Journal of Metals, Materials and Minerals, 37(1), e2688, 2027

Comparative study of frictional behavior of Cr-doped and Al:Ti-doped DLC coatings on AISI 4140 steel

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Received date:
26 March 2026
Revised date:
11 September 2026
Accepted date:
23 October 2026

Keywords:
Diamond-like carbon;
Tribology;
Wear;
Ball-on-flat;
Steel

Abstract

Diamond-like carbon (DLC) coatings have attracted significant interest for applications requiring low friction and high surface durability. Consequently, various DLC formulations have been developed to meet different tribological demands. In this study, the frictional behavior of AISI 4140 carbon steel, which is a widely used high strength structural material, was investigated following surface modification with two distinct DLC coating configurations. Specifically, Cr–doped DLC with a graded CrCN interlayer (Cr–DLC/CrCN) and AlTi-doped DLC with a graded AlTiCN interlayer (Al:Ti–DLC/AlTiCN) were compared. Wear tests were performed using the ball-on-flat method. The results show that the Cr–DLC/CrCN coating exhibited a lower coefficient of friction (0.26) than the Al:Ti–DLC/AlTiCN coating (0.30). Laser scanning confocal microscopy also revealed a smaller wear track on the Cr–DLC/CrCN surface, with a reduced depth (2.52 μm) and width (496.68 μm). Raman spectroscopy indicated a higher sp^2 bonding fraction in the Cr–DLC/CrCN coating, as reflected by its increased I_D/I_G ratio, which contributed to its lower friction behavior. Overall, the Cr–DLC/CrCN coating demonstrated superior tribological performance and wear resistance, making it a more promising surface modification for enhancing the durability of AISI 4140 carbon steel in severe sliding wear environments, typical in oil and gas industries.

1. Introduction

Structural alloy steel, such as AISI 4140, is widely employed in the manufacturing of gears, bolts, couplings, spindles, tool holders, sprockets, oil industry drill collars, tool joints, and other critical components due to its excellent balance of strength, toughness, and hardenability. Its mechanical properties can be further enhanced through various treatments such as heat treatment, alloying, and surface modification [1]. Although these properties make AISI 4140 steel highly attractive for engineering applications, a major limitation of this steel is its relatively low frictional resistance, which restricts its performance under severe contact and sliding conditions. Consequently, improving the tribological performance of AISI 4140 steel has become an important research focus. Surface modification through thin-film coatings such as TiN, TiAlN, and diamond-like carbon has emerged as an effective approach to address these limitations [2].

Among these coatings, DLC is of particular interest because of its outstanding combination of beneficial properties, including high hardness, low friction coefficient, excellent wear resistance, chemical inertness, and superior corrosion resistance [3,4]. These characteristics make DLC an attractive material for extending the service life and reliability of mechanical components subjected to high friction and wear conditions. The performance of DLC is strongly influenced by its microstructure, which is typically characterized by the ratio of sp^3 (diamond-like) to sp^2 (graphite-like) carbon bonds [3,5,6]. A higher sp^3 content generally leads to higher hardness, whereas sp^2 bonding contributes to electrical conductivity and low friction. To further optimize DLC performance, several strategies have been investigated, including elemental doping and the incorporation of interlayers, both of which have been shown to improve film adhesion, reduce residual stress, and enhance overall tribological performance [3–5].

Previous studies have demonstrated that interlayers and doping elements such as Ti, Cr, Nb, W, Mo, and Al play a significant role in improving the adhesion of DLC films. Among these elements, Cr also exhibits excellent wear resistance at elevated temperatures [7]. These elements form strong carbides through bonding with carbon and help reduce internal stress. However, the components incorporated into the DLC film, as well as the interlayer design, must be carefully optimized to avoid compromising wear resistance and other functional properties [8–10].

