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Characteristics of Diamond-Like Carbon Films on AISI D2 Using a Gas Mixture of Argon and LPG

Abstract. Diamond-like carbon (DLC) is an essential material for the mechanical industry, especially in producing components of cutting tools, automobiles, and industrial equipment. On the surface of AISI D2, plasma -Liquified Petroleum Gas (LPG) is used to create diamond-like carbon (DLC) films. The goal of the current research was to study the influences of pressure in the deposition process on the tribological and mechanical properties of the DLC films on AISI D2. The research results showed that by an increase in pressure of the process, the DLC film's hardness increased while its wear coefficient decreased. Characterization of the films was conducted using SEM (scanning electron microscope), Raman spectroscopy, and XRD (X-ray diffractometry). The SEM characterization observed the thickness of the film while the optical microscope showed wear debris and a transfer layer on the contact surface. The Raman spectroscopy showed two wavenumbers which were 1322 and 1577 cm-1. The downshift of the D and G peaks of graphite indicated the presence of DLC films. Based on the XRD analysis, five-strong peaks were seen in the spectra. We achieved a better tribological and mechanical performance for the DLC coating by adjusting the deposition pressure. These details have led to the conclusion that DLC films make an affordable coating material for car parts and cutting devices.

Keywords: AISI D2; *Chemical vapour deposition*; *Diamond-like carbon*; *Liquified Petroleum Gas* (LPG); Ratio sp3/sp2

1. Introduction

Microelectromechanical system (MEMS) is one of the miniaturization products of micro-forming (Vollertsen et al., 2009). Electronic equipment, sensors, and hard disk devices are few examples of MEMS products. According to MEMS Pressure Sensor Market and Technology, the demand for MEMS has grown over the past five years (Lin et al., 2017). In producing MEMS, accuracy is required to obtain results with high precision. The precision of the MEMS product is greatly influenced by the MEMS die itself. The obstacle faced by microforming die is the high value of wear resistance. This is due to the fact that a smaller mold size results in a higher coefficient of friction which increases friction output. (Saotome and Iwazaki, 2001).

Tool steel is increasingly used in machinery and other applications, including machining technology for MEMS. In general, the properties of steel such as tensile strength, resistance to wear, and corrosion resistance are required for machining technology (Abdul et al., 2021). Many tool steels are used as dies for microforming, one of which is AISI D2 (Kara et al., 2020). This is because AISI D2 has a good ability to withstand frictional forces, but in the fact, AISI D2 has a bad life time (Saotome and Iwazaki, 2001). The bad life time of AISI D2 is due to the small size of the microforming dies causing a large friction coefficient value. The greater the friction coefficient value, the greater the force required, resulting in damage to the dies (Sulaiman et al., 2019).

In the industrial world, various improvements in material science to manufacturing techniques especially for microforming are urgently needed due to current technological

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advances (Kosasih et al., 2023). Thin film coating is one of the material processes (Al Hijri et al., 2022). Therefore, thin coating techniques are used to protect the material from an abrasive environment, which can improve the material's mechanical properties and appearance (Modabberasl et al., 2019). Due to its low coefficient of friction, low hardness, low chemical stability, low optical transparency, low electron affinity, and high electrical resistivity, diamond-like carbon (DLC) coating is given special attention (Bewilogua and Hofmann, 2014). DLC is carbon with an amorphous form having a large percentage of sp3 bonds (Tyagi et al., 2019). It is usually applied in optoelectronic devices, automotive parts and tools, protective coverings for tribological or chemical applications, coatings for castings or moulds, and biological components(Kashyap and Ramkumar, 2022). DLC coating has been one of the most interesting research topics in the fields of materials science and structural engineering over the past decade (Thirumalai et al., 2018, Dalibón et al., 2017, Solis et al., 2016). Several technologies for preparing DLC have also been devised, including plasma-enhanced chemical vapour deposition (PECVD), magnetic filtered cathodic arc, magnetron sputtering, and ion beam deposition (Nakao et al., 2018, Wang et al., 2017). All of them have their own advantages, but the PECVD technology is the most popular because it does not need a high deposition temperature and it can deposit carbon on a variety of complex-shaped components (Xu et al., 2017). Plasma surface modification technology was indicated to resolve inner protection in recent decades because of its outstanding performance, including low friction, high density, and low energy consumption (Wang et al., 2019, Mariano et al., 2017, Xu et al., 2017, Pillaca et al., 2015). Recently, some researchers have discovered that various source gases can enhance the coating's tribological performance (Bewilogua and Hofmann, 2014). When a DLC thin film is produced using plasma discharge, the gas usually used is a mixture of hydrocarbon gas and argon (Ar). Benzene (C_6H_6), butane (C_4H_{10}), propane (C_3H_6), ethane (C_2H_6), and methane (CH_4) are some of the most commonly used hydrocarbon gases (Bewilogua and Hofmann, 2014, Erdemir et al., 2000, Erdemir et al., 1999).

