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BETA-TITANIUM BIOMEDICAL ALLOY: EFFECT OF THERMAL PROCESSING ON MECHANICAL PROPERTIES

STOP BETA-TYTANU DO ZASTOSOWAŃ BIOMEDYCZNYCH: WPŁYW OBRÓBKI CIEPLNEJ NA WŁAŚCIWOŚCI MECHANICZNE

A new β-titanium alloy (Ti-3Al-5V-6Cr-3Mo-3Zr) was investigated as a function of heat treatment to evaluate its mechanical properties. The cold drawn beta-titanium alloy was subjected to β-annealing as well as solution treatment and aging treatments. The mechanical properties were evaluated using MTS Landmark-servo hydraulic Universal Testing Machine. The beta-titanium alloy demonstrated an excellent combination of strength and ductility for both β-annealing and solution treatment and aging conditions. The influence of thermal treatments on microstructure was studied with HiRox digital microscope. The fracture morphology investigated revealed predominantly cup and cone/dimpled fracture surface features demonstrating excellent toughness in addition to high strength and low stiffness that are suitable for biomedical applications.

Keywords: thermal processing, beta-titanium biomedical alloy, mechanical properties, microstructure, fractography

Badano wpływ obróbki cieplnej na właściwości mechaniczne nowego stopu β-tytanu (Ti-3Al5V-6Cr-3Mo-3Zr). Ciągniony na zimno stop β-tytanu poddano wyżarzaniu, a także przesycaniu i starzeniu. Właściwości mechaniczne badano za pomocą uniwersalnej maszyny wytrzymałościowej MTS Landmark-servo. Stop β-tytanu wykazał doskonałe połączenie wytrzymałości i ciągliwości zarówno po wyżarzaniu, przesycaniu i starzeniu. Wpływ obróbki termicznej na mikrostrukturę badano przy użyciu cyfrowego mikroskopu HiRox. Badania morfologii przełomów wykazały głównie wgłębienia w kształcie kielicha i stożka co wskazuje na doskonałą twardość w połączeniu z wysoką wytrzymałością i niską sztywnością, które są odpowiednie do zastosowań biomedycznych.

1. Introduction

Titanium alloys are the most efficient metallic alloys used in aerospace, aircraft, military, chemical and biomedical applications [1-2]. Amongst titanium grades, α and $\alpha + \beta$ titanium alloys have poor workability, low fracture toughness, and large directionality of strength primarily due to their HCP crystal structures [3]. Newly developed β-titanium based biomaterials consist of non-toxic elements such as Zr, Nb, Mo, Ta and others [4-5]. These alloys are particularly useful because they increase strength and have excellent cold deformability. As a result, they are now recognized as advanced materials for orthopedic applications. β-titanium alloys have a good combination of high strength, high fracture toughness, good workability (because of their BCC crystal structure), low elastic modulus, and excellent corrosion resistance as opposed to $\alpha + \beta$ type titanium alloys [6-7]. As a result, β-titanium alloys allow a greater load transfer from the artificial implant to the adjacent remod-

eled bone. The bone resorption is then minimized and a possible loosening of the prosthetic device is avoided [8-19].

Ti-3Al-5V-6Cr-3Mo-3Zr alloy resembles close to a beta-C alloy (a type of metastable beta alloy having a nominal composition: Ti-3Al-8V-6Cr-4Zr-4Mo) has high strength with excellent ductility not available in other beta alloys. This is in addition to their excellent cold-working characteristics and fair weldability [20]. The purpose of this investigation is to evaluate the mechanical properties of Ti-3Al-5V-6Cr-3Mo-3Zr alloy as a function of heat treatment/microstructure.

2. Material and methods

$\beta-$titanium alloy (Ti-3wt%Al-5wt%V-6wt%Cr-3wt%Mo-3wt%Zr) was used in this investigation. This alloy was supplied in the cold drawn condition.

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2.1. Heat treatment

One of the β-titanium alloy samples was solutionized at 850°C (in vacuum) for 1 hour and quenched in water and then aged at 500°C for 10 hours. The other sample was annealed at 850°C (in vacuum) for one hour and then air cooled.

2.2. Tensile test

Tensile tests were carried out at a crosshead speed of 0.1 inch/minute (equivalent to a strain rate of 10^{-2}) on test specimens having 6.25 mm gage diameter and 25 mm gage length according to ASTM standard E8.

