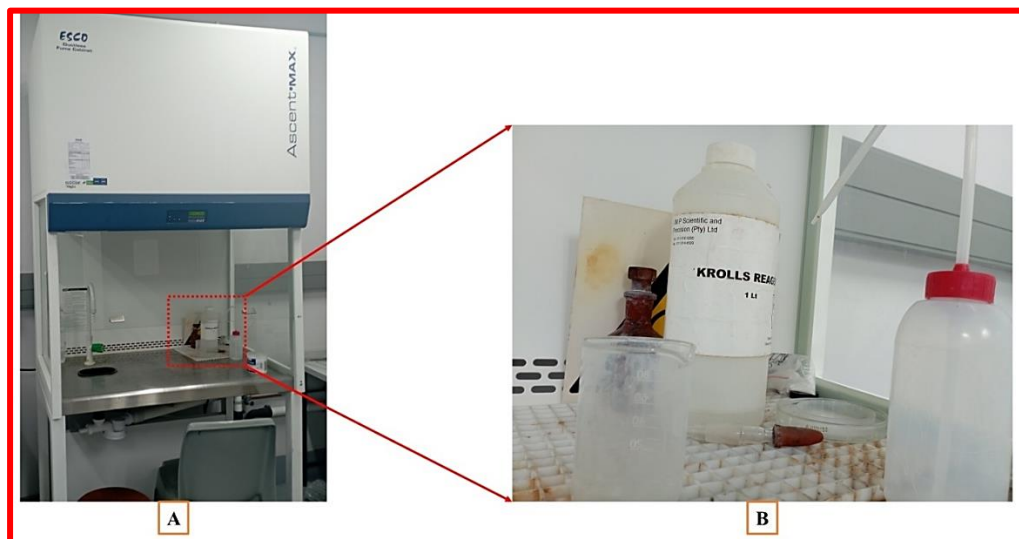


Figure 3. 16: (A) The ESCO Ductless Fume Hood. (B) Kroll's reagent for etching Ti-6Al-7Nb samples in the Metallography laboratory at CUT, Bloemfontein, South Africa.

3.8. Objective 1: Microstructural examination of the as-built and heat-treated LPBF Ti-6Al-7Nb specimens.

The LPBF fabricated Ti-6Al-7Nb specimens were characterized in three distinct states: as-built, stress-relief annealed, and fully annealed. Comprehensive microstructural and phase composition examinations were conducted on both as-built and heat-treated LPBF Ti-6Al-7Nb specimens through SEM and XRD techniques.

3.8.1. Scanning Electron Microscopy (SEM).

A high-performance JEOL JSM – 6610 SEM (**Figure 3. 17**), which comprises of a tungsten electron column and an Oxford Instruments Inca X-Act EDS was used in this study. In addition to its high-resolution imaging, typically several hundred times greater than that of an optical microscope, it also offers compositional or elemental analysis through its EDS component. In SEM, when electrons from an electron gun are focussed into a beam and rastered across the specimen's surface, x-ray signals, secondary electrons (SE), and backscattered electrons (BSE) are generated. Originating deep within the specimen, backscattered electrons (BSE) have a strong interaction with the material and provide compositional information (phases) of the material. Conversely, secondary electrons (SE) originate from the atoms of the specimen and are due to inelastic interactions of the sample and the electron beam. SE are very sensitive to surface structure and provide topographical information thereby identifying the morphology of the specimen (Farayibi, 2014; Eyitayo Olatunde Olakanmi, 2008). Additionally, the generated x-ray signals are thought of as fingerprint of the sample material and elemental composition of this material is determined by measuring the energy of the x-rays emanating from the specimen. SEM was utilized in this study to characterize the microstructure of the as-built and heat-treated LPBF Ti-6Al-7Nb specimens.



Figure 3. 17: JEOL JSM – 6610 Scanning Electron Microscope used in this study.

3.8.2. X-ray Diffraction Characterisation.

For the analysis of the as-built, and heat-treated LPBF Ti-6Al-7Nb samples in this study, a Bruker D8 Advance X-ray Diffractometer with monochromatic Cu K α radiation and higher

performance compound silicon energy-dispersive strip detector, LYNXEYE XE, was used (see **Figure 3. 18**) ("[Empyrean | XRD Instrument | Malvern Panalytical](#)," 2024). An incident X-ray beam was focused onto the mounted specimen on the XRD machine, which was subsequently rotated to obtain the diffraction angles as defined by the Bragg's law, **Equation 3. 1**.

$$n\lambda = 2d_{hkl}\sin\theta \quad \text{Equation 3. 1}$$

In this expression, n represents an integer denoting the reflection order, λ corresponds to the wavelength of the incident X-ray, d signifies the interplanar spacing within the crystal structure, and θ denotes the angle of incidence at which the X-ray beam interacts with the material.



Figure 3. 18: X-ray Diffractometer Bruker D8 Advance utilised in this research at BIUST, Palapye, Botswana.

In this study, the longitudinal section oriented along the build direction (BD) was examined for both as-built and heat-treated LPBF Ti-6Al-7Nb specimens. This analysis was executed using Cu-K α with a wavelength of $\lambda=1.5418 \text{ \AA}$, operating at a working current of 40 mA and a voltage of 40 kV. The scan was conducted at a rate of 0.01°s^{-1} within a 2θ range of 30° to 70° , with a 0.01° step size. There is no joint committee on powder diffraction standard (JCPDS) card for Ti-6Al-7Nb, hence the Ti (00-044-1294) and $\text{Ti}_{0.70}\text{Al}_{0.30}\text{Nb}_{0.07}$ (00-053-0484) cards were used to identify the XRD peaks.

3.9.Objective 2: Compressive behaviour and Tribological performance Analysis.

3.9.1. Investigation of compressive behaviour.

The primary goal of the compression experiments was to examine how LPBF process and annealing treatment impacted the mechanical properties of Ti-6Al-7Nb specimens. These tests help in evaluating the performance of the as-built and heat-treated LPBF Ti-6Al-7Nb samples to be used in acetabular implants, ensuring they can handle the stresses and strains of daily activities. The Schenck POZ 160 Universal Testing Machine with displacement control (**Figure 3. 19**) at the Centre for Scientific and Industrial Research (CSIR) in South Africa, was utilized to perform compressive mechanical testing on samples as per ASTM E9 (E. Committee) standard at different strain rates of 0.015 mm/sec, 0.028 mm/sec, and 0.04 mm/sec. The load was recorded using a load cell, while the displacement data was obtained from the crosshead movement of the testing machine. Results of these tests determined by the built-in load-cell include only the load-displacement data.

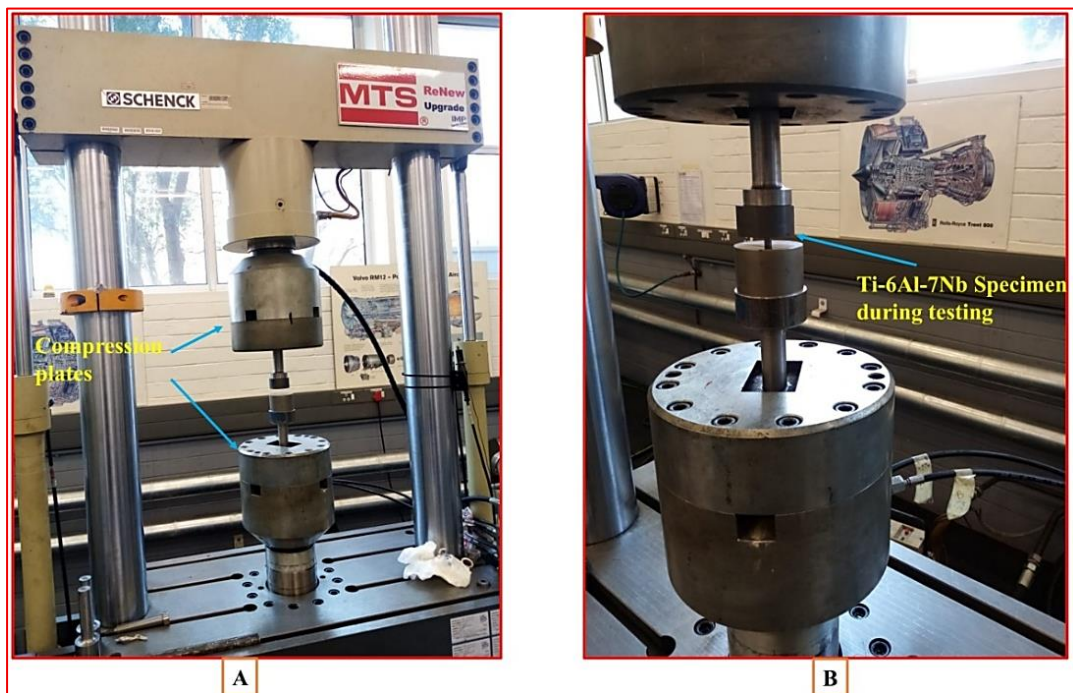


Figure 3. 19: (A) The Schenck POZ 160 hydraulic compressive testing machine. (B) The Ti-6Al-7Nb specimen during compression test.

The specimen was unrestrained and set upright on the lower plate. The bottom compression plate was then driven upward until the top compression plate nearly contacted the specimen's surface. Thereafter, the bottom compression plate moved upward to compress the specimen at a velocity determined by the specified strain rate. The specimen underwent

uniaxial compression until such time that the test was stopped automatically after reaching the ultimate stress point without complete failure of the specimen.

These strain rates were used to replicate the realistic physiological strain rates which varies in the range of $0.005 - 0.08 \text{ s}^{-1}$ during different activities like walking, sprinting, and running (Burr et al., 1996; Hansen et al., 2008; Lanyon et al., 1975). Three strain rates were considered in this study since the acetabular implant experiences different strain rates depending on the loading circumstances hence it is impossible to specify a single loading strain rate suitable for compressive strength analysis. The specimens were tested in as-built, stress-relieved, and fully annealed conditions at all these three different strain rates. To guarantee the validity and reliability of the findings, each test was repeated twice. Specimens were compressed parallel to the LPBF building direction. The compression test matrix for this study is outlined in **Table 3. 6**.

Table 3. 6: The compression testing matrix used in this study.

Category	Sample Condition	Strain rate (mm/sec)	Number of Samples
A	As-built	0.015	2
		0.028	2
		0.04	2
B	Stress-relief annealed	0.015	2
		0.028	2
		0.04	2
C	Full annealed	0.015	2
		0.028	2
		0.04	2

3.9.2. Vicker's Microhardness Test.

Microhardness measures the material's resistance to plastic deformation mostly from an indentation or scratch. Struers Duramin-40 Vickers microhardness tester (Struers ApS, Denmark), which utilizes a pointed diamond pyramid with a square base as the indenter (having an included angle of 136°), was used to conduct microhardness test along the build direction of LPBF Ti-6Al-7Nb samples. Equipped with a 5MP evaluation camera, this microhardness tester has been automated by linking the indenter apparatus to an integrated computer for touch screen or mouse operation. Important system functions such as indent position, indent spacing, hardness value computation, and data plotting are managed by the software package. In this study, indentations spaced 200µm apart were made along the SLM specimens build direction using 500gf load and a dwell time of 10s (see **Figure 3. 20**).

After pressing the indenter on the specimen at a specified load of 500gf, the average diagonal dimension (L) of the indent generated on the test sample was measured. These details were used to calculate the Vicker's Microhardness number as shown in **Equation 3. 2**;

$$HV = 2000P \frac{\sin\left(\frac{a}{2}\right)}{L^2} \quad \text{Equation 3. 2}$$

Where P represents the applied load (gf), a denotes the indentation face angle, and L indicates the average diagonal length (µm).

A Vickers indenter with a face angle of 136° is used to measure Vickers microhardness in this study, determined by simplifying **Equation 3. 2** to **Equation 3. 3** for the calculation.

$$HV = 1854.4 \frac{P}{L^2} \quad \text{Equation 3. 3}$$

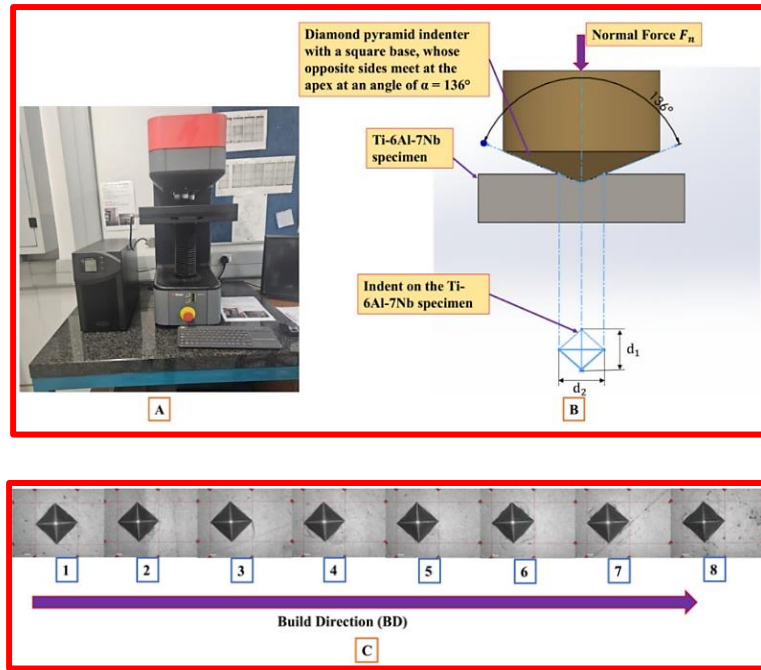


Figure 3. 20: (A) Struers Duramin-40 Vickers microhardness tester. (B) Details of the indenter and indentation on the Ti-6Al-7Nb specimen. (C) The Vicker's microhardness test points (1-8) along Ti-6Al-7Nb specimens' build direction.

3.9.3. Tribological performance analysis.

This test was conducted to investigate the wear volume (V_w), coefficient of friction (COF), wear rate (WR), and wear mechanisms in as-built, and heat-treated LPBF Ti-6Al-7Nb specimens under contact stresses replicating those experienced by the acetabular implant. Prior to wear testing, the specimens underwent the same grinding and polishing procedures as were used for metallographic preparation, as described in section 3.7.2, to maintain a uniform contact area.

Linearly reciprocating ball-on-flat sliding wear tests, simulating the wear condition of the human hip joint, were conducted using an Rtec Multi-Function Tribometer equipped with Rtec MFT17 software, (MFT-5000 Universal Tribometer, Rtec Instruments, USA), as per G 133-05 (2016) standard (Committee, 2016). **Figure 3. 21** shows the schematic wear test set up and the Rtec Multi-Function Tribometer used in this study. The wear depth (z), and coefficient of friction (COF) at every 10 seconds were automatically computed by the Rtec Tribometer's MFT 17 software while its white light profiler provided the 2D and 3D images of the wear tracks. This tribological test was done along the Ti-6Al-7Nb specimens' build direction.

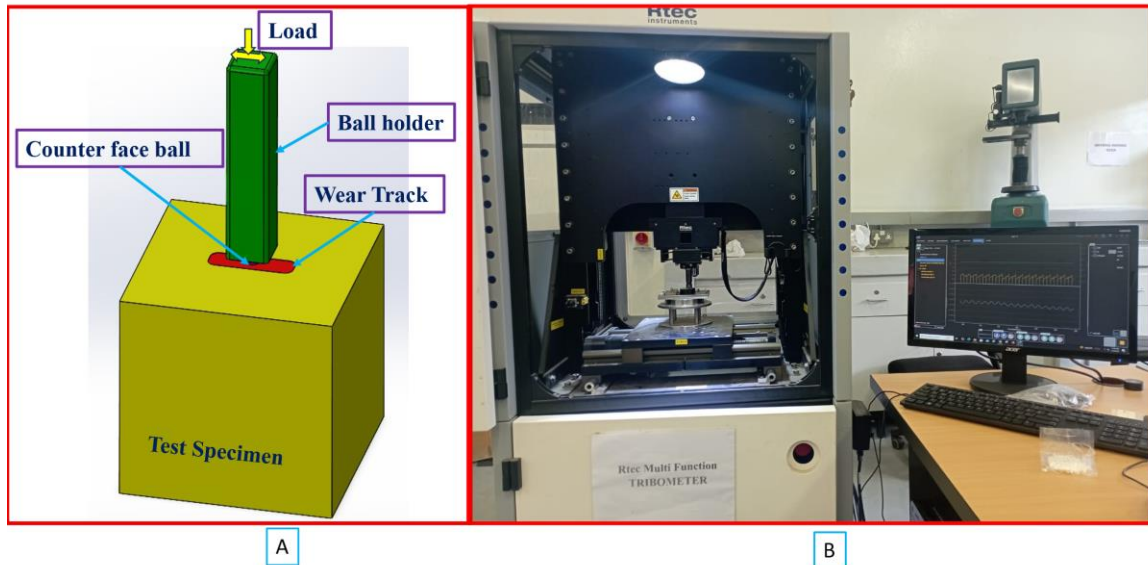


Figure 3. 21: (A) Schematic representation of the tribometer set up. (B) Rtec Multi-function Tribometer used in this study at Materials Laboratory in BIUST, Palapye, Botswana.

The contact stresses applied during the wear test needed to correspond to those experienced at the acetabulum of the normal hip joint. Literature ([Dantas et al., 2023](#); [Hodge et al., 1986](#)) has confirmed that the hip joint experiences 2 to 18 MPa contact pressures when the acetabulum and femoral head are in conforming contact under optimal concentric loading. However, non-conforming articulation of the femoral head and acetabular socket attributable to joint laxity causes edge loading on the acetabular implant resulting in abnormally high, damaging contact stresses reaching more than 950 MPa ([Cho et al., 1992](#); [Sanders & Brannon, 2011](#)). The chosen loads of 10, 15, and 20 N corresponds to high loading conditions and results in very high contact pressures as per the Hertz contact theory ([Hertz, 1881](#)). The human hip joint functions as a tribological system with a ball-in-socket configuration. This design characterizes the femoral head and acetabular cup as highly conforming articulating surfaces, which are denoted by radii, R_1 and R_2 , respectively. Theoretically, the hip joint's contact geometry is often approximated as a sphere-on-flat surface or a ball-on-plate configuration resulting in a circular contact area, refer to **Figure 3. 22**.

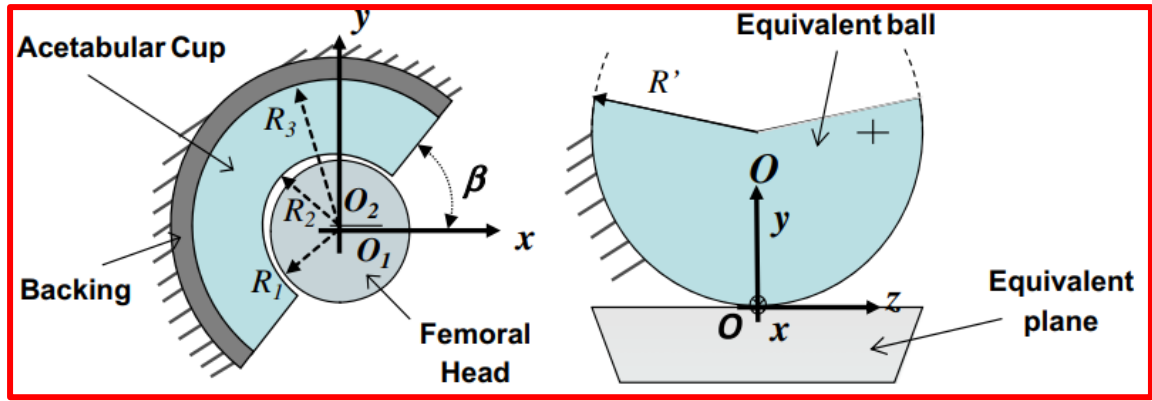


Figure 3. 22: (A) Ball-on-socket configuration of the hip joint. (B) Sphere-on-plate Hertzian geometry for the calculation of contact stresses (Mattei et al., 2011).

Using classical Hertzian theory, an equivalent area was calculated to determine this femoral head and acetabular cup contact area using **Equation 3. 4**.

$$a = \left[\frac{3PR_{\text{eff}}}{2E_o} \right]^{\frac{1}{3}} \quad \text{Equation 3. 4}$$

Where a represents the circular contact area, P denotes the applied load, R_{eff} represents the effective radius, and E' represents the effective modulus elasticity of the two contacting materials.

Equation 3. 5 was used to calculate R_{eff} , the effective radius of the sphere.

$$R_{\text{eff}} = \frac{R_1 R_2}{R_2 - R_1} \quad \text{Equation 3. 5}$$

Where R_1 and R_2 denotes the radii of the femoral head and the acetabular cup accordingly.

