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VARIATION OF TRIBOLOGICAL PARAMETERS DEPENDING ON THE THICKNESS OF THE THIN FILMS USED FOR BIOMEDICAL APPLICATIONS

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Abstract: *This paper presents the results obtained from a project that had as main objective to improve the tribological characteristics of biomedical components in order to increase their lifetime. A solution for optimizing the constructive characteristics and increasing the operational duration for orthopaedic implants components is the deposition of micro and nanostructured thin films through intelligent mechatronic processes and technologies.* TiO_2*, TiN and CrN thin films with thicknesses of 0.5 µm, 1 µm and 1.5 µm were deposited by Direct Current (DC) sputtering, High Power Impulse Magnetron Sputtering (HiPIMS) and cathodic arc, on two types of substrates: CoCr and M30NW steel. The variation of the friction coefficient as a function of time and the wear traces profiles for all samples in each type of thin film were analysed. Following the tribological characterization, and the comparative analysis of the experimental results, information regarding the quality of the deposited material and the degree of influence of thin films thickness were obtained. The main conclusion of the study was that the friction coefficient and the wear rate decrease with an increase of the layer thickness. This means that a thicker layer will be more sustainable and can be used to improve the quality of biomedical components, like hip prostheses.*

Keywords: *Thin films, tribological characterization, friction coefficient, wear rate, implants*

1. Introduction

Substantially improving the quality of life of patients with severe motor disabilities by using partial or total arthroplasty is an important topic of current research in the field of materials engineering and biotribology. [1] This because, over time, the movement of the body causes the wear of the material, loose of fixation and destruction of the implant, most failures occurring due to improper properties or incorrect choice of materials. [2]
Deposition of micro and nanostructured thin films on the biomedical metallic implants and their components, is a solution for reducing the wear rate and increasing the operational time without changing the surface geometry. [3, 4, 5] Generally, coatings applied for improving the implants resistance must have a high adhesion to the substrate and a low friction coefficient to prevent detachment from the substrate, overheating and accelerated wear. Presently, there are studies regarding the use of nitride-based compounds [6], amorphous carbon [7], calcium-phosphate [8], diamond-like carbon [9] as coatings for increasing the implants resistance.
Based on this information and with the aim to improve the technical and functional properties of implants, micro- and nanostructured thin films with various thicknesses, deposited on materials with high mechanical strength and low wear, currently used in orthopaedics, were tested and analysed in the present project.
Tribological tests were performed to determine the friction coefficient and the wear rate of these micro-nanostructured thin films and the dependence on the thickness was assessed by characterization and comparative analysis of the experimental results.

2. Experiments

2.1 Preparation of the samples

In this study, materials with an increased wear resistance were tested and analysed in order to determine their capability to improve the tribological properties of biomedical components. Thus, thin layers of TiN, CrN si TiO_2 were deposited on substrates with a composition similar to the prostheses one, respectively CoCr and M30NW steel substrates [10, 11]. Studying nitrides and oxides with antimicrobial properties, it is possible to determine which of these have an increased resistance and will be more efficient for biomedical components.
Thin films were deposited on substrate disks of 8 mm thick and 10 mm in diameter. CoCr samples disks had been produced by 3D printing technique, using a laser metal sintering prototyping equipment – the Shining 3D EP M250 3D Printer (Fig. 1) – from the Research Centre for Intelligent Mechatronic Systems used for Securing Objectives and Intervention – CERMISO of INCDMTM. [12]

Fig. 1. Shining 3D EP M250 3D Printer

M30NW steel samples disks had been produced by mechanical processing techniques, respectively machining from M30NW steel bars, using the MECATOME T202 micro-cutting machine and polishing by using the MECATECH SPI machine. [12]

2.2 Thin films deposition

Micro and nanostructured biocompatible thin films were deposited on CoCr and M30NW steel substrates by physical techniques. TiO_2, TiN and CrN layers with thicknesses of 0.5 μm, 1 μm and 1.5 μm were deposited by Direct Current (DC) sputtering, High Power Impulse Magnetron Sputtering (HiPIMS) and cathodic arc techniques.
Cathodic arc is a technique in which the material from a cathodic target is vaporized, in vacuum, using an electric arc, then condenses on the substrate, forming thin film. [13]
Sputter deposition is based on ion bombardment of a source material, the target, where ion bombardment results in a vapor due to a purely physical process, i.e., the sputtering of the target material. DC sputtering is a deposition technique where a target material is bombarded with ionized gas molecules that hit the target, sputtering off the atoms into the plasma. The vaporized atoms condense, forming a thin film on a substrate. [14]
HiPIMS uses the magnetic field created inside a deposition system. Increased plasma densities are created near the target, which increase the spraying rate. [15]
The deposition of thin layers by cathodic arc, DC sputtering and HiPIMS was performed at SC MGM Star Construct SRL using two equipment: a DC and HiPIMS equipment Leybiold Z-550-S and a Cathodic-Arc UVN-MGM equipment. [12]. Cathodic arc deposition was performed using a power Wt = 1000, Current = 40 A, Voltage = 24-26 V and Pressure = $4x10^{(-1)}$ Pa. For thin films deposition by using the HiPIMS and DC sputtering methods the technological parameters were: power, Wt = 1000, BIAS = 100 V and pressure = $10^{(-3)}$ mbar.
Deposition processes were performed over a period of 5, 10 and 15 minutes to obtain layers of CrN, TiN and TiO_2 with thicknesses of 0.5 μm, 1 μm, and 1.5 μm on CoCr substrate and biocompatible M30NW steel substrate.