2.3. Hardness test

Hardness tests were performed on a standard digital Rockwell hardness tester according to ASTM E18-07.

2.4. Optical microscopy

The optical microscopy was carried out on well-polished and etched samples using Olympus optical microscope and HiRox digital microscope – KH 7700 model. Kroll's reagent (3 ml HF, 6 ml HNO_3 in 100 ml water) was the etchant used for β-titanium alloys.

2.5. Scanning electron microscopy

Tensile tested fracture surface features were investigated using SEM-LEO 1430 VP and PHENOM – a table top SEM. The fractured surfaces were gold coated to obtain enhanced electron emission from the sample surface to obtain quality images.

3. Results and discussion

3.1. Mechanical properties as a function of heat treatment

Figure 1 demonstrates the macroscopic fractures of the tensile tested samples with respect to their heat treatments. Figure 1(a) exhibits cup and cone type of fracture typical of a material showing gross plastic deformation preceding fracture. Figure 1(b) reveals predominantly a ductile macroscopic fracture with shear lips at the edges of the surface. By comparison, it is expected that the heat treatment corresponding to Figure 1(a) has higher ductility but relatively lower strength values. This is confirmed in the tensile test and is presented in Table 1.
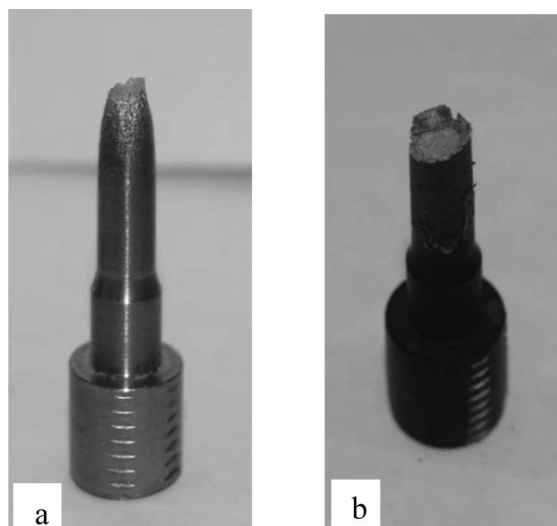

Fig. 1. Tensile fractures of (a) Annealed (b) Solution treated and aged β-titanium alloy

The mechanical properties for the heat treated beta-titanium alloys are shown in Table 1.

TABLE 1

Tensile test data for Ti-3Al-5V-6Cr-3Mo-3Zr alloy

Heat treatment	Yield strength (MPa)	Tensile strength (MPa)	Elong. (%)	Elastic modulus (GPa)	Hardness (HRC)
Solution treatment and aging	965	1031	17	95	34.6
β−Annealed	872	905	24	92	29.3

Cold drawn and annealed samples showed 905 MPa, 872 MPa, 24%, 29.3 HRC as values for tensile strength, 0.2% yield strength, elongation, and hardness, respectively. In comparison, tensile properties of cold drawn, solution treated and aged samples were evaluated as; 1031 MPa (tensile strength), 965 MPa (yield strength), 17% (elongation), and 34.6 HRC (hardness). In addition, β-titanium alloy samples were found to have a relatively lower value of elastic modulus between 92 and 95 GPa that is lower in comparison to $\alpha + \beta$ titanium alloys (for example Ti-6Al-4V alloy). The elastic modulus was experimentally determined based on a standard method using the extensometer as an attachment to the test specimen on a 25T capacity servohydraulic MTS Landmark machine.

3.2. Microstructure as a function of heat treatment

In β-titanium alloys, annealing treatment is specifically referred to as β-annealing. The β-annealing treatment is normally given to the products to improve their fracture toughness. Beta annealing is carried out at temperatures above the β transus of the given alloy. The β

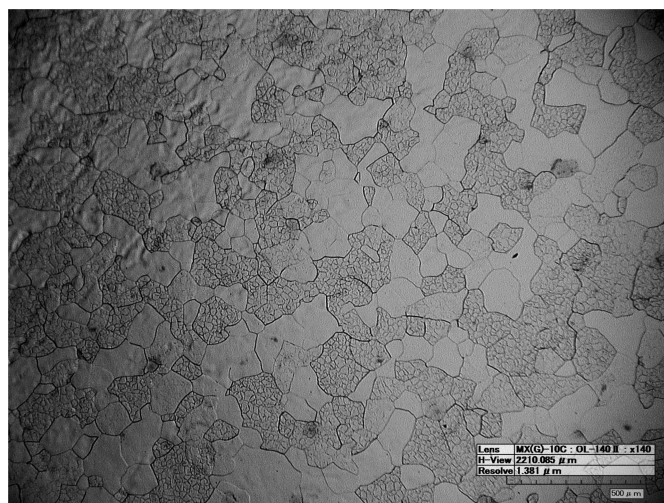transus temperature for the presently investigated alloy is 795°C [15].

polygonized structure, eliminating most of the dislocations.

