

UNIVERSITI TEKNOLOGI MARA

**EFFECT OF POWDER
METALLURGY PROCESSING
PARAMETERS ON MECHANICAL
PROPERTIES AND
DEHYDROGENATION BEHAVIOUR
OF TITANIUM-NIOBIUM ALLOY**

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ABSTRACT

Extensive research has been conducted on P-type Ti alloys that are composed of non-toxic metallic elements, such as niobium (Nb), in order to address the challenges of high elastic modulus and toxicity of specific elements, particularly in the Ti-6Al-4V alloy. Recently, there has been much interest in titanium hydride (TiH₂) due to its densification, oxidation levels, and material costs. This research aims to develop a TiH₂-Nb alloy to obtain high compressive strength and low elastic modulus to prevent the stress shielding phenomenon in bioimplants. Sintering temperature, sintering time, and milling speed serve as the parameters in this study. The fabrication of the TiH₂-Nb alloy involves mechanical alloying and powder metallurgy, followed by compaction at 500MPa and sintering under argon flow. The investigated combination of sintering temperature, sintering time, and milling speed was determined to be 800-1200°C, 1-3 hours, and 100-300 rpm, respectively. The model was analyzed and optimized by the Box Behnken method via Design Expert 7 software to suggest a milling process at 200 rpm before being sintered at 1200°C for 3 hours to achieve optimal results with a desirability of 0.9. The cp Ti-Nb alloy exhibited a higher compressive strength (1887MPa) and elastic modulus (12.4GPa) than the TiH₂-Nb alloy (1768MPa and 8.7 GPa, respectively). The amount of (3 phases for TiH₂-Nb alloy (66%) was slightly higher than cp Ti-Nb alloy (65.7%). Meanwhile, the density of the cp Ti-Nb alloy is slightly lower at 5.43 g/cm³, while the density of the PiPP-Nb alloy is 5.48 g/cm³. The porosity obtained for PiPP-Nb alloy (1.44%) was lower than cp Ti-Nb (2.34%), proving superior densification. Lastly, the decomposition of hydrogen from TiH₂ was investigated using thermogravimetric analysis (TGA) and X-ray diffraction (XRD) analysis. Based on the TG analysis, dehydrogenation process initiated at higher temperatures for TiH₂-Nb powders milled at 100 rpm (TH100) (488°C-582°C), whereas it began at lower temperatures for 200 rpm (TH200) (340°C-410°C) and 300 rpm (PH300) (340°C-430°C). Higher milling speeds increased kinetic energy, which improved Nb diffusion, made grains smoother, and created more defects, lowering the activation energy for hydrogen release. From XRD patterns, the absence of PiPP and NbH peaks demonstrated successful dehydrogenation, stabilizing the final microstructure with a-Pi and (3-Pi phases. Stabilized by Nb, (3-Pi enhanced ductility, whereas a-Pi enhanced strength and hardness. NbH dissolved, releasing pure Nb, while PiPP went through dehydrogenation at 340°C, resulting in a-Pi (HCP structure) and the removal of PiPP peaks. Nb diffused into a-Pi to enhance the a-Pi (3 phase shift and create (3-Pi (BCC structure) at temperatures up to 1200°C. In conclusion, the PiPP-Nb alloy that was successfully developed in this study exhibits superior densification, a low elastic modulus, and high compressive strength, rendering it a promising material for biomedical implants. Milling speed was identified as a critical factor that influenced the final microstructure by influencing dehydrogenation behaviour, Nb diffusion, and phase stabilization. The primary contribution of this work is the optimization of processing parameters for the fabrication of low-modulus, high-strength Ti-based alloys to mitigate stress shielding, as well as the establishment of PiPP as a cost-effective starting material. Additionally, the results offer valuable insights into the relationship between mechanical performance, microstructural evolution, and dehydrogenation kinetics, providing guidance for the development of next-generation titanium biomaterials.

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CHAPTER 1

INTRODUCTION

1.1 Background of Study

Biomaterials involve both synthetic and naturally occurring substances that are utilised to substitute or repair damaged or absent biological structures, with the ultimate objective of reinstating their original form and functionality (Todros et al., 2021). They are employed in heart valves, stents, hip, knee, shoulder, elbow, and dental implants. With advancements in medical science, a wide range of materials, including metals, polymers, ceramics, and composites, have been developed and used for implantation into the human body.

A bone implant, referred to as an orthopaedic implant or bone prosthesis, is a medical apparatus employed for the purpose of substituting or providing assistance to bone tissues in individuals who have experienced bone injuries, fractures, or bone ailments (Al-Shalawi et al., 2023). These implants are intended to restore function, stabilise the affected bone or joint, and mitigate pain. The selection of materials for bone implants is conducted with meticulous care to ensure their biocompatibility, which refers to their ability to be well-tolerated by the human body without eliciting any adverse reactions or experiencing rejection (Marin et al., 2025).

A rapidly increasing global demand for metallic biomaterials is driven by an ageing population and the increased risk of hard tissue failure as individuals age (Oladele et al., 2023). Titanium (Ti), with a density of 4.54 g/cm^3 , closely matches the density of human bone ($1.8\text{-}2.0 \text{ g/cm}^3$) in comparison to stainless steel (8.0 g/cm^3) or cobalt-based alloys (8.5 g/cm^3) (Baroutaji et al., 2022; Govindaraman et al., 2022). Its density, along with superior biocompatibility and corrosion resistance when in contact with human tissues and bodily fluids, makes commercially pure titanium (cp-Ti) an ideal choice for bone implants, replacing 316L stainless steel and cobalt-chromium (Co-Cr) alloys (Azmat et al., 2023). Subsequently, the a+ P type Ti-6Al-4V alloy replaced cp-Ti due to its superior mechanical properties.

Ti-6Al-4V alloy has been extensively used in bone grafts for artificial hip joint replacements due to its biocompatibility, strength, and resistance to corrosion. However, concerns have been raised about the cytotoxic effects of vanadium (V) and