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Effect of solution treatment on the microstructure and mechanical properties of Ti-6Al-6Mo hot-rolled alloy

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ABSTRACT

Titanium and its alloys are commonly used in biomedical applications because of their unique combination of good hot workability, high specific strength and good corrosion resistance. In this study, elastic modulus, hardness and the effects of solution treatment on microstructures and mechanical properties of Ti-6Al-6Mo hot-treated alloy were investigated using Optical microscopy (OM), X-ray Diffractometry (XRD), Scanning electron microscopy (SEM), ultrasonic testing and hardness testing. Previously, the sampels were hot-rolled at 900^{o}C for 1 hour with a total reduction of 50%, and then followed by air cooling. The samples were carried out solution treatment for 1 h at 850^{o}C and 950^{o}C, followed by water cooling and at temperature of 1050^{o}C with water and air cooling. The results indicate that the hardness of the Ti-6Al-6Mo alloy increased with the increasing of solution treatment temperature on the water cooling, while the elastic modulus was decreased. The lowest elasticity modulus value and the highest hardness, 125.4 GPa and 46.3 HRC were obtained at 1050^{o}C with equiaxed micro structure and β' precipitate. XRD analysis exhibited the presence of α and β phases in Ti-6Al-6Mo alloys after solution treatment. Based on the relative intensity of XRD strongest peak analysis, the α phase intensity decreases with the increasing of solution treatment temperature. This phenomenon causes the decreasing of elasticity modulus value with the rising of solution treatment temperature on water cooling medium.

Keywords: Ti-Al-Mo; solution treatment; modulus elastic; α phase, β phase.

INTRODUCTION

Titanium and its alloys are receiving increasing research attention due to their excellence in specific strength, elastic modulus, corrosion resistance and biocompatibility compared to another metallic biomaterial alloys [1-7]. Besides being used for orthopaedic and dental in biomedical applications, titanium alloys as well as cobalt and stainless steel are also being used as materials for cardiovascular stent [8-11]. As one type of titanium alloys, the α+β alloys are widely used because of the various microstructures and mechanical properties can be achieved by elements alloying and thermo-mechanical treatments. Ti6Al64V is one type of α+β alloys that is commercially known as the main titanium alloys [12-16]. Because the toxicity of Vanadium has been reported, Vanadium-free titanium alloys like Ti–6Al–7Nb and Ti–5Al–2.5Fe have been recently developed by several researchers [17,18]. Moreover, metal elements such as Al, V and Fe have higher cytotoxicity based on Kawahara's study [19]. Similarly, because of the special mechanical properties such as super elasticity and shape memory effect, TiNi alloys have been clinical applied for years [20-22]. However, lately Ni Nickel is classified as a toxic and incompatible element [23,24].

Recently, two types of α + β type Ti-6Al-6Mo alloys are developed. This alloy has good hot workability, hardness, strength and good corrosion resistance. Molybdenum as substitute of Vanadium serves as a β stabilizer element which can increase strength and decrease the elastic modulus of the alloy [25]. In order to observel the microstructure of titanium alloys, most of the studies in α+β type alloy focus on the formation of the primary α phase. Hot rolling accompanied with post heat treatment is the most effective method. This method can be used for strengthening of alpha-beta titanium alloys and to refine its morphology [26,27]. The mechanical properties of Ti α+β type alloys can be controlled by solution treatment [28-30]. Solution treatment is the most common heat treatment method used to obtain Ti alloys with low elastic modulus [31-33]. Important parameters in the solution treatment process include temperature and cooling solution. From previous study, solution treatment temperatures affect the α phase volume fractions and affecting α lamellar phase width [34-36]. Hot deformation also affects the microstructures and mechanical properties. The increased percentage reduction of hot rolling produces elongated grain fibres containing fine-equiaxed sub grains [37-39]. Sub-grain size increases with increasing deformation temperatures and equiaxed fine grains formed with a heating temperature of 973 K. Tensile strength increases with increasing percentage reduction in hot rolling process [40,41]. An α+β type titanium alloys is the most widely used for medical application. The most important problem of this alloy is having a high elastic modulus. So that its use is replaced by β type titanium alloys. A β type titanium alloys that have the lowest elastic modulus are usually produced by adding a β phase-stabilizer elements. In this study, a solution treatment of alloy α+β type alloy is carried out to reduce the elastic modulus. Ti-6Al-6Mo alloy is studied to determine the effect of solution treatment on the microstructure and mechanical properties after deformation in α+β region. It is expected that solution treatments can reduce the elastic modulus as well as by addition a β stabilizer element to the titanium alloy.

