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Determination of Mechanical Properties of Nanostructured Materials Used for Dental Implants

Stanca Comşa, Adrian Pacioga, Doina Gheorghiu
National Institute of Research & Development for Mechatronics and Measurement Technique, 6-8 Str. Pantelimon, Bucharest, Romania, stanca_comsa@yahoo.com

ABSTRACT

Load applied during the use of devices can result in component failure. Cracks can develop and propagate under tensile stresses, leading to failure. Friction/stiction and wear limit the lifetimes and compromise the performance and reliability of the devices involving relative motion. Stress and deformation analyses are carried out for an optimal design. Mechanical properties include elastic, inelastic (plastic, fracture, or viscoelastic), and strength. The strength property is needed to predict the allowable operating limit. Some of the properties of interest are hardness, elastic modulus, bending strength (fracture stress), fracture toughness, and fatigue strength. Micro/nanostructures have some surface topography and local scratches dependent upon the manufacturing process. Surface roughness and local scratches may compromise the reliability of the devices and their effect needs to be studied.

INTRODUCTION

Nanostructured materials reportedly exhibit unique microstructures and enhanced mechanical performance. As a result, they have attracted considerable attention in recent years and offer interesting possibilities related to many structural applications.

Although nanostructured materials are traditionally defined as those with grain sizes that are smaller than 100nm, grains in excess of 100 nm are typically present in the microstructure primarily as a result of the broad distribution of grain sizes that evolves during processing. Therefore, we define nanostructured materials as those with grain sizes smaller than 200 nm and with an average grain size of less than 100 nm.

Carbon is able to form linear bonds (sp^1), trigonal (sp^2) and tetrahedrite (sp^3). Diamond, graphite, nanotubes and diamond-like carbon (DLC = Diamond-Like Carbon) is in the range of natural and synthetic carbon.

Until the 1980s, the only known forms of carbon were allotropic diamond and graphite (Figure 1).

Last two decades have shown a variety of forms, depending on the dimensionality repetitive unit:

(i) zero-dimensional, with targeted trigonal bonds sp2, with (quasi) spherical form, held together by Van Der Waals cohesive forces, this class includes fullerenes, named after the great English architect Buckminster Fuller, with C60 , C70, C76, C78, C84, C86, etc., isolated experimentally. This also includes structures derived from fullerenes by functionalisation.

(ii) uni-dimensional, with sp^2-trigonal bonds, endless tubular forms (with polyhexagonal covering) or finite (tubulenes, with caps eventually semi- fullerenes), grouped by cohesive Van Der Waals forces.

(iii) two-dimensional, with sp^2-trigonal bonds, graphite sheets with hexagonal cover, attached by Van Der Waals forces.

(iv) three-dimensional, infinite, with sp^3-tetrahedrite bonds, diamond network, held together by covalence.

(v) three-dimensional, finite or infinite, with sp^2-trigonal bonds of carbon foam networks, held together also by covalence.

These forms are framed within the nano scale (10^{-9}m) and can reach to the micro scale (10^{-6} m).

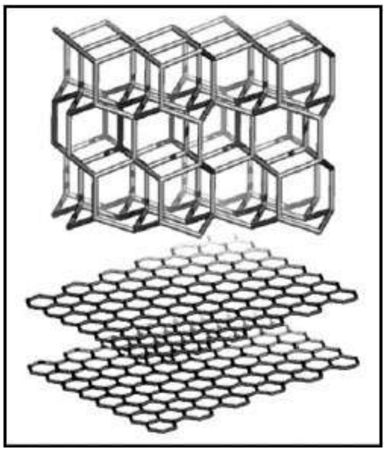


Figure 1 Graphite and Diamond nets

In order to characterize the mechanical properties of nanostructured materials obtained by DLC deposition, for the samples preparation was used pure titanium material, which is one of the more used materials for manufacture of dental implants. Materials used in this area must be biocompatible, not toxic and must not cause allergenic reactions. They also must have the high tensile strength (Rm) and proof strength (Rp), and low density (ρ) and modulus of elasticity (E).

To study the mechanical proprieties of coated materials comparative with not coated ones, three specimens of titanium probes were made.

