DLC FILMS ADHESION ON AISI 1010 STEEL CYLINDRICAL METALLIC SURFACES

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Abstract. *The DLC (diamond like amorphous carbon) films adhesion on AISI 1010 steel cylindrical metallic surfaces was studied. Annealed 10 mm diameter x 14 mm length steel samples were coated with PECVD (Plasma Enhanced Chemical Vapor Deposition) technique. AISI 1010 steel was chosen to perform the present work since it is characteristically ductile and presents low hardness, a consequence of its ferritic structure. Samples were characterized by optical and scanning electron microscopy, scratch test comprising variable and constant load, penetration adhesion testing, microhardness and nanoindentation measurements. Results indicate the DLC films adhesion on the cylindrical metallic surfaces are strongly dependent of the mechanical characteristics of the studied steel.*

Keywords*: DLC, PECVD, Scratch test, AISI 1010 steel*

1. INTRODUCTION

The study of obtainment of DLC (diamond like amorphous carbon) films on steel substrates and the related mechanical behaviour for different applications in the mechanical engineering field has demanded special attention of researchers aiming to optimize the surface characteristics and properties of the treated components (Morshed et al., 2003; Beake et al., 2002; Huang et al., 2002; Jan and Ai, 2001; Chang and Wang, 2001; Sheeja et al., 2001; Lee et al., 2000; Bhushan and Dandavate, 2000; Cellier and Nowak, 1994; Meletis et al., 1995; Podgrnik et al., 2003).

The process of surface deposition involves several variables such as time, temperature exposure, pressure, gas mixtures and the control of these variables is critical to determining the structure of the layer deposited. It should be emphasized that the characteristics and properties of the mechanical component treated superficially are, in general, strongly dependent on the material of the substrate utilized and it is usually a function of response as a whole, that is, from the interaction between the deposited film and the base material coated. Over the last years, thin films of amorphous carbon (aC) and hydrogenated amorphous carbon (aC: H), known as DLC, have been extensively studied because they have properties similar to those of diamond, depsite they have low concentrations of chemical bonds of the sp_3 type, such as high hardness, wear resistance, chemical inertness, and good thermal and optical properties (Michél, 2005). Table 1 presents some typical properties for a-C: H or DLC films.

Table 1. Typical properties for a-C: H or DLC films (Michél, 2005).

Coating material	a-C:H
Micro-hardness (HV 0.05)	>2000
Coefficient of friction against steel (dry)	0,1 – 0,2
Maximum service temperature (° C)	350
Coating color	black

The DLC is suitable for the toughest conditions of wear and with high relative speed, even capable of running dry. The coating provides excellent protection against oxidation, abrasion and adhesion by friction. The coating allows surface pressures that, under regular conditions, it would cause, immediately, the deformation and cold welding. Friction losses are minimized. The good corrosion resistance protects the substrate from damaging attacks. Moreover, the DLC does not attack the food (Michel, 2006). DLC coatings have thicknesses typically on the order of micrometer and are harder than steel. Exhibit high wear resistance and high chemical stability, which ensures obtaining chemically inert surfaces.

As functional aspects of amorphous carbon coatings, one should note the following characteristics (Balzers, 2007):

- low coefficient of friction (adhesion and sliding friction) of 0.05;
- for run dry, as well as in the case of mixed friction, the friction coefficient is reduced to about 0.10 to 0.15 if only one partner of tribological pair is coated; and
- if the coating occurs in both sides of the tribological pair, values of 0.06 to 0.12 can be observed.