To reduce heavy sliding wear in oil-pumping environments, load bearing, adhesion, and surface friction are three key concerns for DLC coatings. The conventional phosphate conversion coating for sucker-rod couplings improved surface friction but failed to meet the load-bearing requirement. For the specific application in this study, a balanced DLC coating design is needed to achieve low friction and load-bearing with strong adhesion. Metal doping facilitates the formation of an adhesive interlayer, building-up a low stress supporting

layer, and activation sp^2 and sp^3 carbon bonds on top. Among various dopant metals, Cr possesses a higher affinity to steel with lower compressive stress, which ensures a well-balanced DLC with a good adhesion strength. Ti- and AlTi-doped DLC excels in higher hardness for wear resistance and oxidation resistance by forming protective surface oxide layers. W-based DLC is hard, but it is also a strong metal carbide former with very high residual stress, which prohibits the formation of carbon sp^2 and sp^3 bonds over W-C bonds. Si is an excellent dopant for reducing the friction coefficient of DLC with lower compressive stress, but load-bearing is an issue.

Based on these considerations, the present work investigates the frictional behavior of DLC coated AISI 4140 steel using a ball-on-flat configuration, with the objective of estimating the service life of DLC films prior to their application on coupling surfaces in oil pumping systems for oil and gas field applications. Two types of DLC coatings were evaluated: Cr–doped DLC with a graded CrCN interlayer (Cr–DLC/CrCN) and AlTi-doped DLC with a graded AlTiCN interlayer (Al:Ti-DLC/AlTiCN). By examining and comparing the tribological behavior of these coating systems, this study provides insights into the influence of interlayer composition and elemental doping on the frictional performance of DLC–coated AISI 4140 steel.

2. Experimental

2.1 Sample preparation

Samples with dimensions of 40 mm × 40 mm × 10 mm were prepared by Aurora Scientific Siam Co., Ltd. A PVD/PECVD hybrid technique, Cathodic Arc Activated Deposition, CAAD [11,12], was used to deposit DLC coatings on AISI 4140 steel. CAAD is a sequential PVD/PECVD technique, which applies a PVD process to deposit the metal interlayer and stress control transition layer, and switches to a PECVD process to deposit the final DLC top layer. Two types of films were deposited: AlTi–doped DLC with an AlTiCN interlayer (AlTi–DLC/AlTiCN) and Cr-doped DLC with a CrCN interlayer (Cr–DLC/CrCN), the coating conditions are shown in Table 1. The surface roughness (Sa) of both films was approximately 1.30 µm. The total coating thicknesses were determined from cross-sectional Scanning electron microscope images (SEM, Hitachi SU3500) of the coatings deposited on tungsten carbide (WC) witness substrates, as shown in Figure 1. The WC witness substrates were coated under the same deposition conditions as the AISI 4140 specimens. WC was selected because its relatively hard and stable surface facilitates cross-sectional preparation and SEM observation. Previous studies have investigated DLC coatings deposited on different substrate materials, including WC–Co and stainless steel, with a chromium interlayer [13]. The measured thicknesses were approximately 3.35 ± 0.16 µm and 2.57 ± 0.11 µm for the Al:Ti–DLC/AlTiCN and Cr–DLC/CrCN coatings, respectively. These values are considered representative thicknesses of the deposited coatings; the actual thicknesses on AISI 4140 may differ because of differences in substrate surface characteristics and coating geometry.

2.2 Film analysis using Raman spectroscopy

The structure of the DLC films was evaluated using Raman spectroscopy (Horiba XploRA PLUS) to determine the I_D/I_G ratio. A 532 nm laser was used as the excitation source, and the spectra were recorded over a wavenumber range of 800 cm^{-1} to 2000 cm^{-1}. The resulting Raman spectra were fitted with four Gaussian peaks using Microsoft Excel software.

2.3 Wear testing

Wear testing was conducted using a ball-on-flat configuration with a universal tribotester (Ducom) to evaluate the frictional behavior of the samples. The test conditions were as follows: a 6.35 mm diameter AISI 440C stainless steel ball as the counterface, a stroke length of

