Influence of heat treatment on microstructure and mechanical properties of titanium alloy grade Ti6AL4V

R Bogucki1 and E Hytros1
1Institute of Materials Engineering, Faculty of Materials Science and Physics, Cracow University of Technology, al. Jana Pawła II 37, 31-864 Cracow, Poland

E-mail: rbogucki@mech.pk.edu.pl

Abstract: In this work, the effect of heat treatment on the control of the volume share of α phase in a two-phase titanium alloy α + β from the Ti6Al4V grade was analyzed. The applied heat treatments resulted in obtaining a volume share of alpha phase in the range of 60% to 96%. The highest hardness was obtained with a sample share 88% α phase, which was characterized by a microstructure consisting of grains of α phase and β phase regions of the phase precipitates of α'.

1. Introduction
Currently, an increase in demand for engineering materials characterized by high strength combined with low weight has been observed. These criteria is meet light metal alloys, which include titanium or aluminum alloys. Titanium has high strength, plasticity, corrosion resistance and low density. The most popular technical alloy of titanium is an alloy of the Ti6Al4V grade with a two-phase structure α + β, which has applications in various industries such as medical, aviation, automotive, space and sport[1-6].

Alloys with the highest mechanical properties currently include beta or pseudo beta alloys[7]. The chemical composition of these alloys makes it possible to control the volume fraction of the alpha phase by applying appropriate heat treatments. This translates into the ability to control the mechanical properties of these alloys in a wide range [7-9]. The widespread use of titanium alloys is limited by technological problems in its manufacturing and processing. Due to the strong affinity of titanium to oxygen, technological processes must be carried out under inert gas or vacuum. The occurrence of hexagonal crystal lattice also hinders plastic forming and forming of elements with complex shapes [3]. Machining is also difficult due to the low thermal conductivity of titanium [3,10]. Due to these problems, titanium alloys are used primarily in strategic industries such as aviation, the military where mechanical properties are the most important. In recent years, there has been a significant increase in interest in titanium alloys due to their unique properties. Work is also underway on defect machining techniques that will increase productivity while reducing costs [10-12].

In this work, an attempt was made to assess the impact of volume control on the alpha phase through the use of various heat treatment schemes on the mechanical properties of Ti6Al4V titanium.

2. Experimental procedure
For the research, a two-phase α + β titanium alloy grade Ti6Al4V was used, with the chemical composition shown in Table 1. The phase transition temperatures were determined on a DIL402C.
dilatometer from Netzsch. The beginning of $\alpha \rightarrow \beta$ transformation was determined at 893 °C and the end at 970 °C. Heat treatment was carried out in a Nabertherm P330 tube furnace under an argon atmosphere. In order to obtain a diversified morphology of the $\alpha$ phase, 6 schemes of heat treatment were performed, Table 2. The first three schemes consisted of heating the samples to a temperature of 1050 °C with a heating rate of 10 °C / min and annealing of 15 minutes, which provided a homogeneous $\beta$ structure. Then the samples were cooled with the oven to the following temperatures: 980 °C, 900 °C, 800 °C, held for 15 minutes, followed by cooling in water (scheme 1, 2 and 3). The next three heat treatment schemes consisted of single-stage heating at temperatures of 980 °C, 900 °C, 800 °C respectively (below the transformation temperature $\alpha \rightarrow \beta$), heating at these temperatures for a period of 15 minutes, followed by cooling in water (scheme 4, 5 and 6). The last heat treatment was aging for 8 hours at 500 °C. The microstructural observations were carried out on a JOEL JSM5510LV scanning microscope with an EDS attachment. Metallographic cuts were made on diamond pastes with a gradation of 6 and 1 µm and etched in Kroll's reagent.

The volume fraction of $\alpha$ phase was determined using the ImageJ program based on electron microscope images with a total analysis of the sample surface of 1 mm$^2$. The average results are summarized in Table 2. The analysis included only the $\alpha$ phase, ignoring the $\beta$ and ($\beta + \alpha'$) areas. This was dictated by the difficulty in separating individual phases, especially in the area of occurrence of fine dispersion particles of $\alpha'$ phase.

**Table 1.** Chemical composition of the Ti6Al4V titanium alloy.

| Element | Mass% |
|---------|-------|
| Al      | 5.5-6.75 |
| V       | 3.5-4.5 |
| Fe      | <0.30  |
| O       | <0.20  |
| C       | <0.08  |
| N       | <0.03  |
| H       | <0.008 |
| Ti      | residue |

Hardness measurements were made using the Vickers method, with a 30 kg load on a Hedort hardness tester. Twelve measurements were made for each sample. The average values are shown in Table 2. The tensile test was carried out at room temperature on the MTS CritritionTM Model 43 testing machine. The elongation and force measurements were recorded using extensometric sensors. Two cylindrical specimens with 5 mm diameter and 25 mm gauge length were prepared.

