Effect of hot isostatic pressing followed by heat treatment on mechanical properties of additively manufactured Ti6Al4V alloy

M. Tech. Thesis

By

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DEPARTMENT OF MECHANICAL ENGINEERING

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A THESIS

Submitted in partial fulfillment of the

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by

Kartik Kumar

DEPARTMENT OF MECHANICAL ENGINEERING
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CANDIDATE'S DECLARATION

I hereby certify that the work which is being presented in the thesis entitled **Effect of hot isostatic pressing followed by heat treatment on mechanical properties of additively manufactured Ti6Al4V alloy** in the partial fulfilment of the requirements for the award of the degree of **MASTER OF TECHNOLOGY** in Advanced Manufacturing and submitted in the **DEPARTMENT OF MECHANICAL ENGINEERING Indian Institute of Technology Indore** is an authentic record of my own work carried out during the time period from July 2023 to June 2025 under the supervision of **Dr. Girish Verma, Assistant Professor Department of Mechanical Engineering, IIT Indore.**

The matter presented in this thesis has not been submitted by me for the award of any other degree of this or any other institute.

02/06/25

Signature of the student with date

(Kartik Kumar)

This is to certify that the above statement made by the candidate is correct to the best of my knowledge.

03/06/25	04/06/2025	
Signature of Supervisor of M.Tech. thesis with date	Signature of Co Supervisor of M. Tech. thesis with date	Signature of Co Supervisor of M. Tech. thesis with date
(Dr. Girish Verma)	**(Dr. Indrasen Singh)**	**(Dr. Mithun Palit)**

Kartik Kumar has successfully given his M.Tech. Oral Examination held on **26th May 2025.**

03/06/25

Signature of Supervisor of M.Tech. thesis

Date:

Convener, DPGC

Date: 05/06/25

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ABSTRACT

The growing application of additive manufacturing (AM) in aerospace, biomedical, and automotive industries has led to increased interest in optimizing the performance of Ti6Al4V alloy components fabricated by the Direct Metal Laser Sintering (DMLS) process. This study investigates the effect of Hot Isostatic Pressing (HIP) followed by post-processing heat treatments on the mechanical properties and microstructural evolution of additively manufactured Ti6Al4V alloy. Test specimens were fabricated via the DMLS technique and subjected to HIP at various temperatures 700°C, 910°C and 1060°C, followed by controlled heat treatments at different temperature. A comprehensive analysis was conducted to assess changes in density, hardness, tensile strength in multiple build orientations, wear resistance, and corrosion behavior. The results demonstrate that HIP significantly reduces internal porosity and promotes a more homogeneous microstructure, while subsequent heat treatment further enhances mechanical integrity. Tensile properties in x and y build directions (parallel to the build plane) showed higher tensile strength and ductility compared to the z direction and due to alteration in microstructure results in changes in the hardness of the material were observed, along with better tribological and corrosion performance in treated samples compared to their as-built sample. This study provides insights into optimizing post-processing protocols to enhance the structural performance of AM Ti6Al4V components.

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CHAPTER 1

INTRODUCTION

1.1 Background and Importance of Titanium Alloys

The increasing demand for advanced materials such as aluminum, steel, nickel, and titanium alloys spans across various industrial sectors—including automotive, aerospace, marine, and biomedical—and continues to rise steadily. In the early 20th century, materials like steel, aluminum, and copper dominated engineering applications, accounting for approximately 80–85% of usage. However, a significant shift began in the mid-1950s with the emergence of Ti6Al4V, a titanium alloy that gained popularity due to its outstanding combination of mechanical strength and manufacturability.

Ti6Al4V became particularly attractive because of its low stiffness, which facilitates forming operations, and its excellent surface wettability, which enhances weldability. These characteristics have positioned the alloy as a valuable material for a wide range of industries. As a result, ongoing research continues to focus on further enhancing the performance of titanium alloys to meet application-specific requirements.

According to industry forecasts, the demand for new large commercial aircraft is projected to exceed 28,000 units globally by 2031, significantly boosting the need for titanium-based components, particularly in aerospace engine systems. Estimates suggest that by 2030, titanium consumption in commercial jet engines could surpass 25,000 metric tonnes.

1.2 Applications of Titanium Alloys

Titanium's light weight, corrosion resistance, and high strength have made it a preferred choice across a wide range of applications:

1. **Aerospace Industry:** Titanium is extensively used in aircraft structures, spacecraft, missiles, and other aerospace components due to its exceptional strength-to-weight ratio, corrosion resistance, and ability to withstand elevated temperatures.

2. **Medical Field:** Owing to its biocompatibility and resistance to body fluids, titanium is commonly used for dental implants, orthopedic devices, and joint replacements. Its mechanical properties and inertness make it suitable for long-term biomedical use.

3. **Automotive Sector:** In high-performance vehicles, titanium is utilized in components such as connecting rods, valves, and gears, where light weight and heat resistance contribute to enhanced performance and fuel efficiency.

4. **Sports Equipment:** Titanium's durability, low weight, and high strength make it ideal for manufacturing bicycle frames, golf clubs, tennis rackets, and other high-end sports gear.

5. **Architecture:** Modern architectural designs incorporate titanium for roofing, cladding, and structural elements due to its aesthetic appeal, corrosion resistance, and long service life.

1.3 Classification and Microstructure of Titanium Alloys

Titanium alloys are traditionally classified based on the dominant phase present—α (alpha), α+β (alpha+beta), and β (beta). However, this classification can sometimes be misleading, as certain α alloys may still contain trace amounts of β phase. For example:

Near-α alloys typically contain small quantities of β-stabilizing elements (less than 10% β phase).

Metastable β alloys (near-β) include higher amounts of β stabilizers (10–15%) and usually consist of over 50% α phase in their processed state..

Several kinds of titanium alloys, which are created by mixing titanium with non-metallic or other metals to improve specific metal properties, have been found by numerous scientists and researchers. Several popular varieties of titanium alloys include:

1. **Ti-6Al-4V**: Because of its remarkable qualities, this common titanium alloy—which consists of 6% aluminium and 4% vanadium—is widely used. This alloy has a high strength-to-weight ratio, excellent resistance to corrosion, and is widely used in biomedical and aerospace applications.
2. **Ti-3Al-2.5V**: This alloy, which is 2.5% vanadium and 3% aluminium, is well known for its advantageous properties. It has a remarkable strength-to-weight ratio, good weldability,
3. **Ti-15V-3Cr-3Sn-3Al**: This titanium alloy, which has 15% vanadium, 3% chromium, 3% tin, and 3% aluminium, is made to make parts for gas turbine engines and aeroplanes. It is

4. designed to fulfil the particular needs of these uses, guaranteeing top performance and longevity.
5. **Ti-5Al-2.5Sn**: Ti is an alloy of titanium consisting of 5% aluminium and 2.5% tin that is utilised in high-performance engines
6. **Ti-6Al-7Nb**: This is a biocompatible titanium alloy used in medical implants, made up of 6% aluminum and 7% niobium.

1.4 Evolution of Additive Manufacturing Technology

In the current manufacturing landscape, producing components with intricate geometries and tight dimensional tolerances using traditional methods poses significant challenges. Conventional processes such as casting, forging, milling, turning, and drilling often struggle to meet the precision and complexity required for modern, high-performance applications. This limits the design flexibility available to engineers, especially when working on parts intended for advanced sectors such as aerospace, biomedical, and automotive.

Fig.1.1 Additively Manufactured Jet Engine blade [5]

One such material is machine-grade titanium alloy, notably Ti6Al4V, which, despite its exceptional material properties, presents considerable machining difficulties. These challenges stem from its:

- High strength at elevated temperatures
- Low thermal conductivity
- Low elastic modulus
- High chemical reactivity, especially when molten

These characteristics result in reduced material removal rates, tool wear, and diminished machining precision. In addition, titanium's reactivity during casting often complicates the process, increasing the likelihood of environmental and mold-related issues.

To address these limitations, Additive Manufacturing (AM) has emerged as a transformative solution. By building parts layer by layer, AM enables the creation of highly complex, dimensionally accurate components that are often impossible—or prohibitively expensive—to produce using conventional methods. It provides engineers and designers with newfound freedom to innovate, iterate, and optimize their designs rapidly.

In particular, the aerospace industry has benefited significantly from AM, as it allows for:

- Accelerated prototyping and design validation
- Production of lightweight, structurally efficient components
- Shorter development cycles
- Lower manufacturing costs for low-volume, high-value parts

AM is thus reshaping the aviation industry by enabling faster innovation, greater design flexibility, and a more sustainable manufacturing approach for the future of air travel.

1.5 Advantages of AM techniques

The advantages of AM process are :

- Part designs have been improved to use less material and waste while keeping functionality; nearly 95% of the material is reusable in future construction projects, and there is little material losses during AM life cycle.
- Design sharing in the digital type file format is simple, making it easy to modify and customize parts and products.
- Weight reduction, which is essential for aerospace applications, is the process of redesigning a part using software to cut weight for certain boundary condition by almost 40% or more.
- Quick production because the entire supply chain is digital before it is manufactured. Put differently, the time it takes to print a 3D file is only a few days, whereas the preparation of a mould takes weeks. Additionally, creating moulds for unique parts is costly, but with additive manufacturing, digital design can be changed quickly.

1.6 Challenges associated with AM techniques

Some of the drawbacks of AM process are :

- Because AM cannot produce many parts as quickly as other production processes due to the build platform's limited area, more work is required to increase the material base that AM can process.
- Metals with powdered forms are more expensive per unit mass in powder-based AM processes than their equivalent bar stock because it is more expensive to compete with traditional forging and casting techniques due to the additional processing required to turn the base metal into a powdered form.
- The microstructure outcomes are determined by process-related high cooling rates and the high temperature gradient that is created when the local level of energy input concentration is concentrated [31].
- Because of the build-up layer formation and consequent stair-stepping, the resulting parts typically exhibit adverse surface roughness, particularly when producing curved or inclined surfaces.
- AM parts become porous as a result of processing parameters that are not ideal.

1.7 Mechanism based classification of Additive Manufacturing

All of additive manufacturing methods listed below have unique benefits and drawbacks. The choice of a particular technique depends on factors like the required accuracy, part size, desired material, and available budget. All of additive manufacturing methods listed below have unique benefits and drawbacks.

- **Material Extrusion (ME):** The most popular method of Additive Manufacturing. To create a part that resembles a stack of layers, thermoplastic materials are melted and extruded through a nozzle.
- **VAT Photopolymerization:** This process builds a solid component layer by layer by curing liquid resin with a laser. It is renowned for its capacity to produce intricate and highly accurate parts.
- **Powder Bed Fusion (PBF):** PBF builds parts layer by layer by fusing tiny powder particles of metal or nylon together with a laser. SLS is widely used to produce functional prototypes and completed parts.
- **DED, or direct energy deposition:** With Directed Energy Deposition (DED), metal wire or powder is melted using an electron or laser beam and then selectively deposited onto the build platform to create the solid part. DED is frequently used to create complex and large metal parts.
- **Binder Jetting (BJ):** BJ is the process of layer-by-layer applying a liquid binding material to a powder bed in the order to form a solid portion. Sand moulds for casting and metal parts are frequently made with BJ.

