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Micro Structural Tests of Titanium and its Alloy Used in the Making of Prosthetic Reconstructions

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Abstract— direct metal laser sintering (DMLS) is a new technology for making constructions based on CAD/CAM techniques. The objective of the present paper is to provide a structural identification of titanium and titanium alloy intended as substructures for permanent prosthetic restorations obtained with computer assisted design and manufacturing techniques. The study material comprised samples of commercially pure titanium Ticp used to make prosthetic constructions and produced with milling technology as well as Ti6Al4V samples manufactured with the additive technology of selective direct metal laser sintering. Micro structural tests were performed with optical and scanning microscopes. These tests confirmed that among the structures produced, sintering of titanium-aluminium-vanadium powders is the preferred technology for making permanent support structures for dental prosthetics and may serve as an alternative to CAD/CAM techniques based on subtractive manufacturing.

***Index Terms*— Microstructure, Titanium and its alloys, Dental prosthetics, CAD/CAM system, Direct Metal Laser Sintering (DMLS).**

I. INTRODUCTION

In the last few years technology based on the Computer Aided Design/ Computer Aided Manufacturing (CAD/CAM) system is becoming increasingly popular as a method for constructing prosthetic restorations [1] - [4]. Substructures for crowns and bridges can be made with this system based on two technologies: milling and laser sintering. The design and construction of a restoration using the CAD/CAM system is preceded by diagnosis and treatment, based on which the physician prepares the conditions for placing a permanent prosthetic reconstruction in the stomatognathic system (SS). When preparing the prosthetic abutments the following rules are observed: secure the path of insertion for the restoration, minimise the amount of tissue removed, eliminate areas of stress concentration conditioned by the shape of the step. The type and structure of the biomaterial must also be taken into account. In the CAD procedure both technologies are divided into similar stages. After the abutments are prepared in the patient's oral cavity, the prosthetic base is reproduced. A conventional impression is taken or highly advanced digital techniques are used with an intraoral scanner that directly scans the abutments in the patient's oral cavity and generates 3D digital models. A plaster model is scanned using light and a mobile base. In both approaches, while scanning and copying the shape of the abutments attention is simultaneously paid to the gingival zone. In addition, it is important to ensure an accurate reproduction of the step and good marginal seal of the planned reconstruction. In the next stage of the procedure a virtual design is made of the external shape of the crown or bridge, taking into accounts its individual anatomic structure and occlusal conditions and making sure to provide optimal support while the soft tissue adapts to the prosthetic reconstruction. Once the material is chosen and the shape of the restoration designed, the CAM procedure begins, during which the substructure of the crown or prosthetic bridge is fabricated. Using technology developed by KaVo Dental GmbH the construction of the restoration is cut out using a NC machine, which can perform milling tasks in five planes. The entire process takes place with cooling. An alternative to milling-based methods is the additive technology - Selective Laser Sintering (SLS), which includes Direct Metal Laser Sintering (DMLS) [4] - [10]. Using a device designed for dental work at the CAM stage the metal substructure is formed via laser beam sintering of titanium-aluminium-vanadium powder. A layer of 0.02 mm thick powder is deposited on the working platform, and then an infrared laser beam is guided over the surface of the powder in accordance with a bitmap, which is a virtual record of the structure to be formed. The working platform is then lowered and another layer of powder is deposited. The cycle is repeated until the entire structure is formed. A constant flow of argon takes place in the chamber. Rapid hardening after melting leads to the formation of an homogenous structure of the material. The objective of the present study was to provide a structural identification of commercially pure titanium – grade 2 – and its alloy Ti6Al4V, intended as substructures for permanent prosthetic reconstructions achieved with CAD/CAM procedures and produced on the basis of two different technologies:

– milling technology preceded by thermal treatment and plastic forming,

– The additive technology of direct metal laser sintering.