**Table 2.** Schemes of heat treatments.

| Sample | Type of heat treatment | Temperature domain | Average phase volume fraction $\alpha$ [%] | HV$_{30}$ | YS [MPa] | UTS [MPa] | Elongation $A_5$ [%] |
|--------|------------------------|--------------------|------------------------------------------|--------|--------|--------|-------------------|
| 1s     | $1050^\circ C/15$ min + | $\beta + (\alpha+\beta)$ | $\sim87.7$ | 573   | 1119   | 1216   | 16.7             |
|        | $980^\circ C/15$ min + |                    |                                          |        |        |        |                   |
|        | $500^\circ C/8$ h      |                    |                                          |        |        |        |                   |
|        | $1050^\circ C/15$ min + |                    |                                          |        |        |        |                   |
| 2s     | $900^\circ C/15$ min + | $\beta + (\alpha+\beta)$ | $\sim91.3$ | 487   | 1079   | 1188   | 6.4              |
|        | $500^\circ C/8$ h      |                    |                                          |        |        |        |                   |
|        | $1050^\circ C/15$ min + |                    |                                          |        |        |        |                   |
| 3s     | $800^\circ C/15$ min + | $\beta + (\alpha+\beta)$ | $\sim96.1$ | 456   | 1053   | 1167   | 4.7              |
|        | $500^\circ C/8$ h      |                    |                                          |        |        |        |                   |
| 4s     | $980^\circ C/15$ min + | $\alpha+\beta$     | $\sim60.1$ | 447   | 1089   | 1148   | 16.3             |


Results and discussion

The applied heat treatment for titanium alloy grade Ti6Al4V enabled control of the alloy's microstructure and its mechanical properties. The first scheme resulted in obtaining the content of phase $\alpha$ in the range from 88% to 96%. The second scheme obtained $\alpha$ phase content from 60% to 76%. In the first scheme, the heating process was carried out to a temperature of 1050 °C, which resulted in the transformation of $\alpha \rightarrow \beta$. The next step cooling to temperatures from 980 to 800 ºC, had an aimed of obtaining a varied content of $\alpha$ phase. It was assumed that as the temperature decreases, the content of the $\alpha$ phase should increase. In scheme 2, the annealing process was carried out below the $\alpha \rightarrow \beta$ transformation temperature. As in the first scheme, it was assumed that lowering the temperature should lead to an increase in the content of the $\alpha$ phase, which is associated with a decrease in the $\beta$ phase stability. The additional heat treatment applied in the form of aging at 500 ºC was aimed at increasing the content of $\alpha$ 'phase precipitates in the $\beta$ phase. Observations of the microstructure revealed a diverse morphology of the alloy structure, Fig. 1.

| 500°C/8 h | 900°C/15 min + 800°C/1 min + 500°C/8 h | $\alpha+\beta$ | ~70,2 | 363 | 1058 | 1123 | 13,7 |
| 500°C/8 h | 800°C/15 min + 500°C/8 h | $\alpha+\beta$ | ~75,7 | 337 | 949 | 1018 | 8,1 |
Figure 1. Microstructure of Ti6Al4V alloy after heat treatment: a) scheme 1s, b) scheme 2s, c) scheme 3s, d) scheme 4s, e) scheme 5s, f) scheme 6s.

| Elt. Line | Intensity (c/s) | Error 2-sig | Atomic % | Conc Wt. % |
|-----------|----------------|-------------|-----------|-------------|
| Al Ka     | 62.10          | 1.582       | 15.454    | 9.331       |
| Ti Ka     | 733.11         | 4.922       | 83.838    | 89.862      |
| V Ka      | 6.04           | 1.735       | 0.708     | 0.808       |

Point 1a

| Elt. Line | Intensity (c/s) | Error 2-sig | Atomic % | Conc Wt. % |
|-----------|----------------|-------------|-----------|-------------|
| Al Ka     | 57.53          | 1.551       | 14.443    | 8.678       |
| Ti Ka     | 736.16         | 4.943       | 84.533    | 90.161      |
| V Ka      | 8.70           | 1.815       | 1.024     | 1.161       |

Point 1b

| Elt. Line | Intensity (c/s) | Error 2-sig | Atomic % | Conc Wt. % |
|-----------|----------------|-------------|-----------|-------------|
| Al Ka     | 66.29          | 1.647       | 15.866    | 9.599       |
| Ti Ka     | 758.46         | 5.019       | 83.585    | 89.774      |
| V Ka      | 4.86           | 1.790       | 0.549     | 0.627       |

