Vol. 03, Iss. 1 (2025) 67-77, DOI: 10.61552/JME.2025.01.004

JME

Journal of Materials and Engineering

www.jme.aspur.rs

Research article

Friction and Wear of Nitrogen Doped DLC Coating and Platinum/Ruthenium/Nitrogen Co-Doped DLC Nano-Composite Coating

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Keywords:

DLC coatings
Pt, Ru and N co-doping
Sputtering deposition
Hardness
Wear

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Received: 3 April 2024
Revised: 25 May 2024
Accepted: 17 June 2024

A B S T R A C T

Nitrogen doped diamond-like carbon (NDLC) coating and platinum/ruthenium/nitrogen co-doped DLC nano-composite (PtRuNDLC-NC) coating deposited on silicon (Si) substrates via magnetron sputtering deposition processes were comparatively investigated to understand the effect of co-incorporation of Pt and Ru on their adhesion strength, hardness, and wear. Compared to the NDLC coating, the PtRuNDLC-NC coating had 6.6% lower hardness, but 9.5% higher adhesion strength. The PtRuNDLC-NC coating had the lower abrasive wear resistance under a dry condition than the NDLC coating. It could be concluded that the co-doping of Pt/Ru/N could not give rise to the higher hardness and abrasive wear resistance of DLC coatings than doping of N.

1. INTRODUCTION

DLC coatings with high hardness, low friction, and high wear resistance are promising for tribological applications [1,2]. DLC coatings have been applied as protective coatings in engine tribo-components (ETCs) such as piston rings, camshaft, crankshaft, valves, etc. [2]. Piston rings provide sealing of the combustion chamber to reduce gas leakage into the crankcase as well as prevent invasion of lubricating oil from the crankcase to the combustion chamber [3]. They play an important role in internal combustion (IC) engines widely used in various industries such as transport, oil and gas, energy and power, agricultural, and household industries. Therefore, their performance, durability, and service life become critical topics for tribologists and engine producers around the world. It has been reported that DLC coated piston rings decrease exhaust gas emission, fuel consumption, and noise level of IC engines with their improved friction and wear losses [2,4]. Reducing the friction and wear of ETCs with DLC coatings is a promising way for effectively reduceing the fuel consumption and exhaust gas emission in IC engines in order to reduce the use of non-renewable fossil fuel resources and the global warming and improve the fuel economy for the more sustainable and greener world [4,5]. However, DLC coatings intrinsically have high residual stresses and thereby poor adhesion strength, which is a major challenge for ETCs operated in extreme environments [2,6]. The poor adhesion strength of DLC coatings can result in their easy delamination in service leading to a failure of DLC coated ETCs and causing a negative effect on the working performance of IC engines such as output, consumption, emission, noise, durability, etc [2,6].

Doping of metals (Cr, Ti, Al, Ni, Cu, Ag, etc.) or non-metals (N, Si, etc.) in DLC matrixes has been carried out to improve the adhesion strength of DLC coatings by releasing their residual stresses [7-9]. Many researchers [7-9] have afforded to improve the adhesion strength of DLC coatings with doping of metals or non-metals. However, doping of metals or non-metals reduces the hardness of DLC coatings and thereby their wear resistance since the coating hardness is closely related to the coating abrasive wear resistance under a dry condition [10,11].

Khun et al. [8,12] developed NDLC and PtRuNDLC-NC coatings to improve their adhesion strength. However, their abrasive wear resistance has not been comparatively studied yet. Therefore, a comprehensive study on the abrasive wear resistance of NDLC and PtRuNDLC-NC coatings should be carried out in order to understand the effect of Pt and Ru co-doping on their abrasive wear resistance under a dry condition by performing their structure-property relationships, which has not been widely reported in literature yet.

In this study, the NDLC and PtRuNDLC-NC coatings were fabricated on the Si substrates via magnetron sputtering deposition processes. Then, their surface chemical compositions, surface wettability, surface roughness, structures, hardness, adhesion strength, friction and wear resistance were comparatively investigated.

