Surface Characterization of DLC Layers PVD Coated on AISI 52100 Steel Substrate

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Abstract. The purpose of this study is to evaluate the influence of substrate and layer deposition parameters on the morphology, topography and adhesion of the DLC layer. The carbonic layer was deposited on the rotary hook body made of AISI 52100 steel. Using the PVD magnetron sputtering technique, the deposition was performed at three different temperatures. Information about the coating was collected using optical, SEM and AFM microscopy and XRD. The DLC hardness and adhesion to substrate was determined. Comparing substrate hardness values, DLC adhesion and topography, it turned out that the best wear resistance was exhibited by the layers deposited at 180 °C.

1. Introduction
The hook body, the bobbin case, the cover ring, the needle guard and the leaf spring are the main components of the rotary hooks. The hook’s role is to form and to realize the stitch (Figure 1).

The large number of rotations that rotary hooks runs during the sewing process, bigger than 2500 rpm, leads to a strong wear stress between the parts being in movement and also between the thread and the hook. During the sewing, the hook comes in constant contact with the thread, thus forming a friction coupler (Figure 2). Some of the special sewing machines, for example those used for automotive airbags are designed to run without lubrication, or a minimal lubrication in the rotary hook area.

Figure 1. 3D model of the rotary hook. Figure 2. Wear traces left by thread on the hook.
Currently, one of the common steels used to make the hook body is AISI 52100, due to its high hardenability properties and wear resistance [1]. For this application AISI 52100 is used in hardened condition, 61-63 HRC (C-Rockwell units).

This hardness provides a satisfactory wear resistance. To increase the wear resistance, an a-c:H DLC layer thin film was deposited using the magnetron sputtering PVD method on the surface of the hook. DLC coatings are well known for their tribological properties (high hardness low friction coefficient) and are used in a wide range of industrial applications [2, 3]. The amorphous carbon layer consists of a mixture of sp² structures with sp³ structures, the proportion in which they are found determines the physical and chemical characteristics of the material [2]. The presence of hydrogen in the DLC layer gives a better behaviour reducing the friction coefficient [4], and increases the adhesion to the substrate [5]. Hardness characteristics of the substrate are influenced by the DLC coating temperature.

A high deposition temperature leads to lower substrate hardness, phenomenon that can reverberate on the adhesion of the coating to the substrate [6, 7].

It has been analysed how the DLC deposition temperature influences the substrate properties, with the purpose of identifying the combination between substrate hardness and DLC layer properties, which can ensure the optimal tribological behaviour of the hook body.

2. Materials and method

For the heat treatment of AISI 52100 steel, a batch of 30 samples (Ø 40 x 15 mm) divided into 3 series of 10 pieces was prepared. The chemical composition of the samples was verified using an OBLF QS 750 spectrometer (Table 1).

| Standard Symbol | C  | Si  | Mn  | P  | S  | Cr  | Mo  | Ni  |
|----------------|----|-----|-----|----|----|-----|-----|-----|
| ASTM A295-98   |    |     |     |    |    |     |     |     |
| AISI 52100     |    |     |     |    |    |     |     |     |
| Average values % | 0.875 | 0.247 | 0.388 | 0.011 | 0.025 | 1.588 | 0.037 | 0.163 |

Heat treatment was performed in a CBUT 10 batch atmosphere furnace, electrically heated. Samples were hardened and tempered for a hardness of 62-63 HRC (Rockwell Units).

Hardening parameters:
- austenitizing temperature: 820 °C;
- austenitizing time: 60 min;
- Carbon potential: 0.9 – 1.0%;
- quenching media: oil at 90 °C.

Tempering parameters:
- temperature: 180 °C;
- holding time: 60 min.

For the DLC coating, the specimens surface has been grinded, sand blasted with 40-60 μm diam. glass pearls, polished, cleaned and ultrasonic degreased, thus attempting to reproduce how the hook body is made under industrial conditions. The a-c:H DLC coating was performed using the magnetron sputtering (PVD) deposition technique in the Eifeler Vacotec Alpha 400C system at three different temperatures: 180 °C, 200 °C and 250 °C (Figure 3). For each deposition temperature there were used 10 samples, named series. Working pressure 3 mPa, argon consumption 45 l/charge. A small negative voltage (-35 V) has been applied to the substrates to increase ion assistance during coating deposition.