METHODS AND MATERIALS

Table 1 shows the chemical composition of the alloy used in this study. Ingots of the alloy were re-melted eight times in order to get chemical homogeneity.

Table 1. Chemical composition of the alloy (% wt).

Al	Mo	Fe	Cr	Ti
5.76	5.10	0.067	0.13	balance

Plates with a thickness of 10 mm cut from the ingots were homogenized (HM) at 900ºC for 1 h in an argon (Ar) gas atmosphere, followed by furnace cooling. Then, the plates were hot-deformed at 900ºC until 50% reduction. Hot-deformed alloy were subjected to solution treatment at 850ºC, 900ºC and 1050ºC for 1 h in an argon gas atmosphere, followed by Water Cooling (WC); 850ºC, 900ºC and 1050ºC and Air Cooling (AC); 1050ºC. The surfaces of the specimens were mechanically ground with emery paper and polished with alumina paste to a mirror finish and etched in Kroll solution (85% distilled water, 10% HF dan 5% HNO_3). An optical microscope and a scanning electron microscope were used to observe the microstructures of alloy. An energy dispersive X-ray spectrometer was used for the compositional analysis of the microstructures. The phases of the heat-treated alloy were identified using X-ray diffraction and the XRD patterns were obtained by Cu Kα radiation. The hardness was determined by Rockwell C hardness tester with an applied load of 150 kgf with six measurements for each sample. Ultrasonic tests were performed on hot-rolled alloy before and after solution treatment to determine the elastic modulus (E) of the alloy using the thickness gage of DM4 DL and DA301 probe. Based on the ultrasonic test, ultrasonic longitudinal velocities and shear wave velocities will be obtained. The relationship between ultrasonic longitudinal velocities (V_L) and shear wave velocities (VT) with elastic modulus values is shown in Eqn. (1) and the relationship between ultrasonic longitudinal velocities (V_L) and shear wave velocities (V_T) with Poisson ratio (σ) is shown in Eqn. (2) [42].

$$E = \frac{\rho V_T^2 (3V_L^2 - 4V_T^2)}{V_L^2 - V_T^2} \tag{1}$$

$$\frac{V_T}{V_L} = \sqrt{\frac{1-2\sigma}{2(1-2\sigma)}} \tag{2}$$

RESULTS AND DISCUSSIONS

In Figure 1(a), the microstructure of the Ti-6Al-6Mo as-cast alloy was a Widmanstätten type, consisting of two phases, α and β. Widmanstätten type structure was a microstructure with unequal α-phase growth direction. The microstructure of the Ti-6Al-6Mo homogenized alloy was a lamellar microstructure with α-phase with plate-like morphology formed at the β prior-β grain boundaries, as shown in Figure 1(b). The lamellar microstructure was obtained as a result of air cooling from the β phase (solid solution) to (α + β) phase. The transformation of β phase into α phase begins with the formation of nuclei at the β prior grain boundary. The microstructure of Ti-6Al-6Mo hot-rolled alloy (perpendicular from rolling direction) was the α plate-like deformed phase in the (α + β) phase matrix, as shown on Figure 1(c).

The microstructure of Ti-6Al-6Mo of alloy solution treated resulted at 850˚C/Water Quenching (WQ) shows the existence of α phase with plate-like shape which has elongated during hot rolling in (α + β) phase matrix (Figure 2(a)). Solution treatment performed at 950˚C WQ reduced the number and also increase the size of α-plate-like phase in the α + β matrix as well as the formation of β-phase precipitate (Figure 2(b)). In the water quenched and air-cooled specimens, when the temperature was raised from 850˚C to 950˚C, it tends to decrease the volume fraction of primary a and modify the matrix morphology.

