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MECHANICAL CHARACTERIZATION OF NANOSTRUCTURED THIN FILMS USED TO IMPORVE MECHATRONIC COMPONENTS

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Abstract. *Taking into account the importance of mechatronic applications, researches regarding the possibility to improve the lifetime of mechatronic components were made. Nanostructured metallic thin films (Ti, Cr, Al and Ti/Al multilayer) were deposited on different types of steel substrates, because nanomaterials have exceptional properties in relation to the common materials. In this paper a part of the results obtained after mechanical and topographic characterization of the thin films are presented. Cr is the deposited thin film showing the highest hardness on the surface of steel substrate type OSC. After the scratch tests realized, Ti layer presented the best adhesion on all types of steel substrates used in experiments. The results of these researches could be extremely useful for engineers in the mechatronic field.*

Keywords: *thin films, nanomaterials, wear, topographic characterization, mechatronics*

1. INTRODUCTION

Mechatronics, as science of mechanical-electronical-computer systems is the result of technological development of the recent decades. All high-tech products made today are mechatronic products: modern cars, industrial robots, microrobots used in military industry, nanorobots used in medical investigations, computers, printers, office equipment, equipment for medical investigations, prostheses and artificial organs, audio, video recording systems, etc [1].

The surfaces of these systems, made of the most suitable materials are subjected, in time, to some tribological processes. As a result, friction and wear appear – processes that manifest at micro- or nano-scale and are heavily dependent on surface interactions.

From tribological point of view, the wear is a process of progressive loss of material arising from the interaction of friction couplings surfaces. Between the wear and friction processes there is a close interdependence, meaning that the wear is a result of friction, and the state of surfaces resulting from friction influences the wear.

Wear is a mechanical process in that the strains associated with the destruction process of the surface may exceed the material strength and, thus, wear particles occur.

There are several wear mechanisms [2], which in some systems can be combined. These are: adhesive wear, abrasive wear [3], abrasive wear with the third body [4], superficial fatigue wear, corrosion wear and fretting wear.

Taking into account these processes that have important effects on mechatronic systems, and the importance of the lifetime of components and systems in this area, the project researches presented in this paper focuses on the use of materials for tribological improvement of mechatronic components (high precision gears in miniature constructions, high precision bearings, components of mechatronic equipment for measuring, positioning and adjustment: bearers, guides, grippers, etc.).

The materials commonly used in the mechatronic field must have certain properties: excellent resistance to degradation for minimizing the particles generation which can adversely influence the systems. The surfaces must be hard to keep the wear to a minimum value.

Currently pure metals, stainless steels, and metal alloys are used.

The need to have resistant components, with anticorrosive composition and improved mechanical properties have led to the application of thin films of materials with superior properties on different surfaces used in mechatronic applications. Nanomaterials have exceptional properties in relation to the common materials, for example: the tensile strength is 20-50 times greater than the stainless steel; Young's modulus is 5 times higher than the stainless steel [5].

The main objective of this project was the characterization of thin films surfaces with micro and

nanometric structures deposited by physical methods. Researches on materials were directed mainly to study the mechanical properties of their surface.
Studies and progress have been done by the use of very hard alloys and materials to keep the wear to a minimum value.

2. MATERIALS AND EXPERIMENTAL METHODS

2.1. Materials deposition

Thin films are deposited on mechatronic systems using different techniques.

Deposition methods must take into account the nature of phenomena that take place in the deposition process, diversity of thin films forming parameters (composition, structure, thickness, components spread in volume) and their application parameters (adhesion, wear resistance, corrosion resistance, porosity).

Therefore, under this project, studies have been conducted on thin films deposited by electrons beam evaporation.

Electrons beam evaporation is a technique that deposits metallic thin layers for the transposition of geometries from the mask in the substrate followed by lift-off.

Using the deposition process by electrons beam evaporation, nanostructured thin layers of Ti, Cr, Al and Ti/Al multilayer were deposited on four types of steel: OLC45, Rul1, C120 and OSC. OLC45 and Rul1 are used in mechatronics for the production of gauges, bushes, actuators, measuring dowels, opposite dowels, positioning stands, body of gauge. C120 and OSC are used in mechatronics for the production of screwed calibre and ledges, sensing heads, standards, punches. All these mechatronic components are subjected, over time, to wear [6].