Additionally, the equivalent elastic modulus E_o , and the contact stresses Sc were determined using

Equation 3. 6 and **Equation 3. 7** respectively.

$$E_o = \left[\frac{(1 - \nu_1^2)}{E_1} \right] + \left[\frac{(1 - \nu_2^2)}{E_2} \right] \quad \text{Equation 3. 6}$$

$$Sc = 0.918 \left[\frac{P}{D^2 E_o^2} \right]^{\frac{1}{3}} \quad \text{Equation 3. 7}$$

Where; $E_{1,2}$ are the Young's moduli of the two contacting materials (Pa), $\nu_{1,2}$ are the Poisson's ratios of the respective materials in contact (dimensionless), P denotes the applied load (N), D is the sphere's diameter (m) and E_o corresponds to the effective elastic modulus.

In order to attain comparable, even higher contact stresses than those recorded in both the normal hip joint and hip joint simulators, the aforementioned formulae, **Equation 3. 4** to **Equation 3. 7** were used to determine the appropriate applied normal load and radius of the counter ball. Thus, 10, 15, and 20 N loads producing contact pressures more than 950 MPa were chosen to simulate all these contact conditions within the hip joint (refer to **Table 3. 7**)

Table 3. 7: The ball-on-disk sliding wear testing parameters.

Experiment Number	Sample Condition	Load (N)	Hertz Contact Pressure (MPa)	Number of Samples
1	As-built	5	977	3
2	As-built	10	1231	3
3	As-built	15	1409	3
4	Stress Relieved	5	898	3
5	Stress Relieved	10	1132	3
6	Stress Relieved	15	1295	3
7	Full Annealed	5	1043	3
8	Full Annealed	10	1314	3
9	Full Annealed	15	1504	3

The counter-wear body was a spherical alumina (Al_2O_3) ball indenter having a diameter of 6.35 mm. Several factors led to the use of Al_2O_3 balls counter face. Firstly, most hip implants use a ceramic-on-polymer (CoP) bearings comprising of either zirconia or Al_2O_3 femoral heads and conventional UHMWPE liner backed by Ti-6Al-4V alloy acetabular cup ([Beckmann et al., 2015](#)). Secondly, Al_2O_3 has a higher micro-hardness (1500-1700 HV) than Ti-6Al-7Nb hence it is anticipated that deformation processes during sliding will, in this

instance, be limited to the Ti-6Al-7Nb alloy (Mu, 2024). The wear test parameters included a sliding distance of 4 mm, a duration of 10 mins, an acceleration of $0.1 \frac{mm}{s^2}$, and a velocity of $5 \frac{mm}{s}$, under varying loads of 10, 15, and 20 N.

The experiments were replicated three times using a new Al_2O_3 ball per each test condition to observe repeatability. The wear volume (mm^3) was calculated using **Equation 3. 8** and **Equation 3. 9**, adopting the approach used in the analyses performed by Malikongwa et al. (Malikongwa et al., 2021) and Nyakundi et al. (Nyakundi et al., 2024).

$$V_w = \frac{a\pi h^2}{3b} [3R - h] \text{ (mm}^3\text{)} \quad \text{Equation 3. 8}$$

$$h = R - \sqrt{R^2 - b^2} \text{ (}\mu\text{m)} \quad \text{Equation 3. 9}$$

Where: V_w – wear volume, R – alumina ball (indenter) radius (3.175 mm), a is the wear track half length, and b denotes the wear track's width (see **Figure 3. 23**).

Additionally, wear rate, $W_R \left(\frac{mm^3}{N \cdot m} \right)$, was calculated using the Archard's wear model (Ju et al., 2021) (**Equation 3. 10**), taking into consideration the total sliding distance, S_d (4 mm), wear volume V_W (mm^3), and the applied normal load (N_l).

$$W_R = \frac{V_W}{N_l \times S_d} \quad \left(\frac{mm^3}{N \cdot m} \right) \quad \text{Equation 3. 10}$$

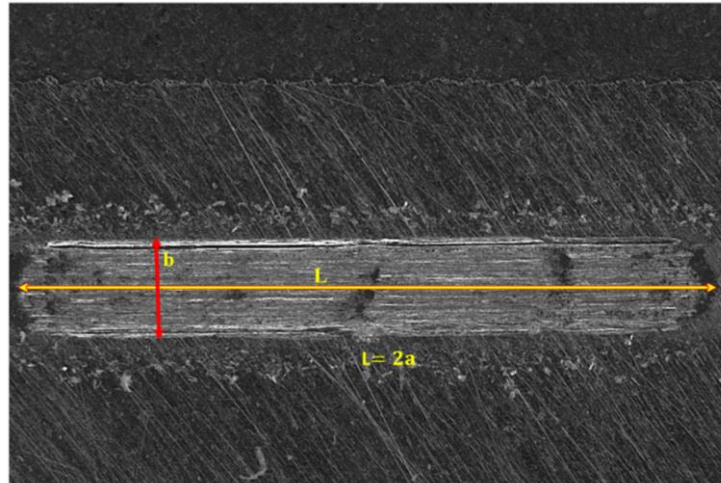


Figure 3. 23: Wear Track parameters used for wear rate analysis after ball-on-flat specimen wear test.

3.9.3.1 Stereo Microscopy Analysis of the Wear Tracks.

Following the wear test, an Olympus SZX7 Stereo microscope (refer to **Figure 3. 24**) equipped with a Preciv imaging software was utilized to examine the wear tracks. This stereo microscope was utilized to accurately measure the length of the wear track. Additionally, the wear track's width was measured at three different points distributed evenly over the track length and averaged. The compiled data was utilized to assess wear performance by analysing volumetric material loss and wear rate in both as-built and heat-treated LPBF Ti-6Al-7Nb specimens as per **Equation 3. 8** to **Equation 3. 10**.

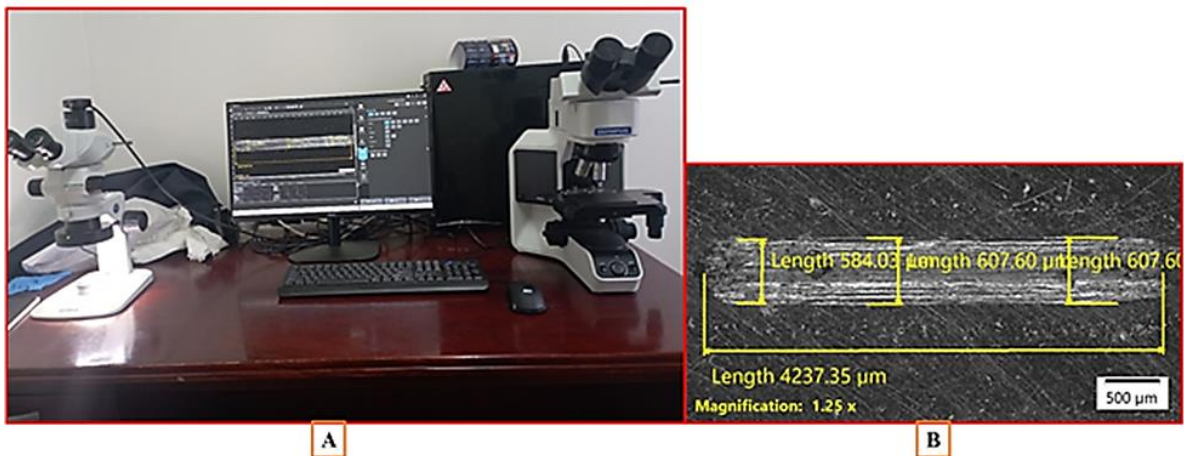


Figure 3. 24: (A) Olympus SZX7 stereo microscope and (B) Measurements on the wear track for calculating the volume loss, and wear rate at the BIUST's Laboratory under the ELbM Project.

3.9.3.2 Ultrasonic cleaning.

Ultrasonic cleaning is a critical step in preparing the wear samples for Scanning Electron Microscopy (SEM) analysis. In our study, the as-built and heat-treated LPBF Ti-6Al-7Nb wear samples were ultrasonically cleaned in ethanol for 10 minutes, refer to **Figure 3. 25**, before mounting on the stage (vacuum chamber) of the SEM. Ultrasonic cleaning ensures optimal sample condition for SEM analysis, providing high-quality and reliable imaging results (Ghassemi et al., 2015).



Figure 3. 25: Ultrasonic cleaning of the Ti-6Al-7Nb specimens after wear test prior to SEM analysis of the wear tracks.

3.9.3.3 SEM and EDS Analysis of the wear tracks.

The wear tracks' surface morphology and elemental composition were examined in detail using a JEOL JSM-6610 SEM (Japan) integrated with an EDS system. This arrangement allowed for precise imaging and elemental characterization, unveiling important details about the material's surface properties and chemical composition.

A comprehensive evaluation of wear tracks through SEM and EDS is critical to understand the wear mechanisms of LPBF Ti-6Al-7Nb specimens. Surface morphology analysis reveals important features like grooves, pits, and cracks in the wear tracks which helps to determine the dominant wear mechanisms in the material, such as abrasive, adhesive, and fatigue wear (Ju et al., 2021). The shift in material performance and wear resistance due to the heat treatment process can easily be observed with SEM analysis (Su et al., 2022).

The EDS component of SEM (JEOL JSM-6610, Japan) provides the chemical composition of the wear tracks, critical for detecting any transfer of material between the contacting surfaces or the presence of wear debris. Some suspected oxidized patches within the wear tracks were analyzed using the spot EDS analysis to measure their elemental composition and confirm if they indeed underwent some oxidation to confirm oxidative wear mechanism (Su et al., 2022). While EDS identifies which elements are present in the wear scar, it does not provide accurate data for the wt.% of each element.

3.10. Objective 3: Corrosion resistance and in vitro biocompatibility analysis through immersion in simulated body fluid.

In vitro biocompatibility and corrosion resistance study were performed through immersion corrosion testing in simulated body fluids conducted as per ASTM G31-72 (2004) standard procedure (Pa & Materials, 2004). The unmounted as-built and heat-treated LPBF Ti-6Al-7Nb specimens having dimensions of $L \times W \times H = 10\text{mm} \times 5\text{mm} \times 5\text{mm}$ were metallographically prepared through grinding and polishing as explained in section 3.7.2.

3.10.1 Ultrasonic cleaning.

A total of thirty six Ti-6Al-7Nb specimens were prepared for analysis, consisting of twelve as-built, twelve stress-relief annealed, and twelve fully annealed samples. Then, ultrasonic cleaning was performed in three sequential stages: acetone for the first stage, ethanol for the second, and deionized water for the third, with a cleaning duration of 15 minutes per cleaning stage (**Figure 3. 26**). The choice of the ultrasonic cleaning solvents was made as per recommendations in ASTM G31-72 (2004) standard procedure (Pa & Materials, 2004) to avoid contamination and ensuring cleanliness for valid and credible results.

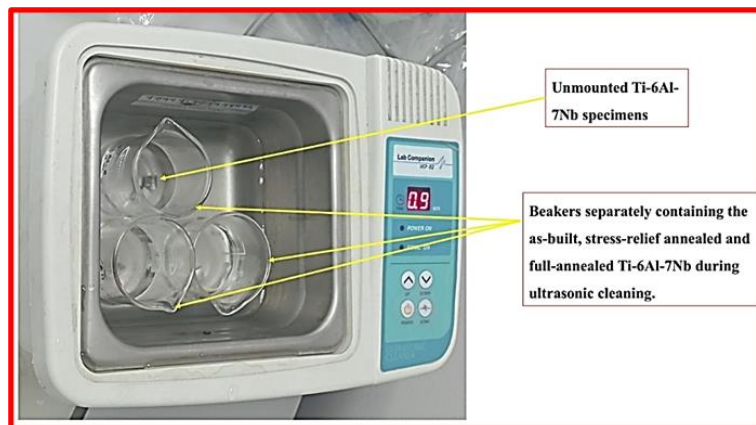


Figure 3. 26: Ultrasonic cleaning of the as-built, stress-relief annealed, and fully annealed Ti-6Al-7Nb specimens for immersion corrosion.

After ultrasonic cleaning, the samples were dried using a blower and subsequently weighed on an analytical balance of $\pm 0.001\text{g}$ precision (see **Figure 3. 27**). In the subsequent stages, the samples were carefully handled using clean gloves to avoid contaminating the specimen's surfaces.

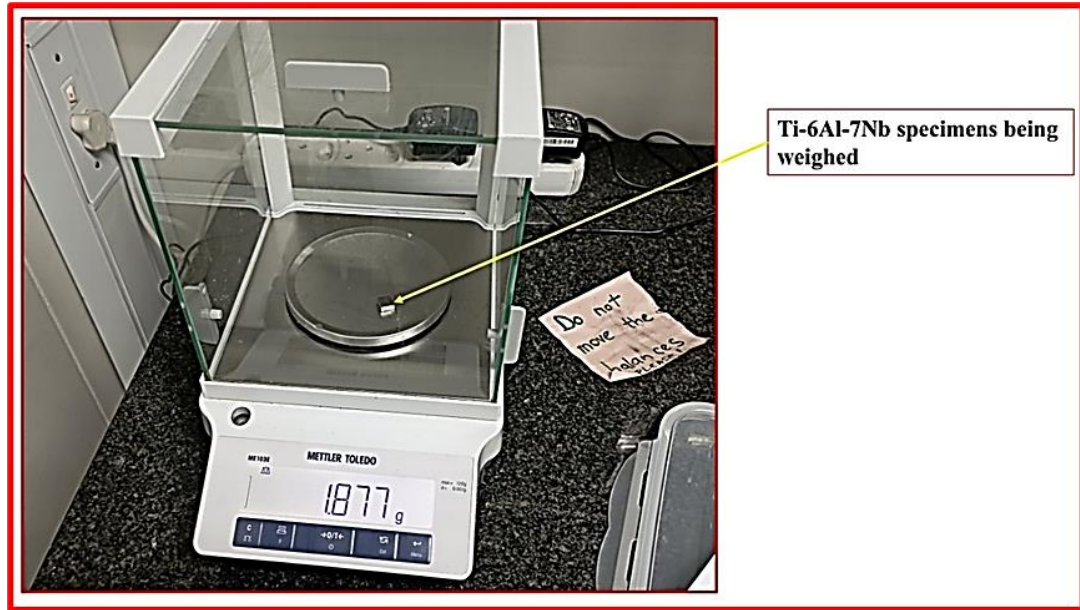


Figure 3. 27: Weighing of Ti-6Al-7Nb specimen on analytical balance prior to immersion in body simulated fluid.

3.10.2 Immersion of the LPBF Ti-6Al-7Nb samples and composition of the simulated body fluid.

The simulated body fluid, Dulbecco's phosphate buffered saline solution (DPBSS) with a pH~7.4 was utilized for this in vitro biocompatibility and corrosion resistance study. By mimicking the osmolarity and ion concentrations of extracellular fluid as can be seen in **Table 3. 8** ([Balasubramanian Gayathri et al., 2022](#); [Vaira Vignesh et al., 2020](#)), DPBSS, offers a physiologically realistic environment for studying the corrosion behaviour of hip prostheses. To replicate the human physiological conditions, a digital thermostatic water bath maintained the DPBSS temperature at $37^{\circ}\text{C} \pm 0.1^{\circ}\text{C}$. The specimens were split into six batches with each batch containing 2 as-built, 2 stress-relief annealed (HT650), and 2 full annealed (HT850) samples making a total of 6 specimens per batch. This was purposely done to guarantee test reproducibility.

Table 3. 8 Dulbecco's phosphate buffered saline solution chemical composition.

Component	Chemical Formula	Concentration mg/L
Anhydrous calcium chloride	CaCl_2	100
Anhydrous magnesium chloride	MgCl_2	47
Potassium chloride	KCl	200
Potassium phosphate monobasic	KH_2PO_4	200
Sodium chloride	NaCl	8000
Anhydrous sodium phosphate dibasic	Na_2HPO_4	1150

The samples were immersed in DPBSS at different durations of 24 hours, 72 hours, 120 hours, 168 hours, 240 hours, and 336 hours (1, 3, 5, 7, 10, and 14 days respectively) using McCartney tubes immersed in water baths depicted in **Figure 3. 28**.

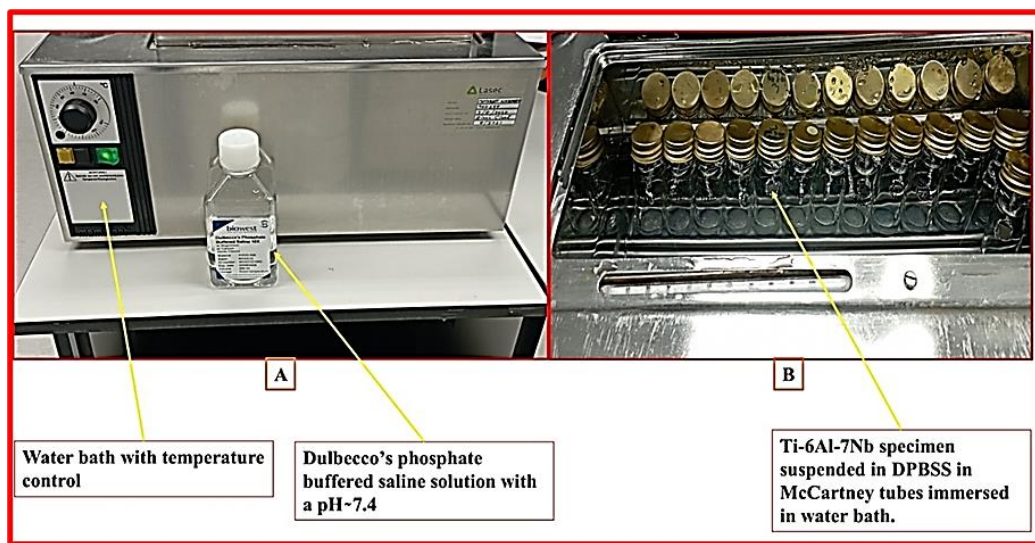


Figure 3. 28: (A) Dulbecco's phosphate buffered solution used to mimic the ionic concentration and pH of the body fluids and water bath used to control the test environment temperature to the body temperature of 37°C. (B) Ti-6Al-7Nb specimens exposed to the simulated body environment.

Following a 24-hour immersion, the first batch was removed from the simulated body fluid. The second, third, fourth, fifth, and sixth batches were removed after 72 hours, 120 hours,

168 hours, 240 hours, and 336 hours respectively. After removing the specimens from the water bath, they were washed with ethanol, dried using a blower, and weighed using a precision balance to determine the mass difference. The corrosion rate was determined using **Equation 3. 11** (Pa & Materials, 2004).

$$\text{Corrosion rate} = \frac{K \times W}{A \times T \times D} \quad \text{Equation 3. 11}$$

Where;

- K is a constant, 8.76×10^6 , used when the corrosion rate units are millimeters per year (mm/y).
- W denotes mass loss in grams, recorded to the nearest 1 mg.
- A signifies the exposed surface area in cm^2 , measured to the nearest 0.01 cm^2 .
- T represents the exposure duration in hours, rounded to the nearest 0.01h.
- D corresponds to the density of Ti-6Al-7Nb specimens in $\frac{\text{g}}{\text{cm}^3}$. ($D = 4.52 \frac{\text{g}}{\text{cm}^3}$ for Ti-6Al-7Nb ("TIMET TIMETAL® 367 (Ti-6Al-7Nb) Titanium Alloy datasheet,")

3.10.3 Scanning Electron Microscopy (SEM) and EDS Analysis of the corroded surface.

Titanium and its alloys, Ti-6Al-7Nb inclusive, fall within the bioinert biomaterials class as described in section 2.2.3.2.1. Their superior biocompatibility originates from the formation of a surface layer that facilitates hydroxyapatite mineralization, imparting osteoconductive properties within the physiological system (Nishiguchi et al., 2001). Calcium phosphate, especially hydroxyapatite HA_p , is a natural inorganic material present in the human bones and teeth which significantly contributes to the structural strength and regenerative capabilities of the bone (Orlovskii et al., 2002). A ceramic biomaterial, Synthetic Calcium HA_p [HA_p , $\text{Ca}_{10}(\text{PO}_4)_6(\text{OH})_2$], has undergone extensive research for prosthetic applications due to its physicochemical resemblance to the hydroxyapatite mineral inherent in bone tissue. Most of these applications have been on coatings of orthopaedic implants to improve bone osseointegration (Ciobanu et al., 2009).

The spontaneous accumulation of calcium phosphate on titanium alloy surfaces from simulated physiological fluids like Dulbecco's phosphate-buffered saline, has been documented in literature. This process is believed to improve the interfacial integration and bioactive properties of implants, fostering their compatibility within the biological

environment (Sandrini et al., 2007). Hence, this methodology section primarily aims to evaluate the hydroxyapatite mineralization capability of as-built and heat-treated LPBF Ti-6Al-7Nb samples within simulated physiological environments.