2.3 Tribological tests

Tribological tests were performed at National Institute for Research and Development in Optoelectronics (INOE 2000) with the aim to determine the friction coefficient and wear rate of the biocompatible thin films deposited.
The TriboLab UMT Bruker ball on disc tribometer for universal mechanical and tribological testing was used to perform these tribological tests (Fig. 2). The main types of measurements involve parameters like resistance to breaking, bending and scratching, and offer the possibility of measuring very low friction forces.

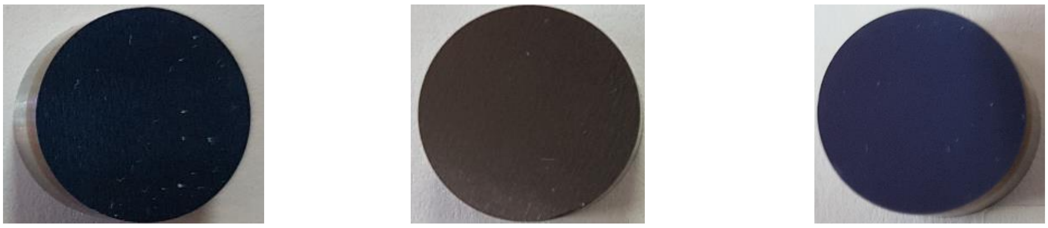
Fig. 2. Ball on disc tribometer

The variation of the friction coefficient as a function of time was determined applying a pressure force of 0.7N, on a sliding distance of 7,9m (300 cycles), during 80sec, with a speed of 10cm/s. The wear traces profiles were analysed using a Dektak 150 electro-mechanical probe profilometer, performing the tests on a scanning length of 1000 μm and applying a contact force of 49 μN.

3. Results

3.1 Thin films

Using the deposition systems with the technological parameters presented, TiO_2, TiN and CrN thin films were obtained, with thicknesses of 0.5 μm, 1 μm and 1.5 μm, deposited on CoCr and M30NW steel substrates (Fig. 3).



0.5 μm TiO_2 deposited on M30NW steel by DC sputtering | 1 μm CrN deposited on CoCr by DC sputtering | 1.5 μm TiN deposited on CoCr by DC sputtering

Fig. 3. Thin films deposited on CoCr and M30NW steel substrates by cathodic arc, DC sputtering and HiPIMS

3.2 Tribological tests

Following the tribological tests, the average value of the friction coefficient μ and the average value of the wear rates k were obtained. The variation of the friction coefficient as a function of time (Table 2) was also obtained for all samples from each type of layer (respectively each layer thickness). The wear traces profiles (Table 3, Table 4) were obtained for each type of deposition (respectively each layer thickness).

