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To cite this article: D Košťáliková *et al* 2020 *IOP Conf. Ser.: Mater. Sci. Eng.* **776** 012093

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Study of microstructure of selected dental restoration parts

D Košťaliková, M Janeková, A Dubec, P Kováčiková and F Pešlová

Faculty of Industrial Technology in Púchov, Alexander Dubček University of Trenčín,
I. Krasku 491/30, 020 01 Púchov, Slovakia

E-mail: daniela.kostialikova@fpt.tnuni.sk

Abstract. The given paper as well as the whole complex of problems is closely connected with the investigation of the selected materials which are used for dental implants manufacture. The investigated implants had to be removed prematurely from the patient's mouth cavity due to the occurrence of critical states (CS) in relation to the manufacturing material. These critical states of the material led to the malfunction and unacceptability of the dental implants from the aspect of the health condition and the hypersensitivity of the patient to degraded implant material. This work is mainly based on the combination of the theoretical as well as practical knowledge along with predetermination and specification of the needed testing procedures for materials which are commonly used for dental implants. Samples were prepared from the cut sections of the implants in a metallographic (ceramographic) way and subsequently, they were investigated in the terms of their structural properties. The main aim of this paper was to present the microscopic evaluation of the materials for implants and to analyse the undesirable defects that led to their destruction.

1. Introduction

The aim of this work is a detailed study of the concerned parts of dental replacement in which a common problem occurred - the formation of critical states that caused the dysfunction of the replacement to be used by patient. The investigated implants had to be removed prematurely from the patient's mouth cavity due to the occurrence of critical states (CS) in relation to the manufacturing material. These critical states of the material led to the malfunction and unacceptability of the dental implants from the aspect of the health condition and the hypersensitivity of the patient to degraded implant material. Initiation of degradation was investigated for the individual structural elements from a microscopic point of view to determine the critical states which are essential phenomena for subsequent degradation. The oral cavity of the patient is an environment that superposes the wear process and causes the accumulation of critical states that can be exhibited by sensitivity to dental replacement or unacceptable patient's pain. In the event of an undesirable exposure of the used dental replacement, it is necessary to remove the element from the oral cavity immediately. From a medical point of view, it is a complicated procedure for the patient because it may cause undesirable bone damage during removal of the dental replacement. The constant contact of their restorations with mucosa, saliva, periodontal tissue and bone highlights the importance of an in-depth analysis of their chemical and physical characteristics and biocompatibility assays for ensuring patient safety [1].

The production for metallic restorations in the dental laboratory has conventionally been carried out using the traditional cast method based on the lost wax process [2]. Although the alloys of choice were gold-based when the casting technique was first developed, they were gradually replaced by base metal alloys such as nickel-chromium (Ni-Cr) and cobalt-chromium (Co-Cr) alloys [3]. Ni-Cr alloys



containing beryllium (Be), are no longer recommended because of allergic reactions and potential carcinogenic effects of Ni and Be. Most Ni-Cr alloys are formed by Ni (68% to 89%) and smaller percentages of other elements such as Mo, Be, Si, although it has an important role in passive capacity of alloy tends to increase hardness and melting temperature, the latter being important feature to increase the safety margin during ceramics firing [4, 5].

Commercially pure titanium (cp-Ti) and its alloys are widely used for manufacturing dental implants due to their superior mechanical and physical properties, such as corrosion resistance and high modulus of elasticity in tension, and their excellent biocompatibility [3]. There are four grades of cp-Ti depending on their content to oxygen and iron [3]. Following cp-Ti, Ti-6Al-4V, which is also known as Ti6-4 and Ti-grade 5, became commonly used for biomedical applications, because of its enhanced mechanical strength [4]. It is well known that one main reason for the excellent physical and biological properties of titanium and its alloys is the native oxide film (TiO_2) that is created spontaneously on its surface upon air exposure. This film, having only a few nanometers thickness (4.3 ± 0.2 nm for the mechanically polished cp-Ti surface), appears to be responsible for the chemical stability, chemical inertness, corrosion resistance, and even biocompatibility of titanium [6].