II. MATERIAL AND METHODS

The study material comprised samples made with commercially pure titanium grade 2 (Ticp) labelled Everest T Blank (KaVo Dental GmbH, Biberach/ Riß, Germany), intended for the CAD/CAM procedure in the KaVo-Everest system, and samples made with a Ti6Al4V alloy labelled EOS Titanium 64 (Electro Optical System GmbH, München, Germany) - Ti64 made with DMLS technology (Table I). Cuboids measuring 15mm x 5mm x 2mm were made from both materials. The Ticp samples were taken while cutting factory blanks produced in the form of cylinders and small blocks, while Ti64 samples were taken from blocks cut following the additive process of selective metal sintering using an EOSINT M 270 device.

Table 1. Material composition

Elt.	Conc
Titanium	balance
Aluminum	5.5 – 6.75 wt.-%
Vanadium	3.5 – 4.5 wt.-%
Oxygen	< 2000 ppm
Nitrogen	< 500 ppm
Coal	< 800 ppm
Hydrogen	< 150 ppm
Iron	< 3000 ppm

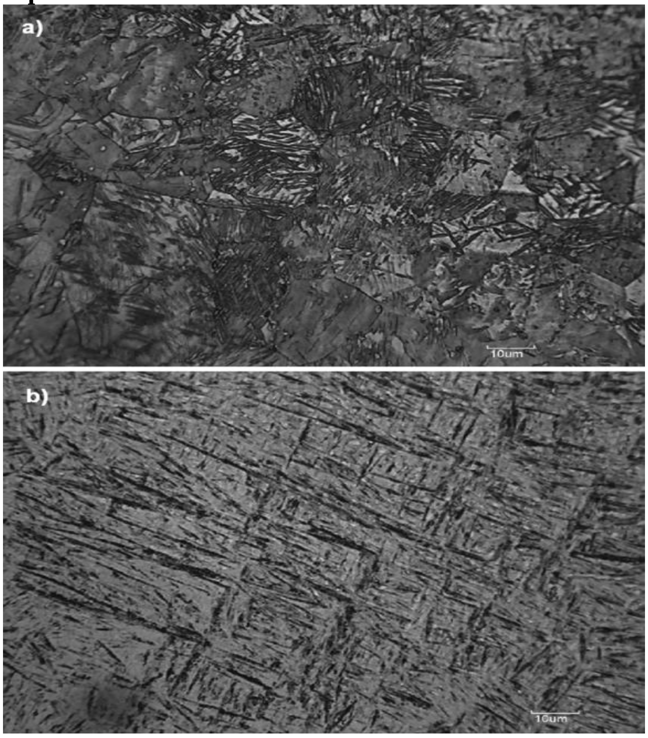

Fig. 1 Microstructure images from an optical microscope: (a) commercially pure titanium – grade 2, (b) Ti64 alloy

Structural analyses were carried out on cut, polished and etched samples. With this aim in mind, the samples were imbedded in resin and polished using a Struers TegraForce-5 device, on which, with the aid of pre-programmed operations, the surface layers required for micro structural tests were obtained. In the next stage the samples were etched in a 10% HF solution with the following composition: 10ml HNO_3 +20ml HF + 20ml glycerine. Micro structural tests of Ticp and Ti64 were conducted using an optical microscope from the company Nicon Eclipse ME 600 with digital image recording as well as a scanning microscope from the company JOEL JSM 5510LV coupled to a digital processing accessory (IXRF System 500 Digital Processing) for X-ray analysis. The accessory made it possible to determine the qualitative and quantitative chemical composition in micro-area at the marked points.

III. RESULTS

The results of the titanium Ticp and Ti64 alloy tests conducted with a Nicon Eclipse ME 600 optical microscope took the form of microstructure images in different magnifications. The present study presents representative images of Ticp and Ti64 alloys in the same magnification (Fig. 1). Ticp made with milling technology was shown to possess a single-phase granular microstructure. The average sizes of grains in cross-sectional and longitudinal profiles were similar, i.e. 20 μm. The Ti64 alloy from laser sintering had a two-phase, fine-grained microstructure with an acicular-lamellar character. Tests carried out on both materials with an optical microscope revealed no discontinuities.

