Experimental investigation on chromium-diamond like carbon (Cr-DLC) coating through plasma enhanced chemical vapour deposition (PECVD) on the nozzle needle surface

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Abstract

This study emphases the reduction of droplet defect in nozzle needle during the application of Cr-DLC coating on the substrate. The coatings were carried out in the highly sophisticated chamber with attached coating setup under the vacuum atmosphere. The materials were assessed in terms of their adhesiveness, hardness, wear resistance, and microstructural morphology. The dry wear test result reveals the appreciable outcome of nitrogen (N_2) flushing on quality of interfacial bonding. The indentation confirms the superior morphology of coated substrates but yet insufficient to resist crack propagation due to weak in adhesiveness of chromium (Cr) layer under non-N_2 flushing. Scanning electron microcopy (SEM) embedded with energy dispersive x-ray (EDX) was deployed for analysis of cross-sectional coated of substrate. The statistical results claimed that the samples rejection without N_2 flushing was showed up to 1.2 %. However, the results revealed that Cr-DLC coating with N_2 flushing reduced droplet defect and gain remarkable performance over a wide range of samples. Consequently, this novel methodology was employed to assess the coating performance by using a performance index based on tribological properties.

Keywords: Adhesion; Deposition; Droplet defect; Cr-DLC coating; N_2; Needle; PECVD.

1 INTRODUCTION

Diamond-like carbon (DLC) coatings are commonly used for tribological and thermal applications in automotive industries. DLC coating possesses excellent mechanical, thermal, and tribological properties. In addition, they have greater physical properties due to its superior properties of hardness, thermal stability, low friction coefficient, and excellent wear resistance. DLC also provides a good diffusion barrier to moisture, gas and excellent properties which make the DLC coating appropriate to be used for use in anti-wear field [1]. They are applied as wear protection coatings for specific components such as modern diesel injection systems. Thin-film coatings, deposited with the help of plasma technology, are very hard and suitable as wear-resistant. Intense plasma cleaning and ion bombardment during deposition result in formation of excellent adhesion and high density films. Thin-films are highly suitable for parts with low tolerances. The introduction of automotive coatings was started with the development of the high-pressure fuel injection pump. Due to increased injection pressures of up to 2500 bar (with operating temperatures to 300°C), an increase in the mechanical and thermal steadiness is required. Eventually, there, the common rail system became the ruling system since the constantly high pressure in the rails allows for multiple injections which lead to enhancement of fuel economy. These coatings may well contribute to reducing the CO_2 emissions, in turn, they lead to the development of environmentally friendly systems [2]. The main characteristic of DLC is represented by the ratio between the sp3 and sp2 bonded carbon atoms and the hydrogen content. They can possess high hardness, low coefficients of friction against materials such as steel, and are chemically inert [3-5]. The adhesive strength may have superior bonding, which allows an increase of the tribological characteristics [6-7]. Further, these coated surfaces promote a higher adhesive strength with an increased wear resistance corroborated with superior mechanical properties, corrosion resistance and bioactivity [8-9]. However, to avoid any weakness on DLC adhesiveness during coating of steel substrate, the chromium layer was considered as an intermediate adhesive layer. It was deposited between C-H and its substrate. To improve the quality of coating, a new methodology was developed for the selection of coating performance by using a performance index. It was observed that Cr-DLC coating shows better adhesion behavior with high carbon steel substrate [10]. The CrN single layer and Cr-CrN multilayer coatings were observed having no significant improvements in the tribological properties as that of uncoated carbon fiber reinforced epoxy composites using ball-on-disc testing. It is due to highest value of $\mu > 0.6$ in dry contact between AISI 5210 steel and Al_2O_3. However, Cr-CrN multilayer coatings with DLC top layers may significantly improve the tribological material properties. It allows obtaining maximum of sliding cycles, due to reduced shear forces with low-friction behavior of DLC-steel system [11].