In cases where there is excessive cyclical load and micromotion of the implant in the bone, it come up to the effect of an acidic environment (or various pH), which promotes the formation and growth of corrosive layers on it, that can break off. Loosen sharp articles then violate the tissue and, when the metal part is exposed, it leads to inflammatory processes because the metal ions are actively decomposed. It is known from expert studies that titanium corrosion leads to implant failure or initiation of periimplantitis (a serious complication during implant treatment, inflammatory changes in the area of the implant of the plantar neck) and thus bone loss [7–9].

2. Experiment

In the work, the attention is paid to the wear of materials of selected dental replacement that was prematurely removed from the oral cavity (figure 1). The investigated parts of the replacement were provided by the dental clinic, which did not have any knowledge on the composition of the materials and production technology. From an ethical point of view, there was not specific information about a particular patient (age, disease, or duration of use) given. This meant that the solution of the problem was based on a reciprocal approach, where it was necessary to determine the results on the basis of degradation. Threaded parts from implants that interacted with the patient's bone required further investigation. After extraction from the oral cavity and purification of these segments (tested samples), the degradation of the material has been already visually observed on the threads.



Figure 1. Part of dental replacement.

To obtain information on the used materials, prior to the microscopic examination of each structure, the chemical analysis by the EDX detector was performed: x-act, Oxford Instruments. The obtained mass percentages of the individual elements served to incorporate them into the specific equilibrium binary

diagrams. On the basis of the predominant elements in the individual parts of the replacement, it was possible to determine the mutual solubility or insolubility of the basic elements forming the particular alloy. According to areas where the analyzed elements occurred and the comparisons of concentration with the basic binary diagrams, particular phases at a given temperature and concentration could be identified.

3. Chemical analysis of individual parts of dental replacements

Dental replacement is a multi-component composite consisting of a metal and ceramic part, joined with a special bond. In order to evaluate the quality of the materials used, microscopic evaluation has always to be made separately for the individual micro-structures. Chemical analysis was performed from randomly selected sites – figures 2 and 3 (in the form of spectra in figures 4 and 5) and it can be seen in tables 1–6.

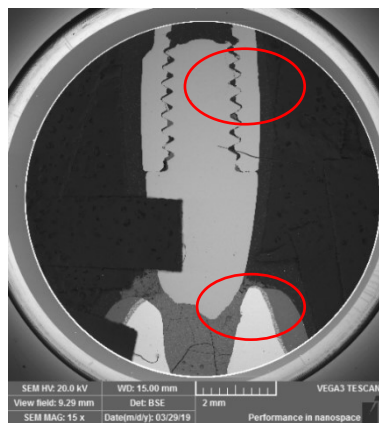


Figure 2. Selecten sites of replacement for chemical analyses.

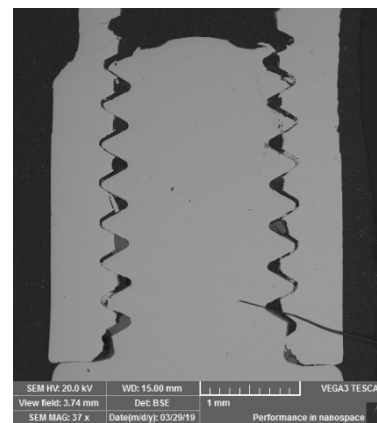


Figure 3. Metal screw joint of dental replacement.

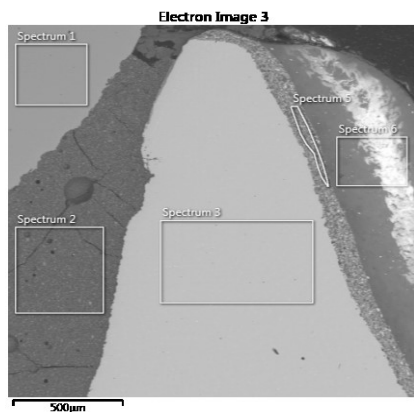


Figure 4. Spectra for chemical analyses.