Table 2: Variation of the friction coefficient as a function of time

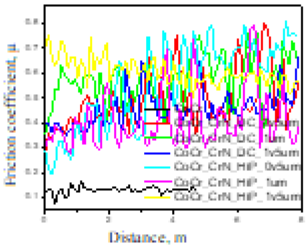
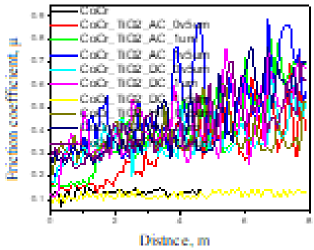
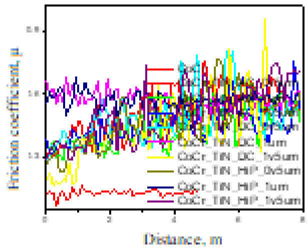
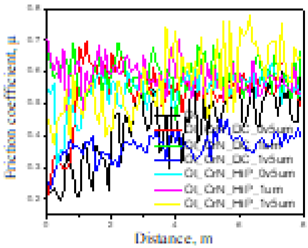
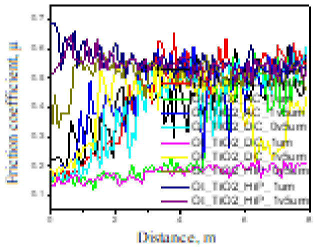
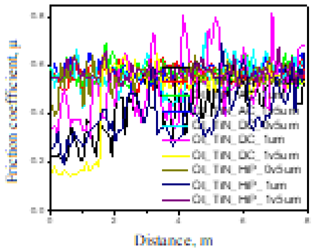
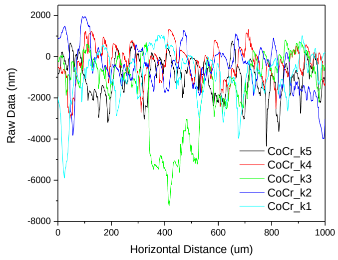
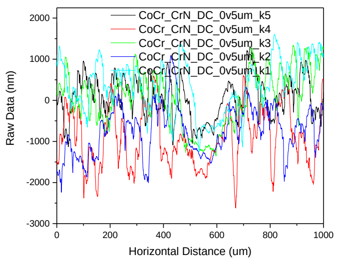
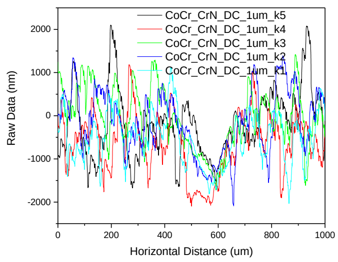
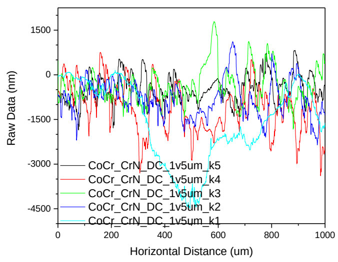
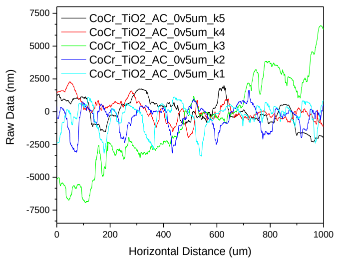
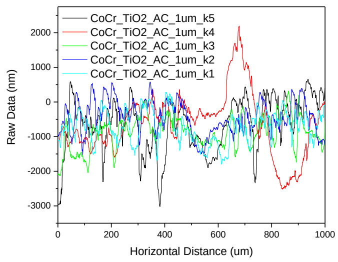
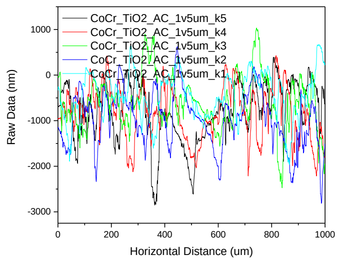
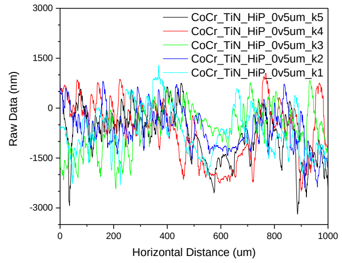
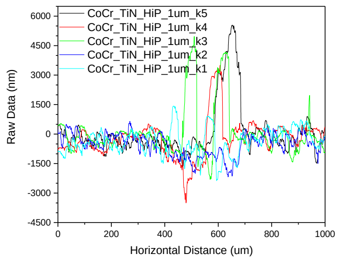
		
CoCr_CrN	CoCr_TiO_2	CoCr_TiN
		
Ot_CrN	Ot _TiO_2	Ot _TiN

Table 3: Wear traces profiles of the thin films deposited on CoCr substrate

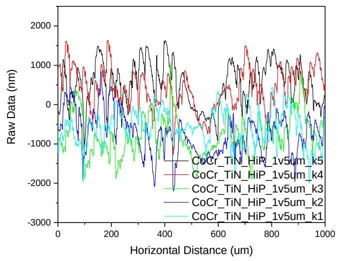
		
	CoCr	
		
CrN - DC 0.5	CrN - DC 1	CrN - DC 1.5
		
TiO_2 - AC 0.5	TiO_2 - AC 1	TiO_2 - AC 1.5



TiN - HIP 0.5

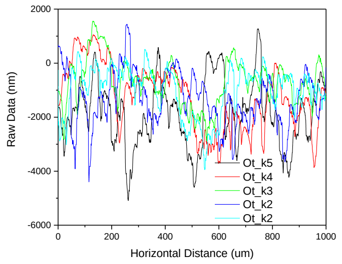


TiN - HIP 1

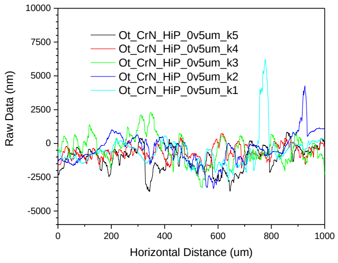


TiN - HIP 1.5

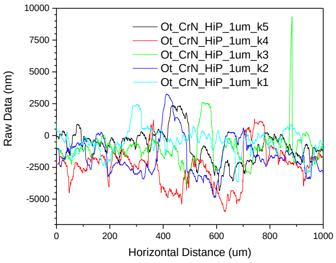
Table 4: Wear traces profiles of the thin films deposited on M30NW steel substrate