2. EXPERIMENTAL DETAILS

2.1 Sample preparation

Prior to the coating deposition, the Si substrates were etched with an Ar^+ plasma in the vacuum chamber at a pressure of 10 mTorr and a substrate bias of -250 V for 20 min to remove oxidized surface layers and other contaminants. The PtRuNDLC-NC thin coating was deposited on the p-Si (100) substrates using a custom-made DC magnetron sputtering deposition system by co-sputtering graphite (99.99%) and $Pt_{50}Ru_{50}$ (99.99%) targets of 4 inch in diameter with DC powers of 850 W and 40 W in the reactive deposition chamber where nitrogen and argon gases were introduced as reactive and working gases at flow rates of 15 sccm and 50 sccm, respectively, for 30 min. The NDLC coating was prepared under the same conditions without co-sputtering of the $Pt_{50}Ru_{50}$ target. The substrate bias, substrate rotation speed, and deposition pressure optimized for all the coatings were -90 V, 20 rpm, and 3 mTorr, respectively. Their estimated thickness was about 110 nm.

2.2 Characterization

The surface chemical compositions of the coatings were measured using X-ray photoelectron spectroscopy (XPS, Kratos-Axis Ultra) with a monochromatic Al Kα radiation (hυ = 1486.71 eV) powered with 10 mA and 15 kV. Distributions of Pt 4f and Ru 3d on the PtRuNDLC-NC coating surface were mapped with a pass energy of 80 eV for 15min.

The bonding structures of the coatings were characterized using confocal micro-Raman spectroscopy (Renishaw RM1000) with a He-Ne laser (632 nm). It had a spectral resolution of 1 cm^{-1} and a spatial resolution of 1 µm. The Raman spectra were fitted using a Gaussian function. Five random measurements on each coating were carried out to get average Raman parameters.

The surface morphologies of the coatings were captured using scanning electron microscopy (SEM, JEOL-JSM-5600LV). Their surface topographies were scanned using atomic force microscopy (AFM, Digital Instruments S-3000) with which their surface roughness was evaluated in terms of an arithmetic average, R_a. Five random measurements on each coating were carried out to get an average R_a value.

The water contact angles of the coatings were measured using a sessile liquid drop method (FTA 200). An average water contact angle was taken from five random measurements on each coating.

The adhesion strength of the coatings was measured in terms of a "critical load" that was associated with an instant adhesive failure between the coating and substrate using a micro-scanning-scratch-tester (Shimadzu SST-101) to scratch the coating surfaces with a 30 μm diamond stylus under progressive loading. The oscillation amplitude, oscillation frequency, scratch rate, and down speed optimized were 50 μm, 30 Hz, 10 μm/s, and 2 μm/s, respectively. Five random measurements on each coating were conducted to get an average critical load.

The hardness of the coatings was measured with a NHT (Anton Paar) indentation tester under a load control up to a maximum load of 250 mN at loading and unloading rates of 200 mN/min with a pause for 5 s at the maximum load. Sixteen random measurements on each coating were carried out to get average hardness and reduced elastic modulus. Oliver & Pharr method was applied for the hardness measurement.

The friction coefficients of the coatings were measured using a ball-on-disc micro-tribo-test (CMS). A fixed 6 mm alumina ball was loaded on a rotating coating surface in a circular path of 2 mm in radius for 3,000 laps (about 0.038 km) at a sliding speed of 3 cms^{-1} under a normal load of 1 N. Their wear widths and depths were measured using a white light confocal imaging profilometry to calculate their wear volumes. Three random measurements on each coating were carried out to get average tribological results. Alumina balls were used instead of steel balls since the former could generate the severe wear of the coatings. In this study, all the measurements were carried out at room temperature (RT ~ 22-24°C).

3. RESULTS AND DISCUSSION

The surface N contents in the NDLC and PtRuNDLC-NC coatings are 20.7 and 18.1 (12.6% lower) at.%, respectively. The surface Pt and Ru contents in the PtRuNDLC-NC coating are 3.2 and 4.9 at%, respectively.

Distributions of Pt 4f and Ru 3d on the surface of the as-deposited PtRuNDLC-NC coating are shown in Figures 1a and b, respectively, from which the PtRuNDLC-NC coating surface enriched more with Ru than Pt is found since Pt/Ru aggregates have a core-shell-structure possessing an inner-core enriched with Pt and an outer-shell enriched with Ru [12,13].