Otherwise, the AISI 1010 steel was chosen for this study because of its low carbon content and the absence of alloying elements, which gives the same a predominantly ferritic structure, thus ensuring the attainment of a matrix substrate rather ductile, of low hardness. So, in the present work, the adhesion of DLC films on AISI 1010 steel cylindrical metallic surfaces obtained by PECVD (Plasma Enhanced Chemical Vapor Deposition) technique (Michler et al., 1998; Roth, 1995; Choi et al., 2004) was studied, aiming to determine the characteristics and the homogeneity degree for which the DLC films are deposited on the samples surface, and emphasizing the mechanical behaviour and the interaction of the hard coated film to ductile steel matrix pair. To achieve the objective proposed in this paper, the samples were characterized by different techniques, involving metallographic analysis by optical microscopy and scanning electron microscopy, adherence determination by scratch tests covering the application of constant and variable force, adherence tests by penetration, determination of microhardness as well as the characteristics of nanomechanical properties using the nanoindentation technique.

2. EXPERIMENTAL PROCEDURE

Sample of AISI 1010 steel in annealed condition, presenting in its microstructure the constituents ferrite and cementite, were machined according to the conditions shown in Figure 1, and the external cylindrical surfaces were submitted to grinding process.

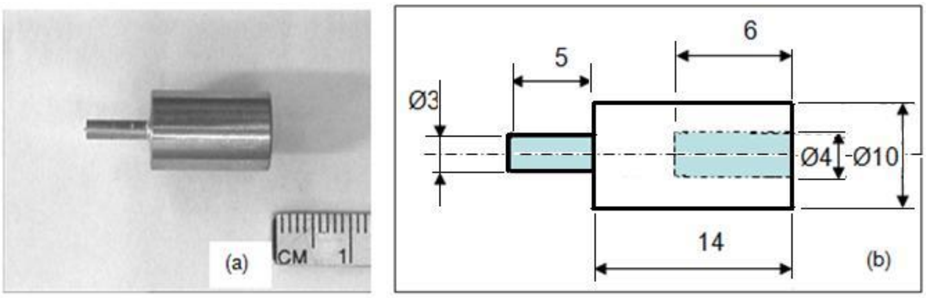
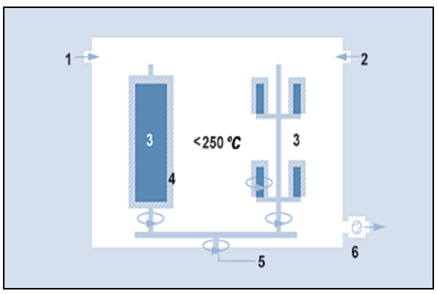


Figure 1. (a) Surface aspect of the sample prior to the depositon step; (b) Design and dimensions of the sample.

The DLC film deposition of AISI 1010 steel samples was performed by PECVD technique using microwave source of 1200 W. Figure 2 shows schematically the electric discharge chamber where the components are positioned to be treated. At first, an inert process gas, argon in the case, enters into the chamber. The cleaning of the components surface is carried out through the mechanism of ion bombardment. In sequence, the deposition of a chromium base intermediate metal film is carried out by means of magnetron sputtering technique. Subsequently, the reactive gas, for acetylene (C_2H_2), is introduced, leading to the formation of amorphous carbon film.

Once the electrical discharge is obtained, the typical reactions of dissociation, ionization and excitation, basically involving these different collisional processes, allow the creation of atoms and carbon ions, hydrogen, and various radicals (C_xH_y) possible, which are responsible for the formation of thin DLC film. It should be emphasized that the variation in voltage applied to the substrate ("bias") also influences on the final properties of the deposited film.

The process parameters were adjusted to ensure the achievement of a film conjugated, consisting of an intermediate layer of chromium, ranging between 0.3 and 0.6 μm, followed by the deposition of DLC film comprising a thickness ranging between 1.6 and 2.0 μm. Prior to the coating treatment, the samples were subjected to two cleaning steps: a) cleaning in a bath of petroleum ether under ultrasound for 35 minutes; and b) cleaning performed under hydrogen electrical discharge at a temperature of 150 °C for 30 minutes, a pressure of 1 Torr, bias voltage of 100 V and a flow of 100 cm^3s^{-1}.



1	An inert gasprocess (argon)
2	Reactive process gas (acetylene)
3	Components to be coated (cathode)
4	Electrical discharge or plasma
5	Connection for high-frequency
6	Vacuum pump

Figure 2 - Schematic representation of the electrical discharge chamber to obtain DLC films by PECVD technique using microwave source.

