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Adhesion of thin layers testing by the inclined cyclic impact

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Abstract

In this paper, the authors try to evaluated some contact mechanism characteristics at the interface between a cylindrical indentor with spherical head that exercise repeated inclined impacts at an angle of 30^0 over some thin layers from TiN. The coatings with thin layers were obtained by laser pulses deposition process and had different thickness, depending on the number of pulses al that were made the coatings. Were used two impact testers, one at low frequency and other at high frequency. It was analyzed the evolution of the failure process of coatings, as well as the appearance and development of critical events. By chance, after a cyclic inclined impact test at 30^0 over 10^4 cycles, it has produced a long imprint of wear, very similar to that obtained by another author, and declared as resulting as a consequence of an "inclined cyclic impact test with sliding".

Keywords: cyclic impact, inclined impact test, SS316L, TiN, thin coating failure, inclined impact with sliding.

1. INTRODUCTION

Pioneers of instrumented indentation tecnique, W. C. Oliver and G.M. Pharr [1] who introduced in 1992 the measuring of hardness and elastic modulus by instrumented indentation technique that was adopted and used widely in mechanical behavior characterization of materials at the small scales. Also, K.-D. Bouzakis et al [2] have examined the stresses - deformation laws of treated substrates were studied by means of a finite element methods (FEM) based on evaluation of the coating nano - indentation and adhesion results through the inclined impact test. The continuous improvement of technologies of PVD (physical vapor deposition), chemical or mechanical treatments of cemented carbide substrates, such as microblasting, have contributed to the significant growth of the cutting performance [3, 4].
Studying the effect of mechanical treatment on the topography of cemented carbide inserts and resistance properties of the surface, was polished micro - sandblasting substrates at different process pressures and roughness data, for two lengths of cutting. To count the stress - deformation laws and properties of hardness in the exposed layers of the material, the measurements were made of nano-hardness at different indent loads, and the corresponding results were evaluated using the methods described in [5, 6].
In the nano - indentation procedures, because deviations of the indentor tip shape from its ideal geometry and indentation depth limited, the contact region between indentor and sample cannot be determined accurately [7]. Thus, the contact area between the indentor and sample during the phases of loading and downloading of nano - indentation are defined by calculations based on FEM, thus allowing the determination of hardness from different methods. Methods used until now to assess the results of the nano-indentation to determine the hardness values are based on empirical equations describing the course of loading of indentation versus indentation depth during nano - indentation stages of loading [8] and unloading [9-12]. These methodologies involve among other things, that the material under examination provides uniform mechanical properties compared to the depth of the indentation, which excluded the determination of hardness of thin films, due to the effects of the substrate, or materials with surface classified mechanical properties. The nano - deviations due to the limits of the accuracy of indentor manufacturing [10, 13-14] are difficult to accurately recorded. Even through the observations of atomic force microscopy (AFM) of the tip, real geometry cannot accurately be mathematically described and is treated as a spherical surface [15-16].
The perpendicular impact test was documented as a effective and convenient methodological tool for the caracterisation of the fatigue properties of coatings. In addition, by this test can be investigated the cohesive and adhesive failure models of PVD coatings [17-20]. Adhesion strenght at interface coating - substrate was described in [20], through a simulation based on FEM, using the corresponding contact elements. According to these calculations and experimental results, in the case of a weak film-substrate adhesion, position of the equivalent maximum stress and start of the failure superficial film was displaced from his neighbourhood to imprint center. However, the critical fatigue load, i.e. the maximum load which avoid a fracturing of the coating after 1 million impact, remained virtually unchanged.To calculate the stress distribution that occurs in the loaded region of the film and the substrate, is developed a proper

method of simulation with finite element (FEM), which takes into consideration the elasto-plastic mechanical properties of all the materials involved.
In this paper we report about the testing in inclined cyclica normal l impact or, with high frequency and the results obtained on two thin coatings deposited on SS316 stainless steel, medical grade. The authors try to evalued some contact mechanism characteristics at the interface between a cylindrical indentor with spherical head that exercise repeated inclined at an angle of 30^0 and over some thin layers from TiN.

