EVALUATION OF THE APPLICATIVE-TRIBOLOGICAL CAPACITY OF THIN FILMS USED TO INCREASE THE LIFETIME OF HIP PROSTHESES COMPONENTS

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Abstract - The assessment of the applicability of micro-nanostructured thin films intended for biomedical applications and finding a layer of biocompatible material with a specific thickness useful for improving the tribological properties of hip prostheses was the main objective of the experiments. Thin films of TiN, TiO_2 and CrN were deposited on femoral heads of CoCr and M30NW steel, through physical processes – cathodic arc, DC sputtering and HiPIMS sputtering - being obtained layers with thicknesses of 0.5 µm, 1 µm and 1.5 µm. Following the nanoindentation and tribological characterization tests, the hardness and wear rate of the thin films were determined and the degree of influence of each deposition method or of the material used was analysed. Taking into account the values of the obtained parameters, the conclusion is that the TiN and CrN thin films deposited by DC sputtering had better wear resistance and the best tribological characteristics compared to TiO_2, the first one being further tested on real biomedical components to evaluate their applicability. To study the quality of the thin films, some wear tests simulating the movement in an environment similar to the real one and a new topographical analysis of the worn films were made. Following all these studies, TiN was determined to be the most suitable material in the form of a useful thin film for recuperative biomedicine. The protection offered by the thin films against wear was proven by the fact that, at the macroscopic level, traces of material were observed on some uncoated components, and at the microscopic level, a decrease in the surface roughness of the coated femoral head, in comparison to the uncoated femoral heads. From the experimental data obtained, a main conclusion was drawn, regarding the thin films thickness with the highest degree of applicability and wear resistance for biomedical applications.

Keywords: Thin films, Durability, Wear rate, Hip prostheses, Roughness, Tribological parameters.

1. Introduction

The human locomotor apparatus is made up of all the bone structures linked together by joints, and of muscles, encompassing three main systems: the bone system, the joint system and the muscle system. The basic function of this apparatus is locomotion, i.e. the movement of the individual in space, as well as the mobilization of the various body's segments, one in relation to the other [1].

Bipedal walking is the main form of locomotion necessary for human activities. This activity is characterized by complexity and dynamism, thus fulfilling important functions for the human body: unipodal stability, preventing falls, avoiding obstacles, moving the human body in space by moving the center of mass and adapting to avoid painful movements. The human gait cycle contains several stages, namely: one-legged support – right lower limb, left lower limb swing, left lower limb partial step length, right lower limb swing, one-legged support - left lower limb, right lower limb partial step length [2].

Normal walking involves the appropriate morpho-functional adaptation of the nervous, locomotor, cardiovascular and respiratory apparatuses, requiring a perfect coordination of the movements of the structural elements involved in this activity, in conditions of energy and functional economy, on different substrates with different textures.

Walking restrictions can appear at any age, following inflammatory and degenerative conditions, as well as traumas that affect the integrity and functionality of the elements of the locomotor, respiratory and cardiovascular apparatuses, central and peripheral neurological conditions. After a certain age, the bone skeleton undergoes a demineralization process that leads to a decrease in resistance to external stress, which results in the production of diseases such as osteoporosis. Osteoarthritis and rheumatic arthritis affect the structure of mobile joints such as the hip, shoulder, knee, elbow or ankle. Following cerebrovascular accidents, patients are affected by sudden or progressive paralysis and it mainly manifests itself on motor or sensory actions in the limbs of the human body on one of its parts.

In order to reduce disabilities and improve patients' live, walking rehabilitation systems were used, including orthopedic implants, which replace functional structures affected by diseases or traumas, with an artificial component, made of materials that allow the restoration of tissue's function, as well as its appearance. [3] To realize it, several types of materials are needed, depending on how damaged the tissue is and the behavior of the material under various static and dynamic demands. Considering that, over time, implants wear out due to corrosion, friction or abrasion, it is important to use materials as resistant as possible, in order to increase the duration of their use. [4] The choice of appropriate materials to be used in the prostheses' construction has been the subject of study by many researchers [5] and it has been observed that from a mechanical point of view, metallic and ceramic materials seem to be the best choice for the medical applications in the case of hard biological tissues, while polymers can be used in the case of soft biological tissues. [6], [7], [8]

Ceramics used for the reconstruction of the damaged parts of the musculoskeletal system are alumina, zirconia, tricalcium phosphate, or calcium phosphates.