The physical and metallurgical characteristics of the treated samples were obtained by the following analysis techniques:

- chemical analysis by the technique of X-ray spectroscopy;
- metallographic analysis and microhardness profiling;
- scanning electron microscopy with X-ray dispersive energy microprobe;
- scratch adherence tests involving the application of constant and variable force;
- adherence tests by penetration;
- determination of the nanomechanical characteristics using the technique of nanoindentation.

The chemical composition of the materials used in this study was obtained by chemical analysis in an optical emission spectrophotometer, Spectrolab model LAX-X7. The determination of the carbon content was performed by direct combustion in a equipment Quimitron QCS model 7000, with ranges of carbon from 0.0001 to 6%. Sample preparation for metallographic analysis was performed using 2% Nital etchant for revealing the microstructure.

The determination of the profiles of microhardness in Vickers scale, was performed using a LECO microhardness model LM 700 AT, with a load of 300 grams (HV 0.3).The measurement was performed every 100 μm. The procedure used complied with the technical standard ASTM E384-04. For each sample were performed three microhardness profiles uniformly distributed to 120° along the circumference of the cross section. The results presented are the averages of the profiles obtained.

The determination of the microstructures, as well as the depth of the different deposited layers, was performed using an Olympus microscope and the technique of scanning electron microscopy SEM. The determination of the thickness of films deposited, DLC and chromium, and the chemical composition of these films, qualitatively, were made using a microscope FEI, Model Quanta 200 (low vacuum) equipped with X-ray dispersive energy microprobe, Oxford model 6427, with resolution of 137 eV.

To determine the thickness of the deposited films, it was specified a magnification of 5000x. The procedure adopted in selecting the magnitude (of 5000x) and the preparation of the samples, complied with the technical standard specified in ASTM B 748-90. This standard aims to determine thickness of metallic coatings by SEM, take into account that the field of vision must be chosen between 1.5x and 3x the thickness of the coating to be measured. Still in the process of determining the thickness of deposited films, each sample was divided into four sectors, 5 measurements were conducted in each of the four selected billets along the diameter of the sample. Finally, it was determined the chemical profile of the films deposited through the X-ray dispersive energy microprobe. Chemical analysis was made punctually every 0.25 micrometers into the film.

The equipment used for the testing of adhesion by scratch ("Scratch Test") covering application of the constant and variable force was Revetest CSM, which has in its structure a Zeiss microscope attached to record surface conditions, since the charges can range between 1 and 150 N, and the choice depends on the characteristics of film-substrate pair. The test consists of applying a certain force (expressed in Newton), using a conical diamond indenter (identical to the Rockwell C indenter) with a radius of 200 μm at the tip, which is translated along a path of 10 mm for checking the quality of adhesion of the deposited layer. This technique allows obtaining a result which indicates the presence of regions with some irregularity of that layer, from the occurrence of detachment of the deposited film. For the refered equipment, the applied force may be constant or variable. A constant force is applied to check the homogeneity of the film along the route. Moreover, it is possible to verify the occurrence, along the measured route, points of detachment the obtained film. In turn, the technique of applying a variable force is used to verify the occurrence of rupture in the film for a given force and distance which the break occurs.The sound of the diamond indenter tearing the film is recorded continuously during the test. After testing, the scratch printed by the diamond is characterized by an optical microscope or a scanning electron microscope. The scratch is then evaluated for the type and size of the deformation occurred in the deposited layer, which is a function of applied load. It is used as a measure of the cohesion of the set of film/substrate, and the load below which a pattern of displacement of the film, to be defined, happens, is determined.

Typical modes of detachment obtained during scratching test, in accordance with standard DIN1071-3, can be listed as follows:

a) detachement of the film;
b) fissuring of the film ahead of the indenter and subsequent detachment;
c) fissuring formation along the scratch and lateral detachment of the film;
d) occurrence of fissures all over the scratch;
e) occurrence of adaptation fissures along the film.

