

PHASE AND MECHANICAL CHANGES OF Ti5Al4V ALLOY AFTER HEAT TREATMENT IN VACUUM

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Abstract

Exploration of biocompatible materials with improved mechanical properties imposes a change in the chemical composition of the ($\alpha+\beta$) titanium alloys used in the modern implantology. The increased quantity of some toxic alloying elements could eventually cause undesirable biological effects in the human body. The high Al content could lead to reducing implant in vivo survival. Simultaneously, the low Al concentration will decrease the mechanical strength of the alloy but the alloy is expected to have lower cytotoxicity and improved plasticity. The present study aims at determining the maximum vacuum heat treatment response at various cycles of solution treatment, water quenching and precipitation of Ti5Al4V alloy. For this purpose, the critical limits for the heat treatment technological parameters were defined according to the XRD analysis and Vickers hardness test results and they were taken into account for determining the optimum heat treatment process.

Keywords: ($\alpha+\beta$) titanium alloys, vacuum heat treatment, XRD, hardness

1. INTRODUCTION

The major drawbacks of the titanium alloys used as orthopedical materials are that they are relatively soft and have poor wear resistance [1]. It is possible to increase the mechanical strength by controlling the composition, grain size, and microstructure of the alloy. A way to influence the microstructure and phase composition of ($\alpha+\beta$) titanium alloys is to apply different heat treatment cycles for achieving different properties for a certain application. The β -transus temperature of a particular alloy depends on the content of the alloying elements and their distribution between both α and β phases of the Ti alloy. The decrease in the content of the α -strengthening element Al as opposed to conventional Ti6Al4V alloy will reduce the hardness as Al is a solid solution hardener but simultaneously, the alloy is expected to have lower cytotoxicity and improved plasticity as well as low α -phase precipitation hardening ability at low-temperature precipitation. The isomorphous β stabilizers like V, Nb, and Mo depress the transformation temperature and increase the corrosion potential because β -phase is known to be nobler than α [2]. Mo and Nb are used to increase the strength and to reduce the elastic modulus of the alloy [3]. V is not found to participate in the formation of the surface oxide film which even destroyed in the organism is afterward regenerated [4].

In contrast to the well-examined Ti6Al4V, the variation in the composition of the modified Ti5Al4V alloy will change the temperature of the solution treatment and that of the decomposition of the quenched structures. These changes are necessary to be examined in order to increase the strength of the implant material. The current work is aimed at determining the optimum heat treatment cycle for Ti5Al4V alloy by examining the mechanical properties, microstructure, and phase transformation.

2. MATERIALS AND EXPERIMENTAL PROCEDURES

Samples with dimensions 14×14×4 mm were cut out of 16 mm thick sheet material using the electro-erosion cutting method. The chemical composition of the alloy given in **Table 1** was measured by JEOL JXCA-733

Microprobe scanning electron microscope (SEM) coupled to a WDX detector. The focused electron beam with 50 μm diameter was operated at 19.9 kV for the acquisition of the chemical composition.

Table 1 Chemical composition of the Ti5Al4V alloy (wt.%)

Chemical element	Al	V	Fe	Mn	Co	Cr	Mo	Pd	Nb	Hf	Ti
Ti5Al4V	5.2	4.4	0.14	0.11	0.065	0.002	0.17	0.15	0.36	0.036	Bal.

The as-received alloy samples were single solution treated (ST) for 30 min at 890, 920 and 950 °C and water quenched (WQ) (**Figure 1a**). The specimens with the highest hardness received by the quenching were precipitated (P) for 4 hours at three different temperatures - 500, 520 and 540 °C and air cooled (**Figure 1b**). All treatments were carried out in vacuum ≤ 1 Pa. The surfaces of the samples were grounded and polished to be examined.

