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Research Article

The Influence of Deposition Pressure from the Mixture of Argon and Liquefied Petroleum Gass on the Properties of Diamond-Like Carbon Films

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Abstract: Diamond-like carbon (DLC) is an important material for the mechanical industry, specifically in producing components for cutting tools, automobiles, and industrial equipment. The films of DLC can be formed on the surface of AISI D2 using plasma-plasma-liquified petroleum gas (LPG). Therefore, this research aimed to determine the influence of pressure from deposition process on the tribological and mechanical properties of DLC films on AISI D2. The results showed that an increase in the pressure led to an increment in the hardness and a reduction in the wear coefficient. Moreover, the films were characterized using scanning electron microscope (SEM), Raman spectroscopy, and X-ray diffractometry (XRD). SEM characterization was used to determine the thickness of the films while the optical microscope focused on wear debris and a transfer layer on the contact surface. Raman spectroscopy also analyzed the two wavenumbers which were 1322 and 1577 cm^{-1} with the downshift in D and G peaks of graphite applied to determine the presence of DLC films. Based on XRD analysis, five strong peaks were observed in the spectra. Furthermore, this research achieved a better tribological and mechanical performance for DLC films by adjusting deposition pressure, leading to the conclusion that the material could be made affordable for the production of car parts and cutting devices.

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

1. Introduction

Microelectromechanical system (MEMS) is part of the miniaturization products of micro-forming (Vollertsen et al., 2009) with a few examples reported to be electronic equipment, sensors, and hard disk devices. The information provided by MEMS Pressure Sensor Market and Technology showed that the demand increased over the past 5 years (Lin et al., 2017). In producing MEMS, the accuracy required to obtain high precision is significantly influenced by the die. This is because the micro-forming die is often affected by the high value of wear resistance associated with the higher coefficient of friction in smaller mold sizes, leading to an increase in the friction output (Saotome and Iwazaki, 2001).

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The use of tool steels in machinery and other applications, including machining technology for MEMS, is increasing. Moreover, properties such as tensile strength, resistance to wear, and corrosion resistance are generally required for the application of these steels in machining technology (Abdul et al., 2021). An example of the tool steels often used as dies for micro-forming is AISI D2 (Kara et al., 2020) due to the ability to withstand frictional forces. The disadvantage is the bad lifetime (Saotome and Iwazaki, 2001) caused by the small size of the dies which leads to a large friction coefficient value. A higher friction coefficient value needs more force which further damages the dies

In the industrial world, several improvements are urgently needed in the material science field concerning manufacturing techniques, specifically for micro-forming, due to current technological advances (Kosasih et al., 2023). An example of the process to be enhanced is thin film coating which is used to protect materials from an abrasive environment in order to improve the mechanical properties and appearance (Roza et al., 2024; Ningrum et al., 2023). Special attention is required on diamond-like carbon (DLC) coating due to the low coefficient of friction, hardness, chemical stability, optical transparency, and electron affinity as well as high electrical resistivity (Bewilogua and Hofmann, 2014). DLC is defined as carbon with an amorphous form and has a large percentage of sp3 bonds (Tyagi et al., 2019). It is usually applied in optoelectronic devices, automotive parts and tools, as protective coverings for tribological or chemical applications, coating for castings or molds, and in biological components 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). This has led to the introduction of several technologies for the preparation process such as plasma-enhanced chemical vapor deposition (PECVD), magnetic filtered cathodic arc, magnetron sputtering, and ion beam deposition (Nakao et al., 2018; Wang et al., 2017). All the technologies have some advantages but PECVD is the most popular because it does not need a high deposition temperature and can deposit carbon on different complex-shaped components (Xu et al., 2017). Furthermore, plasma surface modification technology was reported to have been effective in resolving inner protection in recent decades because of the outstanding properties, 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). Several sources of gases have also been observed recently to have the capacity to enhance the tribological performance of coating (Bewilogua and Hofmann, 2014). For example, the gas normally applied when DLC thin films are produced using plasma discharge is a mixture of hydrocarbon gas and argon (Ar). Moreover, 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)

The die life of AISI D2 can be increased through coating using DLC films. Therefore, this research applied DLC coating to AISI D2 substrates and investigated the influence of pressure in deposition process conducted using the mixture of argon and liquified petroleum gas (LPG) through PECVD technology. The main purpose was to examine the influence of deposition pressure on the mechanical and wear performance of DLC on AISI D2. Moreover, the morphology and microstructure were characterized using SEM, Raman spectroscopy, and X-ray diffractometry (XRD) to determine the behavior of DLC coating deposited under different pressures. This research was considered to be new and had not been investigated by previous scholars.

