Abstract
Titanium and its alloys are highly successful materials for the fabrication of dental and orthopedic implants, on account of their favorable combination of properties such as low specific weight, high strength to weight ratio, low modulus of elasticity, very high corrosion resistance and excellent general biocompatibility. This paper describes manufacture of ultra-fine grain titanium, its structure and properties. Ultra-fine grain titanium has higher specific strength properties than ordinary (coarse-grained) titanium. Ultra-fine grain titanium was produced by the ECAP process.

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. 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. Titanium is at present preferred to stainless steels and cobalt alloys namely thanks to its excellent bio-compatibility. Together with high biocompatibility of Ti its resistance to corrosion evaluated by polarisation resistance varies around the value \( 10^3 \, \Omega \cdot m \). 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 [1]. That’s why it is appropriate to use for dental implants rather fine-grained Ti instead of coarse-grained Ti. Use of ultra-fine-grained concerns numerous fields including medicine. 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
Strength properties of ultrafine-grained titanium must have the following values: \( R_m > 1000 \, MPa, \ R_{0,2} > 850 \, MPa \). 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 \( (N\cdot m/g) \), for the alloy Ti6Al4V it varies around 200 \( (N\cdot m/g) \), and for \((n)Ti\) it is possible to predict the values \( R_m/\rho = 270 \, (N\cdot m/g) \). As a matter of interest it is possible to give the specific strength also for some other dental materials: steel AISI 316L \( R_m/\rho = 65 \, (N\cdot m/g) \), cobalt alloys \( R_m/\rho = 160 \, (N\cdot m/g) \). 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. 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 [2]. 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.

Titanium alloy (Grade 5-Eli). 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 [3].

Zircon Dioxide of zirconium (ZrO₂) 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 4.5 mm but several factors can make necessary the use of different width implants [4]. The dentist must evaluate properly the condition of the patient's jaw and the position of the missing tooth in the mouth and in relation to the adjacent teeth.

**Wide form implants (large diameter), Fig. 1a**

Back teeth 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 - 6.0 mm in diameter.

**Shorter implants, Fig. 1b**

If there is close proximity with a facial nerve, a shorter implant has to be used to avoid the risk of nerve damage. 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.

**Narrow form implants (small diameter), Fig. 1c**

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 3.5 mm.

The use of mini dental implants 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.
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-compability, resistance to corrosion, strength and other mechanical properties from the viewpoint of its application in dental implants [5]. 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 [6,7]. The specimens were deformed at room temperature with different initial strain rates. After testing, the deformed specimens in order to preserve the microstructure Fig. 2- 4.
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.

Tab. 1. Chemical analysis titanium Gr. 2, w. %

<table>
<thead>
<tr>
<th>N</th>
<th>O</th>
<th>C</th>
<th>Fe</th>
<th>Al</th>
<th>Cr</th>
<th>Ti</th>
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<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>
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Tab. 2. Mechanical properties cp titanium after annealing 650 °C/ 1 h.

<table>
<thead>
<tr>
<th>Tensile strength [MPa]</th>
<th>Yield strength [MPa]</th>
<th>Elongation [%]</th>
<th>Reduction of area [%]</th>
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<tr>
<td>365</td>
<td>212</td>
<td>51</td>
<td>71</td>
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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. ECAP technology and drawing was made in variants:

- 8 passes ECAP at a temperatures of 280 °C; with annealing between individual passes.
- rotation re-forging to a diameter of 10 mm (cold forming: $\varepsilon = 2.2$).
- 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. 5. The structure was analysed apart from light microscopy also by the X-ray diffraction. Tab. 4 summarises the obtained basic mechanical properties.

![Microstructure of titanium after: a) initial structure, b) after equivalent strain 7.1 (8 passes ECAP)](image-url)
6. 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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REFERENCES