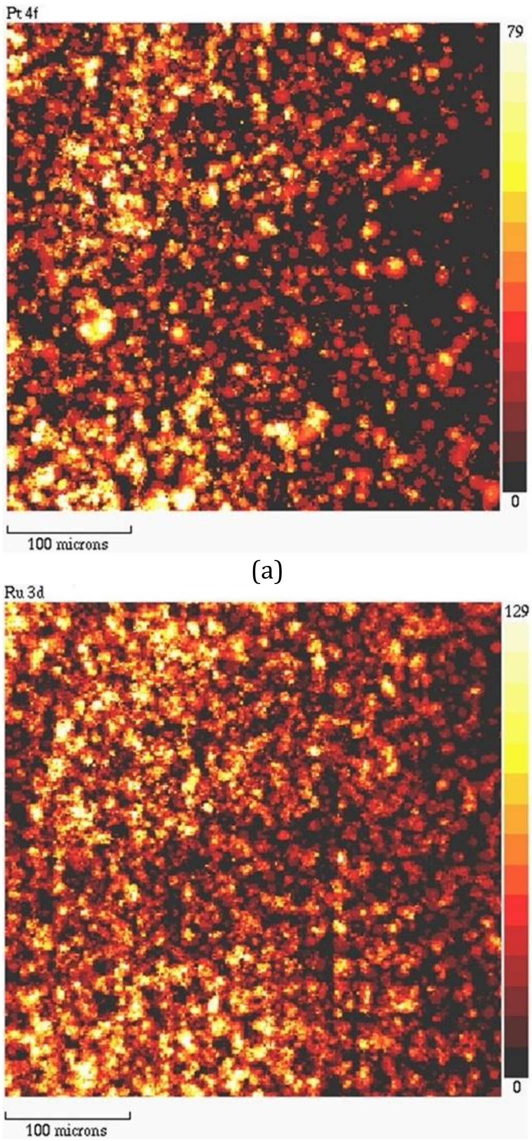


Fig. 1. XPS mapping images showing distributions of (a) Pt 4f and (b) Ru 3d on surface of as-deposited

PtRuNDLC-NC coating. Bright spots in Figures 1a and b represent Pt and Ru elements, respectively.

Figure 2 shows the Raman spectra of the NDLC and PtRuNDLC-NC coatings. Comparison of their Raman spectra shows that the co-incorporation of Pt and Ru apparently depresses the Raman spectrum because Pt/Ru aggregates are inactive phases to Raman excitation [12,14]. The Raman spectrum is mainly attributed to sp^2 bonds in a DLC matrix [14]. Their Raman spectra are mainly composed of G and D peaks. Stretching vibration of any pair of sp^2 sites contributes to the G peak while breathing mode of aromatic rings is responsible for the D peak [14].

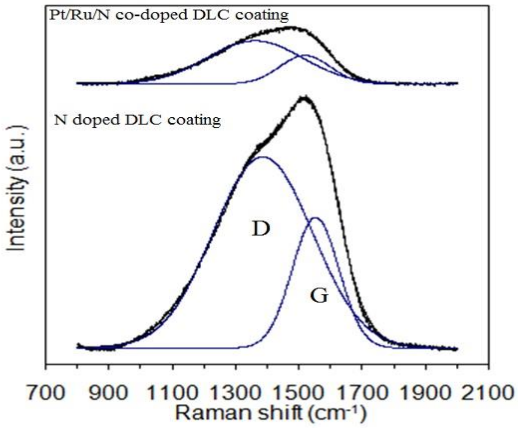
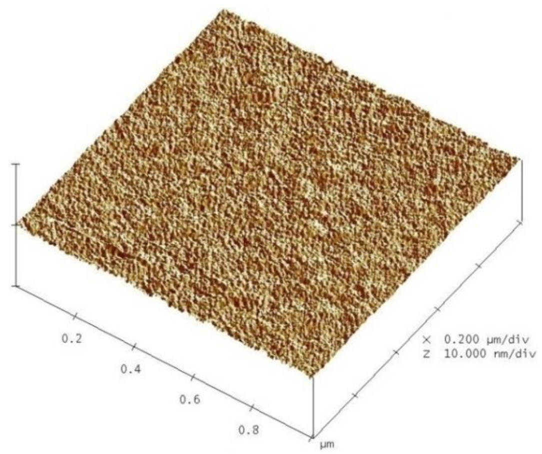


Fig. 2. Raman spectra of NDLC and PtRuNDLC-NC coatings.