The main metals used in implants are vanadium steel – used in flat bone fractures and screws; titanium, iron, tantalum, chromium, cobalt, molybdenum, niobium – used to create the alloys used in implants tolerated by the body.

Over time, alloys based on cobalt and chromium were used, with maximum resistance to fatigue and traction, titanium alloys, which have very good mechanical (such as high elasticity), chemical, physical properties, and it was based on non-toxic alloys and alloys with shape memory.

Stainless steels are also used to make implants, having an increased resistance to demands, high temperatures and pressures in corrosive and aggressive environments.

Over time, the body's movement or the replaced segment causes wear of the material, causing defects due to excessive deformation, breakage, corrosion and electrochemical attack, a process that causes the destruction of the prosthesis.

To eliminate wear problems and increase the prostheses' durability, coatings were made [9], [10], [11] with thin biocompatible metallic (such as Ag, Cu, Zn, Ti, Mg, Ca), polymeric (hydroxyapatite) or antimicrobial (metal oxides) films of the prosthesis components using variants of technologies derived from the CVD (chemical vapor deposition) and PVD (physical vapor deposition) processes. The main methods are thermal evaporation, cathodic arc deposition, electron beam physical vapor deposition (EBPVD), Direct Current (DC) magnetron sputtering, Radio Frequency (RF) magnetron sputtering and the sol-gel method. [12]

Coating with hydroxyapatite is considered a solution to ensure the achievement of osseointegration in conditions of suboptimal mechanical fixation. Active antimicrobial coatings of inorganic metal oxides (ZnO, TiO_2, MgO, CaO, etc.) reduce the risk of infections associated with implants and improve the life's quality, without altering their properties. In addition, antimicrobial coatings reduce problems related to surfaces' corrosion, contribute to the reduction of allergies caused by Cr and Ni in steels, and prevent or reduce superficial and deep local surgical infections due to improper use of medical instruments.

Starting from the ever-increasing importance of coatings in orthopedic implants, the authors realized researches to determine the appropriate material and method for depositing a thin film on the surface of a hip prosthesis components, in order to increase their hardness.

2. Experiments

2.1. Materials

The researches carried out within the project were based on the mechanical and functional analysis of micro-nanostructured thin films of TiO_2, TiN and CrN with 3 different thicknesses (0.5 μm, 1 μm and 1.5 μm) deposited by DC magnetron sputtering, HiPIMS (High Power Impulse Magnetron Sputtering) and Cathodic Arc (thermal evaporation with an electric arc) on two types of substrates currently used for orthopedic prostheses – CoCr and M30NW steel. [12], [13]

The tests were initially carried out in laboratory on substrate samples with a thickness of 8 mm and a diameter of 10 mm, produced by the mechanical processing of CoCr and biocompatible M30NW steel bars, using a MECATOME T202 cutting machine and a MECATECH SPI grinding machine. [14]

2.2. Thin Films Deposition

Deposition of the thin films by cathodic arc, DC sputtering and HiPIMS was carried out at SC MGM Star Construct SRL using a DC and HiPIMS sputtering Leybiold Z-550-S equipment and Cathodic-Arc UVN-MGM equipment. [14] The deposition processes through the three methods had the technological parameters presented in Table 1.

Table 1. Technological parameters of the deposition methods

Technological parameter	HiPIMS sputtering	DC sputtering	Cathodic Arc
Power, W_t	1000	1000	1000
BIAS, V	100	100	
Current, A			40
Voltage, V			24-26
Pressure, mbar	$10^{(-3)}$	$10^{(-3)}$	
Pressure, Pa			$4x10^{(-1)}$

The TiN and TiO_2 thin films were deposited by the three methods using N_2 and O_2, respectively, as process gas. CrN layers were deposited by DC sputtering and HiPIMS sputtering, using N_2 as process gas. The deposition time varied between 5, 10 and 15 minutes to obtain layers with thicknesses of 0.5 μm, 1 μm and 1.5 μm.

2.3. Nanoindentation and Tribological Tests

Physico-mechanical, structural, topographic and tribological characterizations of the deposited micro-nanofilms were carried out, in order to highlight the properties of the obtained thin films, and to observe the defects that can appear in these films, following the production processes.