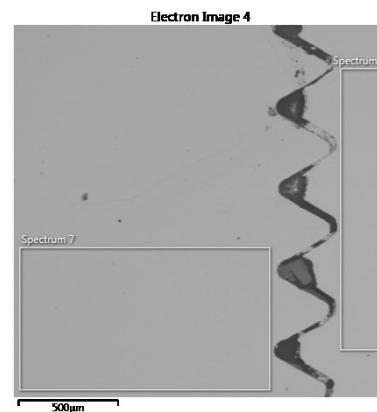


Figure 5. Spectra for chemical analyses.

The metal parts form a screw connection (includig threads) that connects the ceramic part with the bone. Components analysis was done from min. seven randomly selected sites and the average values can be seen in the tables.

Evaluation of ceramic material has shown that it is a silicon-based oxide ceramic (table 1) containing aluminum and other elements.

Metal part in ceramics consists of nickel chromium molybdenum (Ni-Cr-Mo) alloy (table 2). Undesirable elements such as aluminum and manganese were also present in the analysed alloy. The nickel content, which represents that the human organism may be allergic, exceeds 60 wt. %.

The chemical composition of the abutment of the metal part of the implant corresponds to the composition of the Ti-6Al-4V titanium alloy with the designation VT6 (table 3). This part is screw-connected to another part which is implanted into the bone (figure 5).

The inner screw part of the implant (figure 3) is made of pure titanium (table 4). On the basis of measurement, it can be assumed that the complementary elements (up to 100 %) may also be hydrogen, oxygen, or other undesirable non-metallic elements (elemental analysis did not show these elements).

The interlayer that connects the ceramic with the crown part of the implant has the chemical composition shown in table 5. It can be seen that this joint consists of a large number of different elements that may not have positive effect on all living cells.

The next one joint (interlayer) represents the connection between the crown and the metal abutment and it consists of a set of elements listed in table 6. The chemical composition indicates that it is a chemical compound with the function of good adhesion between two different materials, regardless of the living organism.

There is a danger that in the case of a loosening implant in the gums, food or toothpaste elements may precipitate on the surface in the form of so-called plaques. This phenomenon can lead to undesirable chemical reactions in the oral cavity, leading to subsequent dental replacement degradation.

Table 1. Chemical composition of ceramics (wt. %).

Element	O	Si	Al	Na	K	Ba	Ca	In
Spectrum 6	60.4	21.2	6.5	5.9	3.9	0.8	0.6	0.6

Table 2. Chemical composition of metal part (wt. %).

Element	Ni	Cr	Mo	C	F	Si	Nb	Mn
Spectrum 4	61.3	21.7	9.4	2.7	2.7	1.0	0.7	0.5

Table 3. Chemical composition of abutment screw area (wt. %).

Element	Ti	Al	V	C
Spectrum 1	88.6	5.7	3.6	2.1

Table 4. Chemical composition of the Ti part of implant (wt. %).

Element	Ti	C
Spectrum 8	97.9	2.1

Table 5. Chemical composition of intelayer for crown – ceramics system (wt. %).

Element	O	Pt	Si	Al	Na	K	Ca	Ba
Spectrum 5	52.4	17.0	15.4	5.5	4.6	3.9	0.6	0.6

Table 6. Chemical composition of intelayer for crown – abutment system of implant (wt. %).

Element	O	C	La	F	Al	Si	Ca	K	P	Na	Ni
Spectrum 4	36.5	18.4	11.4	9.9	7.1	6.9	6.1	1.6	1.2	0.6	0.2

4. Microscopic evaluation of materials

The metallographically prepared samples of individual materials were used for investigation of the purity and morphology of microstructures because it is reflected in the quality of dental replacements. If the required parameters of the microstructure are not satisfactory, critical states occur, leading to destruction of the dental replacement functionality.

The ceramic material was characteristic by fine pores that were unevenly distributed throughout the whole volume of the material. The thickness of the ceramics and interlayer was not constant in the crown. The ceramics had a thickness ranging from 1387.8 μm to 1652.9 μm , the interlayer was from 74.7 μm to 87.1 μm in thickness (figure 6).

