Characteristics of Diamond-Like Carbon Coating Using Plasma Assisted Chemical Vapor Deposition on Steel Tool

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Abstract

High Speed Steel (HSS) tool is commonly used in engineering applications, especially as cutters. The shortfall of this materials are wear and corrosion. However, these can be reduced by coating the surface of the material. Therefore, the purpose of this research is to investigate the effects of Diamond Like Carbon (DLC) coating, quenching heating treatment, and tempering on the physical and mechanical properties of HSS surface. The physical characteristics which will be investigated is the micro structure, while the mechanical characteristics are hardness, wear and corrosion rate. HSS has variations in their chemical composition (% mass): 0.75–1.5 C, Co >12, V > 5, 4–4.5 Cr, 10–20 W and Mo. Furthermore, DLC coating uses Plasma Assisted Chemical Vapor Deposition (PACVD) technique with variation in the duration of coating (1,2,3,4,5 and 6 hours) at temperature of 300 ℃, with pressure variations of 1.0, 1.2, 1.4, 1.6, 1.8 and 2.0 millibar. DLC coating material be treated from methane or ethane gas, which is streamed into the fire with Argon (Ar). The result shows variations in DLC coating and the hardness grade depends on the coating time and pressure variation. DLC coating for a duration of 4 hours under 1.8 mbar pressure can reduce the surface hardness of HSS tool by 62% accompanied by increased ductility.

Keywords: HSS tool, Diamond like carbon, Surface hardness, PACVD

Abstrak

Perkakas dari baja kecepatan tinggi (HSS) umumnya digunakan dalam aplikasi teknik, terutama sebagai pemotong. Kekurangan bahan ini adalah keausan dan korosi. Namun, ini dapat dikurangi dengan melapis permukaan material. Oleh karena itu, penelitian ini bertujuan untuk menginvestigasi efek lapisan Diamond Like Carbon (DLC), perlakuan panas quenching, dan tempering pada sifat fisik dan mekanik permukaan HSS. Karakteristik fisik yang akan diinvestigasi adalah struktur mikro, sementara karakteristik mekanisnya adalah tingkat kekerasan, keausan dan korosi. HSS memiliki variasi dalam komposisi kimianya (% massa): 0,75–1,5 C, Co >12, V > 5, 4–4,5 Cr, 10-20 W dan Mo. Selanjutnya, lapisan DLC menggunakan Plasma Assisted Chemical Vapor Deposition (Teknik PACVD) dengan variasi durasi pelapisan (1,2,3,4,5 dan 6 jam) pada suhu 300 ℃, dengan variasi tekanan 1,0, 1,2, 1,4, 1,6, 1,8 dan 2,0 millibar. Bahasa pelapis DLC diolah dari metana atau gas etana, yang dialirkan ke api dengan Ar (Ar). Hasilnya menunjukkan variasi dalam lapisan DLC dan tingkat kekerasan tergantung pada waktu dan variasi tekanan lapisan. Pelapisan DLC selama 4 jam di bawah tekanan 1,8 mbar dapat mengurangi kekerasan permukaan alat HSS sebesar 62% disertai dengan peningkatan daktilitas.

Kata-kata kunci: Baja kecepatan tinggi, Diamond like carbon, Pengerasan permukaan, PACVD
1. Introduction

The research of the material surface coating using Diamond Like Carbon (DLC) has a essential conducted in engineering and medical sectors. The reason is DLC coating has high hardness, low friction coefficient, high wear resistance, biocompatible [1], and chemical inertness. Because of high resistance to corrosion, the DLC coating can be applied in the metallurgy sector, aeronautic and biomedical [2]. Moreover, It can be used on magnetic devices [3] and increasing fatigue strength [4].

DLC thin coating increases the corrosion resistance of orthopedic prostheses and suitable applied to the biological environment [5]. The material produces a high elastic modulus [6] and increases life period by 250% from drill bit on oil drilling [3].

Previous study show that coating the surface of tungsten (W) metal using interlayer [7]. The interlayers are Cr, CrN, CrNC, CrC or WC and DLC, which are also coated. The result shows that the low friction coefficient (of 0.19), low specific wear (8.36 x 10 mm/Nm), with a stiffness variation of 13.28 to 32.13 GPa. There is stiffness variation because of the tungsten (W) metal content and the variation in layer outcome. The stiffness and roughness of metal surfaces which gives DLC thin coating uses Plasma Enhanced Chemical Vapor Deposition (PECVD). The results depend on the parameters of the coating process [3, 8].