The hardness of the thin films deposited on the substrate samples was determined following nanoindentation tests performed with a Hysitron TI Premier nanoindenter using a maximum pressing force of 0.7 mN (except for the CrN layer – force of 2 mN). [14]

Figure 1: Ball on disc tribometer TriboLab UMT Bruker

To perform the tribological tests, in order to determine the wear rate, a ball on disc tribometer (Figure 1) was used – TriboLab UMT Bruker for universal mechanical testing and tribological testing.

The wear track profiles were investigated using a Dektak 150 electro-mechanical profilometer.

2.4. Evaluation of the Applicative-Tribological Capacity

In order to carry out experimental researches to determine the applicability of micro-nanostructured thin films, hip prostheses components and femoral heads coated with thin films were made.

CoCr femoral heads were produced by 3D printing using the metal laser sintering prototyping equipment, Shining 3D EP M250 3D Printer (Figure 2) – from the Research Center for Intelligent Mechatronic Systems for Security Objectives and Interventions – CERMISO of INCDMTM. UHMWPE acetabular cups and Rul type biocompatible steel femoral stems were made by mechanical processing in the laboratories and workshops of INCDMTM.

Figure 2: Shining 3D EP M250 3D Printer

Evaluation of the ability to use the nanostructured thin films in the real environment was carried out by testing in an environment similar to the real one the femoral heads obtained and coated with the thin films that presented superior tribological properties following the tests performed on the samples.

The MTS Bionix Implants and Prosthetic Components Testing Equipment (Figure 3) allows testing of ankle-foot devices and foot units according to standardized methods. The wear subsystem with several degrees of freedom, which performs movements of flexion/ extension, abduction/ adduction, internal/ external rotation, was used to test the hip prosthesis.

Figure 3: MTS Bionix Implants and Prosthetic Components Testing Equipment

Values of the parameters used for the tests carried out in the case of prostheses with femoral heads coated with TiO_2, TiN and CrN thin films are presented in Table 2.

Table 2. Values of the parameters used for the tests carried out

Test Summary Table 1

Name	Value	Unit
Test Reference		
Specimen Refere...		
Level Multiplier	100.000	%
Number of Cycles	5000000	count
Maximal Force	-0.300	kN
Minimal Force	-3.000	kN
Flexion Angle	25.000	°
Extension Angle	-18.000	°
Abduction Angle	-4.000	°
Adduction Angle	7.000	°
External Rotation	-10.000	°
Internal Rotation	2.000	°

2.5. Determining the Wear Degree of the Deposited Thin Films

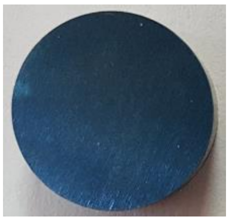
In order to determine the wear degree of the deposited thin films, the femoral heads were characterized after application tests, with the help of the KLA TENCOR-MICRO XAM-100 surfaces' microgeometry measurement system, which allows obtaining the values of the main tribological parameters (Figure 4).

Figure 4: KLA TENCOR-MICRO XAM-100 surfaces' microgeometry measurement system

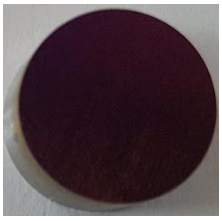
3. Results and Discussions

3.1. Thin Films

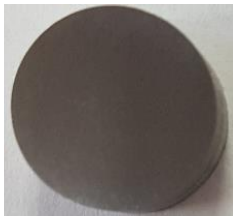
TiO_2, TiN and CrN thin films with thicknesses of 0.5 µm, 1 µm and 1.5 µm deposited on CoCr and M30NW steel substrates by DC sputtering, HiPIMS and cathodic arc were obtained. (Figure 5) Their characterization demonstrated the uniform surface of these substrates.



0.5 µm TiO_2 thin film deposited on CoCr substrate by DC sputtering



1 µm TiN thin film deposited on M30NW steel substrate by HiPIMS



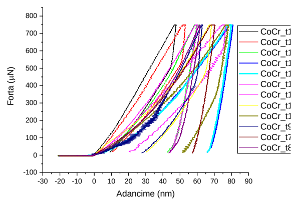
1,5 µm CrN thin film deposited on CoCr substrate by HiPIMS

Figure 5: Thin films deposited on CoCr and M30NW steel substrates.

