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# CHARACTERIZATION OF DLC COATING IN DRILLING TOOLS

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**Abstract.** *An experimental study on the mechanical properties and adhesion of diamond-like carbon (DLC) coatings was carried out in this work. For this, tools used in machining systems for drilling of high speed steel (HSS) and cemented carbide (WC-Co) were coated by technical PECVD. All analyzes were done in the own tools, being prepared specially by this. In sequence, were made: thickness micrographs and visual analysis of grip of DLC by scanning electron microscopy; topographical images to mark the silicon middle layer in micrometer scales by atomic force microscopy (AFM); wettability tests to identify the type of interaction with water; nanoindentation tests on the coating for hardness and modulus of elasticity measurement; hardness of the substrate and the film adhesion test on a Rockwell hardness test. By topographical analysis (AFM) was confirmed the silicon middle layer with a thickness greater than the DLC itself (1.8 and 1.0 micrometers respectively). The DLC coating presented higher hardness compared both substrates, corresponding to approximately 5 times the hardness of high speed steel (~7.6 GPa) and 2 times the hardness of WC-Co (~13.4 GPa). The analyzes of the pairs substrate/coating showed that the relationship between their interfaces should be considered, even the WC-Co being harder, its coating obtained lower hardness compared to the HSS. This is associated with the status of substrate before deposition process and its individual properties. It's important too that the interface has high adhesion by coating, so it can work in the best condition to increase wear resistance, thereby improvement the load capacity of the surface. The properties and therefore the quality of the coating varied with the substrate.*

**Keywords:** *Characterization, DLC, drill, HSS, WC-Co*

## 1. INTRODUCTION

The deposition of the coating Diamond-Like Carbon (DLC) on cutting tools and sliding components is interesting to the mechanical and rocks drilling industries. This is due to the unique properties of high hardness and good resistance to wear. Furthermore, DLC layer is effective to improve corrosion resistance and fatigue (Chowdhury *et al.*, 2004; Choi *et al.*, 2012; Suzuki *et al.* 2013; Sutton *et al.*, 2013).

DLC is a hard film formed by carbon produced first time in 1960s by Aisenberg and Chabot (Lettington, 1998; Nakahigashi *et al.*, 2004). These were pioneers in the use of energy species for deposition, developing, in principle, the work of deposition with silicon (production of Si ions by spraying electrodes in on argon plasma). Finally, using the same device to spray carbon electrodes. Thus, the deposition of the transparent carbon film with high hardness is created. Although the film has properties like diamond, its inventors did not find it appropriate to give the name "diamond" but rather "diamond-like" or "i-C", because its structure was amorphous and not crystallized as the diamond (Nakahigashi *et al.*, 2004; Lifshitz, 1999).

The development of the technique continued with important results in 1970s, with the hydrogen-containing (hydrogenated) amorphous carbon film deposited using ionized beam of hydrocarbon gas (Weissmantel and Reisse, 1979). Radio frequency plasma assisted chemical vapor deposition (RF-PCVD) was also developed for hydrogenated amorphous carbon film. Non-hydrogenated amorphous carbon film is also deposited using cathodic arc physical vapor deposition (CA-PVD), cathode sputtering or laser ablation. In the middle 1980s, metal containing amorphous carbon film was developed using simultaneous sputtering of metal and carbon targets (Dimigen *et al.*, 1987). In the 1990s, plasma immersion ion implantation and deposition (PIII and D) method was developed for hydrogenated amorphous carbon film (Nakahigashi *et al.*, 2004).

Main difficulties encountered in steel deposition processes, which are widely used in industry, are as high residual compressive stresses and the incompatibility of the coefficient of thermal expansion. These characteristics directly influence the growth limitation of the film thickness with good adhesion in a maximum of 0.25  $\mu\text{m}$  of the DLC. Silva, *et al.*, 2011 had a thickness of 1  $\mu\text{m}$  using carbonitriding and cementation surface treatments before to the application of DLC in SAE 6150 steel samples. The aim of surface treatments is to approximate substrate and coating characteristics. For this, studies on structure, composition and processing of materials were intensified.

The use of carbon coating improves the life of the drills by increasing its hardness and decreasing its coefficient of friction. Another advantage is the possibility of working with minimal quantity of lubrication (MQL) or without it. Bhowmick and Alpas (2008) evaluate the advantages that are possible when DLC coated tools are used with the MQL during the drilling of Al-6% Si (329 Al) alloy. Experimental studies with DLC coatings showed better performance during dry drilling due mainly to attenuated adhesion properties. Dry drilling of aluminum alloys is an environmentally friendly process but also an extremely difficult task due to the tendency of aluminum to adhere to drills made of conventional materials such as HSS.

Technological developments are increasingly leading to the use of self-lubricating coatings due to the need to reduce polluting waste. A trend in the development of wear resistant coatings is to make them harder and thinner through improvements in the deposition processes (Tuck *et al.*, 2001). Recent studies have shown the influence of hardness and adhesion on film behavior, however the mechanisms behind these effects are still not well understood. Therefore, this work is justified to contribute with information about mechanical properties, reliable, resulting from the characterization of the coatings in drilling tools.