The die life of AISI D2 can be increased through coating using DLC film. Therefore, this study applied DLC coatings to AISI D2 substrates by investigating the influence of pressure gases in the deposition process with the mixture of argon and liquified petroleum gas (LPG) through PECVD technology. The main purpose of current study was to examine the influence of pressure of the deposition process on the mechanical and wear performance DLC on AISI D2. The coating morphology and microstructure were characterized using SEM, Raman spectroscopy, and XRD to determine the behaviour of DLC coatings deposited under different pressure. This study is new and has not been reported by previous studies.

2. Methods

2.1. Materials

15 mm in diameter and 5 mm in thickness, respectively, AISI D2 materials were used as specimens. The specimen surfaces were then polished with metal polish after being polished with 100-to-5000-mesh abrasive paper. This study used a plasma glow discharge device system developed at the National Research and Innovation (Indonesia). The hydrocarbon source was a mixture of argon and LPG containing up to 80% propane (C_3H_8) which is produced by PT PERTAMINA in Indonesia. The ratio of argon to LPG in the DLC coating was 5:1(v/v), argon serves as a carrier.

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2.2. Experimental set-up

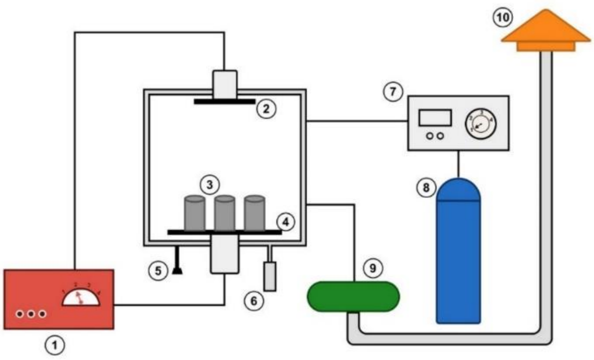

Figure 1 Schematic diagram of plasma deposition for diamond-like carbon. Information: 1) DC/RF power supply, 2) Anode, 3) Specimen, 4) Cathode, 5) Grounding, 6) Pirani Gauge, 7) Control, 8) Source Gas, 9) Rotary Pump, 10) Exhaust

As illustrated in Figure 1, the deposition system used in this study consisted of 4 components: 1) the gas supply system, 2) the deposition chamber (pipe), 3) the voltage source, and 4) the vacuum-exhaust system. This gas system consisted of 2 inlets namely LPG gas and argon gas. The deposition chamber was built using stainless steel with a height of 40 cm and a diameter of 32 cm. An anode and cathode system was placed in the deposition chamber. The vacuum system was generated from an Edward E2M2 rotary pump with a capacity of 2.8 m^3/hour (0.1 Pa) which was equipped with a Pirani meter series 1001 vacuum pressure measuring device. The high-voltage electrical system was designed with a high DC voltage of 1-20 kV with a current of 1-50 mA. In this study, the operating temperature was 450°C and the operating pressure was varied to 1.2, 1.4 and 1.6 mbar.

2.3. Experimental procedures

The specimens were inserted into the chamber and then the vacuum pressures were adjusted to 1.2, 1.4, and 1.6 mbar. Then, the input gas was ignited with a composition ratio of argon gas and LPG of 5:1 (v/v) until the desired temperature was reached (450°C). Once the temperature was reached, the deposition process was carried out for 4 hours.