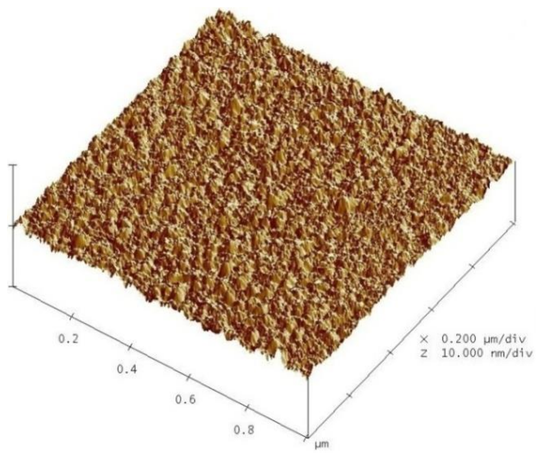
It is clear that an intensity (I_D/I_G) ratio of the D and G peaks is informative of graphitization of an amorphous carbon structure [8,12,14]. The I_D/I_G ratios of the NDLC and PtRuNDLC-NC coatings are 1.3 and 1.6 (23.1% higher), respectively, which indicate that the PtRuNDLC-NC coating has the higher sp^2 fraction as a result of its metal-induced-graphitization associated with the incorporation of Pt and Ru [10,12,14].

The R_a value of the Si substrate is 0.13± 0.03 nm. The R_a values of the NDLC and PtRuNDLC-NC coatings are 0.81± 0.05 nm and 1.18± 0.1 nm (45.7% higher), respectively. It implies that the surface roughness of the underlying Si substrate does not influence those of the both coatings. The PtRuNDLC-NC coating has the higher surface roughness attributed to its surface PtRu-aggregates and metal-induced-graphitization than the NDLC coating [10,12].

Figure 3a and b show the surface topographies of the NDLC and PtRuNDLC-NC coatings. The surfaces of the both coatings have good continuity with homogenous surface features, but the PtRuNDLC-NC coating has coarser surface asperities for its rougher surface.

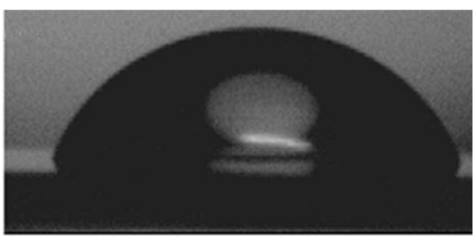


(a)

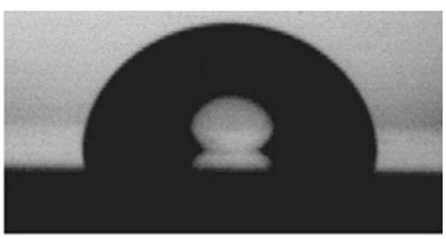


(b)

Fig. 3. Surface topographies of (a) NDLC and (b) PtRuNDLC-NC coatings.



(a)



(b)

Fig. 4. Water droplets on (a) NDLC and (b) PtRuNDLC-NC coating surfaces.

The water droplets on the NDLC and PtRuNDLC-NC coating surfaces are presented in Figure 4 with their water contact angles of 59±0.9° and 68.1±0.9° (15.4% higher), respectively. A hydrophobic surface is normally characterized with a water contact angle larger than 70° [15-17]. Since the water contact angle of the PtRuNDLC-NC coating is close to 70°, its surface can be considered as a hydrophobic surface. The surface of the PtRuNDLC-NC coating is more hydrophobic than that of the NDLC coating because its reduced N content with the incorporation of Pt and Ru lowers polar C-N bonds on its surface [12,18,19]. In addition, the chemical inertness of exposed Pt/Ru aggregates on the PtRuNDLC-NC coating surface could be also responsible for its more hydrophobic surface [12]. Since air trapped in surface asperities below a water droplet is responsible for a large water contact angle, the higher surface roughness of the PtRuNDLC-NC coating gives rise to its larger water contact angle by trapping more air at the water liquid/coating interface [20,21].

The hardness and reduced elastic modulus of the NDLC coating are 15.2±2.3 GPa and 186.4±10.8 GPa, respectively. However, the PtRuNDLC-NC coating has the lower hardness and reduced elastic modulus of 14.2±1.4 GPa (6.6% lower) and 179.6±5.8 GPA (3.6% lower), respectively, which can be correlated to the co-incorporation of softer Pt and Ru metal elements and metal-induced graphitization [22,23].

The critical loads of the NDLC and PtRuNDLC-NC coatings are 359±7 mN and 393±9 mN (9.5% larger), respectively, indicating that the PtRuNDLC-NC coating has a certain improvement in its adhesion strength because promoted sp^2 fraction via metal-induced graphitization and formed Pt/Ru aggregates in its matrix lower its residual stress by degrading its rigid sp^3 bonded cross-linking structure [7,24].