This paper presents an experimental methodology to measure the mechanical properties of DLC coating in HSS and cemented carbide (WC-Co) drilling tools. As well as evaluate its visual aspects, thickness, adhesiveness, wettability and wear resistance.

## 2. EXPERIMENTAL PROCEDURE

Most of the work related to coating characterization is performed on specially prepared flat samples. The samples analyzed in this work were taken from the HSS and WC-Co drills with DLC coating performed on the tool deposition company CVD-Vale located in São José dos Campos / SP. Whose coating process is carried out by plasma enhanced chemical vapor deposition (PECVD).

### 2.1 Sample preparation

The samples analyzed were taken from DLC coated drill bits, of which the size and preparation of the drills were designed to better match the characterizations.

For analysis of the thickness of the DLC coating, the cross sections were carried out on the straight shank of the drill, as in Figure 1 (a), carefully to preserve the free coating of peeling or edge burning. These were hot-coated with resin durafast and subjected to a sanding and polishing sequence mainly to meet the atomic force microscopy (AFM), which requires low surface roughness for the approximations (Figure 1 (b)).

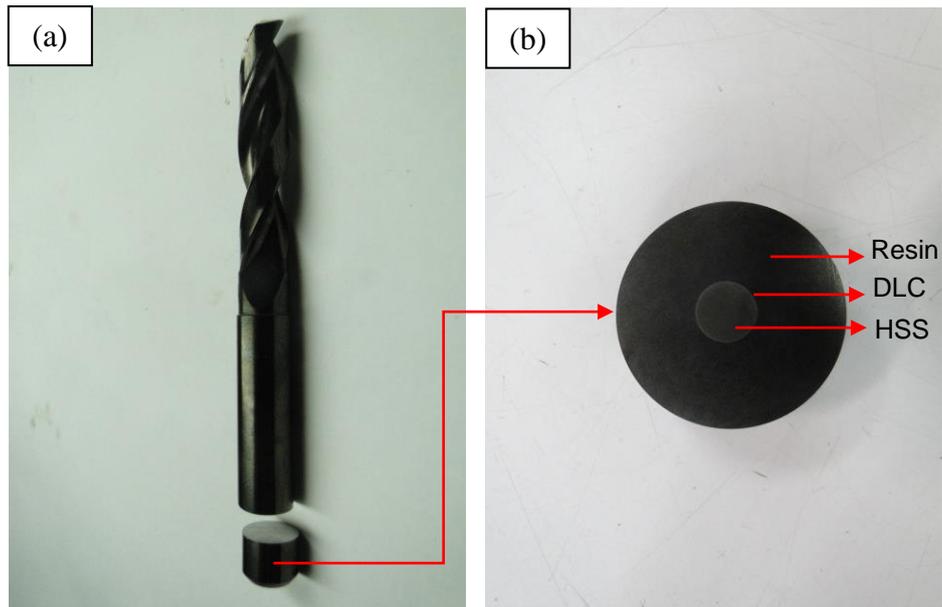


Figure 1. (a) Cross section on the straight shank of the drill and (b) sample after hot-coated with resin and subjected to a sanding and polishing sequence.

The preparation of samples for the nanoindentation tests to hardness and modulus of elasticity of the coating required a sequence of cuts of straight shank of the drill, that allowed direct measurements on the coating maintaining a flat support base, as shown in Figure 2. The same care was taken during cutting to prevent material properties from being altered during the procedure. A support base of the sample is required for the execution of the tests, as it aims to position the indenter on the cylindrical surface, and the techniques used are very sensitive and designed to work on flat surfaces specially prepared for these tests. However, since the areas analyzed in nanoindentation are of micrometer scales, the upper end region meets the test specifications.

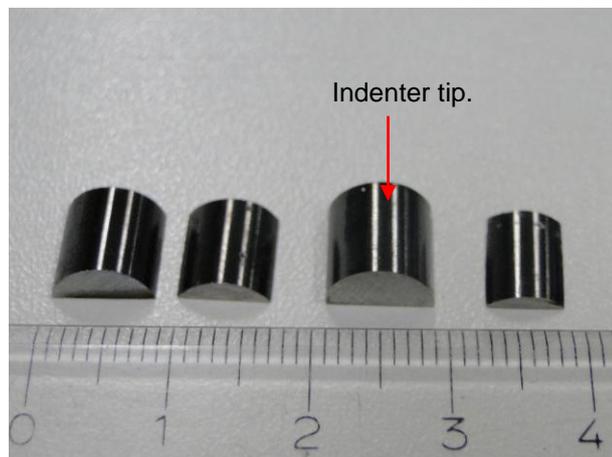


Figure 2. Sample for nanoindentation tests from straight shank of the drill.

To perform surface wettability testing, HSS and WC-Co samples of dimensions 22 x 11 x 7 mm (Length x width x thickness) and coating on only one face were used.

For microindentation, no specific sample preparation is required, and such tests can be performed on the tools themselves. As will be described in the next item.

