Researches on the Influence of Thermal Treatment on the Mechanical Properties of Titanium Dental Prostheses

DIANA DIACONU POPA, ANCA VITALARIU*, CATALINA HOLBAN CIOLOCA, ANDRA AUNGURENCEI, ODETTE LUCA, MONICA TATARCIUC
Grigore T. Popa University of Medicine and Pharmacy Iasi, Faculty of Dental Medicine, Department of Implantology, Removable Restorations and Dental Technology, 16 Universitatii Str., 700115, Iasi, Romania

The longevity of dental prostheses depends on the strictness of following the clinical-technological algorithm. The main method of obtaining the metallic dental prostheses is casting, which involves a succession of stages that may produce modifications in the alloy structure, that will lead to alteration of its biological and mechanical behaviour. The modification of some technological parameters influences the internal microstructure and, implicitly, the mechanical properties of the metallic dental prostheses. The paper aims to investigate the influence of cooling rate on the mechanical properties of the titanium moulded pieces. We realized 40 samples divided in four groups consisting of 10 samples each: one group as control, and three test groups. The 30 test samples underwent a thermal treatment of homogenization, represented by a heating process up to 400 °C on 0.1 °C per second. The samples were maintained at 400 °C for 2 min, and, after that were cooled at different rate: group 2: 70°C/s, group 3: 30°C/s, and group 4: cooled with an average rate, varying from 30 to 70 °C/s. Group 1 was the control group consisted in 10 samples without thermal treatment. The results showed that mechanical properties of the control group are inferior comparing to the samples submitted to the thermal treatment. Among the three groups of samples that have been thermal treated, the samples that have been cooled at an average rate, varying from 30 to 70 °C/s (group 3), have optimal mechanical properties.

Keywords: titanium, cooling rate, mechanical properties

Titanium has come more and more to prominence in dentistry, due to its special properties in comparison with other based metals and alloys [1-2]. Due to its particular chemical stability, high resistance to corrosion, and excellent biocompatibility, Ti has been extensively used in dentistry, for fixed and removable dental restorations [3-7], and orthodontic treatments [8,9]. Pure titanium is composed by 99.5% of titanium and 0.5% of interstitial elements (carbon, oxygen, nitrogen, hydrogen and iron) and the proportion of these elements directly affects the metal properties. The ASTM (American Society of Testing and Materials) Standard F1295 specifies titanium in different grades according to its purity, which is evaluated according to the amount of oxygen. The titanium melted only from titanium sponge is known as titanium grade 1, which is considered the most pure grade. When titanium sponge is mixed with titanium fragments, the amount of oxygen (O) and iron (Fe) increases and titanium becomes harder. The more fragments are added, the harder the titanium becomes (titanium grades 2, 3 and 4) [10].

Casting represents the conventional technique used for making titanium dentures, but, due to the higher melting temperature of titanium, near 1,672°C, requires special melt procedures, cooling cycles, investments and equipment to avoid its contamination [11]. These requirements must be followed because titanium suffers a change in its crystalline state at 883°C, where the alpha-hexagonal phase changes to beta-cubic phase[12]. Together with alloy composition, all the algorithm's steps (the type of investment and the investing technique, the metal melting, casting and cooling conditions, type of casting machine), have great influence on the final quality of casts[13-15].

Our paper aims to investigate the influence of one technological parameter-cooling rate-on the mechanical properties of the commercially pure titanium (cpTi) moulded pieces: elastic limit, maximum elongation and ultimate tensile strength.

Experimental part

Material and method

For this research 40 rectangular cross-section samples (for tensile testing) were made using commercially pure titanium Rematitan M (Dentaurum/Germany) with the following composition (table 1).

<table>
<thead>
<tr>
<th>Ti</th>
<th>Fe</th>
<th>O</th>
<th>N</th>
<th>C</th>
<th>Mg</th>
</tr>
</thead>
<tbody>
<tr>
<td>99.6 %</td>
<td>0.15 %</td>
<td>0.12%</td>
<td>0.005 %</td>
<td>0.006 %</td>
<td>0.013%</td>
</tr>
</tbody>
</table>

The samples were obtained through the lost-wax technique, using rectangular cross-section wax patterns (0.7 x 4 mm), gauge length of 20 mm and total length of 44 mm (fig. 1). The gating system consists in sprues and air vents of 3 and 1 mm in diameter, respectively. The investing material used was Rematitan Ultra (Dentaurum/Germany), a spinel-based investment material specially developed for titanium casting technique, in order to reduce the alpha case layer formed at the contact zone between the titanium and the investment (10-30 µm).