Fig. 2. Digital microscopic structure of the cold drawn and β annealed β-titanium alloy demonstrating equiaxed grains of metastable β phase
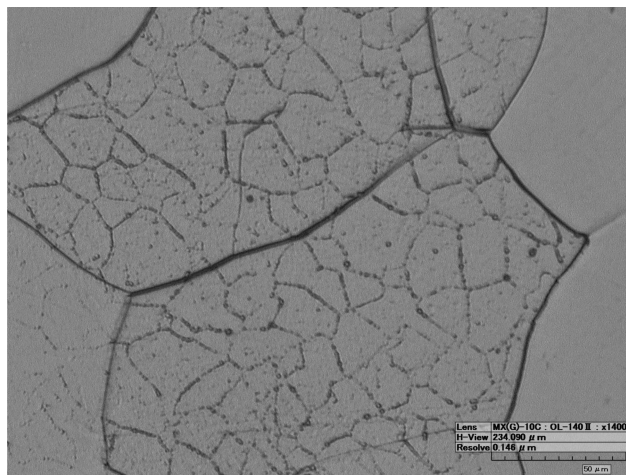

Fig. 3. Digital microscopic structure of cold drawn and β-annealed sample (at a higher magnification) showing β grain/subgrain structure with α precipitates

Alloys that contain sufficient quantities of beta stabilizers to suppress the martensitic transformation thereby retaining the bcc crystal structure on quenching to room temperature are known as metastable β-titanium alloys. Theoretically, excessive stabilizer elements could be added to the alloy so that beta-transus temperature is reduced to below room temperature yielding a stable β-titanium alloy. Metastable β-titanium alloys are typically given a thermomechanical treatment to precipitate additional phases. During solution treatment and aging, precipitation of alpha phase and/or intermetallics improves yield strength and fracture toughness. The morphology, size and distribution of these precipitates determine the mechanical properties of the alloy. Homogeneous precipitation of a fine alpha phase in metastable β-titanium alloys leads to increased yield strength and ductility. These thermomechanical treatments show promise in controlling the microstructure of the final β-titanium alloy with improvement in mechanical properties [21].

Figure 2 shows the presence of equiaxed grains in the β-titanium base metal matrix. The overall microstructure depicts both recovery and recrystallization stages of annealing treatment of a cold worked metal/alloy. The present β-titanium alloy was cold drawn, and had the microstructure composed of deformed grains containing a large number of tangled dislocations. This is a well-established phenomenon for any metal/metal alloy during/after cold working. During annealing, the thermal energy permits the dislocations to move and form the boundaries of a polygonized subgrain structure as can be clearly seen in Figure 3. In the recrystallized grains, new small grains nucleate at the cell boundaries of the
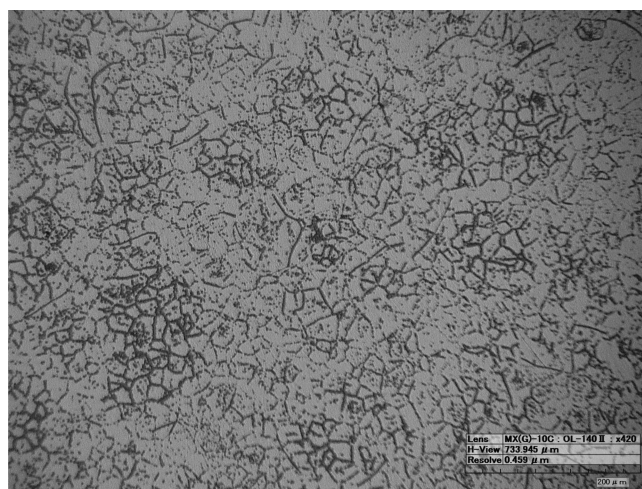

Fig. 4. Digital microscopic structure of the cold drawn and solution treated and aged sample exhibiting α precipitates (dark) in β grains

A wide range of strength levels can be obtained by solution treatment and aging. The origin of heat treating responses of titanium alloys lies in the instability of the high temperature β phase at lower temperatures. Solution treating of β-titanium alloys is normally carried at temperatures slightly above the β transus temperature. Aging causes the decomposition of the supersaturated β phase retained upon quenching. The time/temperature combination depends on required strength.