The load for which occurs a determined pattern of detachment is called the critical load. If different patterns of detachment occur, there may be several critical loads. For this paper it was used two different possibilities in the mode of load application: a) use of constant load for four different values, namely 5, 20, 50 and 80 N; and b) use of variable load for three different ranges: from 5-20, 20-50 and 50-80 N, depending on the results obtained for the samples studied here.

The equipment used for the adherence test for penetration was the CSM Revetest. In the analysis of the tests it was used for comparison purpose reference patterns evaluating the size and the detached type of the layer displaced around an indentation made on the deposited surface. It is considered as perfect film deposition condition, surface aspects presenting only fissures and no detachment of the film around indentation. In the present work such adherence class (AC) is named AC1. The worst condition would be the whole detachment of the deposited film around the indentation, which would correspond to an adherence class of AC6. For intermediary classes, AC2 would comprise an increase of fissures density with no film detachment; AC3 the start of the film detachment; and AC4 and AC5 simultaneous occurrence of fissures and detachment, comprising a crescent intensity of film removal, respectively. Above the class AC3 it is considered that the film did not have a perfect adhesion and consequently there is the occurrence of detachment of the deposited layer.

The study of film adhesion by penetration tests, applying a constant force value was conducted in two distinct ways, using the equipments CMS Revetest and Rockwell hardness tester. In the first case, the load (expressed in Newton) was apllied using a conical diamond indenter with a tip radius of 500 μm. After applying the charge, the amount of points on which there was some detachment of the deposited material is determined. For this purpose, it was used in this case the CSM Revetest equipment, which enables the application of three distinct forces, 50, 80 and 150 N, which presents a Zeiss microscope to record the condition of the surface tested. In this equipment the charge is applied intermittently, in stages, rather than continuous mode. In the second case, the study of the displacement of the film by a constant value applied force was performed using a Rockwell Durometer in the scales HRA and HRC. For this case it was used the equipment of the brand Stiefelmayer Reichert, with a conical diamond indenter with an angle of 120° to check the hardness of the material. It was performed two tests for each sample, employing different charges of: a) 150 kgf (HRC scale); and b) 60 kgf (HRA scale), using a Zeiss microscope to record surface conditions. In this equipment, unlike, the force is applied continuously and not intermittently.

Finally, the determination of the hardness of DLC films was obtained by nanoindentation technique. In this case, the equipment used was a nanoindenter XP, MTS brand, with a maximum force capacity of 400 mN. The tests were conducted using maximum force of 400 mN, for a total of 10 charge-discharge cycles. The tests were performed in ambient temperature and atmosphere, using a Berkovich tip of pyramidal base. It was performed 3 indentation tests for each deposited film. During the indentation cycle it was used a time of 15 s for loading and 15 s for unloading, and between the cycle of loading and unloading the force was kept constant for a period of 10 s.

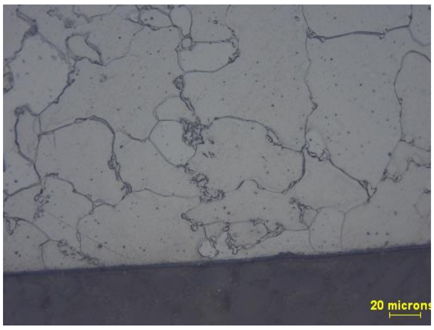
3. RESULTS AND DISCUSSION

Table 2 presents the chemical analysis obtained from samples taken from cylindrical rods of AISI 1010. The result indicates that the raw material is within specifications for that steel.

Table 2. Result of chemical analysis performed for samples of AISI 1010 steel.