## 2.2 Procedure

Initially an analysis of status of the coating over the drills was carried out using scanning electron microscope (SEM) images from XL30 PHILIPS equipment. And the total thickness of the DLC was measured by same equipment,

but now with the sample properly prepared for the session view, as shown in the previous item. However, according to the manufacturer who covered the drills, it was necessary to create an interface layer of an intermetallic material, more specifically Si, to obtain adequate adhesion of the film to the substrate. This layer is not differentiated in SEM images.

To measure the silicon interface between substrate/DLC, the AFM technique was carried out in the Asylum Research equipment model MFP-3D-SA, from which a region was swept in the same location as the cross section of the sample used in the SEM. Generating the three-dimensional topography of the region, distinguishing the Si interface. These measurements are essential for the nanoindentation test because it is necessary to know the maximum indentation depth allowed so that the properties of the substrate do not influence the measurements of the coating. In the literature, it is indicated that the indentation depth does not exceed 10% of the thickness of the coating (Silva *et al.*, 2011).

The hardness and modulus of elasticity measurements of DLC coatings were performed on the nanoindentador NanomechPro™ with diamond tip type Berkovich. The tests were performed on both the HSS and the WC-Co samples. With the coating thickness results already predefined, it was possible to establish the maximum indentation depth values around 280 nm. The loading values were initially based on the work of Cooper *et al.*, 1994.

To evaluate the adhesion of the DLC coating, with the silicon middle layer, on the metallic substrate of the HSS and WC-Co was performed the method known as Mercedes (Vidakis *et al.*, 2003). The test was accomplished with a standard microdurometer (Wilson® Rockwell® Series 2000) adjusted to perform the Rockwell C hardness measurements (see Figure 3), with 150 kgf on the coated tool surface, from which 3 tests were performed on each sample.

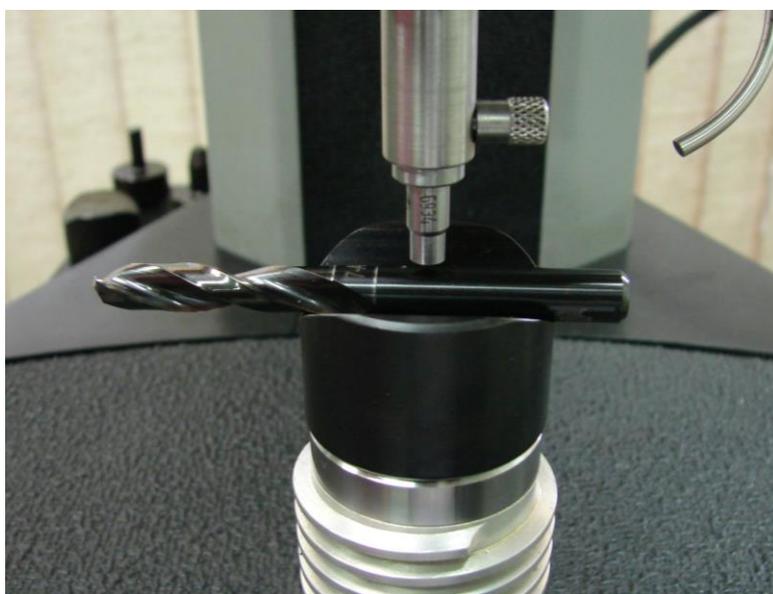


Figure 3. Rockwell C microindentation test (Mercedes test).

A visual observation of the indentations formed after the tests was performed. From which, with the aid of an optical microscope, the images are recorded and evaluated by the failure type established by the standard, VDI 3198 (Figure 4).

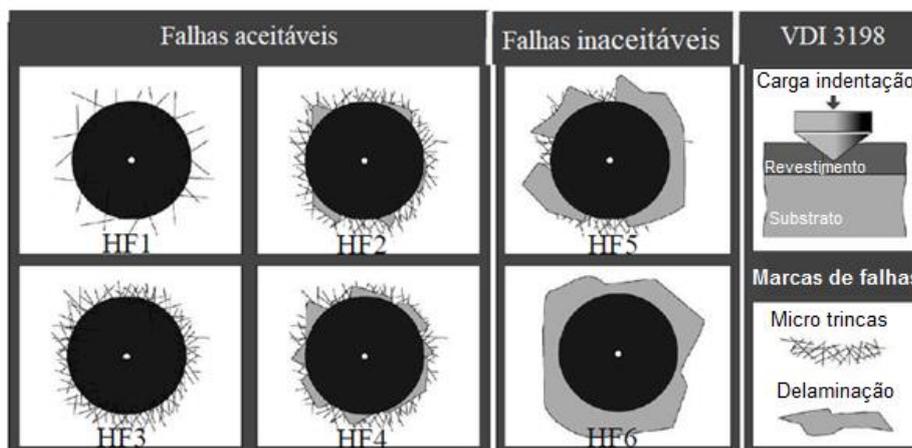


Figure 4. Indentation test evaluation standards according to VDI 3198

To check the wettability both surfaces, with and without coating of each material, a Goniometer was used, an equipment composed of a camera that records the image of a drop (volume lower than 10  $\mu\text{L}$ ) on the surface that will be studied. The sample were properly cleaned with high purity ethanol, thoroughly dried in air and carefully wiped with a cloth to remove potential physically adsorbed remaining alcohol molecules. The interaction between the surface and a given liquid, according to Ferreira (2013), can be studied by measuring the so-called contact angle  $\Theta$ . This is defined as the angle between a plane tangent to a drop of the liquid and a plane containing the surface where the liquid is deposited, as shown in Figure 5.