- **Material Jetting (MJ):** Material Jetting (MJ) deposits liquid photopolymer droplets onto a build platform using a technique akin to an inkjet. After being cured by UV light, these droplets allow layer by layer construction of solid parts. MJ is widely used in the production of intricate and highly accurate parts.
- **Sheet lamination:** Layers of material sheets are laminated together to construct a part in the process of sheet lamination. Usually composed of paper, plastic, or metal, the sheets are joined together by means of an adhesive. Layer by layer, the procedure is repeated until the last section is finished.

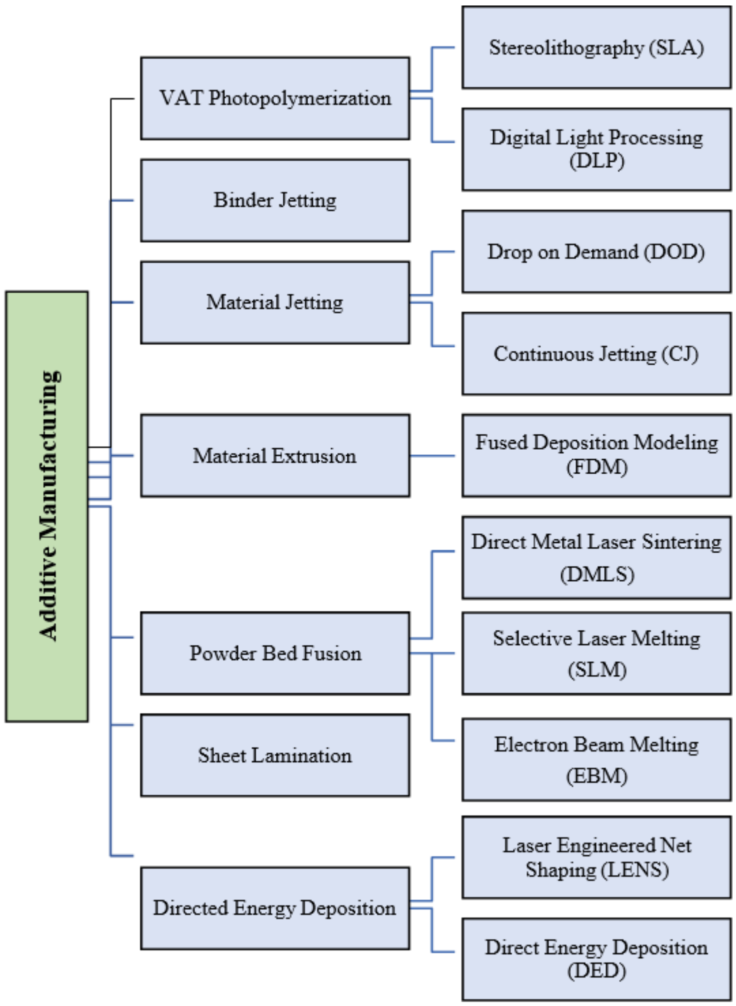


Fig.1.2: Flow Chart showing different types of Additive Manufacturing process.

1.8 Direct Metal Laser Sintering (DMLS)

Direct Metal Laser Sintering (DMLS) is one of the most prominent and widely adopted technologies under the umbrella of Powder Bed Fusion (PBF) in Additive Manufacturing (AM). It is often considered a preferred method due to several distinct advantages:

- **Exceptional Accuracy:** DMLS offers outstanding dimensional precision, enabling the production of highly accurate components. This makes it especially suitable for demanding applications in aerospace, medical devices, and automotive engineering**.**
- **Broad Material Compatibility:** Although primarily used with metal powders, DMLS technology is adaptable and can work with a variety of materials, making it versatile for different industrial needs.
- **Rapid Production:** The process enables fast manufacturing, which is ideal for low-volume production runs and rapid prototyping, reducing lead times and accelerating product development cycles.
- **Design Flexibility for Complex Parts:** DMLS excels at fabricating parts with intricate features, including internal cavities, undercuts, and lattice structures that are typically unachievable through traditional manufacturing techniques.
- **Efficient Material Usage:** DMLS is a cost-effective and environmentally friendly approach, as it minimizes material waste. Unused powder can often be recycled and reused, further enhancing sustainability.

Overall, the method is effective and adaptable, providing material versatility, high precision, and the capacity to create complex geometries. As a result, it is a preferred option for a variety of applications across multiple industries[4][5].

1.8.1 Principal and Procedure of DMLS

Direct Metal Laser Sintering (DMLS) operates on the principle of using a high-powered laser to selectively melt and fuse metal powder, layer by layer, to form a fully solid component. The process begins with a three-dimensional digital model of the desired part. This model is then digitally sliced into thin horizontal layers, typically ranging in thickness from 20 to 50 microns, which guides the laser during the building process.

The procedure of DMLS is as follows:

- **Create a 3D graphical model of the component :** The creation of a 3D CAD model of the component that needs to be manufactured is the first step in the DMLS process.
- **Slice the graphical model:** Next, the 3D model is divided into thin layers that range in thickness from 20 to 50 microns. This generates a sequence of two-dimensional pictures that will be utilised to direct the laser while printing.
- **Get the machine ready**: After that, the DMLS printer is configured for printing. This process includes levelling the build platform, adjusting the laser, and loading the machine with the metal powder.
- **Printing the part:** Once the machine setup is complete, the printing process begins. A high-powered laser is used to selectively heat and melt the metal powder, causing the particles to fuse and solidify into the desired shape. Guided by a computer-controlled system based on a 3D digital model, the laser targets only the specific regions of the powder bed that need to be fused.

- **Post-processing:** The part might require post-processing once the printing process is complete. To improve the part's strength and durability, this might involve heat treating it, increasing the surface finish, and removing support structures.

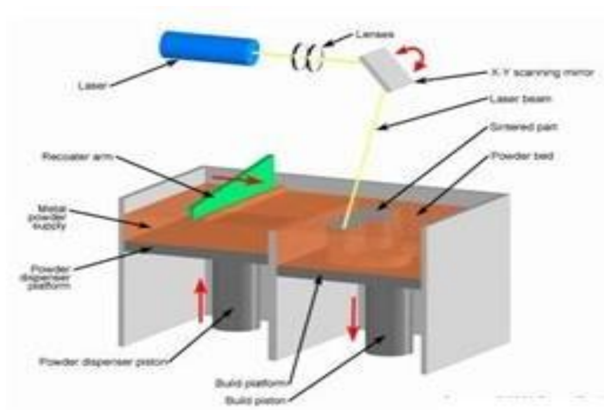


Figure 1.3 Direct Metal Laser Sintering (DMLS) Setup [6]

The benefit of DMLS is that it can produce parts with intricate internal structures and geometries that are hard or impossible to fabricate with conventional manufacturing techniques.

1.9 Hot Isostatic Pressing (HIP)

Hot Isostatic Pressing (HIP) is an advanced manufacturing and post-processing technique widely utilized for producing high-performance components across industries such as aerospace, nuclear, marine, medical, automotive, and defense. Originally developed in 1955 for diffusion bonding (DB) applications, HIP has since evolved to serve various purposes, including powder consolidation (PM HIP), densification of castings, compaction of pre-sintered parts, and more recently, as a post-processing method for additively manufactured (AM) parts.

1.9.1 Key Benefits of the HIP Process

- **Design Flexibility:** HIP allows for tailored mechanical properties by varying parameters such as temperature, pressure, and dwell time, enabling customized material behavior to suit specific applications.
- **Mechanical Property Enhancement:** The process produces fine-grained, isotropic microstructures, which contribute to improved strength, fracture toughness.
- **Material Efficiency and Cost Reduction:** The Near Net Shape (NNS) PM HIP approach reduces material waste and machining needs, thereby lowering the buy-to-fly ratio and accelerating production of parts with intricate shapes.

1.9.2 Mechanism of Densification in HIP

HIP promotes pore elimination through the reduction of surface energy, driven by a decrease in surface area. Densification occurs via three primary mechanisms:

- Yield collapse
- Creep deformation
- Atomic diffusion

These mechanisms are primarily controlled by the temperature, applied pressure, and holding time during the process. Additionally, factors such as the material of the encapsulating canister (for NNS HIP applications) and the heating and cooling rates also significantly affect the overall densification response.

1.9.3 Hot Isostatic Pressing (HIP) System

The Hot Isostatic Pressing (HIP) system is built around a sealed steel pressure vessel, which is closed at the top using a secure cover. Inside the vessel, the furnace is encased with thermal insulation, acting as a barrier to separate the hot zone from the water-cooling system used in the HIP unit. To achieve the required high-pressure environment, a compressor introduces inert gas—commonly argon, nitrogen, or helium—through a gas inlet positioned at the top of the vessel. A significant portion of this gas is recovered and reused in subsequent HIP cycles, enhancing efficiency and reducing operating costs.

One of the most vital components of a HIP system is its heating element, which is selected based on the desired temperature range and application requirements. These heating elements ensure uniform temperature distribution within the chamber, which is critical for effective densification and microstructural consistency during the HIP process.

1.10 Ultimate Tensile Test (UTS)

The Ultimate Tensile Strength (UTS) is one of the most critical mechanical properties used to evaluate the performance of structural materials. It represents the maximum stress a material can withstand while being stretched before failure. For Ti6Al4V alloy—particularly in its additively manufactured form—this test is essential for understanding how post-processing treatments like Hot Isostatic Pressing (HIP) and heat treatment influence its load-bearing capability. Tensile testing was carried out on samples oriented along the X, Y, and Z directions to evaluate the anisotropy in mechanical behavior. The experiments were performed using a Universal Testing Machine (UTM) under well-defined and consistent testing conditions. The primary parameters measured during the tests included:

- Yield strength
- Ultimate tensile strength (UTS)
- Elongation at break (percentage)
- Fracture characteristics

1.11 Heat Treatment of Ti6Al4V

Heat treatment is a thermal process used to alter the microstructure, phase composition, and mechanical properties of alloys. For Ti6Al4V, heat treatment is critical in transforming the as-built martensitic structure (α′) into more stable and ductile α+β phases.

1.11.1 Key Heat Treatment Parameters:

- **Temperatures used:** 850°C, 950°C, 1060°C, 1080°C
- **Holding time (soak time):** Varied from 30 minutes to multiple hours
- **Cooling methods:** Air cooling and water quenching

- **Heating rate:** ~3.5°C/min
- **Cooling rate:** ~2°C/min (except for quenching)

1.12 Corrosion Testing

Ti6Al4V is well-known for its natural resistance to corrosion, primarily due to the formation of a stable oxide layer (TiO_2). However, the corrosion performance of additively manufactured parts can be compromised due to porosity, microstructural inhomogeneity, and surface roughness. Therefore, evaluating corrosion behavior post-HIP and heat treatment is essential.