* email: ancavitalariu@yahoo.com
The especially strong affinity of titanium to oxygen is taken into consideration in the melting and casting process. Titanium is highly reactive at its melting temperature of 1720°C, therefore an automatic argon-arc vacuum-pressure casting machine (Castmatic-S, Iwatani, Japan) was used to cast the metal [16]. This machine has an upper chamber where the metal is melted, and a lower one, containing the mold. After the chambers are evacuated with a vacuum pump, the upper chamber is filled with compressed argon gas. An argon arc is generated between a tungsten electrode and the metal, melting the metal which then, due to the pressure differential, fills the mold [17-19].

The castings were allowed to bench cool to room temperature. After divesting, the samples were sandblasted with 50 µm Al₂O₃ particles and desprued with an ultrathin separating disc, at a speed of 15,000 rpm. The samples were divided in four groups consisting of 10 samples each: one group as control, and three test groups. The 30 test samples underwent a thermal treatment of homogenization, represented by a heating process up to 400°C on 0.1°C per second. The samples were maintained at 400°C for 2 min, and, after that were cooled at different rate: group 2: 70°C/s, group 3: 30°C/s, and group 4: cooled with an average rate, varying from 30 to 70°C/s. Group 1 was the control group consisted in 10 samples without thermal treatment.

To evaluate the mechanical properties, the cooled samples were subjected to a tensile testing in order to analyze the elastic limit (yield strength), maximum elongation and ultimate tensile strength. The elastic limit represents the maximum pressure that the material can undergo without a permanent deformation. Elongation is the deformation resulting from the application of a tensile force. The elongation of the material can be conventionally divided into two stages: an increase of the sample's length, before the limit of proportionality, which is not permanent and is proportional to the applied force, and the elongation from the limit of proportionality until fracture, which is a permanent deformation. Ultimate tensile strength is measured by the maximum stress that a material can withstand while being stretched or pulled before breaking.

The tensile testing was carried out according to ISO 6892-1: 2009, at room temperature using a computer controlled testing machine (Instron 3382) with a constant crosshead speed of 1 mm/min (fig.2).

Results and discussions
The elastic limit
Our results for elastic limit (yield strength) are presented in table number 2. We noticed that the elastic limit was higher in samples cooled at rate of 30°C/s, in comparison with the samples cooled at the higher rate (70°C/s). The control group showed the highest value of elastic limit. The higher the elastic limit values, the more fragile the structures will be.

Maximum elongation
Permanent deformation (elongation ε) was determined by measuring the dimensional increase between two fixed points of the sample, and calculated according to the formula:

$$\varepsilon = \frac{|\text{deformation}|}{\text{initial length}} \times 100.$$  

The results obtained were illustrated in the table number 3. The lowest value of elongation was noticed in samples of Group 4 (cooled at average rate, varying from 30 to 70°C/sec) and the highest one in the control group. If the value of elongation is lower, it also means that the deformation of that structure will be lower; therefore, metals and alloys with lower values of maximum elongation will be more indicated for making dental prostheses [20,21].

Ultimate tensile strength
Ultimate tensile strength (UTS), often shortened to tensile strength (TS) is defined as a stress, which is measured as force per unit area. Our values were determined by the ratio between the maximum tensile strength and the section area of the sample. The results showed that the highest tensile strength was in group 4, and the lowest in control group (table 4).

<table>
<thead>
<tr>
<th>Table 2</th>
<th>THE ELASTIC LIMIT OF THE SAMPLES</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Group 1 (control group)</td>
</tr>
<tr>
<td>Elastic limit</td>
<td>398.21 Mpa</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Table 3</th>
<th>MAXIMUM ELONGATION OF THE SAMPLES</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Group 1 (control group)</td>
</tr>
<tr>
<td>Maximum elongation</td>
<td>22.7%</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Table 4</th>
<th>TENSILE STRENGTH VALUES</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Group 1 (control group)</td>
</tr>
<tr>
<td>Tensile strength</td>
<td>386.2 Mpa</td>
</tr>
</tbody>
</table>
Conclusions
The longevity of prosthetic constructions depends on the strictness of surveying the technological algorithm. Our study proves that variation of cooling rate after heating determines modifications of studied mechanical characteristics: elastic limit (yield strength), maximum elongation and tensile strength, the samples submitted to thermal treatment showing better values than the control group. From the three groups cooled after the heating, the ones that were cooled at an average rate, from 30 to 70°C, have the best mechanical properties.

Based on these data, we can conclude that under appropriately adjusted conditions, high quality titanium prosthetic appliances may be produced.

References
5. MARECI, D., CHELARIU, R., IVANESCU S., GORDIN, M., CRETESCU, I., GLORIANT, G. Rev. Chim (Bucharest), 66 no. 8, 2009, p.787
15. CHAN D, GUILORY V, BLACKMAN R, CHUNG KH. J Prosthet Dent 1997;78, p.:400
19. ZINELLIS S., J Prosthet Dent.;84, 2000,p.575

Manuscript received: 5.04.2017