Sui et al. [12] investigated the performance of a single and multilayer plasma which enables to enhance the chemical vapor deposition (PECVD) process. It forms the CrN/DLC/Cr-DLC having superior adhesive wear resistance as compared with the single CrN and DLC coating. The investigation revealed that the multilayer coatings were superior in toughness. It is caused by the very fine-grained that is in contrast to the single layer coatings, which contain coarse-grained structure. Also, multilayer PVD coatings provide high wear resistance and low friction coefficient. The friction coefficient of multilayer-coated surface is seven times lower as compared to single-layer coated surface and shows as well as a reduction in the adhesive wear, of multi-layer coatings, under sliding test against ISI 440C steel [12]. The dominant adhesive behaviour and thermal stability permits to increase the stress corrosion resistance at interface due to its characteristics, lubricity of DLC [1]. The PECVD-Si coatings on Ti substrate surface strongly adhered on the substrate and was noted having superior resistance to delamination [13]. However, surface defects of delamination and droplet defect on the coating process are necessary to be understood. In most cases, the type of coating process, thickness of coating, an ultrasonic vibration, spray flow rate and spray droplet size, a continuous thin liquid film and substrate temperature was revealed to influence the surface quality of the coating. The impingement of multiple droplets on a substrate using a spray coating endorse that the spray process is highly random and stochastic. There, a higher substrate temperature provides a more uniform and defect-free coating forms [13]. The thickness of the coating was over 35 µm which led to delamination defects, over the coating surface, and the results showed that the deposition rate may drive the quality of deposition [14].

In the light of all above literature review and in order to overcome the current quality of coating, in this paper, Cr-DLC coatings were produced. It can develop a superior adhesion DLC multilayer on the nozzle needle made of high carbon steel that is used as base material, commonly used for rail fuel injector. The injector has to assure the exact fuel metering by tight closing of the nozzle valve and has to operate with very high speed because of the multiple injections during an individual cycle of the engine. The objective of this study was to investigate whether the Cr-DLC dual layer coatings could reduce droplet defect in nozzle needle and to increase the coating hardness.

2 EXPERIMENTAL METHODS

2.1 Materials and coating deposition

The high carbon steel substrate was chosen as a nozzle needle material (substrates) with a dimension of 42 mm in length. The substrates are arranged in a tray and cleaned in an ultrasonic cleaner containing cleaning agent as Isopar-H for 15 min. Yet, in the case of mass production, the manufacturing rate will be higher for fully loaded system. For that, a substrate rotation table is developed; where its base is equipped with a vertical spindle distributed over the table circumference. The design of the table allows to form a satellite table, where the epicyclical gear drive system is located on the circumference of base table. The vertical spindle with substrate holders is individually driven by the planetary gears and are connected to a rotation axis under the table. Now, since the process is PECVD, the rotation of the table plays a very important role in the distribution of coating. The negative bias voltage will be connected to the substrate table through the rotation feed by software controller (fully or semi-automatic). It is necessary to load the chamber with the highest possible number of substrates to minimize the coating cost per substrate, as taking into account that all these substrates must be in compliance with the specification properties of the coating. Prior, the substrate is being loaded in the loading fixture followed by the table; the fixtures are being cleaned with the help of N_2 gas, also known as N_2 flushing. This process is carried out at a pressure of 5 bar with the help of a flushing gun. The sole purpose of carrying out this process is to flush out the loose as well as sticky carbon particles from the fixtures, which were deposited when the previous batch was being coated. It ultimately causes the non-reparable defect on the substrate called as droplet defect. Vacuum cleaning, post N_2 flushing, is done on the entire loading fixture and the table so as to suck the remaining loose particles of carbon. After these processes, the table and fixture are ready to load the substrate for coating. Now, once the batch is loaded in the coating chamber, the first step performed is the evacuation process, which is the vacuum formation process. The vacuum pressure maintained inside the chamber is of the order of 10^{-4} millibar. This process is followed by the heating and cooling of the batch for 2.5 h. Once the cooling is done, the fully automated coating chamber does the leak rate test, to ensure the complete vacuum inside the chamber. Then is conducted the most important processes before coating deposition taking place, called the etching process. The etching process is done with the assistance of plasma source, giving a line-of-sight effect for the argon (Ar) ions. The attraction of the argon ions enforces etching of the biased substrates. The Ar flow rate is set around 35 sccm. Once the etching of the entire batch is done, chromium (Cr) deposition starts taking place on the substrates, since, it acts as an excellent adhesion agent between the high carbon steel and the carbon layer.