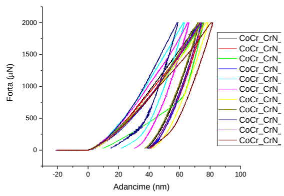
3.2. Nanoindentation and Tribological Tests

To determine the hardness and the wear rate of the thin films deposited on the samples, nanoindentation and tribological tests were carried out, the obtained values being detailed in the form of nanoindentation graphs (Table 3, Table 4) and tribological graphs.

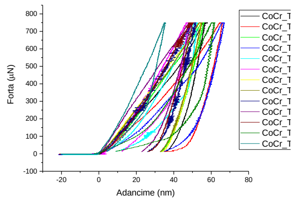
Table 3. Nanoindentation graphs of the thin films deposited on CoCr substrate



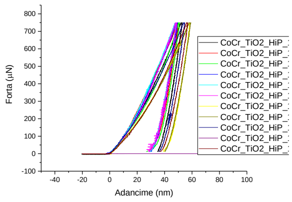
CoCr substrate



0.5 µm CrN thin film deposited by DC sputtering

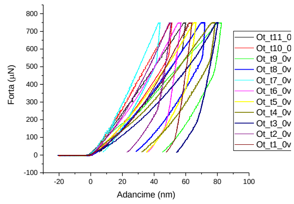


1 µm TiN thin film deposited by DC sputtering

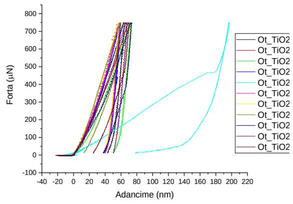


1.5 µm TiO_2 thin film deposited by HIPMS sputtering

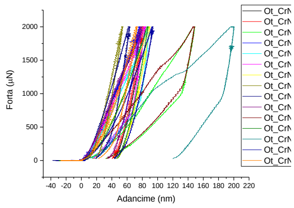
Table 3. Nanoindentation graphs of the thin films deposited on M30NW steel substrate



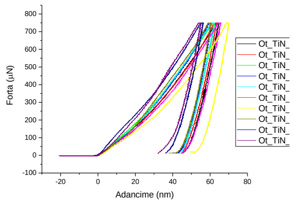
M30NW steel substrate



0.5 µm TiO_2 thin film deposited by cathodic arc

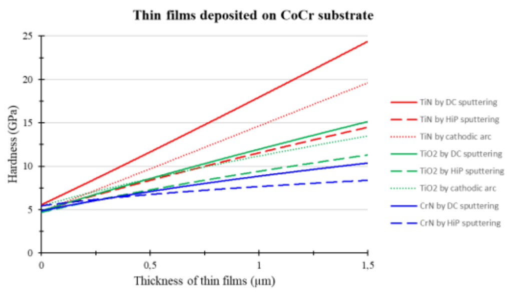


1 µm CrN thin film deposited by DC sputtering

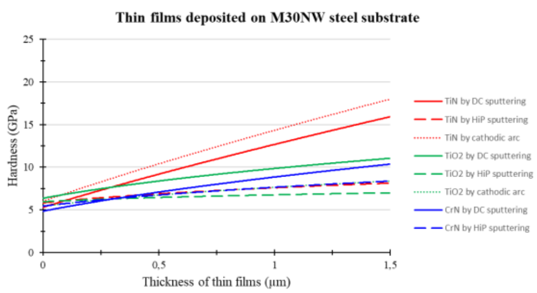


1.5 µm TiN thin film deposited by HIPMS sputtering

The hardness values of the deposited thin films were analysed to determine the influence of the film's thickness of each material and of the deposition method. Following the tests carried out, the variation graphs of the average hardness were developed according to the thickness of each type of thin film deposited by 3 different methods on both substrates' types used in the tests (Figure 6).



(a)



(b)

Figure 6: The variation of the hardness depending on the thickness of each type of thin film deposited by DC sputtering, HiPMS sputtering and cathodic arc on (a) CoCr and (b) M30NW steel substrates.

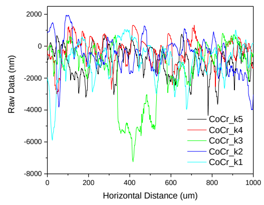
The hardness of the TiN thin films deposited on both types of substrates (CoCr and M30NW steel) increased uniformly as the film's thickness increased. All these values were also influenced by the three deposition methods that were used, so that the hardness values of the TiN thin films deposited on both types of substrates were the highest when cathodic arc deposition method was used.