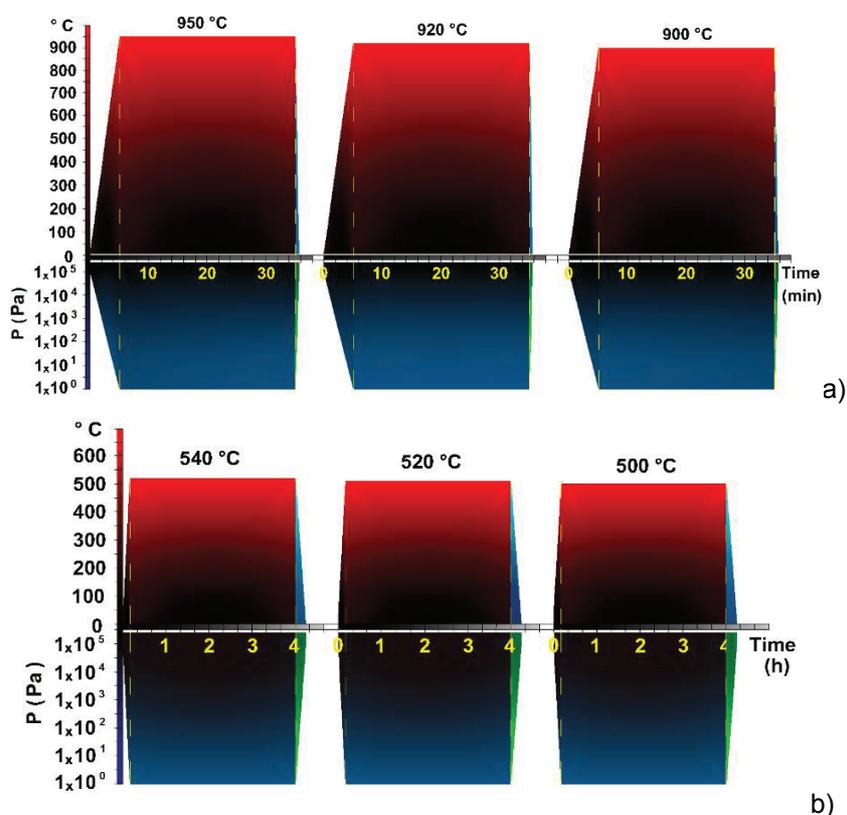


Figure 1 Cyclograms of the applied heat treatment processes: a) solution treatment; b) precipitation

A Vickers Hardness tester 432 SVD by Wilson-Wilpert was used for the hardness measurements of the alloy according to the standard ISO 6507-1. In order to compare the hardness changes, the research was made on the top (rolling direction) of each sample with a load of 10 kg and dwell time of 10 s. Ten sequential measurements by increments of 500 μm between the imprints were made. The light optical micrographs of the Kroll's reagent etched samples were obtained using a Nikon microscope with the adapted 14-megapixel digital camera. To ensure the comparability of the survey results for all samples, one and the same magnification was utilized. The X-ray phase identification was performed with URD-6 diffractometer, applying Bragg-Brentano geometry and $\text{Cu-K}\alpha$ radiation at 30 kV and 20 mA. The scanned diffraction angle range was $2\theta = 30\text{-}100^\circ$ with a step size of 0.05° and counting time of 2.5 s/step. The lattice response was measured in the rolling direction after a horizontal and vertical scan of each sample because of the initial texture of the alloy. The qualitative and quantitative phase analysis was held using Match!3 software.

3. RESULTS AND DISCUSSIONS

The maximum heat treatment response was evaluated by the changes in hardness values and microstructure. The strengthening results after solution treatment at three different temperatures are shown in **Figure 2**.

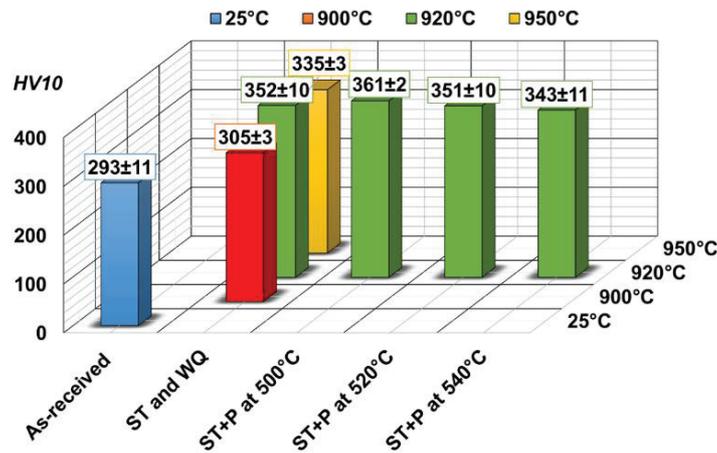


Figure 2 Comparison of the hardness values of the Ti5Al4V samples after ST at three different temperatures and WQ, as well as after P at three different temperatures. All measurements were made at the rolling direction (RD) of the material.