This process is followed by transition-1 and transition-2 phases. Carbon (C) deposition starts through transition-1 phase, still having a majority of Cr deposition. In transition-2 phase, the % of C is higher than transition-1 phase and % of Cr which is lower in this phase as compared to transition-1. The sole purpose of these two transition phases is to progressively increase the deposition of C, followed by decreasing the deposition of Cr. Further, is conducted the final phase of coating, called as C-deposition, in which purely C-coating is done on the substrates, without the presence of Cr in. This entire coating process is carried out at a constant temperature of 150ºC and for a duration of 7 h. The deposition pressure is maintained in the range of 10-3 Pa. The Cr layer with a thickness of about 0.4 to 0.7 µm was deposited on the surface of substrates and followed by DLC coating at the above the Cr layer, with a thickness of about 1.7 to 2.2 µm. **Figure 1** shows the schematic representation of plasma enhanced chemical vapour deposition (PECVD).

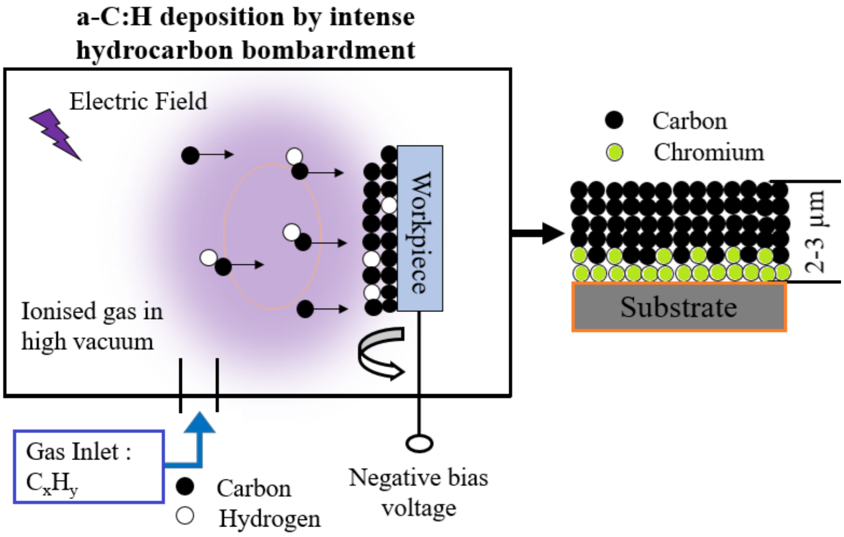


Fig. 1 Schematic construction of PECVD sputtering process

2.2 Coating characterization

In order to evaluate the adhesion quality, some disruptive tests were performed on the substrate samples. The strength of cohesiveness was obtained by penetrating the surface with a diamond indenter on a macro scale. The macro indentations were performed on rockwell hardness tester (Make: ATMO rockwell hardness tester, Model: BRIO) which is used for evaluating the hardness of substrate material, Cr layer, and DLC layer as well. The macro indentation was performed using a rockwell C hardness tester with a tip radius of 2 μm. Micro-abrasive wear test (calowear) using a simple ball catering method is generally used in tribological experiments for quality assessment of superficial up to 100 μm thick coatings. The thickness of coated samples was measured by calowear abrasion tester (Make: CSM instruments). It is a straight forward method to characterize the resistance to abrasion of a surface. The spherical ball of 30 mm diameter is kept on the guide of the needle with the help of suitable fixture and then the ball is rotated over it with a speed of 800 rpm for a span of 25 s. There, a small drop of diamond slurry (0.5 - 1 μm) is put on the spherical ball. The SEM and EDX analysis have been performed on the substrate to understand the surface characteristics and compositions of the coated substrate. The N_2 flushing was performed before and after each batch of coating to provide a quality coating.

3 RESULTS AND DISCUSSION

3.1 Morphology of coated sample

The coating was done at the guide and seat of nozzle needle by Cr-DLC layers via PECVD technique. However, the deposited coating was considered to be dense and the uniform. The needle was examined after coating using SEM morphology (**Figure 2**).