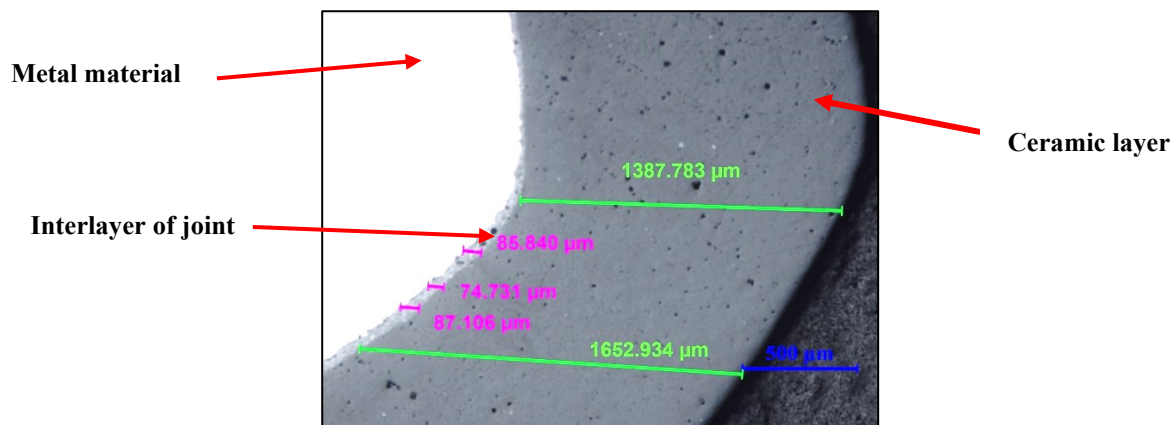


Figure 6. Individual layers of dental replacement.

The interlayer had good adhesion to the ceramics. It can be assumed that the interlayer penetrated also into the pores of the ceramics, thereby stabilizing its attachment to the surface. From the above mentioned fact, it can be seen that it was a piece of hand-made manufacture. These components are made directly to fit the patient's required conditions resulting in patient's oral cavity.

The NiCrMo alloy (figure 7) was evaluated from the point of view of micro-purity and surface layer character. The occurrence of spot oxides and local agglomerates of complexes of various elements was reflected in the microstructure (figure 7a). Detailed examination of the area around the surfaces (at closer magnification) shows that the surfaces have been damaged by either corrosion or diffusion reaction of chemical elements from the surroundings [10–12].

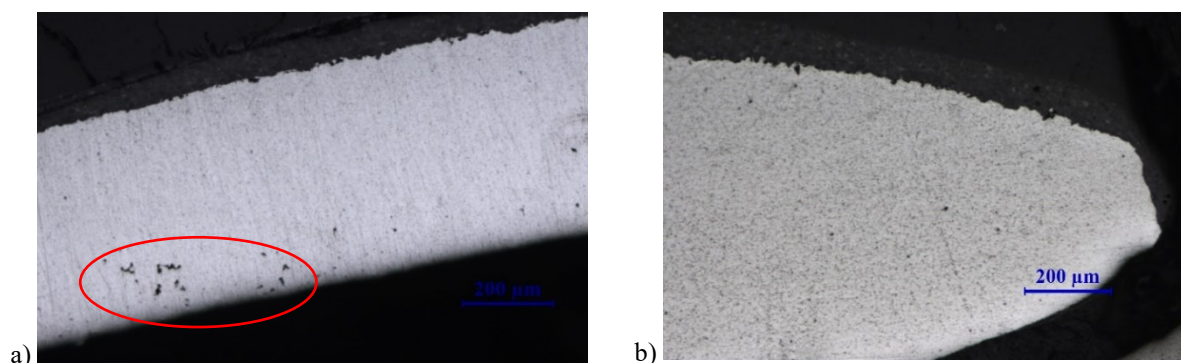


Figure 7. Microstructure of the NiCrMo alloy.

The figure 8 shows the ceramic layer and interlayer. The dimensions of the ceramic layer were ranging approx. from 630.2 μm to 717.6 μm . The interlayer in figure 8 exhibited the dimensions ranging from 100.9 μm to 119.3 μm , but in the other site (figure 9a), it was in the range from 52.7 μm to 75.64 μm .

