Cryogenic rolling induced microstructural evolution, mechanical behaviour, and phase stability in Ti-6Al-4V and CP-Ti alloys

by

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This thesis is

dedicated to my family

for

their unwavering support and endless sacrifice

for

whatever I am today and will be in the future…

Certificate

This is to certify that the work incorporated in this Ph.D. thesis entitled, *“***Cryogenic rolling induced microstructural evolution, mechanical behaviour, and phase stability in Ti-6Al-4V and CP-Ti alloys**”, submitted by **Vaibhav Gaur** to the Academy of Scientific and Innovative Research (AcSIR) and Royal Melbourne Institute of Technology (RMIT) in fulfillment of the requirements for the award of the Degree of **Doctor of Philosophy (Ph. D)** the AcSIR-RMIT Joint PhD (Cotutelle) Degree Program*,* embodies original research work carried out by the student. We further certify that this work has not been submitted to any other University or Institution in part or full for the award of any degree or diploma. Research material(*s*) obtained from other source(*s*) and used in this research work has/have been duly acknowledged in the thesis. Image(*s*), illustration(*s*), figure(*s*), table(*s*) etc., used in the thesis from other source(*s*), have also been duly cited and acknowledged.

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I **Vaibhav Gaur**, a Ph.D. student of the Academy of Scientific and Innovative Research (AcSIR) with Registration No.: **20EE19A31001** hereby undertake that, the thesis entitled **"Cryogenic rolling induced microstructural evolution, mechanical behaviour, and phase stability in Ti-6Al-4V and CP-Ti alloys"** has been prepared by me and that the document reports original work carried out by me and is free of any plagiarism in compliance with the UGC Regulations on "Promotion of Academic Integrity and Prevention of Plagiarism in Higher Educational Institutions (2018)" and the CSIR Guidelines for "Ethics in Research and in Governance (2020)".

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Table of Contents

List of Figures

List of Tables

List of Abbreviations

HCP	Hexagonal Close-Packed
BCC	Body-Centred Cubic
FCC	Face-Centred Cubic
BF	Bright Field
BSE	Backscattered Electron
CPFEM	Crystal Plasticity Finite Element Modelling
CP-Ti	Commercially Pure Titanium
DF	Dark Field
DFT	Density Functional Theory
DIC	Digital Image Correlation
EBSD	Electron Backscattered Diffraction
ECAP	Equal Channel Angular Pressing
ECD	Equivalent Circle Diameter
EDM	Electrical Discharge Machining
EL%	Percentage Elongation
FCC	Face-Centred Cubic
FEG	Field Emission Gun
GND	Geometrically Necessary Dislocation
HAGB	High Angle Grain Boundary
HPT	High-Pressure Torsion
HRTEM	High-Resolution Transmission Electron Microscopy
IPF	Inverse Pole Figure
KAM	Kernel Average Misorientation
LAGB	Low Angle Grain Boundary
ND	Normal Direction
ODF	Orientation Distribution Function
RD	Rolling Direction
SAED	Selected Area Electron Diffraction
SE	Secondary Electron
SEM	Scanning Electron Microscopy
SFE	Stacking Fault Energy

SPD	Severe Plastic Deformation
SSD	Statistically Stored Dislocation
TD	Transverse Direction
TEM	Transmission Electron Microscopy
Ti	Titanium
TMP	Thermomechanical Processing
TRIP	Transformation-Induced Plasticity
UFG	Ultrafine Grain
UTS	Ultimate Tensile Strength
XRD	X-Ray Diffraction
YS	Yield Strength

Synopsis

This study investigates the effects of cryogenic rolling on Ti-6Al-4V and commercially pure titanium (CP-Ti) alloys, known for their high specific strength, corrosion resistance, and biocompatibility. Ti-6Al-4V, a dual-phase α+β alloy, is commonly used in aerospace and biomedical applications for its balance of strength and ductility. In contrast, CP-Ti, a single-phase α alloy, is mainly used for its formability and corrosion resistance. The research examines the effects of cryogenic temperatures on microstructural evolution, mechanical properties, and phase stability, with a focus on the relationship between initial microstructure, mill-annealed (MA) versus heat-treated (HT), influences deformation mechanisms. Cryogenic rolling, performed at liquid nitrogen temperatures (-196°C) caused significant grain refinement in both alloys. For Ti-6Al-4V, the complex deformation behaviour of the dual-phase structure involved the α-phase (HCP), which developed dislocation accumulation and sub-grain formation due to limited slip systems, while the β-phase (BCC) accommodated strain through active slip. After cryo-rolling, grain size reduced from approximately 24 µm to 9.5 µm in MA samples, and HT samples achieved even finer, more uniform microstructures (around 8.5 µm) due to recrystallized starting microstructure. Electron Backscatter Diffraction (EBSD) analysis showed an increase in low-angle grain boundaries (LAGBs) from approximately 46% to 69% in MA Ti-6Al-4V, indicating dislocation-driven sub-grain formation. HT samples showed an increase in LAGBs from around 39% to 79%, suggesting suppressed dynamic recovery.