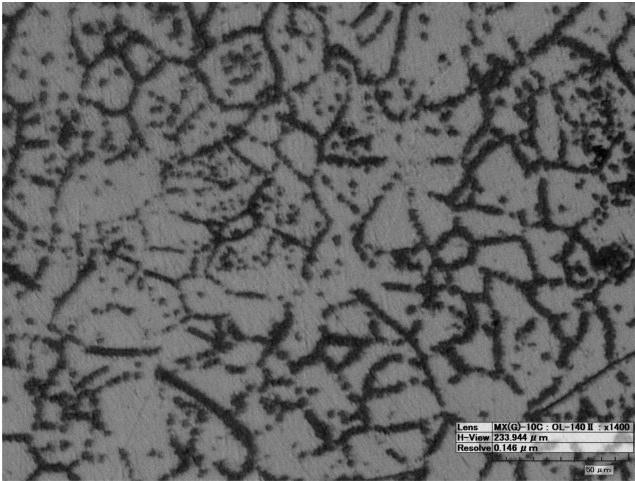Figures 4 and 5 demonstrate the presence of dark precipitated particles of α in β grains.

Fig. 5. Digital microscopic structure of the cold drawn and solution treated and aged sample

3.3. Tensile fracture surface features

Figure 6 clearly demonstrates the formation of voids, void coalescence and growth for the β-annealed sample. These features demonstrate the ductile characteristics of the material. This is supported by the elongation value at 24% from the tensile test. The homogeneous precipitation of a fine alpha phase in metastable β-titanium alloys leads to increased yield strength and ductility [21] that is evident in Figure 6 in the form of dimpled/ductile structure. The typical ductile characteristics along with isolated regions of brittle fracture for the sample that was solution treated and aged are shown in Figure 7.
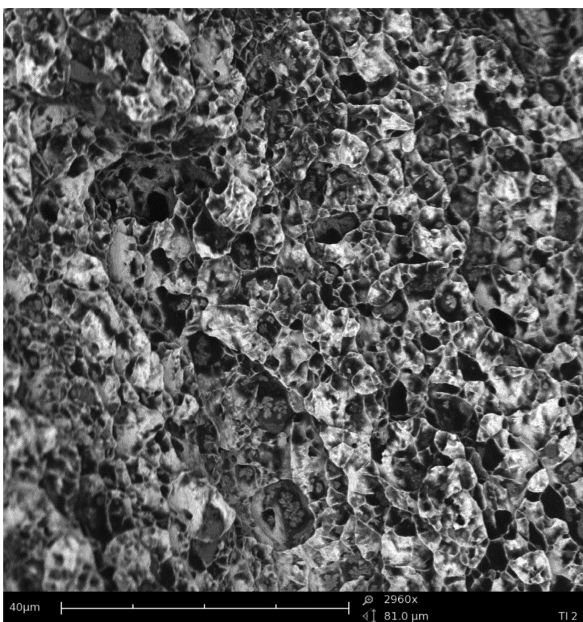

Fig. 6. SEM fracture surface feature of β-annealed sample demonstrating ductile fracture by void coalescence and growth

The isolated region of brittle fracture is the evidence for the relative decrease in ductility (in terms of 17% elongation) as reported in Table 1.
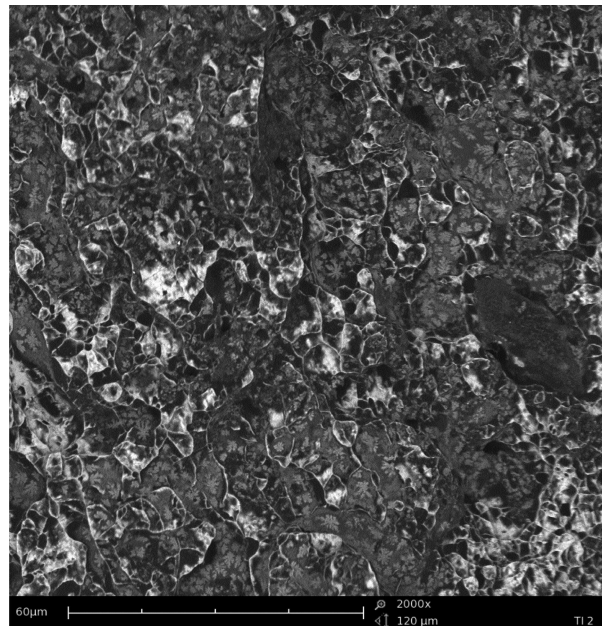

