STRUCTURE AND PROPERTIES OF TITANIUM FOR DENTAL IMPLANTS

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Abstract

This paper describes manufacture of ultra-grained titanium, its structure and properties. Ultra-grained titanium has higher specific strength properties than ordinary (coarse-grained) titanium. Ultra-grained titanium was produced by the ECAP process. The research itself was focused on physical base of strengthening and softening processes and developments occurring at the grain boundaries during the ECAP process at half-hot temperature. Strength of ultra-fine grained titanium varies around 960 MPa, grain size around 300 nm.

Keywords: ultrafine-grain titanium, ECAP process

1. INTRODUCTION

It is required that a material for dental implants is bio compatible, it must not be toxic and it may not cause allergic reactions. It must have high ultimate strength $R_m$ and yield value $R_p$ at low density $\rho$ and low modulus of elasticity $E$. Metallic materials used for dental implants comprise alloys of stainless steels, cobalt alloys, titanium (coarse-grained) and titanium alloys [1, 2]. Semi-products in the form of coarse-grained Ti or Ti alloys are used as bio-material for medical and dental implants since the second half of the sixties of the last century [3, 4]. Titanium is at present preferred to stainless steels and cobalt alloys namely thanks to its excellent bio-compatibility [5]. Together with high biocompatibility of Ti its resistance to corrosion evaluated by polarisation resistance varies around the value $10^3 \Omega \cdot \text{cm}$ [6]. For these reasons pure titanium still remains to be a preferred material for dental applications. Development trend in case of this material is oriented on preservation of low value of the modulus of elasticity and on increase of mechanical properties, especially strength. According to the Hall-Petch relation it is possible to increase considerably strength properties of metals by grain refinement [7]. That’s why it is appropriate to use for dental implants rather fine-grained Ti instead of coarse-grained Ti. Use of ultra-grained concerns numerous fields including medicine [8]. Bulk ultra-grained structural metallic materials are used for dental applications. These are materials with the grain size smaller than approx. 100 to 300 nm. High-purity titanium is used for dental implants.

2. MECHANICAL PROPERTIES OF DENTAL MATERIALS

Ultra-grained titanium is characterised by exceptional mechanical properties, among which high ultimate strength and high yield value are of utmost importance. Strength properties of ultrafine-grained titanium must have the following values: $R_m > 1000 \text{ MPa}$, $R_{0,2} > 850 \text{ MPa}$. Apart from the strength, another important properties of dental implants is their so-called specific strength (strength related to density). Mechanical properties of metallic material for implants are evaluated in relation to its density as so-called specific properties. In case of classical coarse-grained titanium the relation $R_m/\rho$ varies around 70 to 120 (N·m/g), for the alloy Ti6Al4V it varies around 200 (N·m/g), and for (n)Ti it is possible to predict the values $R_m/\rho = 270 \text{ (N·m/g)}$. As a matter of interest it is possible to give the specific strength also for some other
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dental materials: steel AISI 316L - $R_m/p = 65 \text{ (N.m/g)}$, cobalt alloys $R_m/p = 160 \text{ (N.m/g)}$. Disadvantage of dental implants based on steel or cobalt alloys is their high tensile modulus of elasticity: $E = 200$ to 240 GPa, while in case of titanium and its alloys this value varies between 80 and 120 GPa [9]. At present only few companies in the world manufacture commercially bulk nano-materials.

2.1 Materials of dental implants

The use of endosseous implants for replacing missing teeth became possible, only when scientists found a suitable material such as titanium that could integrate with human bone. The materials used for implantation must have some specific characteristics such as immunity to corrosion, bio-compatibility, strength, damage tolerance and capacity for joining with bone and other tissues (osseointegration).

**CP Titanium**

Commercially pure Titanium is the first material found that combines the most favorable mechanical and physical properties for successful use in dental implants. It is immune to corrosion by body fluids, acids and oxygen, it is bio-compatible, hard enough to withstand the forces of chewing and osseointegrates well with the jawbone. Titanium also is almost completely nonmagnetic and is extremely strong for its weight. The majority of dental implants are made of commercially pure titanium which ~99% titanium and small amounts (0.18-0.40%) of oxygen with trace amounts of iron, carbon, nitrogen, and hydrogen. The concentration of carbon and iron determines the grade of the alloy (1-4).

