Development of Implementable Diamond-like Carbon Coating for Improving Scratch, Wear and Optical Properties for Practical Industrial Applications

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Abstract

Diamond-Like Carbon (DLC) coatings have existed since many years mainly being developed at lab scale. Till date only cutting tools and certain automobile parts are being coated with DLC on an industrial scale. This is mainly due to the deposition of DLC coatings at temperatures greater than 300°C to achieve a particular hardness making it unsuitable for coating on cutlery, glass and plastics. This work deals with the research activity to coat high hardness DLC with hardness greater than 30 GPa at room temperature using Plasma Enhanced Chemical Vapour Deposition (PECVD) on various substrates like glass, plastics, steel etc. In the current work, several substrates like Stainless steels (SS), Poly Methyl Methacrylate (PMMA), Cutting tool materials, Glass, Carbon Steel, Germanium materials were coated at room temperature to observe adhesion and hardness of DLC on the above-mentioned substrates. Atomic Force Microscopy (AFM) has been used to study the plasma treated surfaces with various gases which have yielded in improved adhesion. It was observed that the adhesion of DLC was good and cleared the peel-off test as per MIL-C-48497A. The evaluated hardness of the DLC film deposited at room temperature exceeded 30 GPa when measured using Nano Indentation. Performance tests on Germanium for IR transmission with DLC coating was carried and found to be greater than 90%. Performance tests were also carried out on cutting tools and was found that DLC coated tools lasted 2.5 times more than tools that were not coated with DLC.

Keywords: DLC, AFM, Plasma Surface Treatment, Performance Test of DLC, PECVD.

1. INTRODUCTION

Diamond-like carbon (DLC) has been a widely researched topic since past two decades. DLC possesses properties that make it hard, wear resistant and have good IR optical transmission capability. In order to imbibe hardness in DLC, it is essential to heat the substrate during deposition to high temperatures as it leads to crystalline formation. However, in practical applications like coating on glass, plastics and other materials that are susceptible to heat, require DLC to be coated on room temperatures. Dekempeneer et. al [1] have deposited at 200°C which yielded a hardness of 14 GPa at 500 V bias. Kutsay et. al [2] have deposited DLC using RF-PECVD with hardness of 3.6 GPa. Fedonsenko et. al [3] have deposited DLC at 150°C at 400 W resulting in a hardness of 17.5 GPa. Kalin et. al [4] have used RF PECVD to deposit DLC and obtained a hardness of 22 GPa. Heeg, Jan et. al [5] have used a bias voltage of 100 V and heated to a temperature of 160°C to deposit DLC and obtained a hardness of 25 GPa. Safaie et. al [6] have deposited DLC using methane with oxygen incorporation using RF PECVD and a hardness of 19.6 GPa was observed. During deposition of DLC using methane as precursor gas yielded a hardness of 7.1 GPa. Ebrahimi, Mansoureh et. al [7] [8] have deposited DLC using acetylene gas and achieved a hardness of 16.23 GPa at 60°C was recorded. With the use of methane gas during deposition at 150°C, a hardness of 22.05 GPa was noted.

Yang, Won Jae et. al [9] have demonstrated that DLC exhibited a COF as low as 0.04-0.05. Ladgwig et. al [10] have observed a COF of 0.24 when DLC was deposited at higher temperatures. Xiao et. al [11] have deposited DLC using PVD and achieved COF of 0.067. Dai et. al [12][13] have observed COF by varying parameters during deposition of DLC using HIPIMS of 0.12 and 0.05. They have noted a peak hardness of 14 GPa when COF was 0.05. The authors also observed amorphous nature of the film using TEM studies. Bai et. al [14] have observed the critical load of DLC to be 90 nN using computer simulations.

This research comprises of the deposition of diamond like carbon films at room temperature using Radio Frequency based PECVD on various substrates for practical applications.

2. EXPERIMENTATION

The substrate was initially cleaned. Then, the sample was loaded in the vacuum chamber and cleaned using Argon and Oxygen plasma to remove contaminants on the surface that induce stresses in the film. RF-PECVD was used to synthesize the Diamond-like Carbon (DLC) films. Acetylene was used to deposit DLC films. Characterisation was done to qualify the quality of the DLC films.