Interaction	Superhydrophilic	Hydrophilic	Hydrophobic	Superhydrophobic
Drop diagram				
Contact angle	$\theta < 10^\circ$	$\theta < 90^\circ$	$\theta > 90^\circ$	$\theta > 150^\circ$

Figure 5. Diagram of drops on surfaces with different degrees of wettability and corresponding values of contact angles (Ferreira, 2013).

### 3. RESULTS AND DISCUSSION

#### 3.1 Visual analysis of coating after deposition process

The experimental work began with the visual analysis to evaluate the coating adhesion in the new tools. From the Figure 6., it is possible to see a change of appearance at the ends near the main edge, passing through the tip and through secondary edge. This for both HSS and WC-Co coated with DLC layer, but the latter was more intense (Figure 6 (b)).

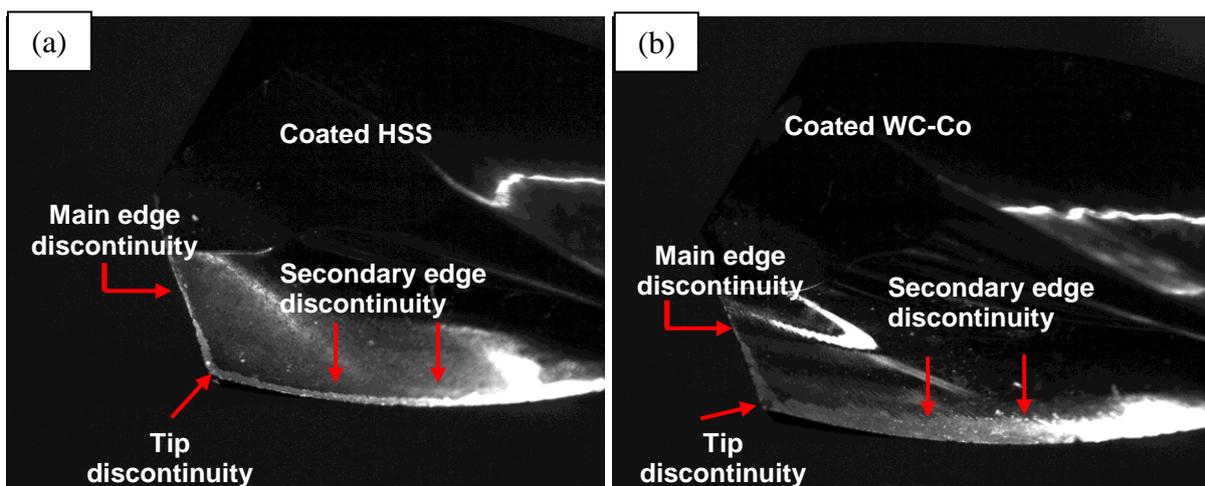


Figure 6. Discontinuities observed in drills immediately after coating.

Afterwards the zone of the secondary edge was chosen to be observed in more detail in the SEM and the images of the Figure 7 were obtained. In the image of the Figure 7 (a) and noticeable the lack of coating near the secondary edge by the inner side (flutes), while the outside (secondary surface of clearance) is free of discontinuities. The magnification in zone 1 of the Figure 7 (a), in Figure 7(b), it is possible to observe a pattern of lines that forms the topographic profile

observed. This profile is followed by the coating and is probably derived from drill manufacture, precisely the step of machining the flute. Then, the drill manufacture should be observed. The magnification in zone 2 of the Figure 7 (a), in Figure 7 (c) shows the secondary edge as the interface between adhesion and lack of adhesion.