2. METHODS

To characterize the cohesion and adhesion strength of coatings, were made many tests of normal impact and inclined impact with a cylindrical pin with spherical head. Impact test is a more accurate simulation of a real situations that affect the life of the material. There are two common test procedures at multiple impact, namely the cyclic impact testing of low frequency and impact testing of high frequency. In this paper we report about a new setup for the cyclic impact testing, normal or inclined, with high frequency and the results obtained on three thin coatings of TiN deposited on stainless steel SS 316L, medical degree.
Samples used as substrates for thin films deposition of TiN were 316L stainless steel discs with a diameter of 22.5 mm, height of 10 mm and hardness of 464 HV30. The hardness measured values of 316L stainless steel samples before coating and layers of TiN micro-hardness were:
- Sample TIN/316L stainless steel; 5000 pls: Substrate material hardness: 277; 299; 286 *HV* 5 gf, HV_5; Average hardness: 287 HV_5; Coating micro-hardness: 352; 345 *HV* 50 gf; Average micro-hardness: 348.5 $HV_{0.5}$.
- Sample TIN/316L stainless steel; 10000 pls: Substrate material hardness: 407; 429; 423 *HV* 5 kgf, HV_5; Average hardness: 420 HV_5; Coating micro-hardness: 532; 545 *HV* 50 gf; Average micro-hardness: 539 $HV_{0.5}$.
- Sample TIN/316L stainless steel; 20000 pls: Substrate material hardness: 453; 473; 423 *HV* 5 kgf, HV_5; Average hardness: 420 HV_5; Coating micro-hardness: 748; 727 *HV* 50 gf; Average micro-hardness: 737.5 $HV_{0.5}$.
It was noticed the increase in surface hardness of TiN coating deposited depending on its thickness. For the determination of thickness of thin layers of TiN deposited were made ground slides of SS316L steel samples on were deposited layers. To characterize better the cohesion and adhesion strength of coatings, were made and tests of normal impact and inclined impact with a cylindrical pin with spherical tip. Impact test is a more accurate simulation of a real situations that affect the life of the material. There are two common test procedures at multiple impact, namely the cyclic impact testing of low frequency and impact testing of high frequency.
In this paper, the authors try to evalued some contact mechanism characteristics at the interface between a cylindrical indentor with spherical head that exercise repeated inclined at an angle of 30^0 and over some thin layers from TiN deposited on stainless steel SS 316L, medical degree.
In the case of the normal impact, where a target suffering from plastic straining, the largest part of the initial kinetic energy W_1 is dissipated in target, as mechanical work, W_p, with small amounts recovered by the elastic forces, at the kinetic energy of indenter W_2. In addition, a possible source of this energy loss seems to lie in the dissipation of energy into the test piece, in the form of elastic vibrations W_v caused by transient nature of collision. An expression of this energy can be written as follows:

$$W_1 = W_2 + W_v + W_p \tag{1}$$

W_v estimatation was derived theoretically by Hutchings, assuming that contact pressure that acts on the contact area is constant and then:

$$W_v = \frac{\beta(1+\nu_d)}{\rho_d C_d^3} \cdot \left(\frac{1-\nu_d^2}{1-2\nu_d}\right)^{1/2} F_z^2 \omega_d \alpha \tag{2}$$

where:

$$C_d = \left(\frac{E_d}{\rho_d}\right)^{1/2};\ \omega_d = \frac{2\omega}{1+e};\ e = \left(\frac{W_2}{W_1}\right)^{1/2} \text{ and } \omega = \frac{\pi}{2t}, \tag{3}$$

where ν_d is Poisson's ratio for substrate; ρ_d and E_d are the density and Young's modulus. F_z is the maximum load of normal impact, *e* is the coefficient of restitution, and *t* is the loading time obtained from the relation of the curve

between load and time. β is a dimensionless amount which depends only on the Poisson's ratio. For $\nu = 0.25$, $\beta = 0.537$ and for $\nu = 0$, $\beta = 0.639$.
The scheme of the inclined impact exercised by a cylindrical pin made of Cr carbide, with spherical head of 3 mm diameter is presented elsevhere [21]. Normal force initially produces an elastic deformation of the coating and of the base material, which respond as a whole.
To study the evolution of coating failure, have been applied an impact force (F_i) of 40 N, resulting in a compression component F_c ($F_c = F_i \cdot \cos 30^0 = 40$ N $\cdot$ 0.866 = 34.64 N) and a tangent component F_t ($F_t = F_c \cdot \text{tg } 30^0 = 40$ N $\cdot$ 0.5774 = 23.1 N), at 5 Hz and at three different impact cycles (200, 400 and 600). Appeared tangential force, (F_t) exerts an additional important stress on the ball-coating contact. The tangential force increases, as the angle of inclination is larger (figure 1).

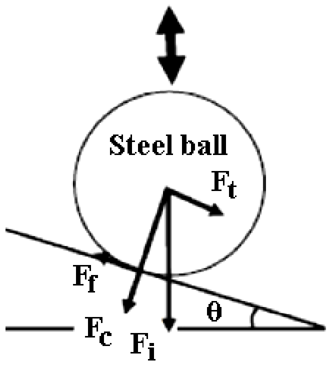
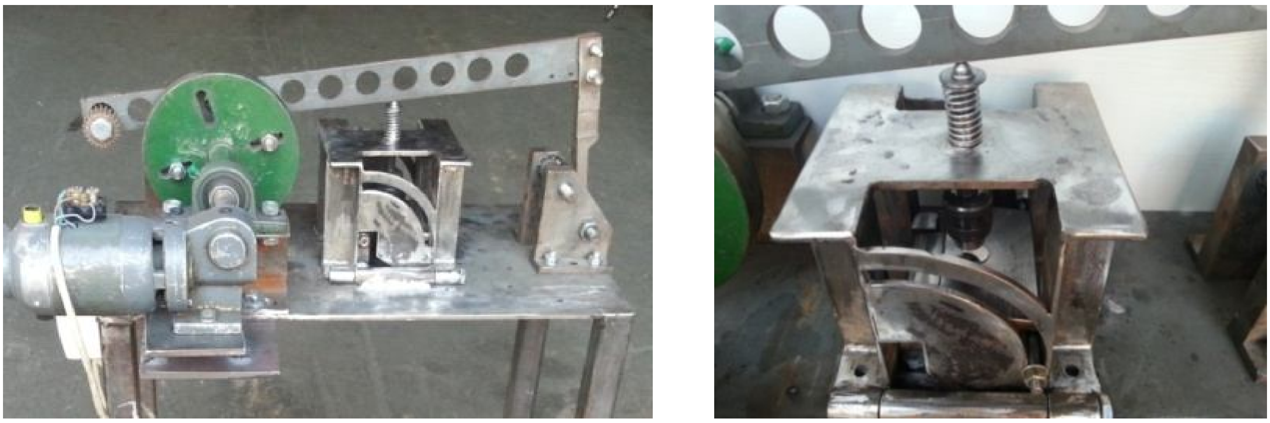