1.12.1 Methodology:

Corrosion tests were carried out using an electrochemical three-electrode setup involving:

- **Working Electrode:** Polished Ti6Al4V sample
- **Reference Electrode:** Saturated Calomel Electrode or Ag/AgCl
- **Counter Electrode:** Platinum wire
- **Electrolyte:** 0.9% NaCl solution

The test involved two key measurements:

1. **Open Circuit Potential (OCP):** To assess the passive behavior
2. **Potentiodynamic Polarization (PDP**): To evaluate corrosion current density and corrosion potential

1.13 Organization of the Thesis

There are 5 Chapters in this thesis.

Chapter 1 A thorough overview of the study is given in this chapter with a particular emphasis on Direct Metal Laser Sintering's (DMLS) current state of technology. It provides an explanation of DMLS's operation and literature on titanium alloys.

Chapter 2 delivers the research background, motivation, problem statement as well as objective and scope of research**.**

Chapter 3 The main goal of these tensile tests is to investigate ultimate tensile strength of Ti6Al4V, which is an additively manufactured material made by Direct Metal Laser Sintering, under the worst possible circumstances.

Chapter 4 explores in detail whether Hot Isostatic Pressing (HIP), which was used in the study, is a superior post-processing technique. The chapter also includes the results of tensile tests on Ti6Al4V that was produced using Direct Metal Laser Sintering.

Chapter 5 provides a thorough synopsis of the thesis, outlining the principal discoveries, outlining the major conclusions derived from the study and pointing out significant research gaps that need to be filled in the order to indicate potential directions for future study.

CHAPTER 2: LITERATURE SURVEY AND OBJECTIVES

Ti6Al4V (Grade 5) is recognized as one of the most widely used titanium alloys due to its remarkable combination of strength, toughness, and resistance to corrosion. Despite these advantages, its mechanical behavior is strongly influenced by the presence of interstitial elements such as hydrogen, oxygen, nitrogen, and carbon.

These impurities impact the alloy in different ways:

- Hydrogen, when present in small amounts within the titanium matrix, has little effect on strength or ductility. However, if it accumulates and forms hydride compounds, it can cause hydrogen embrittlement, leading to a significant loss in material toughness.
- Oxygen and nitrogen act as strengthening agents, but they simultaneously reduce impact resistance, with oxygen having the most pronounced strengthening effect.
- Carbon, on the other hand, has a relatively minor influence on both strength and toughness compared to the other interstitials.

Chemically, Ti6Al4V (Grade 5) is composed of about 90% titanium, 6% aluminum, and 4% vanadium. It features a dual-phase structure, consisting of:

- The α-phase, which has a hexagonal close-packed (HCP) crystal structure.
- The β-phase, which is body-centered cubic (BCC).

The distribution, volume fraction, and orientation of these phases significantly affect the alloy's mechanical and physical performance.

- Aluminum serves as an α-phase stabilizer, reducing density and increasing strength.
- Vanadium stabilizes the β-phase, improving toughness and ductility.

This unique blend of α and β phases allows Ti6Al4V to deliver a well-balanced set of mechanical properties, making it ideal for demanding engineering applications. With an average density of about 4.3 g/cm³, it offers both lightweight characteristics and high structural integrity, which is particularly valuable in sectors such as aerospace, biomedical implants, and automotive design. [1].

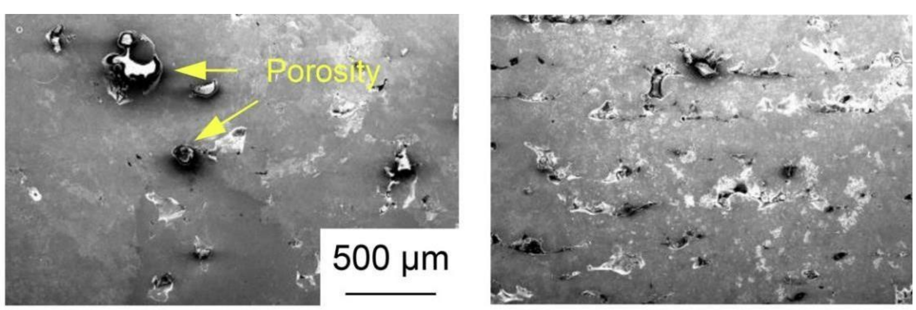


Fig. 2.1. FESEM images of DMLS Ti6Al4V specimen demonstrating porosity. [7]

Xingchen et.al.,[8] study examined The influence of Hot Isostatic Pressing (HIP) on microstructure and mechanical performance of lattice structures composed of selective laser melted (SLM) Ti6Al4V. The results of the study showed that HIP converts the original acicular α′ martensite into a combination of α and β lathes. Furthermore, the lattice structure's internal porosity was considerably decreased by HIP, going from 5.9% to 0.5%. As such, the samples treated with HIP demonstrated significant improvements in both fracture strain and compressive strength when compared to the as-fabricated (AF) samples. The enhanced resilience of the materials under compressive loads can be ascribed to their refined microstructure and reduced porosity, which are the results of HIP treatment. Eklund et.al.,[9] conducted the experiment to optimize HIP (Hot Isostatic Pressing) parameters for EBM (Electron Beam Melting) Ti-6Al-4V in order to enhance material strength. It was found that using a lower HIP temperature of 800 °C and a higher pressure of 200 MPa resulted in the highest strength while effectively eliminating internal defects. By deliberately introducing porosity during printing and applying an optimized HIP cycle, the material achieved its maximum strength.

Benzing et al. [10] addressed the issue of internal porosity in Ti-6Al-4V components produced through electron beam melting (EBM) powder bed fusion, where such defects are commonly found. While traditional Hot Isostatic Pressing (HIP) methods are effective in eliminating porosity, they can lead to grain coarsening, which compromises the material's strength. To overcome this, the authors introduced an improved HIP technique, which includes holding the material above the β-transus temperature, followed by rapid quenching and subsequent tempering. This modified cycle not only effectively eliminates internal voids but also causes a microstructural transformation—changing the grain structure from columnar to equiaxed, adjusting the α-lath aspect ratio, and improving overall microstructural uniformity. Remarkably, this approach preserves the yield strength and ultimate tensile strength comparable to that of the as-built material.

Chen et al. [8] focused on the porosity formed within Ti6Al4V components manufactured via cold spray additive techniques, emphasizing the detrimental effects these internal voids have on material performance. In this study, HIP treatment was applied to cold spray Ti6Al4V bulk samples to improve density, microstructural uniformity, and mechanical behavior. Using optical microscopy and X-ray computed tomography (XCT), the evolution of pore structures was analyzed. The 3D reconstructions from XCT revealed that HIP's combination of high pressure and elevated temperature facilitates diffusion-driven densification, resulting in a fully consolidated microstructure. Post-HIP analysis showed grain growth in previously deformed regions, with β-phase precipitates uniformly surrounding equiaxed α grains. Mechanical testing confirmed a notable increase in strength, attributed to enhanced metallurgical bonding and improved microstructural integrity. Overall, HIP significantly improved the density and mechanical performance of the cold-sprayed Ti6Al4V samples.

Balasubramaniam et al. [11] utilized Hot Isostatic Pressing (HIP) to manufacture integral turbine rotors from AISI 304 stainless steel and the nickel-based superalloy Inconel 718 for critical applications such as liquid propulsion systems in the Prithvi missile and cryogenic engines used in the Geostationary Satellite Launch Vehicle (GSLV). Before moving to full-scale rotor fabrication, initial research was conducted to understand the relationship between microstructure and mechanical properties in HIP-processed 304 stainless steel and Inconel 718. The materials treated with HIP achieved nearly full theoretical density and exhibited a uniform, fine-grained microstructure. Their mechanical performance satisfied the necessary standards for turbine rotor applications, thereby supporting the viability of producing near-net-shape, full-scale components using HIP technology.

Alegre et al. [12] described HIP as a highly effective thermomechanical post-processing method, commonly employed to improve the quality of additively manufactured (AM) parts by removing internal imperfections such as unfused areas and entrapped gas porosity, which otherwise reduce mechanical strength. The study focused on evaluating a non-standard HIP treatment cycle and its effect on the tensile performance of Ti-6Al-4V alloy made through Selective Laser Melting (SLM).

This experimental HIP process was performed at 850 °C and 200 MPa pressure, maintained for a duration of two hours. To minimize grain coarsening, which is detrimental to tensile strength, the cooling phase was deliberately accelerated beyond what traditional furnace cooling can achieve.

The study assessed three types of samples:

- As-built SLM specimens
- SLM specimens treated with the modified HIP cycle
- Wrought reference specimens, processed by rolling and annealing

A comprehensive set of tensile tests, along with microstructural and fractographic analysis, was conducted before and after HIP to study crack initiation behavior and its relation to tensile strength. The findings showed that the modified HIP cycle effectively improved material density, preserved microstructural integrity. These results validate the potential of tailored HIP cycles in significantly enhancing the durability and reliability of SLM-fabricated Ti-6Al-4V parts.

Masuo et.al.,[15] analyzed that the majority of the defects were caused by gas porosity and lack of fusion. The tensile strength of a Ti-6Al-4V produced by AM was examined in relation to surface roughness, the elimination of numerous pores, and the impact of the Hot Isostaic Pressing (HIP) process. Then, using the actual 3D mesh that was retrieved from the blade scrap, the rectangular block shape was used to better establish the model.

Du Plessis et al. [17] highlighted the significant role of Hot Isostatic Pressing (HIP) in enhancing material density and minimizing porosity in Ti6Al4V components produced through the Laser Powder Bed Fusion (LPBF) additive manufacturing processThey explain that the tensile strength of AM parts is increasingly linked to internal pores, which HIP can effectively close, making it essential for high-reliability components, particularly in aerospace. However, the study also notes challenges in completely sealing near-surface pores, which can persist or reappear during post-HIP heat treatment.

Using X-ray tomography, the authors evaluated pore closure efficiency across different pore types, such as lack of fusion, keyhole pores, contour defects, and even artificially induced voids. The imaging was done before and after HIP, allowing direct comparisons. While HIP demonstrated high closure efficiency for internal defects, near-surface pores were more problematic, occasionally penetrating the surface. In some cases, annealing after HIP led to the reopening of previously sealed pores and even a “blistering” effect—a phenomenon observed for the first time in this study.

Additionally, the research presents early evidence suggesting that HIP could potentially consolidate unmelted powder regions, which may open up avenues to increase build rates in LPBF by relaxing melting requirements in select areas.

Bin Zhou et.al.,[18] used selective laser melting, Ti6Al4V components with intricate structures, superior functionality, and high surface quality can be produced (SLM). To prevent oxidation at high temperatures, the traditional SLM process is performed in a chamber filled with an inert gas at a pressure slightly higher than the surrounding air. However, the as-fabricated parts may develop pores due to the inert gas. In this work, the SLM procedure was performed in vacuum to enhance the quality of the Ti6Al4V samples that were SLM-fabricated. Following fabrication, the Ti6Al4V samples were heated to isostatic pressure (HIP). X-ray computed tomography (CT) was used to assess the remaining porosity. Following fabrication, the Ti6Al4V samples were heated to isostatic pressure (HIP). X-ray computed tomography (CT) was used to assess the remaining porosity. The samples' mechanical characteristics and microstructures were assessed both with and without HIP. The test results demonstrated that, in comparison to material produced using the traditional SLM process, SLM under vacuum could reduce the porosity of Ti6Al4V samples. Porosity could be further decreased and better elongation could be achieved by the Ti6Al4V samples after HIP.