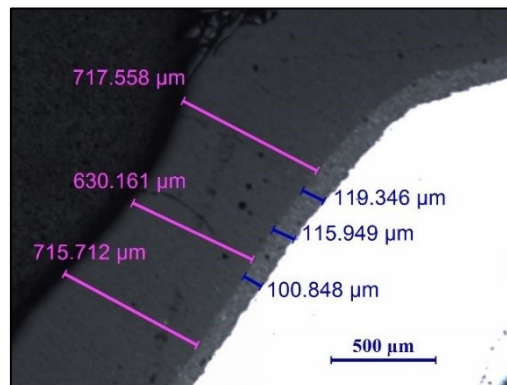


Figure 8. Layers of dental replacement.

Under the interlayer, there was the occurrence of particles with dimensions of 16.76 μm and 10.55 μm (figure 9b). Probably, it is the penetration of ceramic particles migrating through the interlayer but it could also be the presence of impurities or corrosive particles and their presence would significantly affect the functionality of the ceramic-alloy joint. There is also a high probability of Cr and Mo-based carbide phases occurrence.

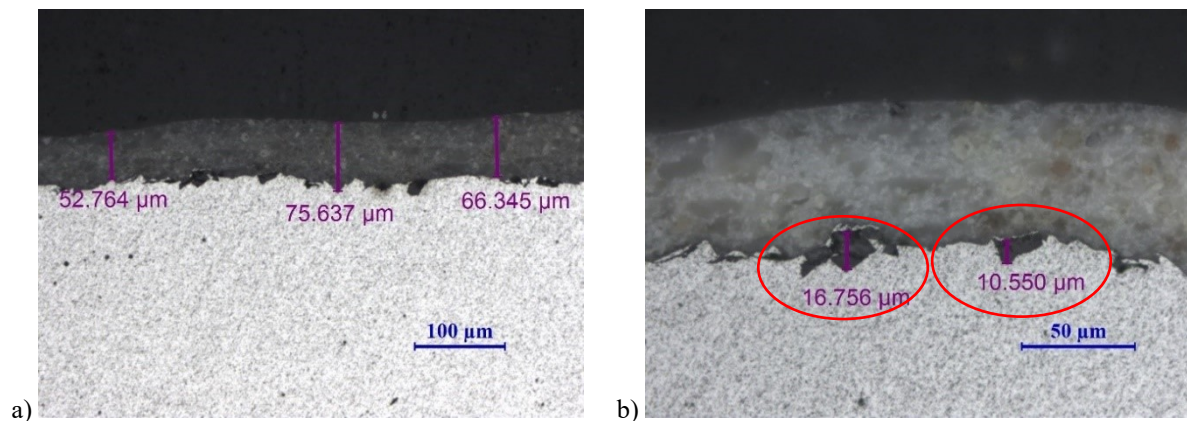


Figure 9. NiCrMo alloy surface area.

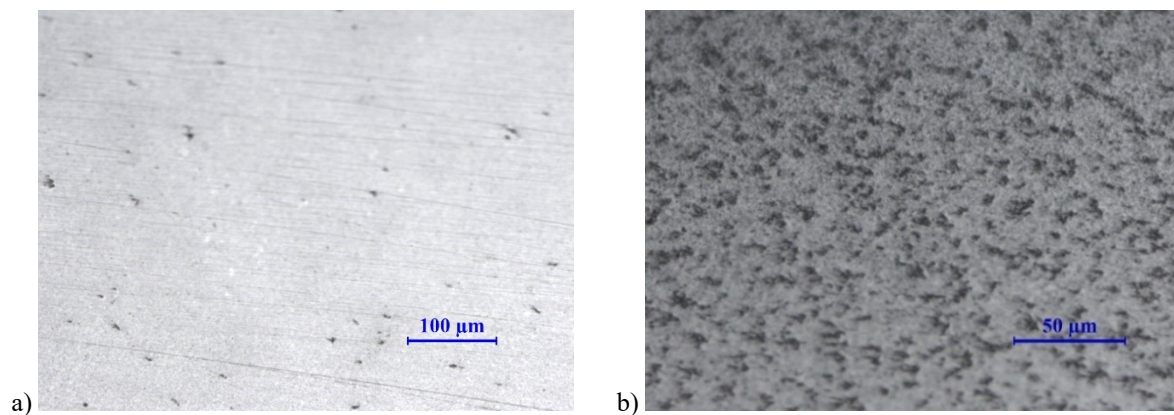


Figure 10. Evaluation of microstructure of Ti-6Al-4V alloy.