2.4. Analysis

The film thicknesses were measured with HITACHI FLEXSEM 100 scanning electron microscopy equipped with energy dispersive spectroscopy (EDS) with a magnification of 3000 X, which was used to determine the elemental composition of material substrates following the deposition procedure. The characteristics of the film were evaluated using RAMAN iHR320 HORIBA with the laser at 514 nm in wavelength. The phase composition of the layers was investigated using a Cu K and PAN analytical AERIS X-ray diffractometer (XRD), in most of the cases it is sufficient to start around 10 or even around 100° in 2theta.. The tribological properties of the specimen were analysed using an Ogoshi High-Speed Universal Wear Testing**.** The wear rate of the films was measured as the amount of material removed per unit weight and sliding distance. The substrate's microhardness was measured using a Matsuzawa MMT-X7 microhardness tester with a 10 gf indenter load.

3. Results and Discussion

Figure 2 displays the coating film's XRD peaks. There were five diffraction peaks at 2θ values of 35.5, 45.3, 49.5, 65.5, and 82.7. In detail, three strong peaks dominate the XRD spectrum at 2θ, 45.3, 65.4, and 82.7, reflections from the [1 1 1], [2 2 0], and [3 1 1] diamond planes (Roy et al., 2002). Pang et al. deposited DLC films on Cu at low voltage (800-300 volts) through electrolysis of a water-ethanol solution and discovered three peaks corresponding to the diamond at 2θ values of 43.2 (the 111 diamond plane), 74.06 (the 220 diamond plane) and 89.9 (the 311 diamond plane) of diamond (Pang et al., 2010). Carbon may have contributed to the peak at 2θ of 35.5. The weakest diffraction peak at 49.5 of 2θ may be identified as graphite [102].

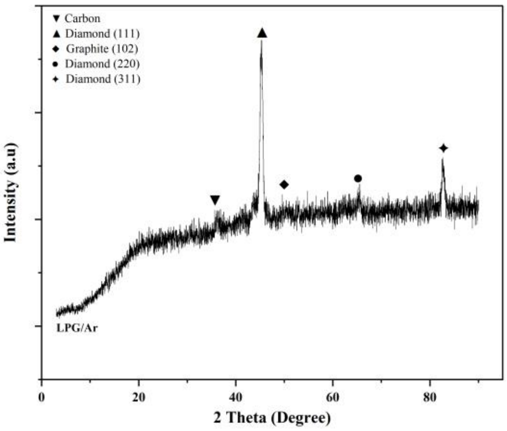

Figure 2 XRD for representative deposition

Figure 3(a) demonstrates that the lower the deposition pressure was, the higher the hardness would be. In this study, the optimal hardness (445.51 HVN) was reached at operating conditions of 1.6 mbar and 450°C. During the coating process, results in a higher hardness value and greater wear resistance at lower pressures. Therefore, higher performance will also come from a reduced gas pressure(Bewilogua and Hofmann, 2014). Because the atoms move more quickly at higher pressure, there will be more collisions. The larger the collision results in the deposition being inhomogeneous. This inhomogeneity causes the surface to have poor performance as indicated by the hardness value decreasing as the process pressure increases. Ward L used C_2H_2 gas as a carbon source in the coating process for 45 minutes. He discovered that an increase in pressure and bias voltage can accelerate the deposition rate and then result in a thick layer (Ward et al., 2014). In this study, after the DLC deposition process using the plasma method, the hardness value of the AISI D2 increased about 2 times, namely from 235 VHN to 448 VHN. Furthermore, based on a previous study, the DLC deposition has been carried out on AISI D2 substrate material using the Deep Oscillation Magnetron Sputtering (DOMS) method and resulted in a hardness increase of about 3 times (Ferreira et al., 2021). However, compared to the DOMS method, the plasma deposition method has several advantages, namely it is more environmentally friendly, cheaper and easier to use.