Test Specimens

The test specimens were obtained from laminated pure titanium rods with 100mm length. On 2 of them a diamond-like carbon coatings was deposited using thermionic vacuum arc method (TVA)

Deposition by thermionic vacuum arc method (TVA) is made by evaporating the coating material by an indirectly heated cathode surrounded by a Wehnelt cylinder. The evaporation material state is obtained by heating it with thermal electrons generated by the circular shape filament and indirectly heated and situated above the anode. The used anode was a carbon rod with a 6mm diameter.

In figure 2 is presented the experimental assembly of TVA system. The simetric anod-cathode arrangement permits an perpendicular electrons bombardment on anode surface.

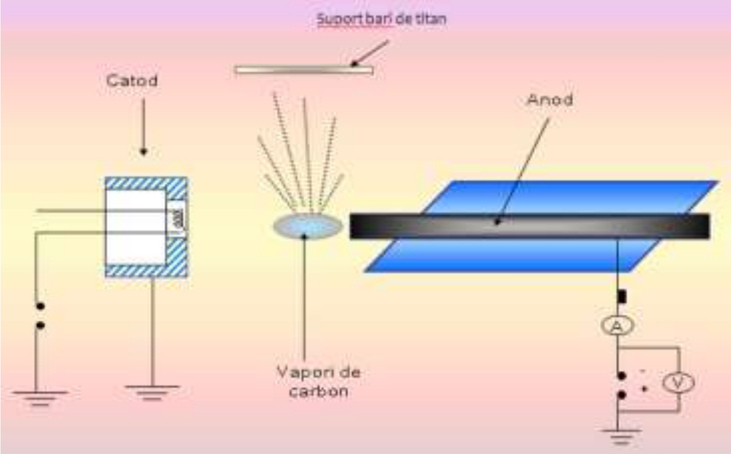


Figure 2 Experimental assembly of TVA system

Due to large energy dissipated in the plasma volume, the deposited material is completely dispersed without drops. The obtained film is very fine and under certain conditions appears as a nanostructures deposition.

After a 20nm layer deposition the process was stopped and the titanium rod was rotated with 180°. After rotation the process was restarted and another 20nm layer was deposited.

Mechanical proprieties evaluation

For determination of mechanical resistance the specimens were tested at bending and tensile. The tests were made on a static test machine type Hounsfield H10KT.

BENDING TESTS

The bending test procedure was realized according to the methods described in the following standards: STAS 1660:1980 Tests of metals. Bend test of iron; SR EN ISO 178:2003 Plastic materials. Bending properties determination.

For the bending test the specimens were mounted on the testing machine and the load was applied until the test preset extension (f=2,5mm) as shown in figure 3.

Figure 3 – Samples during bending test

Initial specimen parameters:

• Initial diameter: d_0	d_{01}=2,97mm	d_{02}=2,95mm	d_{03}=2,94mm
• Original gauge length : L	100 mm		
• Spam distance: l	90 mm		

Initial setting test parameters:

• diameter of cylindrical bearing: D	6 mm
• radius of loading piece : R	3 mm
• Cross head speed: v_D	20 mm/min
• Maximum load range: F_{max}	1000 N
• Maximum extension range: f_{max}	3 mm
• Preset extension: f	2,5 mm

Test results:

• Maximum force: F_{max}	F_{max1}=79,9N	F_{max2}=78,7N	F_{max3}=77,7N
• Original gauge length : L	100 mm		
• Spam distance: l	90 mm		
• Flexural moment: $M_i = \frac{F_{\max} l}{4}$	M_{i1}=1797Nmm	M_{i2}=1771Nmm	M_{i2}=1748Nmm
• Bending strength modulus: $W_{ef}=\pi d^3/32$	W_{ef1}=2,57mm^3	W_{ef2}=2,52mm^3	W_{ef3}=2,49mm^3
• Bending strength $\sigma_i=M_i/W_{ef}$	σ_{i1}=699,22MPa	σ_{i2}=702,77MPa	σ_{i3}=702,01MPa

Bending test graph: $F = F(f)$ is shown in figure 4 for the uncovered specimen and in figures 5 and 6 for the carbon coated probes.