The surface characteristics were examined using SEM analysis to observe the surface morphology while EDS characterized the chemical composition of the new phases deposited on the Ti-6Al-7Nb samples after immersion in DPBSS. Additionally, the SEM and EDS analyses presented in this chapter will provide further insight into how heat treatment processes influence the surface morphology and elemental composition of the deposits developed on the LPBF Ti-6Al-7Nb surfaces following immersion in the simulated body fluid.

3.11. Conclusions.

This chapter has presented a comprehensive description and discussion of the current research methodology. The research's materials and methodology have been documented for repeatability and justification of the selected material and methodological approach. The Ti-6Al-7Nb powder materials, the LPBF process, heat treatment, and the characterization techniques of the as-built and heat-treated LPBF Ti-6Al-7Nb samples with regard to acetabular implant application have been thoroughly explained. All the processes and equipment used in this study have been presented.

Chapter 4: Ti-6Al-7Nb Powder Properties and Microstructural Analysis of the As-built and Heat-treated LPBF Ti-6Al-7Nb Specimens.

4.1 Chapter Summary.

The microstructural analysis results of Ti-6Al-7Nb powder, LPBF fabricated Ti-6Al-7Nb specimens, through SEM and XRD, and the impact of annealing treatment on microstructural properties of processed specimens is hereby described and discussed. This chapter aims to examine the processability and annealing treatment response of LPBF Ti-6Al-7Nb samples, enabling the selection of an optimal heat treatment procedure to develop a targeted microstructure for acetabular implant applications.

The first section of this chapter (**section 4.2**) details the findings from the microscopic examination and XRD analysis of Ti-6Al-7Nb powders, highlighting their microstructural characteristics and phase composition. Following this, **section 4.3** and **section 4.4** present an in-depth investigation of the microstructural properties of the as-built and heat-treated LPBF Ti-6Al-7Nb specimens through SEM and EDS. **Section 4.5** focuses on the phase identification in the as-built and heat-treated LPBF Ti-6Al-7Nb samples through XRD analysis. A comparative evaluation of the SEM and XRD findings for these specimens is discussed in **section 4.6**. Lastly, **section 4.7** highlights the key findings of the chapter and concludes with predictions of the optimal microstructure for acetabular implant applications, drawing from both the results of this study and relevant literature.

4.2 Properties of Ti-6Al-7Nb powders.

The results of the powder morphology analysis from SEM, elemental composition analysis, phase composition analysis of the powder through XRD, and particle size distribution are presented in this section.

4.2.1 Scanning Electron Microscopy (SEM) Observation of Ti-6Al-7Nb Powders Morphology.

Gas atomized Ti-6Al-7Nb alloy powders (20-45 μm) sourced from Suzhou Joroyce Products Co. Ltd; China was utilized in this work. SEM images of the Ti-6Al-7Nb powders are presented in **Figure 4. 1**.

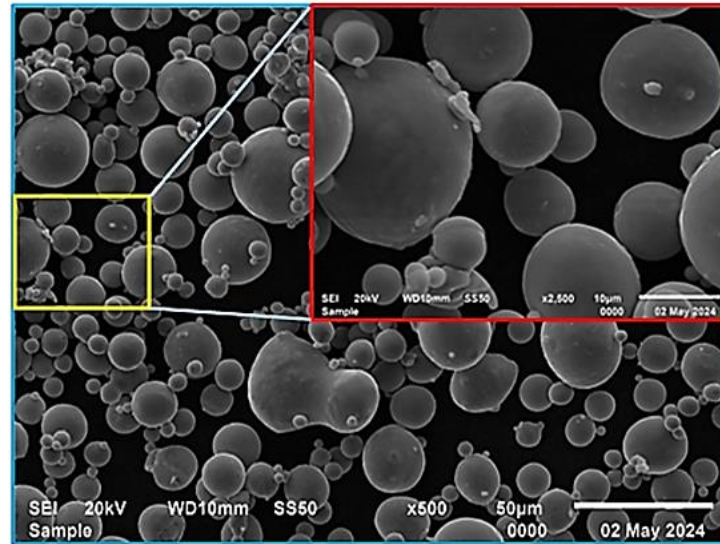


Figure 4. 1: SEM micrographs depicting the particle morphology of the gas-atomized Ti-6Al-7Nb alloy powders.

The powders have a spherical morphology with a few satellites. Spherical powder particles are typically favorable for additive manufacturing due to their excellent flowability and capacity for adequate laser energy absorption ([Jang et al., 2020](#); [Mahamood & Akinlabi, 2017](#)). The generally good flowability observed in spherical powders is primarily attributed to their low frictional interactions, as smooth, curved surfaces glide more easily over one another. However, the adhesion between particles, which can significantly hinder flowability, is influenced less by particle shape and more by other factors such as moisture content or electrostatic forces, especially in powders composed of very fine particles. Moisture can increase inter-particle cohesion through capillary forces, while electrostatic charges arise from particle size reduction and contact interactions, both contributing to reduced flowability ([Schulze, 2007](#)). This distinction is critical to understanding powder behavior and has been well-documented in classical powder technology literature ([Enferad et al., 2021](#); [Hulme, 2022](#)).

Furthermore, the high magnification insert clearly shows the presence of a few satellites, small powder particles adhering to the larger spherical powder particles. Different studies have reported on the presence of satellites in titanium alloys produced by gas atomization process ([Derimow et al., 2022](#); [Popov et al., 2018](#)). Though satellites have potential benefits of reducing unmelted zones in the LPBF product as satellites melt more easily reducing the risk of unmelted powder on the surface, their fraction in the powder should be minimized

as they can markedly impact flowability and packing density on the powder bed leading to defects in the printed part (Popov et al., 2018).

4.2.2 Particle Size Distribution of Ti-6Al-7Nb Powder.

The particle size distribution of as-received Ti-6Al-7Nb powders was determined by laser diffraction method and its findings are presented in **Figure 4. 2**. The PSD analysis revealed the equivalent particle diameter of $D_{10}=19\ \mu\text{m}$, $D_{50}=34.8\ \mu\text{m}$, and $D_{90}=56.8\ \mu\text{m}$ and $36.3\ \mu\text{m}$ average size.

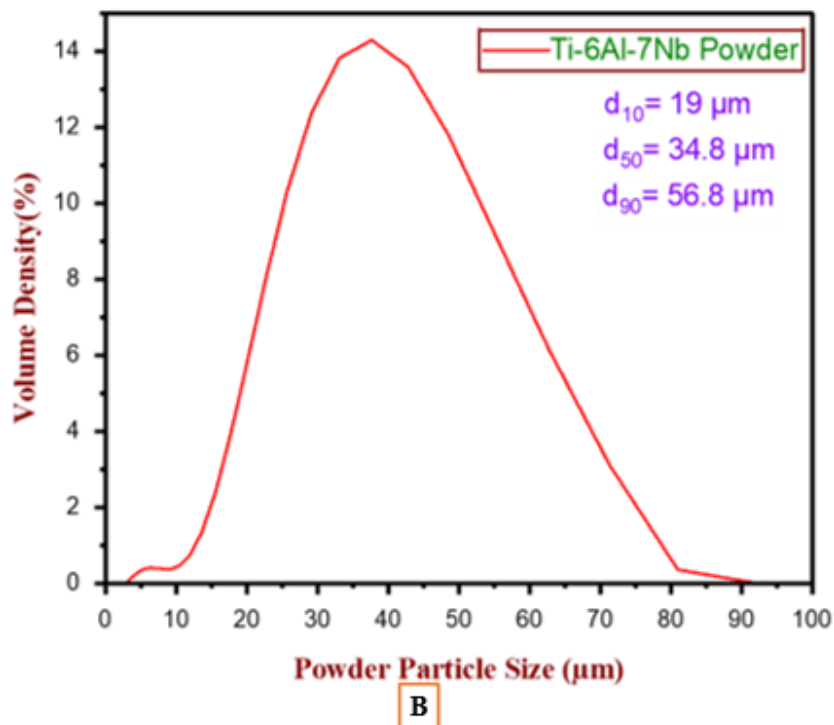


Figure 4. 2: The Ti-6Al-7Nb powder's particle size distribution..

4.2.3 Chemical Composition of Ti-6Al-7Nb Powders.

The elemental composition of the Ti-6Al-7Nb powders, determined by EDS analysis, is detailed in **Table 4. 1**. The EDS provides semi-quantitative data and has limited accuracy for light elements such as carbon, oxygen, hydrogen, and nitrogen due to their low atomic numbers and weak characteristic X-ray signals ("Energy-Dispersive X-ray Spectroscopy (EDS)," 2019). The regulatory reference standard for Ti-6Al-7b alloy in surgical implants is F1295 (F. Committee), presented in the second row of **Table 4. 1**. The data indicate that this powder complies with the medical regulations' standard for surgical implant applications. This compliance confirms that the supplied Ti-6Al-7Nb powder does not

contain harmful elements or contaminants that could cause toxicity or adverse reactions in the body system.

Table 4. 1: Elemental composition of Ti-6Al-7Nb alloy powder (wt.%).

Elements		Ti	Al	Nb	Ta	Fe	C	O	N	H
Ti-6Al-7Nb Powder		Bal.	6.12	7.08	0.01	0.15	0.01	0.175	0.024	0.0012
Wrought (F1295)	Min	Bal.	5.50	6.50	0.00	0.00	0.00	0.00	0.00	0.00
	Max	Bal.	6.50	7.50	0.50	0.25	0.08	0.20	0.05	0.009

4.2.4 Phase Composition of Ti-6Al-7Nb Powders.

The XRD diffractogram for Ti-6Al-7Nb powder is presented in **Figure 4. 3**. The diffraction pattern reveals the presence of α peaks at $2\theta = 35.35^\circ, 38.59^\circ, 40.47^\circ, 53.30^\circ$, and 63.63° and also a β peak observed at $2\theta = 42.48^\circ$ (crystallographic plane (111)). This confirms the $\alpha + \beta$ crystallographic structure of Ti-6Al-7Nb powders.

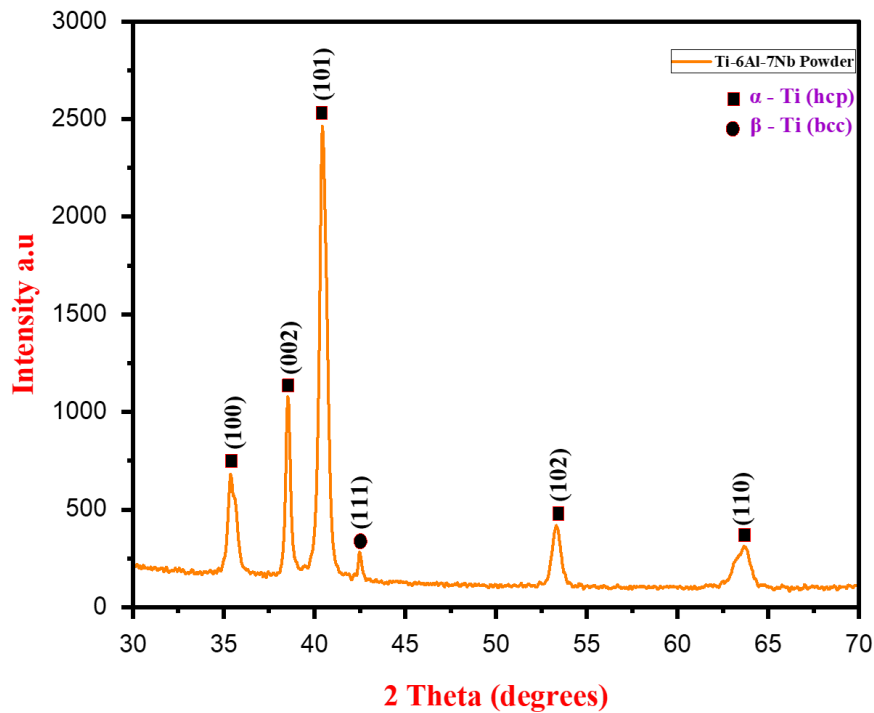


Figure 4. 3: The XRD diffractogram for Ti-6Al-7Nb powder.

4.3 Microstructure of the As-built Ti-6Al-7Nb Specimens.

Figure 4. 4 gives the microstructure of the as-built Ti-6Al-7Nb samples observed under SEM along the build direction. In this SEM image, the α' phase is depicted as dark regions,

whereas the β phase appears as small bright spots. Additionally, prior β columnar grains are clearly growing parallel to the build direction, a formation directly influenced by the thermal processing history of LPBF. During LPBF, the laser beam's heat is primarily dissipated downward through the previously built layers and the build plate (Eshawish et al., 2021; C. Xu et al., 2020). This process creates a temperature gradient, with the highest temperature at the molten region and decreasing temperatures along the solidified layers below. The prior β grains align with the maximum temperature gradient, leading to their epitaxial growth across the deposition layers. This epitaxial growth means that the prior β grains maintain their crystallographic orientation through successive layers, reinforcing the columnar structure.

Upon cooling the samples down the β -transus temperature, the body centred cubic (BCC) β -phase transforms to the hexagonal close packed (hcp) α' -phase. This transformation is primarily dictated by the rapid cooling rate, which typically ranges from $(10^3 - 10^6 \frac{^{\circ}\text{C}}{\text{s}})$ (Eshawish et al., 2021; C. Xu et al., 2020) in the LPBF process. Such extreme cooling rate prevents the development of equilibrium α -phase and instead produces α' -phase, a metastable martensitic variant of α -phase. Thus, the prior β -grains serve as the parent material from which the needle-like acicular α' martensite laths precipitates. This concurs with the research results of Chao et al. (C. Xu et al., 2020) and Yang et al. (J. Yang et al., 2016).

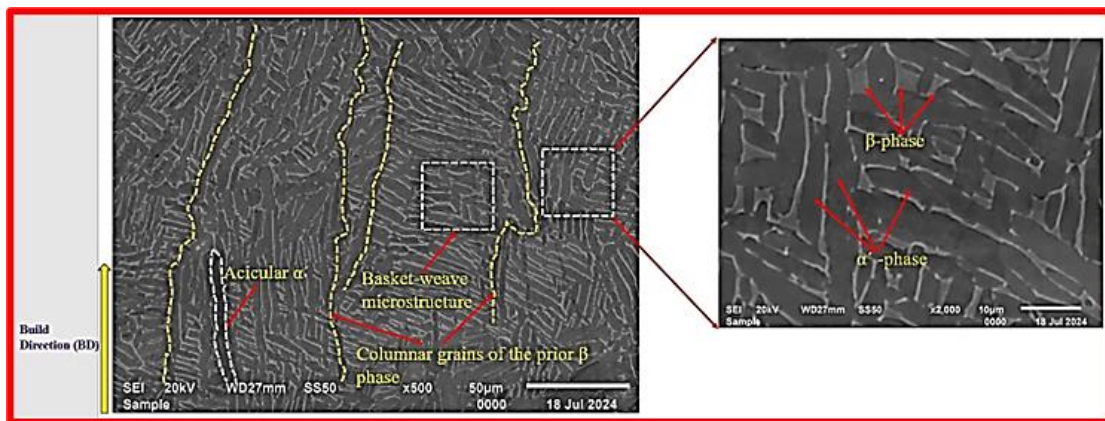


Figure 4. 4: SEM micrographs displaying the microstructure of the As-built Ti-6Al-7Nb Specimen. A higher magnification insert on the right shows the basket-weave microstructure arrangement.

Additionally, the microstructure inside the prior columnar β -grains was characterized by a fine, interlocking pattern of α' -phase lamellae within the β -phase matrix, resembling a

woven basket hence called the basket-weave microstructure. Ren et al. (Ren et al., 2017) reported similar observation during LPBF of Ti-6Al-4V. This microstructure's significance in orthopaedic implants application was highlighted by Spotose (Spotose, 2022) and Gheorghe (Gheorghe et al., 2019) who noted that it enhances the mechanical properties of titanium alloys, particularly strength and corrosion resistance. However, they reported lower ductility resulting from this microstructure due to more grain boundaries which inhibits the material's ability to plastically deform and poses significant implications on acetabular implants' performance and longevity. Less ductile acetabular implants facilitate stress shielding effect, increase the risk of fracture, and compromise stability as they are not flexible enough to accommodate natural movements of the hip joint (Ziranu et al., 2024).

4.4 Microstructure of the Heat-treated LPBF Ti-6Al-7Nb Specimens.

In this section the transformation of α' to $\alpha+\beta$ during the annealing process at two different temperatures (650°C and 850°C) having a holding time of two hours and subsequent air cooling was investigated. **Figure 4. 5** presents the microstructure of the stress relief annealed (HT650°C) and fully annealed specimens (HT850°C) at various magnifications along the build direction.

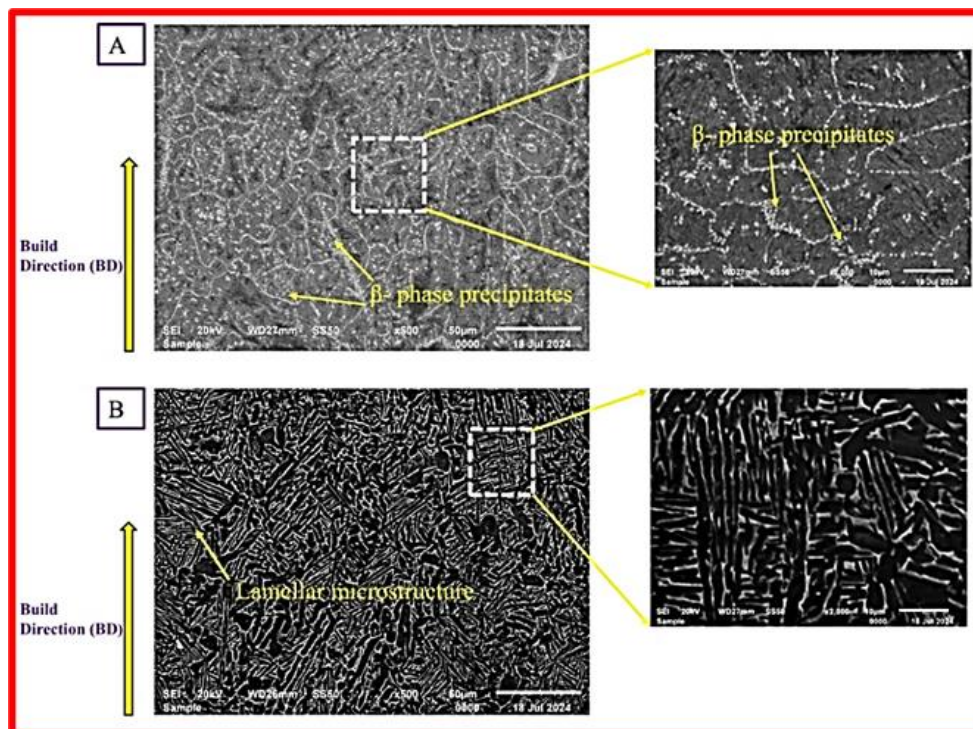


Figure 4. 5: Microstructure of the heat-treated Ti-6Al-7Nb Specimens. (A) Stress-Relief Annealed (at 650°C for 2hours followed by air cooling) specimen, and (B) Full Annealed Specimen (at 850°C for 2hours followed by air cooling). The higher

magnification inserts on the right present details of the precipitation of the β -phase in stress-relief annealed and lamellar arrangement in Full annealed specimen.

Microstructure of the stress-relieved (HT650°C) specimen (see **Figure 4. 5A**) is characterized by the nucleation of β -phase particles along grain boundaries, a clear indication that stress-relief annealing treatment facilitated martensite decomposition and β phase nucleation. However, annealing the specimens at 650°C limits the β -phase growth to nanoparticle size precipitates and the presence of α' structure could still be observed. Additionally, the nucleation of the β -particles is heterogenous; where particles preferentially form at the grain boundaries possibly because these regions exhibit a higher energy state than the grain interior which provide the thermodynamic driving force needed for nucleation (Etesami et al., 2022).

Increasing the annealing temperature to 850°C promotes the β -phase's growth and causes the β -phase's shape to change from precipitate to more of a lamellar structure. The full-annealing heat treatment process (HT850°C) alters the microstructure substantially compared to the as-built (**Figure 4. 4**) and stress-relief annealed (**Figure 4. 5A**) samples. Compared with the as-built microstructure, the fine acicular α' martensite has been thoroughly transformed into a refine lamellar $\alpha+\beta$ structure ($\alpha' \rightarrow \alpha + \beta$). One key benefit of lamellar microstructure is its ability to enhance wear and fatigue resistance, contributing to the extended durability of acetabular implants (Correa Rossi et al., 2024).

Table 4. 2 summarizes the microstructural properties for all the LPBF Ti-6Al-7Nb specimens' condition. Microstructure development in LPBF Ti-6Al-7Nb varies across as-built, stress-relief annealed, and fully annealed conditions, leading to the variation in the type, and shape of the constituent phases observed. The volume fractions were calculated using MATLAB image analysis tools while the microstructure sizes were measured using the line intercept method.