2.1 **Problem Statement**

This study focuses on analyzing the mechanical properties, microstructural evolution, and deformation behavior of the Ti-6Al-4V alloy under different manufacturing techniques and processing conditions. A key objective is to understand the influence of interstitial impurities—including oxygen, nitrogen, carbon, and hydrogen—on the alloy's strength, ductility, and resistance to impact.

Additionally, the research aims to assess how various advanced manufacturing methods, such as Selective Laser Melting (SLM), Electron Beam Melting (EBM), and other additive manufacturing (AM) techniques, along with post-processing treatments like heat treatment, affect the alloy's mechanical performance, physical characteristics, and microstructure.

The ultimate goal is to provide insights into optimizing processing parameters, such as laser energy input and heat treatment temperatures, to enhance the material's performance for specific engineering applications. Furthermore, the study seeks to deepen the understanding of Ti-6Al-4V's fracture behavior, response to deformation, and mechanical response under different strain rates, loading directions, and thermal conditions.

2.2 Main Aim of the Study

The primary aim of the study is to show effectiveness of Hot Isostatic Pressing and Heat Treatment on mechanical property of Ti6Al4V fabricated via DMLS manufacturing process.

2.2.1 Objectives of the Study:

- To study the microstructure of HIP treated Ti6Al4V after heat treatment.

- Evaluate the tensile stress of HIP treated Ti6Al4V after Heat Treatment.
- Evaluate the tensile stress of Ti6Al4V in X, Y and Z build direction.
- To investigate the tribological behaviour of before and after HIP treated Ti6Al4V.
- Comparatively study on the corrosion rate of before and after HIP treated Ti6Al4V

CHAPTER 3: MATERIAL AND METHODOLOGY

3.1 Introduction

This study focuses on evaluating the mechanical, tribological, and corrosion behavior of Ti6Al4V alloy manufactured using Direct Metal Laser Sintering (DMLS), followed by Hot Isostatic Pressing (HIP) and various heat treatment protocols. A systematic experimental methodology was adopted to fabricate, process, and characterize the material.

3.2 Material

Ti6Al4V, one of the most versatile and widely accessible alloys on the market, was the material used in the study. Table (2) displays the chemical composition of Ti6Al4V. Vanadium (V), Aluminium (Al), Nickel (Ni), Tin (Sn), Zirconium (Zr), Carbon (C), Molybdenum (Mo), Silicon (Si), Chromium (Cr), Iron (Fe), Copper (Cu), Niobium (Nb), and Titanium (Ti) are the elements that make up Ti6Al4V, according to this. Ti6Al4V specimens were produced using a DMLS system (EOS M290). The powder used conformed to the composition specified for Ti6Al4V. Key process parameters included:

- Laser power: 340 W
- Scanning speed: 1400 mm/s
- Spot diameter: 140 µm
- Layer thickness: 30 µm
- Shielding gas: Argon

After fabrication, the samples were cleaned and prepared for post-processing.

Table 3.1: Chemical composition of Ti6Al4V.

Chemical Compounds	Percentage (By wt.)
V	4.26
Al	5.43
Sn	0.0621
Zr	0.0026
Mo	0.004
C	0.366
Si	0.0220
Cr	0.0097
Ni	<0.0012
Fe	0.114
Cu	<0.04
Nb	0.0388
Ti	balance

3.3 DMLS Sample Preparation

The specimens were fabricated using a Direct Metal Laser Sintering (DMLS) machine at IIT Delhi. The processing parameters used during fabrication are summarized in Table 3 below. Among the various parameters, laser power, scanning speed, and layer thickness are the most critical factors influencing the quality of the built components.

Table 3.2: Process Parameter of DMLS

Building Parameter	**Values**
Laser Power (W)	145
Spot Size(μm)	140
Scanning Speed(mm/s)	1000
Layer Thickness (μm)	30
Hatching distance (mm)	0.082
Wavelength -nm	1060

3.4 Hot Isostatic Pressing (HIP)

To minimize internal porosity and enhance microstructural uniformity, the as-built samples underwent HIP treatment at three different temperatures:

- 700 ℃
- 910 ℃
- 1060 ℃

  Each HIP cycle was conducted at 200 MPa for a duration of 2 hours in an inert atmosphere. This process densifies the material and reduces internal voids.

A manufacturing technique called hot isostatic pressing (HIP) applies high pressure and temperature simultaneously. Since its invention in 1955, it has been widely used in various fields, initially for diffusion-bonding applications in the nuclear industry. An overview of hot isostatic pressing is given in this paper, which also examines the temperature and pressure cycles, as well as the tools employed. The use of HIP in ceramics, powder metallurgy, diffusion bonding, and casting healing is also taken into consideration. Lastly, a discussion of the most recent developments in technology, research, and application.

By simultaneously applying isostatic pressure and a high temperature to a workpiece, a process known as hot isostatic pressing (HIP) causes the workpiece—which is typically a powder—to solidify. An inert gas, such as nitrogen or argon, is pumped into a pressure vessel to serve as the pressure medium and up to 200 MPa of pressure, while a furnace inside the vessel reaches temperatures of up to 2000 degrees Celsius. Typically, the workpiece is enclosed in an evacuated glass, ceramic, or sheet metal capsule. It is important to exercise caution when designing the capsule and filling it to prevent distortion from compression. For castings, the workpiece's surface acts as a self-contained capsule.

HIP process is a process in which Temperature and Pressure both are applied simultaneously. The HIP machine used in the research is Engineered Pressing System International (EPSI) at DMRL,Hyderabad.

3.4.1 Hot Isostatic Pressing (HIP) Procedure

The Hot Isostatic Pressing (HIP) process followed in this study involved a series of controlled steps to ensure the effective consolidation and densification of the Ti6Al4V alloy:

- **Initial Vacuum Cycling:** To prepare the furnace for HIP, a vacuum cycle was performed three times. This involved alternately introducing argon gas into the pressure chamber and then evacuating it using a vacuum pump. This step is essential to eliminate atmospheric contaminants and ensure an inert processing environment.
- **Pressurization:** Once the vacuum cycle was complete, the internal pressure of the chamber was gradually increased to approximately 350 MPa using high-purity argon gas.
- **Controlled Heating:** After achieving the desired pressure, the furnace began heating at a controlled rate of 10°C per minute, starting from room temperature (approximately 28°C) up to the target HIP temperature specific to each test condition.
- **Dwell Period:** Upon reaching the specified HIP temperature, both temperature and pressure were maintained steadily for a predetermined duration (known as the dwell time). This is the critical stage for diffusion and microstructural homogenization.

- **Cooling Phase:** Once the dwell period ended, the system was allowed to cool. The cooling rate varied naturally based on the temperature gradient between the furnace interior and the external environment.

  The complete HIP cycle—from initial vacuum creation through heating, holding, and cooling—required over 48 hours to return the samples to room temperature under controlled conditions.

3.4.2 HIP Equipment Components

A typical HIP system consists of several integrated subsystems, each playing a crucial role in ensuring precise control of the pressurization and heating process.

- **Pressure Vessel**

The pressure vessel is the core of the HIP system and must be designed to handle extreme stress over long operational periods. Key considerations include its wear resistance, ability to withstand creep, and compliance with allowable stress limits over the equipment's lifetime. Pre-stressed wire-wound vessels with non-threaded end closures and an external yoke system.

- **Furnace System**

The furnace maintains a stable and controlled high-temperature zone during HIP. Heat is supplied by either convection or radiation, with radiation becoming more dominant at elevated temperatures. A thermal shield is used to protect the pressure vessel walls from excessive heat

.

- **Gas Handling System**

The gas system provides and maintains the pressurizing medium inside the chamber. While gases like helium and nitrogen can be used, argon is the most commonly preferred due to its inertness and availability. For ceramic materials, oxygen-argon mixtures may be employed. The system must be capable of delivering high-purity gas at pressures typically ranging from 100 to 400 MPa.

- **Control System**

The control unit manages and synchronizes all components of the HIP system. It can range from basic relay-based circuits to advanced programmable logic controllers (PLCs) or microprocessor-based systems. At basic levels, operators initiate each step manually, whereas high-level systems offer full automation, including real-time control, data logging, maintenance alerts, error diagnostics, and process optimization.

- **Auxiliary Systems**

These systems support the main HIP operation by providing essential functions such as cooling systems, safety interlocks, temperature and pressure sensors, and emergency shutdown mechanisms. They ensure smooth and reliable operation of the overall HIP unit.

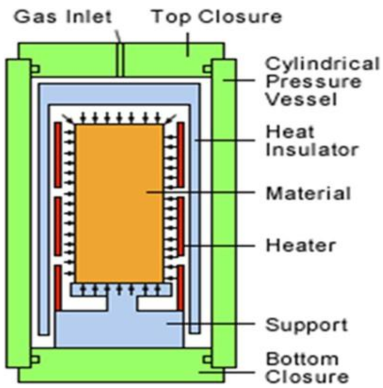


Fig.3.1 Hot Isostatic Pressing

3.5 Heat Treatment

Post-HIP samples were subjected to multiple heat treatment protocols to investigate their effect on mechanical performance. The variables included:

- Treatment temperature (e.g., 950 °C, 1060 °C, 1080 °C)
- Number of cycles
- Soaking time
- Cooling method (air cooling or water quenching)

The aim was to induce desirable microstructural transformations, such as bi-modal and lamellar structures.

Heat treatment is a post-processing method used to enhance the mechanical properties and microstructural characteristics of Ti6Al4V alloy

Fig.3.2 Tubular Furnace

3.5.1 Purpose of Heat Treatment

Heat treatment is an essential post-processing step aimed at enhancing the mechanical, tribological, and microstructural properties of Ti6Al4V components fabricated via Direct Metal Laser Sintering (DMLS). In this study, heat treatment was conducted after Hot Isostatic Pressing (HIP) to further optimize the material's ductility, strength, while promoting phase transformation and stress relief

3.5.2 Objectives of Heat Treatment

The key goals of applying various heat treatment cycles were to:

- Improve tensile strength.
- Refine the microstructure, specifically to attain bi-lamellar or equiaxed grain structures
- Enhance homogeneity and reduce residual stress

- Tailor hardness and ductility balance for application-specific performance

3.5.3 Heat Treatment Parameters

The heat treatment conditions applied to the HIP-treated Ti6Al4V samples varied in terms of:

- **Soaking temperature:** 850 °C, 950 °C, 1060 °C, and 1080 °C
- **Number of cycles:** Single to multiple repeated cycles (up to 8)
- **Soaking time:** Maintained depending on the selected cycle
- **Cooling method:** Either air-cooled or water-quenched
- **Heating rate:** 3.5 °C per minute
- **Cooling rate:** 2 °C per minute (except during water quenching)

3.5.4 Heat Treatment Cycles Used

- **Repeated Cycle Treatment (at 910 °C for 8 Cycles):**

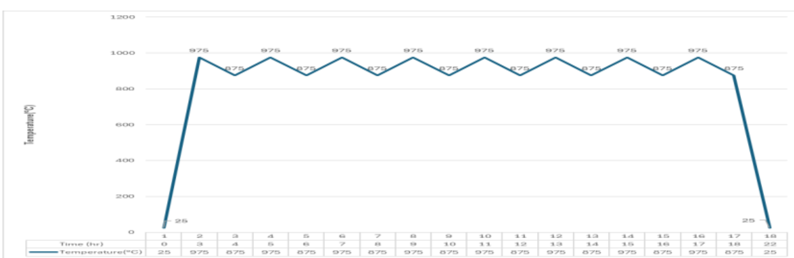


Fig. 3.3 Heat cycle for repeated heat treatment of Ti6Al4V

This multi-cycle treatment aimed to stabilize the microstructure and promote grain refinement. It facilitated the development of a more ductile and uniform structure through controlled cyclic thermal exposure.

