

# Experimental Substantiation of the Use of Nickel-Free Superelastic Titanium Alloys in Implant Dentistry

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## Abstract

Superelastic titanium alloys (titanium-niobium-zirconium and titanium-niobium-tantalum) are considered to be the more advantageous choice for dental implants' production compared to titanium due to their physicomechanical properties close to those of the bone tissue. Morphological studies on biocompatibility of these alloys in comparison with the titanium had been carried out in animals. Control examination of interaction between titanium alloy samples and bone was performed at the 30th and 90th day after implantation; the methods used for examination were the scanning electron microscopy and microprobe elemental analysis of titanium alloy - bone interface. Tissue along the interface between bone and titanium and superelastic titanium alloys was similar in morphology and elemental composition both at the 30th (at the surface of samples of titanium alloys and in contact with them the poorly mineralized connective tissue was detected) and 90th day (the surface of the titanium alloy samples was covered with mineralized tissue similar in composition to the surrounding bone) after surgery.

Keywords: implant, titanium-niobium, bone tissue, morphology, elemental composition.

# INTRODUCTION

Despite the excellent biocompatibility of titanium alloys and ceramics, the search for new constructional materials for dental implants still continues [1, 2, 3]. This is due to a significant difference in the physicomechanical properties of the implants and the receiving bone tissue, able to lead to bone overload, especially in case when there are too few implants. Currently, comprehensive studies are under way to substantiate the possibility of use in implant dentistry of superelastic titanium alloys: titanium-niobium-tantalum (Ti-22Nb-6Ta) and titaniumniobium-zirconium (Ti-22Nb-6Zr), obtained by casting in the National University of Science and Technology (MISiS) [4, 5, 6, 7, 8]. Along with the properties of superelasticity, generally typical for titanium nickelide (Ti-50Ni), new titanium alloys do not have nickel in their composition.

The purpose of the research was to carry out experimental and morphological study of the osseointegration of nickel-free superelastic titanium alloys Ti-22Nb-6Ta and Ti-22Nb-6Zr.

## MATERIALS AND METHODS

The ability of superelastic titanium-niobium-tantalum and titanium-niobium-zirconium alloys to osseointegration was evaluated in comparison with titanium in experimental animals at the Kazan Federal University (laboratory of laser confocal scanning microscopy of the Interdisciplinary Center for Analytical Microscopy, Interdisciplinary Center of Shared Facilities of Kazan Federal University (KFU ICSF)). 18 rabbits of Gray Giant breed with an average weight of 2,500 g were used in the study. The anesthesia of animals was performed by intramuscular injection of 2% solution of Rometar, a 4 cm incision was made in the submandibular region, the jaw surface was skeletonized and holes of 4 mm in diameter and 2 mm in depth were drilled. Samples of alloys were inserted with the torque into the prepared bone sites, the wound was irrigated with 3% hydrogen peroxide solution and closed in layers. The animals were withdrawn from the experiment at the 30th and 90th day by intramuscular injection of 6 ml Calypsol. The bone blocks were harvested and placed in 10% neutral buffered formalin solution; X-ray examination was performed using Kavo Pan eXam Plus. The bone blocks were previewed under the optical microscope at 50x, 100x and 200x magnification. The interface zone between bone and titanium alloy samples was analyzed using an auto-emission highresolution scanning electron microscope Merlin (Carl Zeiss) after processing the bone blocks in a Q 150T ES vacuum sputter coater (Quorum Technologies). In the vacuum unit the bone blocks were coated with conductive Au/Pd alloy layer at a ratio of 80/20 and thickness of 15 nm by cathode sputtering technique. The microscope is equipped with AZtec X-Max energy dispersion spectrometer (Oxford Instruments) with 127 eV resolution; the measurement accuracy is 0.01-1%. Elemental X-ray microprobe analysis, including spectrography, was carried out using EVO GM (Carl Zeiss) electron microanalyzer at an accelerating voltage of 20 keV and working distance of 10 mm with a set of reference standards for quantitative microanalysis; the probing depth was about 1  $\mu$ m; the detection limit of elements was 1,500-2,000 ppm.

