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CLINICAL STUDY

Mechanical properties and microstructure of Ti-35.5Nb-5.7Ta beta alloy

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Abstract: *Objective:*Titanium and titanium alloys represent generally accepted metallic biomaterials for clinical dentistry and dental implantology. In this paper, we present a Ti-35.5Nb-5.7Ta alloy with a special respect to its microstructure and mechanical characteristics, such as Young modulus of elasticity.
*Methods:*Three thermal treatments differing in temperature and time of annealing were used during the Ti-35.5Nb-5.7Ta processing in order to evaluate the effects of ageing, melting annealing, and annealing on mechanical characteristics and microstructure.
Results: Using microscopy, the alloy was analyzed and the differences in shares of beta phase grains, alpha particles and precipitates evaluated. The three thermal treatments were evaluated also from technological point of view.
*Conclusion:*The following thermal treatment was found optimal for the Ti-35.5Nb-5.7Ta alloy: melting annealing at 800 °C for 0.5 hour followed by a cold swaging with a 52–79 % deformation, and final hardening at 500 °C for 2 hours in water*(Tab. 2, Fig. 3, Ref. 24)*.Text in PDF *www.elis.sk.*
Keywords: titanium alloys, thermal processing, precipitation grains.

Introduction

Pure titanium as well as titanium alloys are frequently used in dental clinical practice for dentures, dental implants, crowns and constructions of partial removable dentures. A great number of material science institutions and dental clinics are involved into development and testing of newly-designed titanium alloys. To reach desired mechanical material properties, chemical characteristics and biocompatibility, numerous additive elements, their proportion in the alloy, respectively, are tested. Niobium (Nb), tantalum (Ta), and zirconium (Zr) are considered main additive (nobel) elements that can improve physical/chemical properties of resulting titanium alloys (1). In general, titanium alloys may form, according to crystal structure, four basic categories: (a) α alloys, (b) β alloys, (c) intermediate α + β alloys, and (d) intermetalics (e.g. Ni-T). Recently, it is widely accepted that β alloys represent the most prospective material for medical applications from the above-specified alloys (2).

Our earlier studies (see e.g. 3–5) carried within the framework of Stomatological Research Centre (Masaryk University, Brno) focused on tests of mechanical properties and biocompatibility of titanium alloys with addition of Nb, Mo, Ta, Va and Fe. In these studies, it was shown that proportion of additive elements affected microstructure of the alloys, formation of phase of the alloy, in particular.

Recent requirements for titanium β alloys as materials for dental implantology comprise: (a) high biocompatibility including no or minimized toxicity, (b) good osseointegration, and (c) optimal mechanical properties. There are several major mechanical characteristics that titanium alloys must fit before being used in *in-vitro* testing and clinical studies. Among them, low value of Young's Modulus of Elasticity (E) represents one of the most common requirement. In dental titanium alloys, E varies within the range of 63–128 GPa. Generally, E of dental implant should not be too high compared to E of bone tissue (6) so that bone cells damage and resulting osteoporosis and/or poor osteointegration is avoided (7). High resistance to corrosion is another mechanical property that titanium alloys must have. Thanks to TiO_2 layer formed on the upper surface of titanium β alloys, dental implants made of titanium alloys exhibit high resistance (for review see 8). However, due to saliva and galvanic effects corrosion may happen to a certain extent as proved in *in vitro* (e.g. 9) and clinical studies (e.g. 10). Corrosion titanium alloy may be accelerated by inflammatory-induced decrease in pH in a neighbourhood of dental implant that may cause even small particles release from dental implant (11). In such cases, the rate of corrosion increases. The risk of corrosion-induced metal ion release decreases when some additional elements are included into titanium alloy. Some authors (12) report that Ta addition decreases corrosion in titanium alloys

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thanks to formation of Ta_2O_5 on the upper surface that strongly reduces exchange of ions between material and surrounding tissue. There is a significant reduction of the exchange rate when compared to those titanium alloys forming only TiO_2 (13). Therefore, titanium alloys having Ta are considered promising dental materials with minimized corrosion behavior (14). Moreover, recent techniques that exploit coating of ceramics over dental implants (see e.g. 15) allow to suppress metal ion release from the alloy as well as creation of proper micro/nanostructure of dental implant surface for optimal biointegration (16).