Figure 1 The forces that act upon the inclined cyclic impact plane

Increasing tangential force means the increase of the shear stress in the coating layer, what can lead to a situation in which this overcomes the adhesion force between the coating and substrate. In this situation, dislocation of the coating will take place, initially by agglomeration of its material in front of the contact mark, and then by piercing the coating and its removal by peeling.
Figures 2 shows two images of this impact tester with mechanical driving (a), where the impact is applied to the sample with a Cr carbide pin with spherical head, clamped in a chucking (b).



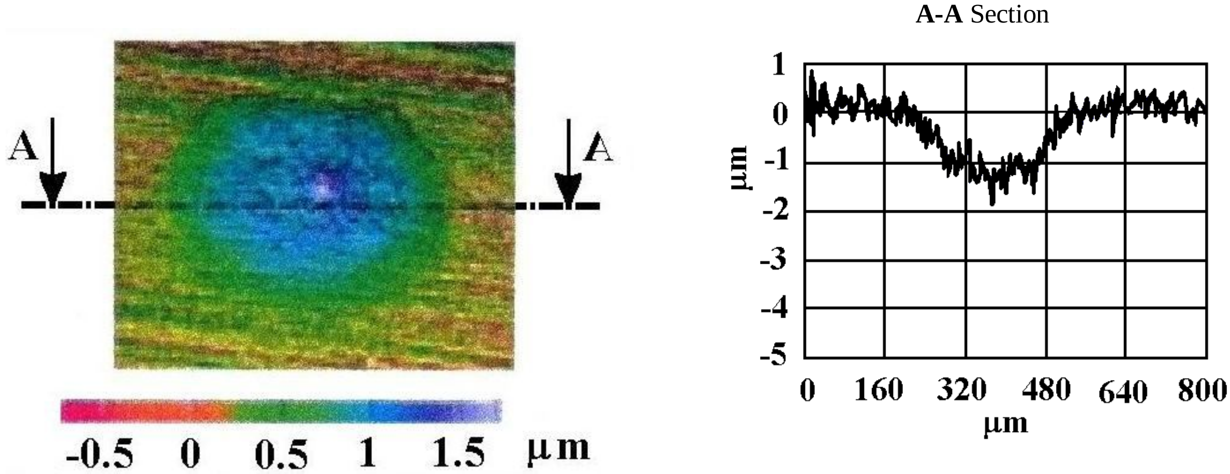
(a) (b)

Figure 2 Images of the impact tester with mechanical driving (a), where the impact is applied to the sample with a Cr carbide ball, clamped in a chucking (b)

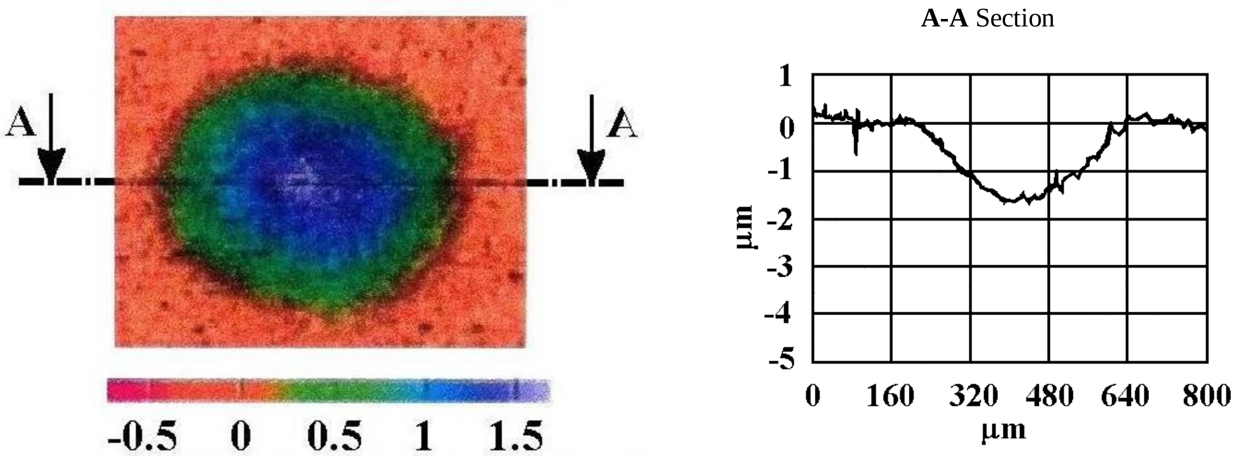
For the impacts at very high frequency it was built another tester, that in principle consists in a $X - Y$ table where a sample of different materials is fixed. The sample will be hit (impacted) by a large number of times, but always monitored by a cylindrical impactor, with spherical head, extremely hard, mechanically actuated by knocking with a hammer, presented elsevhere [19].