2. Method

2.1. Materials

AISI D2 materials with 15 mm diameter and 5 mm thickness were used as specimens. The surfaces were polished using 100-to-5000-mesh abrasive paper and metal polish. Moreover, a plasma glow discharge device system developed at the National Research and Innovation (Indonesia) was used. The hydrocarbon source was a mixture of argon and LPG containing up to

80% propane (C_3H_8) produced by PT PERTAMINA in Indonesia. The ratio of argon to LPG in DLC coating was 5:1(v/v) with the argon designed to function as the carrier.

2.2. Experimental set-up

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

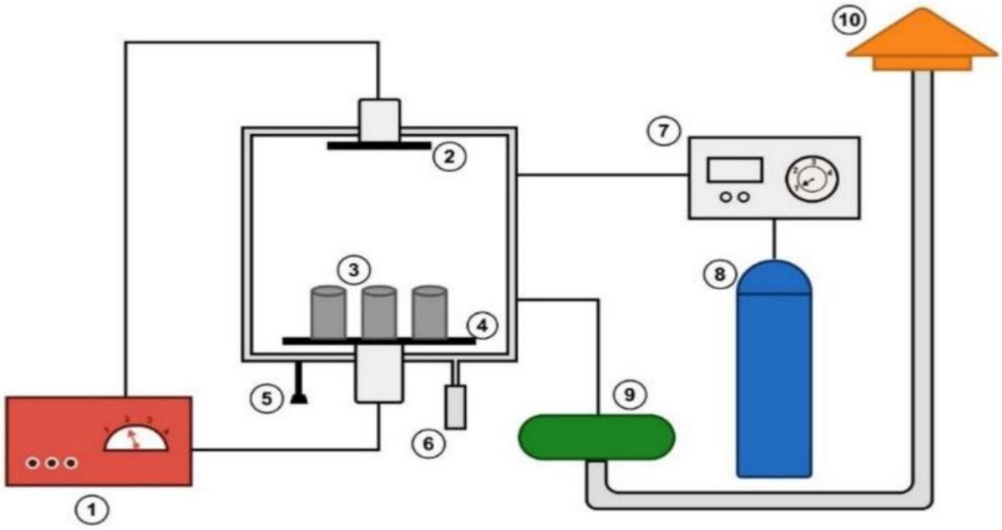


Figure 1 Schematic diagram of plasma deposition for DLC. Information: 1) DC/RF power supply, 2) Anode, 3) Specimen, 4) Cathode, 5) Grounding, 6) Pirani Gauge, 7) Control, 8) Source Gas, 9) Rotary Pump, 10) Exhaust

2.3. Experimental procedures

The specimens were inserted into the chamber and the vacuum pressure was adjusted to 1.2, 1.4, and 1.6 mbar. The input gas was later ignited at a composition ratio of 5:1 (v/v) for argon gas and LPG until the desired temperature of 450°C was achieved followed by deposition process for 4 hours.

2.4. Analysis

The thicknesses of the films were measured using HITACHI FLEXSEM 100 scanning electron microscope (SEM) equipped with energy dispersive spectroscopy (EDS) and a magnification of 3000 X. The device was also used to determine the elemental composition of material substrates following deposition process. The characteristics of the films were evaluated using Raman iHR320 HORIBA with laser at 514 nm in wavelength. Moreover, the phase composition of the layers was investigated using a Cu K and PAN analytical AERIS XRD which was considered to be initiated around 10 or even 100° in 2 theta in most cases. The tribological properties of the specimen were analyzed using an Ogoshi High-Speed Universal Wear Testing and the wear rate of the films was measured as the amount of material removed per unit weight and sliding distance. Furthermore, the microhardness of the substrate was determined using a Matsuzawa MMT-X7 microhardness tester with a 10 gf indenter load.

