# DLC FILMS ADHESION ON ABNT M5 STEEL CYLINDRICAL METALLIC SURFACES

**Jairo Müller Wolff, jairo.wolff@br.bosch.com**
Graduate Program of Materials Science and Engineering (PIPE), *Universidade Federal do Paraná* (UFPR), 81531-980, Brazil

**Silvio Francisco Brunatto, brunatto@ufpr.br**
Department of Mechanical Engineering, *Universidade Federal do Paraná* (UFPR), 81531-980, Brazil

***Abstract.*** *The adhesion of DLC (diamond like amorphous carbon) + chromium base intermediate metal film on cylindrical metallic surfaces of ABNT M5 steel was studied. Annealed 10 mm diameter x 14 mm length steel samples were coated with PECVD (Plasma Enhanced Chemical Vapor Deposition) technique. ABNT M5 steel was chosen due to its large application in the metal-mechanical field and to present high hardness and good toughness in tempered condition. 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 that the adhesion of the DLC + Cr films on the cylindrical metallic surfaces is strongly dependent of the mechanical characteristics of the studied steel.*

***Keywords****: DLC, PECVD, Scratch test, AISI M5 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, such as high hardness, wear resistance, chemical inertness, and good thermal and optical properties (Michél, 2005), depsite they have low concentrations of chemical bonds of the $sp_3$ type. 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 ABNT M5 steel was chosen to realize this study due to its large application in the metal-mechanical field and to present high hardness and good toughness in tempered condition, a consequence of its microstructure comprised by fine alloying element carbides dispersed in a cubic martensitic matrix. So, in the present work, the adhesion of DLC films on ABNT M5 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 hard and tough 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

Samples of ABNT M5 steel were machined from a tempered bar according to the conditions shown in Figure 1, and the external cylindrical surfaces were submitted to grinding process.

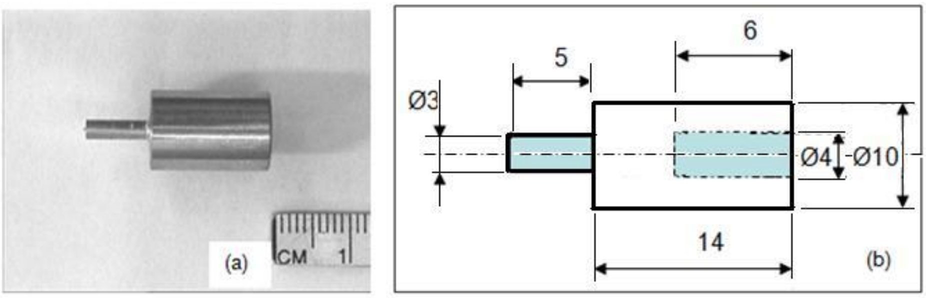
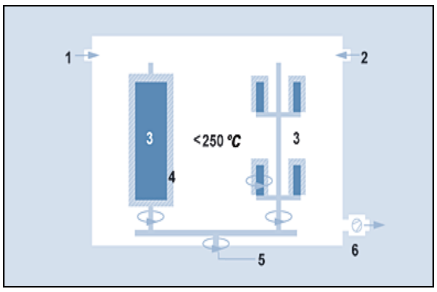



Figure 1. (a) Surface aspect of the sample prior to the depositon step; (b) Design and dimensions of the sample.

The DLC film deposition of ABNT M5 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 carrying out the metallographic analysis followed the steps outlined in the standard ASM-11 using to 2% Nital reagent 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, five 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) detachment 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 samples of ABNT M5. The result indicates that the raw material is within specifications for that steel.

Table 2. Result of chemical analysis performed for samples of ABNT M5 steel.

| Component | C | Si | Mn | P | S | Cr | Mo | V |
|---|---|---|---|---|---|---|---|---|
| wt.% | 0,8 | 0,42 | 0,23 | 0,019 | 0,01 | 3,94 | 4,43 | 0,97 |

Figure 3 presents the micrograph of ABNT M5 steel used in this study, confirming the presence of a tempered martensitic matrix, a typical microstructure obtained from quenching-tempering treatment, as expected. It is also indicated by SEM characterization the Cr and DLC films thicknesses deposited on the substrates of ABNT M5 steel. Measurements indicate thicknesses of 2.166 and 0.632 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 18 GPa (equivalent to 1800 HV), which is as expected for DLC films.