3. RESULTS

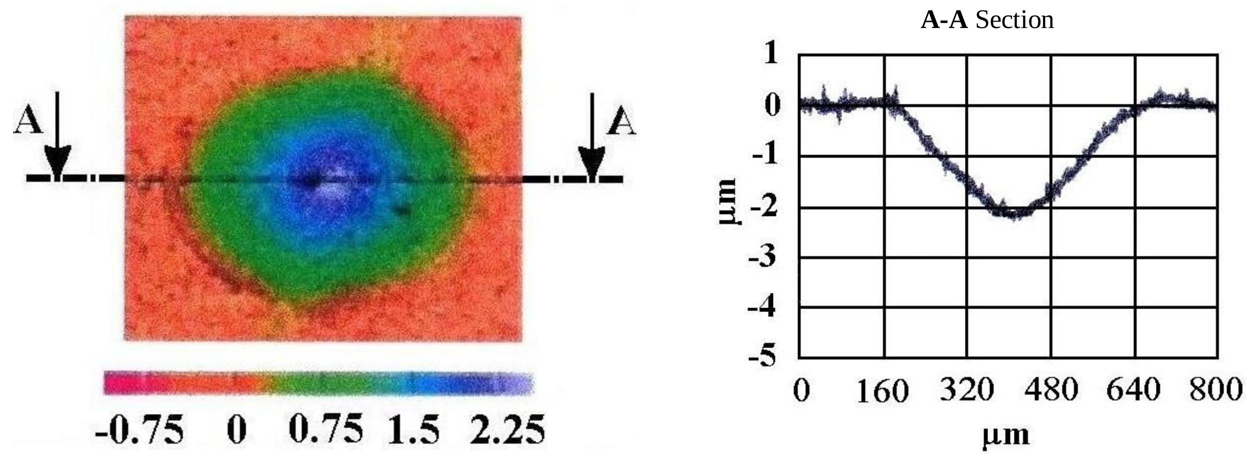
Figures 3 show the depths of some remaining craters on TiN / SS316L determined by measurements of confocal 3D microscopy (left) and profilometry (right), depending on the duration of the impact.



10^4 impacts at 1400 N impact force; impact duration: 0.3 ms; scar depth: ≈ 1.4 µm



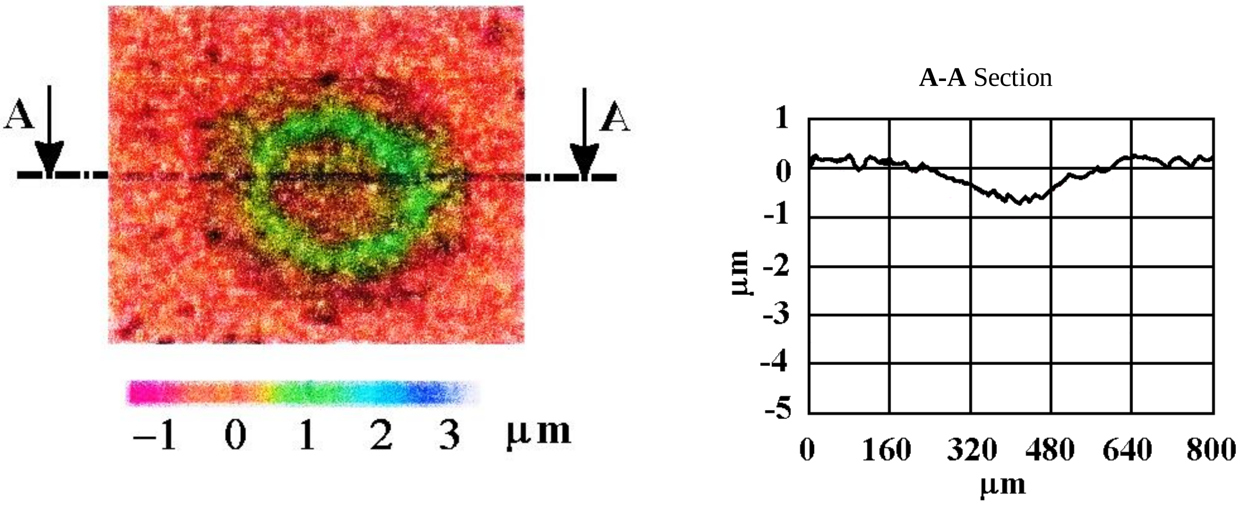
10^4 impacts at 1400 N impact force; impact duration: 5 ms; scar depth: ≈ 1.75 µm