3. Results and Discussion

XRD of the coating films presented in Figure 2 showed five diffraction peaks at 2θ values which were 35.5 θ, 45.3 θ, 49.5 θ, 65.5 θ, and 82.7 θ. A comprehensive analysis showed that the three strong peaks dominating XRD spectrum at 2θ were 45.3, 65.4, and 82.7 reflections from the [1 1 1], [2 2 0], and [3 1 1] diamond planes respectively (Roy et al., 2002). Meanwhile, Pang et al. deposited DLC films on Cu at a low voltage of 800-300 volts through electrolysis of a water-ethanol solution and reported three peaks corresponding to the diamond at 2θ values were 43.2, 74.06, and 89.9, respectively (Pang et al., 2010). It was possible that carbon contributed to the 35.5 peak recorded at 2θ while the weakest observed at 49.5 could be identified as graphite [102].

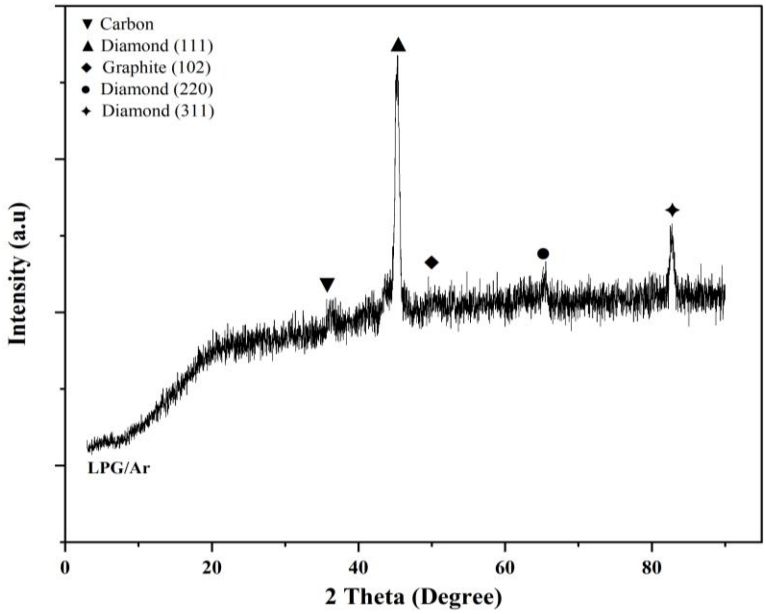


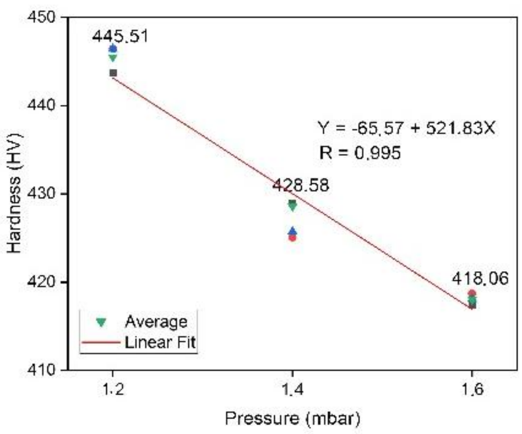
Figure 2 XRD spectrum for the 1.2 mbar

The information presented in Figure 3(a) showed that lower deposition pressure led to higher hardness. In this research, the optimal hardness of 445.51 HVN was reached at operating conditions of 1.6 mbar and 450°C. During the coating process, lower pressure was also observed to have led to greater wear resistance. The trend showed that the performance of the material would be better at a reduced gas pressure (Bewilogua and Hofmann, 2014).

The quicker movement of atoms at higher pressure led to more collisions which subsequently caused inhomogeneous deposition. The condition caused poor performance of the surface as observed in the reduction in hardness value as the process pressure increased. This was in line with the results of the previous research on the application of C_2H_2 gas as a carbon source in the coating process for 45 minutes that an increase in pressure and bias voltage accelerated deposition rate, causing a thick layer (Ward et al., 2014). In the same pattern, DLC deposition process conducted in this present research using the plasma method increased the hardness value of AISI D2 by approximately 2 times, from 235 VHN to 448 VHN. The observation was supported by the application of Deep Oscillation Magnetron Sputtering (DOMS) method to the same materials and films which produced a three times increase in the hardness value (Ferreira et al., 2021). However, compared to DOMS, the plasma deposition method has several advantages which include being 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. This is necessary because wear resistance is important for estimating material surface damage, specifically caused by wear and tear. The results presented in Figure 3(b) showed coefficient of friction (CoF) under different pressures. It is important to state that CoF is the ratio of the resistive force of friction divided by the normal or perpendicular force pushing the objects together (Hakim et al., 2021). The results showed that CoF value of DLC layer with a pressure of 1.2 mbar was the smallest at 1.3 x 10^{-3} compared to 1.4 and 1.6 mbar. This low value showed that the surface of DLC coating was becoming more resistant to wear and strain. Moreover, the trend was observed to be influenced by the elevated hardness of the material as confirmed by the highest value recorded for 1.6 mbar in Figure 3(a). This showed that a higher hardness value increased the ability of the material to withstand frictional forces, leading to a reduction in the wear resistance. Meanwhile, low hardness allowed the wear debris on the contact surface to penetrate and move which subsequently caused significant wear.