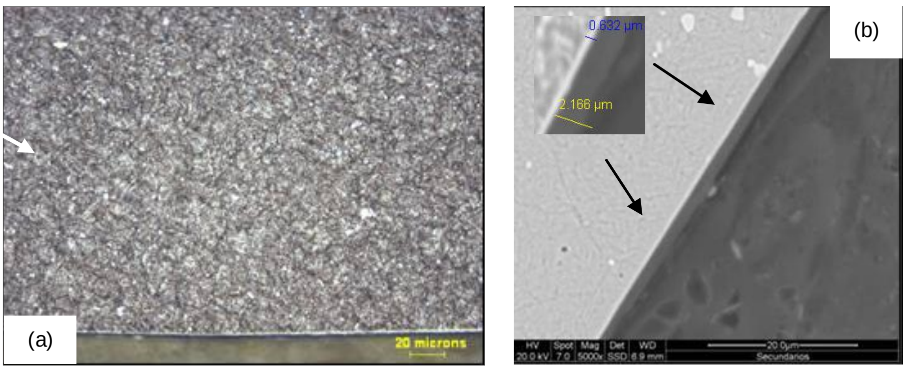



Figure 3. (a) Microstructure of ABNT M5 steel sample, obtained by optical microscopy; and (b) aspect of the DLC and Cr films deposited on the sample surface, obtained by SEM.

Figure 4(a, b, c, d) presents the qualitative results of the adhesion obtained by scratch testing with the application of constant load of 5, 20, 50 and 80 N, respectively, performed on the amorphous carbon films (DLC) deposited on the

ABNT M5 steel samples. The result for the load of 5 N indicates the occurrence of a very 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. The plastic deformation of the substrate surface is increased as the load is changed for 20, 50 and 80 N (see Fig. 4b, c, d, respectively). The results indicate 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,c, d). The evidence of no detachment at the scratch edges is indicative of the perfect adhesion of the deposited film. Otherwise, the evidence of small points of carbon removal indicated by white regions within the scratchs (Fig. 4b, c, d) is a fact which may be directly related to the low ductility of the substrate material. Finally, as the load is increased, a tendency of the whole film/base material to yield in the refered test conditions is evidenced inside the scratch.

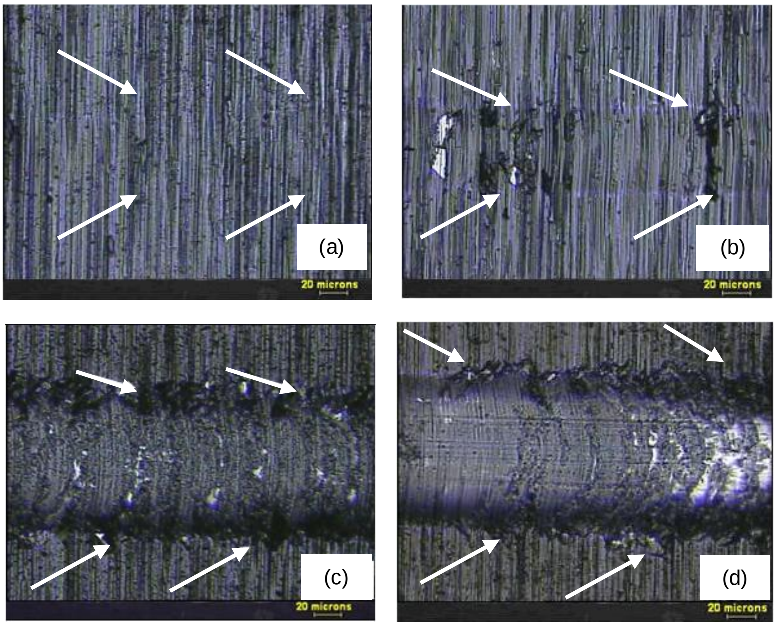



Figure 4. Adhesion by scratch test with application of a constant load of: a) 5 N; b) 20 N; c) 50 N; and d) 80 N.