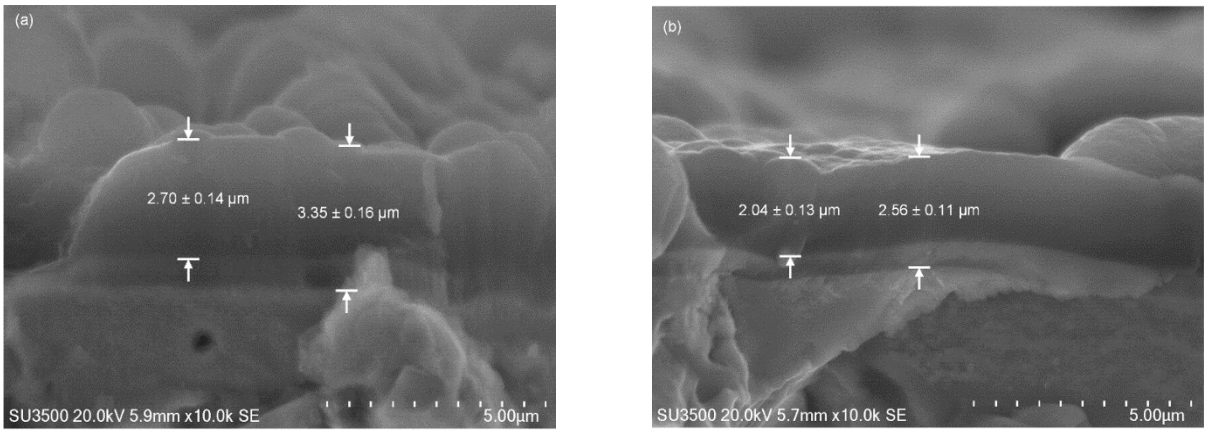


Figure 1. SEM images illustrating the film thickness of (a) Al:Ti–DLC/AlTiCN, and (b) Cr–DLC/CrCN coatings.

Table 1. Coating conditions for the Al:Ti–DLC/AlTiCN, and Cr–DLC/CrCN samples.

Sample	Pressure [Pa]	Bias [V]	Temperature [°C]	Reactive gas	Time [h]	Hardness [Hv]
Al:Ti–DLC/AlTiCN	2.66	100	350	N_2, C_2H_2	5	2791
Cr–DLC/CrCN	1.50	100	200	N_2, C_2H_2	5	2546

10 mm, a test duration of 20 min, and a sliding speed of 20 mm·s^{-1}. A normal load of 20 N was selected to produce measurable wear and to represent a demanding contact condition relevant to engineering and oil and gas applications, consistent with previous studies on DLC coatings. All coatings were tested under identical conditions to allow direct comparison, although wear behavior depends on the applied load. After testing, the wear tracks on the samples were characterized using laser scanning confocal microscopy (LSCM; Olympus LEXT OLS5000, Japan) and SEM.

3. Results

3.1 Raman spectroscopy

After fitting the Raman spectra with four Gaussian peaks using Microsoft Excel software, as shown in Figure 2, two main bands (G and D) and two sub-bands (I and D3) were clearly identified. The first main band, the G band (E2g mode) located at approximately 1580 cm^{-1}, corresponds to the bond stretching of pairs of sp^2 carbon atoms in the graphitic structure. The second main band, the D band (A1g mode) at around 1360 cm^{-1}, is attributed to the breathing modes of disordered graphitic carbon rings, which typically indicate the presence of structural defects or disorder in the carbon network [14–16].

In addition to these main peaks, two sub-bands were observed. The I band at ~1212 cm^{-1} and the D3 band at ~1499 cm^{-1} are both associated with the stretching vibrations of amorphous carbon, suggesting the coexistence of sp^2 and sp^3 related disorder within the films [14]. These additional bands provide further evidence of the complexity of the carbon bonding environment.

To compare the films, the areal ratio of the main bands was calculated, as this parameter is often used to evaluate the relative proportion of disordered to ordered sp^2 bonding in carbon-based films. As shown in Figure 2, the I_D/I_G ratio of the Al:Ti–DLC/AlTiCN film was 1.50, whereas the Cr–DLC/CrCN film exhibited a slightly higher ratio of 1.71. This increase in the I_D/I_G ratio for Cr–DLC/CrCN indicates a greater degree of sp^2 carbon bonding compared with the Al:Ti–DLC/AlTiCN film [15].

