Study of the Titanium Alloys Surfaces Used in Orthopaedic Systems

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***Abstract* — Total hip prosthesis (THP) is a highly successful orthopaedic device. However, its durability is generally limited to a few decades due to difficult conditions in the human body and huge demands it is subjected to. A hip prosthesis is deteriorating due to high surface pressures caused by mechanical movements of the body.**

The aim of this project is to improve the characteristics of hip prostheses, in order to increase their functionality and their life span. This paper presents an analysis regarding the topography and tribological parameters of femoral heads structures and of femoral heads coated with TiN. We studied the tribological properties of the surfaces of some femoral heads made of Ti alloys or coated with TiN. These femoral heads were obtained from some prostheses after revision surgery. Afterwards, we used TiN nanostructured coatings for reducing the wear process. TiN thin films were deposited using physical vapour deposition (PVD) and some scratch tests have been realized on these coatings surfaces. The study of coatings surfaces was made using atomic force microscopy (AFM) that offers the possibility to obtain nanometric 3D control of thin films.

Main result of these researches is that used coatings offer the possibility to improve the system properties.

***Index Terms* — atomic force microscopy, coatings, titanium alloy, total hip prosthesis, wear.**

I. INTRODUCTION

The hip is a ball and socket joint, formed by the articulation of the rounded head of the femur and the cup-like acetabulum of the pelvis. It forms the primary connection between the bones of the lower limb and the axial skeleton of the trunk and pelvis. The head of the femur attaches directly to the acetabulum and by a thin neck region to the shaft.

A hip joint can realize six different kinds of movements: flexion and extension (on or from the spine and on or from the thigh), abduction and adduction of the femur, internal (medial) and external (lateral) rotation of the pelvis, thigh or spine.

Normal function of hip joints may be disturbed due to illness and under the negative influence of other factors determined by the mode of human life and professional activity. Surgical problems (e.g., problematic orientation or problems in wound healing), host abnormalities or diseases, infection, material fracture, wear, and corrosion are the most common failures of hip prostheses. For this reason damaged bones are replaced by total hip prosthesis (THP) consisting of (Fig. 1):

- a cup that replaces the hip socket;
- metallic encasing;
- a ball that replaces the fractured head of the femur;
- a stem that is attached to the shaft of the bone to add stability to the prostheses.

Total hip prosthesis is also a highly requested bio-tribo-system, on which many mechanical, thermal, chemical and biological factors act.

Durability and stability of this prosthesis depends on mechanical stress, movements and heat [1] that support artificial hip joint, bone-cement and cement-stem interfaces strength for cemented stems [2]. It also depends on the growth of bone inside for uncemented stems [3], and on the resistance to wear of femoral stems, acetabular cups and femoral heads [4]. High superficial pressures produced by mechanical movements of the body have an important influence on the articular surfaces deterioration.

Taking into account the hip prostheses components, there can be acetabular cup – ball wear and acetabular cup – encasing wear.

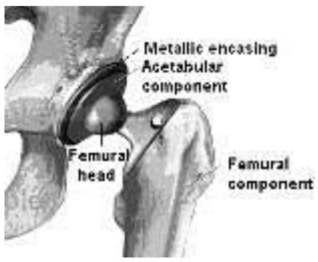


Fig. 1. Components of a total hip prosthesis.

These deterioration processes have a complex mechanism, combining abrasive wear, adhesive wear, third body wear and fatigue wear [5]. Abrasive wear [6] represents the removal of material from one surface by the other. Adhesive wear is produced where localized bonding of the two surfaces occurs, such that the attachment force is stronger than the yield strength of the material. A small piece of material is removed from one surface and is attached to the other. Third body wear refers to the insertion of a wear particle between two moving surfaces [7]. Fatigue wear can lead to subsurface cracks propagating and flaking off of particles from the surface. High subsurface stresses can also be caused by third bodies between the two articulating surfaces leading to accelerated fatigue wear.