The results of scanning tests on Ticp and Ti64 alloys included SEM images taken with a JOEL JSM 5510LV microscope as well as analyses of their chemical composition in micro areas and at randomly selected points of these micro areas using an EDS accessory. The sequence of Fig. 2 and 3 show selected representative SEM images of the microstructure of Ticp and Ti64 samples. Titanium Ticp possesses a single-phase structure. The grains have similar sizes.

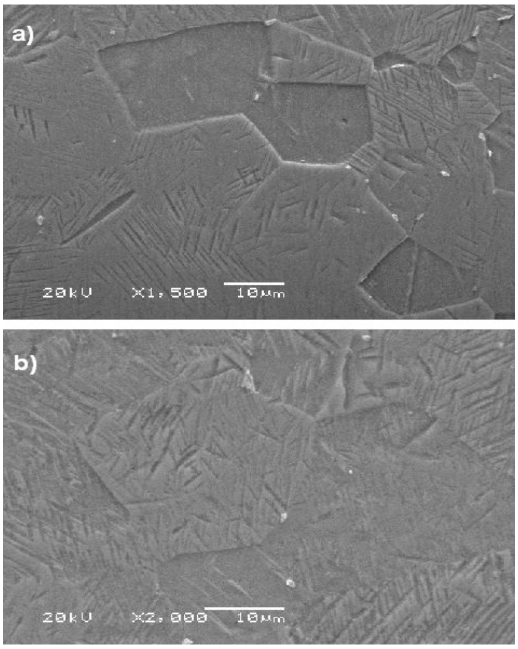

Fig. 2 SEM images of the microstructure of the Ticp sample – grade 2

Visible in the titanium grains is acicular marten site (Fig. 2). The structure of the two phase Ti64 alloy consists of a β matrix as well as released α phase deposits in the shape of extended needles (Fig. 3). The chemical composition of both biomaterials was analysed using an EDS accessory. The presence of titanium was identified in the micro-area of the Ticp surface. Three selected points were tested (Fig. 4).

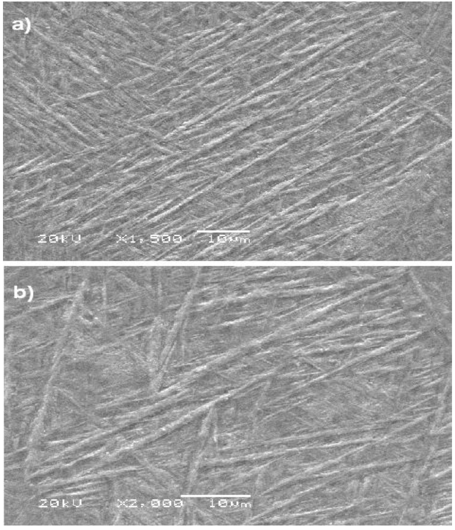

Fig. 3 SEM images of the microstructure of the Ti64 sample

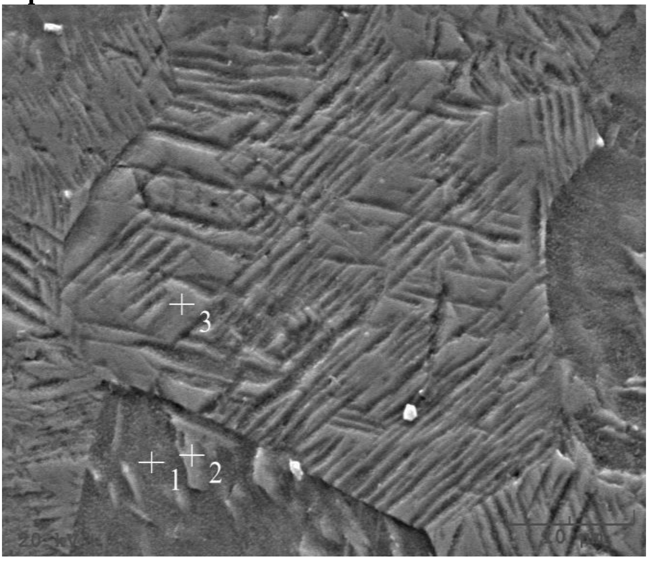

Fig. 4 SEM images of Ticp – grade 2 marked with points for analyzing the chemical composition