Fretting and sliding wear resistance is another feature that dental implant materials based on titanium alloys should exhibit. Stress transmission between hard tissue and dental implant surface which are in contact plays a major role since further bone degradation and bone adsorption should be avoided. Within last decades, several research studies have been devoted to improvement the performance in terms of the wear behaviour of the biomedical titanium alloys (for review see 6). Although the wear resistance of β-Ti alloys has shown some improvement when compared to α + β alloys, the ultimate utility of titanium alloys as wear components will require more complex studies including all wear mechanisms involved.

It is well established that majority of mechanical properties as well as microstructure of titanium alloys may be affected by processing of the material during manufacturing, heat treatment in particular (17). Cooling rate (e.g. 18) as well as ageing (e.g. 19) may play an important role in final microstructure and shape memory properties of titianium alloy (20). In our study, we focused on changes in mechanical properties and microstructure as affected by thermal treatment of titanium β alloy. For our study, the Ti-35.5Nb-5.7Ta β alloy was selected because the material is new and was subjected to a series of tests recently (21) in order to evaluate its applicability in dental implantology.

Material and methods

Alloy manufacturing

Titanium alloy Ti-35.5Nb-5.7Ta used in this study was developed and manufactured in UJP Prague. The alloy was melted in a vacuum arc furnace equipped with water-cooled Cu catalyzer, W electrode at undepressuredHe atmosphere. Annealing was performed at 800 °C for 30 min followed by hardening in water to reach β phase structure of the alloy. Experimental samples were prepared as cylindric elements of a diameter ranging 9–11 mm. Elemental composition was measured on each sample three times by a NORAN Six/300 microanalyzer with nitrogenless detector. Mean values reached 63.5% Ti, 29.7% Nb a 6.85 Ta.

Thermal processing

Three following thermal treatments were used to produce different microstructure of theTi-35.5Nb-5.7Ta alloy: (1) ageing at 500 °C for 2 hours, abbreviated as *A* in the following text, (2) melting annealing at 900 °C followed by ageing at 350 °C for 2 hours, abbreviated as *B*, and (3) melting annealing at 900 °C followed by ageing at 450/500 °Cfor 10 hours, abbreviated as *C*.