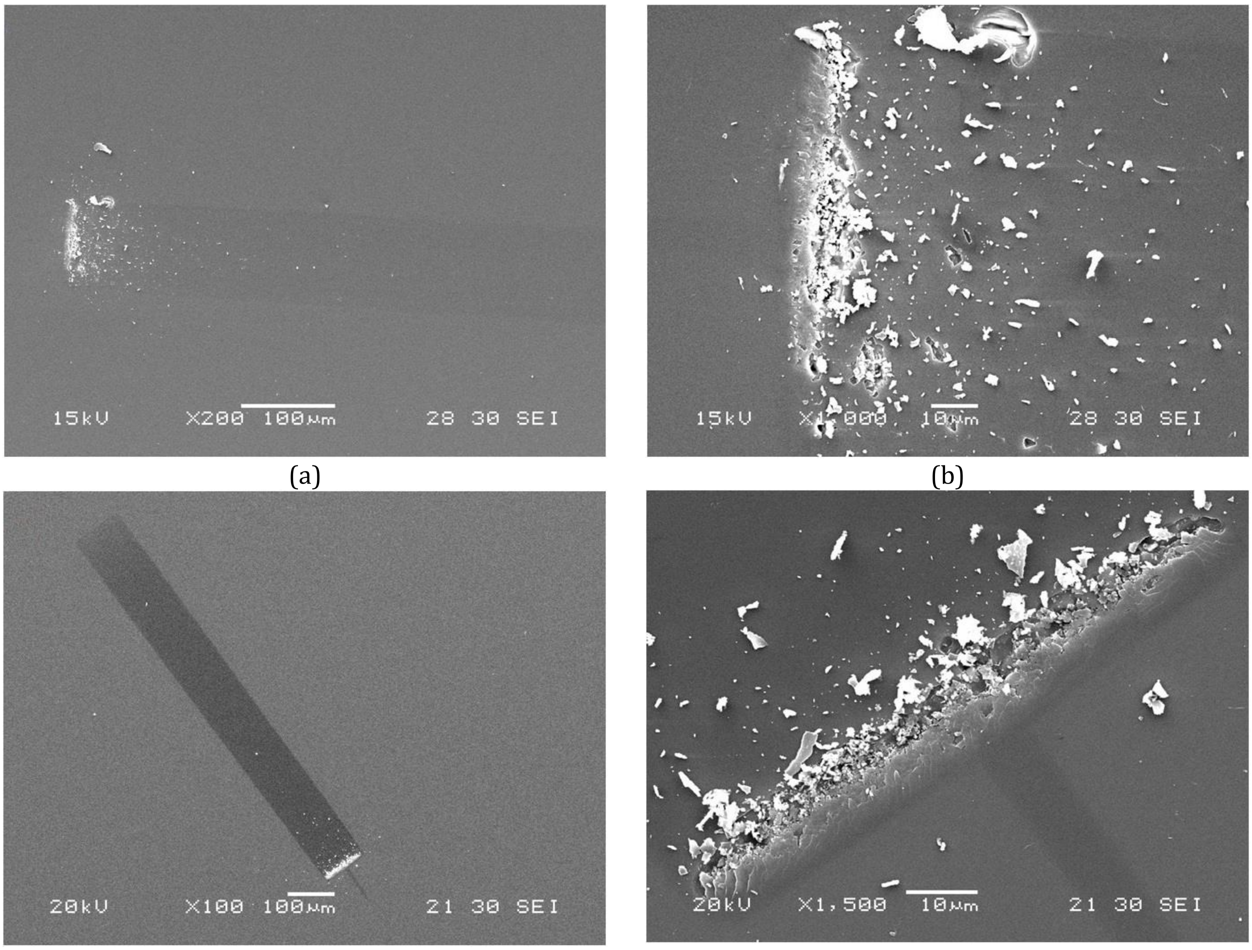


Fig. 5. Surface morphologies of scratched (a and b) NDLC and (c and d) PtRuNDLC-NC coatings observed at different magnifications.

Figures 5a-d show the surface morphologies of the NDLC and PtRuNDLC-NC coatings observed after the scratch tests. In Figure 5a and c, the scratch path of the PtRuNDLC-NC coating is more apparent than that of the NDLC coating probably due to its lower hardness and elastic modulus. However, apparent surface damages are not found on the scratch paths of the both coatings before their adhesive and brittle failures as fragments at their respective critical loads in Figures 5b and d, implying that the cohesive strength of the both coatings are strong enough to withstand their scratch induced surface damages during the scratching [24,25]. It is clear that the both coatings have sufficient scratch resistance to effectively prevent their underlying Si substrates from scratch induced surface damages during the scratching.

