EVALUATION OF MECHANICAL PROPERTIES OF TITANIUM-BASED ALLOY FOR USE IN DENTISTRY

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Abstract

Titanium and its alloys are promising materials in the dental filed because they have good biocompatibility, corrosion resistance and physical and mechanical properties. For that reason, they have many applications as crowns, inlays, bridges, dental implants etc. Titanium can be alloyed with other metals, such as chromium and niobium, to modify its mechanical properties. So, titanium alloys are increasingly being developed with the aim of obtaining a material with excellent biocompatibility and satisfactory mechanical properties for dental applications. In this work four samples of Ti-10Cr-10Nb alloy were produced in a laboratory arc furnace under argon atmosphere in the shape of button. They were re-melted for four times in the same conditions to ensure chemical homogeneity. Casting of four alloys in the shape of plates with different thickness was obtained by standard dental procedure and vacuum-pressure technique. Their Vickers hardness (HV 2) and microhardness (HV 0.2) as well as bending strength were determined. Microstructure was observed by light microscope. Surface of alloys after 3-point bending test was analyzed by scanning electron microscope with energy-dispersive spectrometer. It was found that all alloys have similar two-phase microstructure, hardness which satisfies dental requirements and high bending strengths values. From the obtained results it follows that experimental alloy has potential for further investigation for use in dentistry.

Keywords:
Titanium alloys, dentistry, microstructure, mechanical properties.

1. INTRODUCTION

In dentistry, titanium and its alloys have many applications in orthodontics, endodontics, prosthetics and implantology. Thus, they are applied to dental products such as crowns, inlays, bridges etc. as well as dental implants. The primary benefit of titanium and titanium alloys as materials for dentistry is due to their corrosion resistance and biocompatibility in the oral cavity compared to the other metallic materials [1,2]. However, it is difficult to cast these alloys by conventional dental casting techniques, since titanium is so reactive at high temperatures, easily oxidized, and reacts with the crucible and mold components. The formation of these harden, brittle reaction layers is important in the production of prostheses such as crowns and bridges or removable partial denture frameworks. Investment elements diffused to the interior of the titanium castings will change their mechanical properties. Precise titanium casting depends on the casting system and mold materials [3,4]. When molten titanium is cast into an investment shell, there is invariably some degree of reaction between the metal and the shell, resulting in a contaminated surface layer. Oxygen increases the hardness and brittleness of titanium alloys, while other elements may result in inhomogeneous microstructure [5].

In our previous work [6] new Ti-10Cr-10Nb alloy was designed. Investigation presented in this paper is one step further towards the application of this alloy in dental practice. So, for that purpose four samples of plates, which should serve for implant component production, were prepared in dental laboratory by standard
procedure. Microstructure and mechanical properties of castings considering the investment material impact on them were evaluated.

2. MATERIALS AND METHODS

In this work four samples of Ti-10Cr-10Nb alloy (~8 g each) were produced by melting pure elements (>99.9%) in a laboratory arc furnace under argon atmosphere. A water-cooled copper crucible was used to avoid contamination of reactive materials. Samples were re-melted for four times to ensure chemical homogeneity. Casting of four alloys in the shape of plates with different thickness was obtained by standard dental procedure. Metallic plates were prepared using wax patterns with dimensions 32 x 10 x h mm and invested in a mold ring with alumina-zirconite-bonded investment for titanium alloys. Molds have been preheated for one hour at 750°C. The alloy samples were then melted and casting was completed in a commercial dental vacuum-pressure casting machine (Dentaurum). The molds were cooled at room temperature before divesting. The castings were then sandblasted with Al₂O₃ particles of 110 µm in a sandblasting plant (Manfredi).

3-point bending tests were performed on LRX testing machine with Nexygen software for dynamic testing. The bending strengths were determined using the equation: \( \sigma = \frac{3PL}{2bh^2} \), where \( \sigma \) is the bending strength (MPa), \( P \) is the load (N), \( L \) is the span length (mm), \( b \) is the specimen width (mm) and \( h \) is the specimen thickness (mm) [7]. Plates were then analyzed by scanning electron microscope SEM Tescan Vega TS 5136 MM with integrated energy-dispersive spectrometer (EDS). Fractured and bended plates were embedded in conductive resin and metallographically prepared by SiC-paper grinding and Al₂O₃ water suspension polishing on microcloth. Vickers hardness (HV 0.2 and HV 2) was measured on Leica VMHT 302801 machine. Microstructure of samples etched in Kroll’s reagent was observed by light microscope Olympus GX51 equipped with digital camera.

3. RESULTS AND DISCUSSION

The surface of plates with different thickness was smooth and without cracks. They are subjected to the 3-point bending test. Plates 1 and 2 were bended, but not fractured, while plates 3 and 4 were broken. Bending strength values for all samples are given in Table 1. Plate 1 was quickly bended at load of 172.73 N resulting in the lowest bending strength. It follows that this sample is very ductile. Plate 2 was also bended, but the ratio of bending was much smaller than at plate 1 resulting in the highest strength. Plate 3 shows high strength value but it was broken at load of 843.13 N. Plate 4 was broken too but at lower load of 426.42 N which make this sample very brittle. Bending strengths of plates 2 and 3 are much higher than those for commercially pure titanium and binary Ti-(5-30)Cr alloys [8] and very similar to those for ternary Ti-5Nb-(1-13)Cr alloys [7].