Depositions were made with a Temescal FC-2000 system (Figure 1), a versatile evaporation system that supports a variety of accessories to meet almost any requirement. FC-2000 is a system with rapid cycle, blocked charging, which allows the source to remain in vacuum during recharging of the substrate.

Figure 1. System of deposition by electrons beam evaporation Temescal FC-2000 [7]

Standard components of this system are: control system, electrons beam source, power supply, vacuum pump and control, air system, water system, vacuum chamber.

Careful control of the electrons beam of Temescal offers completely digital operation, internal storage of up to 64 user-defined patterns and compatibility with almost any electrons beam gun commercially available.

2.2. Mechanical characterization of deposited thin films

Nanostructured thin films deposited for improving the resistance of mechatronic components in this project were physico-mechanically, structurally and topographically characterized.

Following these characterizations, information about the degree of influence of the material used were obtained, for a future enhancement of substrates resistance. Mechanical characterizations are important, highlighting important properties of deposited thin films, such as hardness and adhesion [8].

Determination of deposited thin films hardness
To determine the hardness of deposited thin films, the system for micro-hardness measurement HMV-2 was used, a system used within metallographic researches.

The automatic reading system for Vickers hardness tests (Figure 2) is designed to automatically measure the distance between opposite corners of the fingerprint to find the Vickers hardness based on the fingerprint image realized on the testing surface and captured by the CCD camera.

Figure 2. Automatic reading system for Vickers hardness tests

In the project, this system has been used to make measurements under the following conditions T = 24°C; H = 50%, F = 98.07mN, $HV_{0,01}$, 10 sec.

Determination of deposited thin films adhesion
By using the scratch tests it is possible to detect premature failure of the coatings adhesion in real applications. Scratching test method is a very reproducible quantitative technique, where the critical

loads at which errors occur are used to compare the cohesion or adhesion properties of the coatings or of the substrate material.

A CETR-UMT 2 system (Figure 3) was used to perform measurements and tests in order to determine the adhesion of thin films. The device allows two ways of sliding contacts: with alternative reciprocating or unidirectional sliding. It also allows to control the ambient temperature and humidity of air.

Figure 3. CETR-UMT 2 system

The device performs various types of tribological tests (pin-on-disk, ball-on-disc, block-on-ring, pin-on-ring and micro-indentation) having an interchangeable module, which can be mounted on a base structure.
This device has a modular design, with a basic module that contains the positioning system, the charging system, the driving system, the measurement system, the electronic system of control and data acquisition-processing. The device provides protection against vibrations.
The tests involved the execution of a measuring cycle in three steps, with a normal force of 5 N, a length of 5 mm, speed of 0.2 mm/s, having duration of 25 s.

2.3 Topographic characterization of thin films surfaces deposited and subjected to the scratch-tests

Topographic characterization of thin films deposited and subjected to scratch tests was carried out using atomic force microscopy (AFM). The working principle of the atomic force microscopy is the measurement of interaction force between a tip and the sample surface using special probes, made of a cantilever with a sharp tip at the end. The force applied on the tip by the surface leads to bending of the cantilever. Measuring the deflection of cantilever, it is possible to evaluate the interaction force tip – surface. Acquisition of AFM surface topography can be made by registering the small deviations of the elastic cantilever [9].

AFM measurements were performed using a microscope type NT-MDT NTEGRA Probe NanoLaboratory (Figure 4) [10]. This type of microscope has the following basic systems and modules: base unit, base modules (probes, exchange support, scanner, heating platform, fluid cells, etc.), protective hood, optic viewing system, vibration isolation system and control system (SPM controller, termocontroller, computer with interface panel).
Surfaces of 50/50 μm situated in different locations of samples were scanned and topographic parameters were determined using NOVA SPM Software – the software of NT–MDT NTEGRA Probe NanoLaboratory microscope.

Figure 4. AFM microscope, NT–MDT NTEGRA Probe NanoLaboratory. 1 – base unit; 2 – probe; 3 – vibrations isolation system; 4 – optical viewing system

Following this investigation, it was possible to identify more clearly the nature of the damage and to determine the roughness parameters of all characterized surfaces.