The hardness of the TiO_2 thin films deposited on CoCr substrate and on M30NW steel substrate had a uniform increase with the increase of the films' thickness when these were deposited using DC sputtering method.

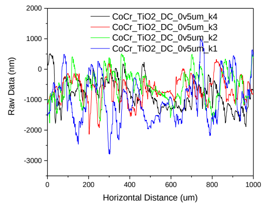
CrN thin films deposited on both substrates had a uniform increase of hardness with the increase in of the film's thickness. Taking into account the influence of the deposition methods, the hardness values of the CrN layers deposited on both substrates were the highest when DC sputtering deposition method was used.

After the tribological tests, the wear traces' profiles were made for all the tested samples, obtaining the average values of the wear rate *k*. (Table 5, Table 6)

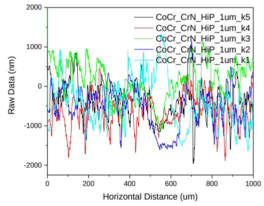
Table 5. Wear traces' profiles of the thin films deposited on CoCr substrate



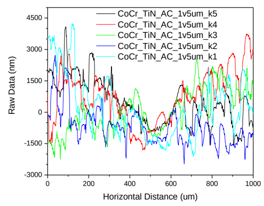
CoCr substrate



0.5 µm TiO_2 thin film deposited by DC sputtering



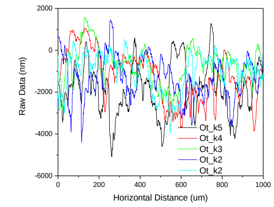
1 µm CrN thin film deposited by HIPMS sputtering



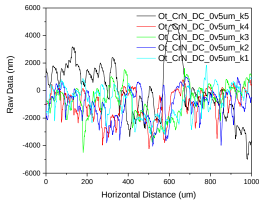
1.5 µm TiN thin film deposited by cathodic arc

Among the curves obtained, those with smaller errors were selected and the average values of the wear rate *k* were calculated. The wear rate decreased exponentially with the increase of the deposited film's thickness depending on the substrate and on the deposition, method used (Figure 7).

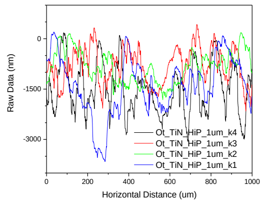
Table 6. Wear traces' profiles of the thin films deposited on M30NW steel substrate



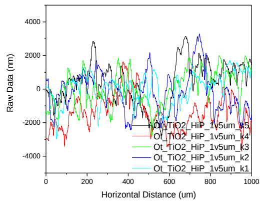
M30NW steel substrate



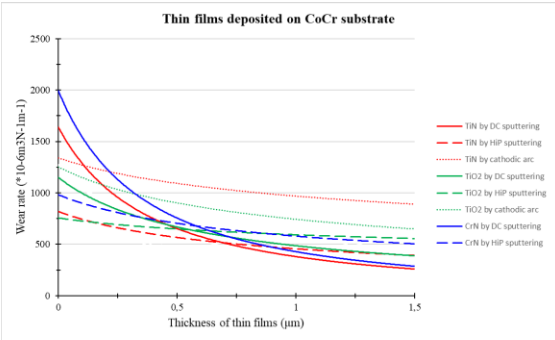
0.5 µm CrN thin film deposited by DC sputtering



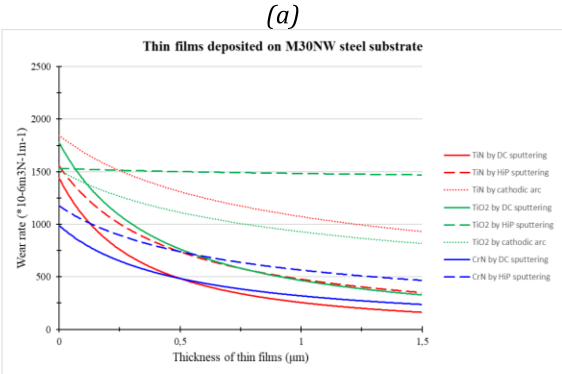
1 µm TiN thin film deposited by HIPMS sputtering



1.5 µm TiO_2 thin film deposited by HIPMS sputtering



(a)



(b)

Figure 7: The variation of the wear rate depending on the thickness of each type of thin film deposited by DC sputtering, HiPMS sputtering and cathodic arc on (a) CoCr and (b) M30NW steel substrates.