Ti-6Al-4V alloy was used for the screw part of the dental replacement. The evaluation of the micro-purity of the titanium alloy revealed a relatively high occurrence of fine sharp-edged inclusions with

varying size and minor oxides phases, in the whole volume (figure 10a). The microstructure consists of two basic phases. They are α (light) and β phase (dark), without significant grain boundaries (figure 10b).

In the area which is surrounding the screw (figures 11a and 11b), a light layer with different thicknesses from 49.5 μm to 57.4 μm was observed. It can be assumed that in accordance with the thermodynamic analysis of the ternary system of Ti-Al-V alloys (according to Rault's Law), the Ti-V system has a higher activity than the Ti-Al and Al-V systems. This means that during the temperature treatment, vanadium or aluminium may precipitate from the surface of the titanium alloy [7].

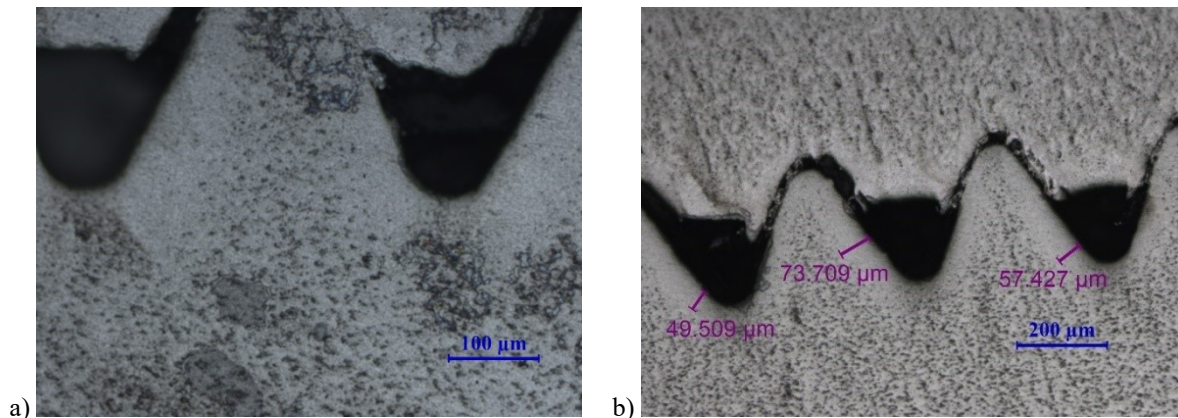


Figure 11. Microstructure of Ti-6Al-4V alloy.

This phenomenon results in occurrence of areas where the precipitate (β -phase) has a finer structure and the individual grains have a different size (figure 11). This structure could have been formed during the heat treatment while the material was being cooled.

Cp-Ti screw consists a large number of impurities, cavities and pores in microstructure and it can create stress in the structure and lead to the cracks initiation (figure 12).