The Universal Wear Testing Machine was used to measure the wear resistance of DLC-coated and uncoated materials. Wear resistance is important for estimating material surface damage, especially caused by wear and tear. Figure 3(b) shows the results of wear tests indicating the CoF at various pressures. The coefficient of friction is a number that is the ratio of the resistive force of friction divided by the normal or perpendicular force pushing the objects together (Hakim et al., 2021). Based on Figure 3(b), Compared to layers with pressures of 1.4 and 1.6 mbar, the CoF value of the diamond-like carbon layer with a pressure of 1.2 mbar has the smallest value of 1.3 x 10^{-3}. The low CoF indicates that the diamond-like carbon coating's surface is becoming increasingly resistant to wear and strain. This is also impacted by the material's elevated hardness. This was because a pressure of 1.6 mbar resulted in the highest hardness value (Figure 3(a)). The higher the hardness value, the better the material was to withstand frictional forces, so the wear resistance value was smaller. The low hardness makes the wear debris on the contact surface penetrate and move, which leads to a lot of wear. The frictional oxidation process on the surface of DLC coating increased the coefficient of friction during the first stage of friction by converting C-C and C-H bonds into C-O bonds and C=C double bonds (Wang et al., 2019, Li et al., 2005). As a transfer layer formed between two contact surfaces, the coefficient of friction began to decrease. With continuous friction, the transfer layer achieved a critical thickness, resulting in a stable coefficient of friction. This indicates that machine parts, particularly those that come into contact with each other, can be coated with DLC. The decrease in the coefficient of friction increased the mechanical effectiveness of the machine, in a term of DLC-coated cast iron (Banerji et al., 2016, Suprapto et al., 2018). Based on this study, the DLC coating may be applied to friction machine components to extend their lifetime and mechanical efficiency, resulting in a more efficient operation due to a reduction in friction losses. The high mechanical