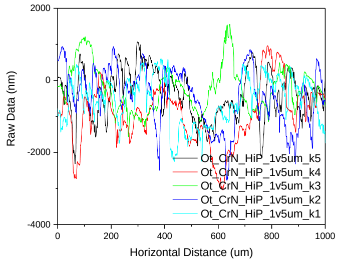
M30NW steel



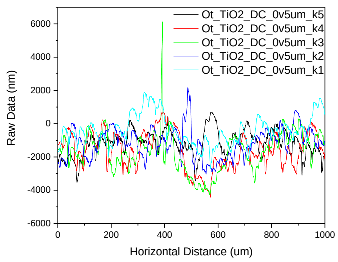
CrN - HIP 0.5



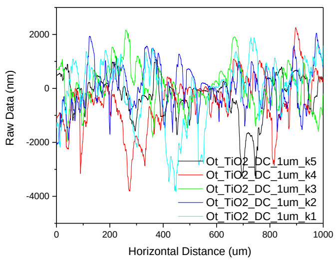
CrN - HIP 1



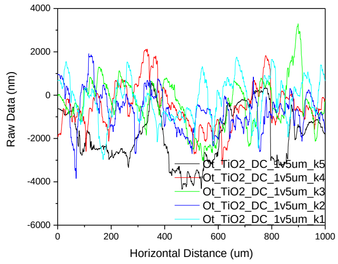
CrN - HIP 1.5



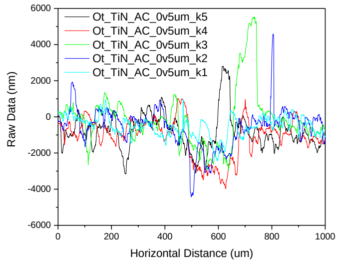
TiO_2 - DC 0.5



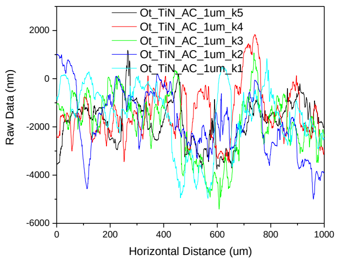
TiO_2 - DC 1



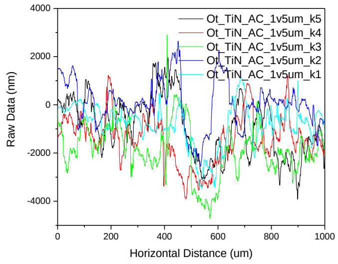
TiO_2 - DC 1.5



TiN - AC 0.5



TiN - AC 1



TiN - AC 1.5

Among the curves obtained, those with smaller errors were selected (presented grouped in the graph) to calculate the average values of the friction coefficient μ and of the wear rate k.
The friction coefficient and the wear rate of the deposited thin layers were analysed from the influence of the layer thickness and the deposition method point of view.

Friction coefficient μ – the influence of the thin films' thickness. The friction coefficient of the TiN layers deposited on the two types of substrates (Fig. 4) had a small variation, oscillating with the increase of the layer thickness (between 0.5 and 1.5 μm), having values between 0.132 and 0.649. The TiN layer with a thickness of 0.5 μm, deposited on the CoCr substrate by cathodic arc, has a maximum value of the friction coefficient. As the thickness of this layer increases, the value of the friction coefficient decreases. Increasing the layer's thickness from 1 μm to 1.5 μm also leads to a decrease of the friction coefficient when DC sputtering deposition method is used. However, the friction coefficient increases with the increase of the thickness, when the HiP sputtering method is used.
Regarding the friction coefficient of this layer when it is deposited on the M30NW steel substrate, although its values fall within a smaller range (0.504 and 0.593), there is a much more oscillating variation for the layers deposited by all the three methods. The TiN layer deposited by DC sputtering is the only one that shows a decrease of friction coefficient as the thickness increases. In the case of the other two layers, an increase of friction coefficient is observed as the layer thickness increases. If a uniform variation is taken into account, the increase of the friction coefficient also occurs proportionally to the increase of the thickness.

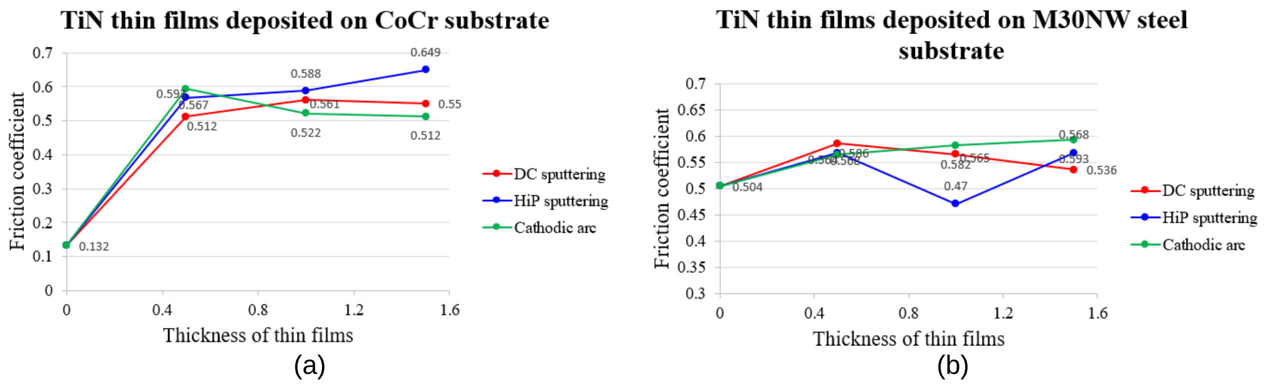
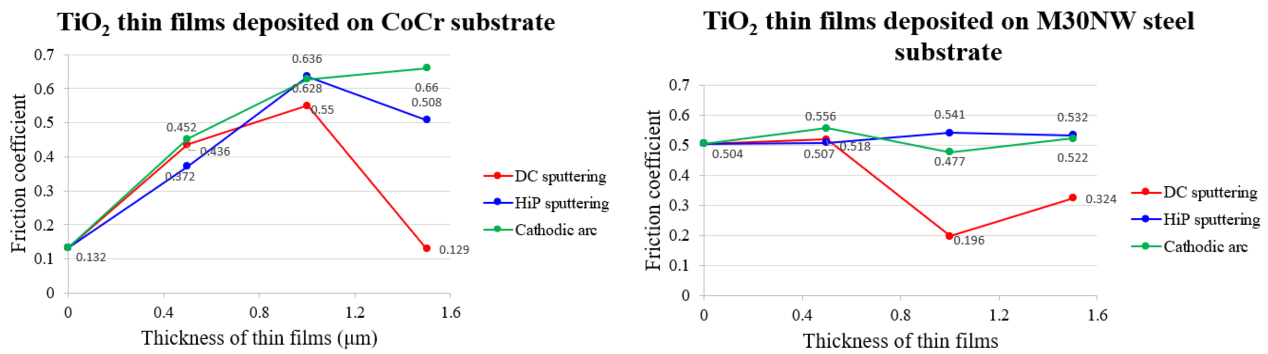


