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STRUCTURE AND MECHANICAL PROPERTIES ULTRA-FINE GRAINED TITANIUM

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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 it self 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: titanium, properties and structure

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 ρ 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 R/Ωm [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. 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 % HNO_3 and 80 % H_2O for 20 second. Chemical analysis and mechanical properties titanium are given in the Table 1-3.

Table 1. Chemical analysis commercially pure titanium (CP), (weight %)

N	O	C	Fe	Al	Cr	Ti
0,004	0,068	0,008	0,03	0,01	0,01	Rest.

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

Tensile strength	$R_{p0,2}$ Yield strength	Elongation 2“ gage	Reduction of area
[MPa]	[MPa]	[%]	[%]
365	212	51	71

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

Label	Diagonal of indention d [mm]	HV30
Sample 1	659	128
	632	139
	652	131
Sample 2	658	128
	655	130
	658	128
Sample 3.	527	200
	525	202
	535	194

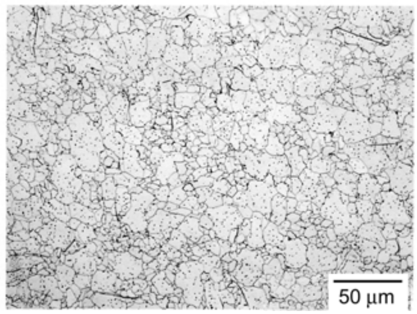


Fig. 1. Initial microstructure of titanium

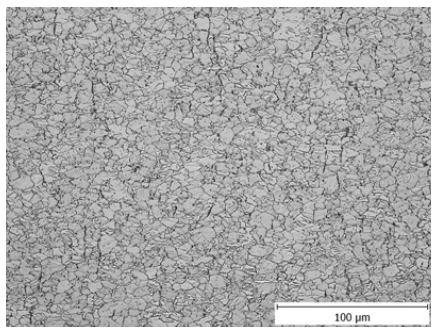
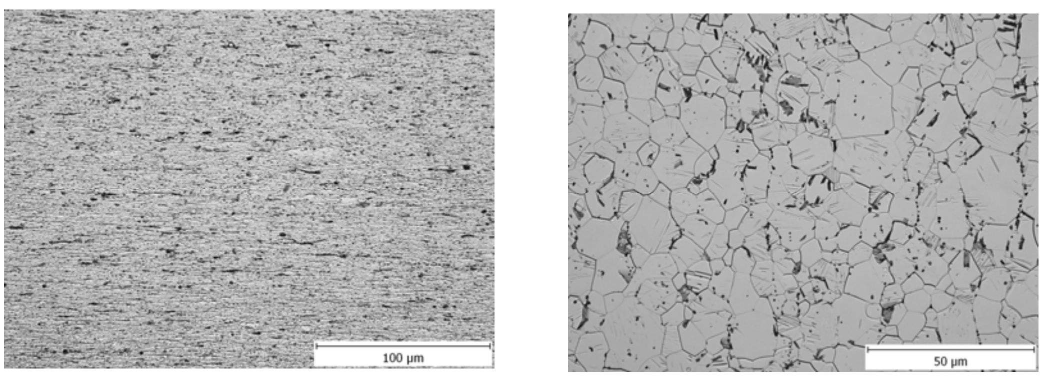


Fig. 2. Microstructure of titanium after cold rolling (deformation e = 46 %)



a) b)

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

3. PROPERTIES OF ULTRA-GRAINED TITANIUM

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: $R_m > 1000$ MPa, $R_{p0,2} > 850$ 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/ρ) 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 $\tilde{R}_m\rho = 270$ (N·m/g. As a matter of interest it is possible to give the specific strength also for some other dental materials: steel AISI 316L - $\tilde{R}_m\rho = 65$ (N·m/g), cobalt alloys $\tilde{R}_m\rho = 160$ (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.

4. 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. 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.

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 4 - 5 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 Figure 4, 5 and 6. The structure was analysed apart from light microscopy also by the X-ray diffraction. Table 4 summarises the obtained basic mechanical properties.

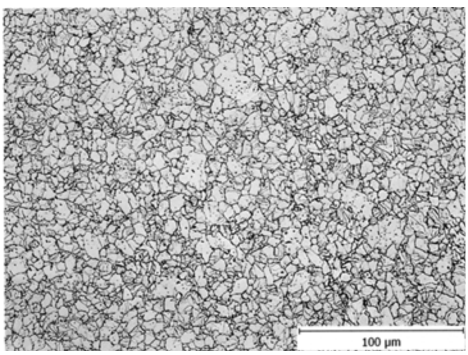


Figure 4. Microstructure of titanium after 2 passes ECAP

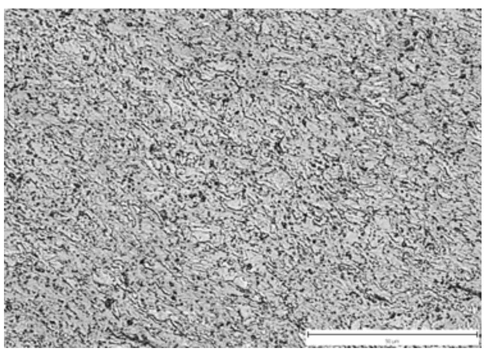


Figure 5. Microstructure of titanium after 4 passes ECAP

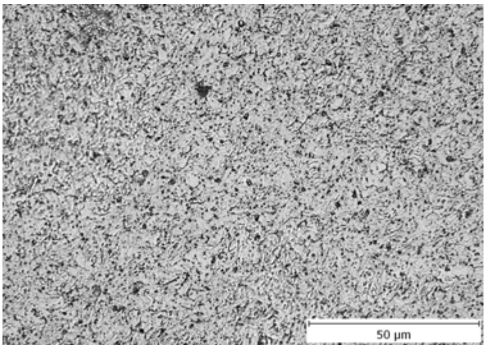


Figure 6. Microstructure of titanium after 4 passes ECAP

Table 4. Mechanical properties titanium after ECAP and drawing

Forming processed	R_m [MPa]	A [%]	E [GPa]	d_z [nm]
ECAP (8 passes)	960	12	100	100 to 300
Drawing (D_d = 6 mm)	1030 to 1050	9	100	100 to 300

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 varies around 1250 MPa, grain size around 300 nm.

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