Chapter 1 Introduction

Titanium and its alloys are widely recognised for their exceptional properties, including an unmatched strength-to-weight ratio, excellent corrosion resistance across various environments, remarkable structural performance in extreme conditions, and notable biocompatibility [1-4]. These extraordinary combinations of properties place this material into the category of strategic materials for advanced engineering applications. Right after their commercialization in the late 1940s [5], this material revolutionises the market by setting high standards of performance, primarily in aerospace propulsion and airframe design [6], marine systems [7], chemical processing infrastructure [8], and energy generation technologies [9]. While conventional alloys like Ti-6Al-4V continue to dominate industrial applications due to their proven reliability [10], contemporary research targets next-generation solutions, including metastable β-phase formulations [11], innovative thermomechanical processing routes [12], and engineered surface architectures [13] to overcome persistent limitations such as high production costs [7], tribological vulnerabilities [14], and elevated-temperature performance constraints [15]. This chapter establishes the multidisciplinary framework for understanding titanium's metallurgical evolution, processing-property relationships, and expanding role in next-generation engineered systems.

1.1 Background

Titanium (Ti), with atomic number 22, was discovered in 1791 by William Gregor. It ranks as the ninth most abundant element in the Earth's crust and fourth among structural metals after aluminium, iron, and magnesium. Its commercial viability emerged only after Wilhelm Kroll developed a magnesium reduction process for titanium tetrachloride ($TiCl_4$) in 1937, which replaced the inefficient sodium reduction method and enabled industrial-scale production by the 1950s [16]. This breakthrough coincided with Cold War aerospace demands, driving the U.S. Department of Defence to classify titanium as a strategic material in 1951 and fund capacity expansions that increased global output from 2 tons per year in 1948 to 20,000 tons per year by 1960 [17, 18].

1.1.1 Metallurgical Fundamentals

Titanium exhibits two allotropic forms critical to alloy design [19] , as shown in Figure 1.1:

- ***α*-phase**: Hexagonal close-packed (HCP) structure stable below 883°C, characterized by superior creep resistance but limited slip systems, leading to anisotropic mechanical behaviour [20].
- ***β*-phase**: Body-centred cubic (BCC) structure stable above 883°C, offering enhanced formability and isotropic properties [21].

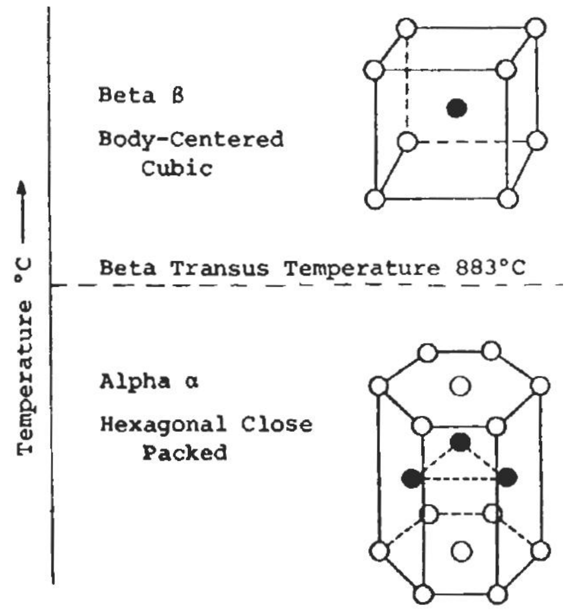

Figure 1.1: Crystal structures of titanium showing *α*-phase (hexagonal close-packed, HCP), stable below 883°C and *β*-phase (body-centred cubic, BCC), stable above 883°C. These structures underpin the mechanical and processing behaviour of titanium alloys [19].