Fig. 4. Variation of the friction coefficient depending on the thickness of the TiN thin films deposited on CoCr substrate (a) and M30NW steel substrate (b)

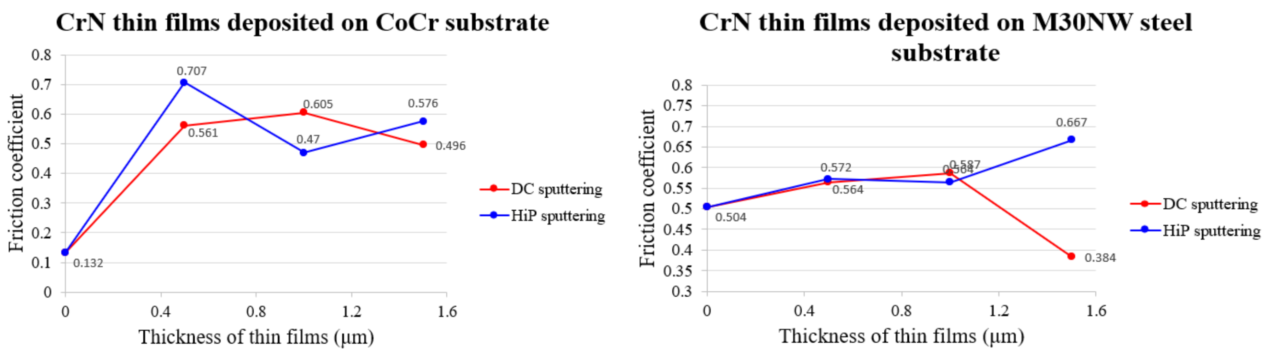
The friction coefficient of the TiO_2 layers deposited on the CoCr substrate (Fig. 5) had a uniform increase with the increase of the layer thickness (between 0.5 and 1 μm) and a decrease with the increase of the layer thickness up to 1.5 μm.
In the case of TiO_2 layers deposited on M30NW steel substrate, it was observed that as the layer thickness increases from 0.5 μm to 1.5 μm, there is a decrease in the friction coefficient of the layers deposited using the cathodic arc and DC sputtering methods. The decrease in the value of the friction coefficient occurs with the increase of the layer thickness from 1 μm to 1.5 μm when using the HiP sputtering method.

The friction coefficient of CrN layers deposited on both types of substrates (Fig. 6) had different variations with the increase of the thickness in both cases. Thus, increasing the thickness of the CrN layer deposited on the CoCr substrate led to a decrease of friction coefficient when both deposition methods were used. In the case of the layer deposited on M30NW steel, friction coefficient decreases as the layer thickness increases when the DC sputtering method is used. The value of the friction coefficient increases for the layer deposited by HiP sputtering once the layer thickness increases up to 1.5 μm.



(a) (b)

Fig. 5. Variation of the friction coefficient depending on the thickness of the TiO_2 thin films deposited on CoCr substrate (a) and M30NW steel substrate (b).



(a) (b)

Fig. 6. Variation of the friction coefficient depending on the thickness of the CrN thin films deposited on CoCr substrate (a) and M30NW steel substrate (b).

Wear rate k – the influence of the thin films' thickness. The wear rate of the deposited TiN layers (Fig. 7) had an uneven variation as the thickness increased (between 0.5 and 1.5 µm) for both types of substrates, the values being between $207 \cdot 10^{-6} m^3N^{-1}m^{-1}$ and $3510 \cdot 10^{-6} m^3N^{-1}m^{-1}$. The 1 µm thick TiN layer, deposited on the CoCr substrate by DC sputtering, has a maximum wear rate value. Once the thickness of this layer increases to 1.5 µm, the value of the wear rate decreases, being an important result in the use of this material, demonstrating the increased resistance to wear. The wear rate of the TiN layers deposited by the other two methods had an oscillatory increase depending on the layer thickness.