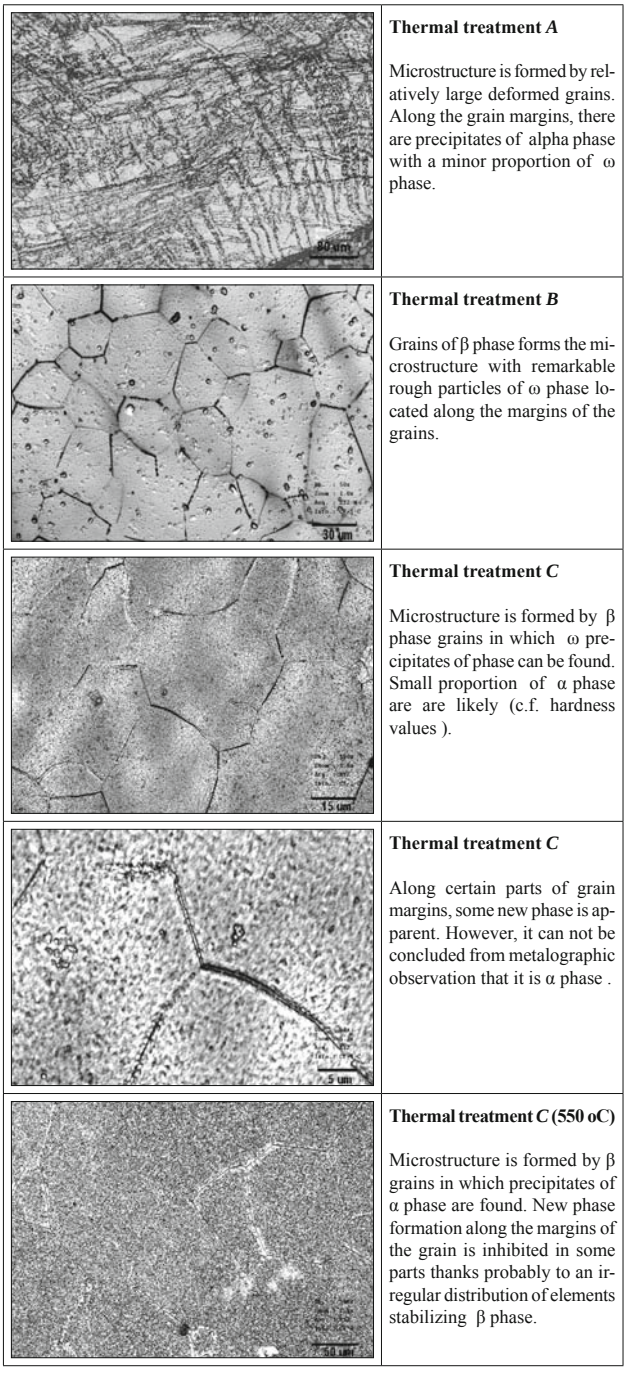

Fig. 1.The effect of ageing temperature on the microstructure of Ti-35.5Nb-5.7Ta alloy.

Mechanical properties

Titanium alloy was tested before (initial state) and after the *A*, *B*, and *C* thermal treatments. Hardness was measured by a micrometer LECO M 400 by Vicker‘ s method using a load of 9,81 N. The measurement was done in a cross line through the alloy samples using a 0.5 mm distance between hits.

After thermal treatments, pressure and tensile tests were done using a ZwickRoel Z 100. Cylindric samples of a height of 5.0 mm

