Characteristic of Ti-6Al-6Nb alloys following solution treatment with cryogenic cooling for implant applications

F Rokhmanto¹, H Arief², Alfirano², and C Sutowo¹

¹) Research Centre for Metallurgy and Material - Indonesian Institute of Sciences Bd. 470 Kawasan Puspiptek Serpong, Setu – Tangerang Selatan.
²) Teknik Metalurgi, Fakultas Teknik, Universitas Sultan Ageng Tirtayasa, Cilegon, Indonesia

E-mail: fendy.rokhmanto@gmail.com

Abstract. Titanium alloys are widely used as implant materials, due to the mechanical properties, one of which is the low elastic modulus. The differences in the elastic modulus of Titanium alloys and Bones are wide enough, therefore it’s necessary to investigate how to obtain a lower Titanium elastic modulus. In this paper used Ti-6Al-6Nb alloys as an implant material. The Ti-6Al-6Nb alloys Solution Treated at 1100 °C, following cryogenic cooling with liquid Nitrogen. Before that The Ti-6Al-6Nb alloys Homogenized at 1100 °C for 12 hours, then Hot rolled with preheated at 1100 °C for 30 minutes. As comparison Solution Treatment also done in the ice water cooling and air cooling. The Ti-6Al-6Nb alloys characterized with a hardness test, metallographic and ultrasonic test to obtain the elastic modulus. The analysis shows that the lowest elastic modulus was 100.19 GPa and highest hardness was 52.54 HRc is in the cryogenic cooling process due to the beta phase formed.

1. Introduction

Titanium alloys especially alpha-beta Titanium widely used as implant materials because of an excellent corrosion resistance, an excellent biocompatibility and good mechanical properties, such as the low elastic modulus. Ti-6Al-4V known as commercially alpha-beta alloys implant material, but Vanadium is also known as a toxic element and caused allergic in the human body [1,2]. Vanadium in Ti-6Al-4V is a beta phase stabilizer, while Aluminum is an alpha phase stabilizer [1]. Therefore Vanadium as a beta phase stabilizer should be substituted, for example with Molybdenum or Niobium [3].

The mechanical properties, such as hardness, strength, and elastic modulus is depend on the alloying materials and the microstructure. Where to get the suitable mechanical properties, such as hardness and elastic modulus, material can be heat treated above the temperature alpha-beta region (beta transfuse temperature) [4]. The beta transfuse temperature of Ti-6Al-4V is 995 °C [5].

In previous studies of Ti-6Al-6Nb, the influence of solution treatment temperature on mechanical properties was studied [6], but the influence of cooling medium after solution treatment on mechanical properties has not been studied. In previous studies of Ti-6Al-6Mo, the elastic modulus affected by cooling medium [7], but the cryogenic cooling has not been studied. In this paper will be studied the influence of cryogenic cooling after solution treatment on the characteristic of Ti-6Al-6Nb for implant applications.
2. Experiment
In this paper, the composition of titanium alloys are 6% Al, 6% Nb (wt.%), the material purity, that is Titanium, Aluminum and Niobium is 99.9%. The materials, re-melted used vacuum arc furnace in the argon atmosphere. The alloys ingot dimension is ø 7.5 x 80 mm. The ingot homogenized at vacuum tube furnace at 1100°C for 12 hours and furnace cooling in the argon atmosphere. After that, thermomechanical treatment held with hot rolled up to 80% reduction, with preheating at 1100°C for 30 minutes. The solution treatment held at 1100°C for 1 hours, followed by quenching with various media, liquid Nitrogen (cryogenic cooling), water, and air cooling. The Ingots, characterized with a hardness tester, an optical microscope and SEM-EDX to investigate microstructure, and the elastic modulus obtained with an ultrasonic approach by measuring ultrasonic velocity (ASTM E494-95).

3. Result and Discussion
Figure 1. shows the microstructure of Ti-6Al-6Nb alloy in various condition, it has two phases, an alpha phase (light) and a beta phase (dark). Figure 1a. is the microstructure of the as cast, the structure is a lamellar or needle-like structure. The structure called needle-like because of the shape are like a needle (lujering). Figure 1b, 1c and 1d show the microstructure in condition after solution treatment with a various cooling medium. In the air cooling medium the structure is equiaxed or globular, but in the faster cooling, water quenching (Figure 1c) and cryogenic cooling (Figure 1d), the globular structure looks flatter and along shape.
Figure 2. Microstructure of Ti-6Al-6Nb in condition, (a) As cast, (b) Air Cooling Solution Treatment, (c) Water Cooling Solution Treatment, (d) Air Liquid Nitrogen Solution Treatment