Composition of hip prostheses (e.g. ceramic, metals) and materials properties have a great influence on the period of time that these systems are used. Stainless steel, titanium alloys, polymers and ceramic composites undergo degradation after 10–15 years of use.

Metallic materials are resistant to breakage, even if they are relatively soft. Generally, pure metals are rarely toxic. Metallic biomaterials have different influences on the human body, is distinguishing different forms of biological reactions, according to: concentration of metal, exposure time and route of administration. Two compounds of the same metal can induce strong, but different responses.

Titanium and titanium alloys are commonly used for THP due to their high mechanical strength, good corrosion resistance and biocompatibility. Ti6Al4V is a compound with thermo-mechanical processed microstructure to create the desired amount of two phases with fine particles for optimum mechanical properties. In particular, Ti6Al4V alloy has high resistance to stress and fatigue, an excellent corrosion resistance, a very high biocompatibility and high strength relative to weight. For these reasons it is an alloy widely used for advanced biomedical applications.

However, the compounds have poor tribological characteristics, especially in abrasive and sliding conditions. High friction coefficient and low wear resistance occurs frequently when Ti6Al4V alloy is sliding against other engineering materials.

In order to improve mechanical properties of hip prostheses, these have been coated with different materials, which have superior properties.

Taking into account the problems regarding THP's deterioration presented before, we are trying to improve their tribological properties using nanostructured coatings.

II. MATERIALS

We analysed a series of total hip prostheses with modular femoral heads of Ti6Al4V alloy and PVD coated with TiN. These were recovered following revision surgery. All of the studied prostheses had acetabular cups made of ultra-high molecular weight polyethylene (UHMWPE).

Wear processes produce different changes in total hip prostheses that are impossible to see with the naked eye. A methodology of ascending degrees of resolution was established using macroscopic (resolution millimeters), microscopic (resolution microns) and nanoscale (resolution nanometers) measurements. Different methods of investigation were used during the years to establish the surface topography of total hip prostheses.

We realized topography of hip prostheses components by atomic force microscopy (AFM), because its images display high quality and dense nanocrystalline structure of the surfaces. An atomic force microscope (NTEGRA Probe Microscope – Fig. 2) working in the noncontact mode was used in our experiments. It is composed of a base unit where we have the sample, a special measuring head made of a cantilever with a pointed end, and an optical viewing system. AFM images are processed using Nova SPM software. In this way, tribological parameters that offer information about the uniformity of femoral heads surfaces were obtained, like:

- maximal and average height of surface;
- roughness of the studied surface describing its uniformity;
- ten point height (S_z) expressing surface roughness by the selected five maximal heights and hollows, nm;
- surface skewness (S_{sk}) characterizing the non-symmetry of distribution;
- coefficient of kurtosis (S_{ka}) characterizing the distribution spread.

Fig. 2. NTEGRA Probe NanoLaboratory: a) 1 – base unit; 2 – measuring head; 3 – vibration isolation system; 4 – optical viewing system.

In order to improve resistance of femoral heads we made studies about deposition of TiN thin films on stainless steel disks by physical laser deposition (PLD). We used TiN for coatings due to the fact that it is a hard biocompatible material with excellent resistance to abrasion.

PLD experiments were realized using a KrF* laser (λ = 248 nm, $\tau_{FWHM} \approx 25$ ns, ν = 10 Hz), into a deposition chamber, with stainless steel reaction chamber to 5000, 10000 and 20000 pulses. Disks samples (22.5 mm in diameter and 10 mm in height), made of 316L stainless steel were used as substrates.

TiN thin layers obtained after PLD have been also characterized using NTEGRA Probe Microscope, working in the nonocontact mode.

Adhesion resistance of deposited TiN layers was evaluated by scratch testing with the system presented in Fig. 3. This system has a diamond spherical tip and the movement is pure sliding. The tested body is the 316L stainless steel disks coated with TiN. The applied loads were in the range of (2.5 – 125) N. Each scratch was analysed at the end by optical microscopy (OM) and AFM.