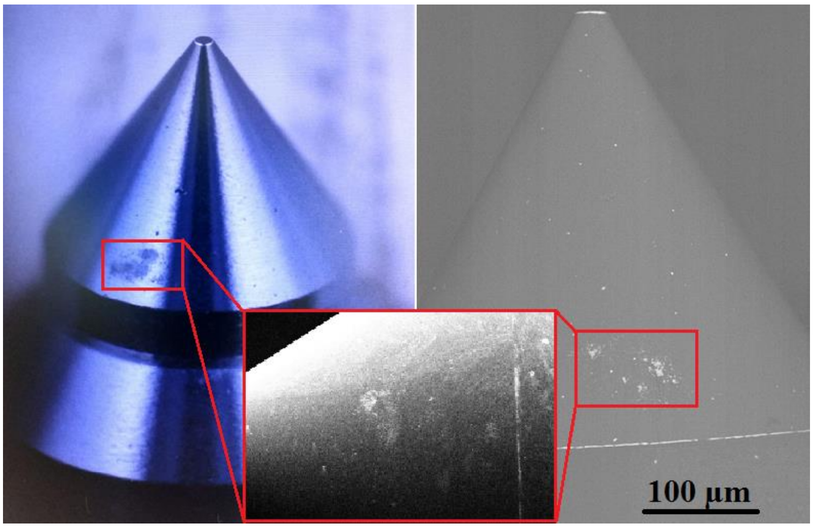


Fig. 2 Droplet defect on the surface of coated nozzle and view of defected portion using SEM morphology

It was observed that few white spots were agglomerated on the surface. Those are defects generated due to the improper deposition during transition layer after Cr deposition. Therefore, the rate of deposition was considered to be an important parameter which can affect the quality of deposition [13]. To understand the surface of white spots were conducted chemical composition scanning using Energy-dispersive X-ray spectroscopy (EDX) (**Figure 3)**. The EDX analysis has revealed that the spectrum 1 contains 24.70 wt.% C, 59.11 wt.% Cr and 10.13 wt.% Fe; spectrum 2 contains 50.23 wt.% C, 42.94 wt.% Cr and 4.86 wt.% Fe while spectrum 3 contain 24.93 wt.% C, 59.15 wt.% Cr and 10.16 wt.% Fe, respectively. It was observed that white spots were found having more concentration of Cr particles and the grey surface having an accumulation of C particle on the surface as shown in the **figure 3.** The occurrence of this type of defect may be due to the transition layer where Cr percentage became lesser than C, as poor adhesiveness or uneven coating on the substrate. Because, a single DLC coating may have poor adhesion with the substrates, a Cr layer is added to achieve better adhesiveness behavior with the steel substrate [1]. The presence of Fe in a higher amount of 4 wt.% could be related to delamination of coated particles. However, the deposition of particles was influenced by rate of deposition and surface condition before coating. Therefore, the N_2 flushing is considered as potential route to reduce these defects and maintain a proper flow rate of deposition.