Figure 2. is an SEM picture of Ti-6Al-6Nb alloy in various condition, it has two phases, an alpha phase (dark) and a beta phase (light). The lamellar structure observed in as cast condition as shown in Figure 2a. However the equiaxed structure observed in the SEM picture after solution treatment with air cooling is Figure 2b, water quenching is 2c and cryogenic cooling is 2d. The SEM shows higher magnification than optical microscope. In rapid cooling condition (Figure 1c and 1d) the equiaxed or globular structure looks flatter and along shape, however when observed with higher magnification under SEM looks the equiaxed structure in a smaller shape (Figure 2c and 2d) than air cooling (Figure 2b). Figure 2b, 2c and 2d show the beta phase dominates the microstructure and the grain size structure became smaller when the cooling rate is faster. The cooling rate was prohibits the beta phase transform to alpha phase, because of that the alloys dominated by beta phase. The alfa phase is also formed, it is promoted by the Aluminum as known as alfa stabilizer.

Figure 3. shows the hardness of Ti-6Al-6Nb alloy in various condition. The as cast ingots have the highest hardness number 54.2 HRC, after solution treatment process the hardness is decreased. The cooling rate after the solution treatment process enhanced the hardness from 49.9 HRC in air cooling became 50 HRC in water quenching and 52.5 HRC in cryogenic cooling (liquid Nitrogen cooling). The highest number in as cast ingot promoted by the acicular structure (needle-like) that similar with martensitic structure in steel, besides that in this condition the microstructure dominated by an alpha phase (Figure 1a and 2a) that had HCP crystal structure [8]. The enhanced hardness number after the solution treatment process was promoted by the grain size structure. The smaller grain size enhanced the hardness although the Ti-6Al-6Nb dominated by a beta phase that had BCC crystal structure [8].
Figure 4. Elastic modulus of Ti-6Al-6Nb

Figure 4. shows the elastic modulus that obtained with an ultrasonic approach by measuring ultrasonic velocity that calculated with formula below: (ASTM E494-95)

\[ E = \frac{\left[ \rho \left( V_T^2 \left( 3V_T^2 - 4V_L^2 \right) \right) \right]}{V_L^2 - V_T^2} \]  \hspace{1cm} (1)

\[ \frac{V_T}{V_L} = \sqrt{\frac{1 - 2\sigma}{2(1 - \sigma)}} \]  \hspace{1cm} (2)
Where:
\( \sigma = 0.36 \)
\( \rho \) = density (4.25 g/cm³)
\( VL \) = longitudinal velocity
\( VT \) = transversal velocity
\( E \) = elastic modulus

Figure 4. shows the elastic modulus had decreased when the cooling rate is faster. In the air cooling the elastic modulus is 106.7 Gpa, than decreased to 102.25 Gpa in water quench and became 100.2 Gpa in cryogenic cooling. The decreasing elastic modulus is inversely proportional to the hardness as shown in Figure 3. Thus it can be understood that the smaller grain size can decrease the elastic modulus, as well as the phase formed, the more beta phases are formed, the smaller the elastic modulus.

4. Conclusion
The microstructures, hardness and elastic modulus of Ti-6Al-6Nb in solution treatment process with cryogenic cooling medium have been investigated. From the investigated can be concluded that the alloys had equiaxed microstructures with alpha-beta phases that dominated by beta phase. As compared with air cooling and water quenching process, the cryogenic cooling had the smallest grain size structure, lowest elastic modulus 100 GPa and highest hardness 52.5 HRc

References
[1] Keizo H, et.al Nippon Steel Technical Report No 62, 1994.
[2] Mitsuo N., Elsevier Material Science and engineering A 243 231-236, 1998.
[3] Marsumi Y and Pramono A W., Research Centre for Metallurgy – Indonesian Institute of Sciences 2014.
[4] Sutowo C, et al. 2017 Prosiding Semnastek 1-6
[5] Obasi G C, et al. 2012 Acta Materialia 60 (17), 6013–6024
[6] Senopati, G, et al. 2018 AIP Conference Proceedings. 1964
[7] Rokhamanto, F. et al. 2018 AIP Conference Proceedings. 1964
[8] Matthew J. and Donachie, Jr, 2000 The Material Information Society, Second Edition

Acknowledgement
This paper financially supported by 2018 thematic program of Indonesian Institute of Sciences.