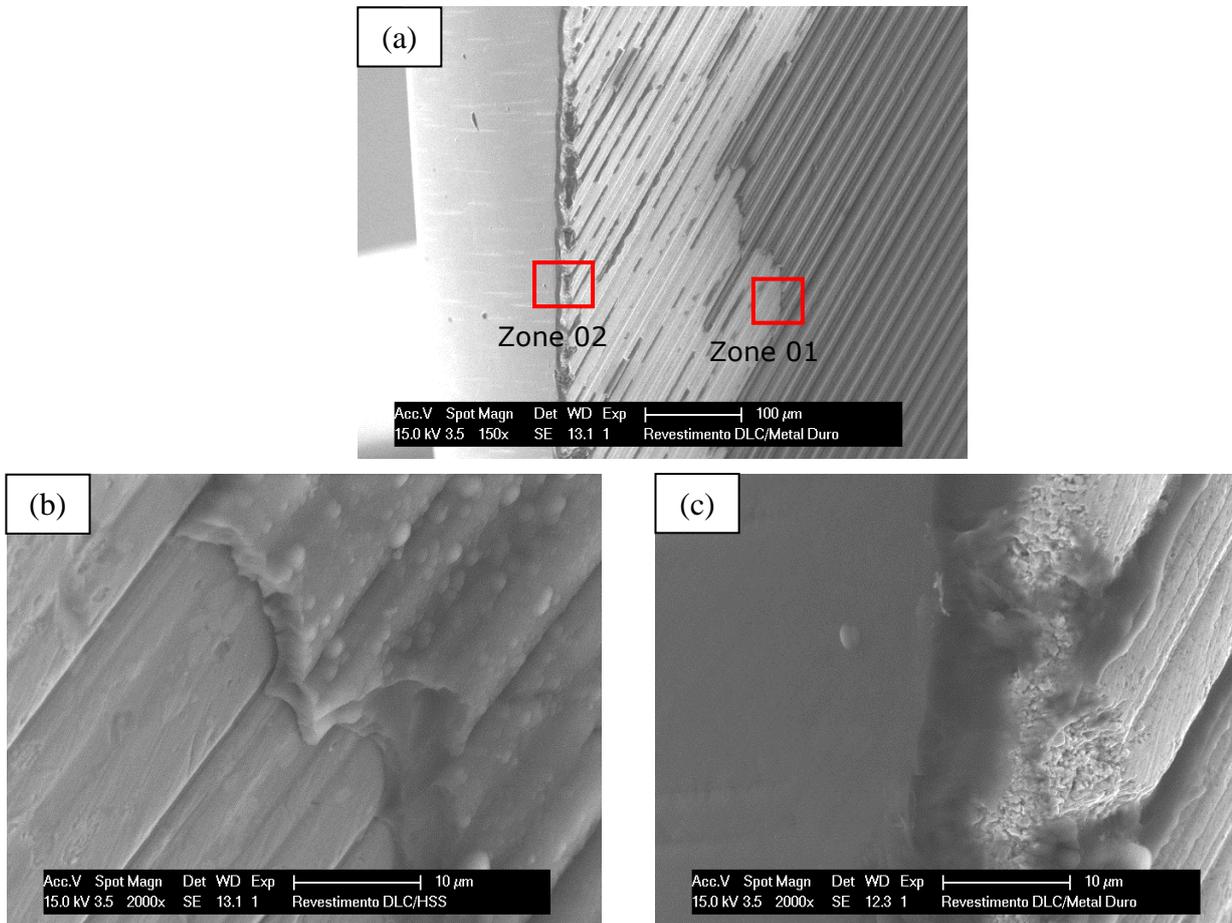


Figure 7. (a) Magnification by (SEM) of the secondary cutting edge of the tool. (b) Magnification of zone 01 in (a), which shows that the coating followed the surface profile. and (c) Magnification of zone 02 in (a) which shows failure by lack of adhesion at the edge by coating.

### 3.2 Coating thickness measurements and morphological analysis

The coating thickness images were obtained by SEM, through the samples prepared in cross section. In the Figure 8 the resin/coating/substrate layers are observed, with a magnification in the coating/substrate interface region the total coating thickness measured was 2.81 μm.

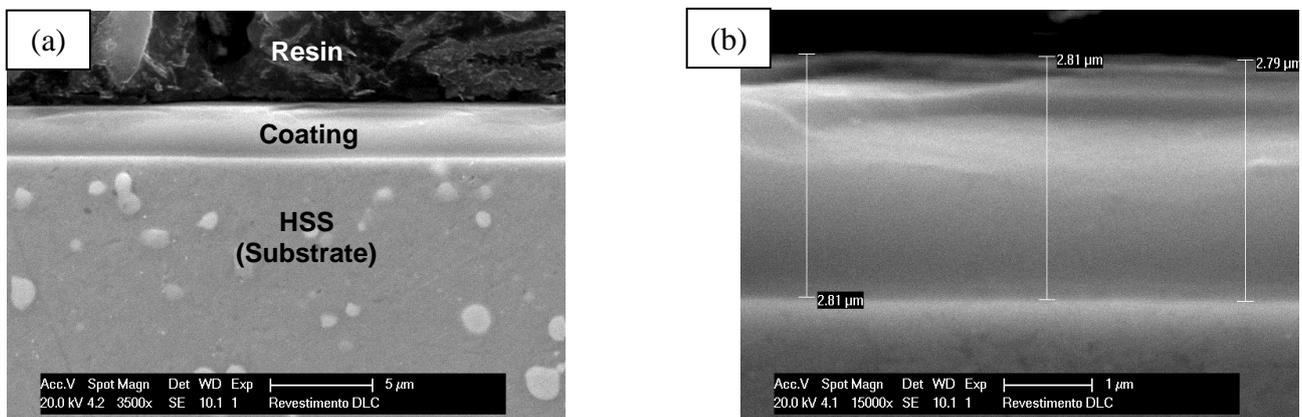


Figure 8. SEM of (a) presentation (resin/coating/substrate) and (b) coating thickness.

These values are an advance for the deposition process, as thicknesses greater than 0.5  $\mu\text{m}$  generate higher residual stresses in the interface region with consequent peeling of the film. Although the peeling was evidenced in some zones of the tool, in the majority, the film remained adhered with this thickness. Cooper *et al.*, 1994 investigated the relationship between adhesion and film thickness of DLC, from which it was observed that larger thicknesses lead to adhesion difficulties. In their studies, samples with 2.0  $\mu\text{m}$  had isolated areas of peeling, while samples with 0.5  $\mu\text{m}$  showed no tendency for spontaneous peeling to the same substrate.