2.1 Plasma Treatment

It has been observed in the past that it is extremely difficult to improve adhesion of DLC on various materials especially on Acrylates and Germanium substrates. Adhesion of DLC is possible only when the total internal stress during deposition of the film is reduced. This reduction can be achieved when the surface of the substrate is modified at the nanoscale. This is attained by bombarding ions of gases like Argon, Oxygen or Nitrogen using their respective gas plasma. In this work, the effect of Argon, Oxygen and Argon with Oxygen plasma on Poly Methyl Methacrylate (PMMA) has been observed under Atomic Force Microscope and the same has been discussed in the results and discussion section. It can be implied that by varying the time and power of plasma exposure to the substrates, the coating can adhere to the substrate. A typical work plan for plasma etching of PMMA has been given. The time for plasma treatment for Ar, O_2 and Ar + O_2 was varied from 5-30 min in steps of 5 min for each of the gas combination. By plasma treatment, usage of any intermediate

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buffer layer can be avoided thereby improving adhesion, transmission, wear properties of the film.

2.2 Deposition

After necessary plasma treatment, DLC was deposited with a flow rate of 300 sccm with a power of 200 W of a Plasma Enhanced Chemical Vapour Deposition. The time of deposition was selected based on the thickness of the film. It was found in previous experiments using ellipsometer that the rate of deposition for 300 sccm flow rate was 32 nm/min. Acetylene gas was used as the precursor gas for deposition of DLC.

The thickness of the film has been selected based on the application. In case of wear resistant applications coating thickness was maintained at about 1.2-1.5 µm. However, for optical applications thickness was calculated using $t=\lambda/4n$ where λ is wavelength region to be transmitted and n is the refractive index of the film.

3. RESULTS AND DISCUSSIONS

3.1 Scanning Electron Microscopy

Scanning Electron Microscope (SEM) is a useful technique to find out grain sizes in any morphology. DLC is mostly amorphous in nature and hence does not show any microstructure. From the SEM image (fig 1), it is also observed that the surface of DLC does not show any grain structure and has a smooth surface. It can be inferred that the carbon atoms are adsorbed on the surface of silicon forming high strength Si-C and Si-H bonds. [15]

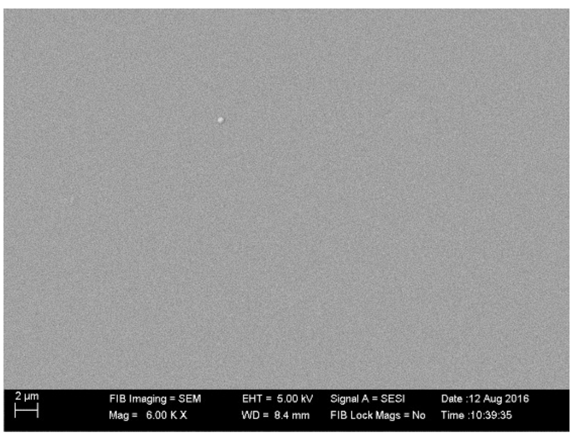


Fig.1. SEM image of a DLC coated surface

3.2 Atomic Force Microscopy

To demonstrate the effect of etching on adhesion on samples where adhesion of DLC is extremely difficult, Poly Methyl Methacrylate (PMMA) was chosen as the substrate material.

The main objective was to increase the bombardment using ions to create variations on the surface of the PMMA at nanoscale level in order to improve the adhesion. Later, PMMA was deposited with DLC. The same can be extended to several substrate materials in order to improve the adhesion. However, depending on the inherent hardness, crystal face or packing density of the material, the etching rate shall differ. Softer materials show higher etching with the above three plasma processes whereas harder materials show lower etching. Plasma from other gas sources and partial pressures within plasma could be varied to create a unique etch pattern on the surface of the substrate. It may also be realized that gases like Argon and Oxygen used for plasma etching have a different effect of creating an end dangling bond. This also enhances adhesion as several atoms latch on to these dangling bond sites to build the film. The graph in fig. 2 shows the change in surface roughness after various kinds of etching were performed on the PMMA sample.