A similar analysis was carried out for the Ti64 alloy. In this case, titanium, aluminum and vanadium were all identified on the tested surface. The composition of the elements in the selected micro-area is presented in Table II. Points on the studied surface were also analyzed. Point 1 constitutes phase α, while points 2 and 3 constitute phase β (Fig. 5). The chemical composition of particular points is presented in Table III.

Table 2. Chemical composition of a selected area on the surface of Ti64 sample

Elt.	Intensity (c/s)	Error 2-sig	Atomic %	Conc (wt.%)
Aluminum	36,96	2,220	12,322	7,329
Titanium	494,31	8,118	85,323	90,027
Vanadium	13,26	1,330	2,355	2,644
			100,00	100,00

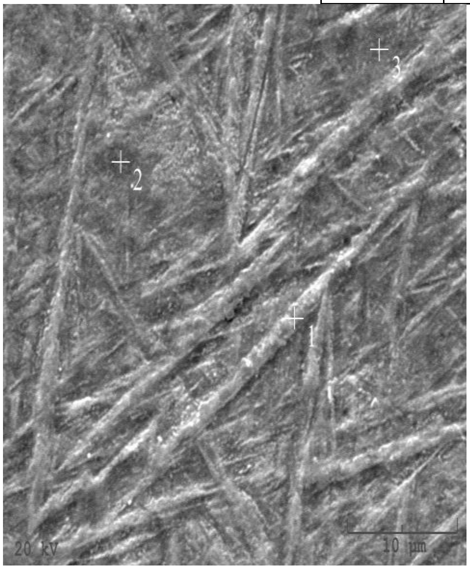

Fig. 5 SEM images of Ti64 alloy marked with points for analyzing the chemical composition

Table 3. Chemical composition of selected points on the surface of the Ti64 sample

Point	Elt.	Intensity (c/s)	Error 2-sig	Atomic %	Conc (wt.%)
1	Aluminum	38,81	2,275	13,658	8,175
	Titanium	459,58	7,827	84,198	89,402
	Vanadium	11,38	1,232	2,144	2,423
				100,00	100,00
2	Aluminum	34,64	2,149	12,002	7,127
	Titanium	477,69	7,980	85,569	90,150
	Vanadium	13,18	1,326	2,429	2,723
				100,00	100,00
3	Aluminum	38,51	2,266	12,942	7,718
	Titanium	483,93	8,032	84,365	89,251
	Vanadium	15,02	1,415	2,693	3,032
				100,00	100,00