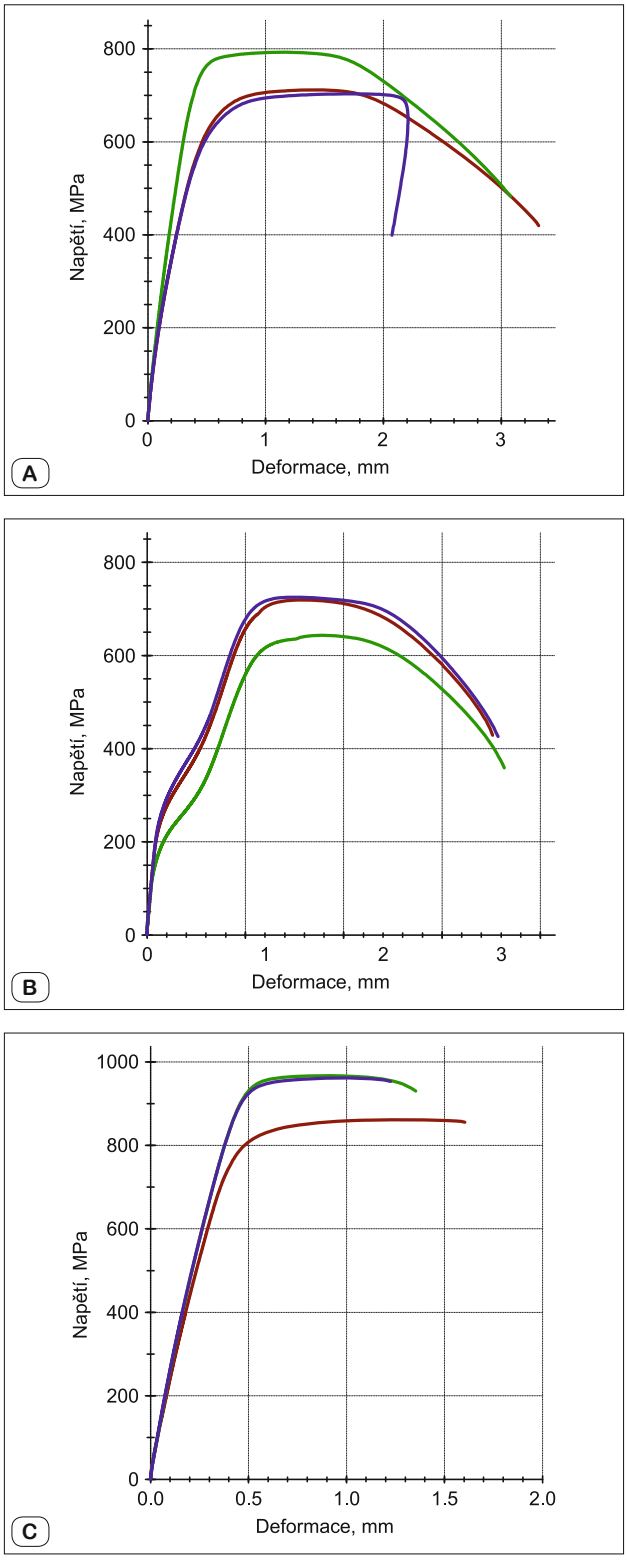

Fig. 2.Dependence of tension on deformation of tested titanium alloy as recorded during tensile tests for individual thermal treatment (ageing): *A, B, C* (for specification see Material and Methods). Three replicates (curves) are shown for an individual treatment.

and a width ranging from 4.0 to 6.8 mm were placed in between two supporting panels and subjected to a load until a plastic deformation was reached. Material response was recorded during a load and the following parameters determined from the curves:E – elasticity modulus in tensile test, $R_{p0,2}$ – sliding limit *(contractual yield),*R_m – breaking point,A_g –elongation (relative deformation) under the influence of the maximum power, A_{30}– Elongation at break rod (maximum relative deformation),Z – contraction (narrowing of the test bar at the quarry).

Microstructure study

Initial material as well as samples after different thermal treatment were subjected to microscopic metallographic study. Samples were etched by Kroll and then their microstructure documented by a confocal laser microscope LEXT OLS (Olympus).

For microstructure study of the samples subjected to the *A*, *B*, and *C* thermal treatments, a REM Tescan VEGA 5135 microscope supplemented by a *Digital Microscopy Imaging* software and X-Ray microanalyserNoran Six/300 (VLSTD detector) were used. For majority of images, surfaces, magnification of 500 was used so that differences in structure could be distinguished.

Results and discussion

Mean values of mechanical characteristics of thealloy treated by three different thermal treatments are shown in Table 1. General trend is that $R_{p0,2D}$increased from *A* to *C* treatments both in pressure and tensile test. In tensile test, E and *Rm* values increased from *A* to *C* treatments showing maximum values of 69.6 GPa and 930 MPa. Contraction of experimental bar at break point (Z) showed decreasing values with *A* to C treatments.

The microstructure of Ti-35.5Nb-5.7Ta alloy treated by the *A* treatment consists of very long grains forming a dentritic structures. The grains of β phase exhibit deformed shape (Fig. 1). Precipitates of β and, to a minor extent, ω phase might be found mainly along the grain margins. Microstructure of the alloy is similar in *B*, and *C* thermal treatment, however, there are some differences due to duration of aging temperature. While *B* treatment led to a formation of β phase grains with rough particles of ω phase and almost

Tab. 1. Comparison of selected mechanical characteristics for between pressure and tensile tests.

	Pressure test		Tensile test				
	$R_{p0,2D}$ [MPa]	E_D [GPa]	E [GPa]	$R_{p0,2}$ [MPa]	R_m [MPa]	A_{30} [%]	Z [%]
Thermal treatment A D79.5% + 500 °C/2h	610	7.2	47.0	626	736	9.4	63.4
Thermal treatment B D79.5% + 900°C/1h + 350 °C/2h	805	5.8	–	–	696	11.9	49.2
Thermal treatment C D79.5% + 900°C/1h + 450 °C/10h	970	4.4	69.6	802	930	4.65	7.03

ED – elasticity modulus in pressure test. E – elasticity modulus in tensile test. $R_{p0,2}$ – sliding limit *(contractual yield).* R_m – breaking point. A_g – elongation (relative deformation) under the influence of the maximum power. A_{30} – Elongation at break rod (maximum relative deformation). *Z* – contraction (narrowing of the test bar at the quarry).