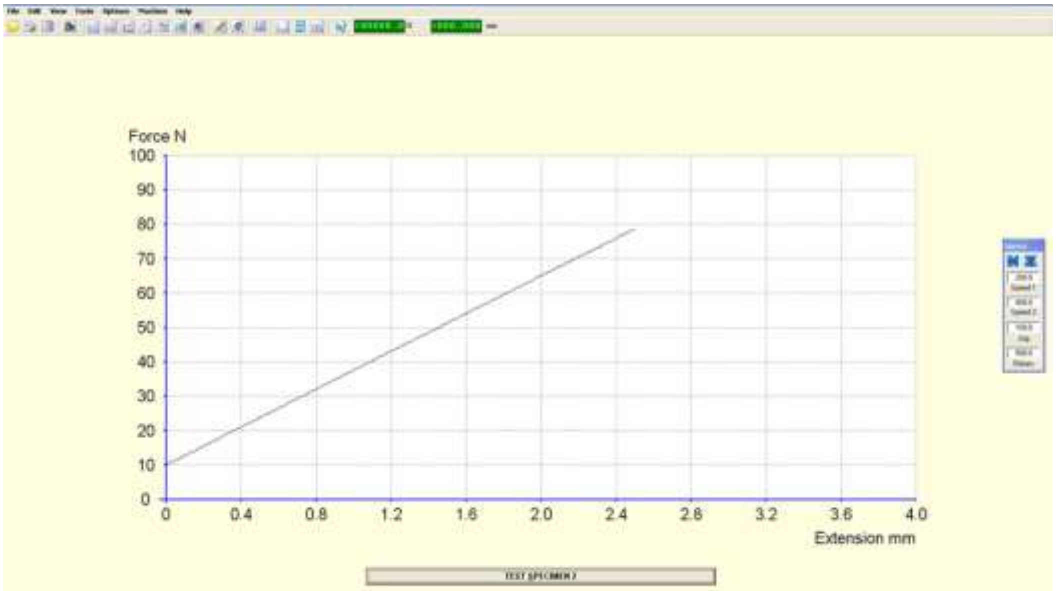


Figure 4 – Bending test graph for uncovered sample

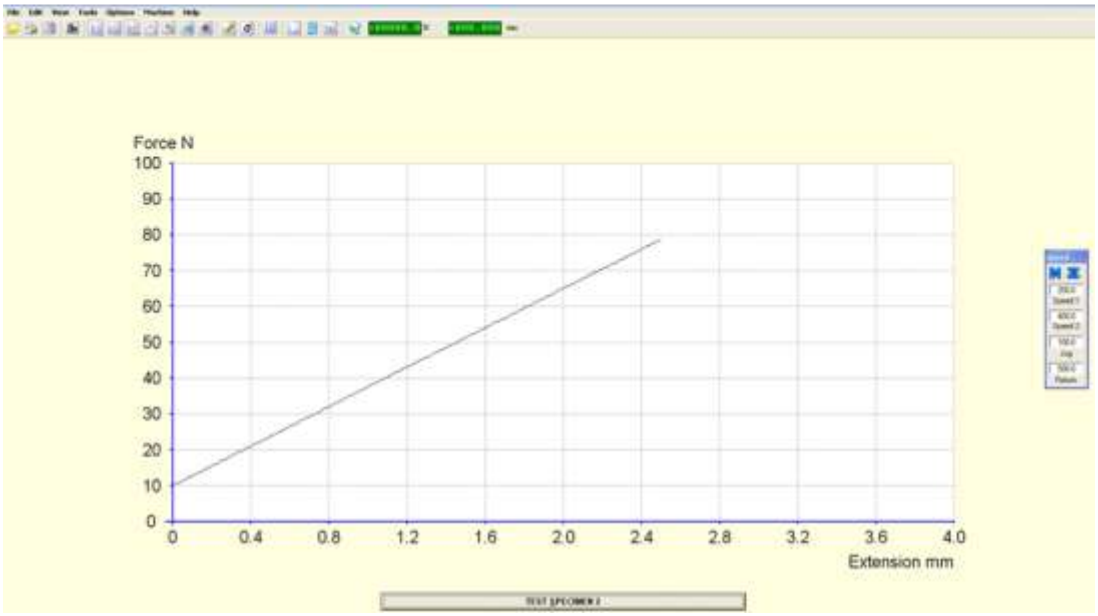


Figure 5- Bending test graph for first covered sample

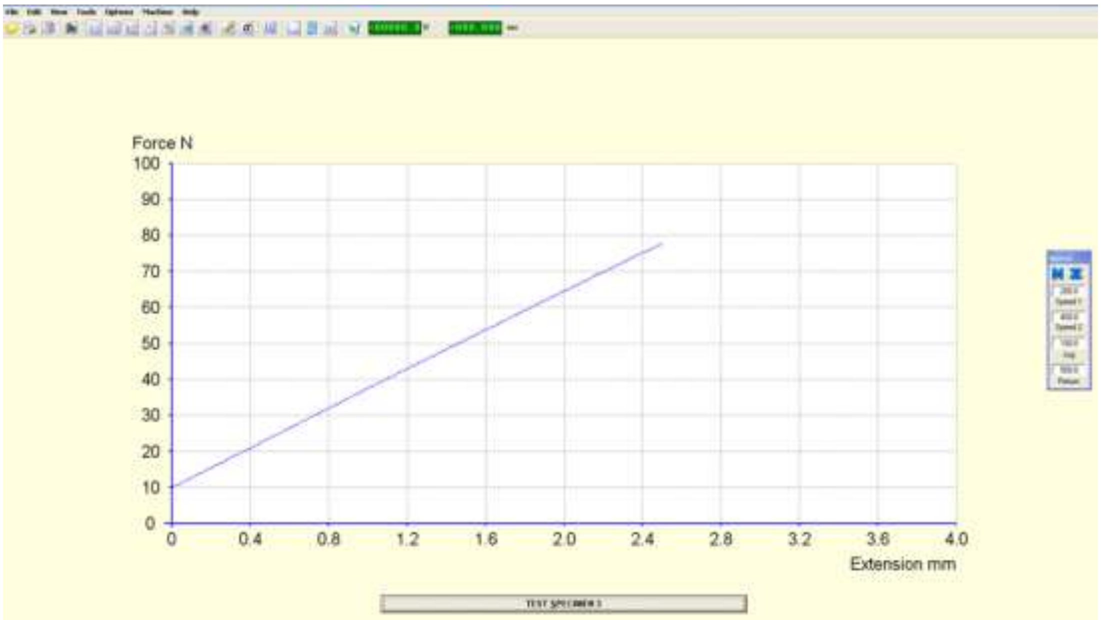


Figure 6-Bending test graph for second covered sample

2. TENSILE TESTS

The tensile test procedure is realized according to the methods described in the standard: SR EN 10002-1:2002 Metallic materials. Tensile testing- Part 1: Method of test at ambient temperature.

For the tensile test the specimens were mounted on the testing machine and the load was applied until break as shown in figure 7.

Figure 7 – Specimen in tensile test

Initial specimen parameters:

• Initial diameter: d_0	d_{01}=2,97mm	d_{02}=2,95mm	d_{03}=2,94mm
• Original cross-sectional area of the parallel length: S_0	S_{01}=6,92 mm^2	S_{02}=6,83 mm^2	S_{03}=6,78 mm^2
• Extensometer gauge length : L_e	L_e=50mm		

