

ANALYSIS OF HEAT TREATMENT RESULTS OF Ti6Al7Nb FOR APPLICATION AS TOTAL HIP ARTHROPLASTY COMPONENT MATERIAL

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ABSTRACT

KEYWORDS

Ti6Al7Nb, Heat treatment Total Hip Arthroplasty Thermal History.

This research is a literature study on Ti-6Al-7Nb material after having heat treatment. This study aims to analyze the results of selected articles reporting the effect of Ti-6Al-7Nb heat treatment. The research method used in this literature study is a qualitative descriptive method. Only several article results from analyses were reviewed that meet the requirement for implementation for future research topics related to materials for total hip arthroplasty. Data sources are obtained through library research techniques (literature study), which refers mainly to online sources, such as scientific journals, websites, and news from trusted sources. The results concluded that materials preparation techniques, heat treatment methods, and results analysis reported in those articles should be appropriate in this paper review. It was suggested from the literature that we need to anticipate that the martensitic phase does not cause significant changes in the Titanium alloys' properties. Moreover, the heat treatment method for α -type Titanium alloys is ineffective; namely, the heat treatment of Titanium alloys is mainly used for $\alpha+\beta$ -type Titanium alloys. The heat treatment should avoid the formation of the ω phase because constructing the ω phase will make Titanium alloys brittle.

INTRODUCTION

Heat treatment is a process of heating and cooling materials to reach expected physical, mechanical, and tribological properties (Banerjee, 2017). The heat treatment of metals and alloys aims at changing microstructures, including changing the defective metallic crystal structure, change in chemical composition, and degree of order (Banerjee, 2017). The changes mentioned above bring the purpose and the chosen thermal treatment of metals and alloys to improve (Banerjee, 2017):

- a) physical properties by refining grain sizes, reducing porosity, electrical, and magnetical properties;
- b) chemical properties, especially in respect of corrosion resistance;

- c) mechanical properties, internal stresses removal, and machineability;
- d) tribological properties, materials' surface properties, especially in respect of wear rate and wear volume release from contacting surfaces;
- e) other unique material functionalities include transformation-induced plasticity and the metamagnetic shape memory effect.

Poor tribological properties such as high friction coefficient, low abrasive wear resistance, and relatively low hardness are the most significant obstacles hindering the broader use of titanium-based alloys and limiting their applications under sliding conditions and contact loads (Suhendra, et al., 2009; S. Izman). Therefore, to improve the tribological properties of titanium alloys, various methods have been applied to improve resistance to abrasive wear (Sandomierski, et al., 2020) and plastic shearing (Borgioli, et al., 2005). Moreover, surface coatings may also improve or modify tribological properties surface layers' microstructure (Kilicay, et changing al., by 2020). Thermochemical treatments improved the wear resistance of Titanium Alloy using thermal oxidation. Although this is a low-cost processing route for wear performance enhancement, it suffers limitations like other processes such as nitriding and boriding (Kilicay, et al., 2020). Recently, a new processing route for a lower-grade titanium alloy has been developed (Alcisto, et al., 2004; Borradaile, et al., 1980; Narayana, et al., 2010; M. Peters, et al., 2003). However, up to now, many efforts have only been dedicated to evaluating the mechanical and microstructural properties of β titanium alloys with Fe and Nb addition. However, very few studies have been systematically conducted to evaluate the tribological behaviour and mechanical properties of α -type Ti-6Al alloy with Nb addition (Fellah, et al., 2014).

Heat treatment application to Ti-6Al-7Nb material, as mentioned above, comes from the purposes of the manufacturing process's background and the material's production purpose. Titanium alloys of this type are generally used as materials for biomaterial applications. Titanium alloys as bone implants require excellent biocompatibility, osseointegration, and non-toxicity. Apart from the above-mentioned, materials used in total hip arthroplasty require corrosion resistance and mechanical and tribological properties (Fellah, et al., 2014; Aniołek, et al., 2022). Therefore, in discussing this literature review, the author tries to figure out the various methods of the titanium manufacturing process in general, then find out the effectiveness of heat treatment related to the phases of titanium alloys. Furthermore, studying the stages of heat treatment work and strategies to get the expected results, including avoiding things that can be detrimental during the heat treatment process. Of course, all information is extracted from information published by researchers in their field.