*Titanium alloy (Ti-6Al-4V or grade 5 titanium).*

Recently there is increased use of this titanium alloy containing 90% Titanium, 6% Aluminum and 4% Vanadium. It is believed to offer better strength and fracture resistance with similar osseointegration performance as commercially pure titanium.

**Zircon**

Dioxide of zirconium (ZrO$_2$) is a new type of material used by some manufacturers of dental implants. Zirconium is a metal with similar biocompatibility properties with titanium. Zircon implants are to be used when there are more aesthetic requirements such as for restoring front teeth but they are much more expensive than titanium ones. The Zircon type of dental implants can offer better aesthetic results because the color of the implant components is completely white and no metal is visible through the ceramic restoration affecting the tooth color.

2.2 Types and size of dental implants

The average width for standard implants ranges from 3.5 to 4.5mm but several factors can make necessary the use of different width implants. The dentist must evaluate properly the condition of the patient's jaw (width and depth of jawbone, bone density) and the position of the missing tooth in the mouth and in relation to the adjacent teeth.

**Narrow form implants (small diameter)**

The implant must not disturb the roots of the natural teeth on its sides. If the empty space is not wide enough, the dentist may decide to use narrower implants to allow adequate space from adjacent roots for better osseointegration of the implant. Narrow implants are also known as mini dental implants and their diameter varies from 1.8 to 3.5mm.

**Wide form implants (large diameter)**

Back teeth (molars and premolars) have to withstand much more load than the rest of the teeth during chewing. If there is enough healthy jawbone in the area, the dentist may prefer to use wide form implants for better stability and force distribution. Wide platform dental implants range between 4.5 - 6.0mm in diameter.
Shorter implants

If there is close proximity with a facial nerve, a shorter implant has to be used to avoid the risk of nerve damage (often used for front teeth). The use of shorter types of dental implants is also recommended in some special cases for the upper jaw to avoid damage to the sinus.

The use of smaller diameter implants (mini dental implants / MDI) has increased significantly over the recent years. Many dentists promote the use of small diameter implants because they involve less surgical time making them a simpler and low cost solution. Patients seeking for affordable implants must be aware that small diameter implants are designed to be used only under certain circumstances that do not allow the use of standard width implants. They are not made to substitute standard implants and only a small number of narrow width implants are approved by FDA for long term use.

3. TECHNOLOGY FOR MANUFACTURE OF ULTRA-FINE GRAINED TITANIUM

The main objective of experiments was manufacture of ultra-fine grained titanium, description and optimisation of its properties from the viewpoint of their bio-compatibility, resistance to corrosion, strength and other mechanical properties from the viewpoint of its application in dental implants. Chemical purity of semi products for titanium was ensured by technology of melting in vacuum and by zonal remelting. The obtained semi-product was under defined parameters of forming processed by the ECAP technology. The output was ultra-grained titanium with strength about 1050 MPa. The obtained ultrafine-grained titanium was further processed by technology (of rotation forging) and drawing to the shape suitable for dental implants.

4. STRUCTURE AND PROPERTIES OF TITANIUM

Commercially pure titanium (CP) bars and sheets were used in this study. The average grain size of the as-received CP titanium is ASTM no. 4. Tensile specimens with a gauge of 50 mm length, 10 mm width and 3.5 mm thickness were machined with the tensile axis oriented parallel to the final rolling direction. The specimens were deformed at room temperature with different initial strain rates. After testing, the deformed specimens in order to preserve the microstructure Fig. 1-3. Specimens were sectioned along the gauge and grip parts of the deformed sample. The samples were then polished etched using 10 % HF, 10 % HNO₃ and 80 % H₂O for 20 second. Chemical analysis and mechanical properties titanium are given in the Table 1-3.