Tab. 2. Tensile test.

Thermal treatment (ageing)	E [GPa]	$R_{p0,2}$ [MPa]	R_m [MPa]	A_g [%]	A_{30} [%]
A					
mean	47.03	626.17	736.03	4.73	9.41
s	13.25	64.03	49.36	1.06	2.20
υ	28.17	10.23	6.71	22.36	23.41
B					
mean	–	–	695.92	5.52	11.91
s	–	–	45.72	0.47	0.20
υ	–	–	6.57	8.55	1.65
C					
mean	69.58	801.63	929.91	3.57	4.65
s	4.48	47.93	59.36	0.61	0.64
υ	6.43	5.98	6.38	17.17	13.82

E – Young modulus of elasticity, – A_g – elongation (relative deformation) under the influence of maximum strength, A_{30} – elongation at break of bar (maximum relative deformation), Z – contraction (narrowing of a bar in breaking point).

no of a phase, C thermal treatment is typical by a proportion some particles probably of α phase. Microstructure of the alloy treated by *B* treatment showed substantial number of ω phase particles that were found mainly along the β phase grains, however, few of them were located inside the grains. If high ageing temperature (500 °C) is used in *C* thermal treatment, α phase is found in high proportion. In this treatment, formation of new phase was found inhibited thanks probably to irregular distribution of the elements that stabilize β phase.

From the analysis of microstructure it is clear that thermal treatment may affect precipitation of ω phase that, in general, decreases elongation of the resulting material. The thermal treatment used in this study, however did not change E too much (Tab. 1). Thus, the alloys, irrespective of thermal treatment, might be considered sufficiently good from clinical point of view.

Structure of fractured material

Tensile test resulted in a fracture of tested material. Dependence of material deformation on tension ranging in the interval of 0–800 MPa is shown in Figure 2. From the data presented for *C* treatment it results that the alloy treated by the *C*treatment exhibited best characteristics in tensile test before final fracture of the material. Images of microstucture of fractured area are shown in Figure 3 for individual thermal treatments. The microstructure was found treatment dependent. In *A* treatment, microholes are less developed and seen less frequently than in the *B* and *C* treatments. This might be attributed to a lower ductility of the material treated by *A*thermal processing. In this treatment, also a preferential fracture along the margins of grains forming the alloy microstructure.*B*treatment let to formation of numerous holes with rough structure on fracture area. In some particular zones, funnel-like structures were formed in between holes. These structures indicated the points at which fracture was initiated due to likely a higher proportion of precipitates. Cavitation started from these funnel-like structures. In spite of the above-described differences in microstructure on fracture area, ductility of the alloy treated by *B* thermal treatmentdid not differed too much from that treated by *A*treatment. In *C*treatment, microstructure resulted in large and rough subsurfaces of fracture area that resulted probably from a high proportion of precipitates.