To investigate the morphology, and adhesion to the substrate, the DLC thin film was SEM studied in cross-section using the JEOL JSM-5200 microscope. The surface porosity of the DLC deposition was assessed, by atomic-force microscopy (AFM) measurements. The sp²/sp³ ratio was investigated.

Figure 3. a-c:H DLC coated sample.
by X-ray Diffraction – XRD and GXRD (grazing incidence) method. Adhesion of DLC coating to substrate was determined using a Rockwell tester (HRC) with a conical indenter with 150 kg load, according to the German standard “VDI Richtlinien 3198” [8].

3. Results and discussion

The results of the applied heat treatment were evaluated using OTTO WOLPERT HT1 Rockwell hardness tester (HRC) with 150 Kg load. The microstructure obtained after the heat treatment was investigated by optical metallographic microscopy using the optical METTALUX II microscope. Surface preparation for metallographic characterization was done using the LECO PR 22 cold mounting press and the JEAN WIRTZ sample polishing machine. For etching was used NITAL in concentration of 3% for 3-5 seconds. A fine-grained tetragonal martensitic microstructure, with uniformly distributed carbides, was observed (Figure 4).

![Figure 4](image.png)

**Figure 4** Hardened AISI 52100 micrograph of the sample 5.

In order not to contaminate the results and to avoid overheating, the samples were not cut with the cut-off machine, they were cut to the size of 15x15x3 mm by electro-erosion machining. DLC layers are clearly defined, adhesion with substrate is generally very satisfactory, a slight fracture in adhesion to the substrate was observed in the sample coated at 180°C. In the case of the DLC sample coated at 200 °C, SEM microscopy highlighted an area where the interface between the substrate and the carbon layer shows a substrate defect resulting from the sectioning and preparation of the sample (Figure 5b).

![Figure 5](image.png)

**Figure 5.** SEM cross-section microscopy of the sample X, DLC layer: a) coating at 180 °C; b) coating at 200 °C; c) coating at 250 °C.

The surface roughness and the morphology of the carbon layers was determined by atomic force
microscopy-AFM shown in Figure 6. It can be seen that the film surfaces are covered with oriented needle-like peaks. The surface of a-c:H film, deposited at 180 °C is more compact, uniform and smooth than the others.

![AFM micrograph of the sample 4:](image)

**Figure 6.** AFM micrograph of the sample 4:
a) coating at 180 °C; b) coating at 200 °C; c) coating at 250 °C.

The results of the determinations can be seen in table 2. It is observed that the roughness values of the DLC layer deposited at 200 °C and 250 °C as well as the general morphological aspect are very close. Better roughness is shown by the DLC coating at 180 °C.

### Table 2. Roughness parameters of the DLC layer.

| Sample                  | Surface  | RMS (nm) (root-mean-square roughness) | Ra (nm) (roughness average) | Surface Skewness (degree of bias of the roughness shape (asperity)) | Surface kurtosis (sharpness of the roughness profile) |
|-------------------------|----------|---------------------------------------|-----------------------------|------------------------------------------------------------------|------------------------------------------------------|
| DLC coating at 180 °C   | 20x20    | 12.64                                 | 9.40                        | 0.9656                                                          | 10.5636                                              |
| DLC coating at 200 °C   | 20x20    | 17.88                                 | 12.99                       | 0.9147                                                          | 6.7271                                               |
| DLC coating at 250 °C   | 20x20    | 16.48                                 | 12.17                       | 0.1947                                                          | 7.0981                                               |

Prior to the thin layer coating, the average value of the Ra roughness parameter on the surface of the steel substrate, measured with the Mitutoyo Surftest SJ-210 roughness tester, was 0.246 μm. The topography of a-c:H DLC coating is highly dependent on the surface roughness of the substrate and deposition parameters [9]. A significant improvement in roughness was found after DLC coating, phenomenon explained by the fact that during the coating process, at the beginning the deposit rate is higher on the peaks and valleys of the rough surface, then as the deposited layer gains in thickness, the surface roughness Ra begins to decrease because the deposition is more pronounced on the slopes and tends to fill the valleys [10].