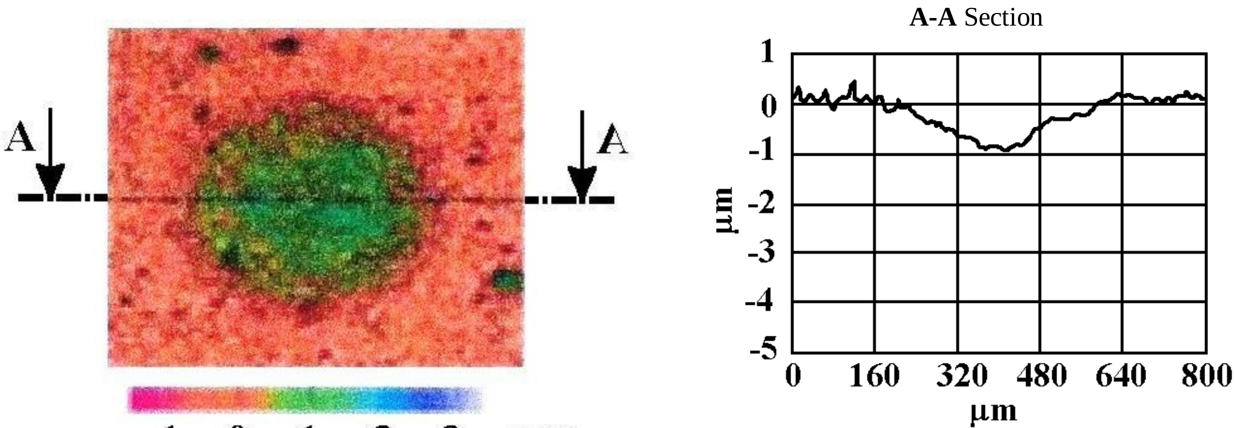
10^4 impacts at 1400 N impact force; impact duration: 10 ms; scar depth: ≈ 2.1 µm

Figure 3 Depths of remaining scars on TiN / SS316L at 1400 N. determined by measurements of confocal 3D microscopy (left) and profilometry (right), depending on the duration of the impact

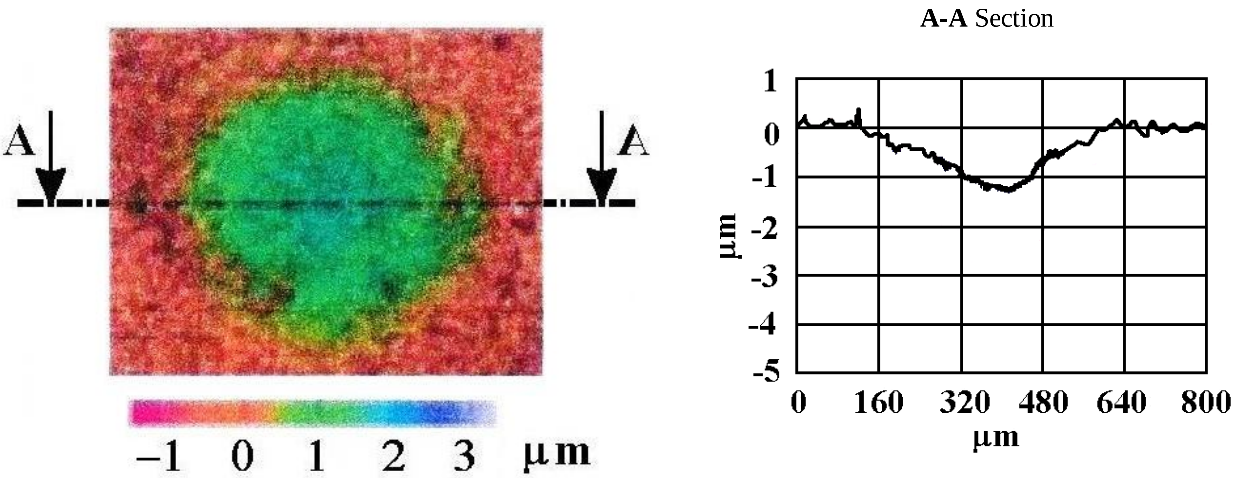
In figure 4 are shown depths of remaining scars on TiN/SS316L determined by 3D confocal microscopy (left) and profilometry (right), at 10^4 impacts of 0.3 ms, depending on the size of the impact force.