![Fig. 1 Initial microstructure of titanium](image1)

![Fig. 2 Microstructure of titanium after cold rolling (deformation e = 46 %)](image2)
Table 1  Chemical analysis commercially pure titanium (CP), (weight %)

<table>
<thead>
<tr>
<th>N</th>
<th>O</th>
<th>C</th>
<th>Fe</th>
<th>Al</th>
<th>Cr</th>
<th>Ti</th>
</tr>
</thead>
<tbody>
<tr>
<td>0,004</td>
<td>0,068</td>
<td>0,008</td>
<td>0,03</td>
<td>0,01</td>
<td>0,01</td>
<td>Rest.</td>
</tr>
</tbody>
</table>

Table 2  Mechanical properties of CP titanium after annealing 649 °C/1 hour (ASTM E8)

<table>
<thead>
<tr>
<th>Tensile strength [MPa]</th>
<th>Yield strength [MPa]</th>
<th>Elongation [%]</th>
<th>Reduction of area [%]</th>
</tr>
</thead>
<tbody>
<tr>
<td>365</td>
<td>212</td>
<td>51</td>
<td>71</td>
</tr>
</tbody>
</table>

Table 3 Initial hardness of commercially pure Ti and hardness after cold rolling

<table>
<thead>
<tr>
<th>Hardness</th>
<th>Initial sample</th>
<th>Sample after cold rolling</th>
</tr>
</thead>
<tbody>
<tr>
<td>HV30</td>
<td>1  2</td>
<td>1  2  3  4  5  6</td>
</tr>
<tr>
<td>128 128</td>
<td>140 139 131 200 202 205</td>
<td></td>
</tr>
</tbody>
</table>

Fig. 3  Microstructure of CP titanium after: a) cold deformation 98 %; b) annealing 670°C / 2 hour

5. OBTAINED RESULTS AND THEIR ANALYSIS

Semi products from individual heats were processed according to modified programs by the ECAP technology and then drawn to a wire. Wire diameter varied about 5 - 8 mm [10,11]. ECAP technology and drawing was made in variants:

a) 8 passes ECAP at a temperatures of 280 °C; with annealing between individual passes.

b) rotation re-forging to a diameter of 10 mm (cold forming : e = 2.2).

c) The following technology of drawing was realised at increased temperatures.

The samples for mechanical tests and for micro-structural analyses were prepared from individual variants of processing. On the basis of the results, particularly the obtained strength values, several variants were
chosen for more detailed investigation of developments occurring in the structure at application of the ECAP and subsequent drawing after heat treatment. Structure of ultra-fine grained titanium after application of the ECAP process is shown in the Fig. 4, 5 and Fig. 6, 7. The structure was analysed apart from light microscopy also by the X-ray diffraction. Table 4 summarises the obtained basic mechanical properties.

![Fig. 4](image1.png)  
**Fig. 4** Microstructure of titanium after 2 passes ECAP

![Fig. 5](image2.png)  
**Fig. 5** Microstructure of titanium after 4 passes ECAP

![Fig. 6](image3.png)  
**Fig. 6** Microstructure of titanium after 6 passes ECAP

![Fig. 7](image4.png)  
**Fig. 7** Microstructure of titanium after 8 passes ECAP

<table>
<thead>
<tr>
<th>Forming processed</th>
<th>$R_m$ [MPa]</th>
<th>$A$ [%]</th>
<th>$E$ [GPa]</th>
<th>$d_z$ [nm]</th>
</tr>
</thead>
<tbody>
<tr>
<td>ECAP (8 passes)</td>
<td>960</td>
<td>12</td>
<td>100</td>
<td>100 to 300</td>
</tr>
<tr>
<td>Drawing (D$_a$ = 6 mm)</td>
<td>1030 to 1050</td>
<td>9</td>
<td>100</td>
<td>100 to 300</td>
</tr>
</tbody>
</table>
CONCLUSION

Technology of manufacture of ultrafine-grained titanium was proposed and experimentally verified. Grain refinement in input materials was obtained using the ECAP process. In conformity with the Hall-Petch relation the strength properties of titanium increased significantly as a result of grain refinement. The obtained mechanical properties correspond with the declared requirements. Ultrafine titanium has higher specific strength properties than ordinary titanium. Strength of ultrafine – grained titanium after drawing varies around 1030 to 1050 MPa, grain size around 100 to 300 nm.

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LITERATURE