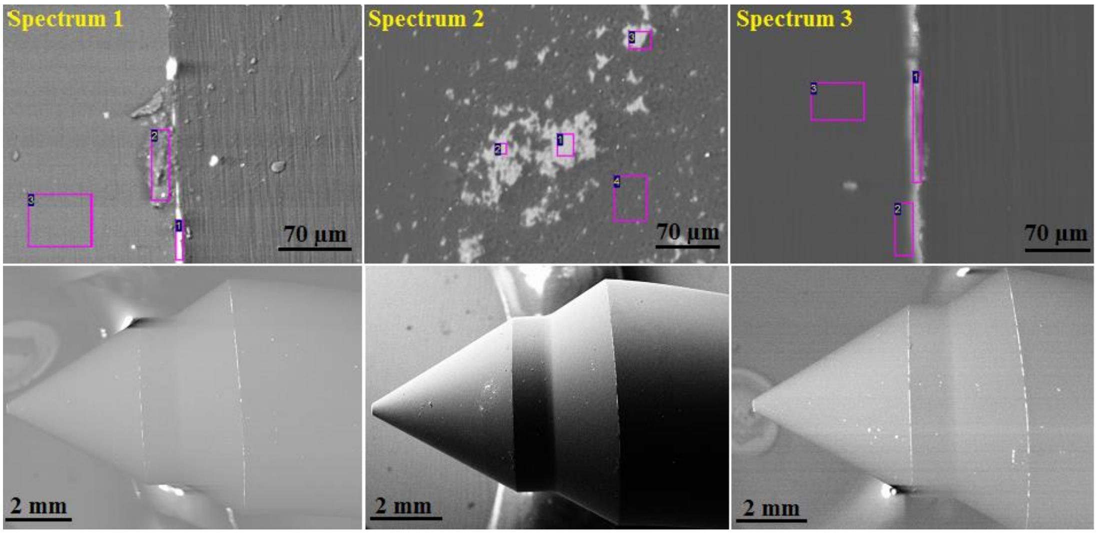
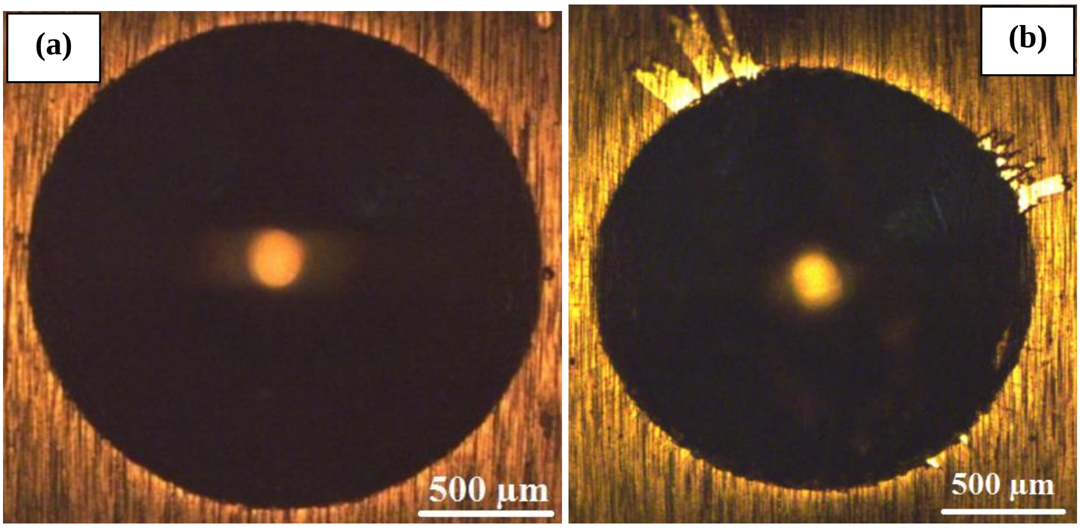


Fig. 3 SEM and EDX morphology showing the droplet defect on the surface of coated nozzle needle

3.2 Adhesive performance

The adhesive performance was examined through indentation technique. Here, the hardness was obtained by conducting trials, on three coated substrates from four different batches, by indentation using rockwell hardness tester as presented in **figure 4**. The adhesion trials made over the coating deposited without N2 flushing indicate that the Cr-DLC coating do not perform very well, hence, it demonstrates low resistance to crack formation as is shown in **figure 4(b, c, d)**.