Figure 1.1 illustrates the hexagonal close-packed (HCP) α-phase (space group: $P6_3/mmc$), which remains stable below 883°C. The HCP structure exhibits a = 0.295 nm, c = 0.468 nm, and a c/a ratio of about 1.58, leading to limited slip systems and anisotropic mechanical properties. Conversely, the body-centered cubic (BCC) β-phase (space group: Im-3m), stable above 883°C, has a lattice parameter a = 0.332 nm. Its symmetry and greater number of slip systems enable more isotropic deformation. The *transition from α to β phase is fundamental to titanium's thermomechanical processing. Adapted from [19].*

Alloying elements are strategically categorized by their phase-stabilizing effects, as shown in Figure 1.2 [11, 22]. In α-stabilized systems, the alloying elements exhibit greater solubility in the α-phase, resulting in an increase in the β-transus temperature. Common α-stabilizers include aluminium, oxygen, nitrogen, and carbon. In contrast, in β-stabilized systems, the alloying elements preferentially stabilize the β-phase, resulting in a decrease in the β-transus temperature. Typical β-stabilizers include iron, vanadium, molybdenum, and chromium.

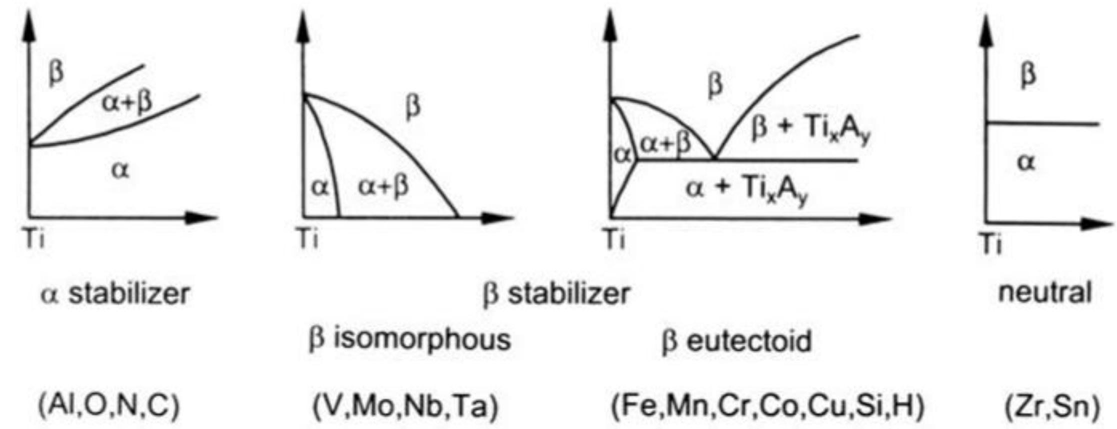

Figure 1.2: Effect of alloying elements on the titanium phase diagram, illustrating how α and β stabilizers shift the β transus temperature and enable dual-phase microstructures [22].

Figure 1.2 shows schematic equilibrium phase diagrams illustrating the effects of α- and β-stabilizers influence the β-transus temperature. α-stabilizers, such as Al, O, and N, raise the β-transus, favouring a dominant α-phase. Conversely, β-stabilizers like V, Mo, and Fe reduce the β-transus temperature, allowing the β-phase to be retained at room temperature. These diagrams serve as useful guides in designing alloys with dual-phase (α+β) microstructures, such as Ti-6Al-4V. Adapted from [22].

1.1.2 Properties and Advantages

Table 1.1 presents some key properties of titanium and its alloys, comparing them with other structural metals [23, 24]. Although titanium offers the best strength-to-density ratio, it is only used in specific applications because of its high cost. Titanium sponge is produced in an inert atmosphere due to its high reactivity with oxygen, which increases the production cost. However, titanium also has greater corrosion resistance in various environments because its high reactivity with oxygen creates a quick and stable oxide layer on its surface when exposed to air. Its high melting temperature, higher than aluminium, its major competitor in lightweight structural applications, gives it a distinct advantage at temperatures above 150 °C.