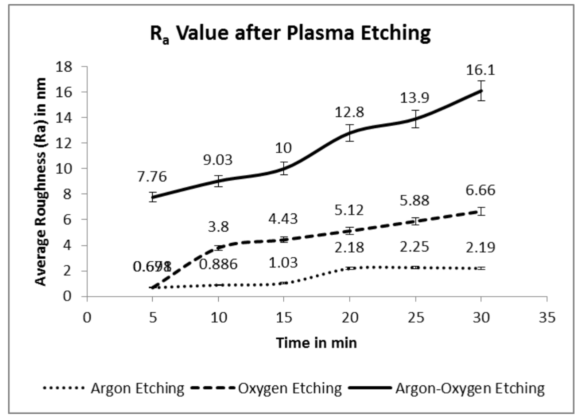


Fig.2. Average Roughness after Multiple Gas Plasma Etching

3.3 Nano Indentation

The hardness of the film was measured using Nano Indentation. Oliver and Pharr technique [16] was used for measurement of hardness of the film. The hardness of the DLC film was measured as 32 GPa when DLC was deposited at room temperature which is better than any of the values reported in the literature survey. The iterations of the measurement stabilized after 150 nm of penetration using a Berkovich indenter. From the load displacement curve (Fig. 3) it is calculated that the elastic recovery of the film is 75% and plastic deformation is 25%.

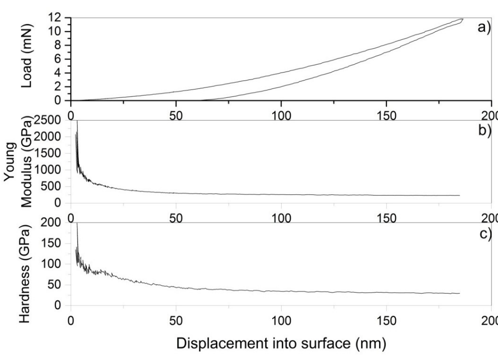


Fig.3. Nano indentation plot of DLC

A scratch test was also performed on DLC film and the average co-efficient of friction was observed to be 0.05 which matches to the values given in literature survey. The length of the scratch was maintained at 500 µm. The critical load was also measured as 222 mN which suggests decent loading capacity for a thin film. The image of the scratch from which the COF was measured has been given in fig 4.

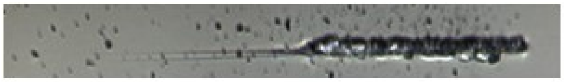


Fig.4. Scratch test of DLC film

4. PRACTICAL INDUSTRIAL APPLICATIONS

An attempt to utilize DLC films in real world applications was made. A few components having industrial/consumer utility were coated. Value addition of a few consumer/industrial components by using DLC films have been explained in the following subsections.

4.1 Kitchen Cutlery Knife

Fig.5. DLC Coated Kitchen Knife

A stainless steel kitchen knife was coated using DLC (fig 5) for to improve cutting life and enhance endurance of the product. As no industry specified standards were found, no testing has been performed on the component.

The coating thickness was roughly measured to be approximately 1200 nm. The steel was subjected to argon plasma treatment for a period of 5 min. This plasma treatment improved the adhesion of DLC as compared to the steel knife that was not treated with plasma.

4.2 Infra-Red Windows

Fig.6.a) Uncoated Germanium b) DLC coated Germanium

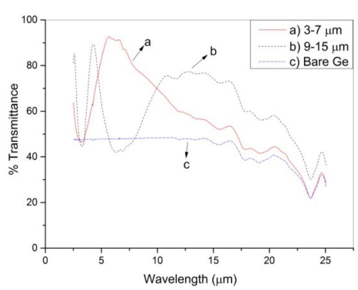


Fig. 7. FTIR Spectra of DLC coated on Germanium [17]

Germanium is used as an infra-red window for IR optics in night vision cameras (Fig 6). Bare germanium has 47% IR transmission, however as observed in this work, DLC improved the transmission efficiency to 93% (fig 7) when measured using FTIR for 3-7 µm wavelength in our previous work [16]. DLC also improves scratch and wear resistance when such optics is installed in vehicles used in desert and marine environment.

4.3 Float Glass

Float glasses are used as a protective layer in solar water heating or solar panels. When such panels are installed in areas that are prone to dry and dusty wind, scratches on the glass panel affect the ability to transmit light or heat. The DLC coated on glass has been given in fig 8.