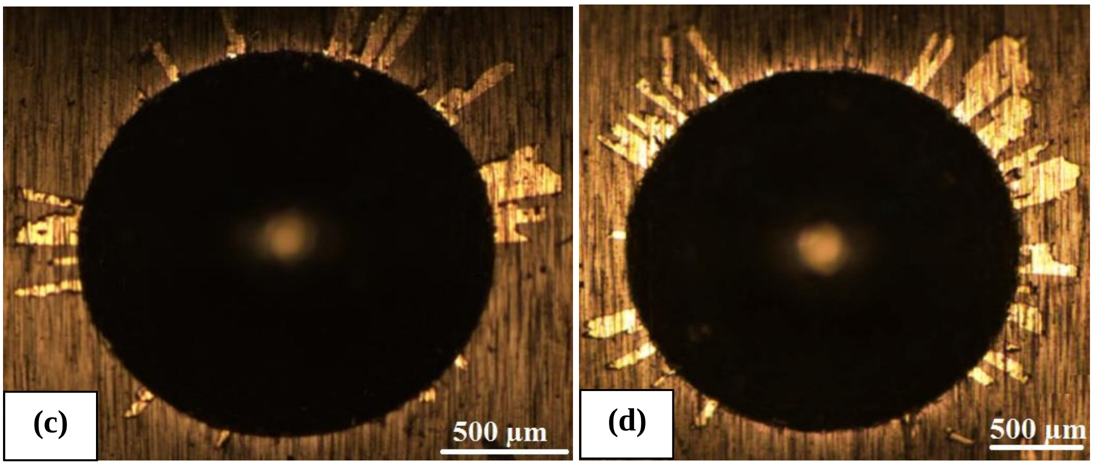


Fig. 4 Optical images presenting the failure patterns of the Cr-DLC coated nozzle needle after indentation: (a) with N_2 flushing, (b, c and d) without N_2 flushing

This observation made on the Cr layer can indicate the poor adhesiveness between Cr layer and C-layers when is associated to high value of load which is responsible for the adhesive failures of coatings [15]. The indented image of Cr-layered samples revealed fine transverse cracks in and around the indentation due to the high hardness and brittle nature of carbon. The result revealed that the hardness was around 60 ± 0.5 HRC. There was no significant difference of hardness, between uncoated and coated substrates. Nevertheless, the Cr-DLC dual layer coating had shown a superior indentation morphology **(figure 4a)**. The evaluation were performed on a scale from HF 1 (stated as very good adhesion) to HF 6 (insufficient adhesion) in accordance to VDI 3198 [16]. The **figure 4a** shows the absence of delamination that is associated to a strong interfacial bond. On the other hand, the identical type of coating produced on similar substrate (**Fig. 4b, 4c**, **and 4d**) indicates very poor adhesion and notable number of radial micro-cracks and delaminated [16-18]. Accordingly, it was noted that N_2 flushing is playing a significant role and enable an increased adhesiveness of interfacial bonding between Cr and its C substrate. In the as-deposited state, the indentation results indicate values in range of HF 2 to HF 3 for system A whereas an HF 4 for system B. They are both rated as acceptable adhesion quality with the gradient system being slightly superior. By the event of indentation, severe tensile stresses may be induced in the azimuthal direction

[16]. Due to this fact, the test cannot be treated as fully reliable adhesion evaluation. Therefore, the results should be considered with an appropriate degree of caution for Cr-layer thickness measurement.

3.3 Cr-layer thickness measurement

The calowear test has been used to assess the tribological properties up to 100 μm thick coatings. **Figure 5** shows the worn-out coated surface after calowear test. Wornout surface was clearly visible, in the direction of sliding, in the all test materials. Microcutting caused by the diamond slurry, on the test surface, was observed but no asperity deformation traces were observed. From the worn-out image, the thickness of Cr-layer was detected as t_{cr}= (D_3-D_2)/2 and C-layer, tc= (D_1-D_2)/2. It was observed that superior wear resistance is achieved in the case of effective flow rate and a proper N_2 flushing**. Figure 5** shows severe wear track due to weakness in the transition layer of the coated surface [19-20, 22-26]. It was revealed that the thin films exhibited a high friction coefficient. From these results, it is concluded that the wear rates of DLC films are influenced by the counter material [21, 27-29]. The adhesiveness of Cr with DLC cause a lower friction behavior due to superior bonding as presented in **figure 5**. The better quality of coating has confirmed the superior adhesiveness between the Cr-C: H layers. It provides as well as high tribological properties when is applied the nitrogen flushing. Similarly, were observed that the DLC specimen shown a prominence in C=O and COOH bonds and better corrosion resistance as that of Nitrogen-doped DLC specimen [30].