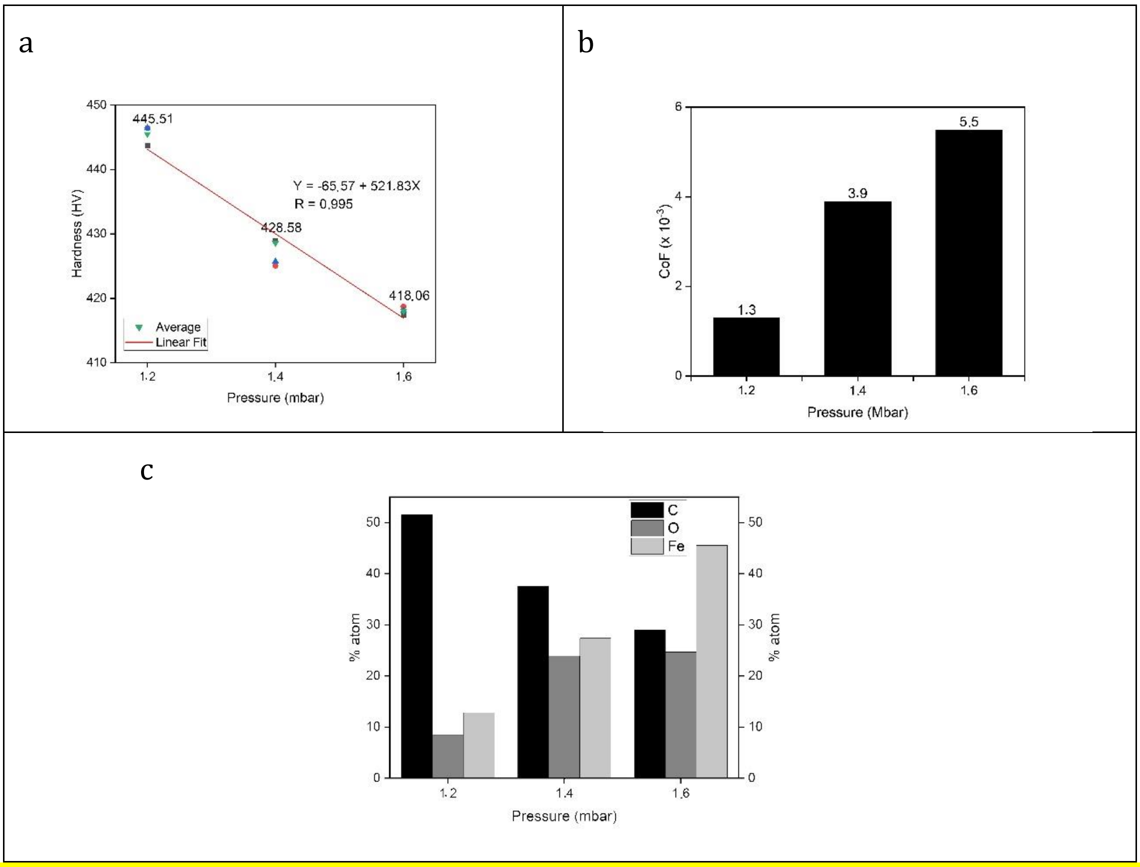

Figure 3(a) Microidentation of The DLC at Different Pressure **(b)** CoF of The DLC Various Pressure **(c)** EDX Chemical Composition of The Surface

properties and hardness of the DLC film are mainly responsible for its wear resistance (Huang et al., 2022). The low wear resistance value of the DLC layer was due to the inert properties of DLC and good adhesiveness. This causes when there is friction, only a little DLC is lost.

To determine the surface elemental composition of the formed diamond-like carbon layer, EDS characterization is carried out. This attempts to determine the effect of the pressure variations that have been applied. Figure 3(c) illustrates the results of an EDS analysis of a diamond-like carbon layer at pressures of 1.2, 1.4, and 1.6 mbar. This image represents the three most abundant surface elements, carbon, oxygen, and iron. Figure 3 (c) shows that the diamond-like carbon layer at a pressure of 1.2 mbar has a higher percentage of C atoms on its surface (51.15%) than at pressures of 1.4 and 1.6 mbar (37.54% and 28.96%, respectively). In accordance with Boyle's law, increasing the pressure parameter will have the effect of reducing the gas volume (Saepuzaman et al., 2019) . The volume of the gas has a linear effect on the deposition rate, so an increase in gas volume will increase the deposition rate. The deposition rate of this diamond-like carbon coating is directly proportional to the surface carbon atom density.