Table 4. 2: A summary of the microstructural features for as-built, stress-relief annealed, and fully annealed LPBF Ti-6Al-7Nb specimens.

Sample Condition	Phase Constituent	Volume fraction		Shape	Size (μm)
		$V_{f,\alpha}(V_{f,\alpha'})/\%$	$V_{f,\beta}/\%$		
As-built	$\alpha' + \beta$	99.2 ± 0.02	0.9 ± 0.05	Basket-weave	$\sim 6.27 \pm 1.03$
HT650	$\alpha + \beta$	84.2 ± 3.85	15.4 ± 3.85	Near spherical	
HT850	$\alpha + \beta$	79.4 ± 1.94	20.3 ± 1.5	Lamellar	$\sim 5.02 \pm 0.97$

4.5 XRD Analysis of the As-built, and Heat-treated LPBF Ti-6Al-7Nb Specimens.

Figure 4. 6 exhibits the XRD profiles of the as-built specimens, stress-relieved specimens, as well as full annealed specimens. The stress-relief annealed, and full-annealed Ti-6Al-7Nb specimens' peaks match both the BCC (JCPDS 01-077-3482) and HCP (JCPDS 04-001-8963) crystal structures, confirming the presence of $\alpha+\beta$ microstructure. On the contrary, the as-built specimen displays XRD peaks pattern associated with the HCP (JCPDS 04-001-8963) crystal structure only.

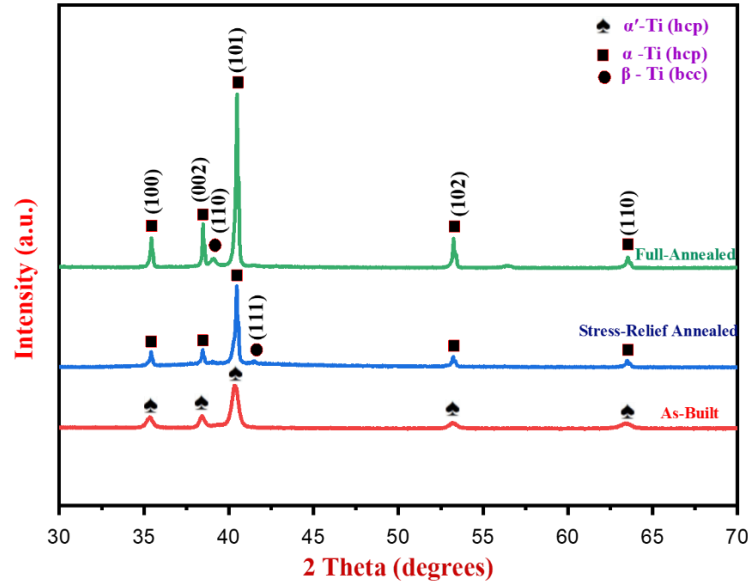


Figure 4. 6: XRD patterns of the as-built, stress relief annealed, and full-annealed Ti-6Al-7Nb alloy specimens.

The crystallographic planes (100), (002), (101), (102), and (110) display the highest diffraction peaks and these planes are linked to the hexagonal close packed (HCP) Ti-crystalline structure. Thus, either the α phase or the α' martensite can be responsible for these HCP peaks, because both phases possess an HCP crystal structure and identical lattice parameters, leading to XRD peaks occurring at the same angular positions (Xu et al., 2020). For the raw Ti-6Al-7Nb powder, a weak β -phase peak can be observed at position $2\theta = 42.5^\circ$ (crystallographic plane (111)). Thus, the raw powder exhibits a biphasic microstructure, incorporating both α and β phases. In the as-built specimen, a β -phase peak at crystallographic plane (111) observed in the raw powder disappears, only (100), (002), (101), (102), and (110) peaks are displayed in the XRD pattern. In this scenario, these peaks should correspond to α' martensite developed by the rapid cooling rates ($10^3 - 10^6 \frac{^\circ\text{C}}{\text{s}}$) in the LPBF process (Yan et al., 2018). Thus, the primary constituent phase of the as-built LPBF Ti-6Al-7Nb specimens is α' martensite. The rapid-cooling rate inhibits diffusion of elements (specifically HCP niobium; Nb, the β -phase stabilizer) consequently, niobium content is higher in the α' than the α phase (Xu et al., 2020). After heat treatment, the BCC (β phase) peaks start to develop at $2\theta = 41.54^\circ$ in the stress-relief annealed (HT650) specimen which becomes more pronounced in the full-annealed (HT850) specimen at $2\theta = 39.17^\circ$. This confirms that the heat-treated specimens possess an $\alpha+\beta$ microstructure, with

the full-annealed specimen containing a greater amount of β -phase compared to the stress-relief annealed specimen.

The hcp peaks in the as-built LPBF Ti-6Al-7Nb specimens are noticeably broader than hcp peaks for the heat treated (HT650 and HT850) samples. As displayed in **Figure 4. 7**, the XRD peak at crystallographic plane (101) of the as-built samples exhibit a broader full width at half maximum (FWHM) than the heat-treated (HT650 and HT850) specimens. This suggests that the as-built specimens contain lattice defects, which raises residual stress. This supports the assertion that these peaks should be of the metastable phase α' martensite. It is possible that these peaks of the non-equilibrium phase are broader due to the higher solute content of the trapped niobium in the atomic structure due to the faster cooling rates. This supersaturation of niobium results in the crystal structure's deformation and the broadening of the peaks as has been previously observed with other β stabilizers like vanadium in the $\alpha + \beta$ Ti-6Al-4V alloy (Dai, Zhang, Zhang, Chen, et al., 2016).

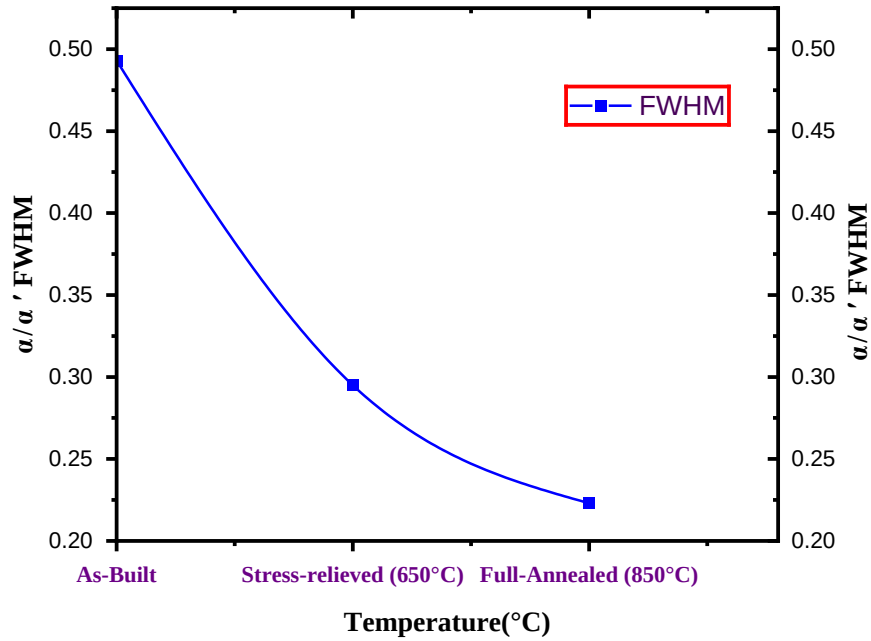


Figure 4. 7: Impact of heat treatment on the Full Width at half Maximum (FWHM) values of selectively laser melted Ti-6Al-7Nb alloy specimen.

FWHM reduction following heat treatment processes confirms that the annealing temperatures are adequate to eliminate the material's residual stresses. Additionally, increasing annealing temperature enhances α -phase peak intensity, indicating the

transformation of $\alpha' \rightarrow \alpha$. Thus, it can be inferred that upon annealing, the metastable phase (α') stabilizes through the development of the stable phases α and β . Since niobium is supersaturated in the α' phase, the heat treatment causes it to diffuse into the β phase which has a greater capacity to dissolve more niobium than both the α and α' phases. The lack of β -phase peaks in as-built samples suggests limited β -phase retention after the LPBF process. The increase in β -phase content from HT650 to HT850 is observed through XRD peak intensities, which indicate the transformation and stabilization of the β -phase occurring during annealing. Thus, the following can be proposed as an ascending order of β phase content in LPBF Ti-6Al-7Nb specimens: $C_{\beta}(\text{As} - \text{built}) < C_{\beta}(\text{HT650}) < C_{\beta}(\text{HT850})$.

4.6 Comparing SEM and XRD Results for the As-built and Heat-treated Ti-6Al-7Nb Specimens.

This study utilized SEM to analyze the microstructure and identify phases developed during laser powder bed fusion process and subsequent heat treatment of Ti-6Al-7Nb alloy. X-ray diffraction (XRD) characterization is then conducted to validate the presence of these phases. The phase constituents observed in as-built and heat-treated LPBF Ti-6Al-Nb specimens, as determined by SEM and XRD, are presented in **Table 4. 3**.

Table 4. 3: Phases in the as-built and heat-treated Ti-6Al-7Nb as identified by SEM and XRD analysis.

Sample Condition	Identified Phases	
	SEM	XRD
As-built	$\alpha' + \beta$	α'
Stress-relief annealed	$\alpha + \beta$	$\alpha + \beta$
Full annealed	$\alpha + \beta$	$\alpha + \beta$

The as-built Ti-6Al-7Nb specimen had two phases as identified by SEM, the dark α' phase and the bright β phase. However, in the XRD analysis of the as-built samples, no peaks for the β phase were detected. Typically, the XRD diffractograms of selectively laser melted $\alpha+\beta$ titanium alloys exhibit α' phase peaks only (C. Xu et al., 2020). This phenomenon can be ascribed to the small fraction of the β phase, 0.86 ± 0.054 , as estimated in this study using MATLAB image analysis tool. Stress-relief annealed specimen displayed α phase and the β phase under SEM analysis and the XRD analysis concurs as both α peaks and β peaks

are detected. Additionally, XRD results strongly agrees with the SEM results as it predicts α as the predominant phase with the diffraction peaks of α , for example α (101) plane, having the highest intensity. The β peak detected at plane (111) confirms that the stress-relief annealed samples are composed of the α and β phases thereby validating the SEM results.

In the case of full annealed specimens, SEM predicted the presence of α and β phases with α as the predominant phase. Similarly, the XRD results agreed with these findings as the diffraction peaks for α planes had relatively higher intensity. The α -phase amount was also higher in the fully annealed samples than the stress relieved specimens as depicted by the increased intensity of the α peaks in the fully annealed samples. This is due to the difference in crystallinity of the material a phenomenon known as the periodicity factor ([Cullity, 1956](#)). Additionally, β phase peak at plane (110) is detected in the XRD analysis suggesting the existence of the β phase in the full annealed specimens. It has to be emphasized that the relative intensity of the β phase's peak is strongly associated with the fraction of stabilized β phase during the heat treatment process ([Bolzoni & Yang, 2024](#)). The β peak intensity in full annealed specimen is higher compared with the stress-relief annealed specimens suggesting a higher volume fraction of the β phase in the full annealed samples than the stress-relief annealed specimens. This is in strong agreement with the SEM analysis results and the calculated β volume fraction results from SEM analysis summarized in **Table 4. 2**.

4.7 Conclusions.

The utilization of SLM Ti-6Al-7Nb alloy in acetabular implants demands excellent characterization techniques to determine microstructure-property relations for this alloy. Designing microstructures possessing excellent properties tailored to suit acetabular implant application requires a thorough understanding of the phase transformations taking place during processing. Our study employed SEM as well as XRD analysis to study the phase transformation and the associated microstructural development in SLM Ti-6Al-7Nb alloy. The SEM and XRD results in our study showed strong correlation and consistency.

The as-built microstructure is largely composed of the metastable acicular α' Ti phase. Though this phase contributes to improved strength, different studies have reported a reduction in ductility and toughness, which are critical for load bearing implants like the acetabular implant ([Chlebus et al., 2011](#)). Furthermore, this phase is associated with low corrosion resistance, Furthermore, this phase is associated with low corrosion resistance, which increases the susceptibility of the acetabular implant to failure due to corrosion failure

mechanism (Dai, Zhang, Zhang, Chen, et al., 2016; D. Zhao et al., 2019). Therefore, it can be predicted from this microstructural analysis that the as-built LPBF Ti-6Al-7Nb specimens are unsuitable for acetabular implant application.

The annealing heat treatment transformed the acicular α' phase and the columnar prior β grains in the as-built specimens to lamellar α and β phase. Excellent properties as per regard to implants application has been reported with the lamellar phase together with the increased content of the β phase in Ti-6Al-7Nb alloy (C. Xu et al., 2020). Therefore, from this microstructural analysis, the full annealed Ti-6Al-7Nb specimen possess favourable microstructure for acetabular implant application. However, further analysis in the following chapters will validate these findings.

Chapter 5: Compressive Mechanical behaviour and Tribological performance of Ti-6Al-7Nb Specimens.

5.1 Chapter Summary.

This chapter presents a comprehensive analysis of the compressive behaviour at various strain rates, microhardness, and wear performance of both as-built and heat-treated LPBF Ti-6Al-7Nb specimens. The chapter is aimed at assessing the compressive load-bearing capabilities as well as presenting the results of investigation on tribological behaviour of LPBF Ti-6Al-7Nb specimens against alumina ball at different loading conditions. **Section 5.2** reports the mechanical property evaluation of LPBF Ti-6Al-7Nb specimens measured from the compression data at different strain rates. Thereafter, analysis on these specimens' microhardness is presented in **section 5.3.1**. Preferable material properties for the LPBF Ti-6Al-7Nb include a material that presents strong wear resistance as this helps to mitigate implant failure and aseptic loosening, while ensuring the longevity of the implant. The wear behaviour is analysed through considering frictional and tribological behaviour of the as-built, and heat-treated LPBF material in **section 5.3.2**. Data from the tribology software as well as wear track analysis using the stereo microscope offered critical insights of the wear behaviour of as-built and heat-treated LPBF Ti-6AL-7Nb specimens (**section 5.3.3**) aiding in the selection of optimum material that reduces wear and enhance durability of acetabular implant. A comprehensive examination of the wear track morphology and wear mechanisms is provided in **section 5.3.4** while **section 5.3.5** discusses and analyses the observed tribological behaviour in relation to acetabular implant applications.

5.2 Compressive mechanical properties.

During different human activities like standing, walking, or running, the upper body's weight generates compressive stresses at the hip joint (H. Hu et al., 2021). Therefore, compressive strength plays a significant role in acetabular implant's longevity. **Figure 5. 1** displays compressive true stress-strain behaviour of as-built, stress-relief annealed, and full-annealed LPBF Ti-6Al-7Nb specimens evaluated at 0.015mm/s, 0.028 mm/sec, and 0.04 mm/sec strain rates. All the samples exhibit two different regions; linear elastic region, and linear plastic region, a typical compressive deformation behaviour of ductile metals (Soro et al., 2019).

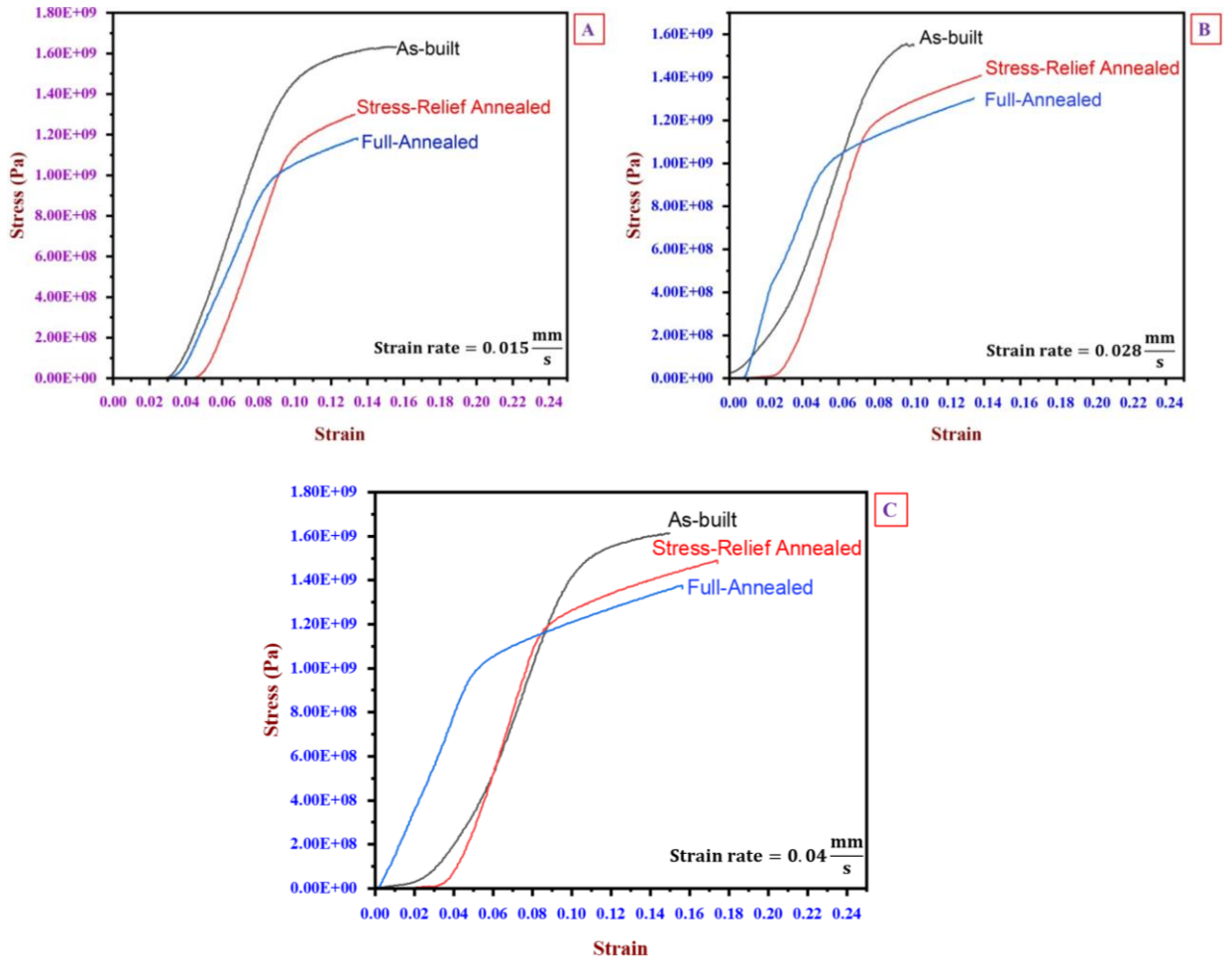


Figure 5. 1: Compressive true stress-strain curves for the as-built, stress-relief annealed, and full-annealed Ti-6Al-7Nb samples at (A) 0.015mm/sec, (B) 0.028 mm/sec, and (C) 0.04 mm/sec strain rates.

Table 5. 1 provides a summary of the as-built and heat-treated LPBF Ti-6Al-7Nb specimen's compression properties.

Table 5. 1: Comparison of the compression properties of as-built, stress-relief annealed, and full-annealed Ti-6Al-7Nb specimens relative to natural bone at strain rates of 0.015mm/s, 0.028mm/s, and 0.04mm/s. $0.2\sigma_y$ and σ_{UCS} denote 0.2% compressive yield strength and ultimate compressive strength of the material respectively.

Compressive Strength, MPa.	Cortical bone		Cancellous bone	Ref
	Longitudinal direction	Lateral direction	2-5	(Dzogbewu et al., 2018; Havaladar et al., 2014; Mirzaali et al., 2016)
	131-224	106-133		

Specimen Condition	Strain rate ($\frac{mm}{s}$)	$0.2\sigma_y(MPa)$	$\sigma_{UCS}(MPa)$
As-built	0.015	1230	1634
Stress-Relief Annealed		1090	1300
Full-Annealed		973	1183
As-built	0.028	1180	1557
Stress-Relief Annealed		1144	1409
Full-Annealed		977	1270
As-built	0.04	1397	1614
Stress-Relief Annealed		1186	1490
Full-Annealed		1000	1377

Despite the decrease in compressive yield strength with annealing treatment, the measured compressive yield strengths are still higher than the typical compressive yield strength of the natural bone (25 MPa to 224 MPa) ([Havaladar et al., 2014](#); [Mirzaali et al., 2016](#)). High compressive yield strength is crucial for acetabular implants since they endure continuous mechanical loading from activities like walking, running, and sitting. ([Attar et al., 2014](#)). However, excessively high yield and compressive strength can cause stress shielding when an acetabular implant bears excessive load, causing bone resorption and weakening over time. Therefore, this reduction in compressive yield strength through full annealing heat treatment would be beneficial in reducing stress shielding in acetabular implants ([Y. Li et al., 2014](#)).