The TiN layers deposited on the M30NW steel substrate using all 3 deposition methods showed a wear rate with a completely uneven variation once the thickness increased from 0.5 to 1.5 µm. The TiN layer with a thickness of 1 µm, deposited on M30NW steel substrate by cathodic arc, has a maximum value of the wear rate. Once the layer thickness increases from 1 µm to 1.5 µm, the value of the wear rate decreases when the cathodic arc and HiP sputtering methods were used. Taking into account a uniform average variation, there is a decrease of the wear rate proportional to the increase of the layers thickness between 0.5 and 1.5 µm, when using the cathodic arc method and the HiP sputtering method. In the case of the layer deposited by DC sputtering, the decrease of the wear rate occurs only up to a layer thickness of 1 µm, the 0.5 µm thicker layer showing an increase of the wear rate value.

The wear rate of the TiO_2 layers deposited on CoCr and M30NW steel substrates (Fig. 8) also had an uneven variation with the increase of thickness (between 0.5 and 1.5 µm) for all the three methods used, the values being between 62*$10^{-6}m^3N^{-1}m^{-1}$ and 3510*$10^{-6}m^3N^{-1}m^{-1}$. The 0.5 µm thick TiO_2 layer, deposited on the CoCr substrate by DC sputtering, has a maximum wear rate value.

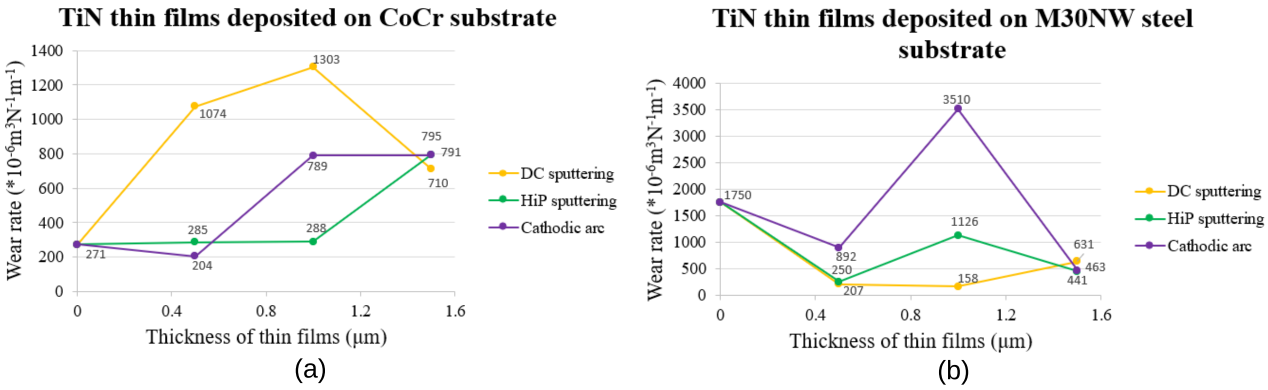


Fig. 7. Variation of the wear rate depending on the thickness of the TiN thin film deposited on CoCr substrate (a) and M30NW steel substrate (b).

Once the layer thickness increases from 0.5 µm to 1.5 µm, the wear rate value decreases when the DC sputtering and HiP sputtering methods were used. The TiO_2 layer deposited on the CoCr substrate by cathodic arc has an increase of the wear rate with the increase of the layer thickness between 0.5 and 1 µm, followed by a decrease with the increase of the thickness up to 1.5 µm. Taking into account a uniform average variation, there is an increase of the wear rate proportional to the increase of the layers thickness, when the cathodic arc method is used.
The wear rate of the TiO_2 layers deposited on the M30NW steel substrate also had an uneven variation with the increase in thickness (between 0.5 and 1.5 µm) for all three methods used. The TiO_2 layer with a thickness of 1 µm, deposited on M30NW steel substrate by cathodic arc, has a maximum wear rate value. Once the layer thickness increases from 1 µm to 1.5 µm, the value of the wear rate decreases when the cathodic arc and HiP sputtering methods were used. The TiO_2 layer deposited on the M30NW steel substrate by DC sputtering shows a decrease in the wear rate as the layer thickness increases between 0.5 and 1 µm, followed by an increase as the layer thickness increases up to 1.5 µm. Taking into account a uniform average variation, all three deposited layers show a decrease in the wear rate proportional to the increase of the deposited layers thickness. These results show that the TiO_2 layers deposited on the M30NW steel substrate present an increase of the wear resistance as their thickness increases.