- **Soak-Time-Based Treatment (at 910 °C):**

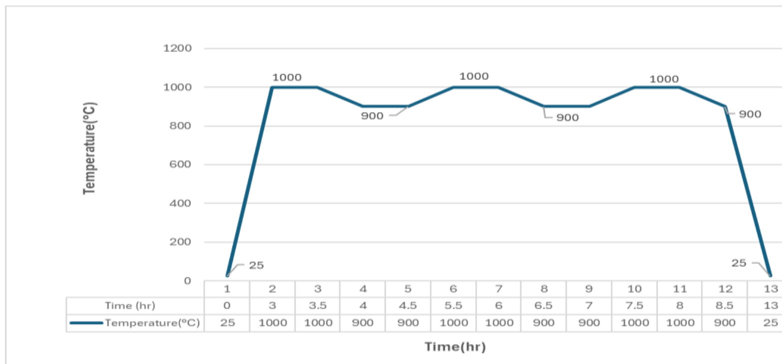


Fig.3.4 Heat cycle for soak time heat treatment of Ti6Al4V

Samples were held at 910 °C for an extended duration to encourage phase transformation and stress relaxation.

- **Heat Treatment at 950 °C:**

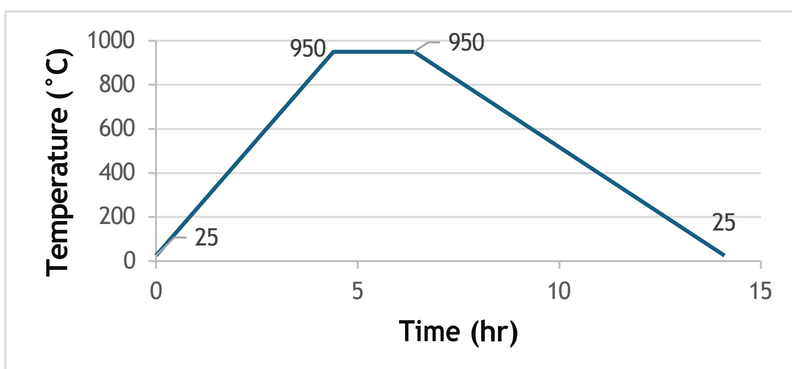


Fig.3.5 Heat cycle for heat treatment at 950°C of Ti6Al4V

A single-cycle treatment targeting bi-modal microstructure formation by coarsening the alpha phase and refining retained beta. Enhanced tensile strength and ductility were observed following this treatment.

- **High-Temperature Heat Treatment at 1060 °C (2 Cycles):** Applied to explore grain growth and its effect on mechanical behavior.

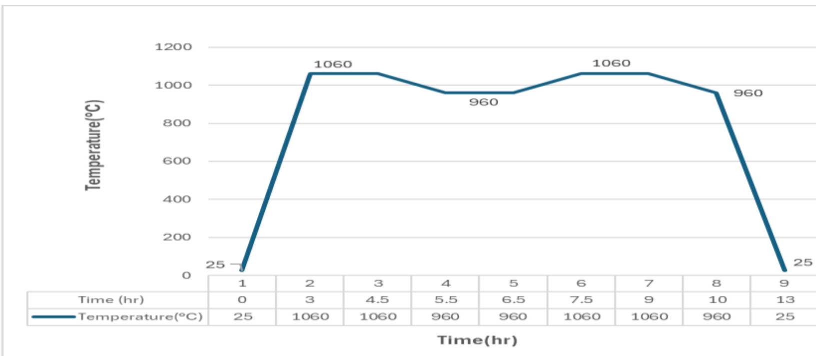


Fig.3.6 Heat cycle for 2 cycle heat treatment of Ti6Al4V

- **Heat Treatment at 1080 °C (3 Cycles):** Aimed at achieving deep recrystallization and potential lamellar transformation, with the goal of balancing high strength and moderate ductility.

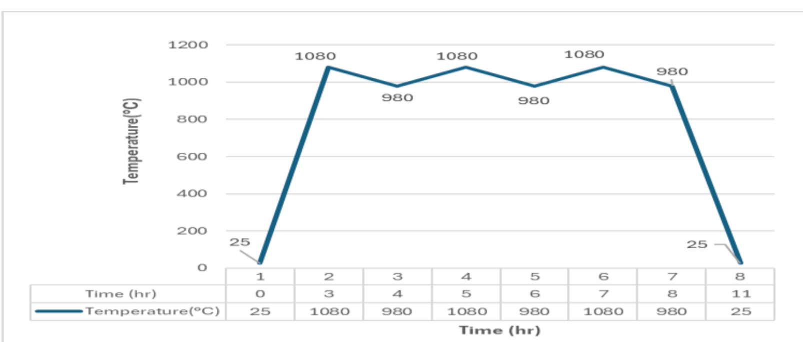


Fig.3.7 Heat cycle for 3 cycle heat treatment of Ti6Al4V

- **Water Quenching at 850 °C:** Rapid quenching was performed to suppress diffusion-controlled phase formation and retain metastable phases such as martensite, which resulted in elevated hardness values

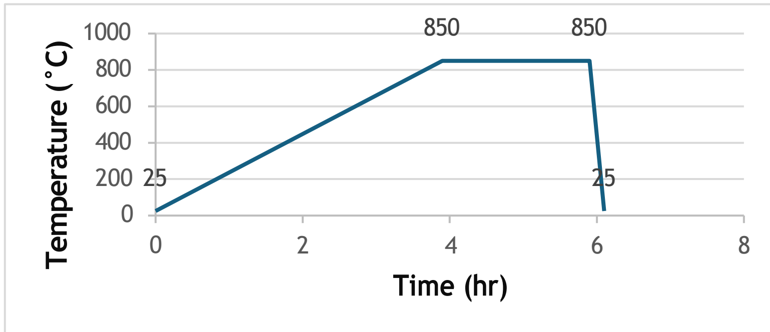


Fig.3.8: Heat cycle for heat treatment at 850°C of Ti6Al4V

3.5.5. Major Components of a Tubular Furnace

- **Heating Tube (Quartz or Alumina):** The central chamber where samples are placed. Made of heat-resistant materials like quartz, alumina, or ceramics to withstand high temperatures.

- **Heating Elements:** Common types include Kanthal (Fe-Cr-Al), Molybdenum, or Graphite, depending on the desired temperature. Embedded around the tube to ensure even temperature distribution up to ~1600 °C or more.

- **Temperature Controller and Thermocouple:** Maintains precise thermal control using PID (Proportional-Integral-Derivative) logic. Thermocouples (usually Type K or R) are inserted near the heating zone to monitor and regulate temperature in real-time.

- **Gas Inlet and Outlet Ports:** Allow controlled flow of protective or reactive gases such as argon or nitrogen. Designed to ensure gas purging and uniform distribution inside the tube.
- **Sample Holder (Boat or Tray):** Made of **heat-resistant ceramics** or **refractory metals** (e.g., Mo or Ta), it holds the specimens during heat treatment.

3.5.6 Working Principle of the Tubular Furnace Setup

- **Sample Placement:** The HIP-processed Ti6Al4V samples are placed in the centre of the heating tube using a ceramic holder.
- **Sealing and Purging:** The furnace ends are sealed using high-temperature resistant flanges. Before heating, the chamber is purged with argon gas to remove residual air and moisture.
- **Heating Cycle:** The furnace is programmed to follow a controlled heating rate (e.g., 3.5 °C/min) until it reaches the target heat treatment temperature (e.g., 950 °C, 1060 °C).
- **Soaking:** Once the desired temperature is achieved, the sample is held at that temperature for a fixed dwell time to facilitate microstructural changes such as grain growth, phase transformation, or stress relief.
- **Cooling:** After the soak period, the furnace is allowed to cool at a regulated rate (e.g., 2 °C/min), either in the argon atmosphere or b y rapid quenching (e.g., water quenching) to retain desired phases.

3.6 Hardness Testing

Microhardness was measured using a Vickers hardness tester with a 2 N load and 20-second dwell time. Surface indentations were taken to assess the hardness variation across different post-processed samples.

Load: 2N

Dwell time: 20 sec

Fig:3.9 computer attached automated Vickers hardness machine

3.7 Tensile Testing

Tensile tests were conducted in the X, Y, and Z build directions to study anisotropic behavior. The ultimate tensile strength, yield strength, and elongation were evaluated using a universal testing machine (UTM).
This is the most basic amplitude sequence that can be produced by repeatedly applying constant-amplitude stress reversals to the test piece until failure.

Tensile testing is a fundamental mechanical test method used to determine how materials behave under axial loading. It provides critical insights into properties such as yield strength, ultimate tensile strength

(UTS), ductility, and Young’s modulus. These properties are essential for evaluating a material’s suitability for load-bearing applications.

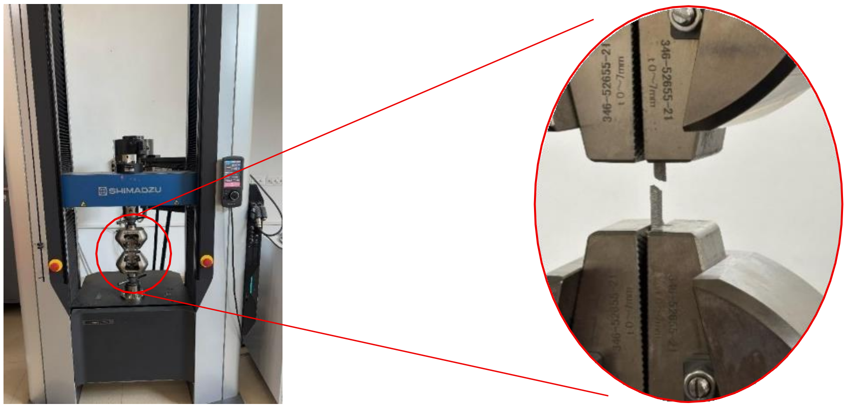


Fig.3.10 UTM with sample

In this study, tensile tests were conducted on Ti6Al4V specimens fabricated using the Direct Metal Laser Sintering (DMLS) process. The specimens were subjected to different post-processing treatments, including Hot Isostatic Pressing (HIP) and heat treatment, to investigate their influence on tensile behavior. Given the anisotropic nature of additively manufactured parts, samples were tested in three orientations: X, Y, and Z.