10^4 impacts at 800 N impact force; impact duration: 0.3 ms; scar depth: ≈ 0.7 μm



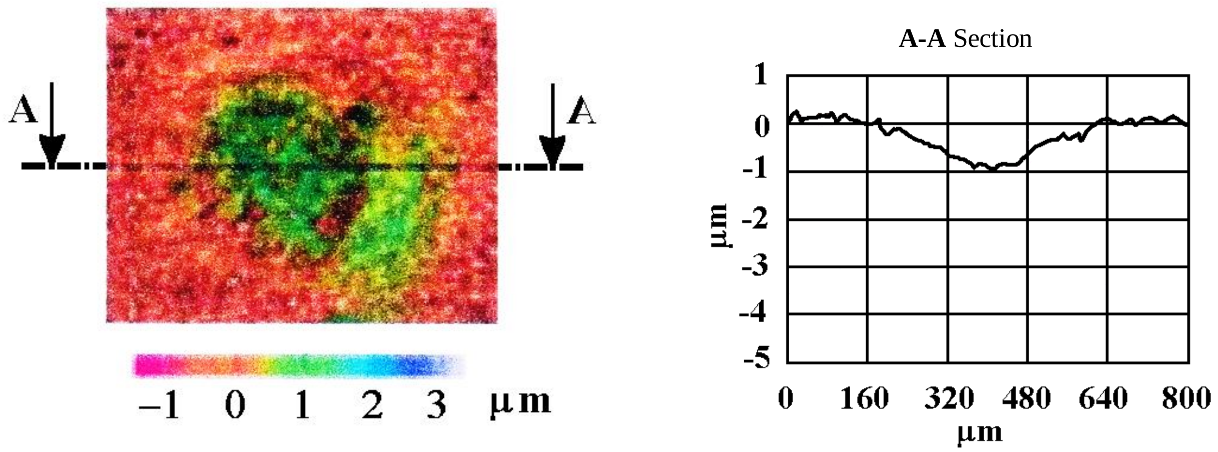
10^4 impacts at 900 N impact force; impact duration: 0.3 ms; scar depth: ≈ 0.85 μm



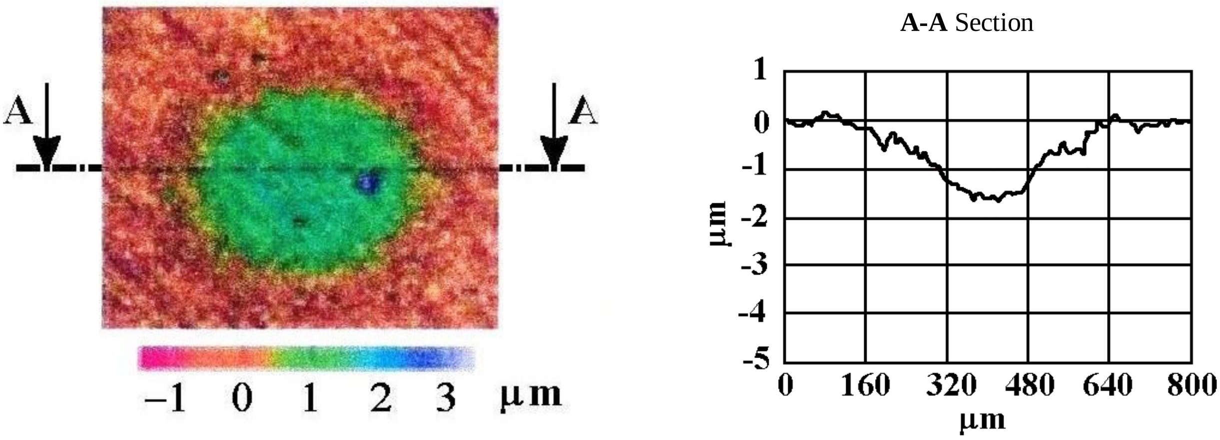
10^4 impacts at 1000 N impact force; impact duration: 0.3 ms; scar depth: ≈ 1.2 μm

Figure 4 Depths of remaining scars on TiN/SS316L at 800 N, determined by measurements of 3D confocal microscopy (left) and profilometry (right), at 10^4 impacts of 0.3 ms, depending on the size of the impact force

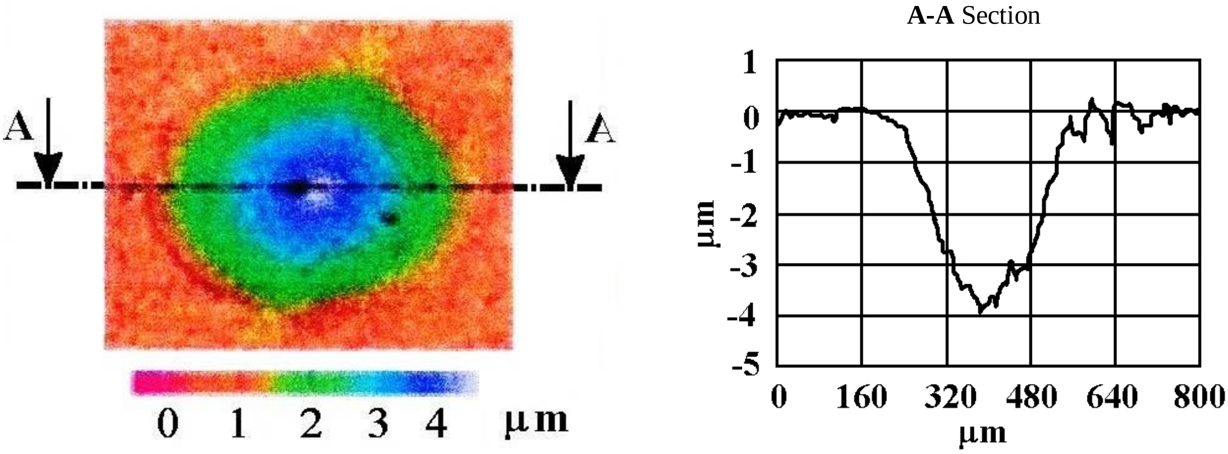
In figure 5 are shown the depths of remaining scars on TiN/SS316L determined by 3D confocal microscopy (left) and profilometry (right), at 10^6 impacts of 0.3 ms, depending on the size of the impact force.