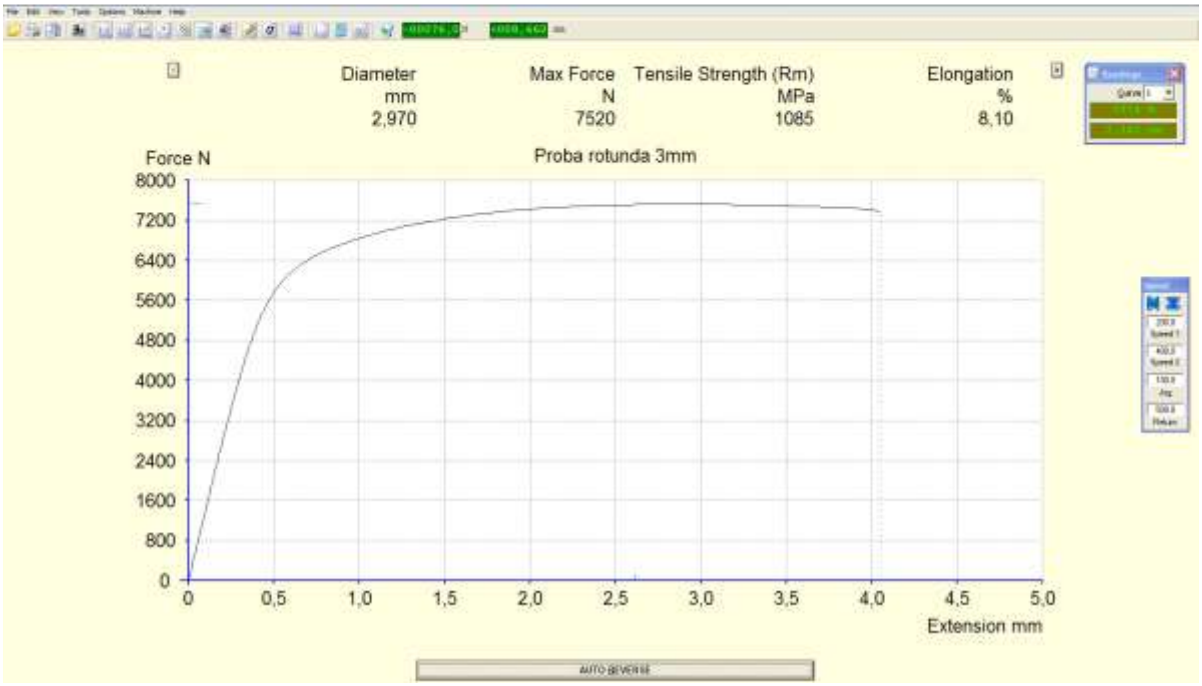
Initial setting test parameters:

• Cross head speed: v_D	20 mm/min
• Maximum load range:	9000 N
• Maximum extension range:	3 mm

Test results:

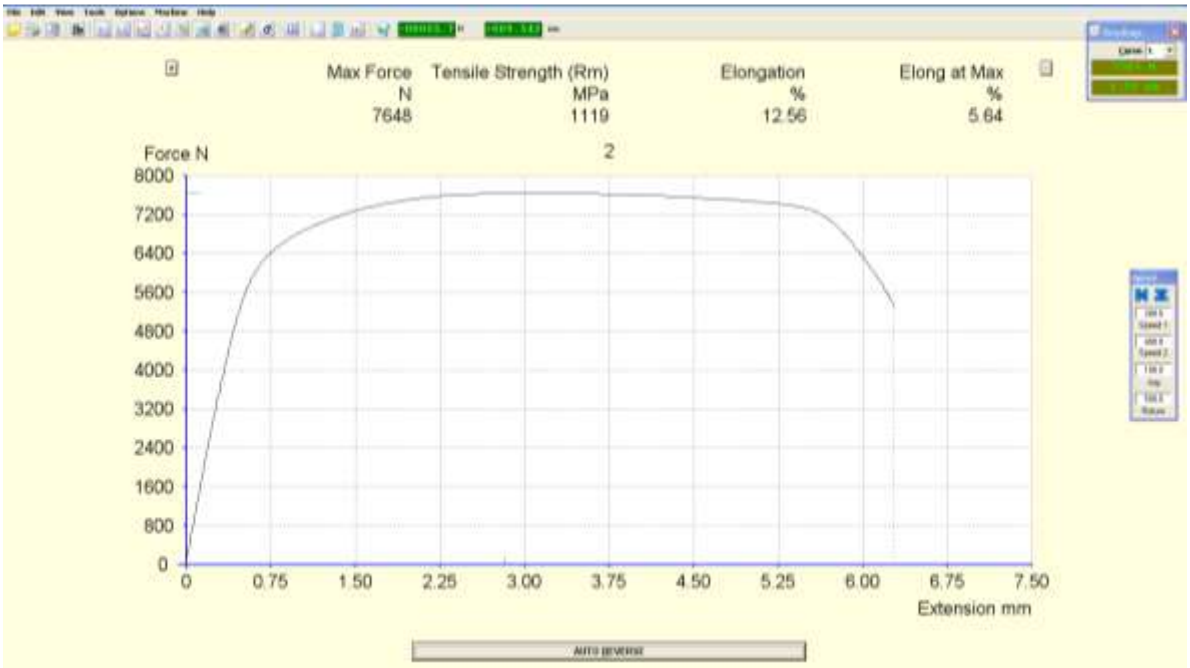
• Final diameter: d_u	d_{u1}=2,2mm	d_{u2}=2,15mm	d_{u3}=2,16mm
• Final gauge length after fracture: L_u	L_{u1}= 54,05mm	L_{u2}=56,28mm	L_{u3}=55,18mm
• Minimum cross-sectional area after fracture: S_u	S_{u1}= 3,79mm^2	S_{u2}=3,63mm^2	S_{u3}=3,66mm^2
• Elongation after fracture: ΔL_c	ΔL_{c1}= 4,05mm	ΔL_{c2}=6,28mm	ΔL_{c3}=5,18mm
• Percentage elongation after fracture: $A=\left(\frac{L_u-L_0}{L_0}\right)\cdot 100$	A_1=8,1%	A_2=12,56%	A_3=10,37%
• Maximum force: F_m	F_{m1}=7520 N	F_{m2}=7648 N	F_{m3}=7200 N
• Percentage total elongation under the maximum force: A_{gt}	A_{gt1}= 5,23%	A_{gt2}=5,64%	A_{gt3}=5,22%
• Percentage reduction of area : $Z=\left(\frac{S_0-S_u}{S_0}\right)\cdot 100$	Z_1=45,2%	Z_2=46,8%	Z_3=46%
• Tensile strength: R_m	R_{m1}=1085MPa	R_{m2}=1119MPa	R_{m3}=1053MPa