Component	C	Si	Mn	P	S	Cr	Mo	Ni	Al	Cu
wt.%	0.09	0.06	0.43	0.01	0.01	0.02	0.01	0.04	0.02	0.00

Figure 3 presents the micrograph of AISI 1010 steel used in this study, confirming the presence of a ferritic matrix with a dispersion of a second phase consisting of spheroidized cementite, a typical microstructure obtained from spheroidize annealing treatment, as expected. It is also indicated by SEM characterization the thicknesses of the Cr and DLC films deposited on the substrates of AISI 1010 steel. Measurements indicate thicknesses of 2.040 and 0.608 μm for the conjugated film of DLC and intermediate Cr layer, respectively. In addition, the determination of the hardness of DLC films by nanoindentation technique indicated, for depths of 180 nm, values around 16 GPa (equivalent to 1600 HV), which is as expected for DLC films.



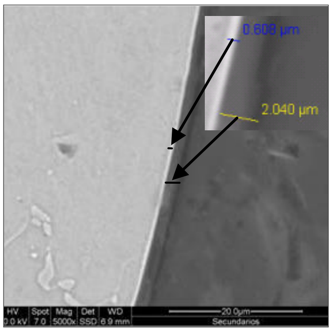


Figure 3. Microstructure of AISI 1010 steel sample, obtained by optical microscopy; and aspect of the DLC and Cr films deposited on the sample surface, obtained by SEM.

Figure 4(a, b) presents the qualitative results of the adhesion obtained by scratch testing with the application of constant load of 5 N and 20 N, respectively, performed on the amorphous carbon films (DLC) deposited on the AISI 1010 steel samples. The result for the load of 5 N indicates the occurrence of a small plastic deformation of the substrate surface (region highlighted by arrows, Fig. 4a), with no evidence of detachment of the deposited DLC film along the edges that separate the regions not scratched of the sample of the test region in contact with the indenter. For 20 N load, the results indicate a strong plastic deformation of the substrate surface, demonstrating the beginning of the pull out of the deposited film, but without any evidence of detachment of the deposited DLC film (see the region highlighted by arrows, Fig. 4b).

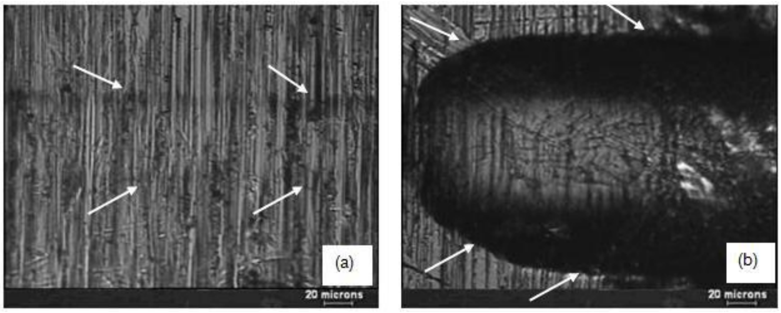


Figure 4. Adhesion by scratching test with application of constant force: a) 5 N; e b) 20 N.

Figure 5(a, b) presents the qualitative results of the adherence test by scratching with variable application of load between 5 and 20 N, for two positions along the course at the beginning and end of the scratching, carried on the surface of amorphous carbon film deposited on the substrate, for samples of AISI 1010 steel. The results confirm the occurrence of strong plastic deformation of the test surface (Fig. 5a, b), with a large removal of the film at the end of the scratching (Fig. 5b), without the existence, however, the detachment close the edges of the scratched area (Fig. 5a, b). The non-occurrence of detachment at the edges is a strong evidence of the perfect adhesion of the deposited film, and the evidence of small points of removal of carbon from the surface tested, represented by white regions present within the scratch (Fig. 5b), is a fact which may be directly related to the high ductility of the substrate material and with the increase of the test charge, when it approaches the value of 20 N, which would lead to the whole film/base material to yield in the test condition.

Because of AISI 1010 steel substrates have shown high plastic deformation and initiation of tearing of the film deposited with a load of 20 N, it was chosen in this work by not continuing to scratch tests with the application of higher loads of 50 and 80 N.