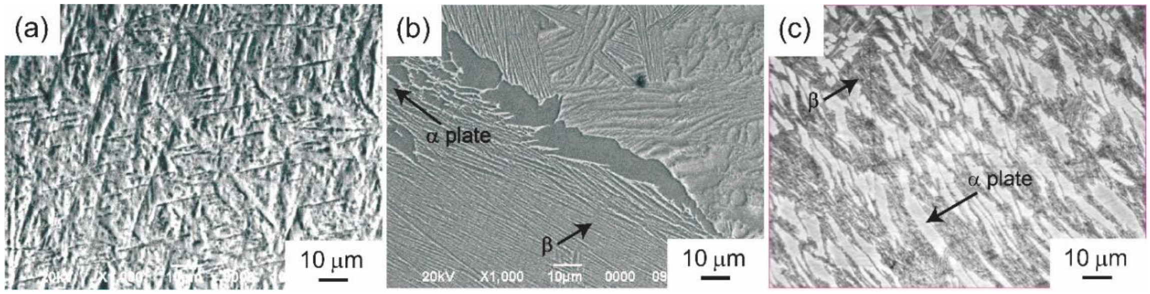

Figure 1. SEM images of Ti-6Al-6Mo (a) as-cast (b) homogenized and (c) hot-rolled alloy.

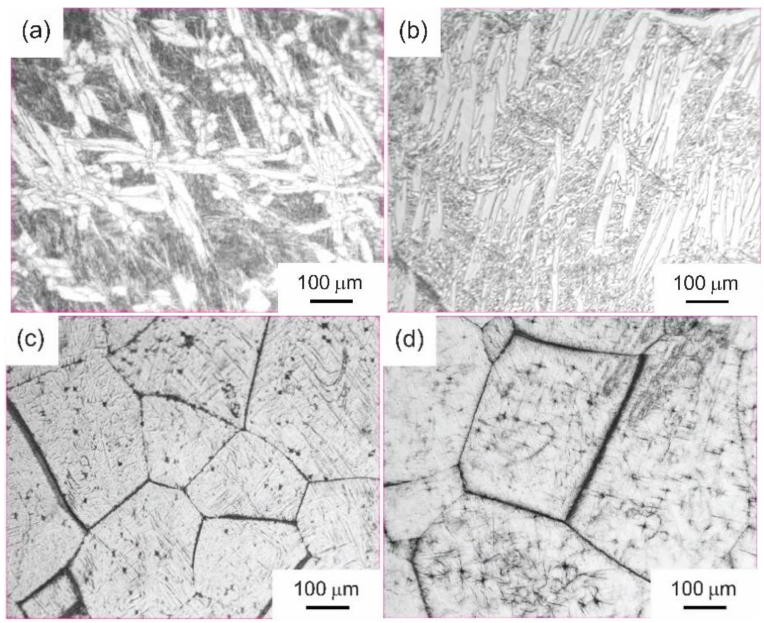

Figure 2. Optical microscope images of Ti-6Al-6Mo alloy after solution treatment (a) 850˚C/WQ, (b) 950˚C/WQ, (c) 1050˚C/WQ and (d) 1050˚C/AC.

Borradaile et.al. [34] confirmed that both solution treatment temperature and the cooling rate from the solutionizing temperature determine the primary a volume fraction matrix. Solution treatment at 1050˚C/WQ gives an equiaxed microstructure of β retained and α matrix (Figure 2(c)). The β phase appears as a precipitate along the grain boundary. Meanwhile, solution treatment at 1050˚C/ Air Cooling (AC), β phase formed along the grain boundaries and tends to be uniformly arranged in the β retained and α matrix (Figure 2(d)).

Figure 3 shows the XRD pattern of before and after solution treatment found the existence of peak shifting in each process. This shifting occurs due to the element of interstitial and substitution on the crystal structure that changes the parameters of the crystal lattice. The diffraction peaks indicate the presence of α and β phases with the relative intensity of each phase shown in Figure 4, obtained from the calculation of the highest diffraction peak ratios of each phase. The highest intensity of α phase at temperature of 850˚C/WQ is 47.41%, because the heating temperature is still under β-transus, so α phase has not passed allotropic transformation into β phase yet.