3.7.1 Objectives of the Tensile Test

- To evaluate the effect of HIP and heat treatment on the tensile properties of DMLS-fabricated Ti6Al4V.
- To compare the tensile strength and ductility of samples in different build orientations (x, y, z).
- To study the correlation between microstructural changes and mechanical performance.

- To determine whether post-processing enhances isotropy and reduces build-direction-dependent variability.

3.7.2 Test Standards and Equipment

The tensile testing was performed in accordance with the ASTM E8/E8M and ASTM E606 standards. The testing setup included:

- **Universal Testing Machine (UTM)**: Computer-controlled with digital data acquisition.
- **Load Cell Capacity:** 100 kN
- **Strain Measurement:** Extensometer or crosshead displacement (depending on availability)
- **Test Environment:** Room temperature under quasi-static loading conditions
- **Sample Geometry:** Cylindrical and flat specimens with gauge length and cross-section as per ASTM specifications

3.7.3 Tensile Testing Machine Components

A tensile testing machine, also known as a Universal Testing Machine (UTM), is used to apply controlled tension to a test specimen to evaluate its mechanical properties, such as yield strength, ultimate tensile strength (UTS), and elongation. The following are the key components of a standard tensile testing setup:

Load Frame The rigid main body of the machine.
Consists of two vertical columns and a base.

Designed to withstand high forces without deformation.

Supports the crosshead movement and the mounted grips.

Crosshead A movable platform that travels vertically along the load frame.

Moves upward or downward to apply tensile or compressive load.

Its speed is controlled to maintain a constant strain rate.

Contains sensors and actuators for precise movement**.**

Grips (Specimen Holders) Securely hold the ends of the test specimen.

Must ensure axial alignment and no slippage during the test.

Load Cell A sensor that measures the applied force (load) on the specimen.

Converts mechanical force into an electrical signal.

Calibrated to provide accurate and real-time load readings.

Extensometer (or Strain Measuring Device) Measures the elongation of the gauge length of the specimen during testing.

Can be mechanical, optical, or laser-based.

Provides accurate strain data needed to calculate Young's modulus and yield strength.

Base Plate or Table Provides support and stability to the machine.

Often vibration-isolated to improve test precision**.**

3.7.4 Experimental Procedure

- **Mounting:** The specimen was securely mounted on the UTM grips.
- **Alignment:** Care was taken to align the specimen to avoid bending stress.
- **Loading:** A uniaxial tensile load was applied at a constant strain rate until failure.
- **Data Collection:** Stress-strain data were recorded in real-time, capturing key parameters:
    - Yield strength (0.2% offset method)
    - Ultimate tensile strength (UTS)
    - Fracture point
    - Percentage elongation
    - Reduction in area (if measured post-fracture)

3.8 Tribological Testing

Wear behavior was studied using a ball-on-disk setup under different normal loads (10 N, 20 N, 30 N). The wear rate and coefficient of friction (COF) were determined to evaluate surface durability and lubrication characteristics.

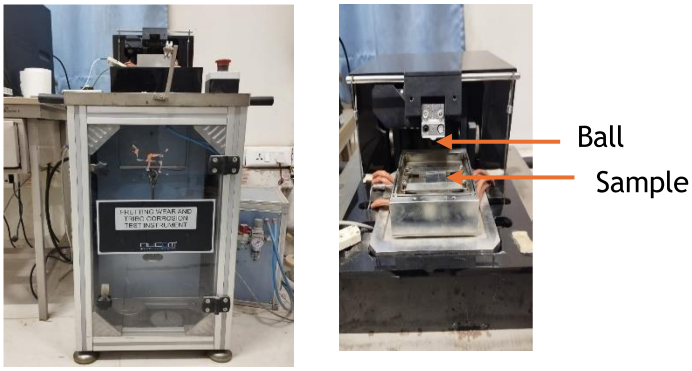


Fig.3.11 Ball and Disk Sliding Wear

Table:3.3 wear test parameter

S.No	Parameters	Value
1	Load	10, 20, 30N
2	Time Duration	20 min
3	Frequency	10Hz
4	Stroke Length	0.5mm

Surface roughness plays an important role in tribology test. It affects the coefficient of friction wear mechanism and wear volume. The relation can be made as higher the roughness, increase in the adhesion and therefore increase in the localized welding, leading to increase in rate of shearing which will ultimately lead to more wear. Adhesion can also be increased by an increase in the load which will increase the micro-welding. As normal load increases the coefficient of friction may decrease but the wear volume will definitely increase. Temperature also plays an important role in wear test. The temperature affects the mechanical properties of the counter bodies, surface film formation and properties of the lubrication used during the test.

3.8.1 Objectives of the Sliding Wear Test

- To evaluate and compare the wear resistance of as-built, HIP-treated, and heat-treated Ti6Al4V specimens.
- To investigate the effect of post-processing on surface degradation under sliding contact.
- To measure the coefficient of friction (COF) and wear rate under different applied loads.

3.8.2 Wear Testing Equipment

The sliding wear tests were conducted using a Ball-on-Disk wear testing machine, which operates in a dry sliding environment. In this setup:

- A hardened steel or ceramic ball (usually 6 mm in diameter) is used as the counterface.
- The test sample, in the form of a circular or rectangular disk, is mounted on a rotating platform.
- A constant normal load is applied vertically on the ball, which slides over the sample surface in a circular path.

3.9 Corrosion Testing

Corrosion resistance is a critical property for materials intended for biomedical, marine, and aerospace applications. Titanium alloys, including Ti6Al4V, are generally known for their excellent corrosion behavior due to the formation of a stable oxide film (TiO_2) on the surface. However, the corrosion performance can be significantly influenced by the manufacturing process and post-processing treatments such as Hot Isostatic Pressing (HIP) and heat treatment.

Electrochemical corrosion behavior was tested using a three-electrode setup in a simulated electrolyte environment. Potentiodynamic polarization (PDP) and Open Circuit Potential (OCP) techniques were used to determine corrosion potential (Ecorr), corrosion current ensity (icorr), and corrosion rate (CR).

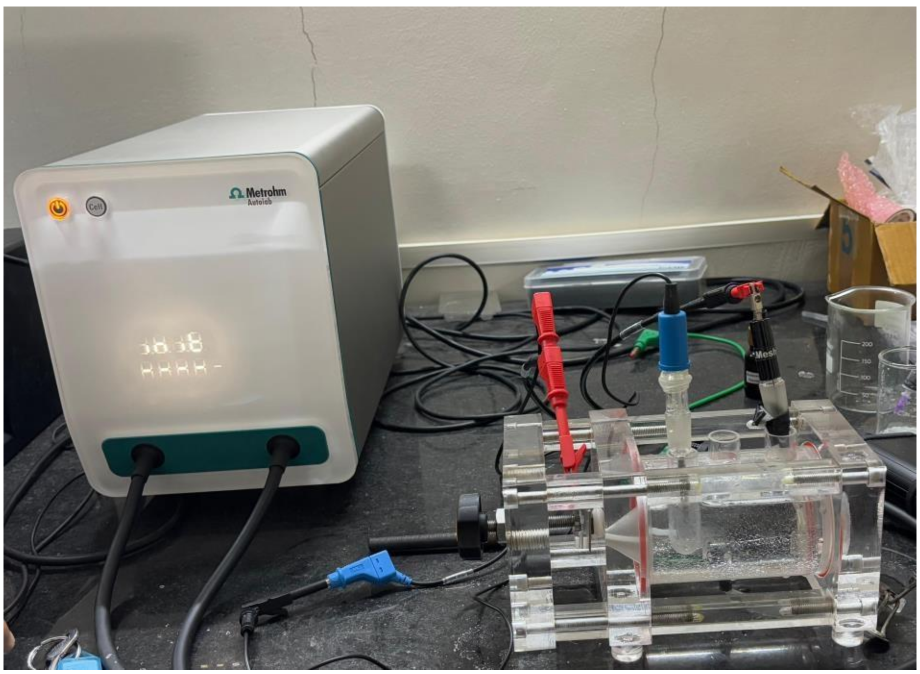


Fig.3.12: Electrochemical experimental setup consisting of three electrodes corrosion cells, electrolyte and monitor.

3.9.1 Objective of the Corrosion Test

- To assess and compare the corrosion resistance of as-built and post-processed Ti6Al4V alloy.

- To determine the influence of HIP and heat treatment on the formation and stability of passive oxide films.
- To obtain key corrosion parameters such as corrosion potential (Ecorr), corrosion current density (icorr), and corrosion rate.

3.9.2 Experimental Setup

The corrosion tests were performed using a three-electrode electrochemical cell, which included:

- **Working Electrode (WE):** The sample of Ti6Al4V alloy (polished and cleaned) and Exposed area: 1 cm^2.
- **Reference Electrode (RE)** A Saturated Calomel Electrode (SCE) or Ag/AgCl electrode was used to maintain a constant reference potential.
- **Counter Electrode (CE):** A platinum wire or graphite rod that completes the circuit and carries the current.
- **Electrolyte Solution:** A simulated environment, typically NaCl solution (e.g., 3.5%) or Phosphate Buffered Saline (PBS) to mimic body fluids or marine conditions.
- **Potentiostat :** An electrochemical workstation that controls the potential and records the current response.
- **Computer Interface:** Software to control test parameters, capture real-time data, and plot Tafel or OCP curves.

Table 3.3 corrosion test parameters

Area (cm^2)	**1**
Potential (V)	-0.5 to 1.2
Scan Rate (mV/s)	0.5

3.9.3 Key Parameters and Calculations

The corrosion rate (CR) was calculated using the formula:

$$CR = K \times \frac{EW \times i_{corr}}{d \times A}$$

Where:

K = corrosion constant (0.00327 mm/year

I corr = corrosion current density (μA/cm^2)

EW = equivalent weight of the alloy

ρ = density of Ti6Al4V (~4.43 g/cm^3)

A = exposed area (cm^2)

Chapter 4: Result and Discussion

This chapter outlines the findings from a series of mechanical, tribological, and corrosion evaluations carried out on Ti6Al4V alloy samples produced using the Direct Metal Laser Sintering (DMLS) technique. The specimens underwent Hot Isostatic Pressing (HIP) followed by varied heat treatment conditions. Key aspects such as mechanical strength, wear performance, microstructural features, and corrosion resistance were comprehensively analyzed to determine the effects of the applied post-processing methods

4.1 Effect of HIP on Microstructure

The microstructure of Ti6Al4V alloy showed considerable variation between the as-built (AB) condition and samples treated with Hot Isostatic Pressing (HIP) at 700 °C (below β-transus) and 1060 °C (above β-transus followed by tempering). In the as-built condition, the structure primarily consisted of martensitic α′ phase, resulting from the rapid cooling rates characteristic of the DMLS process.