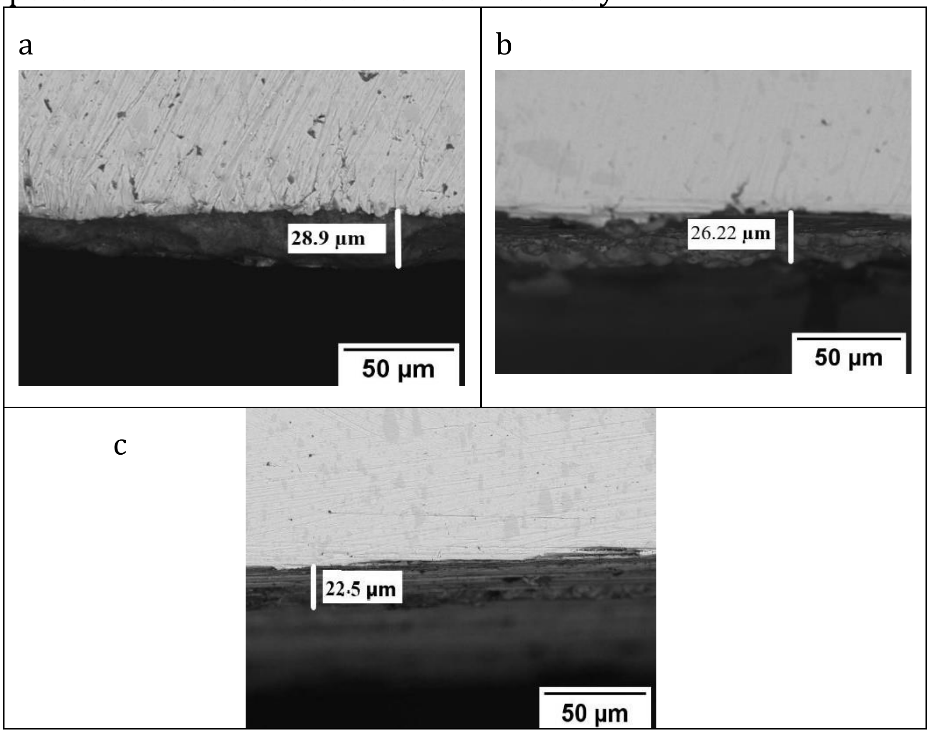

Figure 4 SEM images in cross-section of the DLC films at pressure **(a)** 1.6 mbar **(b)**1.4 mbar **(c)** 1.2 mbar

Determine the influence of pressure on the thickness of the diamond-like carbon coating, thickness tests were conducted. Figure 4 illustrates SEM cross-sectional characterization. Figure 4 demonstrates that a pressure of 1.2 mbar produces the greatest thickness of 28.9 m. This is influenced by the amount of carbon on the surface of the gas, as demonstrated by the EDS analysis in Figure 3(b) The percentage of carbon element composition on the surface of the diamond-like carbon layer at a pressure of 1.2 mbar is 51.15 percent, while the percentage of carbon on the surface of the diamond-like carbon layer at a pressure of 1.4 and 1.6 mbar is 37.54% and 28.96%, respectively.

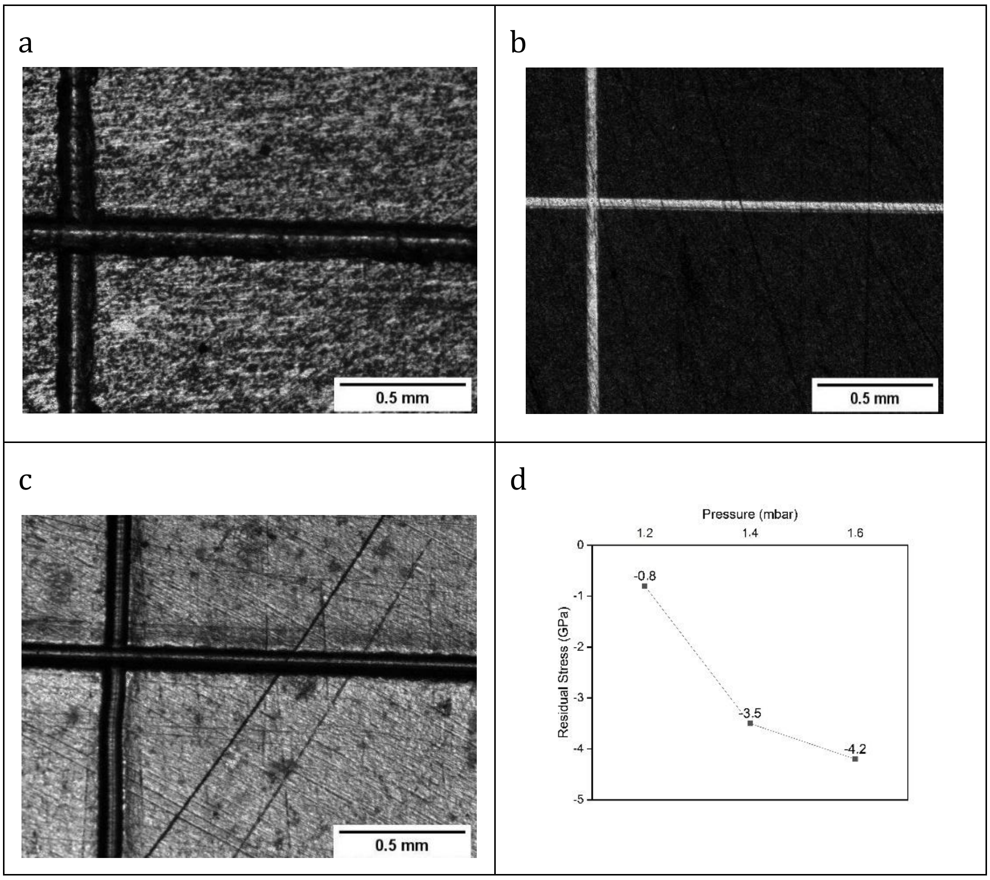

Figure 5(a) Cross Cut Morphology 1.2 mbar **(b)** Cross Cut Morphology 1.4 mbar **(c)** Cross Cut Morphology 1. mbar **(d)** Residual Stress