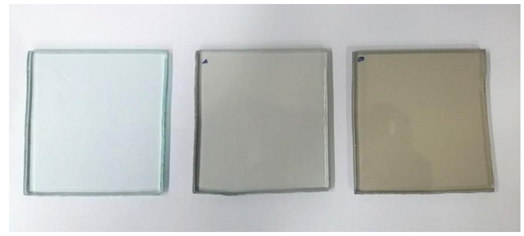


Fig.8. a) Uncoated Glass b) DLC Coated for 40 sec c) DLC Coated for 120 sec

In this experiment, DLC was coated after a very special surface modification of glass using oxygen and argon plasma for a period of one hour. This enabled the formation of free oxygen radicals present in the bond that allowed DLC to adhere to the glass substrate. Without the treatment, DLC was observed to peel off from the glass. The only drawback in the process was the discolouration of glass which may not be admissible for certain applications.

4.4 Poly Methyl Methacrylate (PMMA)

PMMA is used in biomedical components especially in dental and lens moulds. DLC offers wear resistance to such moulds. Acrylates are used in plastic casings of various electronic gadgets that are later dyed to achieve the desired colour. (Fig 9)

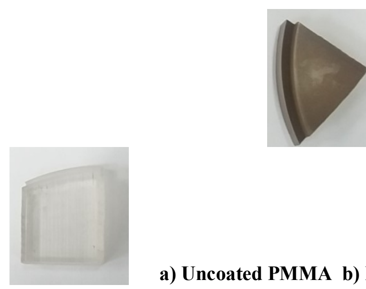


Fig.9. a) Uncoated PMMA b) DLC Coated on PMMA.

4.5 Machine Tool Guideways

Machine tool guide ways made of hardened steel was coated with DLC (fig. 10) to reduce friction as DLC has very low coefficient of friction of 0.05 and also reduces wear. This has the capability of eliminating the usage of grease and oil during sliding motion, thereby, reducing the hazardous environmental effects of these lubricants as well as their corrosive effect on other machine tool elements.

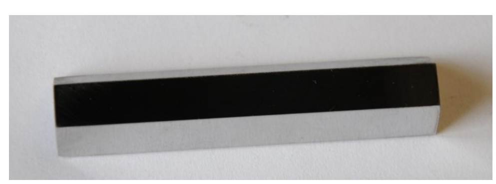
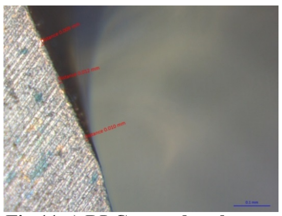


Fig. 10. DLC Coated Prismatic Guide way on Hardened Steel

4.6 Performance Test of Cutting Tools Coated with DLC

Titanium Aluminium Nitride end mill was coated with DLC (fig 11a) to determine the wear rate of the tool. It was compared with an uncoated DLC tool of the same end mill material. The parameters used for machining Cu-Be material are, Speed: 40m/min, Depth of Cut: 0.5 mm, Tool Used: TiAlN, Endmill Ø6 mm, Total cutting time: 35mins, Machine used: Schaublin milling machine. From the microscopy images given below, it can be observed the DLC coated tool wears out slowly when machined under same cutting conditions. It was observed that the peak flank wear on the tool coated with DLC was 10 µm whereas, for an uncoated tool, the peak flank wear was 26 µm (fig. 11b). The wear images were observed under light microscope and measured.



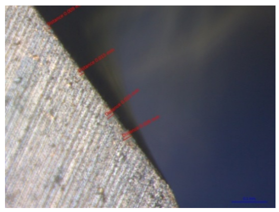


Fig.11.a) DLC coated tool Peak Wear 10 µm **b) Uncoated tool Peak Wear 26 µm**

7. CONCLUSIONS

Achieving high hardness at room temperature using PECVD as compared to existing literature is the novelty in this work. The hardest DLC was achieved when deposited at room temperature at 200 W powers with a hardness value of 32 GPa when measured using nanoindenation. COF was measured to be in the range of 0.04 – 0.06 when DLC film was deposited with a C_2H_2 flow rate of 300 sccm. Highest IR transmission achieved was 93% at 5 µm wavelength. Lowest coefficient of friction value of 0.05 was achieved. Adhesion methodology has been developed to improve adhesion of DLC films on various materials like PMMA, Carbon Steel, Hardened Steel, High Speed Steel, Soda Lime Glass, Germanium, Silicon etc. using plasma treatment techniques without the use of any buffer layer. It was also found that the tool wear reduces by a factor of 2.5 times with DLC coatings

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