Figure 5(a, b) presents the qualitative results of the adherence test by scratching with variable application of load between 50 and 80 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 ABNT M5 steel. The results confirm the occurrence of strong plastic deformation of the test surface, without the existence, however, the detachment close the edges of the scratched area (Fig. 5a, b). The non-occurrence of detachment at the edges confirms the perfect adhesion of the deposited film, as verified in results presented in Fig. 4.

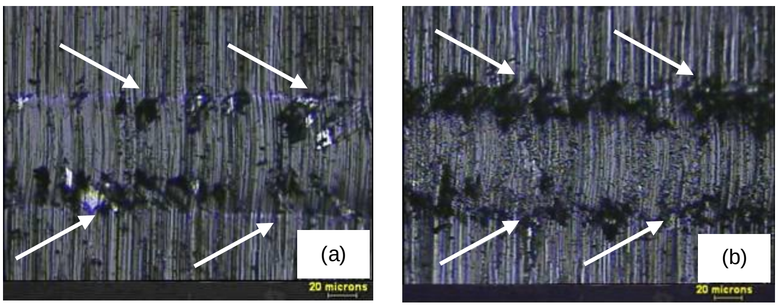



Figure 5. Adhesion by scratch test with variable application strength between 50 and 80 N, for two positions along the route: a) at the beginning and b) at the end of the scratch.

Figure 6(a, b, c) shows the qualitative results of the adherence test by penetration, using the Revetest equipment, applying a constant load of 50, 80 and 150 N on the surface of amorphous carbon films deposited on samples of ABNT M5, respectively. The results indicate that there is no detachment or crack formation along the deposited films for all studied conditions, however, a slight increase in the indentation dimension was verified as the load is increased, as expected. The absence of both crack and detachment around the indentations confirms the good adhesion of the obtained films.

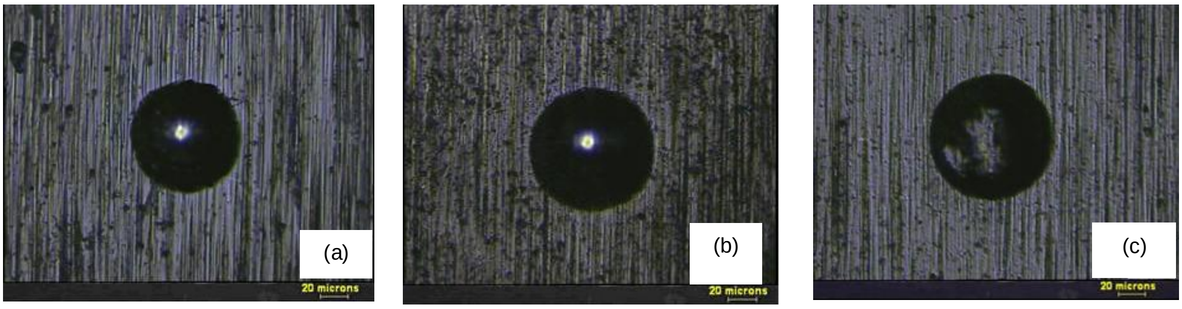



Figure 6. Adherence test by penetration, using Revetest equipment, applying a constant load of: a) 50; b) 80; and c) 150 N.

Figure 7(a, b) shows the qualitative result of the penetration test, using the Rockwell hardness tester by applying constant loads of 60 and 150 Kgf on the surface of amorphous carbon films deposited on samples of ABNT M5, respectively. The results demonstrate there is no crack formation for sample tested with 60 kgf load (Fig. 7a). Otherwise some cracks appear around the indentation for the sample tested with 150 kgf load (Fig. 7b), as indicated by arrow, despite this no detachment was evidenced. Considering the reproducibility of the results, one may observe a very good adhesion of the Cr + DLC films deposited on substrates of ABNT M5 steel cylindrical samples, at least for the test conditions evaluated here. This assertion is based on overall results of all tests involving scratching and penetration achieved. The observed quality levels considering the different possibilities varied between AC1 and AC3 classes, confirming the excellent adhesion of the obtained films.