Fig. 3. General view of scratch tester.

III. RESULTS

Numerous scratches and some local rubbing were observed on the surfaces of hip prostheses extracted from the human body. Such scratching may be attributed to wear resulting in loss of material or plastic deformation of the surface without loss of material. These are possibly caused by a hard particle embedded in the cup.

Microscopic studies of these surfaces revealed that femoral heads and acetabular cups suffered damages by scratching, cracking, peeling, tribocorrosion and material transfer.

We used AFM for a more clearly identification of the damages nature and determination of topographic parameters of the investigated femoral heads surfaces. Various irregularities more or less pronounced can be observed, like particles ripped from the material of the femoral head or scratching. There are also some areas with uniform surfaces.

The mean value of the maximum height (Fig. 4) of the damages from the femoral heads varies depending on the material, in the present case Ti6Al4V (341.19 nm) > TiN (198.79 nm).

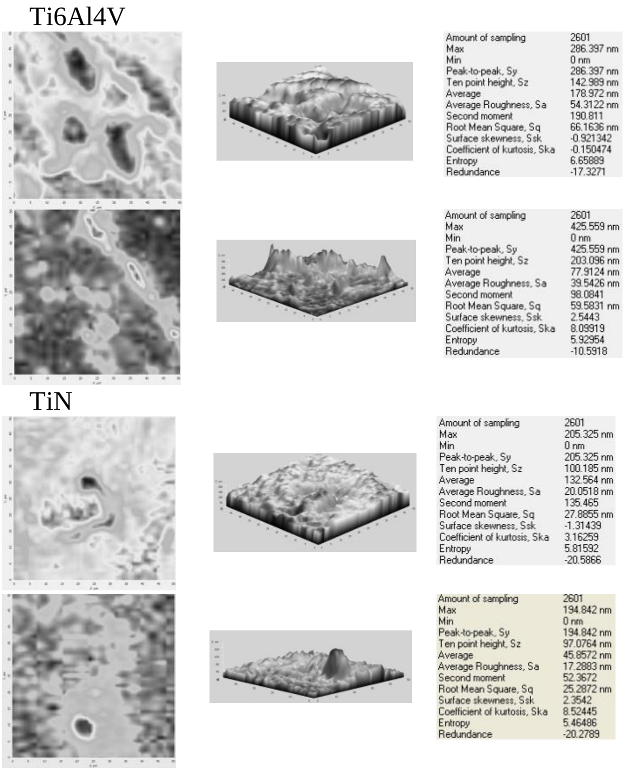


Fig. 4. AFM characterization of femoral heads made up of Ti6Al4V and TiN

The roughness values (Fig. 4) also varied in a wide range, in different parts of the same femoral head, depending on the movements of the body that uses the prostheses. The roughness value of the surfaces also varied depending on the material making up the studied femoral head. This proves the existence of more or less pronounced irregularities, which could have emerged from the wearing process. Largely uniform surfaces were observed, but several pronounced holes left after pulling away some particles appeared. A decrease of the roughness value depending on the material, thus: Ti6Al4V (50.7 nm) > TiN (26.5 nm) was observed from a brief analysis of the obtained mean values.

The mean values of the maximum height and roughness obtained prove the strength of material, wear producing to a much lower depth and a lower alteration of the uniformity.

After the study of used prostheses, TiN thin layers deposited with 5000, 10000 and 20000 pulses have been characterized using AFM. The average thickness of deposited layers was determined by different methods:

- by optical microscopy (OM): 1,1 μm for 5000 pulses and 1,62 μm for 20000 pulses;
- by AFM: 1,67 μm for 5000 pulses, 2,11 μm for 10000 pulses and 2,72 μm for 20000 pulses;
- by SEM: 0,8 – 1 μm for 5000 pulses, 1,2 – 1,4 μm for 10000 pulses and 1,5 – 1,6 μm for 20000 pulses.

