Optimization and characterization of adhesion properties of DLC coatings on different substrates

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Abstract. The Diamond Like Carbon coatings (DLC) are gaining prime importance in the field of surface engineering especially cutting tools technology. The self lubricating property of these coatings makes them unique among other coatings like TiN, TiAlN, CrN etc. Unlike other coatings, DLC coatings give better surface finish and their self lubrication reduces the wear of a part to large extent. In present work, different substrates were selected to study the wear and adhesion behavior of DLC coatings. The coating was produced by physical Vapor Deposition (PVD) technique and the adhesive properties of DLC coatings were analyzed under ambient conditions using nano Scratch testing. Scanning electron microscope (SEM) was used to observe the scratches and their mechanisms.

1. Introduction

The great versatility of carbon materials arises from the strong dependence of their properties on the ratio of (graphite-like) sp² to (diamond-like) sp³ bonds in the films whereas amorphous carbon with a high fraction of sp³ bonds is named as diamond-like carbon (DLC) [1, 2]. DLC coatings have been the subject of extensive study since years because of their potential applications in science and technology. In science, the diamond-like materials preparation and their expanded applications is a subject yet to be explored. In technology, the diamond-like carbon (DLC) films are currently used in the production of hard coatings because of their extreme hardness, chemical inertness and excellent tribological, corrosion and adhesive properties [3]. The most important industrial applications of DLC films are wear-resistant coatings for computer hard-disks and optical coatings for the infrared and for the visible light range [4]. Moreover DLC coatings are being applied upon eye glass frames, pen and cell phone casings and chains because of their scratch resistance and pure black color [5].

Physical vapor deposition (PVD) is fundamentally a vaporization coating technique, involving transfer of material up to atomic level. It is an alternative process to electroplating technique. The process is similar to chemical vapor deposition (CVD) except that the raw materials or precursors, i.e. the material that is going to be deposited starts out in solid form, whereas in CVD, the precursors are introduced to the reaction chamber in the gaseous state [6].

Wear is the deformation and loss of a material in moving contact with another material. Wear and erosion of a film can be measured by: weight loss, material transfer and wear scars. It is extremely sensitive to the application, temperature, material etc [7-8]. The mechanical properties of films are important in their response during subsequent processing and to mechanical stresses [9-10]. The general method for the measurement of adhesion is the scratch test in which a diamond indenter is drawn across the coated surface and cohesive or adhesive behavior of the coating is assessed. The load
on the coated surface during the scratch test can be increased progressively and the load, at which the adhesive failure of the coating appears, is known as the critical load (Lc). Usually three critical loads are defined during a scratch test. First critical load (Lc1) defines the start of initial cracking in the coating surface. Second and third critical loads (Lc2 & Lc3) define the start of buckling by merging the cracks and complete failure of the coating respectively.

In the present study, steel substrates of different types were selected and DLC (diamond like coating) coating was deposited by the process of physical vapor deposition (PVD). Scratch tests were carried out at each coated surface to evaluate the critical loads in order to check which substrate gives the best adhesion with DLC coating. The scratch tracks were also analyzed under the optical microscope to check the adhesion mechanism.

2. Experimental Work

2.1 Sample Preparation

Die steel (D2), high strength steel (HSS), stainless steel (SS), high carbon steel, mild steel (MS), hard steel (H13) were chosen as substrates. Degreasing of the substrates was carried out through ultrasonic cleaning in which samples were dipped in CCl4 for 5 minutes. After that, samples were thoroughly washed with distilled water to remove the traces of CCl4. The samples were then placed in cleano gel solution at a temperature of 60°C for about 10 minutes. Finally the samples were cleaned in ultrasonic bath with acetone followed by careful transfer to coating chamber avoiding any contamination.