Wear rate of the TiN thin films deposited on the CoCr substrate decreased exponentially (Figure 7) as the thickness increased from 0.5 to 1.5 µm. The 1.5 µm thick TiN thin film, deposited on the CoCr substrate by cathodic arc, has a minimal wear rate value. The wear rate's values of the TiN thin films deposited on the M30NW steel substrate were the lowest when the films were deposited by the DC sputtering method.

Wear rate of the TiO_2 thin films deposited on the CoCr substrate by DC sputtering decreases as the thickness of the substrate increases.

TiO_2 thin films deposited on the steel substrate show an increase in wear resistance as their thickness increases. The wear rate values were the lowest when the thin films were deposited by the DC sputtering method.

The wear rate's values of the CrN thin films deposited on the CoCr substrate were the lowest when the films were deposited by the DC sputtering method. The obtained results showed that the CrN thin films deposited on the steel substrate show an increase in the wear resistance as their thickness increases, having the lowest values when DC sputtering deposition method was used.

Following these tests, performed on samples, the TiN, TiO_2 and CrN thin films with a thickness of 1.5 µm were chosen to be deposited by DC sputtering method on the surface of the femoral heads which were tested to determine the applicative capacity.

3.3. Components of the Hip Prosthesis

Following the experimental activities carried out in order to obtain the components of total hip prostheses to be studied in an environment similar to the real one, femoral heads made of CoCr, with an external diameter of 28 mm and acetabular cups made of UHMWPE, were made. (Figure 8)

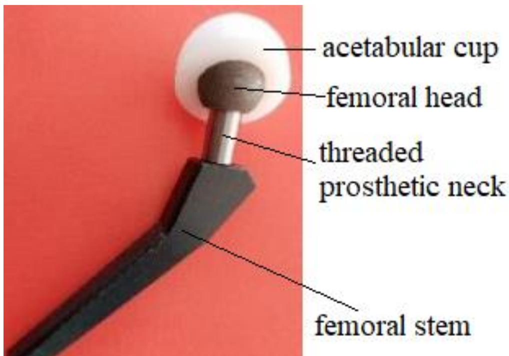


Figure 8: The components of a hip prosthesis realized at INCDMTM.

3.4. Applicative-Tribological Capacity

3.4.1. Femoral Heads Coated with Thin Films

To test the applicative capacity of the micro-nanostructured thin films deposited on the components of a hip prosthesis, coated femoral heads were tested for wear resistance in an environment similar to the real one, according to the procedures previously described.

Thus, the CoCr femoral heads made by 3D printing and the M30NW steel femoral heads were coated with thin films of 1.5 μm thickness by DC sputtering. At the end of these processes, femoral heads coated with TiN, TiO_2 and CrN thin films with a thickness of 1.5 μm were obtained. (Figure 9)

1.5 μm thick TiN thin film deposited by DC sputtering
(a)

1.5 μm thick TiO_2 thin film deposited by DC sputtering
(b)

1.5 μm thick CrN thin film deposited by DC sputtering
(c)

Figure 9: Femoral heads coated with thin films of TiN (a), TiO_2 (b) and CrN (c)

The surface of the M30NW steel femoral heads coated with thin films were characterized at a microscopic level with the KLA TENCOR-MICRO XAM-100 equipment, obtaining the main tribological parameters' values – roughness, skewness, coefficient of kurtosis, before being subjected to tests simulating the movement of the hip prostheses. The average values of these parameters of the deposited thin films are presented in table 7.

On the CoCr substrate, thin films that present a higher roughness value compared to the thin films deposited on the M30NW steel substrate were obtained. These values also depend on the quality of the substrate.

The micro-nanostructured TiN thin film has the lowest roughness compared to the CrN and TiO_2 films, in both cases, which means that the TiN thin film is the most uniformly deposited. All surfaces are asymmetric, but the coefficient values are close to zero, which indicates a small variation in symmetry. The values of the coefficient of kurtosis show a variable distribution of the heights on the two films' surface.