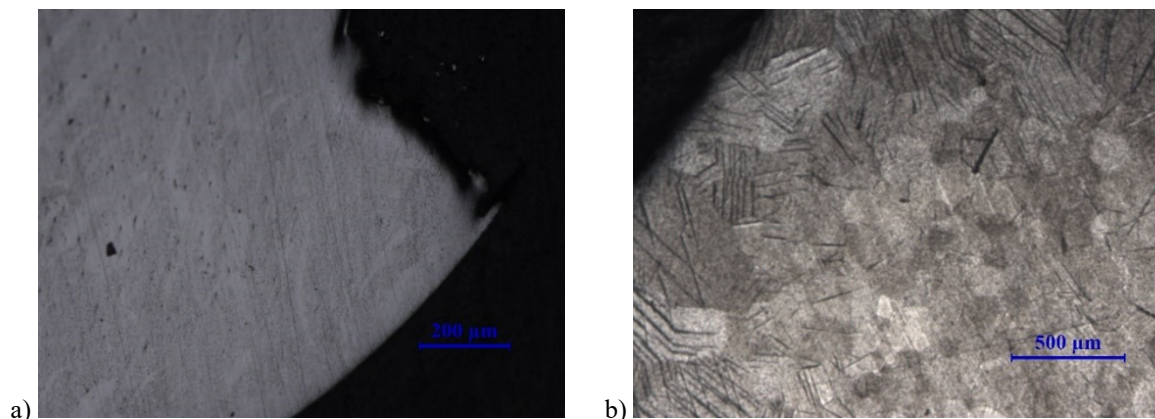


Figure 12. Microstructure of pure titanium a) non-etched b) etched structure.

In the peripheral areas of the screw, there was a noticeable occurrence of twinning and slip lines (figure 11), and it characterizes the plastic deformation process. In some grains of the studied structure, needles (lamellas) were initiated due to polymorphic transformation of the β -phase during cooling.

Since cp-Ti has the ability to form a stable layer of Ti-oxide quickly on its surface, it has excellent osteointegration with the bone because it forms a calcium phosphate-enriched layer. Another

advantageous feature is that in the case of loss of the protective layer (Ti-oxides and Ca-P), the given protective layer is able to be regenerated very quickly [13, 14].

5. Results and discussion

Using the chemical analysis, the aim of the research was to determine which materials were used for manufacture of the individual parts of the dental replacement, while the given chemical analysis was based on the prematurely removed dental replacement from the patient's oral cavity.

The microscopic study of the structure revealed that the quality of the individual component materials of the dental replacement was not satisfactory. Based on the elemental analysis, it was shown that the ceramics and NiCrMo alloy also contained undesirable chemical elements which are dangerous for the living organism. Furthermore, an aluminum-containing titanium alloy was used for the screw connection. Only one component was made of pure titanium, which is inert to living cells. In addition, the micro purity of cp-Ti was not satisfactory.

The implant as a functional structure has to meet the basic requirements from the aspect of materials, both in terms of chemical composition and in terms of wear resistance and especially corrosion. Scientific studies have shown that patients may be allergic to any element that is contained in a dental implant (Ni, V, AL, Cr, Ba, In etc.) when the given element is released into the body. This phenomenon is very difficult to prove. In general, the failure of the dental replacement is due to the occurrence of the critical states or even degradation of any part. If anything is released from the dental structure or even the whole dental replacement is released from the bone, it will lead to malfunction or in worse case it can be dangerous from aspect of the health of the patient. For this reason, dental replacement materials have to be monitored for any change which may result in the critical states. The mentioned critical states are based on the formation of oxides, wear or peeling (flaking) of the material with subsequent release into the human body.

Despite progressive and innovative metallurgical and technological advances in the development of surgical and dental materials, there is permanent occurrence of critical states malfunction and failure. Prevention from the critical states occurrence of dental replacement is to keep the requirements relating to micro-purity of used materials, precise surface treatment of metal and non-metallic parts of dental replacements. And last but not least, a perfect surgical procedure has to be performed from the aspect of the insurances of the correct geometry and placement of the implant in the bone to avoid the undesirable stress in the patient's bone.

6. Conclusions

Based on the examination of prematurely extracted dental replacements, we recommend to keep all predetermined requirements for technological and dental procedures during their application and thus prevent from the occurrence of these undesirable conditions:

- defects which occur during casting and surface treatment,
- unsatisfactory micro-purity of materials,
- deformations of the screw connection,
- incorrect placement of the dental replacement in the patient's bone,
- penetration of food residues into some part of the dental replacement.

It is also necessary to perform a continuous control of these dental replacements in the patient's oral cavity.

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Acknowledgements

This paper was supported by the Slovak Grant Agency – projects KEGA 007TnUAD-4/2017 and VEGA 1/0649/17. This work was created with the support of the project „Advancement and support of R&D for “Centre for diagnostics and quality testing of materials“ in the domains of the RIS3 SK specialization“, code NFP313010W442.