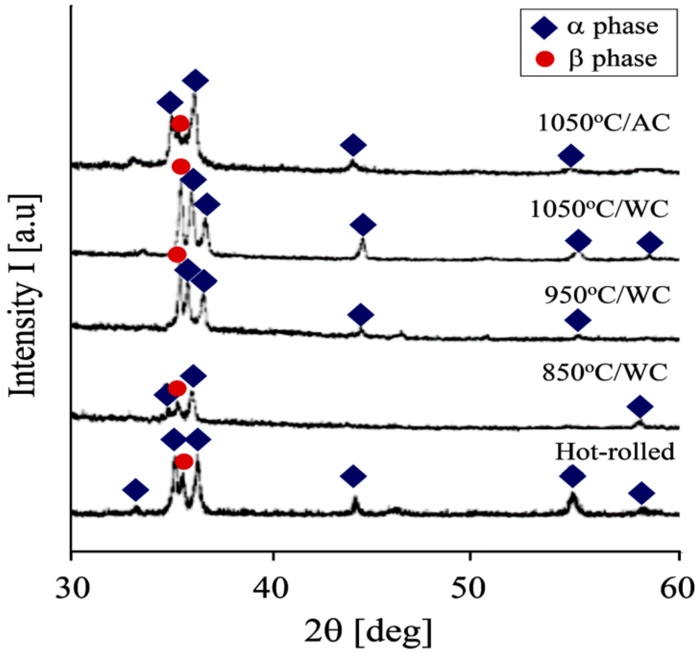

Figure 3. XRD patterns of Ti-6Al-6Mo alloy before and after solution treatment.

The intensity of α phase decreases with raising solution treatment temperature, with increasing solution treatment temperature, the more α phases that undergo allotropic transformation into β phase when heating. The highest intensity of β-retained phase was obtained at solution temperature of 1050˚C/WQ, which was 67.08%. Increasing solution treatment temperature produce more α phases which transformed into β phase. This is agreed with the results of EDS testing in which the increase in the number of phases β was indicated by the presence of Mo in precipitates, as shown in Figure 5. The composition of Ti, Al, and Mo on precipitate was 90.1, 3.38, and 6.52 (in mass %), respectively. Mo is a β phase stabilizer. With high solution temperature and rapid cooling, it did not give enough time for β phase to transform into α phase.

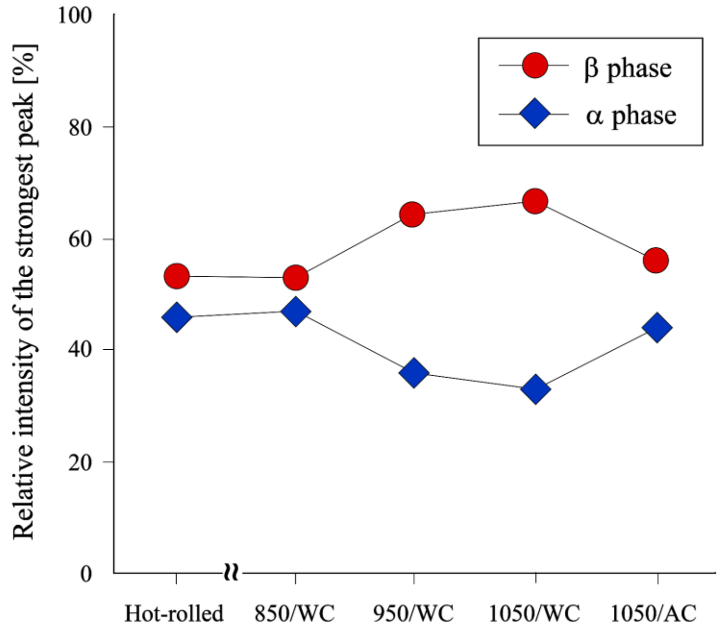

Figure 4. Relative intensities of the strongest peaks in the XRD patterns in the Ti-6Al-6Mo alloy before and after solution treatment.