# **RESULTS AND DISCUSSION**

During the experiment the interaction of superelastic titanium-niobium-tantalum and titanium-niobium-zirconium alloys with the bone tissue demonstrated their osseointegration properties. 30 days after surgery at the interface zone between bone and the surface of integrated titanium-niobium-zirconium samples, gaps of up to 20 µm in size were detected in some loci by means of scanning electron microscope at 50x, 100x, 500x magnification. Further along the interface zone extensive areas of tissue covering the edges of titanium-niobium-zirconium samples were identified (Fig 1-2). Due to the higher content of Ca and P among the 8 analyzed elements (44.27 wt% and 12.89 wt%, respectively), the tissue composition beyond the boundaries of the alloy sample was identified as bone as evidenced by microprobe elemental analysis; the content of C and O was 30.33 wt% and 12.22 wt%, respectively. In the areas of tissue growth on the titanium-niobium-zirconium samples' surface, the main elements identified in tissue composition were C (72.45 wt%) and O (13.45 wt%), which was the evidence of the connective tissue (Table 1). 90 days after surgery, the surface of titanium-niobium-zirconium samples was completely covered with mineralized bone tissue. The results of the microelement analysis showed the following tissue composition: Ca - 27.27 wt%, P - 13.77 wt%, C - 29.68 wt%, O - 25.84 wt% (Fig. 3).

The electron microscopic picture and the microprobe elemental analysis of titanium-niobium-tantalum samples showed during the experiment the results similar to those got with the titanium-niobium zirconium samples.

Element	Ti-22Nb-6Zr		Ti Grade 4		Defener es standard
	30 days	90 days	30 days	90 days	Kelerence standard
С	72.45	29.68	61.12	27.83	C Vit
N	9.49	2.56	3.01	0.00	BN
0	13.45	25.84	21.67	29.60	SiO2
Na	0	0.26	0.10	0.34	Albite
Mg	0.11	0.51	0.20	0.69	MgO
Р	0.81	13.77	3.76	13.76	GaP
S	1.66	0	1.69	1.14	FeS2
K	0.10	0.11	0.08	0.15	KBr
Ca	1.94	27.27	8.37	26.49	Wollastonite
Sum:	100.00	100,0	100.00	100.00	

Table 1. Results of microprobe elemental analysis of tissue composition along the interface between the bone and titanium-niobiumzirconium and titanium samples (wt%).

At the 30th day after the insertion of the titanium sample, a 10- $\mu$ m gap was still visible along the interface between bone and sample surface. Under the higher magnification, multiple areas of tissue spreading from the bone site to the implanted sample surface and covering it were revealed. The elemental composition of this tissue was as follows: C - 61.12 wt%, and O- 21.67 wt% (Table 1). However, the tissue located at some distance from the metal sample surface was mainly comprised of Ca, P, C and O (48.21 wt%, 15.01 wt%, 25.48 wt% and 8.87 wt%, respectively). At the 90th day after implantation, titanium samples were completely covered with mineralized bone tissue of the following composition: Ca - 26.49 wt%, P - 13.76 wt%, C - 27.83 wt%, O - 29.60 wt% (Table 1).



Fig. 1. The interface between bone and titanium-niobiumzirconium sample (at the 30th day of experiment, 50x magnification).



Fig. 2. The interface between bone and titanium-niobiumzirconium sample (at the 30 th day of experiment, 100x magnification).



Fig. 3. Titanium-niobium-zirconium sample in contact with bone (at the 90th day of experiment, 5000x magnification).

#### CONCLUSIONS

Thus, in the experiment conducted on animals, samples of dental implants from titanium-niobium-zirconium and titaniumniobium-tantalum alloys were osseointegrated similar to titanium; osseointegration of samples of dental implants from superelastic alloys was completed in 90 days after their implantation in the bone tissue; osseointegration was accompanied by mineralization of the connective tissue along the interface between the bone and the alloy surface formed in the early postimplantation period; electron microscope analysis and microprobe elemental analysis allowed ranking alloys according to the degree of osseointegration in the following way: titanium, titanium-niobium-zirconium, titanium-niobium-tantalum.

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