The adhesion force between Ti6Al4V metal and DLC layer can be increased using carbonitriding coating (20%C + 80% N2) on Ti6Al4V. Nevertheless, Carbonitriding process produces N2 diffusion on the Ti6Al4V substrate to bond better with DLC layer with a friction coefficient of 0.07 [6]. Also, The simple Electrodeposition technique uses a blend of nickel (Ni). DLC also increases graphitization from DLC layer and the particle from Ni-DLC can be a good distribute. It can also increase density with the timing process of coating. Nickel (Ni) has a strong capacity to reduce the residual stress, and increasing layer bonding of Ni-DLC with steel substrate [9].

According to the explanation above, it can be concluded that DLC layers can be applied in engineering sector. It relates to the needed characteristics such as hardness, high elastic modulus, friction coefficient, wear rate and low corrosion rate. The medical sector can also use it because of the biocompatible property of DLC layer [4]. The outcome properties depends on the materials which it is coated with or the base metal, coating technique and coating parameters such as work temperature, pressure, time of coating process, coating energy and the material source to get DLC layer outcome.

2. Materials and Method

The research materials used include High speed steel, Methane gas (CH4), Argon gas (Ar). While the tools used are CVD plasma, Polish, Hardness test, Wear test machine as well as optics microscope. Also, The range of plasma parameters are effective bias current Ipb peak (as displayed by the d.c. power supply): 0.7 - 4.0 A (pulse current density J = 1.5 to 8.0 mA.cm²). Maximum bias voltage 1000V, Negative pulse duration range of 5–1000s, Negative pulse pause duration range of 5–5000s. However, The pressure variations are 1.4, 1.6, 1.8 and 2.0 mbar with coating duration of 2, 3, 4, 5, and 6 hours. The research flow can be seen in Figure 1.

The test tools are obtained by cutting the materials according to the size and shape of the test standard ASTM E 384-99. Furthermore, the specimen surface was refined using sand paper and Autosol. The research was carried out in two steps. The first test used raw material such as HSS, while a specimen that has been coated with DLC was used for the second test. The test includes wear, hardness, chemical composition test, and micro structure observation using optic microscope.

The plasma system scheme is shown in Figure 2. In principle, the reactor consists of metal sheet in a row, which is in the vacuum space [10]. To obtain the plasma, two metal sheets are made in a row as an electrode system and is connected to DC tension using an anode grounded system. This reactor also consists of a heater system which works to heat the specimen. The temperature of the plasma tube keeps using a temperature control tool of 250-500 °C from the heater system, which is the optimal temperature needs of every application. On highest temperature, the hard surface outcome may be satisfactory, but it will change the dimension, decrease the maximum obtained hardness and reduce the roughness that can be obtained.
In the test, the specimen to be coated is used as the anode. After which methane gas and argon constantly flows to the vacuum space, and the plasma is obtained between a row sheet electrode on DC tension. Then the discharged electron will pound the methane gas until it become ionized. The ions of the methane gas attracts an electric field to the specimen, which then interact with the surface of the specimen.

![Diagram of research flow chart](Figure 1. Research flow chart)

![Scheme of CVD plasma system](Figure 2. Scheme of CVD plasma system: (1) Methane gas, (2) Heater, (3) Temperature control, (4) Vacuum pump, (5) DC high voltage, (6) Plasma tube, and (7) Anode/work piece)
3. Results and Discussion

3.1. Microstructure

The purpose of microstructure research is to study the shape and grain boundary of High speed steel that had been polished using sand paper made from aluminium oxide, water proof of 1500 size, rubbed with diamond pasta and etched with a nital 2%. The microstructure can be seen in Figure 3.

**Figure 3.** Microstructure of high speed steel

**Figure 3** showed that HSS microstructure consists of Martensitic needles in coarse austenite granules. This structure harden when heated above limit and Therefore, HSS material is able to maintain the hardness at a temperature of 300-700°C but cannot defend on ductility [11].