Table 1.1: Properties of Ti-6Al-4V compared to typical structural metals [23, 24]

Property	Ti-6Al-4V	4340 Steel	AA7075-T6	Inconel 718
Density (g/cm^3)	4.43	7.85	2.81	8.19
Yield strength (MPa)	830	1480	503	1100
Ultimate tensile strength (MPa)	900	1790	572	1350
Thermal conductivity (W/mK)	7.2	44.5	130	11.4
Cost ratio (vs. steel)*	15-30x	1x	3x	35x

**Cost ratios indicate raw material costs per unit volume. Titanium's excellent strength-to-weight ratio and corrosion resistance make it suitable for high-value uses, even though it is more expensive [23, 24].*

Key Advantages Driving Adoption:

- **Corrosion resistance:** TiO_2 passive film (Pilling-Bedworth ratio is 1.78) provides resistance to chlorides, acids, and oxidizing media, surpassing stainless steels [25].
- **Cryogenic toughness:** Ductile-to-brittle transition below -250°C enables liquid hydrogen tank applications [26].
- **Bio-inertness:** The TiO_2 layer inhibits ion release, resulting in a survival rate exceeding 95% for orthopaedic implants after 10 years [27].

Primary Fabrication:

- **Ingot metallurgy:** Triple vacuum arc remelting process attains below 10 ppm oxygen/nitrogen control for aerospace billets [28].
- **Powder metallurgy:** Hydride-Dehydride processed powder ($50/kg) enables near-net-shape hot isostatic pressing consolidation [29].

Secondary Processing:

- **Superplastic forming:** Ti-6Al-4V exhibits elongations exceeding 1000% at 900°C, enabling the fabrication of complex aerospace components. [30-33].
- **Additive manufacturing:** Electron beam melting reduces buy-to-fly ratio from 10:1 to 1.5:1 for turbine blades [34].

1.2 Phases and Crystal Structure of Titanium

The phase diagram of titanium, as shown in Figure 1.3, exhibits an allotropic transformation that plays a central role in its processing and mechanical behaviour [19]. At ambient pressure and temperatures below approximately 883 °C, pure titanium stabilizes in the *α* phase, which adopts the HCP crystal structure. This *α* phase is characterized by a *c/a* ratio close to 1.58 and a limited number of independent slip systems. This crystallographic constraint restricts the room-temperature ductility and formability of these metals compared to cubic metals.

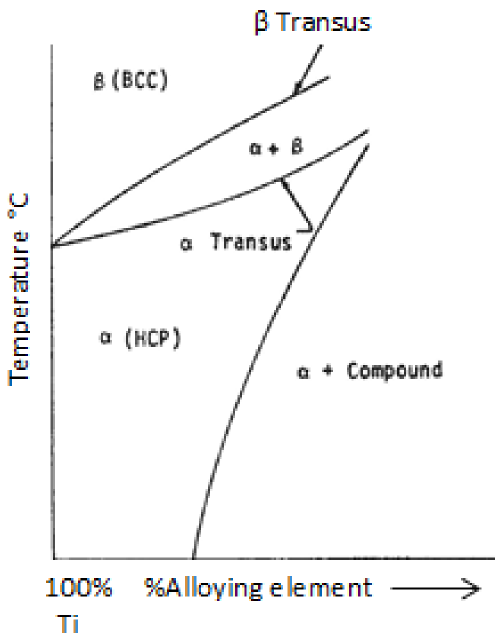

Figure 1.3: The phase diagram of titanium shows its basic equilibrium phases [19].

Figure 1.3 shows that the α to β allotropic transformation occurs at 883°C under ambient pressure. Specific areas represent common processing and service windows for the α-phase and the α+β phase. Alloying elements modify the β-transus temperature, allowing the creation of microstructures tailored for specific uses. Additionally, *metastable phases such as FCC and ω can form under non-equilibrium conditions, such as cryogenic rolling. Adapted from [19].*

When heated above the β-transus (approximately 883 °C for unalloyed Ti), titanium transforms to the β phase with a BCC structure [35, 36]. The β phase offers a higher-symmetry lattice with more available slip systems, thereby enhancing hot workability and enabling substantial microstructural tailoring during thermomechanical processing. Alloying additions strongly influence the α to β equilibrium transformation temperature. Elements such as aluminium stabilize the α phase, whereas vanadium, molybdenum, and niobium act as β stabilizers, lowering the β-transus temperature and enabling dual-phase $\alpha+\beta$ microstructures at room temperature [1, 37, 38]. The widely used Ti-6Al-4V alloy, therefore, contains both α and retained β phases after conventional processing, offering a balance of strength and toughness [39, 40].