The frictional oxidation process on the surface of DLC coating increased CoF 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). However, the formation of a transfer layer between two contact surfaces started to reduce CoF. It was also observed that continuous friction made the transfer layer achieve a critical thickness, resulting in a stable CoF. The trend showed that machine parts, particularly those in contact with each other, should be coated with DLC. This is necessary because the reduction of CoF can increase the mechanical effectiveness of the machine as observed in DLC-coated cast iron (Suprapto et al., 2018; Banerji et al., 2016). The results of the current research show the possibility of applying DLC coating to friction machine components to extend their lifetime and mechanical efficiency in order to produce a more efficient operation.



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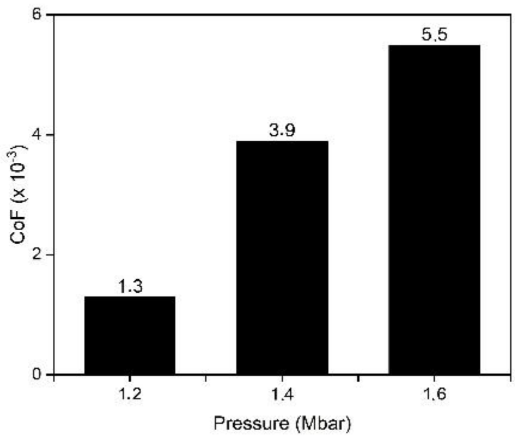

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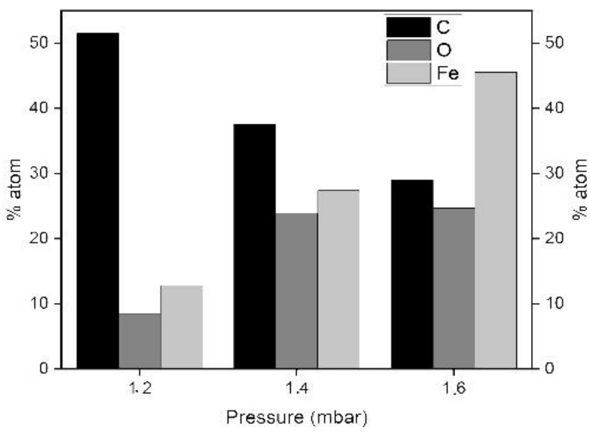
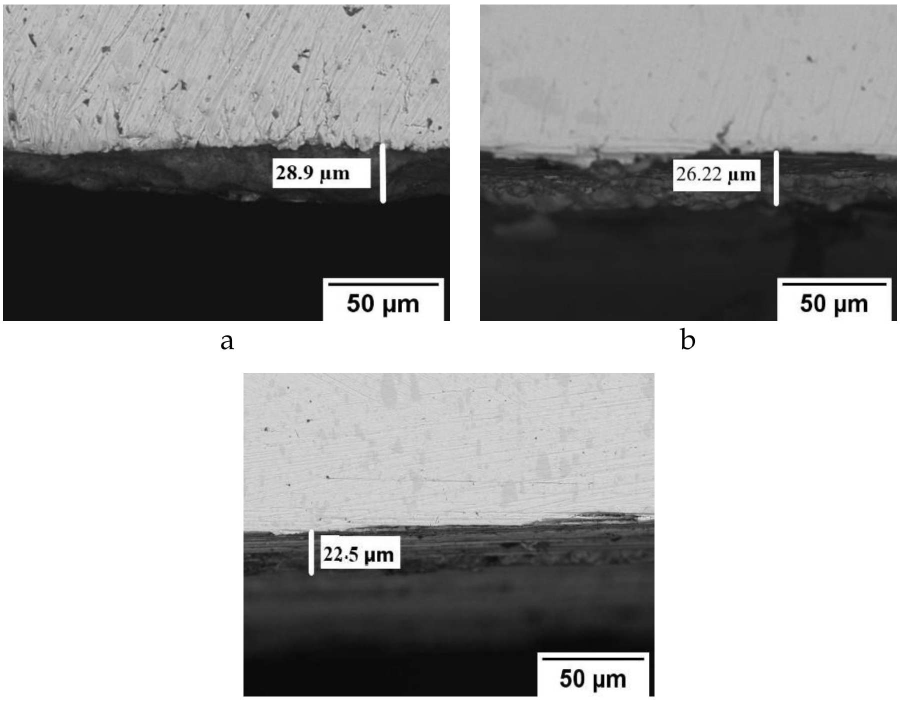