10^6 impacts at 800 N impact force; impact duration: 0.3 ms; scar depth: ≈ 0.7 µm



10^6 impacts at 900 N impact force; impact duration: 0.3 ms; scar depth: ≈ 0.85 µm



10^6 impacts at 1000 N impact force; impact duration: 0.3 ms; scar depth: ≈ 1.2 µm

Figure 5 Depths of remaining scars on TiN/SS316L determined by measurements of 3D confocal microscopy (left) and profilometry (right), at 10^6 impacts of 0.3 ms, depending on the size of the impact force

Primary data collected included measurements of volume and depth and radius ratio of the residual impact crater, h_r depth and residual radius of coating impact crater were measured directly from a cross topography image. Cross image, parallel to the *y* axis was taken into the impact crater center. To calculate the residual impact crater, have used used primary data of the residual imprint impact profile.

In this study was analyzed and a new method of testing reported in [20], the “cyclic inclined impact test with sliding”, to investigate the failure behaviour of various types of biomaterials, (SS316L, Ti6Al4V and CoCr) with different coatings (TiN and CoCr), in terms of extremely high sliding contact dynamic stresses. Figure 1 shows the forces that act upon the sample surface during cyclic impact with an inclination angle of 30^0 front of the sample surface. To study the evolution of coating failure, have been applied an impact force (F_i) of 40 N, resulting in a compression component F_c ($F_c = F_i \cdot \cos 30^0 = 40\ N \cdot 0.866 = 34.64\ N$) and a tangent component F_t ($F_t = F_c \cdot tg\ 30^0 = 40\ N \cdot 0.5774 = 23.1\ N$), at 5 Hz and at three different impact cycles (200, 400 and 600). Figure 6 shows the registration curve of cyclic impact with sliding force depending on the time for one cycle, as in [20].

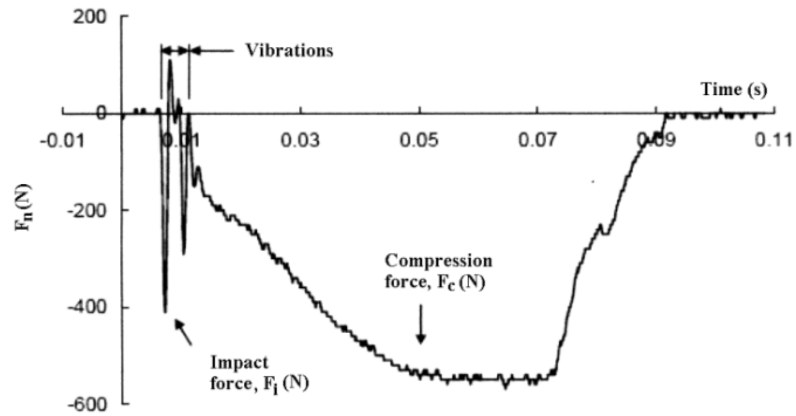


Figure 6 Time dependence of the impact force with sliding curve, for a cycle F_i / F_c = 40 N/34.64 N

The loading curve (figure 6) shows that there are three stages in each inclined impact cycle, i.e., that is the stage of the impact loading, vibrating stage and stage of static loading. After the stage of vibrations, the load continues to increase gradually up to the pre-configured load of compressing (F_c), and then the indenter began to move upward.

It was called the compression stage, in which was formed a long trace (tail) with sliding failures. It was called the pressing stage, in which was formed a long trace (tail) with sliding failures. When the indentor hits the surface of the test sample for the first time, it produces the first peak load, which is the effective impact load, F_i. Once the spheical indentor has completed his first complete contact with coating surface and formed a impact deep impact crater, the indenter had a recoil and again impact the sample. In figure 6, you can see a series of vibratory signals in the impact curve depending on the time in which each process of every of recoil process is a impact process with lowering impact energy between the two contact surfaces. Since there was no compression was imposed, have formed several impact craters (the imprint head) during the vibrrating stage, as a result of low rigidity of the indenter - sample ansamble. It should be noted that the vibrations and recoil may be reduced or eliminated by the application of a pre - compression of system impactor - sample. Figure 7 shows the appearance of the impact and sliding imprint and the distribution of fatigue cracks on the coated surface, after 10000 impact cycles on the SS316L sample coated by the PLD procedure, at 10000 pls. Cross section diagram of the impact scar in the longitudinal direction, is illustrated in figure 7 [19].