3. RESULTS

Ti, Cr, Al thin layers and Ti/Al multilayer were obtained following the deposition by electrons beam evaporation on four kinds of steel: OLC45, Rul1, C120 and OSC. These layers had thickness of 50 nm (Cr and Al), 100 nm (Ti), and Ti/Al multilayer with a thickness of 50 nm each layer (total multilayer thickness is 100 nm).

After scanning 50 × 50 μm areas of the different films deposited on all the substrates, different topographic parameters have been analyzed (surface roughness R_a, surface asymmetry R_{sk}, coefficient of kurtosis R_{ka}) which provide information on distribution and damage of the deposited layers.

The roughness was used as a deterioration indicator of the deposited layer to obtain information regarding the variation in height from one point to another. This is quantified by deviations of the real surface from its ideal form. If these deviations are large, the surface is rough; if the deviations are small, the surface is smooth.

Asymmetry index R_{sk} assesses the asymmetry degree of a distribution and characterizes, together with the coefficient of kurtosis R_{ka}, the shape of the distribution. The asymmetry index R_{sk} is negative or positive as the survey distribution is asymmetrical to the left or, respectively, to the right. A symmetrical distribution, such as the normal distribution, has the zero asymmetry.

The coefficient of kurtosis R_{ka} is part of indices that assess the form of a distribution. A high coefficient of kurtosis shows a distribution with great "tails" (categories far from average are presented), while a small coefficient of kurtosis shows an assignment where are presented less categories far from average. In the case of a distribution close to the normal distribution, the coefficient of kurtosis is around 3. Based on this result, the excess *E* is defined as the difference between the coefficient of kurtosis and 3. For $E > 0$, the distribution is called leptokurtic (the height of the curve is higher compared to the normal), and for $E < 0$, it is called platykurtic (the curve is flattened). If $E = 0$, the division is mesokurtic.

After analysing the roughness average values of deposited films it was seen that on the OSC steel substrate, films with the highest uniform surface are deposited. Titanium has the most uniform surface, and from the thin films deposited with thickness of 50 nm, aluminium is more uniformly deposited on this type of steel substrate. It can be concluded that layers more uniformly were obtained starting from chromium, aluminium and titanium. In the case of Ti/Al multilayer that has a more uniform arrangement on the C120 type substrate.

Taking into account that the average values of the asymmetry index, in the case of all three types of deposited layers, were very close to zero, it can be concluded that they have a symmetrical distribution. The only exception is the Ti/Al multilayer, which has a positive asymmetry index, bigger than 1, so it is a deposition with survey distribution asymmetrical to the left right.

For samples in this project, the excess values obtained after the analysis of coefficient of kurtosis indicate a platykurtic distribution of all thin films deposited on all four substrates types. The exception is in the case of Ti film of 100 nm thickness, the excess of which has a positive value, therefore a leptokurtic distribution.

3.1. Determination of deposited thin layers hardness

As a result of the hardness measurements made at a temperature of 24°C, humidity of 50%, with a force F = 98.07mN, $HV_{0,01}$ in 10 seconds were obtained the results shown in Table 1.

Table 1. Results obtained from measuring the hardness of nanostructured layers deposited on different types of steel

Layer	Substrate	M (The average value of hardness)
Ti	OLC45	91.5
	Rul1	79.0
	C120	86.9
	OSC	91.0
Cr	OLC45	94.8
	Rul1	94.3
	C120	94.4
	OSC	96.6
Al	OLC45	81.4
	Rul1	84.9
	C120	84.8
	OSC	84.4
Ti/Al	OLC45	83.9
	Rul1	81.3
	C120	84.3
	OSC	86.3

These values were analysed and hardness variations of the thin films deposited on a particular substrate type were obtained, variations which can be seen in the following figures.