In contrast, the CP-Ti single-phase α structure enabled simpler grain refinement. Cryogenic rolling reduced grain sizes from about 38µm to 9µm in the MA condition and from 50µm to 17µm after HT. A key observation was the formation of metastable face-centred cubic (FCC) domains within the HCP matrix, confirmed by Transmission Electron Microscopy (TEM). These FCC regions, measuring 10–50 nm, were not present in Ti-6Al-4V and are believed to form due to shear-induced stacking fault accumulation under cryogenic conditions. This phase transition does not occur in bulk titanium at room temperature, indicating a new deformation mechanism driven by stress-induced lattice reorientation. Mechanistically, the lack of FCC transformation in Ti-6Al-4V relates to vanadium's stabilizing effect on the β-phase, which inhibits shear reorientation. Conversely, the single-phase CP-Ti structure allowed localized stress to initiate FCC nucleation. TEM images showed dense dislocation walls and stacking faults near FCC regions, suggesting cryogenic conditions facilitated fault accumulation and enabled the transition from HCP to FCC.

Cryogenic rolling mechanically improved strength in both alloys, but with differing impacts on ductility. Ti-6Al-4V showed a 19% increase in yield strength (YS), from 890 MPa to 1,062 MPa, and ultimate tensile strength (UTS), from 982 MPa to 1,169 MPa, at 30% strain in MA samples. HT samples experienced an even greater 27% increase in YS, from 883 MPa to 1120 MPa, and a 19% increase in UTS, from 957 MPa to 1135 MPa, due to uniform dislocation distribution. Nonetheless, ductility sharply declined from around 12% to 6% in MA and from roughly 13% to 5% in HT conditions, as dislocation tangles and sub-grain boundaries hinder plastic flow. In contrast, CP-Ti showed a more balanced response, with strength increasing significantly with YS rising from 375 MPa to 692 MPa and UTS from 507 MPa to 753 MPa in MA, and YS from 361 MPa to 659 MPa and UTS from 517 MPa to 727 MPa in HT at 30% strain. Ductility, however, dropped abruptly from approximately 28% to 13% in MA and gradually from about 30% to 10% in HT, due to strain-induced FCC phase transformation. The preservation of ductility indicates the potential for Transformation-Induced Plasticity (TRIP) effects, in which metastable FCC domains absorb strain energy via reversible phase changes, thereby delaying fracture.

The research highlights the effect of initial microstructure on cryogenic deformation. HT Ti-6Al-4V with equiaxed grains promotes uniform strain distribution, which improves strain hardening and delays shear localization. In contrast, MA samples exhibited pre-existing deformation anisotropy and accelerated ductility loss. For CP-Ti, HT produced a strain-free α-phase structure, facilitating comparatively uniform FCC formation, while MA samples have random FCC regions due to the initial strain condition. These results suggest CP-Ti could be suitable for TRIP-assisted uses, like biomedical implants or cryogenic storage tanks, where damage tolerance is critical, while Ti-6Al-4V optimized strength-ductility balance via pre-heat treatment, making it ideal for aerospace components that need fatigue resistance.

The identification of FCC domains in CP-Ti offers new insights into metastable alloy development. Future studies should focus on post-rolling annealing to improve high-angle grain boundary (HAGB) formation, use in-situ techniques to monitor FCC phase kinetics, and examine β-stabilized titanium alloys to better understanding of TRIP effects. This work advances titanium metallurgy by demonstrating cryogenic rolling as a method to modify microstructures and mechanical properties, providing a basis for next-generation materials designed for extreme environments. By connecting fundamental understanding with practical applications, the study offers guidance for creating titanium alloys that balance strength, ductility, and phase stability through controlled thermomechanical processing.