Tensile test graph: $F = f(\Delta l)$ - is presented in figures 8-10



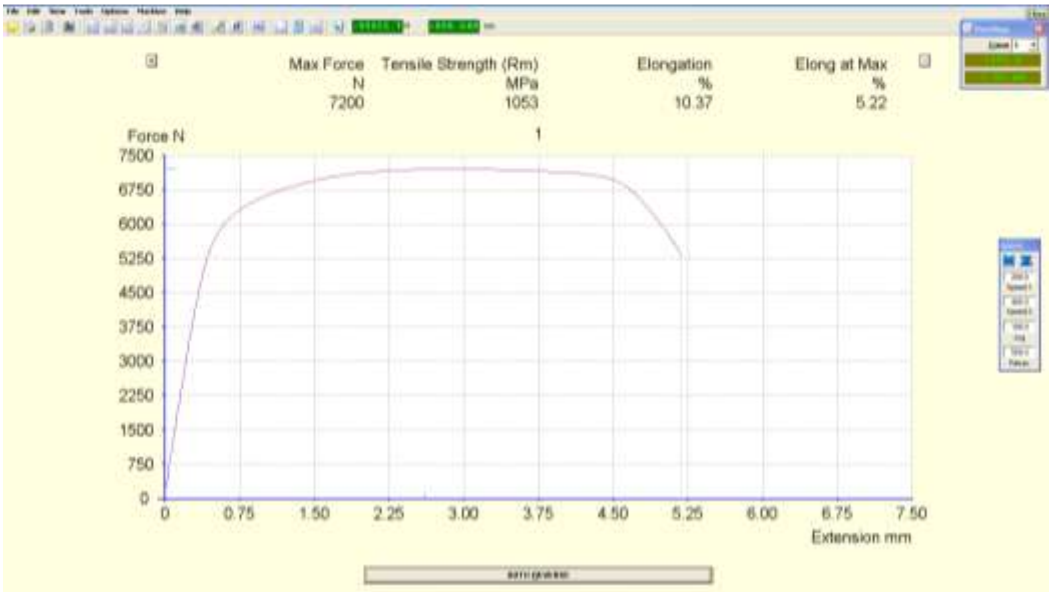
Diameter mm	Max Force N	Tensile Strength (Rm) MPa	Elongation %
2,970	7520	1085	8,10

Figure 8 – Tensile test graph on uncoated sample



Max Force N	Tensile Strength (Rm) MPa	Elongation %	Elong at Max %
7648	1119	12.56	5.64