2.2 Chemical composition of substrates

The chemical composition analysis of the substrates ware carried out by optical emission spectrometer. The following table shows the chemical composition of the substrates used.

| Chemical composition of the substrates used for coating |
|--------------------------------------------------------|
| %C | %Mn | %P | %S | %Si | %Fe |
|-----------------------------------------------|
| Medium carbon steel                           | 0.403 | 1.388 | 0.066 | 0.022 | 0.312 | 97.680 |
| Mild Steel                                   | 0.193 | 0.664 | 0.084 | 0.027 | 0.258 | 98.470 |
| Die steel                                    | 2.358 | 0.563 | 0.061 | 0.021 | 0.643 | 91.415 |
| H13                                          | 0.951 | 0.280 | 0.004 | 0.016 | 0.233 | 97.184 |

2.3 Deposition of Coating

PLATIT π80 coating system was used to deposit DLC coatings upon the above described substrates. The system is fully automated and is controlled by the process computer. The coating deposition was conducted in a flowing pure nitrogen atmosphere. The cleaned coupons were mounted on carrousel substrate holders which rotate continuously around the vertical central axis at a speed of 12 rpm. During deposition, a negative DC bias of -70 V was applied to the substrates. The parameters are shown in Table 2.
Table 2. Technological Parameters of PVD Process

| Parameter    | Coat | Substrate Temperature (°C) | Nitrogen Pressure, (Pa) | Accelerating Voltage, (Volts) | Coat Period (min.) |
|--------------|------|-----------------------------|-------------------------|-------------------------------|-------------------|
| Set Value    | DLC  | 450                         | 1.5                     | 70                            | 120               |

2.4 Characterization of Coatings
The adhesion of the coating was assessed by micro scratch testing. Scratch testing was carried out by a Rockwell diamond indenter of radius 0.1 mm. Three scratches were placed at each load to ensure the accuracy of the results. Loads of 20 and 25N were used to place the scratches at each coated substrate. Multiple scratches of incremental loads were placed in which an area of the coated surface was scratched three times at the same incremental load. Pre-scan and post scans were also carried out that describe the surface profile before and after the scratch testing respectively. The initial load was set at 0.03N at which, pre-scan and post-scan were carried out. The loading rate was adjusted at the half of final load in each test. The length of all scratches was 3mm and the acoustic emission sensitivity was set at 7. All scratches were run at a speed of 1.5 mm/min. The contact force was kept same as the scanning load i.e: 0.03N.
These scratches were then analyzed through an optical microscope to define the critical loads and observe the mechanism of failure as well.

3. Results and Discussions
Figure 1 shows the optical micrographs of a complete multi scratch on DLC coated die steel at 20N load. The first micrograph depicts the region of first critical load where initial cracking in the coated surface appears. Second and third micrographs depict the appearance of first adhesive failure and the end of scratch respectively. It can be observed that there is no appreciable wear on the coated surface, which is the indication of good adhesion of the coating with the substrate.

Figure 1. Optical micrographs of die steel at 20N (20x)

Figure 2 shows the optical micrographs of die steel at a load of 25N and same magnification. It is clear from the micrographs that the wear is much more obvious in this case. But it is evident from the micrographs that there is still no appreciable wear on the coated surface and the traces of coating are still there even at the highest load. The dense cracking within the scratch is the result of excessive amount of load and also because the same area was scratched three times.
The figure 3 shows the comparison of the penetration depth curves for different substrates. It is obvious that mild steel shows the largest penetration depth. This is because the mild steel is the softest among all the substrates and the indenter easily penetrated the surface. Least penetration is shown by high strength steel (HSS) and die steel. This fact is associated to their high hardness and hence showed least penetration depth.

![Figure 3. Load versus penetration depth curves of different substrates at 20N load](image)

The figure 4 shows the acoustic emission curves for different substrates. It can be noticed that die steel and high strength steel show maximum acoustic emission, while H13 steel shows least acoustic emission. This might be because of the brittleness of hard steels, due to which coating produces the heavy noise when it delaminates.

![Figure 4. Load versus acoustic emission curves of different substrates at 25N load](image)
4. Conclusions
The comparisons of penetration depth and acoustic emission curves shows that high strength steel and
die steel show the best adhesion with DLC coating. Least adhesion is shown by the mild steel
substrate. It can be concluded that harder substrates show better adhesion properties with DLC
coatings than the softer ones. The softness of the substrate is also associated to lesser acoustic
emission intensity and vice versa.

5. References
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