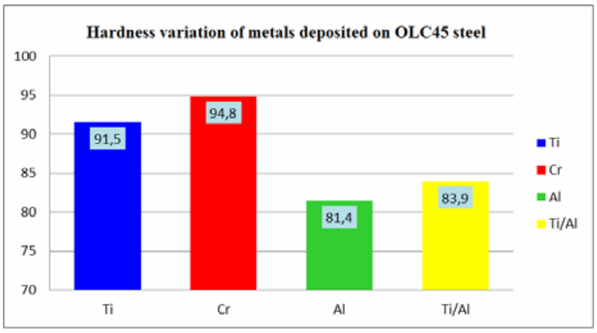

Figure 5. Variation of metallic layers hardness deposited on OLC45 steel

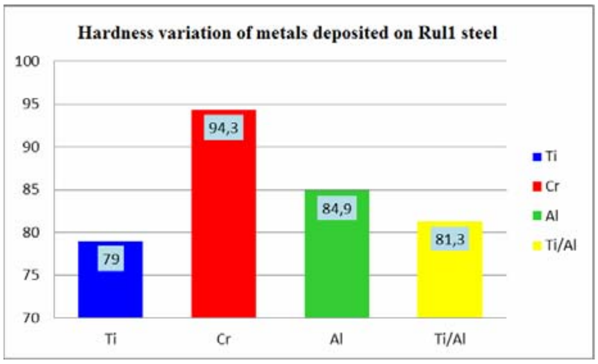

Figure 6. Variation of metallic layers hardness deposited on Rul1 steel

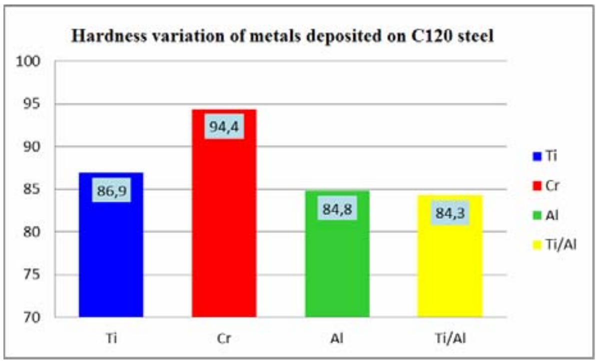

Figure 7. Variation of metallic layers hardness deposited on C120 steel

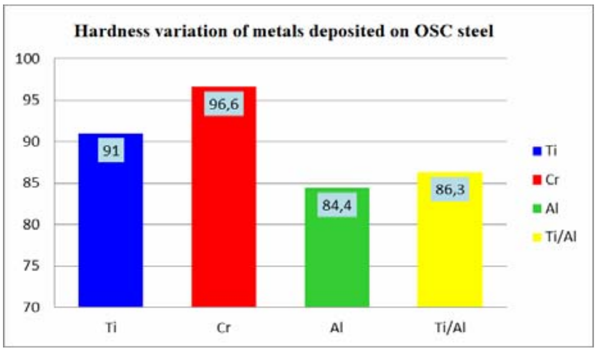

Figure 8. Variation of metallic layers hardness deposited on OSC steel

It is observed that on all the four types of substrate, Cr is the deposited thin film that has the highest hardness, having an average value greater than 90, in all four cases. Ti also has a high hardness, but when it is combined with Al, its hardness decreases given the fact that the top layer is Al, a metal with a lower hardness.

3.2. Determination of deposited thin films adherence by scratch tests

Adhesion of metal layers deposited on various steel substrates was determined by the scratch tests using CETR-UMT 2 system shown in Figure 3.

Tangential force F_x, the normal force F_z, movement in the normal direction Z, movement in the tangential direction Y, time T, friction force F_f (value in module of F_x), friction coefficient COF (ratio F_x/F_z in module) were used and determined.

The average values obtained from these tests are presented in table 2.

Table 2. The average values obtained from tests and measurements of adhesion.

Layer	Substrate	Time [s]	Depth [mm]	Force F_f [N]	COF
Al 50nm	OLC45	3.729	0.016	3.497	0.727
	Rul1	3.933	0.01	3.229	0.664
	C120	2.5	0.009	2.972	0.605
	OSC	4.430	0.02	6.272	1.267
Cr 50nm	OLC45	2.612	0.018	4.876	0.991
	Rul1	1.606	0.01	3.891	0.802
	C120	2.012	0.013	3.557	0.719
	OSC	2.377	0.021	3.461	0.717
Ti 100nm	OLC45	1.931	0.01	3.915	0.81
	Rul1	1.960	0.01	3.139	0.639
	C120	2.052	0.012	4.16	0.846
	OSC	1.656	0.01	4.482	0.91
Ti 50nm + Al 50nm	OLC45	2.053	0.013	3.658	0.739
	Rul1	2.012	0.012	3.199	0.647
	C120	1.859	0.01	3.503	0.702
	OSC	2.082	0.017	3.354	0.701