Furthermore, a noticeable shift of the G band position was observed between the two films. The G band of Al:Ti–DLC/AlTiCN appeared at ~1582 cm^{-1}, while that of Cr–DLC/CrCN was located at ~1578 cm^{-1}. A shift of the G band toward a higher wavenumber is generally correlated with an increase in internal stress within the film, which is typically attributed to a higher fraction of sp^3 bonding [15]. This result suggests that the Al:Ti–DLC/AlTiCN film possesses a relatively higher sp^3 content compared to the Cr–DLC/CrCN film, contributing to different mechanical and structural properties.

3.2 Wear testing

Figure 3 presents the friction force measured under a load of 20 N for 20 min. The results clearly show that the Cr-DLC/CrCN film exhibited a lower CoF than the Al:Ti–DLC/AlTiCN film, with values of 0.26 and 0.30, respectively. A lower CoF indicates that the Cr–DLC/CrCN coating reduces energy loss and surface damage more effectively during sliding, and even a difference of 0.04 in CoF is significant in tribological performance.

To determine which DLC coating underwent more severe wear during testing, the wear-track profiles obtained from CLSM were evaluated in terms of both depth and width, as shown in Figure 4 and summarized in Table 2. These parameters directly reflect the extent of material removal and surface deformation caused by sliding contact. The Cr-DLC/CrCN coating shows a notably smaller wear-track depth (2.52 µm) and width (496.68 µm) compared with the Al:Ti–DLC/AlTiCN coating, which exhibits significantly deeper and wider tracks (3.81 µm and 565.03 µm, respectively). This difference demonstrates that the Cr–DLC/CrCN coating experiences less material loss and maintains surface integrity more effectively under the same test conditions. Moreover, Figure 5 presents backscattered SEM images of the wear tracks of both films, which clearly show that the wear track widths are consistent with the confocal measurement results listed in Table 2.

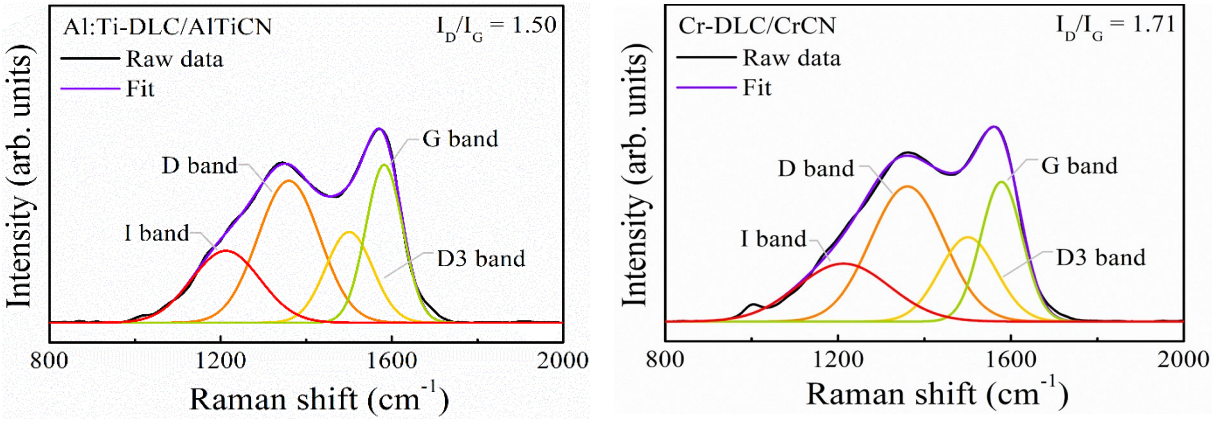


Figure 2. Deconvoluted Raman spectra of Al:Ti-DLC/AlTiCN and Cr-DLC/CrCN coatings.

Table 2. Wear track depth and width of Al:Ti–DLC/AlTiCN and Cr–DLC/ CrCN coatings measured by LSCM.