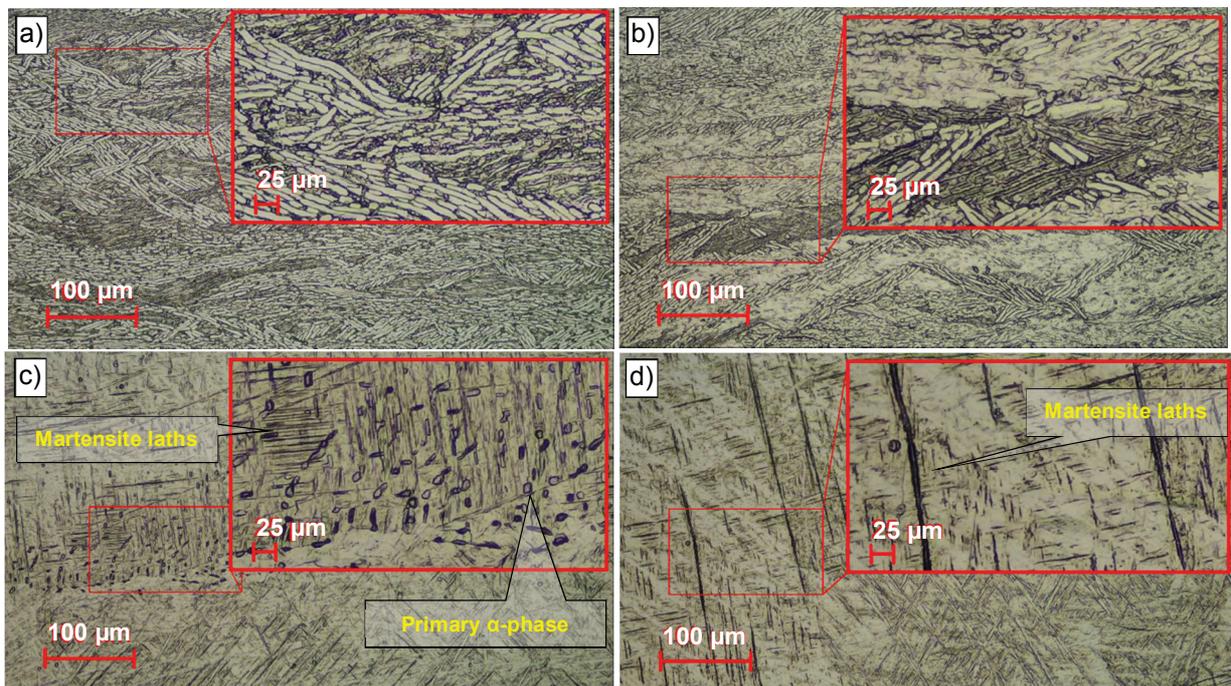


Figure 3 Micrographs of the Ti5Al4V alloy in as-received condition and (a) after ST for 30 min at 890 °C (b); 920 °C (c) 950 °C (d) WQ. Micrographs were made at the RD.

The decreased amount of Al and increased content of β-stabilizers were responsible for the lower hardness values measured within the alloy and consequently for the decrease in the elastic modulus. The welcome lower elastic modulus for biomaterials is known to be obtained after WQ of Ti alloys [5]. As seen in **Figure 2**, the 920 °C ST and WQ samples showed the highest hardness values and therefore, they were used for further

precipitation treatments. With the increase of the precipitation temperature from 500 up to 540 °C the hardness measured after the process decreased.

The heat treatment and the initial processing determine the microstructure and phase composition of the alloy. The initial coarse lamellar microstructure of the sheet material with distorted Widmanstätten α -plates in the prior β -grains is shown in **Figure 3a**. The lamellas inside the colonies were orientated in a parallel way and their texture followed the direction of the prior processing. According to M. Benedetti et al. [6], the lamellas have pronounced microstructure-sensitivity and show less fatigue resistance than the bi-modal structure. The small changes observed after solution treatment at 890 °C (**Figure 3b**) point out that the treatment was carried out in the low α - β range where α' -martensite was not formed. The transformed microstructure with the highest hardness shown in **Figure 3c** consisted of gross acicular narrow α' -martensite laths and low volume fraction of globular primary α phases. The areas containing α -grains probably included more α -stabilizing elements like Al. The obtained bimodal microstructure is more beneficial to fatigue strength because the ductile α -grains give higher resistance to nucleation of voids. The all β -range solution treated sample from 950 °C (**Figure 3d**) possessed enlarged grains with considerably coarser individual long α' -platelets. This structure was generally objectionable because of loss of ductility [7]. It is clear that the β -transus in the examined alloy is around 40-50 °C lower than that of the Ti5Al4V which is equal to 995 °C.