c

Figure 3(a) Microindentation of DLC Films at Different Pressure (b) CoF of DLC Various Pressure (c) EDX Chemical Composition of the Surface

High mechanical properties and hardness of DLC films are mainly responsible for the wear resistance of the material (Huang et al., 2022). Therefore, the low wear resistance value of DLC layer was due to the inert properties and good adhesiveness as observed from the insignificant loss by friction.

The surface elemental composition of DLC layer formed was determined through EDS characterization. The focus was to evaluate the effect of the pressure varied at 1.2, 1.4, and 1.6 mbar and the results are presented in Figure 3(c). The three most abundant surface elements were found to be carbon, oxygen, and iron with the layer at 1.2 mbar recorded to have a higher percentage of C atoms at 51.15% compared to the 37.54% and 28.96% for 1.4 and 1.6 mbar, respectively. In accordance with Boyle's law, an increase in the pressure of gas is expected to reduce the volume (Saepuzaman et al., 2019). Meanwhile, the volume has a linear effect on deposition rate and this means an increase in gas volume normally leads to an increment in deposition rate. It is also important to state that deposition rate of DLC coating is directly proportional to the surface carbon atom density.



a b

c

Figure 4 SEM cross-sectional images of DLC films at (a) 1.6 mbar (b) 1.4 mbar (c) 1.2 mbar

The influence of pressure on the thickness of DLC coating was determined through SEM cross-sectional characterization. The results presented in Figure 4 showed that the highest thickness of 28.9 m was produced under a pressure of 1.2 mbar. This was influenced by the amount of carbon on the surface of the gas, as observed from EDS analysis in Figure 3(b) where the percentage of carbon element on DLC layer at 1.2 mbar was 51.15%. Meanwhile, the content at 1.4 and 1.6 mbar was 37.54% and 28.96%, respectively.

The adhesion of DLC coating was tested at the pressures of 1.2, 1.4, and 1.6 mbar through an optical microscope photo using the cross-cut method according to ASTM D3559. The results presented in Figures 5 (a)-(c) showed that DLC layer formed had good adhesion as observed from the lack of peeling in the area around the intersection of the edge lines and the cross-section. The layer was classified as 5B based on the fact that the cut edges were extremely smooth and no loose parts were identified. Moreover, the residual stress of the surface showed a distortion of the bond angle and length, leading to a weak adhesion. The residual stress was calculated on DLC coating to quantify the adhesive strength based on the criterion that a lower value is an indication of greater adhesion. It was observed that DLC layer with 1.6 mbar had the lowest value of -4.2, thereby indicating a stronger adhesion. Furthermore, an increase in ID/IG ratio was observed to have led to an increment in sp2 content. It is important to state that ID/IG ratio is the comparison of the intensities of D and G peaks in Raman characterization results. The consumption of more sp2 increases the adhesive strength of the films because sp2 has a greater binding energy than sp3 (Sharifahmadian and Mahboubi, 2019). According to previous research, carbon with sp2 hybridization had less distortion which led to a reduction in the internal pressure of DLC layer and a subsequent increase in the graphitic layer causing an improvement in adhesion (Wang et al., 2021).