Table 7. The average values of the tribological parameters of the coated femoral heads' surfaces before the technico-functional testing

Thin film	Roughness	Skewness	Coefficient of kurtosis
TiN thin film on CoCr substrate	226.3	-0.282	-0.43
TiN thin film on M30NW steel substrate	102.4	-0.661	0.547
TiO_2 thin film on CoCr substrate	228.6	-0.254	-0.916
TiO_2 thin film on M30NW steel substrate	195.2	-0.433	-0.406
CrN thin film on CoCr substrate	226.9	-0.275	-0.723
CrN thin film on M30NW steel substrate	123.5	-0.502	-0.124

3.4.2. Characterization of the Thin Films' Surface After the Technico-Functional Testing

At the macroscopic level, following the applicative tests of the prostheses with coated femoral heads, traces of material and destructions of the tested femoral heads' surface were observed on the acetabular cups. (Figure 10, Figure 11)

Figure 10: Acetabular cup with CrN trace, integrated in the module of the MTS Bionix testing system

After the wear testing of the femoral heads coated with TiN, CrN and TiO_2 thin films, it was observed that damages such as scratches, cracks and wear particles detached from the tested film appeared on the surface of the thin film (Figure 12, Figure 13, Figure 14). The presented images demonstrate this and highlight the fact that the TiO_2 thin film deposited on the CoCr femoral head is the most destroyed. The TiN thin film deposited on the M30NW steel femoral head presented the best wear resistance.

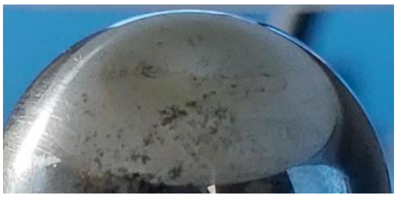
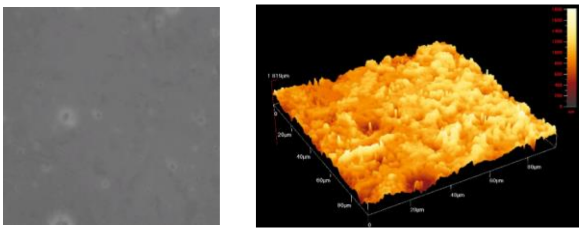
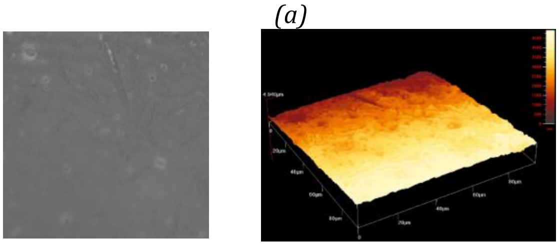


Figure 11: The upper part of the uncoated CoCr femoral head

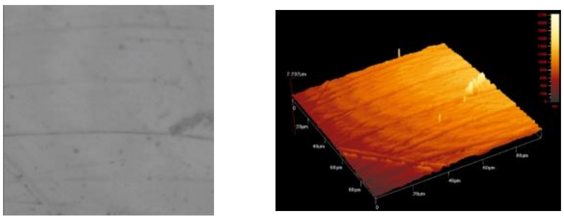


(a)

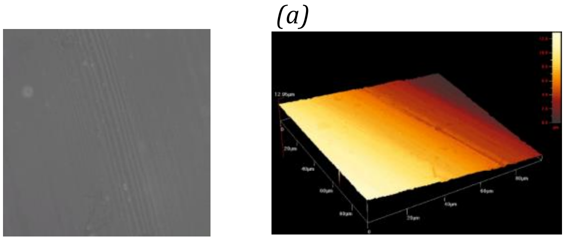


(b)

Figure 12: Surface of the TiO_2 thin film deposited on the CoCr (a) and M30NW steel (b) femoral heads, after wear tests (scale 50:1).

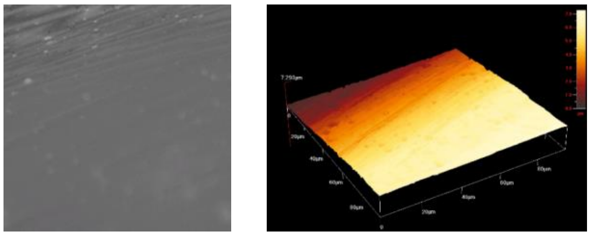


(a)

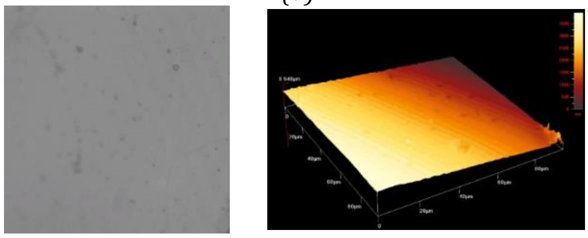


(b)

Figure 13: Surface of the CrN thin film deposited on the CoCr (a) and M30NW steel (b) femoral heads, after wear tests (scale 50:1).