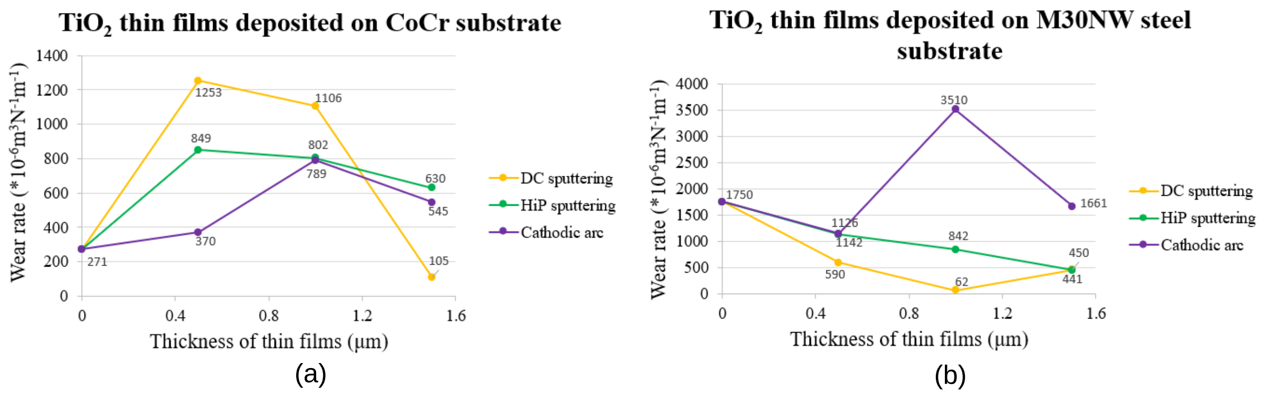


Fig. 8. Variation of the wear rate depending on the thickness of the TiO_2 thin film deposited on CoCr substrate (a) and M30NW steel substrate (b).

The wear rate of the CrN layers deposited on the two types of substrates (Fig. 9) had uneven variations, the values being between 119*$10^{-6}m^3N^{-1}m^{-1}$ and 1750*$10^{-6}m^3N^{-1}m^{-1}$, depending on the

thickness. On the CoCr substrate, the wear rate had an uneven variation once the thickness increased (between 0.5 and 1.5 µm), the CrN layer with a thickness of 1 µm, deposited on the substrate by DC sputtering, having the maximum value of the wear rate. The wear rate of this layer shows an increase with the increase of the layer thickness up to 1 µm, followed by a decrease with the increase of the layer thickness up to 1.5 µm. The value of the wear rate of the CrN layer deposited by HiP sputtering has an oscillatory variation with the increase of the layer thickness between 0.5 and 1.5 µm. Taking into account a uniform average variation, the layer deposited by HiP sputtering has an increase in the value of the wear rate.
The wear rate value of the CrN layers deposited on the M30NW steel substrate decreases proportionally with the increase in thickness (between 0.5 and 1.5 µm) for the two methods used. These results show that the CrN layers deposited on the M30NW steel substrate show an increase in wear resistance as their thickness increases.

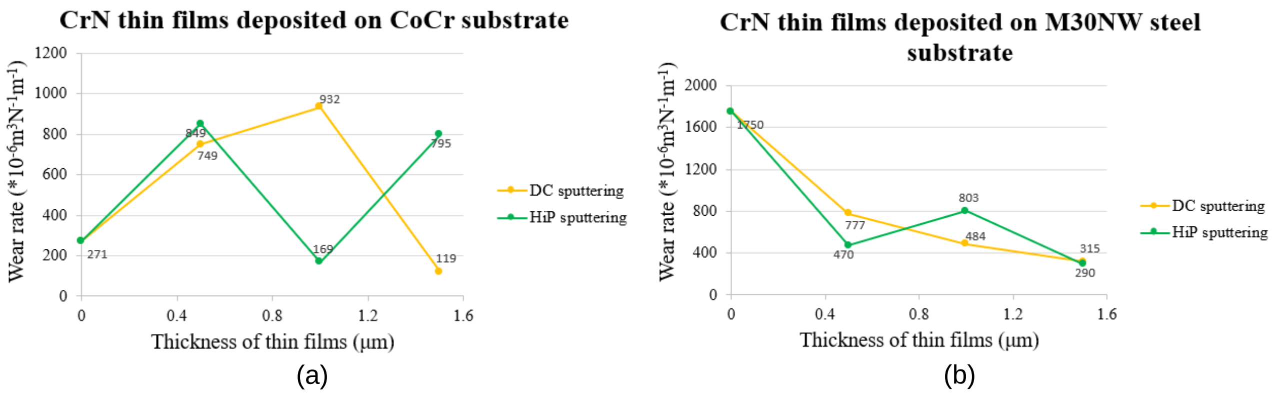


Fig. 9. Variation of the wear rate depending on the thickness of the CrN thin film deposited on CoCr substrate (a) and M30NW steel substrate (b)

For a comparative analysis of the obtained results and to determine the layers with the best tribological characteristics, the lowest values of the friction coefficient and of the wear rate were selected for each layer deposited on the two types of substrates used. (Table 5, Table 6)

Table 5: Comparative analysis of the parameters of the TiN, TiO_2 and CrN layers deposited on the CoCr substrate obtained after tribological tests. (The minimum values of the friction coefficient and of the wear rate. The comparison according to the material is marked with red, and the one according to the method is marked with blue).

Material	Layer thickness	Deposition method					
		DC sputtering		HiP sputtering		Cathodic arc	
		Friction coefficient	Wear rate	Friction coefficient	Wear rate	Friction coefficient	Wear rate
TiN	500 nm	0.512	1074	0.567	285	0.593	204
	1 µm	0.561	1303	0.588	288	0.522	789
	1.5 µm	0.550	710	0.649	795	0.512/met	791
TiO_2	500 nm	0.436	1253	0.372/met	40/849	0.452	370
	1 µm	0.550	1106	0.636	802	0.628	496/ 789
	1.5 µm	0.512/0.129	105/met	0.508	173/630	0.660	392/ 545
CrN	500 nm	0.561	127/749	0.707	849		
	1 µm	0.605	932	0.470	150/169		
	1.5 µm	0.496	119	0.576	795		

Table 6: Comparative analysis of the parameters of the TiN, TiO_2 and CrN layers deposited on the M30NW steel substrate obtained after tribological tests. (The minimum values of the friction coefficient and of the wear rate. The comparison according to the material is marked with red, and the one according to the method is marked with blue).