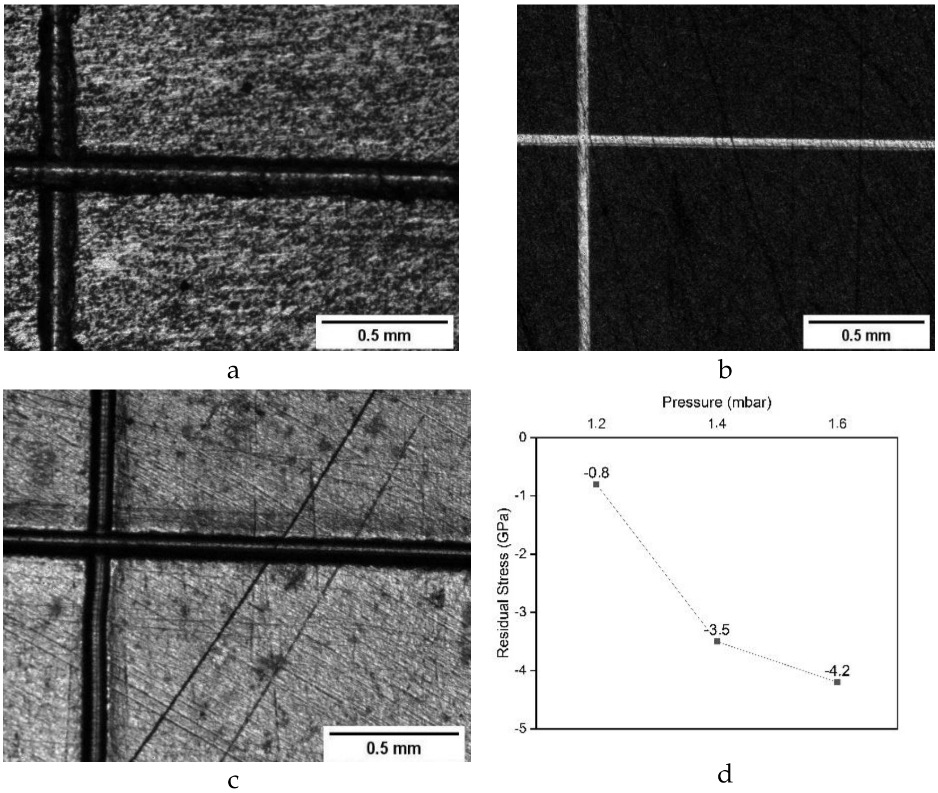


Figure 5(a) Cross Cut Morphology at 1.2 mbar (b) 1.4 mbar (c) 1. Mbar, and (d) Residual Stress

DLC bond structure was analyzed using Raman spectroscopy which was a method considered effective for investigating non-destructive carbon hybridization (Khamnualthong et al., 2012). The results presented in Figure 6 showed two bands at approximately 1322 and 1577 cm^{-1} which corresponded to the disordered graphite band or D-line and the single crystal graphite band or G-line, respectively (Pang et al., 2010). This showed that DLC films were conventional with a mixed structure of sp2 and sp3. G and D bands are well known to generate a typical Raman spectrum for DLC. G band was centered at nearly 1550 cm^{-1} due to the symmetric E2g vibrational mode in graphite-like materials while D band was at nearly 1330 cm^{-1} because of the restrictions on the size of the graphite domain caused by grain boundaries or imperfections (Wang et al., 2017; Ferrari and Robertson, 2001). Raman spectra in Figure 6 showed that all samples of the films exhibited DLC-typical characteristics.

The composition ratio of sp2 and sp3 hybrid carbon atoms significantly influenced the characteristics of DLC layer formed. In the phase diagram, the number of sp3, sp2, and sp formed in DLC layer determined the types of carbon bonds (Furlan et al., 2013). Moreover, the percentage of the relationship between sp3 and the structure of hydrogen bonds was classified (Moriguchi et al., 2016). When the percentage of sp3 was higher, DLC layer formed was closer to the diamond structure. According to Raman spectra analysis, the ID/IG ratio increased as the pressure rose from 1.2 to 1.6 mbar with the values recorded to be 0.84, 0.96, and 1.17, respectively. This led to an increment in sp3/sp2 ratio which made the films to become more diamond-like. The trend was associated with the fact that sp3/sp2 bonding ratio of the films influenced ID/IG value. This was observed from the smaller ID/IG ratio recorded when sp3/sp2 ratio was higher (Huang et al., 2022; Casiraghi et al., 2005; Robertson, 2002).