The analysis of the heat treatment results leads to changes in the material's microstructure, such as grain size, grain shape, cavity size, and surface morphology. All of the above information relates to changes in physical and mechanical properties and even tribological properties. This information will be beneficial as a guide and reference in our current research work. To produce Titanium alloys, according to Hamweendo Agripa, and Ionel Botef (Agripa, et al., 2018), there are three groups of methods: the conventional, advanced and future strategies. Powder metallurgy (PM) is categorised as a conventional sintering method (Tsutsui, 2022; James, 2015). In this method, the mixture of feedstock titanium powder with alloying elements using a suitable powder blender is performed, followed by compaction under high pressure and finally sintered. Advanced production methods use technological methods to improve the processes with the relevant technology to reach better product quality. These technologies are developed conventional techniques to achieve various titanium base alloys and aluminides components (Soliman, et al., 2022). The most widely used cutting-edge method for the production of titanium alloys is atomisation processes (Agripa, et al., 2018). The future methods for producing titanium alloys depend on the demand for these products and to what extent nature will be able to provide them . (Agripa, et al., 2018)

Roughly there are five groups of Titanium, and its alloys can be considered, namely pure (unalloyed), α , near α , $\alpha+\beta$ and β . The unalloyed Titanium exhibits different crystalline lattices at different temperatures. It is called an allotropic material. The packed hexagonal structure (α phase) transforms to a body-centred cubic structure (β phase) at 885°C. The β transus remains at this phase until reaching the melting point of 1,668°C (Gilbert and Shannon, 1991). Alloying elements in Titanium, concerning the allotropic alteration, can be classified as α stabilizers, β stabilizers, or neutral. The presence of α stabilizers affects the increase in the α to β transition temperature.

Depending on the material's response to the heat treating temperature and the alloying elements, the alloys of Titanium can be classified into the following three types (Agripa, et al., 2018):

1. The alpha (α) alloys

These alloys contain α -stabilizing alloying elements with a large amount, such as Aluminium, oxygen, nitrogen or carbon. Aluminium is widely used as the alpha stabilizer for most commercial titanium alloys because it is capable of withstanding the alloy at ambient and elevated temperatures up to about 550°C. This capability, coupled with its lightweight, makes Aluminium an additional benefit over other alloying elements such as copper and molybdenum. However, the Aluminium amount added should be limited because a brittle titanium-

Aluminium compound could occur if it reaches 8% or more by weight of Aluminium. Adding oxygen to pure titanium can increase the strength of Titanium alloy as the oxygen level rises. The non-heat treatable of Titanium α alloys is its disadvantage. However, these Titanium types are generally weldable and have low to medium strength, good notch toughness, reasonably good ductility and good properties at cryogenic temperatures. The addition of tin or zirconium can strengthen Titanium α alloys. These metals have appreciable solubility in both alpha and beta phases, and as their addition does not markedly influence the transformation temperature, they are generally classified as neutral additions. Like Aluminium, tin and zirconium are alloying elements, and the hardening at ambient temperature is retained even at elevated temperatures (Agripa, et al., 2018).

2. The alpha-beta $(\alpha - \beta)$ titanium alloys

These alloys contain four to six per cent of β -phase stabilizer elements such as molybdenum, silicon, tantalum, tungsten, and vanadium. These elements increase the amount of β -phase in the metal matrix, strengthening these alloys by precipitation hardening, and are heat treatable. Solution treatment of these alloys causes the increase of β -phase content mechanical strength while ductility decreases. The most famous example of the α - β titanium alloy is the Ti-6Al-4V with 6 and 4% by weight Aluminium and vanadium, respectively. This alloy of titanium is about half of all titanium alloys produced. Aluminium is added as an α -phase stabilizer and hardener in these alloys due to its solution strengthening effect. The vanadium stabilizes the ductile β -phase, providing hot workability to the alloy (Agripa, et al., 2018).

3. The beta (β) titanium alloys

The elements are β stabilizing elements used in Titanium alloys: chromium, cobalt, copper, iron, manganese, molybdenum, nickel, niobium, tantalum, vanadium, and zirconium. Apart from reducing the resistance to deformation, the β stabilizers strengthen the beta phase and improve alloy fabricability during working operations in hot and cold environments. β stabilizer confers a heat treatment capability that permits significant strengthening during the heat treatment process (Agripa, et al., 2018).