Another observation is that both the compressive yield strength and the ultimate compressive strength of LPBF Ti-6Al-7Nb specimens, whether as-built or heat-treated, increase with an increase in strain rate. This behaviour is referred to as strain hardening and is related to the rate of dislocation generation and dislocation pile up in the material. Dislocations diffuse through the crystal structure from inside grains to the grain boundaries where they can be accommodated, thus providing a softening of the material. At higher strain rates, deformation occurs rapidly, causing dislocation generation to outpace their diffusion to grain boundaries ([Pan et al., 2021](#)). This leads to a higher dislocation density, which restricts further movement of dislocations, thereby increasing compressive yield strength ([H. Liu et al., 2023](#)). This behavior is crucial in acetabular implants as it improves the implant's fatigue strength and its ability to resist fretting damage. For instance, currently hip implant designers and manufactures adopt a surface-enhancement technique known as low plasticity burnishing (LPB) that employs cold work hardening to improve the implant's thermal and biomechanical stability ([Hornbach et al., 2006](#); [Summers et al., 2019](#)). An AcuMatch M-Series implant (Exactech, Inc., Gainesville) adopted this cold work hardening technique and experienced a 10-fold increase of the implant's lifespan ([Hornbach et al., 2006](#)). In the current work, the cold work hardening behavior experienced in as-built and the heat-treated LPBF Ti-6Al-7Nb specimens is essential for improving acetabular implant lifespan without the adoption of this surface treatment. Further studies can be conducted to explore to what extent the lifespan of the acetabular implant can be improved with the adoption of surface treatments like LPB on this SLM Ti-6Al-7Nb alloy that's already displaying important cold work hardening properties.

The low strain rate loading condition of 0.015 mm/s produced a comparatively reduced compressive yield strength in as-built as well as heat treated LPBF Ti-6Al-7Nb specimens. Increased strain rates of 0.028 mm/sec and 0.04 mm/sec produced higher compressive yield strengths that increases the biomechanical properties discrepancy between the acetabular prosthesis and the adjacent bone. More importantly, within the 0.015 mm/s strain rate loading condition the fully annealed LPBF Ti-6Al-7Nb specimens produced the lowest compressive yield strength of 973 MPa. Therefore, the fully annealed LPBF Ti-6Al-7Nb specimens at 0.015 mm/s strain rate can improve the biomechanical compressive properties of the acetabular implant and improve its service life.

5.3 Tribological behaviour.

5.3.1 Microhardness Analysis.

Figure 5. 2 presents the microhardness profiles and the mean microhardness values for the as-built, stress-relief annealed, and fully annealed specimens. The as-built LPBF Ti-6Al-7Nb sample demonstrates a maximum microhardness of $461 \pm 20 \text{ HV}_{0.5}$. The stress-relief annealing process reduces the microhardness by 10.6%, bringing it down to $412 \pm 16 \text{ HV}_{0.5}$. The full-annealed samples display the lowest microhardness value of $370 \pm 5 \text{ HV}_{0.5}$, a significant 19.7% reduction in microhardness from the as-built sample. This indicates that heat treatment significantly impacts the microhardness of LPBF fabricated Ti-6Al-7Nb specimens. These findings demonstrate that increasing annealing temperature reduces the microhardness of LPBF Ti-6Al-7Nb parts. Yuantao et al. (Y. Zhao et al., 2024) documented a similar microhardness reduction in Ti-6Al-4V when annealing temperature increased. The microhardness of titanium alloys is largely dictated by their microstructural phases and grain size. The order of hardness of the phases in titanium alloys is summarized as $\alpha' > \alpha > \beta$ (Han et al., 2015; L.-b. Zhang et al., 2015). The microstructure of as-built Ti-6Al-7Nb specimens is predominantly composed of acicular α' martensite, a hard supersaturated phase with a high dislocation density (Y. Zhao et al., 2024). This improves the as-built Ti-6Al-7Nb specimen's resistance to localized plastic deformation leading to the highest attained microhardness values in this study. During annealing, the hard α' phase transforms to softer α and β phases, resulting in a gradual reduction in hardness of LPBF Ti-6Al-7Nb parts (Galarraga et al., 2017).

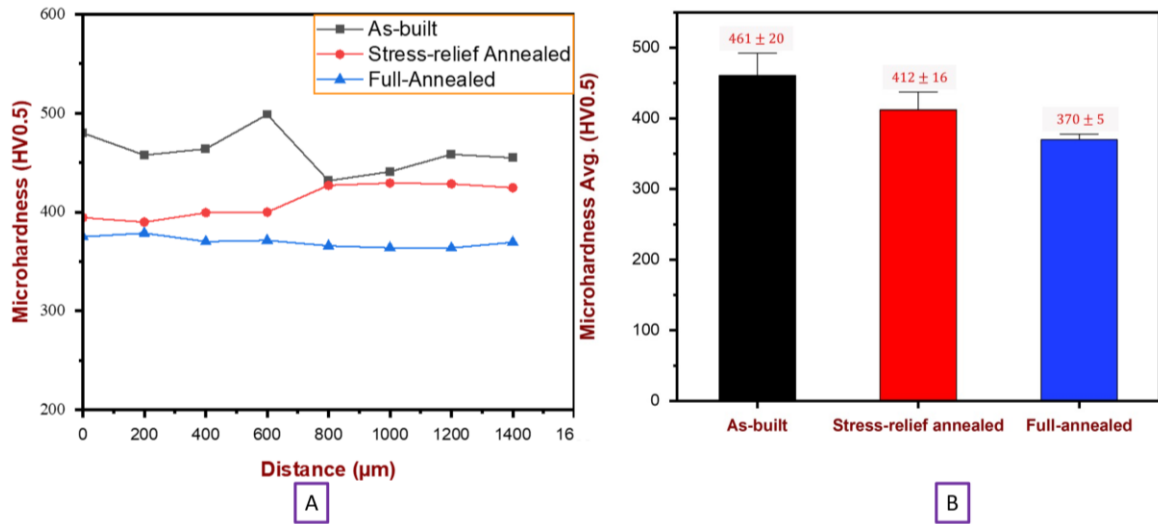


Figure 5. 2: (A) The microhardness profiles of the as-built and heat-treated LPBF Ti-6Al-7Nb specimens along the build direction. (B) Average microhardness values for the as-built and heat-treated specimens.

The stress-relief annealing treatment retained α' phase due to incomplete transformation into α and β phases. This explains its relatively higher microhardness than the full-annealed specimen. At 850°C, the complete transformation of α' phase into α and β phases results in the formation of a lamellar microstructure. The transformation increased the fraction of the softer β phase, reducing the alloy's resistance to plastic deformation and further lowering its hardness.

Acetabulum hardness varies from person to person, and the posterior column is harder than the anterior column. Research has indicated that the overall hardness of the anterior column of the acetabulum varies between 18.9 and 33.8 HV, while the hardness of the posterior column of the acetabulum ranges from 23.1 to 39.1 HV (刘国彬 & guobin Liu, 2020).

Acetabular implants having higher hardness absorb more load reducing stress on the surrounding bone. This leads to bone resorption (loss of bone density) over time, weakening the reamed surrounding bone of the acetabulum (A. Kumar et al., 2022). Additionally, the surrounding softer acetabulum bone may experience accelerated wear due to the harder acetabular implant material ploughing into the bony tissues, leading to debris generation that can cause inflammation and osteolysis (Bezwada et al., 2025). Therefore, to mitigate these risks and guarantee that the acetabular implant works well with the softer bone of the acetabulum surrounding it, the fully annealed LPBF Ti-6Al-7Nb specimen (370 ± 5 HV_{0.5}) presents itself as the best option due to its lowest microhardness than the LPBF as-built

($461 \pm 20 \text{ HV}_{0.5}$) as well as the stress-relief annealed LPBF Ti-6Al-7Nb ($412 \pm 16 \text{ HV}_{0.5}$) specimens.

The degree of homogeneity in LPBF Ti-6Al-7Nb specimens' microstructure also significantly impacted its microhardness behavior. From SEM analysis of the microstructure in **section 4.4**, the fully annealed Ti-6Al-7Nb microstructure consisted of a uniform/homogeneous lamellar structure contrary to the heterogenous microstructure in the stress-relief annealed and as-built LPBF specimens. The homogeneous microstructure of the fully annealed LPBF Ti-6Al-7Nb specimens led to consistent hardness properties, almost a flat microhardness curve in **Figure 5. 2A**, enhancing its reliability. In contrast, the inhomogeneous microstructures in the LPBF as-built as well as stress relief annealed LPBF Ti-6Al-7Nb specimens resulted in fluctuations in the microhardness properties an indication of localized weaknesses where some areas are harder while others are prone to deformation (refer to **Figure 5. 2A**). This signifies the capabilities of the full annealing treatment in optimizing homogeneity to achieve uniform phase distribution and elemental composition to improve consistency in mechanical performance of the acetabular implant material.

5.3.2 Coefficient of friction (COF) Analysis.

The COF for each sample at a specified load were analysed. **Figure 5. 3A-C** shows the COF curves for the As-built, Stress-relief annealed, and full annealed Ti-6Al-7Nb specimens under applied loads of 10, 15, and 20 N versus sliding time. Additionally, **Figure 5. 3D** depicts the average COFs for these samples at 10, 15, and 20 N loads. The COF at each load increases with heat treatment temperature increase. In summary, the COF curves and the average COF bar chart indicate that at all loads the $\text{COF}_{(\text{Full-Annealed})} > \text{COF}_{(\text{Stress-Relieved})} > \text{COF}_{(\text{As-Built})}$.

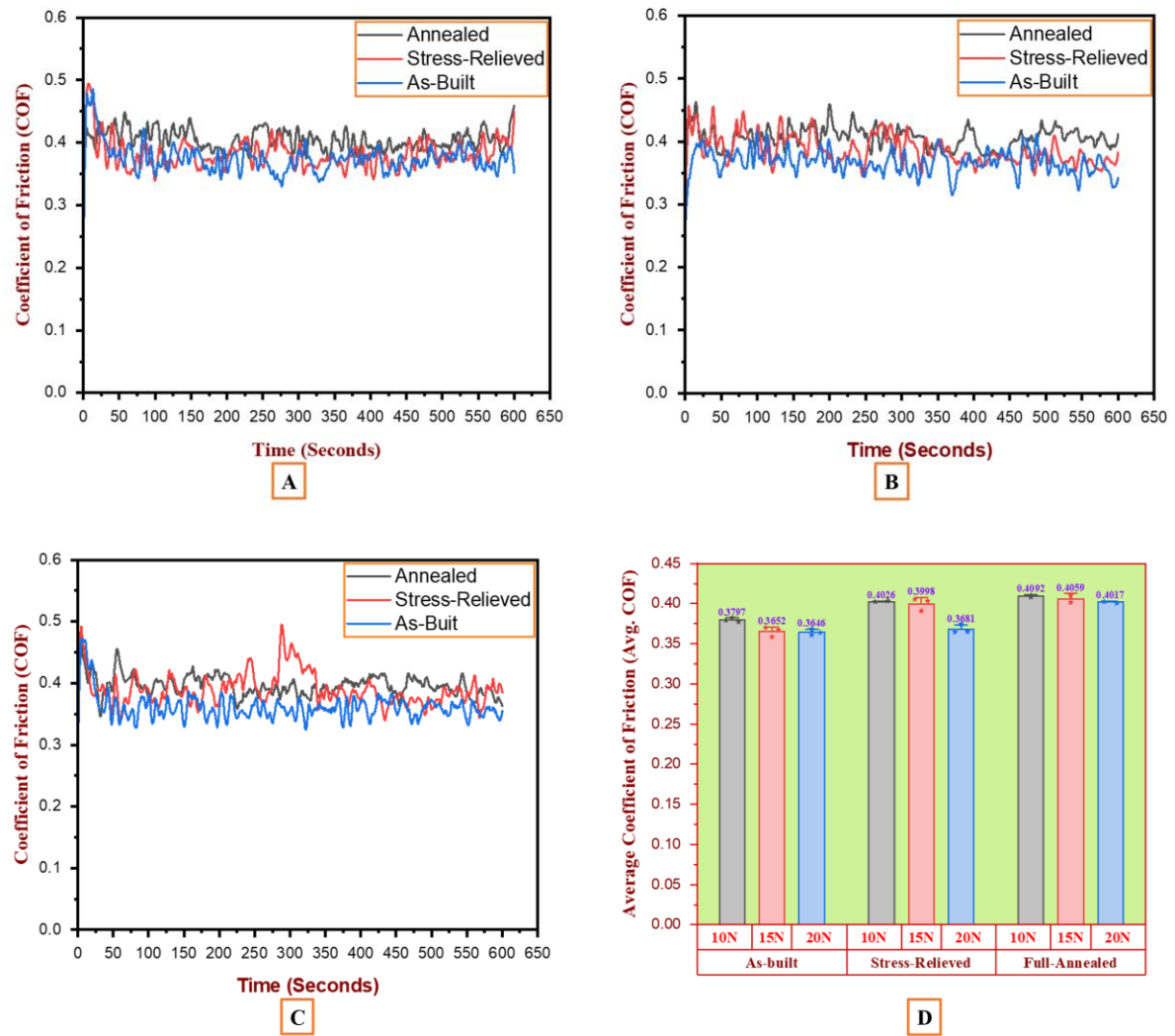


Figure 5. 3: Coefficient of Friction curves for LPBF Ti-6Al-7Nb Alloy at different loading conditions. (A) 10 N. (B) 15 N. (C) 20 N. (D) A bar chart displaying the average COF for As-built, Stress-Relief Annealed and Full Annealed Ti-6Al-7Nb at 10, 15, and 20 N loads.

All the COF curves illustrated in **Figure 5. 3** displayed two wear stages, the initial (running-in) phase and the subsequent stable wear phase. The initial (running-in) phase (approximately 0-25 seconds) is characterised by fluctuations in the COF values. Due to the passive behaviour of titanium alloys, some researchers have attributed these fluctuations in the initial state to the breakdown and removal of the oxide layer that naturally forms on the alloy's surface (Ju et al., 2021). COF values steadily stabilized after sliding for approximately 25 seconds, suggesting that the Al_2O_3 ball and LPBF Ti-6Al-7Nb matrix had reached a stable wear state.

The applied normal load had a significant impact on LPBF Ti-6Al-7Nb's COF. The COF of each sample (as-built, stress-relieved, and fully annealed) decreased as the applied load increased. For instance, the stress-relief annealed Ti-6Al-7Nb specimen had a COF of 0.4026 at 10 N load which decreased to 0.3998 and 0.3681 at 15, and 20 N loads respectively. Previous studies on titanium alloys have also documented a similar pattern ([Ju et al., 2021](#); [Su et al., 2022](#); [W. Yang et al., 2020](#)). This is due to the high frictional heat produced by the high applied load, which softens the metal matrix and facilitates plastic deformation ([Su et al., 2022](#)). This strongly agrees with the research findings of Liu and Sun ([C. Liu & Sun, 2018](#)) on alumina ceramics which concluded that an increase in applied load resulted in high shearing stress at the contact region thereby significantly increasing the temperature of the contact area, facilitating the specimen's plastic deformation. The COF therefore reduced with increasing applied load. Moreover, observed COF reduction at higher loads could be a result of enhanced tribo-oxide layer development on the wear surface, which contributes to friction reduction (as confirmed by EDS analysis in Table 5. 2). The compacted moveable oxide particles at a higher load will provide a rolling effect (playing the role of a lubricant) which transforms part of the sliding friction into rolling friction thereby significantly reducing COF ([X. X. Li et al., 2015](#)). Though the COF differences between as-built, stress-relief annealed, and fully annealed specimens are minimal and should not be overinterpreted, the heat treatment procedure still impacted the COF of LPBF Ti-6Al-7Nb parts where the as-built sample exhibited a lower COF which increased with stress-relief annealing while the full-annealed specimen had a highest COF. This can be linked to the hardness of these LPBF Ti-6Al-7Nb specimens. As observed in **section 5.3.1**, the as-built sample had a higher microhardness which decreased after stress-relief annealing and full-annealing heat treatment. In sliding wear tests, increased hardness can lead to a smoother worn surface with reduced surface roughness, consequently lowering the coefficient of friction (COF). ([Figueiredo et al., 2011](#)) (refer to wear track morphologies in **Figure 5. 6**).

The near-zero COF between 0.002 - 0.01 ([Hodge et al., 1986](#)), provides optimal tribological properties for the articular cartilage of the hip joint, ensuring excellent articulation and load transmission on the joint. However, implementing such near-zero COF in acetabular implants increases implant micromotion, compromises implant stability, leading to issues like migration or loosening of the implant necessitating revision replacements ([Callary et al., 2021](#)). Therefore, in clinical practice an acetabular implant with high COF is preferred

(C. Markel, 2002; Shirazi-Adl et al., 1993). According to existing literature, a friction coefficient between 0.4 and 0.9 at the prosthetic acetabular cup-pelvis interface ensures adequate initial stability for the acetabular implant (Hsu et al., 2007; Y. Zhang et al., 1999). Some studies have even reported good mechanical stability and excellent biological fixation through osseointegration in 3-D printed acetabular implants with a surface COF up to 1.2 (Peterson et al., 2019). The fully annealed LPBF Ti-6Al-7Nb specimen in this study, which has a relatively high average ≈ 0.4 (refer to **Figure 5. 3D**) is a promising candidate for acetabular implants, offering enhanced mechanical stability and better biological fixation than as-built and stress-relief annealed LPBF Ti-6Al-7Nb specimens.

5.3.3 Wear Track Width, Depth, Wear Volume, and Wear Rate Analysis.

Measuring wear track width and depth is a simple yet effective technique for evaluating the LPBF Ti-6Al-7Nb parts' wear resistance (Z. Yang et al., 2013). The bar charts in **Figure 5. 4 (A and B)** indicate that for all LPBF Ti-6Al-7Nb specimens (as-built, stress-relief annealed, and full-annealed) increasing the applied load results in notable increase of both the width and depth of the wear track. For instance, at applied load of 10 N, the as-built sample had a mean wear track width and depth of 426.09 μm and 0.0217 mm respectively. Increasing the applied load to 15 N widened the wear track to 497.1 μm and deepened it to 0.0247 mm. This very same pattern was also observed in stress-relief annealed and full-annealed specimens.

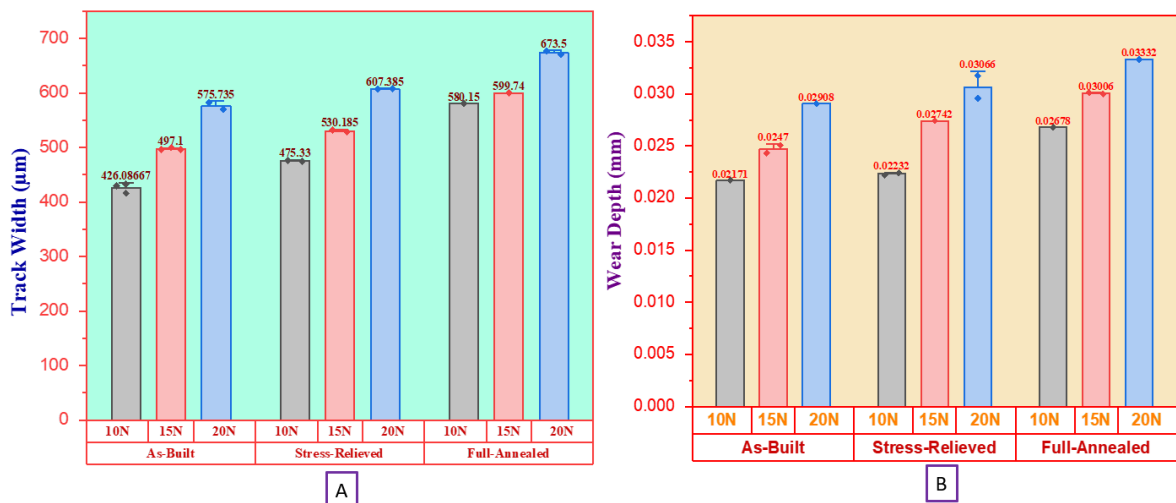


Figure 5. 4: Bar charts showing the effects of heat treatment and applied load on (A) wear track width and (B) wear depth of Ti-6Al-7Nb specimens in as-built, stress-relief annealed, and fully annealed states under loads of 10, 15, and 20 N.

The wear track width and depth also showed how heat treatment affected the LPBF Ti-6Al-7Nb specimens' wear behaviour. The stress-relief annealed specimen wear tracks become deeper and wider, at an applied load of 10 N, the mean wear track width and depth were recorded as 475.33 μm and 0.0223 mm, respectively, compared to the track width of 426.09 μm and wear depth of 0.0217 mm in the as-built specimen under the same load. The fully annealed specimen's wear track width and depth further increased to 580.15 μm and 0.0268 mm, respectively. Jiang et al. (Ju et al., 2021), documented similar tribological behavior in LPBF Ti-6Al-4V parts.