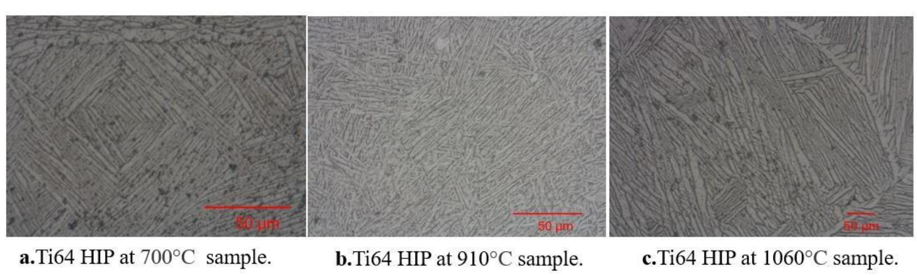


Fig:4.1: (a) Ti64 at 700 °C (b) Ti64 at 910°C (c) Ti64 at 1060°C

A key enhancement observed after HIP at both sub- and super-β transus temperatures was the removal of large spherical pores, which were prominent in the as-built specimens. These internal voids, known to weaken mechanical performance, were effectively eliminated, thereby significantly improving the material's structural reliability.

Additionally, grain structure was affected by the processing temperature. In both the as-built and sub-β HIP conditions, the prior-β grains appeared elongated, aligned with the build direction. This grain elongation contributes to anisotropic mechanical properties, resulting in directional variations in attributes such as strength and ductility.

- In contrast, the super-βtr + temper HIP treatment resulted in equiaxed prior-β grains, which are more favorable for achieving isotropic mechanical performance.

Microstructurally, the AB and sub-βtr HIP samples displayed basket-weave patterns, characterized by dark laths (α-phase) and bright inter-lath regions representing retained or transformed β. Additionally, bright intralath features were visible, likely representing fine precipitates or secondary phase elements.

In terms of α lath thickness:

- The as-built condition exhibited a fine lath structure with an average α thickness of 1.20 μm ± 0.31 μm.
- Following sub-βtr HIP treatment, significant coarsening was observed, with α lath thickness increasing to 2.21 μm ± 0.45 μm.
- After super-βtr + temper HIP treatment, the lath thickness was measured at 1.23 μm ± 0.67 μm, showing only a slight increase compared to the as-built state.

4.1 **Effect of HIP on Hardness**

The HIP has no significant difference on hardness of HIPed samples as compared to AB form. The hardness of AB samples is 384±20HV (0.2kg,10sec) and the hardness of HIPed sample at 1060°C is nearly same. The HIPed sample at 1060°C is 381±23HV (0.2kg,10sec) nearly shame to AB sample. But the sample HIPed at 700°C is 365±13HV (0.2kg,10sec).

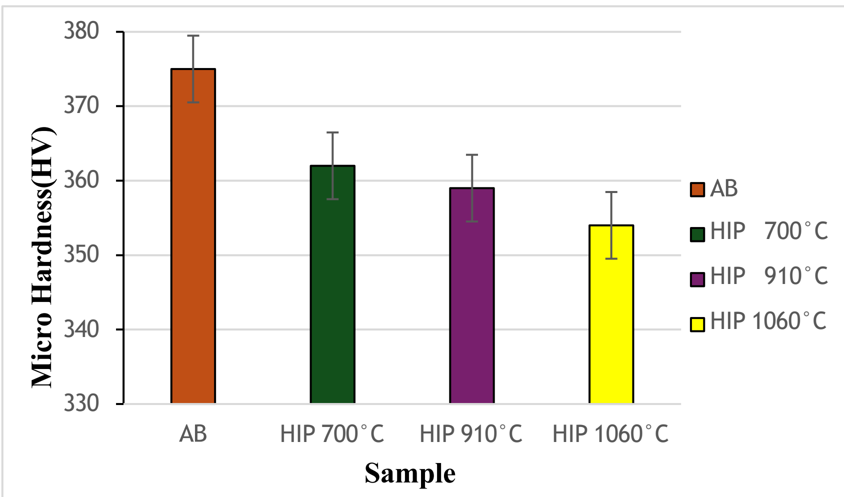


Fig:4.2: Micro Hardness of Ti6Al4V before and after post processing

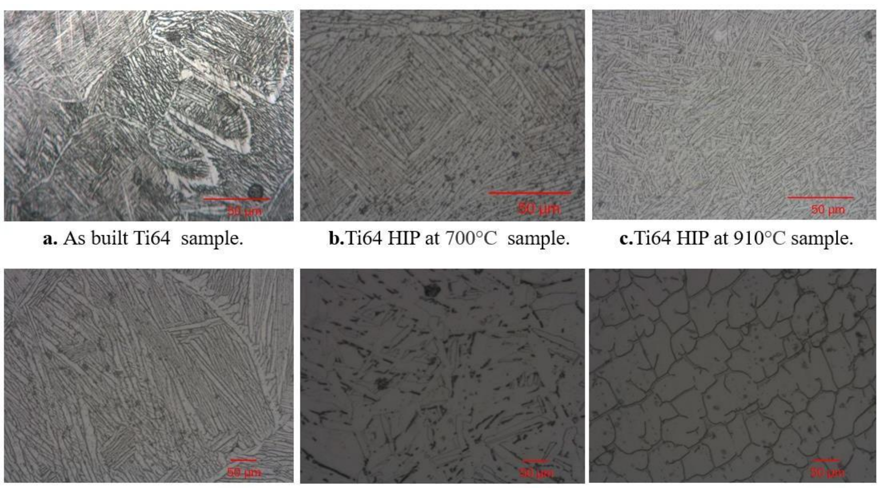
Surface hardness was measured using a Vickers microhardness tester under a 2 N load for 20 seconds.

- As-built samples exhibited the highest surface hardness (~373 HV) due to the presence of metastable martensitic α′.
- HIP at 700 °C and 910 °C slightly reduced hardness (~358 HV and ~354 HV respectively) due to the transformation of α′ into equilibrium α+β phases.

The as-built and HIPed samples differed in micro-hardness by less than 5%. It has previously been reported that the As-Built materials have fine microstructures, whereas the HIPed Ti6Al4V microstructures coarsen after HIP, resulting in decreased material static strength.

4.3 Microstructural Evolution

Optical and scanning electron microscopy (SEM) revealed significant microstructural changes between the as-built and post-processed Ti6Al4V samples.



a. As built Ti64 sample. **b.**Ti64 HIP at 700°C sample. **c.**Ti64 HIP at 910°C sample.

d.Ti64 HIP at 1060°C sample. **e.** Ti64 HIP 700°C post H T at 950°C **f.**Ti64 HIP 1060°C post HT at 950 °C

Fig :4.3 Ti6Al4V microstructures before and after post processing

- As-built specimens displayed a predominantly columnar microstructure with martensitic α′ phase due to rapid cooling during DMLS fabrication.

- HIP-treated samples showed a more uniform, densified structure with reduced porosity. Post-HIP heat treatments transformed the microstructure into bi-modal or lamellar α+β phases, depending on the temperature and holding time.
- Heat treatment at 950 °C and above promoted grain coarsening and phase redistribution, resulting in enhanced ductility.

4.4 Tensile Test Results

Tensile tests were performed on samples in X, Y, and Z build orientations, both in as-built and post-processed conditions.

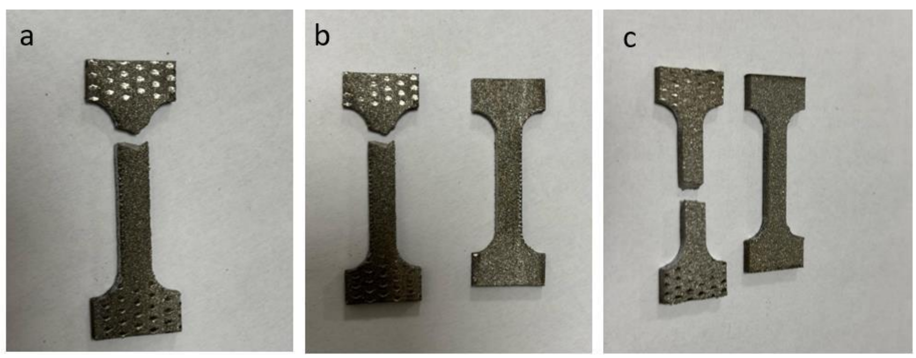


Fig.4.4: (a) x direction (b) y direction (c) z direction

- As-built samples showed directional dependence, with higher strength in X and Y directions (~1200–1220 MPa UTS) and lower values in Z direction (~1150 MPa) due to the layer-wise build structure.
- HIP at 910 °C enhanced tensile strength and elongation across all orientations due to densification and microstructural refinement.

- The combination of HIP + heat treatment at 1060 °C resulted in the best balance of strength (~1100 MPa) and elongation (~14–16%), attributed to the formation of a stable bi-modal structure.
- Ductility was significantly improved in post-processed samples, confirming successful stress relief and phase transformation.

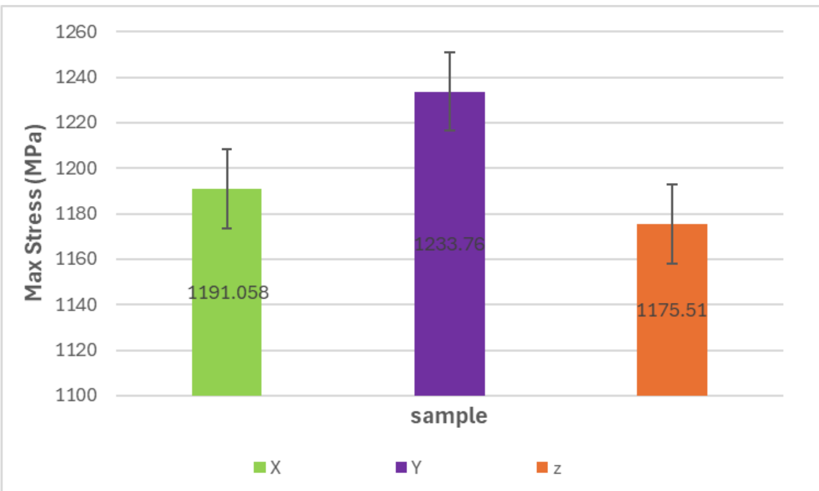


Fig.4.5: Ultimate tensile strength of Ti6Al4V before and after post processing

4.5 Wear Test and Tribological Performance

Wear resistance was assessed using a ball-on-disk sliding wear test under varying loads (10 N, 20 N, and 30 N).