Ti-6Al-7Nb ALLOY PRODUCTION

Powder metallurgy methods are generally used to produce Ti-6Al-7Nb. The most common forms are hot pressing, metal injection mouldering, and blending and pressing. In the production of Ti-6Al-7Nb, a sintering temperature between 900-1400° C is usually used (Figure 1) (Hamweendo, et al., 2016).

The three types of mixed powder, Ti, Al, and Nb, were used in producing Ti-6Al-7Nb alloys by using the metal injection moulding process. The production methods of Ti-6Al-7Nb are

- 1. pre-alloyed powder by mixing Ti and Al-Nb powders,
- 2. the mixture of Ti, Ti-Al alloy, and Nb powders, and
- 3. Mixing of elemental powders of Ti, Al, and Nb.

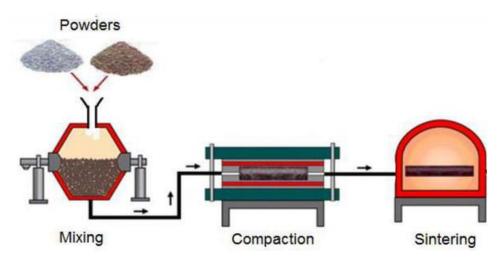


Figure 1: Powder metalurgy process (Hamweendo, et al., 2016)

The first and second methods produce higher density and mechanical properties than the third powder mixture. The third method showed many large pore formations resulting from the Aluminium particle dissolution during the sintering steps. The mixture of Ti+Al-Nb or Ti+Ti-Al+Nb powders produces compacted material with elongation of above 10% with a tensile strength of above 800 MPa. The processing of this alloy using powder metallurgy allows the preparation of parts with complex geometry, which is probably less expensive. Samples of this alloy were obtained from the uniaxial hot pressing of the elemental powders in a vacuum. The pressing was carried out in the temperature range 1000–1500°C with pressures from 10 to 25 MPa.

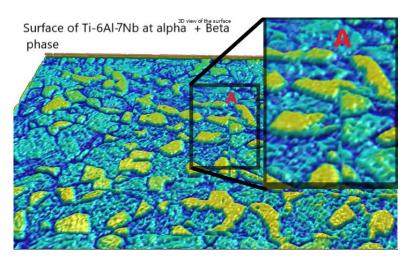


Figure 2: Typical SEM image of Ti-Al-Nb alloys at the α-β phase (*Suhendra, 2022*)

Figure 2 shows the typical surface morphology of the Ti-Al-Nb alloy, where the α , and β phases are recognised from their shapes. The gold colour indicates alloy composition of Ti-6Al-7Nb.

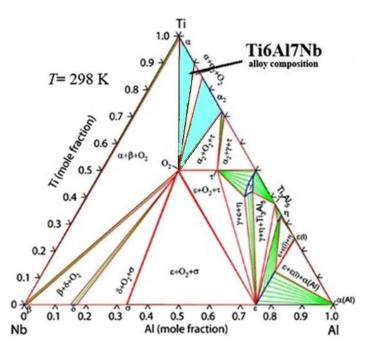


Figure 3: Calculated Phase Diagram for TiAlNb (Stepień, et al., 2016)

Figure 3 shows the calculated phase diagram for Ti-Al-Nb, where the alloy composition of Ti-6Al-7Nb is located at the area as pointed out by the arrow near the peak of the rectangular phase diagram.

Ti-6Al-7Nb HEAT TREATMENTS

The heat treatment of Titanium alloys aims to reduce residual stress developed during fabrication. It can produce an optimum combination of ductility, machinability, and dimensional and structural stability. The treatment also purposes increase strength; optimize unique properties such as fracture toughness, fatigue strength, and high-temperature creep strength [ref]. The research beginning from the literature study aims to obtain insight from reported articles relevant to our future research topics. In several studies found in online sources, such as scientific journals, websites, and news from trusted sources, only five articles' results analyses were reviewed. It is because of the research relevancy, and they could represent other articles we found on the websites. The rest of the articles we cited are relevant for enriching our knowledge in this topic area.

The heat treatment, found by Sarcombe (Sarcombe, et al., 2008), consisted of a moderate cooling rate after solution treatment at 1,055°C (above the β transus) producing a homogeneous structure with a morphology that depended on the post-solution treatment cooling rate. The cooling method following the Ti-6Al-7Nb heat treatment is closely related to the microstructure, and the microstructure after furnace cooling is larger than that after air cooling (Sarcombe, et al., 2008).