Figure 7 Microfotography of cyclic impact with sliding scar after 10000 inclined impacts at 30^0

According to the loading curve in figure 6, there are two peaks of signal monitored in vibrating stage, which means that there are two distinct impacts, before to be sliding wear. This is due to lack of the imposed stiffness of the ansamble impactor - sample, so that after the initial impact, the indenter continue to contact the sample. In this case, after the first full contact between the two surfaces has been completed, the sample has been pushed down and lost the contact with the spherical indentor. However, along with the continuing downward movement of the indenter, formed a second impact crater with a smaller diameter compared to the first, due to the decreasing impact energy. The longitudinal section scheme of the inclined cyclic impact at 30^0 imprint, adapted from [21], is presented in figure 8.

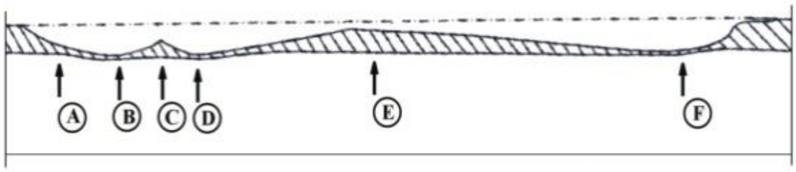


Figure 8 The longitudinal section scheme of the inclined cyclic impact at 30^0 imprint, that is presented in figure 5, adapted from [20]

Due to of the tilted angle of the sample, area where is exerted the greatest impact force was not the focus in the center of first impact crater, but it has gone slightly towards the head of the central area (point A). The impact force to the impact test, is analyzed through the normal component of the F_c and tangent componennt F_t, with their absolute values, according to the angle of inclination θ (figure 1). The normal component F_n of the impact force F_i, caused the bending stress on the edge of the impact - sliding scar, where they were seen in a series of severe cracks (point A). In the area located behind the center of the first impact crater (point B), could be seen cohesive failures with a network of cracks due to elastic deformation of spherical indenter and combined results of the static friction force and normal component F_n of the impact force. Between the two impact craters, there is a ridge point C which delineates an area with fewer failures compared to adjacent areas B and D, because it has a lower bending deformation due to the "shots" of indenter. From figure 8, it may be noted that there are a number of cracks formed in the D - E zone, which intersect with each other. Suppose that they were formed by bi-directional bending deformations in the overlapping impact craters zone.
There is no bending deformation in the initial part of the tail (point E in figure 8). However, once the downward-moving of the spherical indenter, the normal component has kept growing following the load curve in the compression loading stage, until it reaches its maximum value (the point F in figure 8) that is equal to the maximum compressive load, F_c (figure 8).

4. CONCLUSIONS

It is very clear that the impact test provides testing of coatings with thin layers as the base materials (substrates) at very high mechanical stresses, like those of the orthopaedic endoprostheses, and in the case of tools for metal cutting, machines in mining or construction. The impact test ensures the accurate simulation of the cutting edge loading when cutting a difficult to machine material and of shocks patterns acting on the surface of the tool due to interrupted cutting (e.g. milling), or in the case of crushing mills and machines. This test also allows to determine the moment of film failure and its delamination when the substrate is exposed due to dynamic contact, which is very important, especially for cutting tools. At film delamination, the uncovered substrate of the tool is not able to withstand at adverse conditions of the cutting, and the cutting edge is immediately destroyed.
Impact wear mechanisms of the hard metal coatings constitute a current concern regarding the fracturing process of the coating due to cracks propagation, under severe wear conditions. However, research reports do not discuss how the impact wear mechanisms of the coating act in light wear conditions. In terms of impact, it is well known that plastic deformation should appear before the impact wear.
Impact with sliding stresses is an incidentally case, due to some situations in which the necessary rigidity of the indenter – sample assembly is not provided. It cannot be considered an evaluation test, but rather an unsuccessful test of cyclic impact inclined at high angle. In the case presented in this paper, the indenter with spherical head had a 3 mm diameter and great length, of 30 mm, due to construction problems. In the case presented, the impact on the SS316L sample coated with TiN after 10000 impacts inclined at 30° caused that elongated trace shown in figure 7, due to lack of rigidity of the indenter – sample assembly that allowed the impactor recoil after the initial shot.

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Virgil Florescu: He graduated from the Polytechnic Institute of Bucharest, is Ph.d. in engineering sciences. He has worked in academic scientific research and currently is Director of the Mechanical Engineering Department of the Institute of Civil Engineering from Bucharest. He has published over 200 scientific papers, is author of 4 monographs and has participated in many international conferences in the field of "tribology".