For the identification of Si middle layer, which allows an improvement in the adhesion of DLC to steel, the AFM was used to assemble the topographic image of Figure 9 on the interface, distinguishing the Si layer in the coating. The AFM images confirm the total coating thickness values measured by SEM of 2.8  $\mu\text{m}$ .

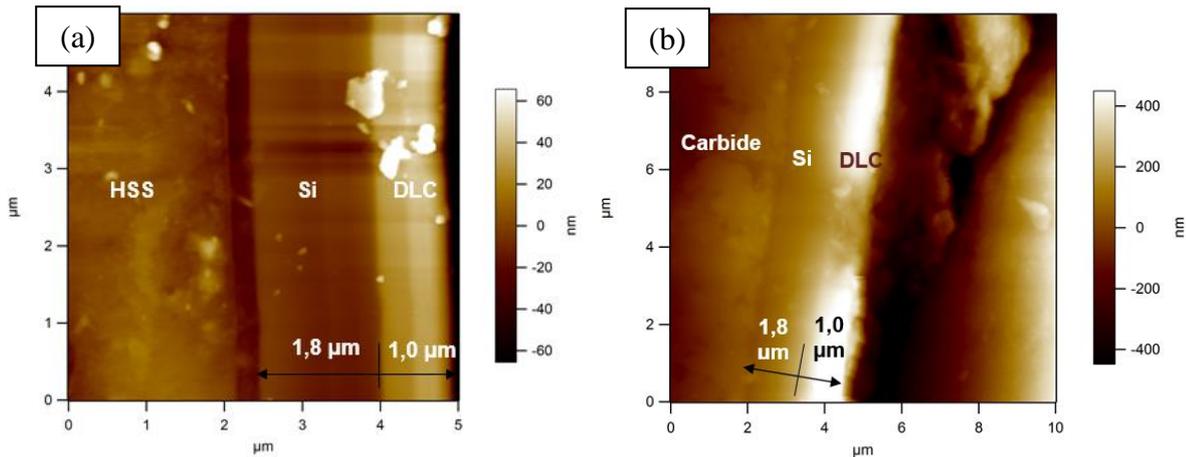


Figure 9. Topography of the (a) HSS/Si/DLC interface and (b) Carbide/Si/DLC interface (AFM).

The topography in both samples allowed to differentiate the Si layer in the coating. However, the better finish of the HSS sample leaves the topography at a sharper resolution, especially at the interface with the resin. The thickness value of the Si layer is approximately 1.8  $\mu\text{m}$  in both samples. This information indicates that the DLC layer has a thickness size smaller than Si, of 1.0  $\mu\text{m}$ .

### 3.3 Hardness and Young's modulus of DLC coating

The nanoindentation technique was chosen because it allows the accomplishment of measurements of surface hardness in the scale of nanometers with penetration depth of some tenth of microns, since the coating in study is very thin with 2.8  $\mu\text{m}$  of total thickness, being 1.8  $\mu\text{m}$  of Si and 1.0  $\mu\text{m}$  DLC, demonstrated in the AFM analysis. Since the aim is to analyze the characteristics of the coating, the maximum recommended indentation depth is 280 nm, being theoretically free from the influence of the substrate. The nanoindentation results for determination of hardness and modulus of elasticity are shown in Tab. 1, of which the method used to calculate the values was based on the method of Oliver and Pharr (Cooper *et al.*, 1994).

Table 1. Results of nanoindentation tests.

Sample	Máx. Load (mN)	Máx. depth (nm)	Hardness (GPa)	Young Module (GPa)
DLC/HSS	8	144 $\pm$ 11	42 $\pm$ 9	266 $\pm$ 43
	12	187 $\pm$ 21	36 $\pm$ 10	236 $\pm$ 65
DLC/Carbide	8	158 $\pm$ 9	26 $\pm$ 4	239 $\pm$ 30
	12	192 $\pm$ 7	27 $\pm$ 3	234 $\pm$ 23

The dispersion associated with the results for the WC-Co sample was lower than for the HSS substrates, their values of both hardness and modulus of elasticity being consistent with Lemoine *et al.*, 2008, which suggest the ranges of 10-30 GPa of hardness and 100-300 GPa of modulus of elasticity for the hydrogenated amorphous carbon (a-C: H).

### 3.4 Adhesion of DLC coating and substrate hardness

For the study of adhesive behavior, the Mercedes test to evaluate the mechanical behavior of coated samples was used. This test is based on a visual evaluation of the indentation formed on the surface of the sample, through a classification of standard VDI 3198 (Figure 4).

The indentations generated in the tests for the DLC coated HSS and WC-Co drills can be seen respectively in the Figure 10 (a) and Figure 10 (b), both of which are classified in the HF6 standard (Figure 4), showing complete delamination of the film. That is, adhesion is shown low even with the presence of deposited Si middle layer.