The properties of materials are dependent upon their structural aspects. The structures may be of different scales of magnitude, from macrostructure to atomic structure. In general, heat treatment of metals and alloys concerns the change in microstructure (Banerjee, 2017). Gallego et al. investigated the effect of equal channel angular pressing on Ti-6Al-7Nb alloy's microstructure after implementing the thermomechanical process on the Ti-6Al-7Nb. The microstructure consists of ultrafine grains ranging from 200 to 400 nm. There was also some evidence of grains with unfavourable orientation to deformation. These grains potentially act as rigid bodies and concentrate the deformation in their surrounding areas as an "open-die grain" mechanism. They also stated that such a deformation mechanism could be attributed to the differences in the plastic behaviour between the alpha and beta titanium phases (Gallego, et al., 2012). The alloying elements affect the structure and properties of titanium alloy. In Titanium alloys, in addition to the type of alloying elements, the microstructure possessed by Titanium alloys affects the performance of the material, through a mechanical process followed by heat treatment, the microstructure of the Ti-6Al-7Nb alloy can be controlled and varied. The process mentioned above is also called the thermomechanics process (Sutowo, et al., 2017). Titanium alloy has four typical microstructure characteristics: equiaxed, basket, dual-phase, and widmanstatten structure. Different phase transformations form different microstructures leading to the various mechanical properties (Damisih, et al., 2018). For example, the plasticity and fatigue strength will change correspondingly as the content variation for primary α phases in the dual-phase alloy. However, the widmanstatten microstr cture with high fracture toughness and high section shrinkage should be avoided as much as possible due to its poor plasticity and corrosion resistance. Furthermore, the strength and plasticity of the equiaxed and the dual-phase structure are better than those of the lamellar structure (Gallego, et al., 2012).

At this stage, which is the core of the discussion in this literature review, the results of the data discussion and analysis of heat treatment carried out by several authors are presented. The purpose of heat treatment on Titanium alloys, especially Ti-6Al-7Nb, each experimental step and the research results are explained here in tabulated form. The goal of tabulating the analysis results related to this heat treatment is to facilitate the identification of the effectiveness of the repair work on the Ti-6Al-7Nb material using heat treatment. It was reported, for instance, that the analysis results on Ti-6Al-6Nb Titanium alloy through solution treatment with different temperature variations gives results (Gallego, et al., 2012):

- The microstructure of the solution treatment results is primer equiaxial in the matrix; the difference is the grain size formed. The higher the solution treatment temperature, the larger the grains formed.
- At a solution treatment temperature of 850°C, grains are ±10.02µm in size. For solution treatment temperature 950°C grains larger, that is, ±10.37µm. The largest grain size was owned by the sample that underwent solution treatment at a temperature of 1050°C, ±12.01µm.
- The higher the solution treatment temperature, the strength of the Ti-6Al-6Nb alloy decreased due to the larger grains produced and the less intensity of the phase. In addition, the high strength of air cooling is not due to the formation of martensite but because the phase has enough time to change to phase.

Another example of a heat treatment procedure reported in the literature is that the heat-treated specimens in a tubular shape of the furnace, and the furnace temperature accuracy was controlled to within $\pm 2^{\circ}$ C. The specimens were introduced at room temperature and heat treated along with the furnace to the required temperature. Some different heat treatments were given to the specimen cut from the rolled sheet. After one hour, the specimens were cooled at various rates, water quenching, air-cooling and slow furnace cooling. The heat treatments were carried out in an inert argon atmosphere. The waterquenched and air-cooled specimens were subjected to ageing treatment in the open-air furnace at 550°C for 4 hours.

The heat treatment process of the Ti-6Al-7Nb and analysis results are summarized in Table 1 below.

Table 1: The effect of heat treatment temperatures on phase transformation, grain size and shape changes, and mechanical/ tribological properties variation