(a)



(b)

Figure 14: Surface of the TiN thin film deposited on the CoCr (a) and M30NW steel (b) femoral heads, after wear tests (scale 50:1).

Following the technico-functional characterization tests, the tribological parameters (Table 8) were obtained with increased values compared to those that the thin films had initially, which indicates their wear. There is a greater difference between the roughness of the thin films before and after the technico-functional testing. This demonstrates the greater wear resistance of the thin films deposited on both types of substrates.

Table 8. The average values of the tribological parameters of the coated femoral heads' surfaces after the technico-functional testing

Thin film	Roughness	Skewness	Coefficient of kurtosis
TiN thin film on CoCr substrate	397.9	-0.158	-0.826
TiN thin film on M30NW steel substrate	160.1	-0.055	0.125
TiO_2 thin film on CoCr substrate	684.9	-0.351	-0.916
TiO_2 thin film on M30NW steel substrate	492.5	-0.898	-0.482
CrN thin film on CoCr substrate	458.9	-0.198	-0.898
CrN thin film on M30NW steel substrate	305.3	-0.432	-0.353

The increase in surface roughness of the uncoated femoral head compared to the coated femoral heads was also determined. This proved that the TiN, TiO_2 and CrN thin films offer protection against wear.

The analysis of the tribological parameters showed that, depending on the material, the surface's wear had a different level, thus TiN had the highest wear resistance compared to CrN and TiO_2. If the method used for deposition is taken into account, it was observed that the thin films deposited by DC sputtering method had a lower roughness after the tests, which means a lower destruction of the film and an increased resistance to wear. Thus, following the obtained experimental results, it was demonstrated that the thin film that was the most resistant to wear, presenting the highest degree of applicability, was the TiN thin film with a thickness of 1.5 μm deposited by the DC sputtering method.

4. Conclusions

The main objective of the experiments was to carry out complex research activities regarding the characterization and evaluation of the application capabilities of micro-nanostructured thin films intended for biomedical applications, deposited on a metal support, through physical processes.

As part of the research, experimental attempts were made to deposit micro and nanometric films of TiO_2, TiN and CrN by DC sputtering, HiPIMS sputtering and cathodic arc on CoCr and M30NW steel substrates. Thus, thin films of TiO_2, TiN and CrN with thicknesses of 0.5 μm, 1 μm and 1.5 μm were obtained.

Nanoindentation tests were carried out to determine the hardness and tribological tests to determine the wear rate. The physico-mechanical and tribological characterization of the samples provided information related to the quality of the deposited material and the influence of the deposition technique.

The surface wear had a different level, so that TiN and CrN thin films had better resistance to wear compared to TiO_2. Taking into account the values of the analyzed parameters, the main conclusion is that the thin films deposited by DC sputtering have the best tribological characteristics, this being considered the best method for layer deposition. CoCr femoral heads were made using the 3D printing process, femoral stems and femoral necks using different physical-mechanical processes.

Following the nanodentation tests and the tribological tests carried out to choose the optimal variants of the film-substrate material couple, simulation tests of the movement of hip prostheses coated with thin films were carried out in order to determine the applicability of micro-nanostructured thin films' depositions.

The information obtained from the comparative analysis of the experimental results proved the fact that the use of thin films, with superior properties, in an environment similar to the real one offers an increased resistance of the hip prostheses on which they are deposited.

After the analysis, the results obtained experimentally on test samples were demonstrated and validated, namely the fact that the most wear-resistant thin film was the TiN film with a thickness of 1.5 μm deposited by the DC sputtering method on the M30NW steel substrate.

Thus, following the research and the results obtained, an increase in the quality of micro and nanostructured thin films with direct applications in recuperative biomedicine can be estimated.

Results obtained in these researches will be developed, in the future, in international projects submitted through the "Support Center for international RDI projects in the field of Mechatronics and Cyber-MixMechatronics", with the aim of improving the quality of biomedical components, that have a great importance in society.

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