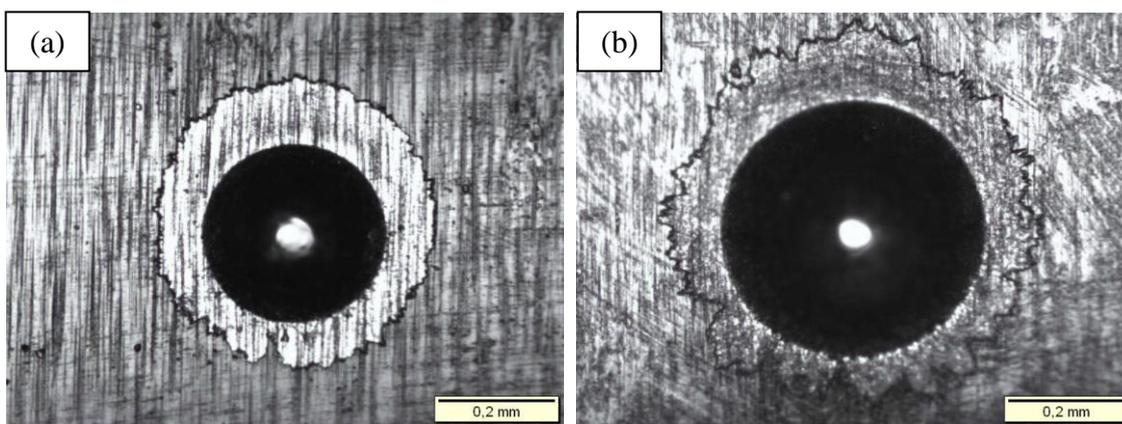


Figure 10. Visual of indentations on the surface (a) HSS and (b) carbide drill. In both is possible to see complete delamination of the coating around the indentation.

Through the microindentations performed, the values of the substrate hardness were determined. Two different measurements were taken: one on the substrate and another on the coated substrate. The values found are listed in Tab 2. The hardness values measured directly on the substrate and on the coating, did not show any significant difference, since the coating has very thin thickness, the top indentation is indifferent to the presence of the film.

Table 2. Hardness of substrate with and without coating

Sample	HSS		WC-Co	
	Substrate	Coated Substrate	Substrate	Coated Substrate
Hardness HRC	64,3	63,4	78,2	77,7
	64,9	62,5	79,0	77,7
	64,8	62,9	78,9	77,6
	64,8	63,2	78,8	78,8
Average	64,7	63,0	78,7	77,9

The hardness values found in the nanoindentation, compared to the substrate, correspond to approximately 5 times the WC-Co (~7.6 GPa) and 2 times the hardness of HSS (~ 13.4 GPa).

### 3.5 Wettability

During the measurements, the analysis program already provides an average between the measurement of the right and left contact angle of the drop. The reading rate was 1 measurement per second for 12 seconds, totaling 12

measurements for each face of the two samples (HSS and WC-Co). This procedure was repeated 3 times for each face. To obtain a final value of the angle, the average of the 3 measurements that each submitted sample was performed.

For the HSS the mean angle was 82.6 and 71.9 for the face without and with DLC respectively. The shape of the drop can be seen in the Figure 11 .

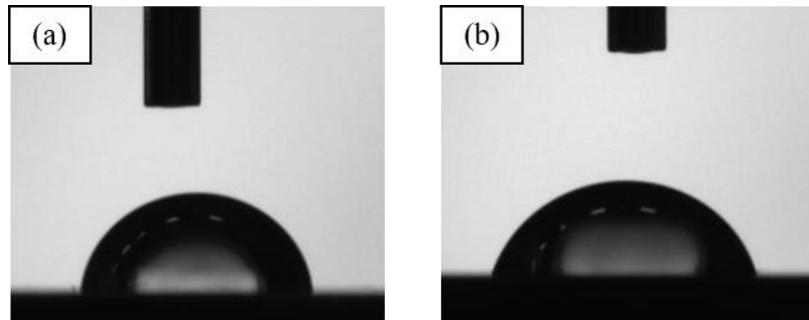


Figure 11. Shape of the drop obtained in the goniometer for (a) HSS and (b) HSS coated with DLC. Hydrophilic coating with respect to the substrate.

For the WC-Co the mean angle was 60.3 and 64.8 for the face without and with DLC respectively. The shape of the drop can be seen in the Figure 12.

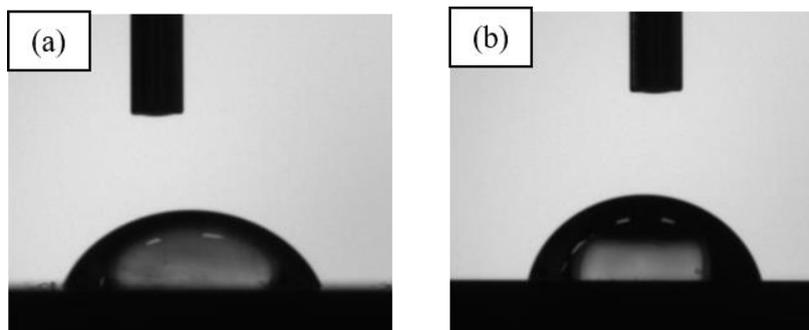


Figure 12. Shape of the drop obtained in the goniometer for (a) WC-Co and (b) WC-Co coated with DLC. Hydrophobic coating with respect to the substrate.