Recent studies have also reported the formation of face-centred cubic (FCC) titanium under specific non-equilibrium conditions. Although FCC titanium is not stable in the equilibrium Ti phase diagram, high-pressure deformation, thin-film deposition, severe plastic deformation, and cryogenic deformation have occasionally produced metastable FCC domains or "ω-like" transitional structures [41-43]. Advanced characterization, including high-resolution transmission electron microscopy (HRTEM) and selected-area electron diffraction (SADP), has been used to identify such regions. The occurrence of these FCC-like signatures is typically linked to high defect densities, stacking faults, or local stress states that promote a change in the stacking sequence of the HCP basal planes [42].

For the present investigation, understanding the interplay of these structural variants is critical. The cryogenic rolling of Ti-6Al-4V and commercially pure titanium (CP-Ti) Grade 2 is expected to generate a very high dislocation density and extensive sub-grain formation. Such conditions favour dynamic recovery and, in some cases, the nucleation of transitional phases. Careful analysis of the α (HCP) matrix, the retained β (BCC) phase in Ti-6Al-4V, and any indications of FCC-type stacking faults provides a foundation for interpreting the mechanical response and for assessing the possibility of non-equilibrium phase formation during severe plastic deformation.

1.3 Motivation

Despite their exceptional specific strength and corrosion resistance, titanium alloys remain challenging to process because of the intrinsic crystallographic limitations of the α-HCP phase. Conventional thermomechanical routes can enhance strength but often compromise ductility, or require complex, multistep procedures [44, 45]. Cryogenic rolling offers a promising alternative. Deforming the material at liquid-nitrogen temperature suppresses dynamic recovery, stores a high density of dislocations, and encourages the formation of sub-grains and high-angle grain boundaries, all of which contribute to grain refinement and strengthening [46, 47].

A systematic understanding of how cryogenic rolling affects different titanium alloy systems is still incomplete [48, 49]. In particular, the mechanical and microstructural evolution of a dual-phase $\alpha+\beta$ alloy, such as Ti-6Al-4V, compared with a single-phase α alloy, such as CP-Ti Grade 2, has not been thoroughly documented under similar cryogenic conditions [50, 51]. The two alloys differ not only in chemical composition but also in deformation mechanisms. Ti-6Al-4V contains a stabilised β phase that can alter strain partitioning and recovery behaviour [35, 44], whereas CP-Ti deforms mostly through slip and limited twinning in the α phase [52, 53].

Furthermore, the influence of the initial microstructure, specifically the comparison between mill-annealed and heat-treated equiaxed grain structures, on the cryogenic deformation response remains underexplored [54, 55]. Equiaxed grains can reduce anisotropy and provide a uniform basis for studying dislocation storage, sub-grain formation, and grain-boundary character during severe deformation [56, 57].

This research is therefore motivated by:

- Quantitative comparison of microstructural refinement and enhancement of mechanical property induced by cryogenic rolling in Ti-6Al-4V and CP-Ti Grade 2.
- Assess the role of initial microstructure on texture evolution, dislocation density, and grain-boundary characteristics.
- Establish a structure-property correlation that can help in alloy design and process optimization for high-performance titanium components.

The study aims to clearly demonstrate how cryogenic rolling conditions and initial microstructures influence the balance between strength and ductility in key titanium alloys.