Heat Treatment Methods	Change in Phase / Microstructure	Affect on Mechanical/ Tribologicsl Properties
The Ti-6Al-7Nb alloy specimens were heated to 970°C, soaked for 1 hour, cooled in water or oil at 20°C, and ageing at a temperature of 450 and 650°C for 5 hours. Specimens were air cooled after ageing .	There is phase transformation/ There is a change in grain size	Change in mech properties (Hv) Affect tribological behaviour
Three solution temperatures (namely 850°C, 930°C and 950°C) were used.	A solution annealing in the $\alpha + \beta$ phase field followed by ageing was carried out.	Hardness: Wear resistance:
Raw materials were used for melting consisted of titanium sponge, and aluminum-niobium master alloy (6%Al-7%Nb). The ingot was obtained in the form of pancake of 600 gm in weight (as cast alloy). The ingot was subjected to deformation (hot rolling) in the in $\alpha + \beta$ phase field (950°C) _(Ajeel, et al., 2007)	Hot rolling of Ti-6Al7Nb alloy at 950°C shows microstructure consisted of globular and acicular a grains (white grains) within a β transformed matrix containing equiaxial grains (dark grains)	N/A
Ti-6Al-7Nb alloy, the heating rate was 20/30 (K/min); the samples were heat treated under vacuum; samples were single solution treated (SST) at 930°C for 1h, heated 830°C for 2 h; Specimens received a precipitation treatment (Ppt) for 6 h at 600°C (Fityan, et al., 2017)	 α-phase became a little coarser after double solution treatment. No martensite was observed after double solution treatment. No much difference was observed in the grain size of a-phase 	The solution treatment could improve the hardness and wear resistance of the alloy
Hot rolling of Ti-6Al-7Nb alloy at 950° C shows microstructure consisted of globular and acicular α grains (white grains) within $\alpha \beta$ -	The XRD analysis of Ti- 6Al-7Nb alloy shows a slight change in the 2θ value of α phase reflections of Ti-	The mechanical properties of these alloys are very sensitive to the

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Heat Treatment Methods	Change in Phase / Microstructure	Affect on Mechanical/ Tribologicsl
		Properties
transformed matrix containing equiaxial grains (dark grains). In the region where deformation was intense, the microstructure of α was elongated with flow lines and this is also confirmed by (Ajeel, et	6Al-7Nb alloy, heat treatment in $\alpha + \beta$ region resulted in recrystallization of the α into an equiaxed morphology designated as primary α in transformed β .	microstructure. The XRD analysis of Cp Ti, Ti-6Al-4V and Ti-6Al-7Nb alloys indicates the presence of a and β
al., 2007). Ti-6Al-6Nb Titanium alloy was preserved by heat and solution treatment with different temperature variations: below T_{β} (850°C), close to T_{β} (950°C), and above T_{β} (1050°C) with a holding time of 1-hour air cooling_(Sutowo, et al., 2017).	The Ti-6Al-6Nb alloy appears to have two phases: α and β . The α phase is lamellar in shape; between these lamellar structures, there is a β phase. In grains containing the prior β phase, colonies of α and β phases are formed, having α lamellar with the same crystallographic orientation.	hases Hardness: Wear resistance:
Selective Laser Melting, Solution treatment at 955°C (below the ß transus), and Solution treatment was performed at 1,055°C (above the ß transus) (Sercombe, et al., 2008)	α 'martensitic structure in a metastable β matrix/ a homogeneous structure was produced, with a morphology that depended on the post-solution treatment cooling rate.	N/A

SUMMARY

The heat treatment results for three different Ti-6Al-7Nb production methods have been studied. Some researchers reported that heat treatment methods manipulated the microstructure in a Ti-6Al-7Nb alloy by reducing the amount of porosity contained in the materials. From this result, the source of preferential crack nucleation and propagation can be minimized; moreover, factors that are the potential to induce the reduction of the fatigue life span can be avoided.

The heat treatment of titanium demonstrated significantly reduced residual stresses. Moreover, heat treatment provides an ideal combination of

ductility, machinability and structural stability. It is due to the differences in microstructure and cooling rates between α and β phases.

The cooling rate has an impact on the morphology of the transformed α . The transformed α increases in thickness and length if the cooling rate is reduced. The α colony size is the most critical microstructural property due to its influences on the fatigue properties and deformation mechanics of β processed α + β alloys. The morphology of the α phase in the β matrix, volume fraction, and distribution of titanium alloys can be controlled by adjusting the temperature, time, and rates in the heat treatment process.

ACKNOWLEDGEMENT

We would like to express our gratitude to the institution that houses the researchers, namely the National Research and Innovation Agency, Republic of Indonesia, which has made the institution a shelter for researchers and innovators in carrying out their duties to create and apply research and innovation results to make Indonesia more advanced. Thanks are also conveyed to the THA and biocompatible team, who have worked together to improve the quality of research results so that they can be applied in the industry.

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