For all samples, the surface has hydrophilic behavior, contact angle  $<90^\circ$  (Figure 5). However, for HSS, the DLC coated surface had a lower average angle than the surface of the substrate. WC-Co, on other hand, the DLC coated surface showed a larger average angle than the surface of the pure substrate. In general that the coatings and substrates will perform well with water-based cutting fluids.

#### 4. CONCLUSION

The methodology developed in the work showed that it is possible to perform characterization of the coating in the tool itself, reflecting the influence of the true conditions to which HSS and carbide substrate are exposed on non-flat surfaces.

Film peeling defects at the cutting edges occurred due to the lack of coating ability to accompany the cutting edge geometry of the tool in the deposition process. Silicon was essential to maintain adhesion on metal substrates, however the thickness found was higher than the manufacturer's expectation.

The hardness values of the coating, by nanoindentation, compared to the substrate correspond to approximately 5 times the hardness of HSS ( $\sim 7.6$  GPa) and 2 times the hardness of WC-Co ( $\sim 13.4$  GPa), showing the potential of the DLC coating to increase the hardness of materials with more accessible prices to the market, such as the HSS reaching values up to the hardness of WC-Co.

The results of a visual evaluation of the indentation formed on the surface by Mercedes test, through a classification of standard VDI 3198, showed complete delamination of the film DLC and low adhesion on both substrates.

For all samples, the surfaces had hydrophilic behavior, contact angle  $<90^\circ$ , by wettability test, which indicates good behavior with water-based fluids.

This work pointed out the importance of the substrate in the final characteristics of the film, because, although both HSS and WC-Co, coated under the same parameters, presented different characteristics quantitatively.

## 5. REFERENCES

- Bhowmick, S., Alpas, A.T., 2008. "Minimum quantity lubrication drilling of aluminum–silicon alloys in water using diamond-like carbon coated drills". *International Journal of Machine Tools & Manufacture*. Vol. 48, p.1429 – 1443.
- Chowdhury, S., Laugier, M.T., Rahman, I.Z., 2004. "Characterization of DLC coatings deposited by rf magnetron sputtering". *Journal of Materials Processing Technology*. Vol. 153–154, p. 804–810.
- Choi, J., Soejima, K., Kato, T., Kawaguchi, M., Lee, W., 2012. "Nitriding of high speed steel by bipolar PBII for improvement in adhesion strength of DLC films". *Nuclear Instruments and Methods in Physics Research B*. Vol. 272, p. 357–360.
- Cooper, C.V., Holiday, P., Matthews, A., 1994. "The effect of TiN interlayers on the indentation behavior of diamond like carbon films on alloy and compound substrates". *Surface and Coatings Technology*. Vol. 63, p.129-134.
- Dimigen, H., Hubsch, H., Memming, R., 1989. "Tribological and electrical properties of metal-containing hydrogenated carbon films". *Applied Physics Letter*. Vol. 50, p. 1056–1058.
- Ferreira, L. M. V., 2013. "Revestimentos Hidrofóbicos". 16 nov. 2014 <[http://run.unl.pt/bitstream/10362/11045/1/Ferreira\\_2013.pdf](http://run.unl.pt/bitstream/10362/11045/1/Ferreira_2013.pdf)>.
- Lettington, A.H., 1998. "Application of Diamond-Like Carbon Thin Films". *Pergamon*. Vol.36, p. 555-560.
- Lifshitz, Y., 1999. "Diamond-like carbon - present status". *Diamond and Related Materials*. Vol. 8, p.1659–1676.
- Nakahigashi, T., Tanaka, Y., Miyake, K., Oohara, H., 2004. "Properties of flexible DLC film deposited by amplitude-modulated RF P-CVD". *Tribology International*. Vol. 37, p. 907–912.
- Silva, W.M., Trava-Airoldi, V.J., Chung, Y.W., 2011. "Surface modification of 6150 steel substrates for the deposition of thick and adherent diamond-like carbon coatings". *Surface & Coatings Technology*. Vol. 205, p. 3703–3707.
- Sutton, D.C., Limbert, G., Stewart, D., Wood, R. J. K., 2013. "The friction of diamond-like carbon coatings in a water environment". *Friction*. p. 210–221.
- Suzuki, Y., Watanabe, M., Toda, T., Fujii, T., 2013. "Development of electrically conductive DLC coated stainless steel separators for polymer electrolyte membrane fuel cell". *Journal of Physics: Conference Series*. Vol. 441.
- Tuck, J.R., Korsunsky, A.M., Bhat, D.G., Bull, S.J., 2001. "Indentation hardness evaluation of cathodic arc deposited thin hard coatings". *Surface and Coatings Technology*. Vol. 139, p. 63-74.
- Vidakis, N., Antoniadis, A., Bilalis, N., 2003. "The VDI 3198 indentation test evaluation of a reliable qualitative control for layered compounds". *Journal of Materials Processing Technology*. Vol. 143-144, p. 481-485.
- Weissmantel, C., Reisse, G., 1979. "Preparation of hard coatings by ion beam methods". *Thin Solid films*. Vol. 63, p. 315–325.

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