Coating adhesion tests were carried out to determine the effect of a diamond-like carbon coating with pressures of 1.2; 1.4 and 1.6 mbar. Figure 5 (a)-5(c) shows an optical microscope photo of the results of the adhesion test using the cross cut method according to ASTM D3559. Figure 5 (a)-5(c) indicates that the diamond-like carbon layer that is formed has good adhesion, as shown by the lack of peeling in the area around the intersection of the edge lines as well as in the cross section. This layer is classified as 5B, based on the fact that the cut edges are extremely smooth and there are no loose parts.The surface's residual stress indicates a distortion of the bond angle and length, which results in weak adhesion. The purpose of calculating residual stress on the diamond-like carbon coating is to quantify the adhesive strength. The lower surface residual stress value indicates greater adhesion. The layer of diamond-like carbon with a pressure of 1.6 mbar has the lowest value, -4.2. This indicates that the adhesion between the diamond-like carbon layer and other pressure variations is stronger. The ID/IG ratio has an effect on this and increasing ID/IG ratio will lead to an increase in the sp2 content. ID/IG ratio is the comparison of the intensities of peak D and peak G in the Raman characterization results. The more sp2 it consumes, the greater its adhesive strength will become. This is due to the fact that sp2 has a greater binding energy than sp3 (Sharifahmadian and Mahboubi, 2019). According to other studies, carbon with sp2 hybridization has less distortion, which can reduce the internal pressure of the diamond-like carbon layer so that the more graphitic layer increases the layer's adhesion (Wang et al., 2021).

The DLC bond structure was analysed using Raman spectroscopy. This spectroscopy is effective for investigating non-destructive carbon hybridisation (Khamnualthong et al., 2012). Figure 6 shows two bands at approximately 1322 and 1577 cm^{-1}, which correspond to the disordered graphite band (also known as the D-line) and the single crystal graphite band (also known as the G-line), respectively (Pang et al., 2010). It implies that the films are conventional DLC films with a mixed structure of sp2 and sp3. It is well known that the G and D bands generate up the typical Raman spectrum of DLC, with the G band centred at nearly 1550 cm^{-1} due to the symmetric E2g vibrational mode in graphite-like materials and the D band centred at nearly 1330 cm^{-1} due to the restrictions on the size of the graphite domain caused by grain boundaries or imperfections (Wang et al., 2017, Ferrari and Robertson, 2001). The Raman spectra in Figure 6 reveal that all samples of the films exhibit DLC-typical characteristics.