At all applied loads, the as-built LPBF Ti-6Al-7Nb specimens exhibited have narrower and shallower wear tracks than stress-relieved specimens and full-annealed specimens where the maximum wear track width and depth were observed (see **Figure 5. 4**). As wear progresses, that is the Al_2O_3 ceramic ball penetrates the Ti-6Al-7Nb specimen, the wear track width and depth increase. Thus, the as-built Ti-6Al-7Nb exhibited superior wear resistance compared to stress-relief annealed and full-annealed specimens across all applied loads.

Figure 5. 5A and B) display the wear performance of as-built and the heat-treated LPBF Ti-6Al-7Nb alloy as a function of wear volume and wear rate at 10, 15, and 20 N loads. The fully annealed specimen exhibits the highest wear volume loss and wear rate, followed by the stress-relieved specimen, while the as-built specimen demonstrates the lowest values across all applied loads. These results are consistent with the attained Vicker's microhardness (refer **Figure 5. 2**), where the as-built samples have higher microhardness than the annealed LPBF Ti-6Al-7Nb specimens.

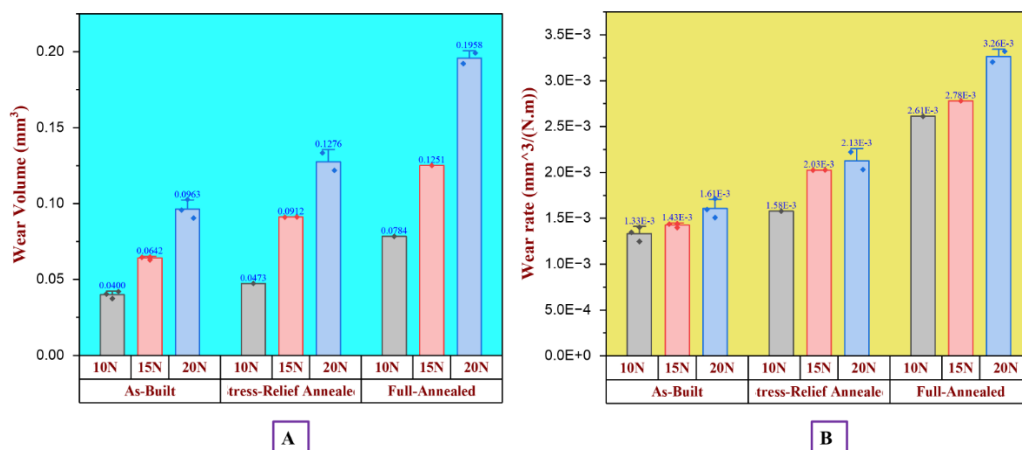


Figure 5. 5: (A) Wear volume and (B) Wear rate of the as-built and heat-treated LPBF Ti-6Al-7Nb specimen at applied loads of 10, 15, and 20 N.

There isn't a universally standardized rate for acetabular implant wear, as wear rates can vary based on factors like the materials used, patient activity levels, and implant design (Bezwada et al., 2025). This study utilized high loading conditions (contact stresses ≥ 950 MPa), to cater for issues of edge wear in acetabular implants. Edge-wear in acetabular implants is associated with higher volumes of material loss (Bergiers et al., 2022). Different acetabular implant retrieval studies have reported varying wear rates. For instance, an average volumetric wear of 49.63 mm^3 was reported for twenty acetabular implants that were retrieved after 90 months in the study of Bergiers et al. (Bergiers et al., 2022). Similarly, twenty-two retrieved acetabular cups by Lord et al. (Lord et al., 2011) exhibited 29.00 mm^3 mean volumetric wear (range of $1.35 - 109.72 \text{ mm}^3$). However, other in vivo radiographic data have reported lower wear rates between $0.055 - 0.3 \text{ mm}$ per year (Bragdon et al., 2002). The mean wear volumes in this study had a maximum of 0.1958 mm^3 confirming the suitability of all the as-built, stress-relief annealed, and fully annealed LPBF Ti-6Al-7Nb specimens designed for use in acetabular implant applications. However, for a more detailed quantitative comparative analysis of the wear rates per year or a million cycles, hip joint simulator studies have to be carried out on the as-built, stress-relief annealed, and fully annealed LPBF Ti-6Al-7Nb samples.

5.3.4 Worn surface morphology and associated wear mechanisms.

5.3.4.1 *The impact of heat treatment on the wear mechanism and worn surface morphology.*

SEM technique was utilized to analyse the impact of annealing treatment on the wear mechanisms of LPBF Ti-6Al-7Nb specimen. **Figure 5. 6** displays high-magnification morphologies of the wear tracks, with elemental composition analysis of the marked spots through EDS summarized in **Table 5. 2**. Transitions in the wear mechanisms were clearly observed in the as-built, stress-relief annealed and full-annealed Ti-6Al-7Nb samples at the same applied load. From **Figure 5. 6A**, the presence of uniform, relatively narrow parallel grooves and resulting debris signify abrasion as the dominant wear mechanism in the as-built samples. The development of flake-like wear points in the wear track is an indication of delamination wear. The layers of the as-built sample are separated or peeled away from the surface, an indication of adhesive wear mechanism. The EDS results in **Table 5. 2**, for spot 1 indicate that the wear debris is rich in O, indicating the occurrence of oxidative wear. In summary, the predominant wear mechanism observed in the as-built sample is abrasive wear with slight adhesive wear as well as oxidative wear mechanisms.

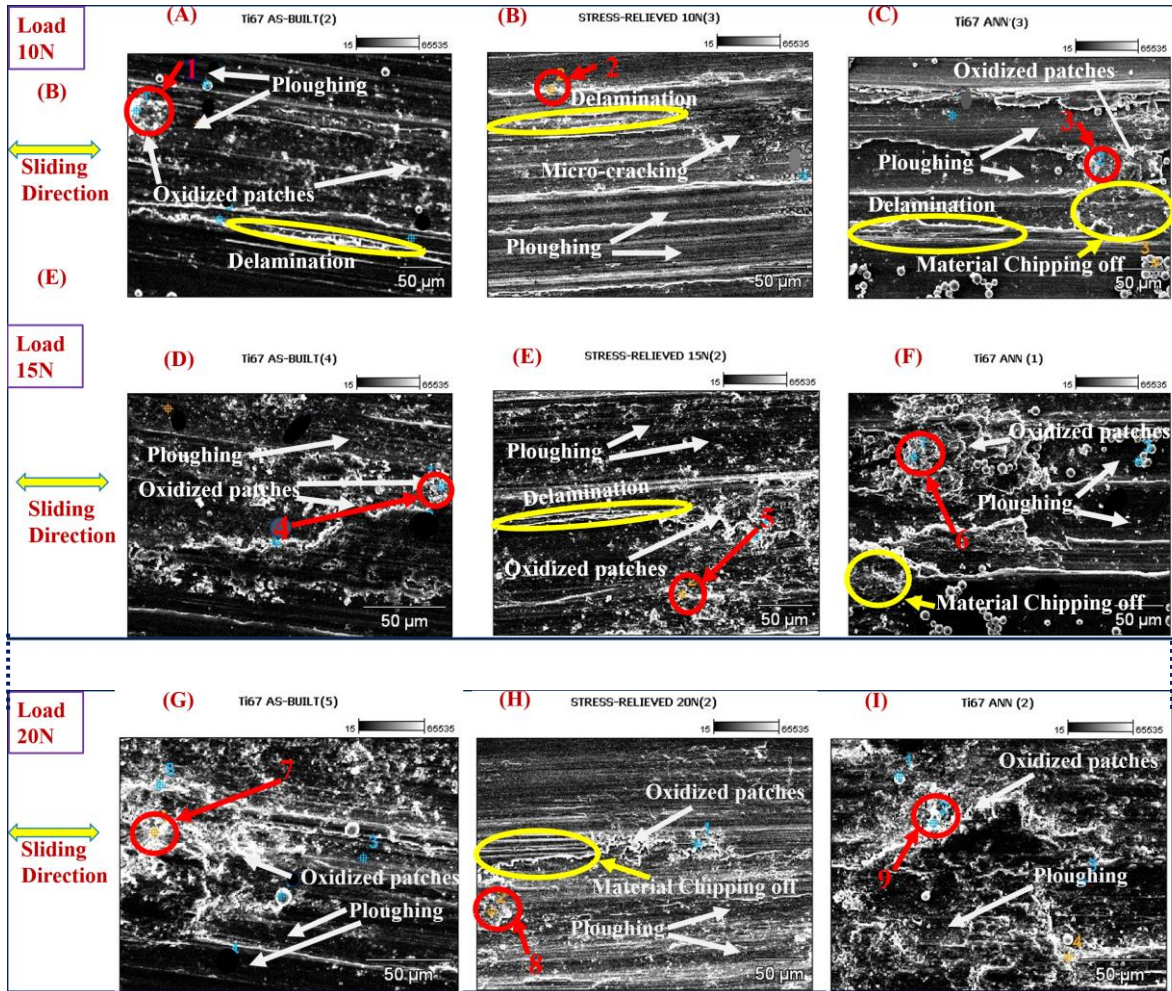


Figure 5. 6: SEM wear track morphologies of as-built (A, D, G), Stress-relief Annealed (B, E, H) and full-annealed Ti-6Al-7Nb (C, F, I) specimens at applied loads of 10, 15, and 20 N.

Table 5. 2: Elemental composition results from the EDS Analysis of numbered spots in Figure 5. 6.

APPLIED LOAD (N)	SAMPLE CONDITION	SPOT IN Figure 5. 6	O	Al	Ti	Nb
10	As-built.	1	9.54	5.04	82.55	2.87
	Stress-Relieved	2	14.98	5.98	73.27	5.77
	Full-Annealed	3	11.79	5.18	76.47	6.56
15	As-built.	4	16.85	3.89	74.29	4.97
	Stress-Relieved	5	9.75	4.36	80.97	4.91
	Full-Annealed	6	8.31	4.17	81.62	5.89
20	As-built.	7	22.64	10.15	62.28	4.93
	Stress-Relieved	8	9.12	5.19	80.27	5.42
	Full-Annealed	9	31.42	13.89	51.40	3.29

Compared to the as-built samples, the ploughing grooves are wider in the stress-relief annealed specimens, attributed to the relatively low hardness which makes it easier for the Al₂O₃ ball to penetrate the material during the sliding process. Furthermore, relative to the as-built sample, the stress-relief annealed specimens exhibit larger delaminated regions. According to Ashby et al. (Kong & Ashby, 1992), during reciprocating sliding, delamination cracks are initiated due to the repetitive work-hardening, which could potentially lead to spalling. Stress-relieved Ti-6Al-7Nb alloy is more susceptible to delamination since it is softer and has less dislocations that can multiply and interact more readily during deformation as a result have more potential to strain harden more quickly than the harder as-built Ti-6Al-7Nb specimens that have higher dislocation density which reduces their ability to accommodate any further deformation (Qayyum et al., 2024). Delamination during strain hardening occurs due to the accumulation and interaction of dislocations near grain boundaries or phase interfaces. As strain hardening progresses, localized plastic deformation and cyclic stresses lead to initiation of microcracks which grow and coalesce as

delamination cracks. Softer, more deformable microstructures like that of the stress-relief annealed Ti-6Al-7Nb specimens experience accelerated dislocation multiplication and interactions that promote this damage mechanism (Fu et al., 2024). The repetitive work hardening under sliding loading thus facilitates delamination, causing surface spalling or failure observed in **Figure 5. 6**.

The elemental composition analysis in the stress-relief annealed sample at spot 2 revealed that the wear debris is rich in O (see **Table 5. 2**) a clear indication of oxidative wear. Therefore, the dominant wear mechanisms in stress-relief annealed samples are abrasion wear and adhesive wear followed by a slight oxidative wear.

The full-annealed LPBF Ti-6Al-7Nb specimen exhibited deeper and wider grooves in its wear track morphology, compared to stress-relief annealed and as-built samples. The low hardness of the full-annealed LPBF Ti-6Al-7Nb specimen causes deeper and wider wear grooves than those in as-built and stress-relieved samples. This indicates that abrasion wear mechanism is more dominant in the full-annealed specimen than in all the tested samples. Adhesion wear was also present as evidenced by the delaminated regions on the wear track. Furthermore, a lot of oxidized patches can also be observed (high O content present at point 3, refer to **Table 5. 2**) a definite sign of increased oxidative wear in comparison to the LPBF Ti-6Al-7Nb as-built and stress-relief annealed specimens.

5.3.4.2 The impact of applied load on the wear mechanism and worn surface morphology.

SEM was utilized to evaluate the worn surface morphology of as-built, stress-relief annealed, and fully annealed Ti-6Al-7Nb alloy specimens under varying applied loads, as illustrated in **Figure 5. 6**. The as-built sample's wear track is relatively flatter than those of the stress-relief annealed and full-annealed specimens under the applied load of 10 N, as observed in **Figure 5. 6A, B, and C**. This explains the low COF encountered in the as-built specimen than the other samples as shown in **Figure 5. 3A**. The stress-relieved and full-annealed specimen exhibit wider and deeper ploughs than the as-built specimen, suggesting low resistance to abrasive wear mechanism by the heat-treated samples. More delaminated regions as well as inhomogeneous distributed oxide patches are found in the stress-relieved and full-annealed samples.

Increasing the applied load to 15 N, widened the ploughing grooves on the wear tracks of all samples as displayed in **Figure 5. 6D, E, and F**. Moreover, the white oxidized patches

with increased O content as evidenced by EDS analysis in spots 4, 5, and 6 (refer **Figure 5. 6**) are further increasing, confirming that oxidative wear is further aggravated with increased load in all samples. This similar observation can be seen with a further increase in load to 20 N in **Figure 5. 6G, H, and I** (refer spots 7,8, and 9 in **Table 5. 2**). This wear mechanism transition was explained by Gain et al. ([Gain et al., 2021](#)) as a result of the softening of the matrix from increased frictional heat under high applied loads which exacerbated oxidative wear.

5.3.5 Implications of the observed wear mechanisms for practical design of Ti-6Al-7Nb alloy using LPBF, stress-relief and full annealing processes in acetabular implant.

In this study, three different wear mechanisms have been observed with respect to the annealing heat treatment procedure and the applied loads. These wear mechanisms are generally; adhesive, abrasive, and oxidative wear mechanisms. From the wear track morphologies in **Figure 5. 6**, adhesive and abrasive wear mechanisms are associated with significant material loss that greatly impacts performance and durability of the acetabular implant. Therefore, these mechanisms result in the release of large amount of debris, which triggers inflammatory responses in the body and compromise the acetabular implant performance ([Sagbas, 2016](#)). Despite wear being typically considered a drawback, some studies have reported intriguing observations regarding oxidative wear mechanism. As observed in **Figure 5. 6**, oxidative wear mechanism leads to the development of tribofilms which serve as a protective layer between the implant surfaces ([Hesketh et al., 2014](#)). This results in improved rolling and articulation of the implant under demanding conditions of the human body.

Consolidating the findings in wear mechanisms at different applied loads and annealing treatment processes, it is clear that full annealing of LPBF Ti-6Al-7Nb enhances its surface properties, that can contribute to improved performance and longer service life for acetabular implants. In contrast to the stress-relieved and the as-built specimens, the fully annealed LPBF Ti-6Al-7Nb specimens' wear surfaces had a lot of oxidized patches or tribofilms. These tribofilms serve as a solid lubricant that enhance the articulation and longevity of acetabular implant while preventing further metal ion release into the body system.

5.4 Conclusions.

This section systematically evaluated the compressive properties as well as sliding wear characteristics of as-built and heat-treated LPBF Ti-6Al-7Nb specimens, tested against Al₂O₃ balls, providing insights into their tribological performance. The compressive properties were evaluated across a strain rate range (0.015 mm/sec, 0.028 mm/sec, and 0.04 mm/sec) similar to the strains experienced at the hip joint. For acetabular prosthesis, it is critical to harmonize the compressive yield strength of the implant and that of the surrounding bone to avoid bone weakening from stress shielding effect. Unfortunately, the bulk metallic biomaterials' compressive yield strengths are very high, and the current trend is to introduce porosity to reduce the compressive yield strength as well as the elastic modulus. However, this work has established that a significant reduction in compressive yield strength (from 1230 MPa in the as-built to 973 MPa in the fully annealed sample) is achievable with full annealing treatment. Hence full annealing is recommended to have a biomechanical match of the implant's compressive properties and the surrounding bone.

Another important finding from this chapter was that the full annealing process resulted in a low microhardness and uniform microhardness curve than the as-built and stress-relief annealed samples. This is ascribed to the increased content of the softer β -phase and homogeneous microstructure in the full annealed specimen which reduces ploughing on the surrounding bone and assures mechanical integrity of the specimens than the as-built and stress-relief annealed specimens. Tribological performance analysis was studied by varying the applied load (10, 15, and 20 N) on the as-built and heat-treated specimens. The high COF and the explored wear mechanisms drove towards the conclusion that the full annealed LPBF Ti-6Al-7Nb specimens display superior tribological performance as with regard to acetabular implant application.

Chapter 6: Corrosion Resistance and In-vitro biocompatibility Analysis in Body Simulated Fluid, Dulbecco's Phosphate Buffered Saline Solution (DPBSS).

6.1 Chapter Summary.

The purpose of this chapter is to present the study's findings on the corrosion resistance and apatite formation capabilities of LPBF Ti-6Al-7Nb specimens. It explores the impact of microstructural design alterations through annealing treatment on the passivated oxide layer, which is critical for enhancing corrosion resistance and biocompatibility in titanium alloys for acetabular prosthesis applications. The chapter begins with an analysis of the corrosion rates for as-built, stress-relief annealed, and fully annealed Ti-6Al-7Nb specimens in simulated body fluids (section 6.2). This is followed by an examination of the surface morphology and elemental composition of the passivated oxide layer (section 6.3). Further analysis is presented in Section 6.4 and section 6.5, where the influence of microstructural phases and morphological characteristics (shape and size) on bio-corrosion resistance and apatite formation ability is thoroughly evaluated. The chapter concludes in section 6.6 by recommending the best LPBF Ti-6Al-7Nb condition for acetabular implant application considering the bio-corrosion resistance and potential for osseointegration through the apatite layer formation in simulated body fluids.

6.2 Corrosion Resistance Characterization.

Figure 6. 1 displays the corrosion rates of as-built and heat-treated LPBF Ti-6Al-7Nb specimens at various exposure times of 1 day (24 hours), 3 days (72 hours), 5 days (120 hours), 7 days (168 hours), 10 days (240 hours), and 14 days (336 hours) expressed in mm/yr. It can be observed that all the samples have the same corrosion behaviour but yield different corrosion rates.

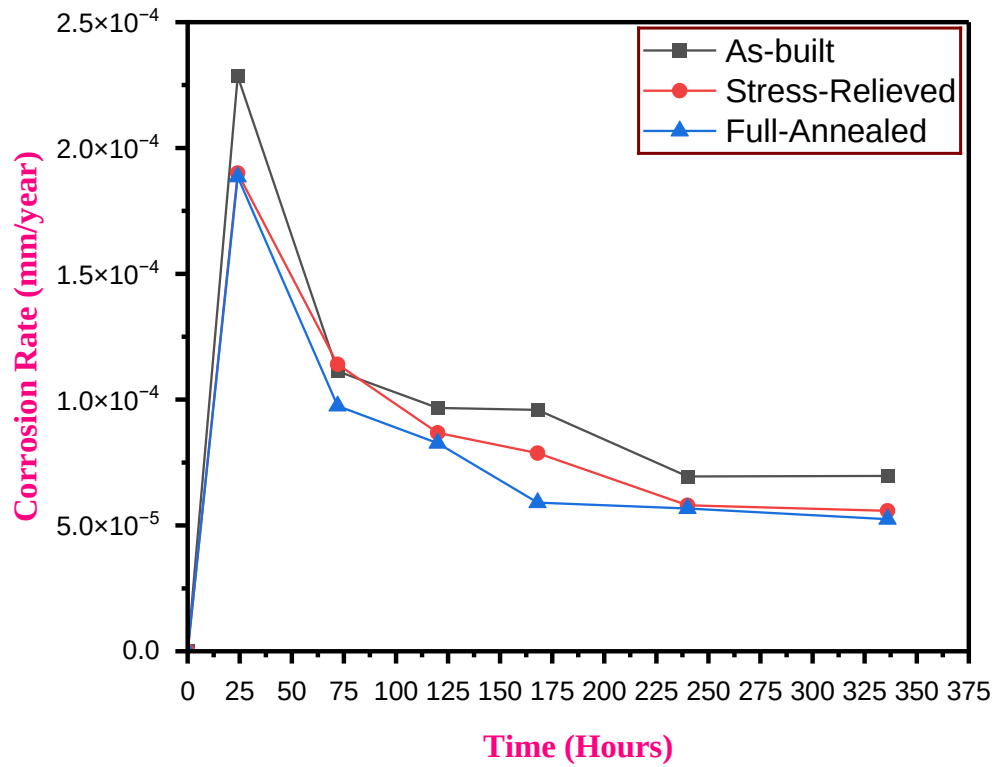


Figure 6. 1: Corrosion rates of as-built, stress-relief annealed, and full-annealed specimen at different immersion durations in phosphate buffered saline solution.