Analysis of these results leads to the following conclusions:

- The time shown in the third column indicates the moment of separation of the deposited superficial layer.
- At this point there is a significant increase in the depth of penetration of the testing device.
- The values of the friction force F_f and the sliding friction coefficient COF are appropriate to the produced phenomenon.

Friction coefficient COF values are decreasing in the order of Al, Cr, Ti, Ti/Al. These values are generated and by adherent structure of the deposited material, and by the peeling strength of the hardened surface layer.

Regarding the moment of separation of the deposited surface layer, Al detaches the fastest from the C120 substrate, Cr from the Rul1 substrate, Ti from OSC substrate, and the Ti/Al multilayer detaches the fastest from C120 substrate.

If are taken into account, mainly, the values of detaching times on the four substrates types it can be see that Al shows the latest moment of separation on all four steels, which may indicate a good adhesion between it and variants of steel.

3.3. Topographic characterization of thin films surfaces deposited and submitted to the scratch tests

After the scratch tests on each sample and examining the scratch traces with the optical microscope, several types of surfaces damages resulted from critical loads were detected: deformations and detachments of material, detached fragments of deposited layers, semicircular cracks of the coating, raising of the material on the edges, cutting of the base material and oxidation.

There are also areas with uniform surfaces, almost without defects.

Identifying more clearly the nature of damages and topographic parameters setting of all the investigated surfaces was performed by atomic force microscopy.

2D images, 3D images, calculated topographic parameters (Figures 9 – 12) and profiles on X and Y axes of these surfaces were obtained.

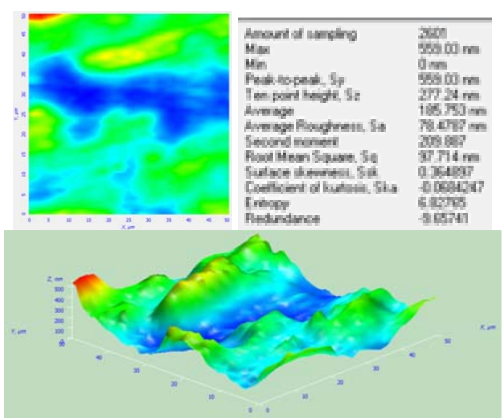

Figure 9. AFM surface characterization of Cr thin films deposited on substrate type OLC45 and submitted to scratch tests

Following these measurements, the values of roughness obtained indicates a damage of the deposited films surfaces. It is about the surfaces where scratches, particles that came out of the surface or cracks were observed. However, taking into account that the averages values of roughness are of the nm order it can be considered that there was not a very high destruction of those surfaces. This allows considering it almost smooth surfaces. Ti layer deposited on the all four types of steel shows the lowest value of the roughness among all the layers deposited. The fact that the scratch tests had not greatly damaged the surface can be interpreted as good adhesion of Ti layer on all types of steel substrates used in experiments.

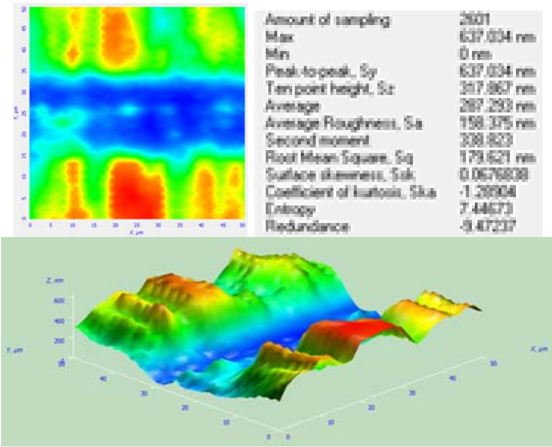

Figure 10. AFM surface characterization of Ti thin films deposited on substrate type Rul1 and submitted to scratch tests