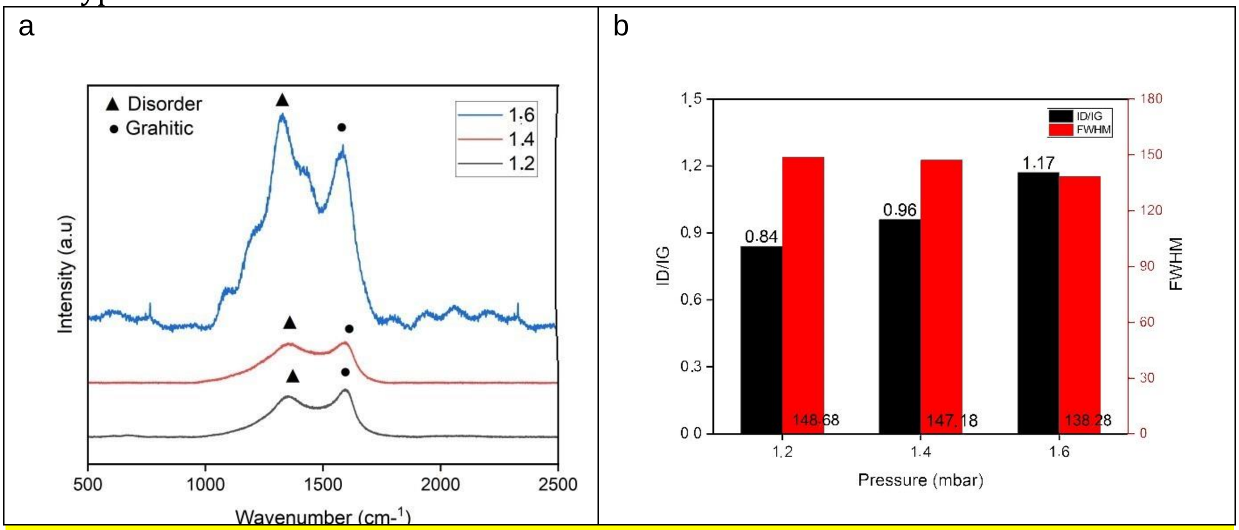

Figure 6(a) Raman Spectra for Diamond-Like Carbon Film (**b)** Relation Between G FWHM and ID/IG Ratio

The composition ratio of sp2 and sp3 hybrid carbon atoms significantly influenced the characteristics of the formed DLC layer. In the phase diagram of diamond-like carbon, the number of sp3, sp2, and sp formed in the DLC layer determined the types of carbon bonds (Furlan et al., 2013). The percentage of the relationship between sp3 and the structure of hydrogen bonds in the DLC layer was classified (Moriguchi et al., 2016). Various types of DLC can be categorized. When the percentage of sp3 is higher, the formed DLC layer gets near the diamond structure. According to the analysis of Raman spectra, the ratio of the intensities of the D and G bands (ID/IG) increased as the pressure rose from 1.2 to 1.6 mbar. The ID/IG values in this study were 0.84, 0.96, and 1.17, which corresponded to a pressure of 1.2, 1.4, and 1.6 mbar, respectively. This suggests that the ratio of sp3/sp2 increased and films became more diamond-like. The sp3/sp2 bonding ratio of the films was determined by calculating the ID/IG value. The higher the sp3/sp2 ratio was, the smaller the ID/IG ratio would be (Huang et al., 2022, Casiraghi et al., 2005, Robertson, 2002)**.** The G peak shows graphitic properties with C=C hybridization on diamond-like carbon [32]. This means that the smaller the FWHM value at G peak, the smaller the change to sp2 C=C hybridization as indicated by Figure 6 (b). Figure 6(b) also shows that the value of the ID/IG ratio increase with increasing pressure. This was because the gas pressure affected the gas input in the chamber, in which the lower the gas pressure at sample , the smaller the gas volume in the chamber, which caused less plasma gas to stick to the substrate. The fewer carbon sources were attached, the smaller the formation of sp3 and sp2 hybridization was, resulting in a smaller ID/IG ratio. The ID/IG ratio—which had previously been 0.95 in the Dalibon research—was successfully reduced by the coating parameter when 1.2 mbar of pressure was applied (Dalibón et al., 2017).

4. Conclusions

On the AISI D2 surface, DLC coatings were produced using the plasma technique at three optimal LPG gas pressures of 1.6, 1.4, and 1.2 mbar. An increase in deposition pressure from 1.2 to 1.6 mbar increased the coating's hardness, thickness, and uniformity in the axial direction progressively, but decreased the wear coefficient. Compared to layers with pressures of 1.4 and 1.6 mbar, the CoF value of the diamond-like carbon layer with a pressure of 1.2 mbar has the smallest value of 1.3×10^{-3}. The bands at 1322 and 1577 cm^{-1} in the Raman spectrum confirmed that the deposited film contained diamond-like carbons. Three intense peaks at 2θ 45.3, 65.4, and 82.7 corresponded to the [1 1 1], [2 2 0], and [3 1 1] diamond planes. By modifying the deposition pressure, we were able to produce a DLC coating with improved tribological and mechanical properties. Based on these facts, it has been determined that DLC films are suitable as an inexpensive coating material for cutting instruments and automobile components.

Acknowledgments

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