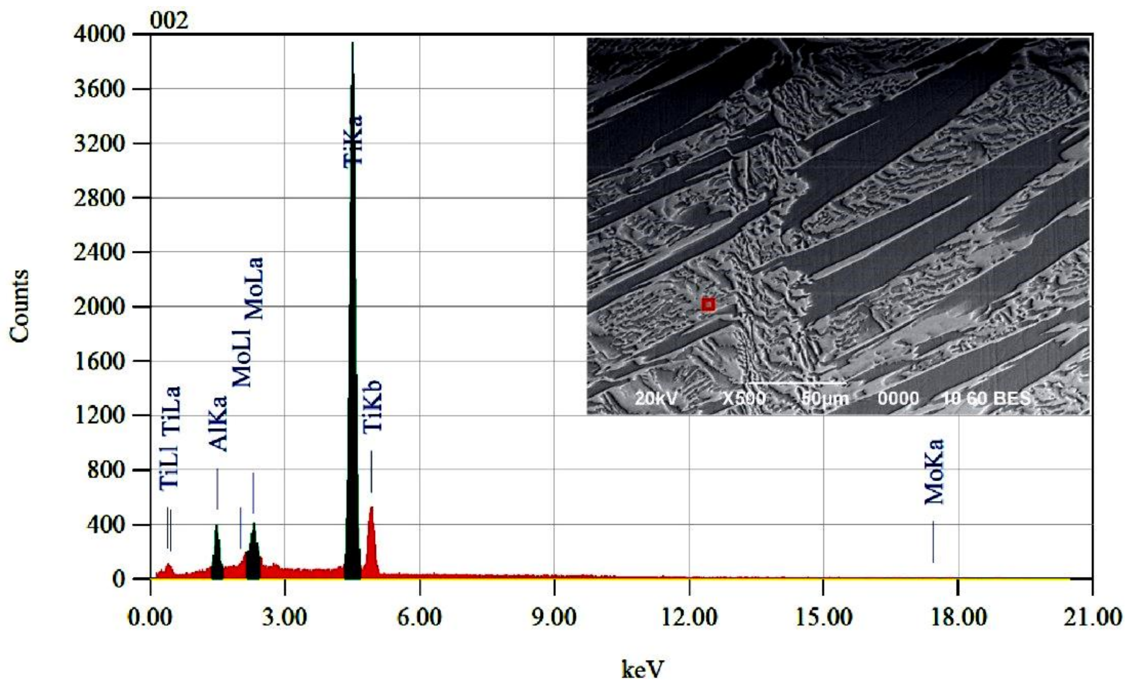

Figure 5. EDS analysis of Ti-6Al-6Mo alloy after solution treatment of 950˚C/WQ.

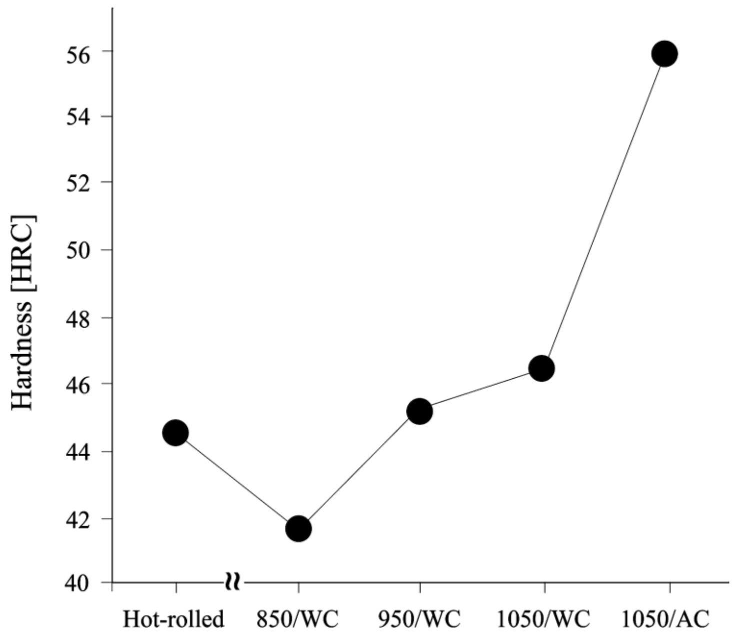

Figure 6. Effect of solution treatment on the hardness of Ti-6Al-6Mo alloy.

The hardness of α phase is higher than that of the β phase [43], meanwhile β phase has a higher hardness than the α' and α" phases [44-46]. Thus it can be concluded that α phase has the highest hardness compared with other phase's hardness. When the intensity of α phase was decreases, the hardness of Ti-6Al-6Mo alloy will also decrease. According to Figure 6, there is an increase in the hardness value after solution treatment at 950˚C/WQ, 1050˚C/WQ and 1050˚C/AC temperatures even though α phase intensity decreases, due to the formation of β-phase precipitate in the β and α matrix. The hardness value of Ti-6Al-6Mo alloys at 1050˚C/AC solution treatment temperature was higher than that of Ti-6Al-6Mo alloy at 1050˚C/WQ, around 55,7 HRC. This was caused by increasing in α phase intensity and formation of β-phase precipitate along grain boundaries. This tend to be uniformly arranged in the β retained and α matrix at the solution temperature of 1050˚C/AC. The precipitates will obstruct the movement of the dislocations. It needs high stress to obtain plastic deformation of the alloy. Thus, the hardness value of the Ti-6Al-6Mo alloy will increase [47].