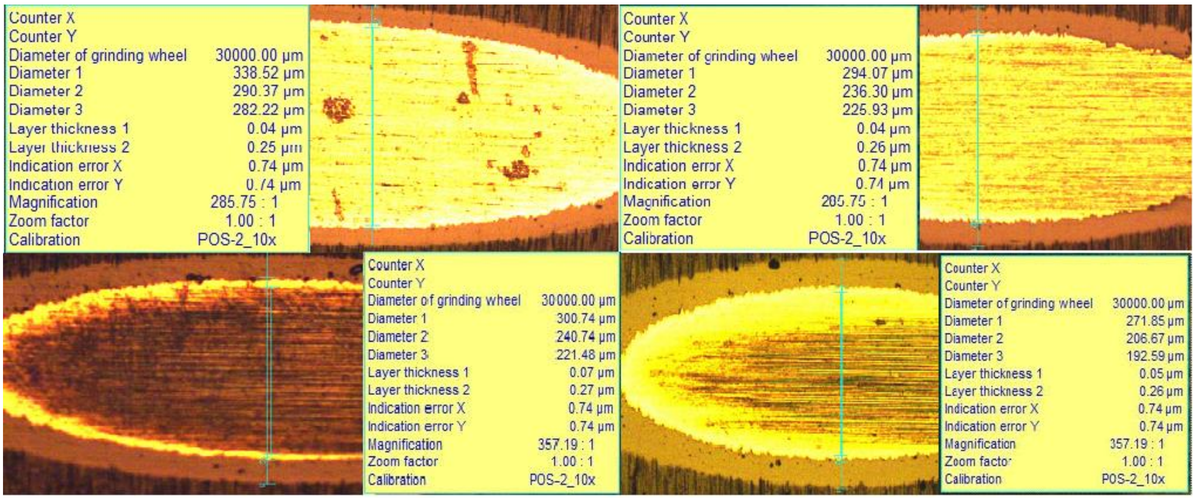


Fig. 5 The failure patterns on the Cr-DLC coatings deposited on high carbon steel after calowear test; with effective flow rate and with N_2 flushing (upper left picture); with uneven effective flow rate and without N_2 flushing

The cross-sectional view was observed having an uniform thickness/quality of coating on the substrates**. Figure 6** shows the cross-sectional view of Cr-DLC coated substrates. There, we can note slightly the weakness occurred at the interface between the Cr-layer and substrate C: H. The insufficient bonding that describe this interface can be responsible of premature failure in adhesiveness when submitted to heavy mass. The large portion of carbon coating on the Cr surface tends to have loose particles on the outside surface. The wear performance achieved on the DLC films, deposited on sintered specimen, is close related to an improvement on the adhesive strength. Adhesive wear and pull-out of hard particles were identified as the main mechanisms of the DLC coatings deposited on the as-sintered substrate [12, 31].