It can be observed that the thickness of the layer increases with the number of laser pulses. Considering that the surface of the substrate has shown some minor defects, an important conclusion is that the layer becomes more uniform along with a more complete coating of initial defects.

The values of layers roughness (Fig. 5) vary depending on the number of pulses the layer has been deposited to (40.013 nm for the sample with 5000 pulses, 26.334 nm for the sample with 10000 pulses, 2.527 nm for the sample with 20000 pulses). A decrease of roughness value with increasing number of pulses was observed.

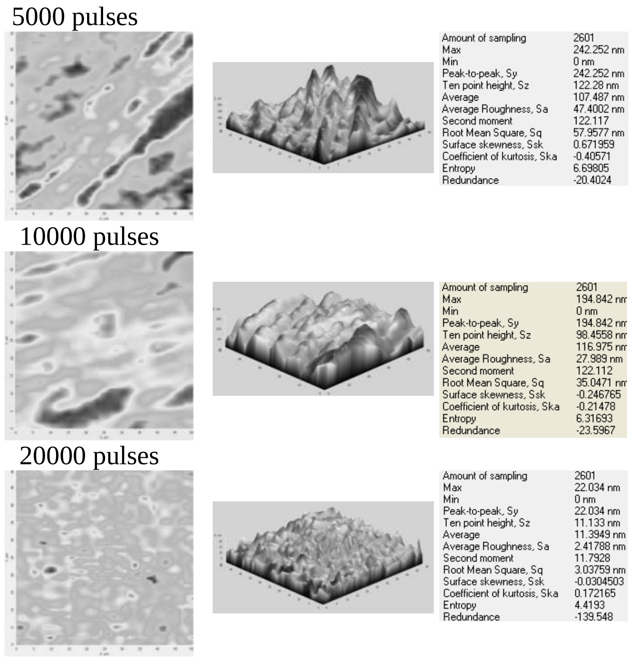


Fig. 5. AFM characterization of TiN layers deposited at 5000, 10000 and 20000 pulses.

TiN layer deposited at 5000 pulses has a surface with lower uniformity, which could be the result of more pronounced irregularity of the substrate. The surface of TiN layer deposited at 10000 pulses has a higher uniformity than that of the layer deposited at 5000 pulses. It can be seen from the images presented small surface defects, but their size is reduced. The surface of the TiN layer deposited at 20000 pulses has the highest uniformity of the all 3 types of realized samples. It is the lowest mean roughness value of these samples, as demonstrated by the uniformity of surface coating, visible from AFM scanning.

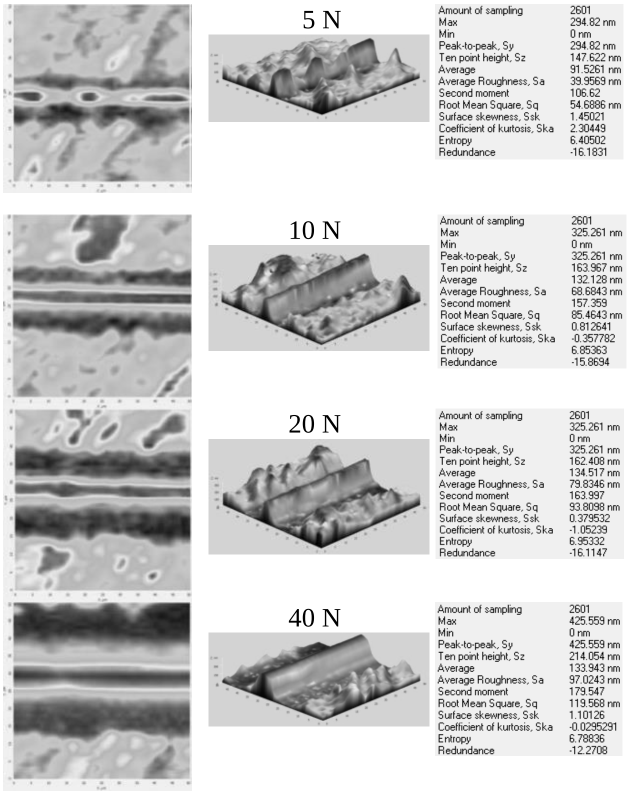


Fig. 6. AFM characterization of TiN layers deposited at 5000 pulses after the scratch test.