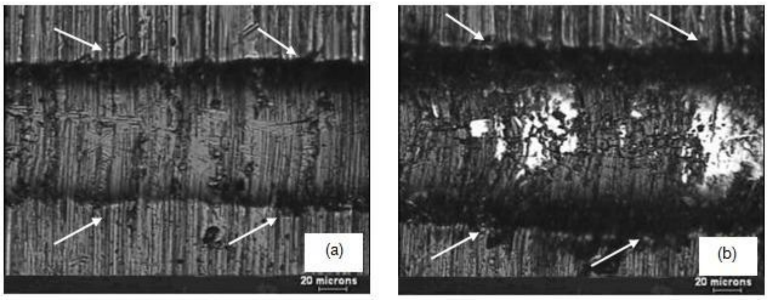


Figure 5. Adhesion by scratch test with application of variable strength between 5 and 20 N, for two positions along the route: a) at the beginning; and b) at the end of the scratching.

Figure 6 shows the qualitative outcome of the adherence test for penetration, using the equipment Revetest, by applying a constant load of 50 N on the surface of amorphous carbon films deposited on samples of AISI 1010. The results indicate that there is no detachment or cracking along the deposited films, however, the large dimension of the print confirms the high ductility of the substrate tested. The results for the charges of 80 and 150 N are not presented here due to the high ductility of the substrate studied, resulting in much larger deformation than those found for the condition presented here.

Figure 7 shows the qualitative result of the test for penetration, using the Rockwell hardness tester by applying a constant load of 60 Kgf (HRA) on the surface of amorphous carbon films deposited on samples of AISI 1010. The results also demonstrate only a significant plastic deformation of the whole film/substrate with no evidence of cracking or displacement of the deposited films.

In qualitative levels and considering the aspects of reproducibility of the results, one may observe a very good adhesion of the film deposited on cylindrical AISI 1010 steel substrates, at least for the test conditions evaluated here. This assertion is based on overall results of all performed tests involving scratching and penetration. Quality levels in terms of the adherence classes varied between AC1 and AC3 considering, of course, the limits imposed by the high ductility of the substrate material used here and its response as a function of the load applied in different tests. In other words, depending on the characteristics of AISI 1010 steel which has low hardness ($\approx$ 100 HV) and high ductility ($\varepsilon \approx$

40%), the charge used in different tests, necessarily had to be limited to 5 N and 20 N in the scratching test and to 50 N and 60 kgf in the penetration tests. For all the cases, only few points of film removal were observed, which are explained by the high involved loads. Despite, no detachment was verified.

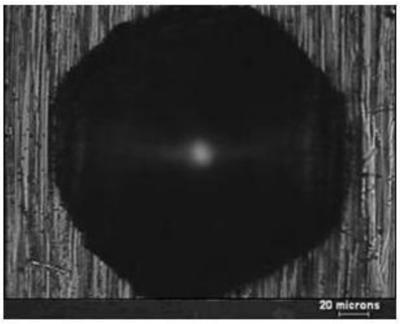


Figure 6. Adherence test by penetration, using the Revetest equipment to a constant load of 50 N.

Additionally, it is necessary to emphasize that the results of adhesion obtained here in a qualitative way differ significantly from those presented by Rahman (2007), which asserts the occurrence of inadequate adhesion to soft substrates. This statement has not been established in this work and deserves to be discussed. In the Rahman (2007) study, the DLC film was deposited directly onto a substrate surface which was changed from the nitriding treatment, unlike the procedure adopted here, which considers an intermediate layer of Cr between the DLC film and the related substrate. Therefore Rahman's statement (Rahman et al., 2007), could not be generalized to study conditions such as those used in this work.