Vickers hardness was examined in cross-section at five spots and the mean values are given in Table 1. It can be seen that all plates exhibit very similar values and typical for dental alloys.

<table>
<thead>
<tr>
<th>Mechanical property / Sample</th>
<th>Bending strength, ( \sigma ), MPa</th>
<th>HV 2</th>
<th>HV 0.2</th>
</tr>
</thead>
<tbody>
<tr>
<td>Plate 1</td>
<td>746</td>
<td>304</td>
<td>344</td>
</tr>
<tr>
<td>Plate 2</td>
<td>2413</td>
<td>306</td>
<td>361</td>
</tr>
<tr>
<td>Plate 3</td>
<td>2200</td>
<td>312</td>
<td>386</td>
</tr>
<tr>
<td>Plate 4</td>
<td>1417</td>
<td>321</td>
<td>394</td>
</tr>
</tbody>
</table>
Surface of experimental plates after 3-point bending test were analyzed using SEM with EDS (Fig. 1). Figure 1a shows that on plate 1 there is no oxide layer on metal surface and EDS shows characteristics peaks for Ti, Cr and Nb only. On plate 2 there is a visible surface layer (Fig. 1b) analyzed by EDS. Resulting spectrum shows presence of various oxides originating from investment material. Thickness of this layer was measured and the results are in Table 2.

Fractographs of broken plates 3 and 4 (Fig. 1c, 1d) show coarse cleavage facets in the fractured surface, which are characteristic of decreased ductility, together with some terrace-type morphology. This is very similar to morphology of Ti-10Cr alloy in paper [8]. The cleavage fractures correspond with the highly brittle nature of the specimens.

![SEM micrographs with EDX spectrum](image1)
![Fractographs](image2)

**Fig. 1** SEM micrographs with EDX spectrum (a,b) and fractographs (c,d) of plates after 3-point bending test
Thickness values of plates together with thickness of metal and oxide layer are given in Table 2. Thickness of plates increases from plate 1 to 4, as well as thickness of oxide layer, but not as thickness of metal. This affects on bending strength. Plate with the highest oxide layer (plate 4) exhibited the lowest bending strength, except plate 1 which has no oxide. Vickers hardness dependence on thickness of oxide layer is shown in Fig. 2. It follows that plate 1 without an oxide layer on its surface shows the lowest hardness values. Plates with higher oxide thickness exhibited higher hardness values due to diffusion of elements from investment material and hardening effect.

**Table 2** Thickness (d) of plates, metallic and oxide layers

<table>
<thead>
<tr>
<th>Sample</th>
<th>d (plate), mm</th>
<th>d (metal), mm</th>
<th>d (oxide layer), mm</th>
</tr>
</thead>
<tbody>
<tr>
<td>Plate 1</td>
<td>0.82</td>
<td>0.82</td>
<td>0.00</td>
</tr>
<tr>
<td>Plate 2</td>
<td>1.26</td>
<td>1.06</td>
<td>0.20</td>
</tr>
<tr>
<td>Plate 3</td>
<td>1.25</td>
<td>1.00</td>
<td>0.25</td>
</tr>
<tr>
<td>Plate 4</td>
<td>1.27</td>
<td>0.95</td>
<td>0.32</td>
</tr>
</tbody>
</table>

**Fig. 2** Dependence HV2 and HV 0.2 on oxide layer thickness

Light micrographs of etched samples show that in microstructure of plate 1 (Fig. 3a) are very coarse grains of matrix. According to [6] it is a beta phase. In other micrographs (Fig. 3b-d) is obvious presence of second phase (alpha, according to [6]) in similar amounts. Microstructure of plate 2 consists of the highest amount of alpha phase which increases the bending strength as well as hardness. It is known that alpha phase has higher hardness values than beta phase of titanium [9].
3. CONCLUSIONS

In this paper the effect of the investment material on the mechanical properties of Ti-10Cr-10Nb alloy castings was examined. According to obtained results it can be concluded that:

1) All samples, except plate 1, had high bending strengths (1417-2413 MPa)
2) Plate 1 without oxide layer on its surface exhibited very high ductility as well as plate 2 with the lowest thickness of that layer
3) Higher thickness of oxide layer on plates 3 and 4 caused their embrittlement
4) Vickers hardness of all samples were similar and satisfied dental requirements
5) Hardness values have tendency to increase as the thickness of oxide layer increased
6) Microstructure of plate 1 was single-phase with coarse grains
7) Oxide layer on surface of plates 2, 3 and 4 did not allow a complete alpha/beta transformation, so their microstructure is two-phases.

Results presented in this paper show that dental casting should produce plates with the required thickness of the metal plates without or with a minimal amount of oxide containing in investment material in order to obtain required mechanical properties for application in dentistry.

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LITERATURE