Sample	Depth [µm]	Width [µm]
Al:Ti–DLC/AlTiCN	3.81 ± 0.14	565.03 ± 2.75
Cr–DLC/CrCN	2.52 ± 0.19	496.68 ± 2.99

These results demonstrate that the Cr–DLC/CrCN coating exhibits higher wear resistance than the Al:Ti–DLC/AlTiCN coating. The superior performance of the Cr–DLC/CrCN coating is supported by its lower coefficient of friction (CoF), smaller wear track depth and width, and lower Fe content detected on the wear track after testing. Collectively, these findings indicate that Cr–DLC/CrCN provides more effective protection of the AISI 4140 steel substrate against frictional wear, better preserves coating integrity, and minimizes substrate exposure under mechanical loading.

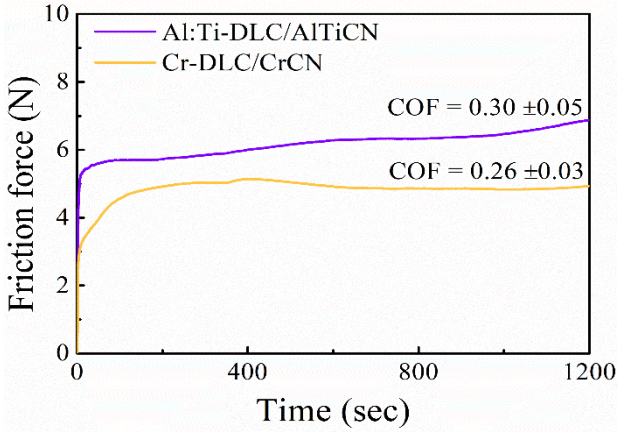


Figure 3. Friction force measured in Ball-on-Flat mode for Al:Ti-DLC/AlTiCN and Cr-DLC-CrCN coating.

4. Discussion

Elemental doping significantly affects the properties of DLC films, including oxidation resistance, wear resistance, corrosion resistance and hardness. Doping also influences the sp^2 and sp^3 bonding structures, as revealed by the Raman results. The Cr–DLC/CrCN film exhibited a higher I_D/I_G ratio, indicating a higher relative sp^2 content, than the Al:Ti–DLC/AlTiCN film, which is consistent with its lower hardness and lower coefficient of friction (CoF). The I_D/I_G ratio reflects the sp^2/sp^3 ratio only indirectly. It is useful for identifying trends, but a more accurate determination requires complementary techniques such as XPS or NEXAFS [3,4,15,17]. Raman spectroscopy can also provide information on internal stress through the G band position. The shift of the G band toward higher wavenumbers in the Al:Ti–DLC/AlTiCN film may indicate higher internal stress, which could contribute to its lower wear resistance, although the G band position is also influenced by sp^2 clustering and doping.

The Cr–DLC/CrCN film showed a smaller wear track than the Al:Ti–DLC/AlTiCN film, as confirmed by confocal microscopy, despite its lower hardness. According to Archard's law (Equation (1)), the wear volume V depends on the wear coefficient K_w, the normal load F_n, the sliding distance S and the hardness H:

$$V = K_w \frac{F_n S}{H} \quad (1)$$

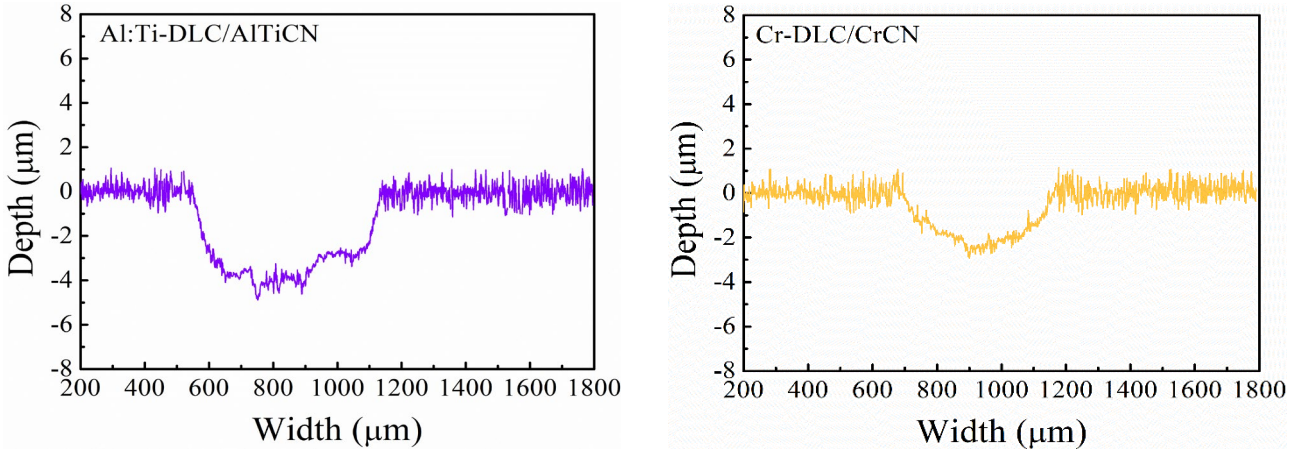


Figure 4. Wear track depth and width of Al:Ti-DLC/AlTiCN and Cr-DLC/CrCN coatings measured by LSCM.

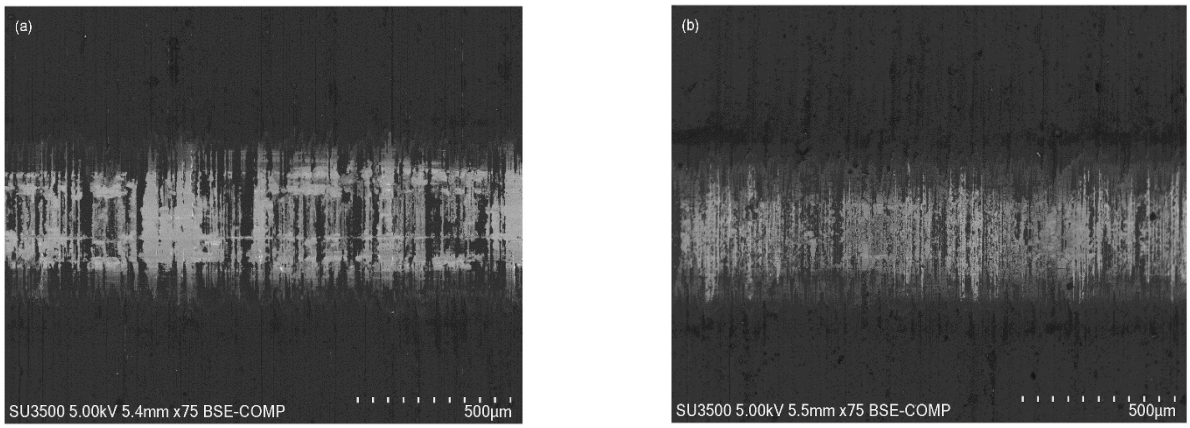


Figure 5. SEM in backscattered mode (a) Al:Ti–DLC/AlTiCN, and (b) Cr–DLC/CrCN coatings.

Because the load and sliding distance were the same for both films, differences in wear are governed by K_w and H, which depend on the coating material. Although a lower hardness tends to increase wear, K_w is strongly influenced by the CoF [19]. A lower CoF reduces interfacial shear stress and frictional energy, which decreases material removal, and may also limit frictional heating and adhesive and oxidative wear. A coating with a lower CoF can therefore show better wear resistance even with a lower hardness. This may explain why the Cr–DLC/CrCN film outperformed the Al:Ti–DLC/AlTiCN film.

More broadly, these observations suggest that wear performance depends not only on hardness but also on carbon bonding structure, internal stress and the evolution of the sliding interface. Previous studies have shown that friction-induced graphitization can form a low-shear-strength transfer film that reduces friction and limits direct contact between the sliding surfaces [18]. However, graphitization does not necessarily improve wear resistance, since excessive graphitization may weaken the coating and increase wear [18]. The lower CoF and higher sp^2 content of the Cr–DLC/CrCN coating are consistent with such an interface contributing to its smaller wear track. Because the transfer film was not directly characterized, this remains a possible mechanism rather than a confirmed finding.