Fig. 7. SEM fracture surface feature of solution treated and aged sample showing ductile fracture features with isolated brittle regions

4. Conclusions

- Ti-3Al-5V-6Cr-3Mo-3Zr alloy exhibited an excellent combination of strength and toughness (ductility) in β-annealed as well as in the solution treated and aged conditions demonstrating its suitability in biomedical applications involving static/dynamic loading conditions.
- The elastic modulus of the new alloy Ti-3Al-5V-6Cr-3Mo-3Zr alloy was experimentally evaluated and found to be relatively low in comparison to other standard titanium alloys used for biomedical applications. This is particularly beneficial in reducing stiffness with synthetic metallic implants.
- The high strength and ductility (toughness) characteristics were demonstrated by dimpled/fibrous fracture surfaces of the alloy.

REFERENCES

[1] P.G. Allen, P.J. Bania, A.J. Hutt, Y. Combress, in, P.A. Blenkinsop, W.J. Evans, H.M. Flower (Eds.), Titanium '95, Vol II, The Institute of Materials, London, 1680-1687 (1996).

[2] R. Boyer et al., Materials Property Handbook: Titanium alloys, ASM International, 1994.

[3] R. Zwicker, K. Buehler, R. Mueller et al., Titanium 1980, Science and Technology, Proc. 4th Int. Conf. on Titanium, Kyoto, Japan, The Met. Soc. AIME, 505-514 (1980).

[4] M. Metikos-Hukovic, A. Kwokal, J. Piljac, Biomaterials **24**, 3765-3775 (2003).

[5] M. Semlitsch, F. Staub, H. Webber, Biomed Techn **30**, 334-339 (1985).

[6] R.R. Boyer, in: D. Eylon, R.R. Boyer, D.A. Koss (Eds.), TMS, Warrandale, PA, 335-346 (1993).

[7] D.M. Brunette, P. Tengvall, M. Textor, P. Thomsen, Berlin, Heidelberg, New York: Springer; 174-76 (2001).

[8] M. Semlitisch, Clinical Mater. **2**, 1-13 (1987).

[9] M. Niinomi, Mater. Sci. Eng. **243**, 231-236 (1998).

[10] H.M. Silva, S.G. Schneider, C.M. Neto, Mater. Sci. Eng. **24**, 679-682 (2004).

[11] D. Kuroda, M. Niinomi, M. Morinaga, Y. Kato, T. Yashiro, Mater. Sci. Eng. **243**, 244-249 (1998).

[12] E.B. Taddei, V.A.R. Henriques, C.R.M. Silva, C.A.A. Cairo, Mater. Sci. Eng. **24**, 683-687 (2004).

[13] R. Banerjee, S. Nag, H.L. Fraser, Mater. Sci. Eng. **25**, 282-289 (2005).

[14] J.I. Qazi, B. Marquardt, H.J. Rack, JOM **56**, 49-51 (2004).

[15] H.J. Rack, J.I. Qazi, Mater. Sci. Eng. **26**, 1269-1277 (2006).

[16] F. Guillemot, F. Prima, R. Bareille, D. Gordin, T. Gloriant, M.C. Porte-Durrieu, Ansel D, Baquey C, Med.& Bio. Eng. & Compu. **42**, 137-141 (2004).

[17] D. Kuroda, M. Niinomi, M. Morinaga, Y. Kato, T. Yashiro, Mater. Sci. Eng. **243**, 244-249 (1998).

[18] J.O. Peters, G. Lutjering, Met. & Mater. Trans. **32**, 2805-2818 (2001).

[19] Y.Q. Zhao, S.W. Xin, W.D. Zeng, J. Alloys Compd. **481**, 190-194 (2009).

[20] ASM Handbook, Heat Treating **4**, 913-923 (1991).

[21] S. Ankem, C.A. Greene, Mater. Sci. Eng. **263**, 127-131 (1999).