All the Ti-6Al-7Nb specimen experienced the peak corrosion rate at 24 hours immersion time followed by a decline as immersion time extended up to 336 hours. This initial rise in corrosion rate from 0 to 24 hours was attributed to the exposure of fresh corrosion sites, while the subsequent reduction was linked to the lack of newly exposed active sites. The as-built LPBF Ti-6Al-7Nb specimens exhibited a peak corrosion rate of $2.285 \times 10^{-4} \frac{mm}{yr}$ while the stress-relief annealed and full-annealed specimens had peak corrosion rates of $1.900 \times 10^{-4} \frac{mm}{yr}$ and $1.885 \times 10^{-4} \frac{mm}{yr}$ respectively. Importantly, these corrosion rates are significantly below the $9 \times 10^{-4} \frac{mm}{yr}$ corrosion rate of additively manufactured Ti-6Al-4V specimens in similar DPBSS obtained from the study of Yashwanth et al. (Balasubramanian Gayathri et al., 2022). This comparison indicates that LPBF-fabricated Ti-6Al-7Nb components possess superior corrosion resistance than their Ti-6Al-4V counterparts.

According to the biomedical standard resistance class, see Table 6. 1, the as-built and the heat-treated LPBF Ti-6Al-7Nb specimens exhibit perfect stability in phosphate-buffered saline solution. Additionally, it is widely acceptable that a metallic implant system's corrosion rate should be less than $2.5 \times 10^{-4} \frac{mm}{year}$ or $0.01 \frac{mils}{year}$ (Manivasagam et al., 2010).

This implies that the LPBF Ti-6Al-7Nb specimens conforms to the biomedical standard in terms of corrosion resistance for acetabular implants application. It is evident that heat-treated LPBF Ti-6Al-7Nb samples exhibit enhanced corrosion resistance, with full-annealed samples showing the lowest corrosion rate, while as-built samples display the highest corrosion rate. This suggests that the LPBF fabricated Ti-6Al-7Nb sample's corrosion resistance is strongly influenced by the adopted heat treatment procedure.

Table 6. 1: The biomedical resistance classification standard (J. Yang et al., 2017).

Resistance Classes	Range of corrosion rates (mm/year)
Perfectly stable	$< 10^{-3}$
Very stable	$10^{-3} - 10^{-2}$
Stable	$10^{-2} - 10^{-1}$
Relatively stable	$10^{-1} - 1$
Less stable	$1 - 10$
Unstable	> 10

6.3 Surface morphology, elemental composition, and in vitro apatite-forming capability analysis.

The prerequisite for an implant, tissue compatibility, is essentially dictated by the reactions that occur between body fluids and the surface of the acetabular implant in the early post-implantation period. The presence of a passivated oxide layer plays a critical role in enhancing corrosion resistance while ensuring tissue compatibility, thereby contributing to implant stability and biocompatibility. A high resolution JEOL JSM-6610 SEM was utilized to assess the post-corrosion surface properties of both as-built and heat-treated LPBF Ti-6Al-7Nb samples. Figure 6. 2 displays the typical surface morphology of the specimens after 24 hours (A, B, and C) and 72 hours (D, E, and F) of immersion.

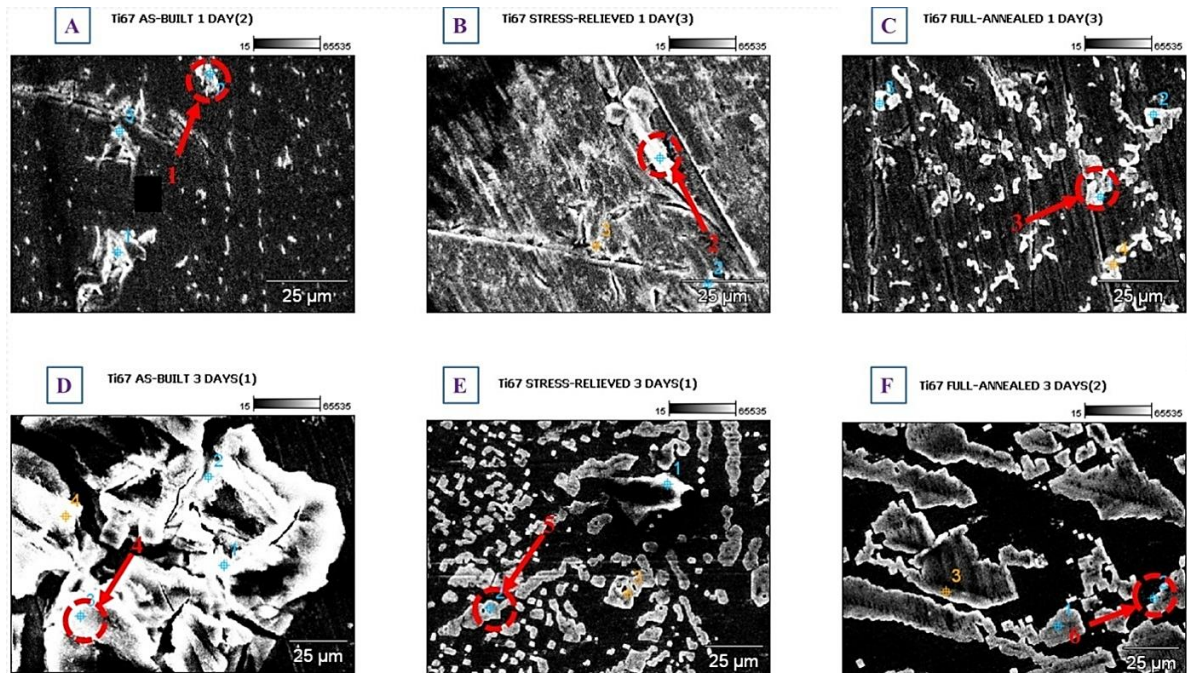
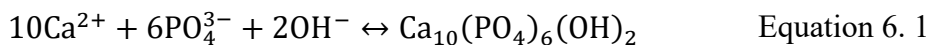


Figure 6. 2: The formation of apatite layer on LPBF Ti-6Al-7Nb alloy after 24 hours immersion; (A) As-built, (B) Stress-relief annealed, (C) Full-annealed, and 72 hours immersion; (D) As-built, (E) Stress-relief annealed, and (F) Full-annealed.

All the specimens displayed the formation of an apatite layer, a stacked flower-like apatite deposit, but at varied intensities. The apatite formation mechanism on titanium implant surfaces in the simulated body fluids is well explained by Jonasova et al. (Jonášová et al., 2004). The aggressive Na^+ ions in DPBSS dissolve the passivated oxide layer, forming an amorphous layer with alkali ions. Alkali ions diffuse from the amorphous layer, while hydronium ions (H_3O^+) from DPBSS penetrate the surface, initiating Ti-OH group formation on the implant within 0.5 hours of immersion. Ti-OH groups on the implant's surface react with calcium ions from the DPBSS to form calcium titanate which significantly enhances surface bioactivity. Calcium titanate absorbs phosphate and calcium ions from the DPBSS, forming apatite nuclei on the implant surface. This apatite grows and spreads across the implant surface using the calcium and phosphate ions from the DPBSS as presented in Equation 6. 1.



Calcium and phosphate ions incorporated within the corrosion product (EDS analysis Table 6. 2) justify that the formed product in this study is apatite. However, there are few apatite precipitate particles on as-built LPBF Ti-6Al-7Nb specimen's surface after soaking in

DPBSS for 24 hours, while more apatite precipitates were deposited on the stress-relief annealed and full-annealed Ti-6Al-7Nb alloy respectively (see Figure 6. 2A, B, and C). The grown island-like apatite layer with some microcracks could be observed in the as-built specimen, while the stress-relieved and the full-annealed specimen displayed a spread-out apatite layer with a dendritic morphology after 72 hours of immersion (see Figure 6. 2D, E, and F).

Table 6. 2: EDS analysis of the corrosion product after 24 hours and 72 hours of immersion.

IMMERSI ON HOURS (H	SAMPLE CONDITIO N	SPOT IN FIGURE 6.2.	O	Al	P	Ca	Ti	Nb
24	As-built	1	10.59	3.26	2.21	0.15	76.76	7.03
	Stress- relieved	2	9.65	3.56	0.93	0.00	79.08	6.78
	Full- annealed	3	5.26	3.52	1.64	0.11	81.14	8.33
72	As-built	4	44.17	13.95	30.35	3.19	1.28	3.88
	Stress- relieved	5	5.43	3.32	1.02	0.00	81.56	8.02
	Full- annealed	6	13.73	3.75	2.83	0.11	70.16	9.41

After 120 hours of DPBSS soaking, all the samples developed a condensed uniform apatite layer on their surfaces (Figure 6. 3A, B, and C), which continued to grow in a “cauli-flower” like morphology after 168 hours of immersion (see Figure 6. 3D, E, and F). At this point, the apatite did not entirely cover the whole surface in all the materials but new precipitate points on the exposed sites of the full-annealed specimen could be observed. EDS analysis in Table 6. 3. confirmed increased calcium and phosphate ion percentages on the implant surface. This suggests that more calcium and phosphorous ions were absorbed from the DPBSS solution to grow the apatite layer with prolonged immersion in both as-built and heat-treated specimens.

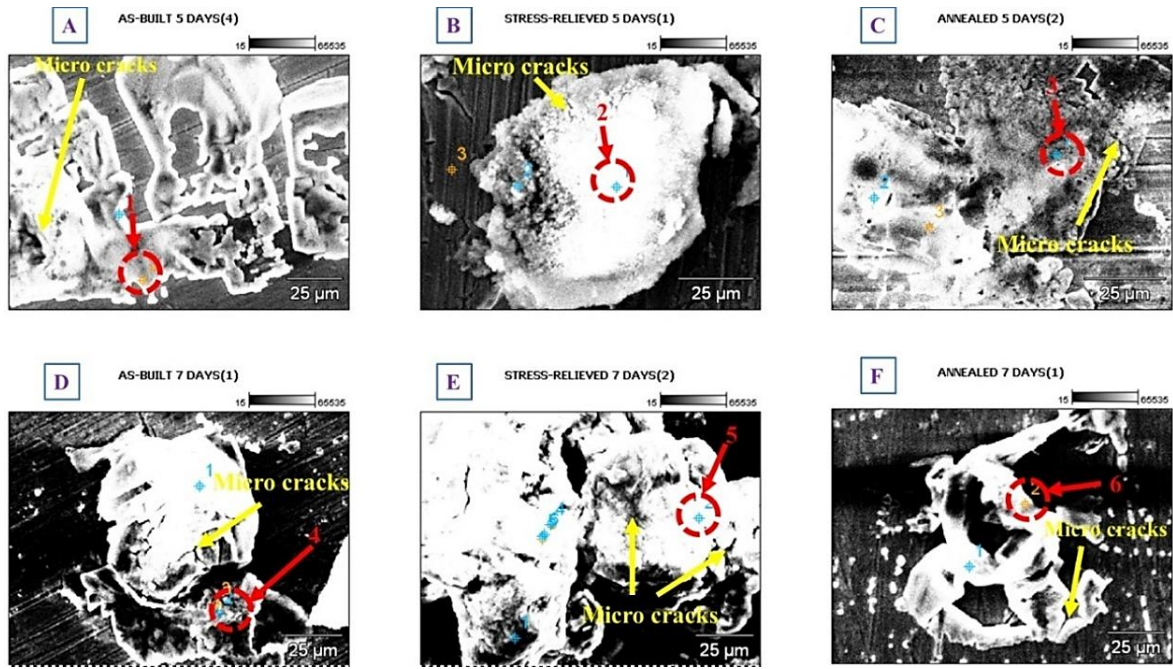


Figure 6. 3: The growth of apatite layer on (A) As-built (B) Stress-relief annealed, (C) Full-annealed after 120 hours of immersion and (D) As-built (E) Stress-relief annealed (F) Full-annealed Ti-6Al-7Nb specimen after 168 hours of immersion in DPBSS solution.

Table 6. 3: EDS analysis of the corrosion product on as-built and heat-treated Ti-6Al-7Nb specimens after 120 hours and 168 hours of immersion in DPBSS solution.

IMMERSION HOURS (Hrs)	SAMPLE CONDITION	SPOT	O	Al	P	Ca	Ti	Nb
120	As-built	1	7.33	3.58	0.87	1.90	78.75	7.57
	Stress-relieved	2	51.49	14.79	27.08	2.75	0.67	0.67
	Full-annealed	3	23.69	5.27	13.10	1.72	39.36	13.54
168	As-built	4	25.94	7.22	33.10	3.86	4.70	16.46
	Stress-relieved	5	10.29	4.55	37.41	2.91	7.06	24.84
	Full-annealed	6	38.40	1.91	8.15	1.09	39.58	6.46

“Cauli-flower” shaped morphology of the deposited apatite layer continued to grow in the as-built and heat-treated LPBF Ti-6Al-7Nb samples after 240 hours of immersion (Figure 6. 4A, B, and C). However, an important observation after 336 hours of immersion was that the full-annealed specimen had its surface fully covered with the apatite layer (Figure 6. 4F) while “cauli-flower” like apatite could still be seen on the as-built and stress-relief annealed specimens with some bare metal surfaces still present (Figure 6. 4D, and E). The increased calcium and phosphorous percentages through EDS analysis summarized in Table 6. 4 confirm that this product is indeed apatite. The LPBF Ti-6Al-7Nb alloy's capacity to induce apatite can thus be ranked as follows: fully – annealed > stress – relief annealed > as – built.

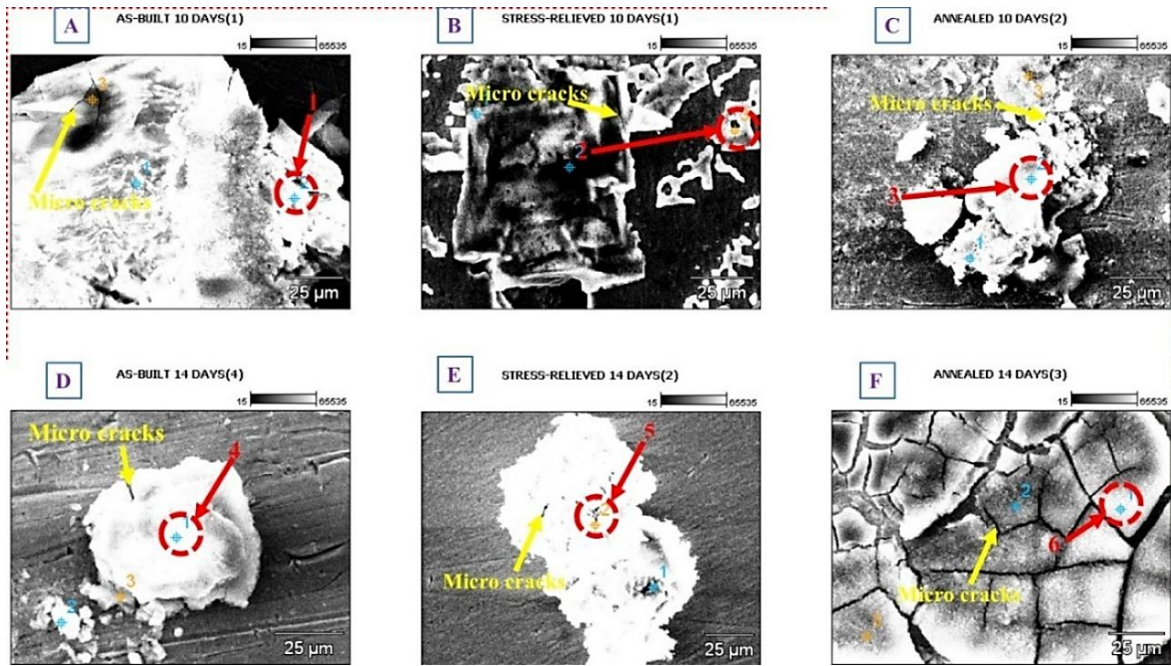


Figure 6. 4: SEM observation of the apatite deposition on the as-built and heat-treated specimens after 240 hours of immersion: (A) As-built, (B) Stress-relief annealed, and (C) Full-annealed; and 336 hours of immersion; (D) As-built, (E) Stress-relief annealed, and (F) Full-annealed Ti-6Al-7Nb specimens.

Table 6. 4: EDS Analysis of the apatite deposit in the as-built and heat-treated LPBF Ti-6Al-7Nb alloy after 240 hours and 336 hours of immersion in DPBSS solution.

IMMERSI ON HOURS (Hrs)	SAMPLE CONDITIO N	SPOT IN Figur e 6. 4	O	Al	P	Ca	Ti	Nb
240	As-built	1	9.85	2.19	7.14	7.18	1.33	2.31
	Stress- relieved	2	3.19	4.86	9.07	1.07	3.26	8.55
	Full- annealed	3	3.58	6.13	7.27	3.92	6.34	3.72
336	As-built	4	3.97	4.33	9.63	9.77	1.76	6.01
	Stress- relieved	5	0.41	11.57	26.15	1.39	10.92	19.55
	Full- annealed	6	45.32	13.66	25.08	10.93	1.65	1.20

6.4 The role of microstructure constituent phase type on apatite formation and corrosion behaviour.

The material's susceptibility to corrosion will depend on its constituent phase ([Usha Rani et al., 2024](#)). Studies indicate that the metastable acicular α' phase, due to its higher energy state, dissolves more readily during corrosion, leading to reduced corrosion resistance in LPBF-produced $\alpha+\beta$ titanium ([Dai, Zhang, Zhang, Zhang, et al., 2016](#); [J. Yang et al., 2017](#)). Additionally, it is established that a greater concentration of the β -phase improves corrosion resistance, whereas an increase in the α -phase reduces it ([J. Yang et al., 2017](#)). As described earlier, the limited diffusion of niobium due to the fast solidification rate in the as-built specimens resulted in the microstructure composed of more acicular α' phase and little β phase content. Therefore, as-built Ti-6Al-7Nb specimens exhibited low corrosion resistance, primarily due to their acicular α' -rich microstructure with limited β phase content, making them more prone to corrosion-induced dissolution.

Despite annealing at 650°C, stress-relief annealed samples exhibit a relatively poor corrosion resistance. This suggests that the 650°C annealing temperature was insufficient to fully transform the metastable α' phase, leaving residual α' martensite due to incomplete recrystallization and diffusion. Thus, the stress-relief annealed specimens had less corrosion resistant β phase, still retained the corrosion prone α' phase and some transformed α phase leading to its inferior corrosion resistance. On the other hand, the 850°C full-annealed specimen exhibited better corrosion resistance. This is linked to the higher β phase content in the fully annealed LPBF Ti-6Al-7Nb specimen which facilitates the development of a stable and strong passive protective layer on the specimen's surface. Thus, the corrosion resistance of Ti alloy phases generally ranks as $\beta > \alpha > \alpha'$, with β offering the highest protection (Dai, Zhang, Zhang, Zhang, et al., 2016). However, from microstructure analysis, XRD analysis, and the observed corrosion behavior in the current research, the ($\alpha+\beta$) phase mixture in LPBF Ti-6Al-7Nb shows higher corrosion resistance than ($\alpha'+\beta$). Therefore, a diffusional phase transformation caused by an appropriate heat treatment procedure is fundamental in improving the LPBF Ti-6Al-7Nb implants' resistance to corrosion in the body.

With regards to biocompatibility through apatite forming ability analysis, the full-annealed samples displayed the fast nucleation and good spread of apatite than the stress-relief annealed and as-built samples (see Figure 6. 2, Figure 6. 3, and Figure 6. 4). Since the full-annealed specimens had a higher fraction of the β phase than the stress-relief annealed and as-built samples, it can be concluded that the β phase has a positive impact on the apatite inducing ability on titanium implants. However, further in vitro studies on biocompatibility through cell viability and proliferation analysis using mouse fibroblast cells or human bone osteosarcoma cell lines have to be carried out to support this assertion.