To determine the DLC coating adhesion to substrate, a Rockwell tester (HRC) with a conical indenter and 150 kg load and was used to imprint the samples surface. It was measured the samples hardness (table 3), and the imprint shape was compared with the reference images designating six adherence indices symbolized from HF 1 to HF 6 [8, 11]. The method has some restrictions:
- the substrate hardness must be at least 54 HRC;
- the thickness of the layer may not exceed 5 μm.

For a thin layer to be considered as adhering to the substrate, traces left on the samples should not exhibit detachments and cracks of the layer around the imprint made by the diamond cone. Samples with adhesion index HF 1, HF 2 HF 3 or HF 4 are considered acceptable (figure 7).
### Table 3. Hardness values before and after DLC coating.

| Samples | Hardened and tempered at 180 °C (series 1) | DLC at 180 °C (series 1) | DLC at 200 °C (series 2) | DLC at 250 °C (series 3) |
|---------|------------------------------------------|--------------------------|--------------------------|--------------------------|
| 1.      | 62                                       | 61                       | 60                       |
| 2.      | 62                                       | 62                       | 59                       |
| 3.      | 62                                       | 61                       | 60                       |
| 4.      | 61                                       | 61                       | 60                       |
| 5.      | 30 pcs. corresponding to series 1, 2, and 3 | 61                       | 61                       | 60                       |
| 6.      | 61                                       | 61                       | 59                       |
| 7.      | 62                                       | 61                       | 60                       |
| 8.      | 62                                       | 62                       | 60                       |
| 9.      | 61                                       | 61                       | 60                       |
| 10.     | 62                                       | 61                       | 59                       |
| Av. value: | 62.5                                     | 61.67                    | 61.22                    | 59.78                    |
| Std. dev.: | 0.527                                    | 0.516                    | 0.422                    | 0.483                    |

**Figure 7.** Adhesion classification according to the six imprint shapes - VDI standard [11].

Imprint determinations according VDI standard, were performed on DLC layer deposited at 180 °C, 200 °C and 250 °C (Figure 8). For this purpose, the Rockwell tester was set at a load of 150 kgf and equipped with a diamond cone penetrator having a peak angle of 120°.
As it can be seen in Figure 8, the obtained imprints after indentation can be categorized as HF1 or HF2 with acceptable adhesion indices. Due to the amorphous nature of DLC deposition, no cracks or combinations of cracks and delamination of the thin layer were observed after the indentation. Microscope images showed only small delamination at the edges of the imprints, proving a good adhesion of the DLC layer to the substrate. Better adhesion to the substrate present the layers deposited at 180 °C, corresponding to HF1 adhesion index.

![Figure 8](image)

**Figure 8.** Imprint shapes of the sample 3: (a) coating at 180 °C; (b) coating at 200 °C; (c) coating at 250 °C.

The crystalline phase composition of the carbonic layers was determined by the X-ray diffraction (XRD). X-ray diffraction obtained on the DLC thin films using conventional θ/2θ scanning methods generally produced a weak signal from the film and an intense signal from the substrate, it can be noticed that X rays have penetrated the DLC layer and the substrate is seen; the peaks presented in Figure 9 come as a result. To avoid intense signal from the substrate and get stronger signal from the film, a GIXRD (grazing incidence) scan was performed, with a lower incidence angle for total reflection of the film without the substrate interference.

![Figure 9](image)

**Figure 9.** X-ray diffractogram of the 10 sample.
After the GIXRD, no obvious peaks can be observed (Figures 9 and 10), the peaks from the substrate have fallen (brown colour curve). Due to few crystallites in the DLC film, one discernible peak was noticed at 2Ө~ 45° that may correspond to a sp³ diamond plane.

![Graph](https://example.com/graph.png)

**Figure 10.** Comparison between GIXRD and symmetrical XRD patterns of the 10 sample - series 1.

### 4. Conclusions

The microstructure obtained after heat treatment consisting on fine-grained tetragonal martensite with uniformly distributed carbides highlighted by optical microscopy and hardness higher than 54 HRC provide wear and mechanical stresses resistance, being a good substrate for the subsequent DLC deposition.

A secondary tempering effect was observed after DLC, hardness values decrease depending on the coating temperature. SEM and XRD analyses revealed the amorphous character of DLC deposition. Surface roughness of the DLC layer is much lower compared to the roughness of the substrate prior to the coating.

Comparing substrate hardness values, DLC adhesion and topography, it turned out that the best wear resistance is exhibited by the layers deposited at 180 °C.

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