IV. DISCUSSION

Micro structural tests of commercially pure titanium Ticp grade-2 showed that the material possesses a uniform, grainy, single-phase structure, which is confirmed by the results of other authors [11], [12]. The sizes of the grains were similar and amounted to 20 µm. The grains exhibited good mutual adhesion, growth and regularity of shape (Fig. 2). The observation revealed the presence of acicular marten site in the area of the grains, which probably developed during the heat treatment. The heat treatment process for the Ticp blanks is not known, since it is a trade secret of the producer. The chemical composition analysed in a selected micro-area and points confirmed that the material was commercially pure titanium (Fig. 4). Micro structural tests carried out on the Ti64 alloy made with DMLS technology led to the conclusion that the material, composed of titanium, aluminium and vanadium, possesses a uniform two-phase construction acicular-lamellar in form and is composed of a β phase matrix and released α phase deposits that take the shape of needles oriented in different directions (Fig. 3). The two-phase Ti64 alloy includes the two most important elements stabilising phase α and phase β. Aluminium dissolves well in a permanent solution α, stabilises and strengthens phase α in a solution and also increases its strength. Simultaneously, it reduces the density of the alloy and increases the thermal stability of phase β. Vanadium, which is an isomorphic element, stabilises phase β and also helps reduce the allotropic transformation $Ti_{\alpha}\leftrightarrow Ti_{\beta}$. The microstructure of the Ti64 alloy is composed of a mixture of phases α and β. [13]. Elemental analysis of the Ti64 alloy confirmed the presence of the following elements: titanium, aluminium and vanadium. An analysis at randomly selected points revealed no uneven percentage distribution of the alloy's metal constituents (Fig. 5, Table III). The microstructure of the material following sintering shows a uniform chemical composition and a lack of porosity. The microstructure formed as a result of the sintering process is a problem that has occupied several authors [10], [14] – [23]. The lamellar microstructure of Ti64 alloy has been confirmed by Ramosoeu et al. [9] and Yadroitsev et al. [15]. The two-phase microstructure of Ti64 produced by laser sintering has also been identified by Rafi et al. [20] as well as Vranken et al. [16]. Using optical micrographs Rafi described the microstructure of Ti64 alloy, which is composed primarily of an α phase and a small amount of β within the prior β columnar grains oriented along the build direction. The α phase posses a lamellar morphology with β surrounding the α lamellae boundary. The substructures of crown- or bridge-type prosthetic reconstructions must have strength properties, which ensure the transfer of occlusal loads, meet high fatigue limit conditions, be resistant to corrosion, have high density and a low Young module, which guarantees optimal contact between the restoration and hard tissue of the SS. These characteristics are determined by the microstructure of the biomaterials, from which fixed prosthetic reconstructions are made. Tests on Ticp and Ti64 microstructures confirmed that the selectively sintered alloy would be the best of these biomaterials to use as a prosthetic substructure. It possesses a two-phase structure with a varying fine-grained orientation. Compared to the single phase granular structure of Ticp such a structure would provide greater strength and durability in prosthetic reconstructions, which must possess a thin wall and a step in the marginal gingiva (Fig. 6). Once the sintering process is complete, the unconsumed powder can be used in the next production cycle. This reduces the production costs and minimises the waste recycling process compared with milling technology.

Fig. 6 Substructure of titanium crown: (a) using subtractive manufacturing technology with milling, (b) following additive laser sintering

V. CONCLUSION

Biomaterial tests on the Ticp and Ti64 alloy using optical and scanning microscopy confirmed a uniform, single-phase granular microstructure of titanium intended for milling in the KaVo Everest system and a uniform two-phase fine-grained structure of the Ti64 alloy produced through the additive technique of selective direct metal laser sintering. The tests showed that the additive technique of selective direct metal laser sintering, which produces a substructure made from Ti64 alloy, is the new preferred technology (in terms of biomaterial structure) for making load-bearing structures for dentistry. It can serve as an alternative to traditional methods based on traditional casting as well as milling-based CAD/CAM methods. This technology does not lead to a loss of material typical of milling procedures and it is pro-ecological.

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I work at Cracow University of Technology in the Faculty of Mechanical Engineering in the Institute of Materials Science. In my research work I am interested in constructional steels especially in plate steels such as HSLA (High Strength Low Alloy) + Cu steels. Moreover I am interested in mechanical fracture. I research the influence of heat treatment on mechanical properties and microstructure.

At present I am interested in the problem of the influence of microstructure on the crack resistance of titanium alloys on the structure of $\alpha + \beta$ and pseudo-β. Moreover I am interested severe plastic deformation techniques which leads to a strong refinement of the microstructure in solid material.

I'm the author and co-author of over 20 articles published in national and international journals (including cited by the Institute for Scientific Information in Philadephia) and conference materials.

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