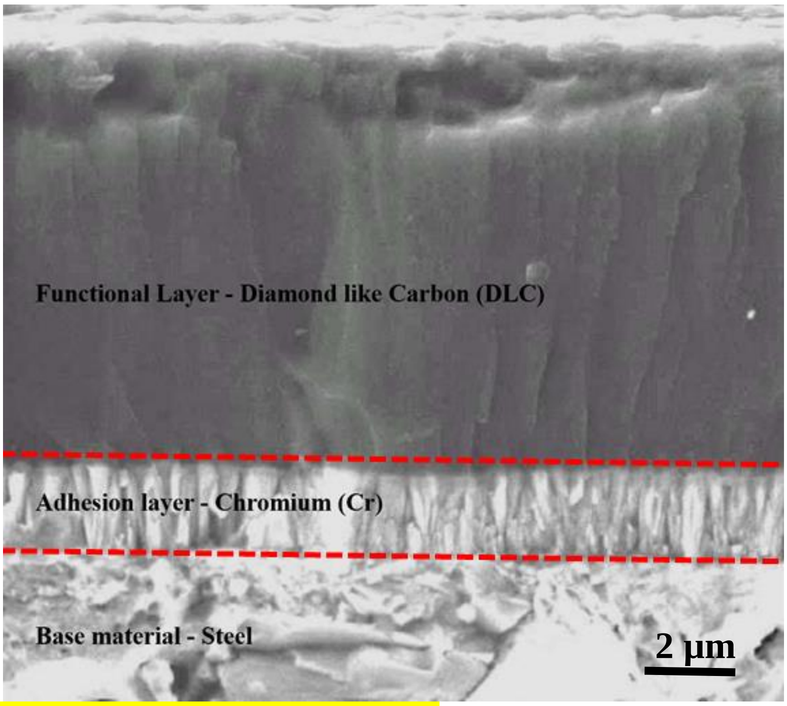


Fig. 6 The cross-sectional view of Cr-DLC coated substrates

3.4 Effect of N_2 flushing

Figure 7 shows the statistical analysis that has been performed for a period of 15 months. It indicates that the N_2 flushing as being a significant route to overcome the adhesives problem and defective outputs, which allows finally to improve the quality of coated substrates. The statistical data demonstrates that the defects rate achieved through N_2 flushing can be reduced to a minimum that is around 0.2 % of rejection. However, the rejections without N_2 flushing can be much higher, up to 1.2 %, and at the peak period when the requirement are higher (during months (March and October)), there, is a possibility to obtain even a more higher rejection rate. These observations clearly demonstrate the importance of N_2 flushing as parameter index to obtain a superior coating quality.

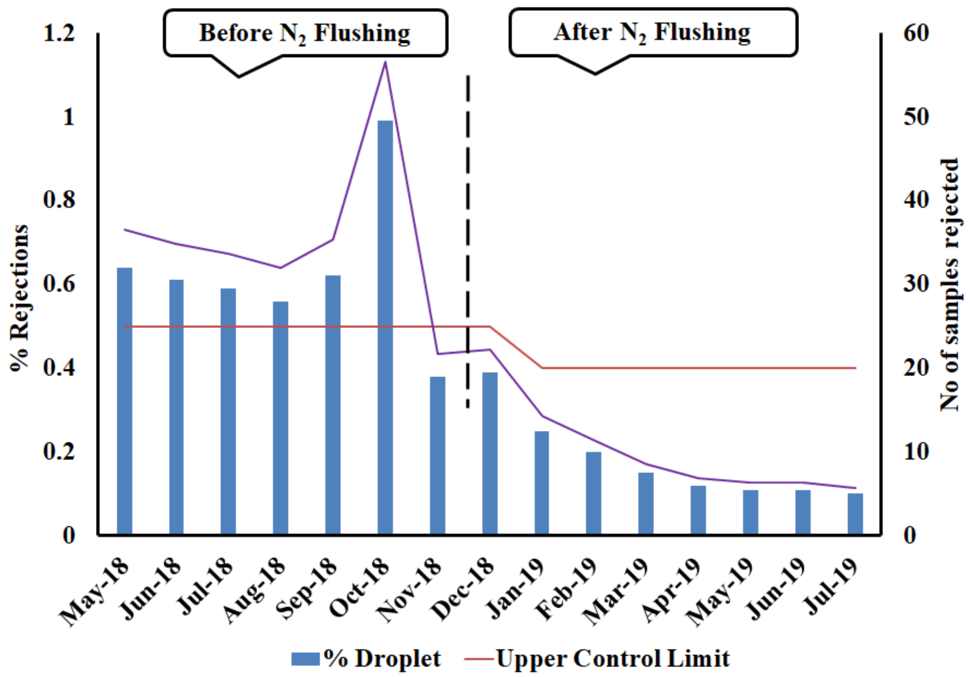


Fig. 7 The failure patterns on the Cr-DLC coatings deposited on high carbon steel after calowear test

4 CONCLUSION

In conclusion, a new experimental methodology with N2 flushing as a performance index to assess the quality of coating was proposed. The main findings are summarized as:

- The coating composition was developed to improve the coating adhesion on a high carbon steel and to increase progressively the coating morphology.

- The indentation result performed on the layer found between the Cr-layer and the DLC multilayer was not obtained successful, as there no measurable differences in hardness were obtained.

- The cross-sectional result shown that the interfaces between the Cr-based adhesion layer and the a-C: H is thermally stable.

- Wear test result endorse a superior interfacial bonding and effective thickness of Cr and C-layer.

- The contribution of Cr on coating performances was verified using SEM and EDX composition analysis. The EDX analysis confirms the weakness of adhesiveness cause of weaker Cr- layer.
- N2 flushing was witnessed to overcome defective outputs through statistical data.
- The overall results reveal that the Cr-DLC coating with N2 flushing abridged droplet and delamination defects. They have overall good performance over a wide range of samples, due to a combination of a thick DLC layer and Cr base which seems to be a good system with thermally stable and wear protection of steel substrate.

Conflict of interest

Conflict of interest: There is no conflict of interest.

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