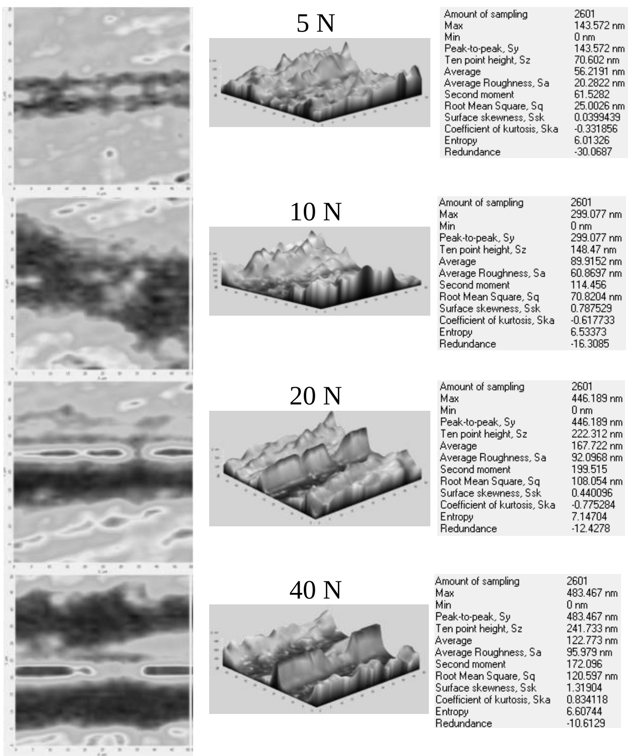


Fig. 7. AFM characterization of TiN layers deposited at 10000 pulses after the scratch test.

Surfaces of TiN layers have been subjected to scratch tests in order to analyse their adhesion to the substrate.

The applied loadings, between 2.5 N and 125 N created scratches with different depth and width. Surfaces obtained after the scratch tests have been characterized using AFM in order to determine different tribological parameters (Fig. 6, Fig. 7, Fig. 8).

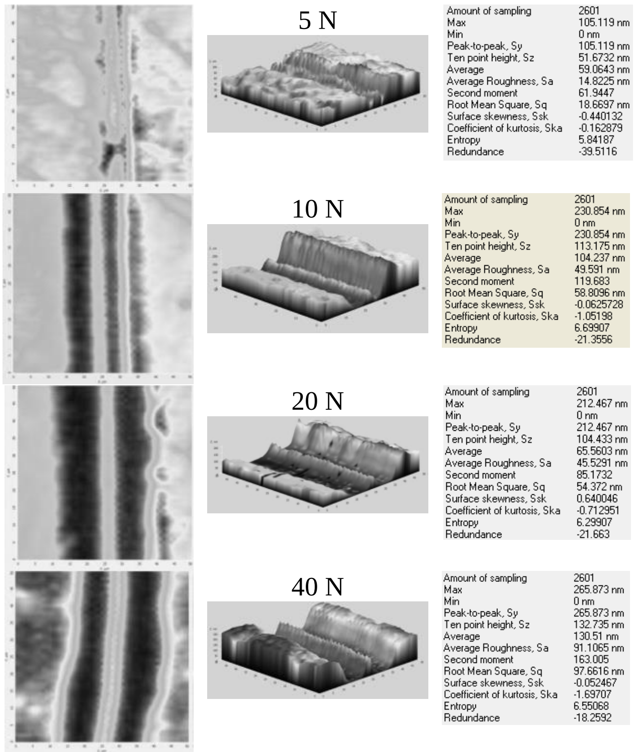


Fig. 8. AFM characterization of TiN layers deposited at 20000 pulses after the scratch test.