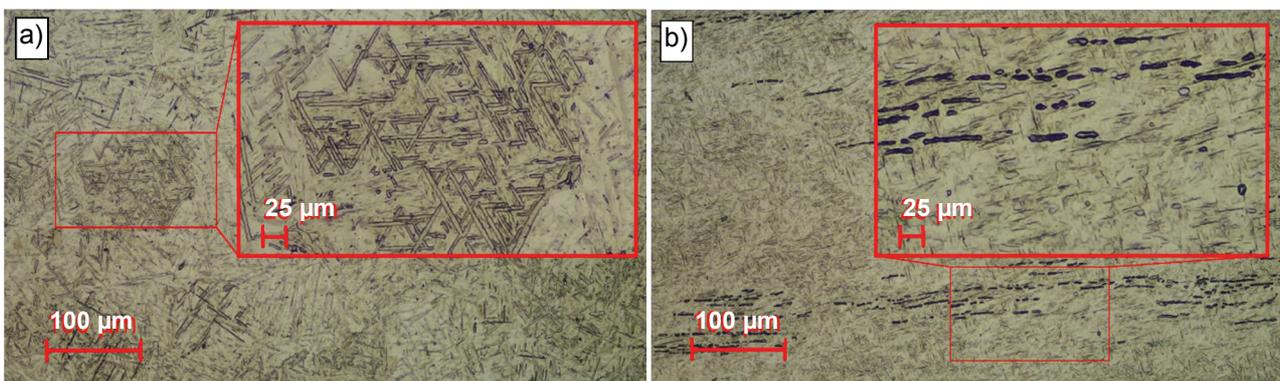


Figure 4 Micrographs of the Ti5Al4V alloy after ST for 30 min at 920 °C and P for 4 hours at a) 500 °C and b) 520 °C

An additional increment of strength was obtained by the alloy element partitioning effect when α' -martensite decomposes to the ordered Ti_3Al phase. This effect will be more pronounced for alloys with higher Al content which explains the slight hardness increase of Ti5Al4V alloy after precipitation. The micrographs of the precipitated samples at 500 °C and 520 °C are shown in **Figure 4a** and **4b**. Because of the submicron size of the precipitates, the latter was not clearly seen by optical microscopy. It is obvious that the coarse-grained structure was retained after the heat treatment process.

The XRD patterns of the as-received alloy (**Figure 5**) revealed the presence of α phase with a hexagonal crystal lattice and high volume fraction of retained β -phases (30%). After the ST and WQ, only α -phases were present because of the diffusionless $\beta \rightarrow \alpha'$ process. The primary α phase and α' -martensite had one and the same inter-planar spacing of the HCP crystal structure and, therefore, it was difficult to distinguish both phases and measure their relative percentage.

After ST and WQ the peaks shifted towards low theta values in all diffracted crystallographic planes after vertical scanning. The strongest shift was observed in (110) and (013) planes - between 0.2 - 0.22°, and the smallest one was seen in (101) plane - 0.11°. These changes could be attributed to the pre-saturation and therefore, expansion of the martensite unit cell because of the presence of dissolved in the transformed β -phase alloying elements. The increased inter-planar spacing was accompanied by hardness increase in the same direction. Compared to ST sample, the α -reflections of the ST+P sample shifted towards higher

diffraction angles and the (013) and (110) planes were highly affected. The smallest was the shift for the (100) and (002) reflections. This phenomenon suggests a contraction of α unit cell caused by the alloy element partitioning effect. At horizontal scan (parallel to the rolling direction), the reflections' shift was also towards the low diffraction values but the shift values were higher, especially in the (012) и (013). The smallest change was observed in the lower theta peaks - (100) and (002). After precipitation, the changes in structure led to a higher theta shift of the reflections compared to the ST sample. The highest change was measured in (012) and (013) planes and the smallest was again in (100) and (002) planes.

The peak broadening is mainly known to arise from the decreased size of the crystallites and microstrains in them. Because of the slight changes in the coarse prior β -grains that pre-determine the size of the martensite laths, the presence of wider peaks implies a larger share of microstrains in the broadening due to the fast cooling rate. In the case of the vertical scan, the full width at half maximum (FWHM) increased mainly for (002) and (012) planes after ST, while for the horizontal scan the (101) и (013) peaks were substantially broadened. For the ST+P sample, the precipitation decreased almost all FWHM values of the peaks as opposed to the ST specimen because of the alloy element partitioning effect.