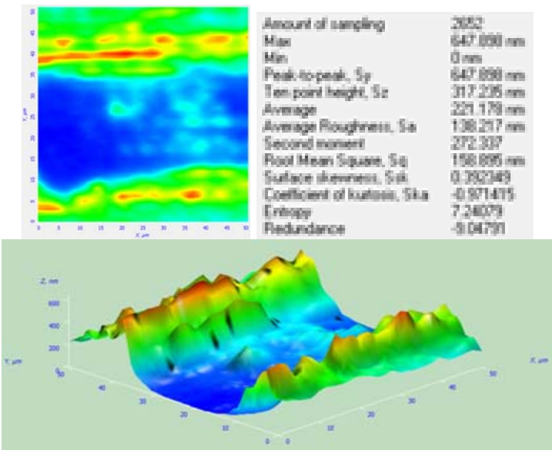

Fig.11. AFM surface characterization of Al thin films deposited on substrate type C120 and submitted to scratch tests

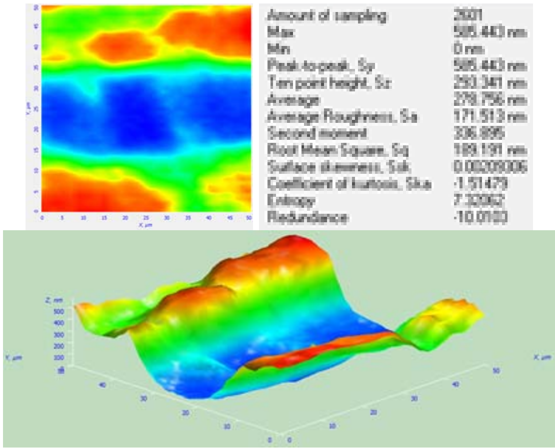

Figure 12. AFM surface characterization of Ti+Al thin films deposited on substrate type OSC and submitted to scratch tests

Asymmetry index R_{sk}, for all the layers deposited, has a positive average value indicating a survey asymmetric distribution to the right. If one takes into account that for some layers the values of this index are close to zero (Al deposited on Rul1=0.3 and Cr deposited on C120=0.25), these could be considered normal distributions.

Coefficient of kurtosis R_{ka} shows for all deposited layers values that lead to a negative excess, indicating a platykurtic distribution. For Ti layer deposited on the OSC substrate, the excess has a value close to zero (0.56), giving the opportunity to be considered as mesokurtic distribution. Also, the Ti layer deposited on the OSC type substrate has a value close to 3 (ie 2.44), which indicates the possibility of considering a normal distribution.

4. CONCLUSIONS

After the study performed the importance of such deposited materials was highlighted for the mechatronic domain:

- tests and measurements have been made for physico-mechanical and topographic characterizations of nanostructured Al, Cr, Ti thin films and of Ti/Al multilayer deposited by the method of electrons beam evaporation on steel substrates type OLC54, Rul1, C120, OSC;
- it was observed that on the all four substrates, Cr is the deposited thin film showing the highest hardness. Ti also has a high hardness, but when it is combined with Al, its hardness decreases, given that the top layer is Al, a metal with lower hardness.
- the highest average hardness for all four types of nanometric layers was obtained on the surface of OSC substrate;
- Al detaches the fastest from the C120 substrate, Cr from the Rul1 substrate, Ti from OSC substrate, and the Ti/Al multilayer detaches the fastest from C120 substrate;
- for the topographic characterization, the values of some tribological parameters, such as roughness,

asymmetry index and coefficient of kurtosis, were analysed;
- taking into account that the average values of roughness are of the nm order it was considered that there was not a very high destruction of deposited layers.
- the fact that the scratch tests had not greatly damaged the surface can be interpreted as good adhesion of Ti layer on all types of steel substrates used in experiments;
- the positive value of the asymmetry index for all layers deposited indicates a survey distribution asymmetric to the right;
- coefficient of kurtosis shows for all layers deposited values that lead to a negative excess, indicating a platykurtic distribution.

The results of these researches could be useful for engineers in the mechatronic field, who are studying the functioning conditions of mechanical parts components and not only, establishing the functional role of each component in the whole assembly and choosing materials that correspond to their proper operation.

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