Figure 7 shows the modulus value of elasticity and phase intensity of Ti-6Al-6Mo after solution treatment. The intensity of α phase in solution treatment 850˚C/WQ is the highest among other solution treatment temperature, that is 47,41%. The α and ω phases have a higher elastic modulus than the β phase. According to Hon et al. [48], the β phase exhibits a much lower elastic modulus as compared to the α phase, Eα = 1,5 Eβ. The elastic modulus of the phases in titanium alloys increase in order β < α" < α < ω [48-50]. Meanwhile, in solution treatment 950˚C/WQ, there was a decrease in elasticity modulus value to 129.26 GPa. Decrease in elastic modulus occurs with the decreasing of α phase intensity in solution treatment 950˚C/WQ to 35.89%. The decrease in α phase intensity can be seen in Figure 7 which shows the decrease of peak pattern of α phase diffraction along with the increasing of solution treatment temperature with water cooling medium. The lowest elasticity modulus value was obtained after solution treatment at 1050˚C/WQ because it has the lowest phase α intensity among other solution treatment temperature, that is 32,92%. At the same solution treatment temperature, which is 1050°C using air cooling, the elasticity modulus value increases significantly to 134.41 GPa. This phenomenon occurs because the intensity of α phase increases to 44.18% caused by a slower cooling rate when compared to water cooling. The low cooling rate will provide an opportunity for the β phase (when heated) to transform into α phase at room temperature [47].

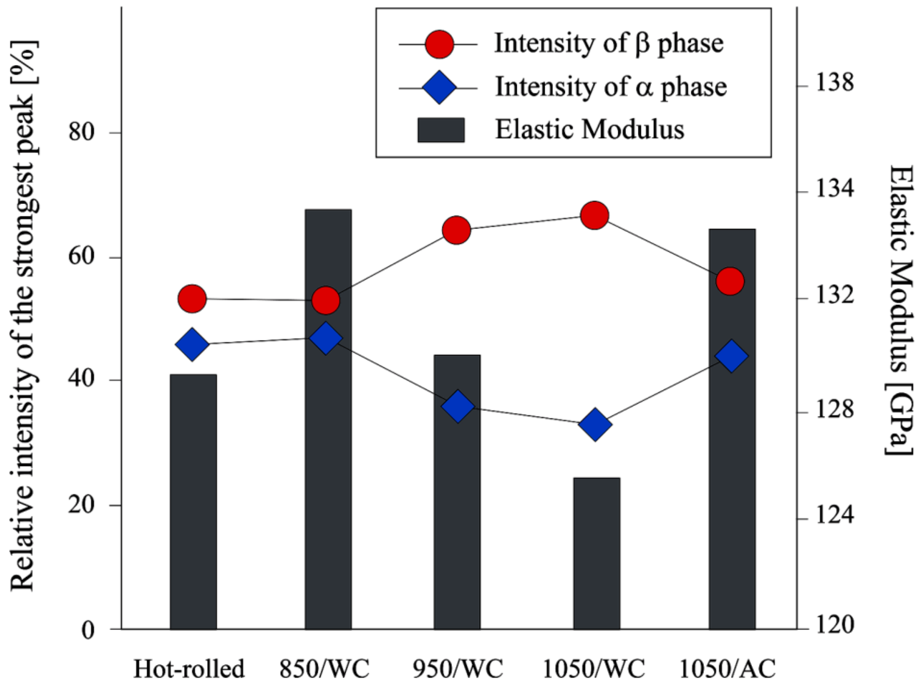

Figure 7. Elastic modulus of Ti-6Al-6Mo alloy before and after solution treatment.

CONCLUSIONS

Solution treatment solution treatment effected to the microstructure changes. The higher solution temperature approaches β-transus, reduces the number of α-grain granules and increases the grain size α in the α + β matrix. While at solution temperature above β-transus resulted in equiaxed micro structure with β retained and α matrix and β phase appear as precipitate in grain. The elasticity modulus value decreases with the increasing of solution treatment temperature on the water cooling medium because α phase intensity decreases with the increasing of solution treatment temperature with water cooling medium. Air cooling has a significant effect on increasing alloy hardness after solution treatment because of increasing in α phase intensity and formation of β-phase precipitate along grain boundaries.

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