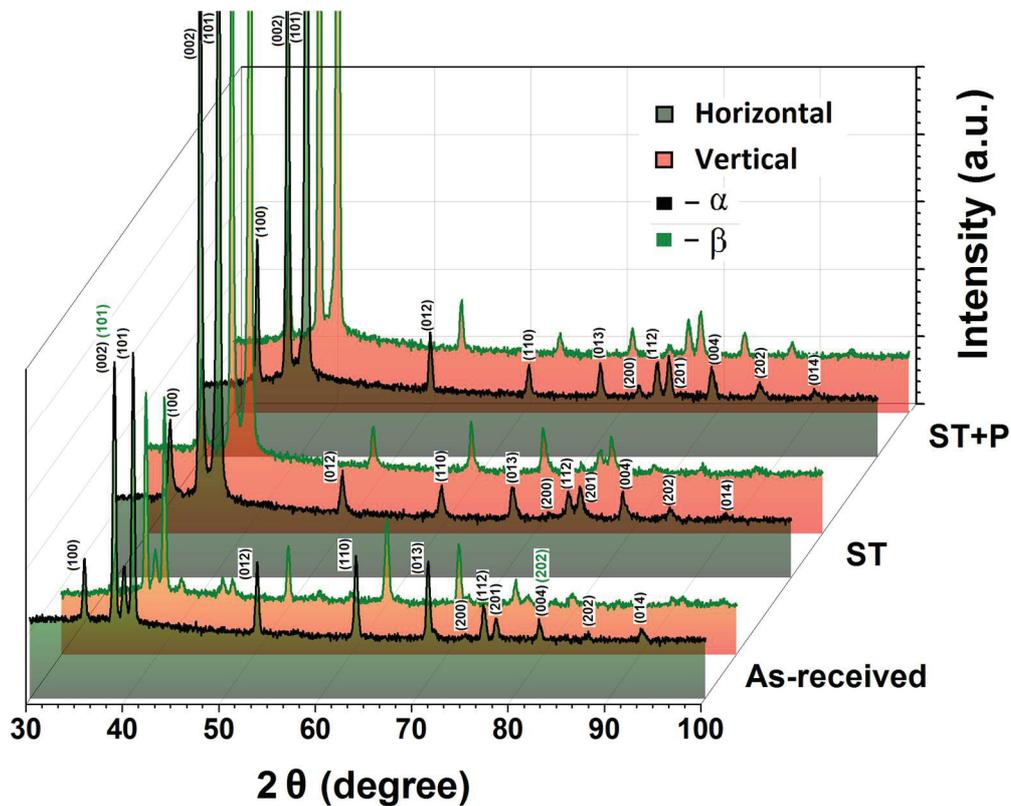


Figure 5 XRD patterns of the as-received, ST for 30 min at 920 °C and P for 4 hours at 500 °C samples scanned in horizontal and vertical direction of the RD

A variation in the texture distribution of the α -reflections with more pronounced increase in the (100), (002) and (101) peaks was also observed. After ST very strong increase in the intensity of (101) and (002) by 260 and 197%, respectively, followed by the (100) increment with 50% was seen in the horizontal direction of scanning. The result suggests the priority alignment of the transformed α' -phase in these particular planes. After precipitation, the highest was the increase in the (002) plane by almost 37% compared to the ST specimen followed by (100) and (101) plane where the increase was about 35 and 23%, respectively.

In the case of the horizontal scan, the introduced changes after ST were lower as the intensities of the (101), (002) and (100) increased by almost 111, 106 and 9 %, respectively. The texture of the precipitated sample

was slightly changed towards the ST sample particularly affecting the intensities of (100) and (101) that rose up with 44 and 31%, respectively, and (002) planes were almost not amended.

4. CONCLUSION

The modified composition of the Ti5Al4V alloy resulted in a change in the physical properties and strength of the alloy. The coarse lamellar structure and initial crystallographic texture contribute to a further reduction of the mechanical properties of the alloy. As the most important substitutional solid-solution straightener, the Al and its low content reflected the measured lower hardness values of the as-received and heat treated material as opposed to the conventional Ti6Al4V alloy. The β -transus temperature decreased by 40-50 °C compared to the Ti6Al4V alloy. It was established that grain refinement of the initial structure could not be obtained by the applied vacuum heat treatment. The maximum age hardening of the alloy was found to be achieved at 500 °C. After the solution treatment at 920 °C and water quenching the martensite was grown predominantly in the pyramidal (101) and basal (002) planes and less in the prismatic (100) ones. In contrast, the ordered structures separated during precipitation were basically formed in the prismatic (100) planes and basal (002) ones. The aging process gave a combination of higher strength and toughness with its tense microstructure that could improve the performance of the implant in vivo.

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