1.4 Research Objectives

The primary objective of this thesis is to explain how cryogenic rolling influences microstructural development and mechanical performance in Ti-6Al-4V and CP-Ti Grade 2, and to develop consistent structure-property relationships that can inform processing techniques for enhanced strength and ductility. To achieve this, the study aims to pursue the following specific objectives.

- **Quantify the effect of cryogenic rolling strain on mechanical properties:** Determine how incremental thickness reductions performed at liquid-nitrogen temperature change yield strength (YS), ultimate tensile strength (UTS), and percentage elongation for both Ti-6Al-4V and CP-Ti Grade 2 in mill-annealed and heat-treated conditions.
- **Characterize microstructural evolution across length scales:** Systematically document grain size, grain-boundary characteristics, and dislocation density produced during cryo-rolling using scanning electron microscopy (SEM), electron backscattered diffraction (EBSD) and transmission electron microscopy (TEM) for the two alloys and both starting conditions.
- **Establish structure-property correlations:** Correlate the results derived from SEM, EBSD and TEM observations with tensile test results to develop analytical relationships that explain strength and ductility trends as a function of deformation.
- **Assessment of initial microstructure effects:** Evaluate how mill-annealed versus heat-treated structures influence deformation uniformity, grain refinement, and ductility retention during severe cryogenic deformation.

1.5 Research Questions

- How does incremental thickness reduction during cryo-rolling influence grain refinement and microstructural evolution in Ti-6Al-4V vs CP-Ti?

- What is the role of initial microstructure (mill-annealed vs. heat-treated) in governing uniformity of deformation and the balance between strength and ductility under cryogenic rolling?
- Does cryogenic rolling induce deformation-driven metastable phases in CP-Ti or Ti-6Al-4V?
- How can measured grain boundary character and dislocation density be quantitatively correlated to the observed macroscopic tensile properties?

1.6 Methodological Approach

The methodological approach of this study has been carefully designed to systematically address the research questions related to microstructural evolution, deformation mechanisms, and structure–property relationships in cryo-rolled titanium alloys. Materials, processing route, and characterization techniques were selected by considering their ability to provide complementary insights across different length scales.

Ti-6Al-4V and CP-Ti Grade 2 were selected as base materials to represent dual-phase ($\alpha+\beta$) and single-phase (α) titanium systems, respectively, in both mill-annealed and heat-treated conditions. This enables a direct comparison of deformation behaviour and phase stability under identical cryogenic rolling conditions, thereby clarifying the roles of alloy chemistry, phase constitution, and initial microstructure in governing microstructural evolution.

Cryo-rolling deformation was employed due to its ability to suppress dynamic recovery and enhance dislocation accumulation at liquid nitrogen temperature. This enables the investigation of grain-refinement mechanisms and defect storage under conditions of limited thermal activation. Incremental thickness reduction was applied to progressively introduce plastic strain, enabling the study of deformation-induced changes as a function of increasing strain.

To evaluate the influence of initial microstructure, both mill-annealed and heat-treated conditions were considered. The heat-treated condition provides a relatively strain-free, equiaxed grain structure, while the mill-annealed condition retains prior deformation features. This comparison is essential for understanding the influence of deformation uniformity, dislocation evolution, and ductility on initial grain morphology and stored defects.

Microstructural characterization was performed using a combination of SEM, EBSD, and TEM techniques. SEM was employed for morphological interpretations and to assess grain elongation and the formation of deformation bands. EBSD was selected to quantify grain size, grain boundary character, and to estimate dislocation density through Kernel Average Misorientation (KAM) analysis. TEM was used for high-resolution characterization of dislocation configurations and phase identification, particularly to investigate the possible formation of metastable phases such as FCC domains.

Mechanical behaviour was evaluated through uniaxial tensile testing, which provides quantitative measurements of yield strength, ultimate tensile strength, and ductility. The combination of microstructural and mechanical characterization enables the establishment of structure–property relationships, directly correlating deformation-induced microstructural features with mechanical properties.