G peak was used to determine the graphitic properties with C=C hybridization for DLC films [32]. It was observed that a smaller FWHM value at G peak led to a smaller change in sp2 C=C hybridization as presented in Figure 6 (b) while ID/IG ratio increased with a rise in pressure. The trend was because the pressure affected the gas input in the chamber. The application of lower pressure reduced the volume of the gas in the chamber, which caused less plasma gas to stick to the substrate. Moreover, the attachment of fewer carbon sources also reduced the formation of sp3 and sp2 hybridization, leading to a smaller ID/IG ratio. It was observed that ID/IG ratio, previously recorded to be 0.95 by Dalibón et al. (2017), was successfully reduced through the coating parameter when 1.2 mbar of pressure was applied.

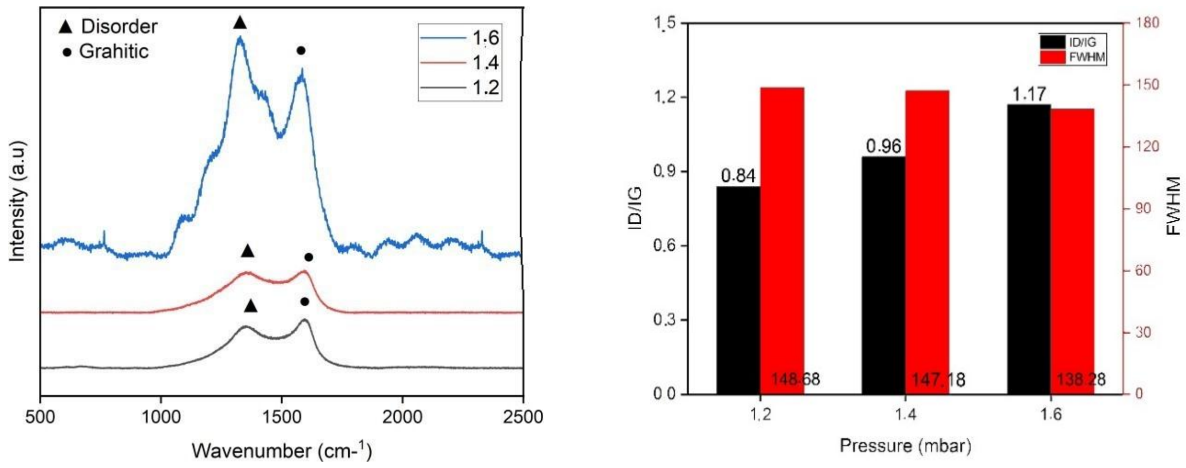


Figure 6 (a) Raman Spectra for DLC Films (b) Relation Between G FWHM and ID/IG Ratio

4. Conclusions

In conclusion, DLC coating was produced on AISI D2 surface using the plasma method at three optimal LPG gas pressures of 1.6, 1.4, and 1.2 mbar. A rise in deposition pressure from 1.2 to 1.6 mbar increased the hardness, thickness, and uniformity of the coating in the axial direction but reduced the wear coefficient. Moreover, CoF value of 1.3×10^{-3} obtained for DLC layer at 1.2 mbar was the smallest compared to the others. The bands at 1322 and 1577 cm^{-1} in Raman spectrum confirmed that the deposited films contained DLC as observed from the three intense peaks of 45.3, 65.4, and 82.7 recorded at 2θ for [1 1 1], [2 2 0], and [3 1 1] planes respectively. This showed that the modification of deposition pressure led to the production of DLC coating with improved tribological and mechanical properties. Based on these facts, DLC films were confirmed to be suitable as an inexpensive coating material for cutting instruments and automobile components.

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Author Contributions

Indah Uswatun Hasanah was responsible for the conceptualization, methodology, investigation, data curation, formal analysis, and preparation of the original draft of the manuscript. Dedi Priadi contributed to the conceptualization, supervision, and project administration. Donanta Dhaneswara participated in the methodology and formal analysis. Suprapto contributed to supervision and manuscript review. Tjipto Sujitno was involved in manuscript review and funding acquisition. Emy Mulyani supported the methodology development and funding acquisition. All authors have read and agreed to the final version of the manuscript.

Conflict of Interest

The authors declare that there is no conflict of interest regarding the publication of this paper. All authors confirm that they have no financial, personal, or professional relationships that could be perceived as influencing the work reported in this manuscript. The research was conducted independently, and the results presented are based solely on the authors' analysis and interpretation.

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