**Figure 4.** High speed steel microstructure on quenching

**Figure 4** shows HSS specimen that has undergone quenching treatment. It is very hard and has a martensit structure and fine grain. The presence of Martensit is because of quenching from Austenite temperature. The effect of quenching using oil causes carbon not to diffuse out but be obtained and trapped in a saturated solution until it changes shape.

**Figure 5** shows HSS micro structure which has been coated with DLC. HSS microstructure consists of Martensitic needles in coarse austenite granules. It has the ability to maintain it’s hardness at a temperature of 300–700°C. But it is brittle, so it cannot maintain its ductility [6].

The brittle characteristics of HSS material can be reduced using DLC coating process at a constant temperature of 300°C. The pressure variation starts at 1.4, 1.6, 1.8, and 2.0 mbar and coating duration of 2, 3, 4, 5, and 6 hours [12]. From figure 5 above, it can be explained that, with coating pressure of 1.8 mbar and 4 hours coating process, the granular form gets bigger and the material become ductile. Therefore, this is relevant to the stiffness test outcome that got hardness reduced to 702.10 VHN.

3.2. Vickers hardness test

**Figure 6** shows the highest outcome of hardness test of a specimen with a coating pressure of 2.0 mbar and 2 hours coating process, which is 921.40 VHN. The lowest hardness is observed on a specimen with 1.8 mbar coating pressure and 4 hours coating process, which is 702.10 VHN. It is accompanied by an increase in ductility of the specimen. **Figure 6** show that hardness on metal surfaces with thin DLC uses Plasma Enhanced Chemical Vapor Deposition (PECVD) technique. This depends on the parameters of the coating process [12].

**Figure 5.** Coating in high speed steel microstructure
3.3. Wears rate

Wear rate shows the amount of lost materials such as mass, volume or thickness on every unit length of sliding or unit of time. A specific wear can be measured with the width of wear of the specimen that is lost by the spinning component. The specific wears (Ws in mm/kg.mm) can be represented in Eq. (1) [10]:

\[ W_s = \frac{B \cdot b^3}{8 \cdot r \cdot P_o \cdot L_o} \]  

Where, \( W_s \) = wear specific (mm\(^3\)/kg.mm), \( B \) = disk width wearer (mm), \( b \) = wear width on specimen (mm), \( r \) = radius of disc wearer (mm), \( P \) = pressure index of wear (kg), and \( L \) = distance from the wear process (mm).

Figure 7 shows that the highest wear occurred on the specimen that was coated with DLC under 1.6 mbar pressure and coating process of 4 hours which is 0.166 Ws. However, The lowest wear occurred on specimen coated with DLC under 2.0 mbar pressure and 4 hours coating process which is 0.010 Ws. This is in accordance with the outcome of the hardness test, which was coated with DLC under 1.4 mbar pressure and 2 hours of coating process which shows the highest hardness value of 921.40 VHN. Therefore, This outcome is relevant to the research of [11] coating HSS metal surface using DLC. The outcome shows that the test specimen has low coefficient of friction (0.19), low specific wear (8.36 x 10 mm/Nm).

4. Conclusion

This research shows the variation value of DLC layers hardness and the relationship between the duration of coating process and pressure variation. DLC coating process of 4 hours and 1.8 mbar pressure reduces the hardness of HSS tool surface by 62% with an increase in ductility. In DLC coating process of 4 hours and 1.8 mbar pressure, the shape of the granules becomes large and the material becomes ductile. The time of coating process and the optimum pressure required to obtain the highest hardness, and the lowest wear rate from HSS using DLC is: 3–5 hours coating process and 1.8 mbar pressure.

Symbol

| Symbol | Description |
|--------|-------------|
| \( b \) | Wear width on specimen (mm) |
| \( B \) | Disk width wearer (mm) |
| \( J \) | Pulse current density (mA.cm\(^2\)) |
| \( L \) | Distance from the wear process (mm) |
| \( P \) | Pressure index of wear (kg) |
| \( r \) | Radius of disc wearer (mm) |
| \( W_s \) | Wear specific (mm\(^3\)/kg.mm) |

Authors’ contributions and responsibilities

The authors made substantial contributions to the conception and design of the study. The authors took responsibility for data analysis, interpretation and discussion of results. The authors read and approved the final manuscript.

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