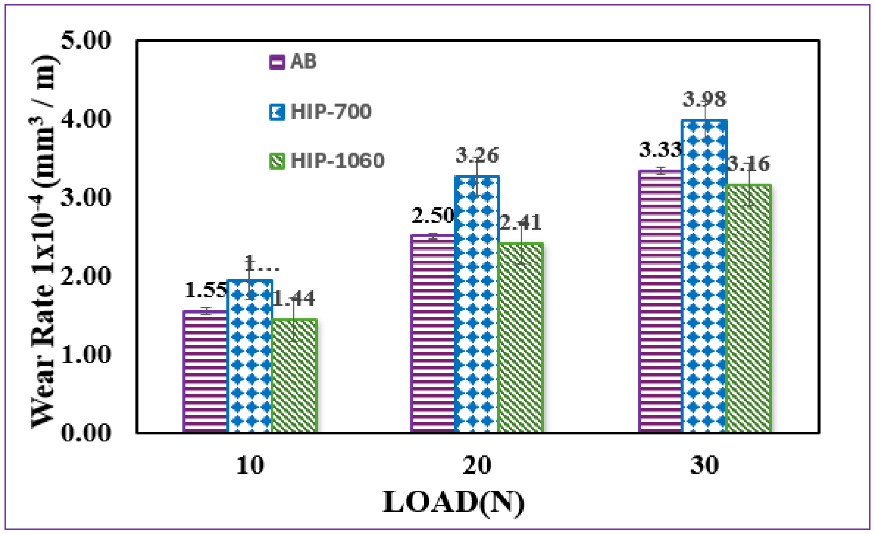


Fig.4.6: Average wear rate of Ti6Al4V before and after post processing

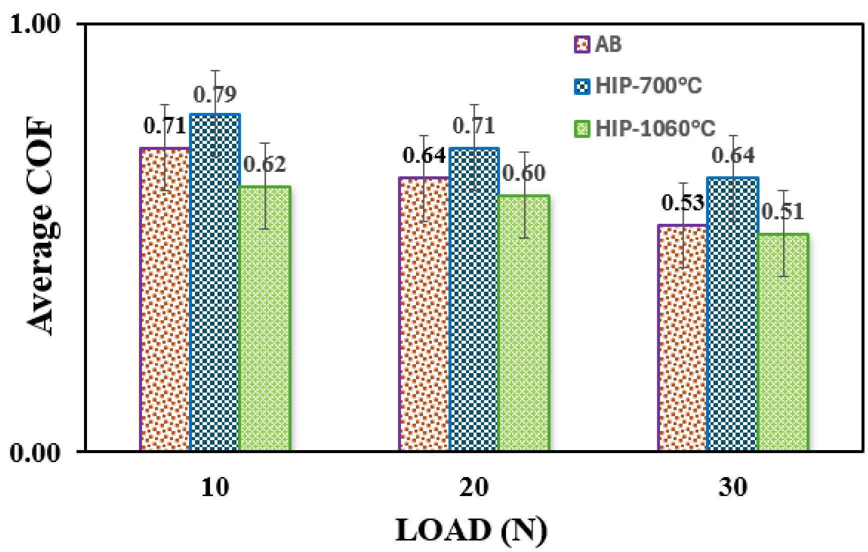


Fig.4.7 average COF of Ti6Al4V before and after post processing

- As-built samples exhibited higher wear rates and greater coefficient of friction (COF) due to surface roughness and unmelted particle defects.

- HIP-treated samples showed improved wear resistance, with smoother wear tracks and lower debris formation.
- Samples subjected to HIP at 910 °C and 1060 °C displayed the lowest wear rates and most stable COF values, owing to surface hardening and enhanced load-bearing capacity.

Microscopic analysis of wear tracks revealed that the wear mechanism transitioned from abrasive and adhesive wear in as-built samples to mild oxidative wear in treated ones.

4.6 Corrosion Test Findings

Corrosion testing was performed using a three-electrode

Table:4.1 Corrosion test of Ti6Al4V before and after post processing

Parameters	**Manufacturing process**		
	AB	HIP910°C	HIP910°C +HT
i_{corr} (µA/cm^2)	0.0629	0.0481	0.0435
E_{corr} (V)	-0.3612	-0.2719	-0.2424
CR (1x10^{-4} mm/y)	4.71	3.26	2.92

electrochemical setup. Key parameters like Open Circuit Potential (OCP) and Potentiodynamic Polarization (PDP) were analyzed.

- As-built Ti6Al4V exhibited higher corrosion current density and a more negative corrosion potential, indicating faster degradation.
- Samples treated with HIP at 910 °C and HIP 910 °C with heat treated demonstrated the lowest corrosion rates, attributed to the formation of a more stable and uniform passive oxide layer (TiO_2).
- These results affirm that defect-free, thermally treated sufaces resist electrolyte penetration, thereby enhancing corrosion resistance.

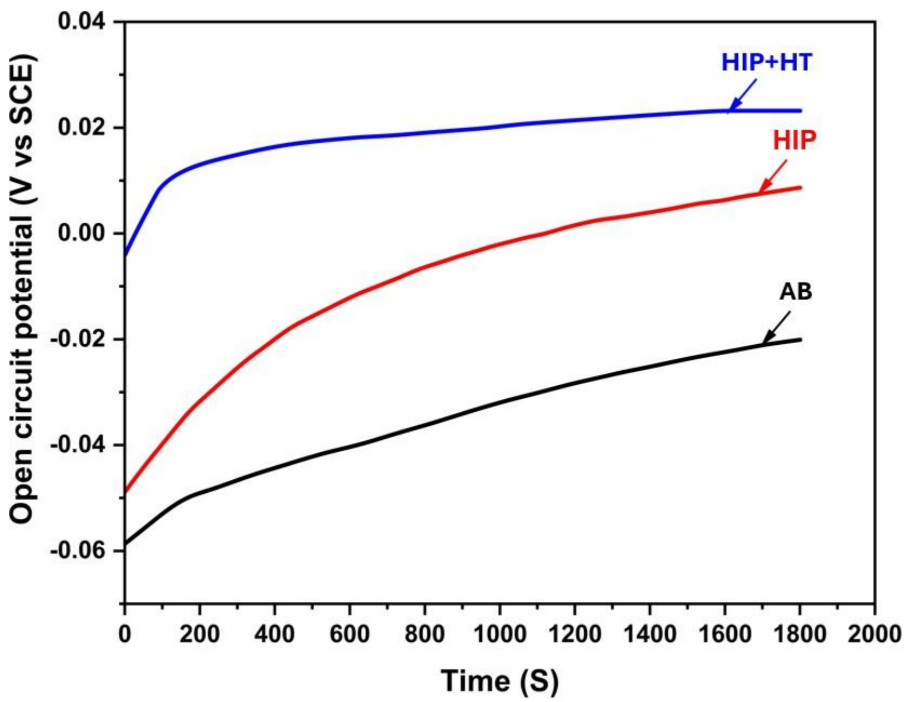


Fig.4.8 OCP plot of Ti6Al4V before and after post processing

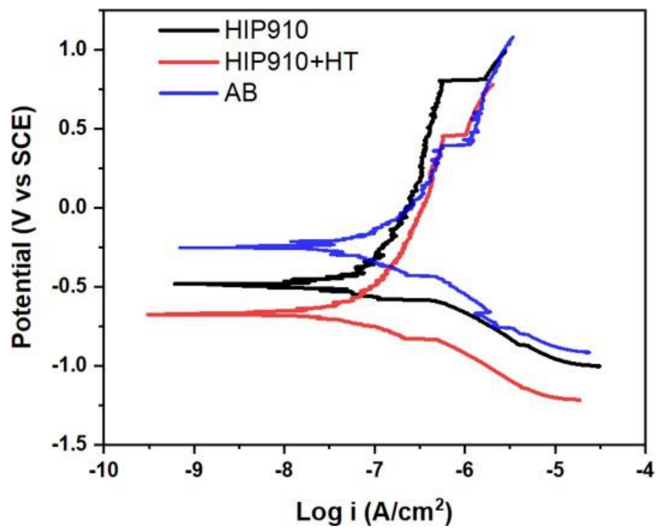


Fig.4.9: PDP curve of Ti6Al4V before and after post processing

Table 4.2: UTS of Ti6Al4V before and after heat treatment

Sample Condition	UTS (MPa)	% Elongation	Hardness (HV)	Wear Rate (mm³/N·m)	Corrosion Rate (mm/year)
As-built	~950	6–8	~373	High	High
HIP at 910 °C	~1020	10–12	~357	Medium	Low
HIP + HT at 1060 °C	~1100	14–16	~335	Low	Lowest
HT at 870 °C (Water Quenched)	~1060	12–14	~381	Moderate	Low

Table:

4.7 Discussion

The results clearly demonstrate that Hot Isostatic Pressing followed by appropriate heat treatment significantly enhances the mechanical, wear, and corrosion performance of Ti6Al4V alloy fabricated by additive manufacturing. The HIP process eliminates porosity and residual stress, while thermal treatment promotes desirable phase evolution, resulting in:

- Improved strength–ductility balance
- Enhanced surface integrity and wear resistance

- Superior tensile strength and corrosion behavior.

These enhancements make post-processed Ti6Al4V alloys well-suited for high-performance and safety-critical applications in the aerospace, biomedical, and automotive sectors.

These findings confirm that the integration of HIP and tailored heat treatment cycles can effectively enhance the performance of Ti6Al4V components for critical engineering applications, such as in aerospace, biomedical, and automotive sectors.

Chapter 5: Conclusion and Future Scope

5.1 Conclusion

This study investigated the influence of Hot Isostatic Pressing (HIP) and subsequent heat treatments on the mechanical, microstructural, tribological, and corrosion properties of additively manufactured Ti6Al4V alloy produced using the Direct Metal Laser Sintering (DMLS) process. The key findings are:

- HIP treatment, particularly at 910°C, significantly improved material densification by eliminating internal pores and defects inherent to the additive manufacturing process.
- Heat treatment, especially at 1060°C, further enhanced ductility and reduced residual stress, resulting in better tensile properties.
- The transformation from a martensitic α′ phase to a more stable α + β lamellar or bi-modal microstructure improved both wear performance and corrosion resistance.
- The combination of HIP + heat treatment resulted in the best overall mechanical performance, balancing strength, ductility, and surface integrity.
- Microstructural evaluation confirmed grain refinement, pore closure, and transformation to equiaxed grains in super-β-treated samples, promoting isotropy in mechanical properties.

5.2 Future Scope

While the present study provides a comprehensive evaluation, several areas remain open for future research:

- Strain Rate Sensitivity: Investigating the deformation behavior under varying strain rates can provide deeper insight into the material's response in dynamic environments..
- HIP at Variable Conditions: Exploring different HIP temperatures and pressures, including real-time pressure-temperature cycles, could optimize the densification process for specific applications.
- Residual Stress Analysis: Quantitative assessment of residual stresses before and after HIP and heat treatment would help refine the process parameters for critical load-bearing components.
- Surface Engineering Integration: Investigating post-HIP surface modification techniques such as laser nitriding, coating, or shot peening could improve surface hardness and wear resistance without compromising internal properties.
- Simulation and Modelling: Developing computational models to predict microstructural evolution, and crack propagation

- under various post-processing conditions can support design optimization.
- Application-Based Testing: Real-world validation of optimized Ti6Al4V samples in aerospace or biomedical prototypes could bridge the gap between research and industrial implementation

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