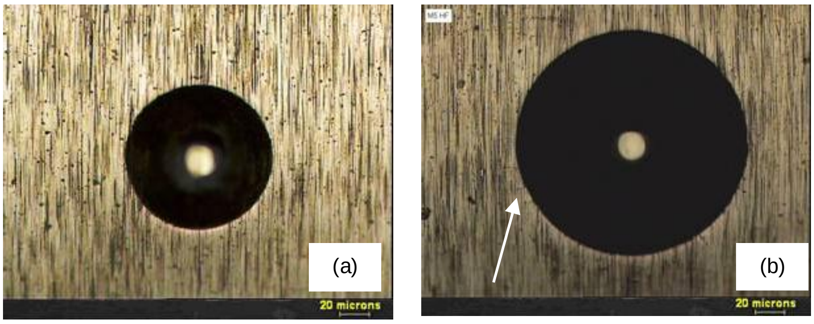
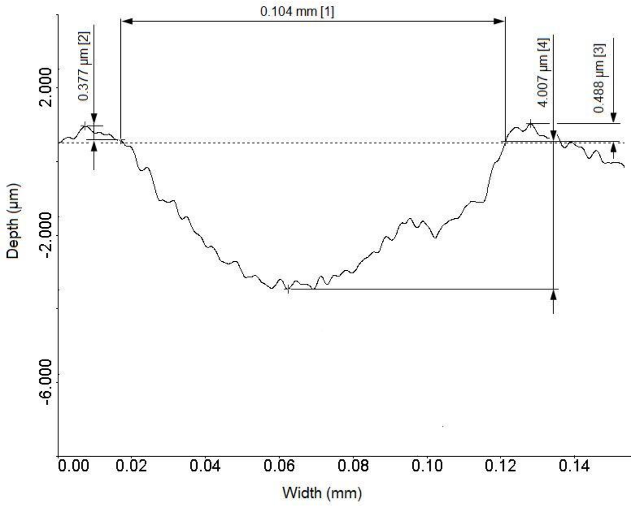



Figure 7. Adherence test by penetration, using Rockwell hardness tester, applying a constant load of: a) 60 kgf (HRA); and b) 150 Kgf (HRC).

Finally, Fig. 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 small amount of material was moved to the lateral regions, or the edges of scratching as a result of the small deformation, leading to a small depth of the obtained scratch. The results indicate the width and the depth values of the scratch were of 104 μm and 4.0 μm (measured in the positions 1 and 4, respectively) when 20 N load was achieved. Measures related to the positions 2 and 3, of 0.38 and 0.49 μm, respectively, indicate that the volume of metal displaced by the action of the indenter depend on the high hardness of the treated substrate, confirming the good tenacity of it.

Returning to the results shown in Fig. 4, 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 ABNT M5 tested with 50 N load, cracks and tearing 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.

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 1800 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.




| Point | Measurement (µm) |
|---|---|
| 1 | 104 |
| 2 | 0,38 |
| 3 | 0,49 |
| 4 | 4,0 |

Figure 8. Determination of the scratch profile by measuring the dimensions of four distinct points (for 20 N load).

Table 3. The width and depth of risk obtained in the scratch test with increasing load, varied 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) |
| 53 | 0.5 | 114 | 4 |

## 4. CONCLUSIONS

In the present work it was studied the influence of ABNT M5 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:

- DLC + Cr conjugated films deposited in ABNT M5 steel substrates present good adhesion;
- the classes of adherence of the deposited films/substrate pair obtained here corresponded to the categories between AC1 and AC3; and
- the typical high hardness and good toughness of tempered steels, such as the verified for ABNT M5 steel substrates, result in the appearance of cracking around the indentation only for very high loads, such as the verified in the Rockwell test, by means the use of 150 kgf load.

## 5. ACKNOWLEDGEMENTS

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## 7. RESPONSIBILITY NOTICE

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