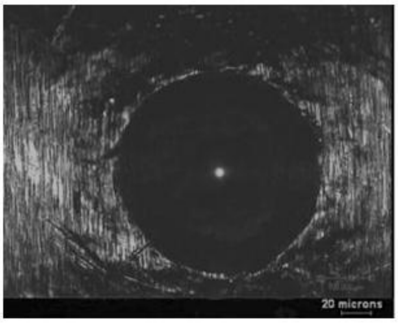
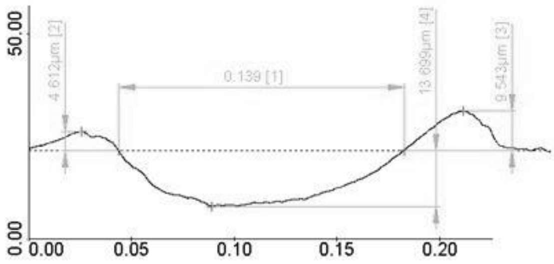


Figure 7. Adherence test by penetration, using Rockwell hardness tester, applying a constant load of 60 Kgf (HRA).

It can be concluded at this point that the presence of an intermediate layer such as used here, in the case a Cr layer, is largely responsible for ensuring excellent adhesion of the deposited film even for soft substrates, which is the case of AISI 1010 steel. This result is consistent with the reported literature (Choi et al., 2004; Rahman et al., 2007; Wei and Yen, 2007), which indicate that an intermediate layer is required to increase the adhesion of the deposited film.

Finally, Figure 8 shows the results obtained using the technique of profilometry, which made it possible to characterize quantitatively the main regions of the risk profile obtained upon application of a load of 20 N, when conducting the scratching test with a force varying from 5 to 20 N. The results indicate that a large amount of material was removed to the lateral regions, or the edges of scratching as a result of large deformation and large depth of the obtained scratch. The results indicate the width and the depth values of the scratch were 139 μm and 13.7 μm (measured in the positions 1 and 4, respectively) when 20 N load was achieved. Measures related to the positions 2 and 3, of 4.6 and 9.5 μm, respectively, indicate that the volume of metal displaced by the action of the indenter depend on the ductility of the treated substrate, confirming the high plasticity of it.

Returning to the results shown in Figure 5, which shows the topography of the risks to different studied areas, as the applied load is changed the scratch characteristics are modified, and the critical load, defined as the load for which the cracks are initiated, depends on the properties of the film/substrate pair. It was found that for AISI 1010 tested with 20 N load, cracks and tearing markedly appears on the scratched surface, indicating that the critical load for the use of this material has already been achieved. Table 3 presents for comparison purpose some measurements taken in the start and in the end of the scratch obtained when the load was changed from 5 to 20 N.



Point	Measurement (µm)
1	139
2	4.6
3	9.5
4	13.7

Figure 8. Characterization of the obtained scratch profile at 20 N load.

Finally, the nanoindentation characterization indicated the obtainment of DLC films with values of hardness and elastic modulus consistent with the tested material, which were on the order of 1600 HV and 180 GPa, respectively. In addition, it should be noted that the results of loading-unloading curves, not presented here, also confirmed the excellent adhesion of the studied film/substrate pair, which was confirmed by the obtainment of continuous curves in the refered graphics.

Table 3. Measures of the width and depth of the scratching obtained in the scratch test with increasing load varying from 5 to 20 N, these measures taken at the beginning and end of the test.

Load of 5N (scratch start)		Load of 20N (scratch end)	
Width (µm)	Depth (µm)	Width (µm)	Depth (µm)
104	2	139	14

4. CONCLUSIONS

In the present work it was studied the influence of AISI 1010 steel substrate in the adhesion of the DLC + Cr conjugated films produced by PECVD technique with microwave source. From the results and discussion it was conclude that:

- the adhesion of the DLC + Cr conjugated films deposited in AISI 1010 steel substrates is very good, and the presence of a Cr intermediate is largely responsible for ensuring excellent adhesion of the deposited film;
- the critical charge for the AISI 1010 in the two studied conditions is on the order of 20 N.
- the classes of adherence of deposited films/substrate pair obtained here corresponded to the categories between AC1 and AC3, according to the specified, and
- the typical high plasticity of ductile metals, such as the verified for AISI 1010 steel substrates, does not necessarily result in the appearance of detachment or cracking along the deposited films; otherwise, in this case, the most common occurrence was the tearing of the deposited material for high applied loads in the scratch test.

5. ACKNOWLEDGEMENTS

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