Point 2a

| Elt. Line | Intensity (c/s) | Error 2-sig | Atomic % | Conc Wt. % |
|-----------|----------------|-------------|-----------|-------------|
| Al Ka     | 53.80          | 1.511       | 12.976    | 7.727       |
| Ti Ka     | 758.88         | 5.042       | 82.912    | 87.650      |
| V Ka      | 36.70          | 2.053       | 4.112     | 4.623       |

Point 2b
In order to confirm the occurrence of $\alpha$ and $\beta$ phases, a microanalysis of the chemical composition was carried out for selected samples using Energy-dispersive X-ray spectroscopy, Figure 2. The difference in vanadium and aluminum content in the alpha and beta phases was observed. The $\beta$ phase stabilizing element is vanadium in the analyzed alloy. Areas richer in this element were a mixture of the $\beta$ phase with the precipitations of the $\alpha'$ phase which was formed during the aging process. The $\alpha$ phase were areas rich in aluminum, which is the basic element stabilizing this phase. The highest level of hardness and strength combined with high toughness was obtained for the lowest percentage of phase $\alpha$ (samples 1s and 4s), table 2. This is probably related to the higher proportion of phase $\beta$ which is characterized by higher mechanical properties due to the body-centered cubic crystal structure. The increase in the volume fraction of $\alpha$ phase with hexagonal close-packed crystal structure led to a decrease in strength properties as well as to a reduction in alloy plasticity. The highest mechanical properties were obtained for the first heat treatment scheme and the content of $\alpha$ phase at 88%. The hardness obtained was 573 HV and the tensile strength and yield strength 1216 MPa and 1119 MPa, respectively, with a relative elongation A5 of 16.7%. Similarly, for the second scheme, the highest strength parameters were achieved for the lowest volume fraction of phase $\alpha$ of 60.1%. The hardness was 447 HV, the tensile strength and yield strength were 1148 MPa and 1086 MPa at 16.3% elongation. Despite the lower content of $\alpha$ phase in the second scheme, lower mechanical properties were obtained. The reasons should be seen in the method of heat treatment. Probably the use of heat treatment with the $\alpha \rightarrow \beta$ transformation led to better distribution of the $\alpha'$ phase precipitates after the aging process. Confirmation of this, however, requires research using transmission microscopy.

Conclusions

1. The analyzed titanium alloy Ti6Al4V has a two-phase $\alpha + \beta$ microstructure, where the volume fraction of the alpha phase and its morphology depends on the heat treatment applied.
2. After applying additional aging at 500 °C, a fine dispersion of $\alpha'$ is precipitated in the supersaturated $\beta$ phase.
3. The best combination of mechanical properties was obtained after heat treatment according to scheme 1s. The alloy had a yield strength of 1119 MPa, a tensile strength of 1216 MPa, and an elongation of 16.4%.
4. The microstructure of the alloy after heat treatment according to the 1s scheme was composed of grains of the $\alpha$ phase and the $\beta$ phase in which the fine-dispersion $\alpha'$ phase nucleated.

Reference

[1] Terlindale G T, Duering T W and Williams J C 1983 Metall. Mater. Trans. A 14 2101-15
[2] Duering T W, Albrecht J, Richar D and Fischer P 1982 Acta Metall. 30 2161-72
[3] Oczkos K E 2008 Mechanik 8-9
[4] Dudek L, Hryniewicz T and Rokosz K 2016 Autobusy : technika, eksploatacja, systemy transportowe 17 (8) 62-66
[5] Titanium Alloy Guide, RMI Titanium Company, Jan/2000 (2)
[6] Boyer R R and Brigs R D 2005 *J. Mater. Eng. Perform.* **14** (6) 681-685
[7] Bogucki R, Mosór K and Nykiel M 2014 *Arch. Metal. Mater.* **59** 1269-73
[8] Dąbrowski R 2011 *Arch. Metal. Mater.* **56** 217-221
[9] Dąbrowski R 2011 *Arch. Metal. Mater.* **56** 703-707
[10] Zębala W, Gawlik J, Matras A, Struzikiewicz G and Ślusarczyk Ł 2014 *Precision Machining* **581** 409-414
[11] Bojko Ł, Ryniewicz A M, Bogucki R and Pałka P 2015 *Prz. Elektrotech.* **91** (5) 29-32
[12] Żyra A, Bogucki R and Skoczypiec S 2019 *Arch. Metal. Mater.* **64** (3) 1005-10