Figure 9 – Tensile test graph on first coated sample

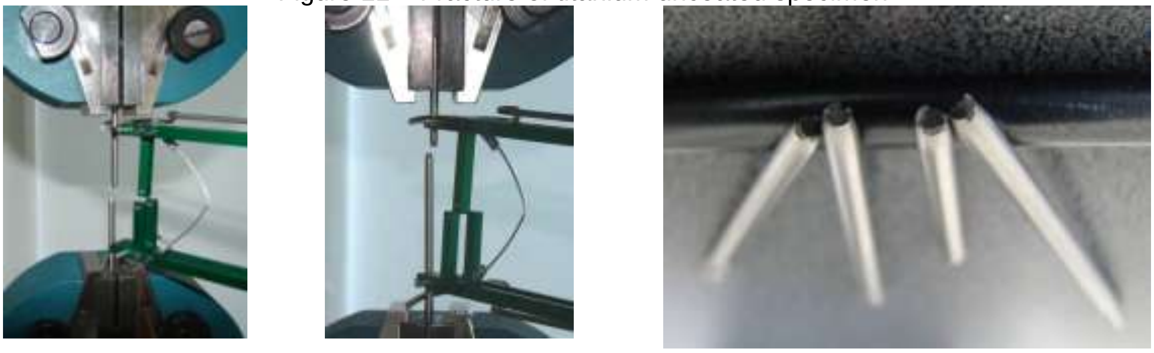


Max Force N	Tensile Strength (Rm) MPa	Elongation %	Elong at Max %
7200	1053	10.37	5.22

Figure 10 – Tensile test graph on second coated sample

The fracture of the samples is presented in figures 11 and 12.

Figure 11 – Fracture of titanium uncoated specimen



a) b) c)

Figure 12 – Fracture of titanium carbon coated specimen
a) Specimen 1 of titanium carbon coated; b) Specimen 2 of titanium carbon coated;
c) Fracture zones of specimen 1 and 2

CONCLUSION

Most mechanical properties are size-dependent. The use of osseointegrated dental implants is a predictable and successful treatment method for functional restoration of the fully or partially edentulous patient. A satisfactory clinical outcome relies on primary stability for load bearing immediately following implantation. This requires osseointegration within a short healing time. One of the most important surface properties is the topography of the surface. Topographical modifications may enhance short- and long term osseointegration.

Different treatment methods aiming a nanostructural topographical respective chemical change can promote stronger and faster bone in-growth and healing.

Mechanical properties of the obtained materials were evaluated by bending and tensile tests using a universal testing machine Hounsfield H10KT, by means of two dedicated software: QMAT XT and QMAT Professional.

The tests were performed using a 20 mm/min crosshead speed and a load cell of 1000N.

Looking at the bending test results, we can see that despite the decrease of the diameters of the specimens, the bending strength slightly increases which means that the dental implants made from titanium with carbon coatings will resist better than uncoated ones.

The maximum force corresponding to the preset extension of 2,5mm decreased at the coated specimens suggesting the increase of the elastic properties. The deformations of the specimens were in the elastic domain and the slopes of the characteristic curve are similar for all test pieces.

The elastic comportment can also be observed in the tensile tests. The increase of the elasticity is shown in the tests results and is given by the ultimate diameter and minimum cross-sectional area after fracture reduction and by the increase of the final gauge length and percentage elongation after fracture.

The coated samples had different values for the percentage elongation after fracture which can be assigned to the method used for preparing the carbon coatings. The method had two stages: in the first step a 20nm layer was deposited then the process was stopped and the titanium rod was rotated with 180°. After rotation the process was restarted and another 20nm layer was deposited. This dividing in two stages of the process could induce non-uniformities of the deposited layer and to produce variation of the elastic properties.

The aim of dental implant is to achieve at least the same percentage of elongation under the same stress in an implant-bone-combination. The increase of the implant elasticity in order to be similar to that of the bone is the most promising way to achieve the desired adapted implant elasticity. Due to the mentioned above considerations the obtained increased elasticity of the specimens is a favourable effect. This can lead to the increase of the implant's life-time.

The deposited carbon coating on the specimens does not seem to have great influence upon the tensile strength because the variation is less than 3%.

The fracture aspect is similar to all probes.

The discovery of novel biomaterials and the refinement of traditional ones is creating a thoroughly unprecedented excitement in the field as nanomaterials designers increasingly confront many of the fundamental challenges of medical science.

The future researches can be orientated to improve the surface depositing process in order to obtain a better homogeneity of the specimen and repeatability of the results.

In order to be able to adequately examine the dental implant materials, *in vitro* biodegradation studies (enzymatic / non-enzymatic bulk hydrolysis) followed by mechanical testing must be carried on after incubation in various physiological media.

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