6.5 Impact of microstructure constituent phase morphology on apatite formation and corrosion behaviour.

Besides phase type, the shape and size of phase constituents impact the corrosion behavior of LPBF Ti-6Al-7Nb parts. In terms shape constituent phases, Chen et al. (X.-M. Chen et al., 2020) concluded that acicular microstructures exhibit poorer corrosion resistance than the spherical and rod-shaped microstructures. Similarly, Iqbal et al. (Iqbal & Fedel, 2018) found that acicular or needle morphology exhibits a lower corrosion resistance than the rod or lamellar like morphology. The acicular morphology in the as-built specimens displayed the worst corrosion in our study which concurs with these prior findings.

As seen in Figure 4. 5A, the stress-relief annealed specimen maintained the acicular structure of the α' phase with some β precipitates along the grain boundaries due to incomplete transformation. Bingjing et al. (Zhao et al., 2017) found that these nano sized β precipitates are susceptible to corrosion due to their larger surface area. Therefore, corrosion easily initiates in these sites and spreads to the whole microstructure resulting in poor corrosion resistance observed in the stress-relief annealed specimen.

Increasing the annealing temperature to 850°C, led to a complete transformation from the acicular morphology to lamellar morphology was achieved as seen in Figure 4. 5B. This morphology produced the best corrosion resistance and apatite formation in our study. Therefore, from a microstructural morphology perspective, the corrosion resistance and apatite-forming ability of LPBF Ti-6Al-7Nb specimens follow the ranking: lamellar > nearly spherical > acicular.

6.6 Conclusions.

This chapter has evaluated the corrosion resistance and biocompatibility of LPBF Ti-6Al-7Nb in body simulated fluids. It has been concluded that the synergetic effect of the metastable acicular α' phase and coarse grain size greatly deteriorated the corrosion resistance of as-built LPBF Ti-6Al-7Nb specimens in body simulated fluids (Dulbecco's phosphate buffered saline solution) resulting in an elevated corrosion rate of $2.285 \times 10^{-4} \frac{mm}{yr}$. This elevated corrosion rate suggests a more rapid degradation of the acetabular implant's structural integrity and an increased release of metallic ions into the body, potentially inducing adverse biological reactions and premature implant failure. Stress-relief annealing slightly improved the corrosion resistance, exhibiting a corrosion rate of $1.9 \times 10^{-4} \frac{mm}{yr}$ while full-annealing treatment greatly improved the corrosion resistance of Ti-6Al-7Nb specimens in DPBSS leading to the lowest corrosion rate of $1.885 \times 10^{-4} \frac{mm}{yr}$. This suggests an important role of full annealing treatment in mitigating the corrosion failure mechanisms of acetabular implants.

In terms of biocompatibility, it has been established in this chapter that the full-annealed Ti-6Al-7Nb specimens displayed fast nucleation and wide spread of the apatite layer on its surface within 24 hours which uniformly spread in a “cauli-flower” and dendrite pattern after long immersion times of 72, 120, 168, 240, and 336 hours. This directly translates to

excellent compatibility and bone tissues can easily attach to this apatite layer and improve the osseointegration process for a longer service life of the acetabular implant.

Chapter 7: Conclusions and Recommendations for Future Work.

7.1 Chapter Summary.

This final chapter provides critical conclusions from this study in relation to the stated aim and objectives. Finally, suggestions for the future work are made to knowledge expansion in this area of research.

7.2 Conclusions and finding from this research work

The following are the main conclusions established by this study in relation to the aims and objectives of section 1.4 of Chapter 1:

7.2.1 *Understanding from the literature review.*

- i. The main failure mechanisms for acetabular hip implants identified are wear, corrosion, and fatigue failure mechanisms. From the human hip joint structural configuration and kinematics analysis, it has been observed that the multidirectional movement of the hip joint combined with varying loads during different loading conditions subjects the acetabular implant to many wear mechanisms. Additionally, the aggressive body environment makes the acetabular implants susceptible to corrosion failure mechanisms. The cyclic loading at the hip joint during activities like walking and climbing stairs, induces fatigue failure in the implant which is further exacerbated by the design flaws, surface defects, and the corrosive bodily fluids which accelerate fatigue crack growth.
- ii. In selecting the potential materials to address premature acetabular implants failure, the commonly utilized Ti-6Al-4V alloy for acetabular prosthesis has demonstrated limited corrosion resistance and insufficient biocompatibility, making it susceptible to the failure mechanisms discussed in (i). Comparative analyses through *in-vitro* and *in-vivo* studies have demonstrated that the Ti-6Al-7Nb alloy offers superior biocompatibility, improved corrosion resistance, and improved tribocorrosion resistance relative to Ti-6Al-4V. Consequently, Ti-6Al-7Nb was the focus material in this research.
- iii. LPBF has proven its capability to fabricate complex near-net-shaped hip implants with high geometric accuracy, closely replicating the structure of natural bone. LPBF is emerging as the premier manufacturing method, surpassing traditional techniques that are often plagued by manufacturing defects, inhomogeneous microstructures, and limitations in producing intricate, bone-mimicking geometries.

7.2.2 *Findings from the Experimental research.*

The primary findings drawn from the research objectives are outlined as follows:

i. Investigate the microstructural features of the as-built, and heat-treated Ti-6Al-7Nb alloy samples

Chapter 4 of this study was devoted to microstructural analysis of the as-built and heat-treated LPBF Ti-6Al-7Nb samples.

- It was established from this study that the as-built LPBF Ti-6Al-7Nb specimens' microstructure comprises elongated prior β grains accompanied by finely dispersed acicular α' martensite as well as a basketweave microstructure displaying an interwoven arrangement of the phases (grains) in a crisscross manner resembling a woven basket. Stress-relief annealing treatment at 650°C causes nucleation of the β phase precipitates while full-annealing at 850°C results in lamellar microstructure due to complete $\alpha' \rightarrow \alpha + \beta$ phase transformation.

ii. Determine compressive biomechanical properties, microhardness, and tribological behaviour of the as-built, and heat-treated LPBF Ti-6Al-7Nb alloy specimens.

Chapter 5 thoroughly explored the compressive mechanical properties, microhardness, and tribological performance of both as-built and heat-treated LPBF Ti-6Al-7Nb specimens.

- It was determined that the relatively lower compressive yield strength of fully annealed LPBF Ti-6Al-7Nb specimens (973 MPa) at 0.015 mm/s strain rate indicates better biomechanical compressive properties and lower stress shielding effect on the surrounding bone than the as-built and stress-relieved Ti-6Al-7Nb specimens. Additionally, the as-built as well as the heat-treated LPBF Ti-6Al-7Nb specimen displayed strain hardening property with increased strain rate which is critical to enhance the fatigue strength and fretting damage resistance of the acetabular implant.
- Accelerated wear or ploughing on the surrounding bone can be expected with the use of as-built and stress-relieved LPBF Ti-6Al-7Nb parts in acetabular implants due to their enhanced microhardness of $461 \pm 20 \text{ HV}_{0.5}$ and to $412 \pm 16 \text{ HV}_{0.5}$ respectively. The full-annealed samples exhibit the lowest microhardness value of

$370 \pm 5 \text{ HV}_{0.5}$, a significant 19.7% reduction in microhardness from the as-built sample necessary to reduce ploughing on the surrounding bone tissue.

- During different loading conditions, the oxidative wear mechanism is dominant in the fully annealed LPBF Ti-6Al-7Nb specimens than the as-built and stress-relief annealed specimens where abrasive and adhesive wear mechanisms associated with more material loss were too dominant. This implies that utilizing fully annealed LPBF Ti-6Al-7Nb alloy generates tribofilms capable of preventing further ion release and lubricating the acetabular implant to improve its articulation and service life.
- iii. Evaluate the corrosion resistance and biocompatibility through in vitro apatite-forming capabilities of the additively manufactured Ti-6Al-7Nb specimen for acetabular implant through immersion corrosion test in body simulated fluids.**

The final part of this work, Chapters 6 and 7 focused on the corrosion resistance and biocompatibility through the formation of a bone-like apatite layer on the specimens' surface upon immersion in simulated body fluids at different time periods. The following conclusions were established:

- The synergetic effect of the metastable acicular α' phase and coarse grain size greatly deteriorated the corrosion resistance of as-built LPBF Ti-6Al-7Nb specimens in simulated body fluids (Dulbecco's phosphate buffered saline solution) resulting in an elevated corrosion rate of $2.285 \times 10^{-4} \frac{\text{mm}}{\text{yr}}$. This accelerated corrosion predicts a faster decline in the structural integrity of the acetabular implant and an increased release of metallic ions into the body, potentially triggering adverse biological reactions and premature implant failure. In contrast, the application of full-annealing treatment markedly enhanced the corrosion resistance of the specimens, reducing the corrosion rate to $1.885 \times 10^{-4} \frac{\text{mm}}{\text{yr}}$. This underscores the critical significance of full annealing in mitigating corrosion-induced degradation mechanisms in acetabular implants.
- The full-annealed Ti-6Al-7Nb specimens displayed fast nucleation and wide spread of the apatite layer on its surface within 24 hours which is uniformly spread.
- and dendrite pattern after long immersion times of 72, 120, 168, 240, and 336 hours. This directly translates to excellent biocompatibility and bone tissues can easily

attach to this apatite layer and improve the osseointegration process for a longer service life of the acetabular implant.

Overall, the lamellar microstructure in the full-annealed Ti-6Al-7Nb specimens produced excellent compressive properties (973 MPa compressive yield strength), lowest microhardness ($370 \pm 4.99 \text{ HV}_{0.5}$) essential for minimizing ploughing on the softer surrounding bone, highest corrosion resistance ($1.885 \times 10^{-4} \frac{\text{mm}}{\text{yr}}$) necessary to maintain the structural integrity and minimizing metallic ion release into the body, thereby reducing the risk of premature acetabular implant failure, and excellent in-vitro apatite-forming capability indicating superior biocompatibility in DPBSS than as-built and stress-relief annealed Ti-6Al-7Nb specimens. This makes the full-annealed Ti-6Al-7Nb a strong candidate for acetabular implant applications.

7.3 Recommendations for Future Work.

Although the study has effectively met its objectives and addressed several knowledge gaps, there is a need for further exploration. To facilitate the adoption of LPBF and annealing heat treatment of Ti-6Al-7Nb alloys to improve service life of acetabular implants, the following recommendations are suggested:

- This study did not carry out fatigue tests on the as-built and heat treated LPBF Ti-6Al-7Nb specimens. Fatigue testing would be a good attempt to understand the fatigue behaviour changes with heat treatment and observe if these specimens can withstand the cyclic loading of the hip joint.
- The corrosion resistance analysis in this study was based on weight loss analysis. However, a detailed study through electrochemical testing would be more informative about the corrosion behaviour of these specimens. Additionally, the bilayer structure observed in other titanium alloys, as documented in the literature, has demonstrated enhanced corrosion resistance and improved osseointegration. To validate its formation and assess its electrochemical stability on LPBF as-built and heat-treated LPBF Ti-6Al-7Nb specimens in this study, electrochemical impedance spectroscopy should be conducted.
- Currently, a lot of efforts are being directed at improving the antibacterial properties of implants to prevent infections, reduce antibiotic use, and support osseointegration. It is hereby suggested that antibacterial studies should be conducted in the future on LPBF fabricated and heat-treated LPBF Ti-6Al-7Nb specimens.

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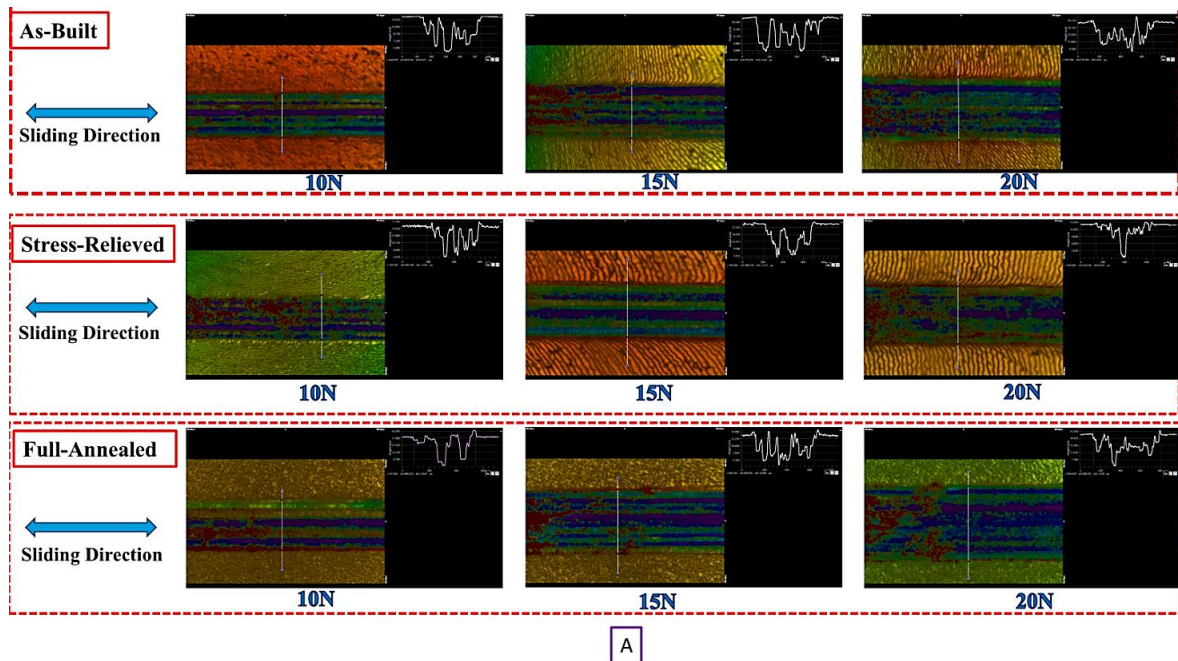
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Appendices.

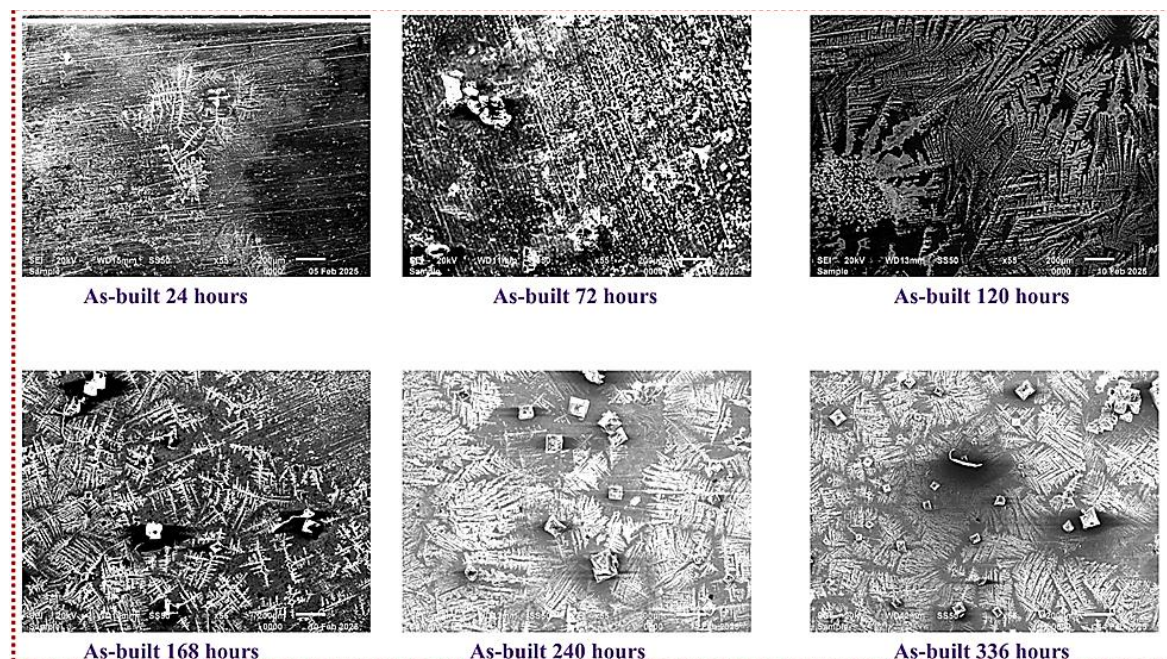
Appendix A: Research equipment and software used in this study.

Equipment/ Software	Tests/ Activities conducted
JEOL JSM-6610 Scanning Electron Microscope equipped with Energy Dispersive X-ray Spectroscopy (EDS)	✓ Analysis of Ti-6Al-7Nb powder morphology ✓ Analysis of powder chemical composition ✓ Microstructural analysis ✓ Wear tracks morphology and wear mechanism analysis
Malvern Mastersizer 3000E	✓ PSD analysis of the Ti-6Al-7Nb powder
The Hyrax SLM Machine	✓ Fabrication or selective laser melting of Ti-6Al-7Nb samples.
Super Series X Vacuum Furnace	✓ Heat treatment of the Ti-6Al-7Nb samples
DK7732 numerical control wire electrical discharge cutter	✓ Cutting samples from the build plate and to their final geometries
Hot mounting press Opal 410 machine	✓ Ti-6Al-7Nb specimens mounting
Qualitest Qualipol-VTD-SA grinding and polishing machine	✓ Ti-6Al-7Nb samples surface grinding and polishing
Lab Companion UC-02 Ultrasonic Cleaner	✓ Ultrasonic cleaning of samples
Bruker D8 Advance powder diffractometer	✓ Phase analysis of the raw Ti-6Al-7Nb powder, as-built, and heat-treated samples.
Olympus SZX7 stereo microscope equipped with PRECiV imaging software	✓ Geometrical measurements of the wear tracks
The Schenck POZ 160 Universal Testing Machine	✓ Compression testing of the as-built and heat-treated Ti-6Al-7Nb samples.
Struers Duramin-40 Vickers microhardness tester	✓ Microhardness test
Rtec Multipurpose Tribometer equipped with MFT 17 software	✓ Wear testing of the samples
ESCO Ductless Fume Hood	✓ Extracts released fumes when etching

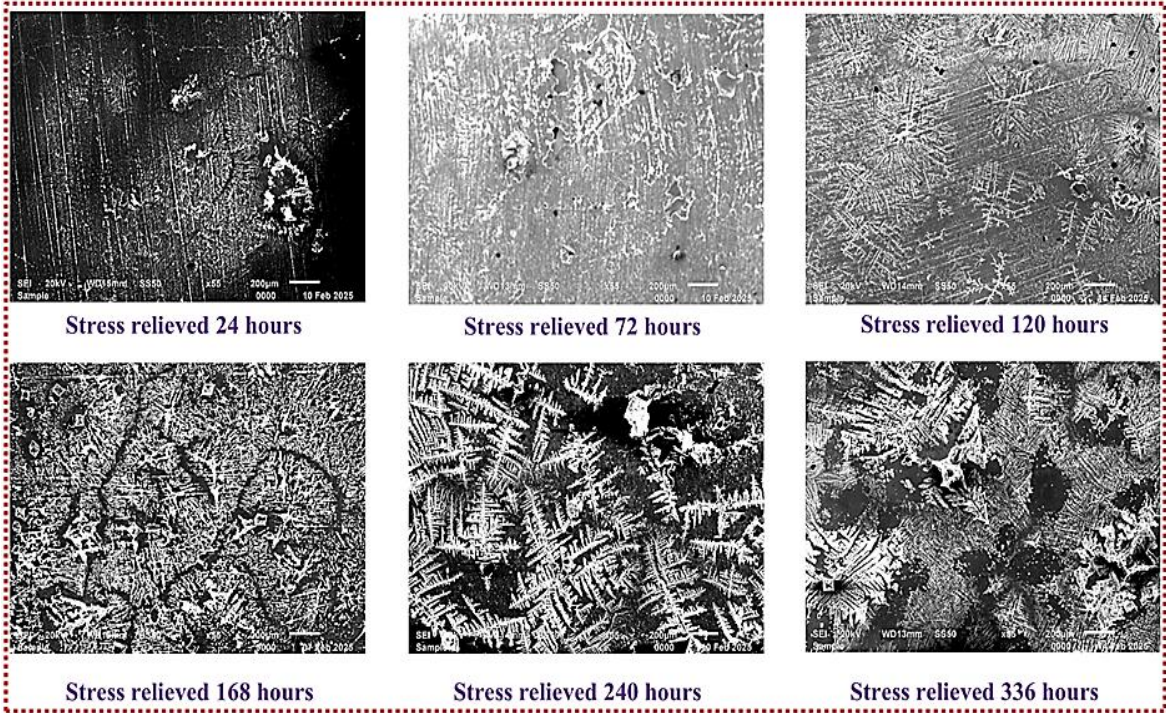
Appendix B: Wear tracks 3D images from the Tribometer's white light interferometer.



Appendix C: Morphology of the Apatite layer development in as-built sample after different immersion hours.



Appendix D: Morphology of the Apatite layer development in stress-relief annealed sample after different immersion hours.



Appendix E: Morphology of the Apatite layer development in fully annealed sample after different immersion hours.