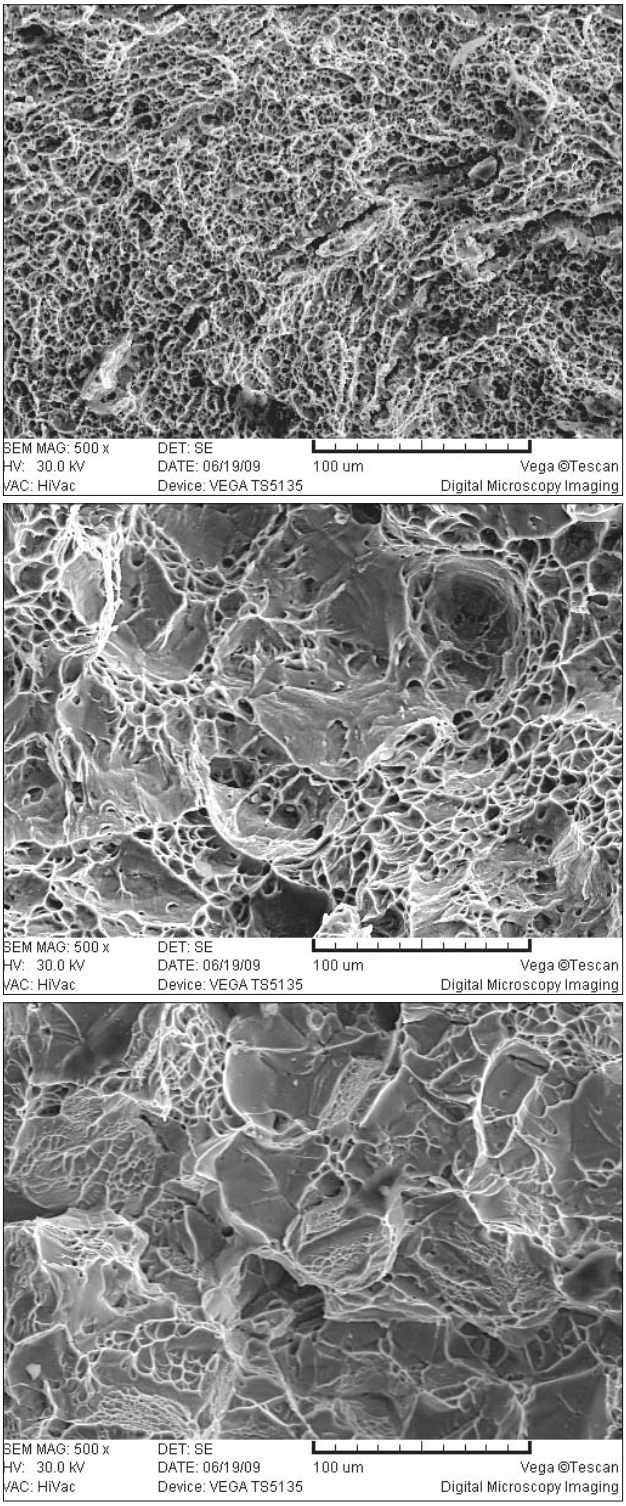

Fig. 3. Microstructure offracture surfaces at breaking tension point for *A*, *B* and *C* thermal treatments (from the top to the bottom). Scale bar represents 100 micrometers.

Holes are seen to only very limited extent, contrastingly to the microstructure of *A* treatment. Reduced number of holes in *C*treated alloy resulted also in a 30 % decrease in ductility (A_{30}) (Tabs 1 and 2).

Conclusions

In our study, microstructure was analyzed in dependence of annealing temperature. It is clear that different proportions between β phase grains and fine α and ω precipitates as well as recrystallization and precipitation processes depended on annealing temperature. Apart from mechanical and microstructure properties, the alloys subjected to three thermal treatment were evaluated also from technological point of view. Machinability and surface roughness were classified (Martikáň, data not shown). It was found that, among the three thermal treatment, only *C*(500 °C) treatmentproduces material with suitable machinability and surface roughness. In conclusion, the following thermal treatment was found optimal for the Ti-35.5Nb-5.7Ta alloy: melting annealing at 800 °C for 0.5 hour followed by a cold swaging with a 52–79 % deformation, and finally, hardening at 500 °C for 2 hours in water. Therefore, similarly to the conclusions of the study (14), the alloy could be recommended for further testing and clinical studies. Future studies will focus further improvement of mechanic properties of the alloy Ti-35.5Nb-5.7Ta that might be reached by addition of some elements. It was reported for Ti35Nb6Ta alloy that boron addition causes grain refinement and influences mechanical properties (22). The final effect on microstructure, however, depended on thermal treatment and boron amount in the alloy (more than 0.05%). It was also demonstrated by the same authors that boron addition influenced recrystallization processes. Moreover, the tensile strength increases with increasing boron addition to alloy. Some authors (e.g. 23), however, report that differences in the amount of added Ta that forms Ti-Nb-Ta alloys may promote mechanic properties and microstructure of the material and thus its applicability in dental implantology. That is the reason why β titanium alloys with tantalum and niobium are further investigated to reach materials with low Young´s modulus of elasticity (24).

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