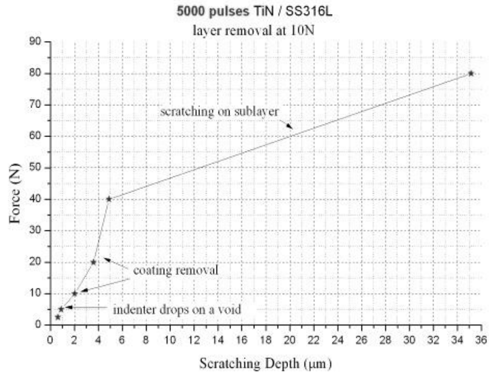
Results showed that the roughness values of the TiN layers surfaces varied depending on the applied loads. These values increased with the increase of the applied load. Lower values of the roughness compared with the values of the uncoated surfaces were determined. In this way it was demonstrated that TiN layers have a more uniform surface even after the scratch tests. This can be a prove for a higher resistance of TiN layers.

Width and depth of the scratches were measured by OM and AFM. Graphic variation curves of scratches depths depending on the applied normal load were drawn.

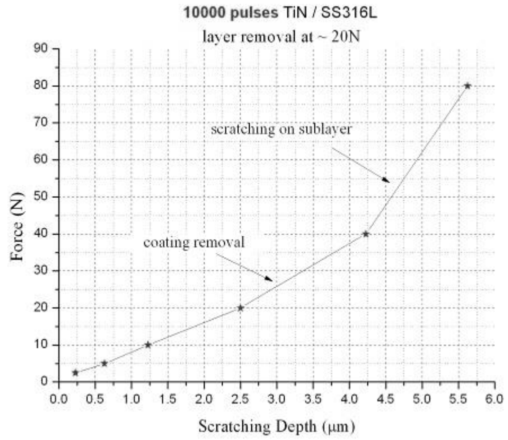
On the OM images were measured in ten places scratch traces widths, then were calculated their depths *h* based on a simplified relationship (indent method), which takes into account the radius of diamond tip (*r*) and the means values of measured widths of the indent (l):

$$h = l / 8r. \quad (1)$$

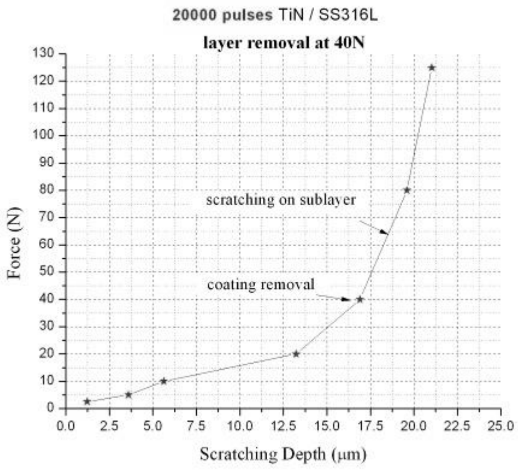
Variation curves of scratching depth function of applied normal load (Fig. 9) show some "indenter drops" due to indentation on voids or other defects of coatings.



(a)



(b)



(c)

Fig. 9. Variation of scratching depth (μm) function of normal load applied over the indenter (N), on SS316L samples, coated with TiN by PLD, (a) at 5000 pulses, (b) 10000 pulses and (c) 20000 pulses, respectively.

IV. CONCLUSION

Atomic force microscopy was a useful technique to characterize the topography of femoral heads surfaces made of titanium alloys. Taking into account the results obtained after these characterization we deposited TiN layers on 316L stainless surfaces in order to improve the characteristics of total hip prostheses.

Following the performed scratch tests and AFM measurements, it was observed that the femoral heads uncovered surfaces show roughness values higher than those of the femoral heads surfaces covered with TiN thin films.

The main conclusion of this study is that TiN nanostructured protective coatings deposited by PLD technique offer the possibility to improve the properties of hip prostheses. Another important thing is that deposition of thin films by PLD with 20000 pulses can be an alternative technology to ensure scratch resistance of femoral heads.

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