Besides hardness and friction behavior, coating thickness and surface roughness may influence the wear performance of DLC coatings. Although the Al:Ti–DLC/AlTiCN coating (3.35 μm) was thicker than the Cr–DLC/CrCN coating (2.56 μm), it showed greater wear damage under the applied test conditions, so greater thickness does not by itself ensure better wear resistance. Surface roughness can also affect the real contact between the coated surface and the counter body. In this study, the roughness of both coatings (Ra ≈ 1.3 μm) was comparable to the coating thickness, which may influence wear through asperity contact, running-in and third-body effects. Because both coatings were deposited on identically prepared substrates and had comparable roughness, they experienced similar initial contact conditions. The comparison between them is therefore considered indicative, while the absolute wear values apply only to this surface condition. The observed difference in wear is likely to arise from the combined effects of thickness, hardness, friction behavior, surface roughness and interactions at the sliding interface.

The discussion above concerns the top DLC layer, which is directly involved in contact during sliding and was the focus of this study. However, the graded interlayers can also affect the adhesion and mechanical integrity of the coatings, and thereby their durability under sliding. Previous studies have shown that interlayer design can influence adhesion and wear performance, and that interfacial debonding or delamination can lead to coating damage [20,21]. Adhesion and interlayer integrity are therefore relevant to interpreting the tribological behavior. Since adhesion was not directly evaluated, its contribution to the observed wear behavior cannot be established.

Finally, the wear resistance and CoF of both films appear to be related to their structural characteristics. Raman spectroscopy indicated that the Al:Ti–DLC/AlTiCN film had higher internal stress and a relatively greater sp^3 fraction than the Cr–DLC/CrCN film. These features may partly explain why it showed lower wear resistance despite its higher sp^3 fraction and hardness. Some limitations however should be noted. Tests were conducted at a single load (20 N) with a 440C counterface, and adhesion and transfer film formation were not directly characterized. The results therefore represent a relative comparison under the tested conditions. Future work should include tests at multiple loads and counterbodies, adhesion testing, and analysis of tribofilm formation on the counter surface for coatings with different dopants.

5. Conclusions

This study comparatively evaluated the structural characteristics and tribological performance of Cr–doped DLC with a graded CrCN interlayer (Cr–DLC/CrCN) and Al:Ti–doped DLC with a graded AlTiCN interlayer (Al:Ti–DLC/AlTiCN) deposited on AISI 4140 steel using a PVD/PECVD hybrid CAAD process. Raman spectroscopy confirmed the presence of characteristic D and G bands in both coatings, together with additional sub-bands associated with amorphous carbon structures. The Cr–DLC/CrCN coating exhibited a higher I_D/I_G ratio (1.71) than the Al:Ti–DLC/AlTiCN coating (1.50), indicating a greater fraction of sp^2 bonding. In contrast, the Al:Ti–DLC/AlTiCN film showed a G–band shift toward higher wavenumbers, suggesting higher internal stress and a relatively higher sp^3 fraction.

Ball-on-flat tribological testing demonstrated that the Cr–DLC/CrCN coating achieved superior frictional and wear performance. It exhibited a lower coefficient of friction (0.26) compared with the Al:Ti–DLC/AlTiCN coating (0.30), along with a significantly reduced wear track depth (2.52 μm vs. 3.81 μm) and width (496.68 μm vs. 565.03 μm). Despite its slightly lower hardness, the improved wear resistance of Cr–DLC/CrCN is attributed to its lower frictional shear stress and favorable sp^2–rich structure, which likely promotes the formation of a stable tribofilm and reduces friction-induced damage.

Overall, the results demonstrate that the combined effects of dopant selection and graded interlayer design strongly influence the structural stress state and tribological response of DLC coatings. The Cr–DLC/CrCN system provides a more balanced combination of low friction and wear resistance, making it a promising candidate for extending the service life of AISI 4140 steel components operating under severe sliding conditions, such as those encountered in oil and gas applications. Future work should include adhesion strength evaluation, corrosion-wear synergy studies, and mechanistic investigations of tribofilm formation to establish a more comprehensive performance assessment.

Acknowledgements

The research was financially supported by PTT Exploration and Production PCL (PTTEP) under contract number SO34550058443. The authors would also like to thank Aurora Scientific Siam Co., Ltd. for their assistance with sample preparation and DLC coatings.

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