Material	Layer thickness	Deposition method					
		DC sputtering		HiP sputtering		Cathodic arc	
		Friction coefficient	Wear rate	Friction coefficient	Wear rate	Friction coefficient	Wear rate
TiN	500 nm	0.586	207	0.568	250	0.564	892
	1 µm	0.565	158	0.47/met	1126	0.582	3510
	1.5 µm	0.536	631	0.568	463	0.593	441
TiO_2	500 nm	0.518	590	0.507	112/772	0.556	627/1142
	1 µm	0.196/met	62	0.541	305/1126	0.477	952/3510
	1.5 µm	0.324	450	0.532	842	0.522	1161
CrN	500 nm	0.564	779	0.572	470		
	1 µm	0.587	484	0.564	803		
	1.5 µm	0.384	290/315	0.667	222/290		

Following the analysis of these values and comparisons, the following results were obtained.

TiN deposited on CoCr substrate

- The layer with the lowest friction coefficient is the one with a thickness of 1.5 µm deposited by cathodic arc, respectively the layer of 0.5 µm deposited by DC sputtering;
- The layer with the lowest wear rate is the one with a thickness of 1.5 µm deposited by DC sputtering.

From a tribological point of view, the TiN layer with a thickness of 1.5 µm deposited on a CoCr substrate by the cathodic arc method has superior properties.

TiO_2 deposited on CoCr substrate

- The layer with the lowest friction coefficient is the one with a thickness of 1.5 µm deposited by DC sputtering;
- The layer with the lowest wear rate is the one with a thickness of 1.5 µm deposited by DC sputtering.

From a tribological point of view, the TiO_2 layer with a thickness of 1.5 µm deposited on a CoCr substrate by the DC sputtering method has superior properties.

CrN deposited on CoCr substrate

- The layer with the lowest friction coefficient is the one with a thickness of 1 µm deposited by HiP sputtering, respectively 1.5 µm deposited by DC sputtering;
- The layer with the lowest wear rate is the one with a thickness of 1.5 µm deposited by DC sputtering.

From a tribological point of view, CrN layers with a thickness of 1 µm deposited by HiP sputtering, respectively 1.5 µm deposited by DC sputtering on a CoCr substrate have superior properties.

Considering these results, the main conclusion is that on the CoCr substrate, the TiN layer with a thickness of 1.5 µm deposited by the cathodic arc method, TiO_2 layer and CrN layer, both with a thickness of 1.5 µm deposited by DC sputtering method, have superior properties and are useful for continuing studies on real systems.

TiN deposited on M30NW steel substrate

- The layer with the lowest friction coefficient is the one with a thickness of 1.5 µm deposited by HiP sputtering;
- The layer with the lowest wear rate is the one with a thickness of 1.5 µm deposited by DC sputtering.

From a tribological point of view, the TiN layer with a thickness of 1.5 µm deposited on M30NW steel substrate by HiP sputtering, respectively DC sputtering has superior properties.

TiO_2 deposited on M30NW steel substrate

- The layer with the lowest friction coefficient is the one with a thickness of 1.5 µm deposited by DC sputtering;

- The layer with the lowest wear rate is the one with a thickness of 1.5 μm deposited by DC sputtering.
From a tribological point of view, the TiO_2 layer with a thickness of 1.5 μm deposited on M30NW steel substrate by the DC sputtering method has superior properties.

CrN deposited on M30NW steel substrate

- The layer with the lowest friction coefficient is the one with a thickness of 1.5 μm deposited by DC sputtering;
- The layer with the lowest wear rate is the one with a thickness of 1.5 μm deposited by DC sputtering, respectively the layer with a thickness of 1.5 μm deposited by HiP sputtering.

From a tribological point of view, CrN layers with a thickness of 1.5 μm deposited by DC sputtering, respectively HiP sputtering on M30NW steel substrate have superior properties.
Considering these results, the main conclusion is that on the M30NW steel substrate, the TiN layer, TiO_2 layer and CrN layer with a thickness of 1.5 μm deposited by DC sputtering method, have superior properties and are useful for continuing studies on real systems.

4. Conclusions

The tribological characterization of tree types of thin films deposited on two types of biocompatible substrates were realized in order to determine and analyse important parameters, like friction coefficient and wear rate. In this way it was possible to determine which is the most resistant substrate material – deposited material couple.
The 1.5 μm thick TiN layer − deposited on the CoCr and M30NW steel substrates – has displayed superior tribological parameters when using the cathodic arc deposition method. The superior quality of the TiO_2 layer deposited by DC sputtering on both types of substrates was demonstrated by the analyses performed. The 1.5 μm thick CrN layer exhibited superior tribological characteristics when deposited on both substrate types using DC sputtering.
The main conclusion of the study was that thin layers of TiN, TiO_2, and CrN, with a thickness of 1,5 μm, deposited on CoCr and M30NW substrates, had superior tribological characteristics. This means that a thicker layer will be more sustainable and can be used to improve the quality of biomedical components, like hip prostheses.

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