Overall, the adopted methodology provides a reasonable approach to achieving the research objectives by correlating processing conditions, microstructural evolution, and mechanical performance in a systematic and reproducible manner.

1.7 Scope of Thesis

This study explores the microstructural evolution and mechanical behaviour of two essential titanium alloys (Ti-6Al-4V and CP-Ti Grade 2) under controlled cryogenic rolling. The scope is deliberately limited to specific experimental variables and characterization methods to ensure a clear and reproducible understanding of structure-property correlation.

- **Chapter 2: Literature Review:** Mechanisms of deformation in HCP metals and crystallographic constraints in titanium, prior deformation studies.
- **Chapter 3: Materials and Experimental Methods:** Precise material specifications, heat-treatment schedules, cryo-rolling procedure, sample geometry, and detailed microstructural characterization (SEM, EBSD, TEM) and tensile testing procedures.
- **Chapter 4: Microstructural Evolution and Phase Analysis of Ti-6Al-4V to Cryo-Rolling:** SEM, EBSD, and TEM analyses were conducted to study the microstructural evolution, grain boundary characteristics, dislocation density and

metastable phase formation in cryo-rolled Ti-6Al-4V for both mill-annealed and heat-treated sample conditions.

- **Chapter 5: Microstructural Evolution and Phase Analysis of CP-Ti Grade 2 to Cryo-Rolling:** SEM, EBSD, and TEM analyses were conducted to study the microstructural evolution, grain boundary characteristics, dislocation density and metastable phase formation in cryo-rolled CP-Ti Grade 2 for both mill-annealed and heat-treated sample conditions.
- **Chapter 6: Mechanical Behaviour and Deformation Response:** Tensile tests were conducted to quantitatively analyze the mechanical properties of both alloys (Ti-6Al-4V and CP-Ti Grade 2) in their mill-annealed and heat-treated conditions. A correlation was established between microstructural analysis and mechanical properties.
- **Chapter 7: Conclusions and future work:** Key findings, scientific contributions, limitations, recommendations for future work and conclusion.

1.8 Significance of the Study

This research addresses key gaps in the processing-structure-property relationship for titanium alloys subjected to cryo-rolling. By conducting a controlled, comparative study of cryogenic rolling on Ti-6Al-4V and CP-Ti Grade 2 in both mill-annealed and heat-treated conditions, the thesis presents several scientifically and technologically significant findings.

- **Clear understanding of cryo-rolling deformation mechanism:** The work provides a systematic study of how suppressed recovery at liquid-nitrogen temperatures affects microstructural evolution, dislocation accumulation and grain-boundary characteristics. Quantitative EBSD and TEM data directly link microstructural refinement to the macroscopic tensile response, thereby developing a systematic understanding of strengthening and ductility trends under cryo-rolling.
- **Role of initial microstructure:** By comparing the mill-annealed and heat-treated conditions, the study shows how grain morphology and stored defects affect grain refinement, deformation uniformity, and the effectiveness of cryo-rolling. These

insights are valuable for designing pre-treatments that maximize favourable property outcomes for specific applications.

- **Application relevance:** The combination of enhanced strength and retained ductility achieved through cryo-rolling, especially when initiated from a heat-treated process, has direct relevance to aerospace, cryogenic storage, and biomedical components where weight, toughness, and dimensional stability are critical. The study, therefore, contributes to closing the gap between laboratory deformation methods and application-oriented component design.
- **Foundation for future research:** The work highlights promising research directions for future, such as HRTEM, synchrotron XRD, and other cryogenic deformation techniques, along with alloying to explore stacking-fault energy. It offers the necessary experimental protocols and decision criteria for pursuing these directions. Additionally, it positions CP-Ti and Ti-6Al-4V as complementary model systems for studying deformation-driven microstructure engineering in HCP materials.

In summary, this thesis enhances both fundamental metallurgy and practical processing of titanium alloys through reproducible experiments, quantitative analysis of microstructure-property correlations, and practical recommendations for thermomechanical conditioning under cryogenic deformation.

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