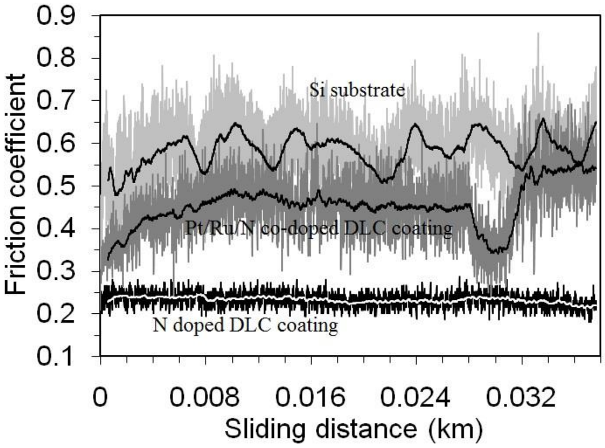


Fig. 6. Friction coefficients of Si substrate and NDLC and PtRuNDLC-NC coatings with respect to sliding distance. The center lines represent their trends of mean friction coefficients versus sliding distance.

Figure 6 presents the friction coefficients of the Si substrate and NDLC and PtRuNDLC-NC coatings tested dry against alumina balls with respect to sliding distance. The Si substrate is used as a reference material to evaluate the wear protective performance of the NDLC and PtRuNDLC-NC coatings. The mean friction coefficient of the Si substrate is 0.58. The friction of the Si substrate apparently increases with its increased wear since a larger contact between two rubbing surfaces results in higher friction via a larger number of contact junctions between them to adhere to each other and then decreases with the formation and growth of tribolayers on its wear track [26-28]. The repeated dry sliding of the alumina ball compacts released wear debris to form and grow tribolayers for decreasing the friction because tribolayers are somewhat harder to prevent a direct contact between two rubbing surfaces and lessen their wear [26-29]. In addition, the tribolayers prevent the wear of the Si substrate to some extent and thereby lessen a contact between two rubbing surfaces, which in turn contribute to its decreased friction. Eventually, the detachment of tribolayers turns to increase the friction of the Si substrate by further increasing its wear. Therefore, the formation and detachment of tribolayers contribute to the wear of the Si substrate and give rise to an up and down variation in its friction throughout the wear test.

Coating of the Si substrate with the NDLC coating apparently reduces the mean friction coefficient to 0.23 that is 60.3 % lower than that of the Si substrate and gives rise to much more stable and much lower friction during the entire sliding. It is hypothesized that N doping forms graphitic clusters in the NDLC coating to reduce its friction during the dry sliding [15,30,31]. In addition, the dry sliding of the alumina ball on the NDLC coating surface probably induces its surface graphitization for its self-lubricating [15,30,31]. Therefore, these effects are responsible for the relatively low friction of the NDLC coating and thereby for its effective wear protective performance.

The PtRuNDLC-NC coating has a mean friction coefficient of 0.45 that are 22.4 % lower and 95.7% higher than those of the Si substrate and NDLC coating, respectively. The reduced hardness and elastic modulus of the PtRuNDLC-NC coating with the incorporation of Pt and Ru are responsible for its higher friction via its larger contact with its counter alumina ball during the dry sliding [26,27,32]. An elastic/plastic contact between soft Pt/Ru aggregates on the PtRuNDLC-NC coating surface and hard asperities on the alumina ball surface gives rise to the higher friction [33]. The rougher surface of the PtRuNDLC-NC coating contributes to its higher friction since mechanical interlocking between mating asperities of two rubbing surfaces in contact generates high friction in a dry sliding process [34,35]. An existence of Pt/Ru aggregates on the PtRuNDLC-NC coating surface probably eliminates its self-lubricating performance by preventing its surface graphitization during the dry sliding. The degraded abrasive wear resistance of the PtRuNDLC-NC coating associated with its reduced hardnes results in its higher wear and hence its higher friction via a larger contact between two rubbing surfaces. Therefore, the friction of the PtRuNDLC-NC coating is relatively higher for all the sliding distance compared to that of the NDLC coating as found in Figure 6. At the near end of the wear test, the PtRuNDLC-NC coating exhibits even higher friction that is close to that of the Si substrate because it is sufficiently worn out to expose its underlying Si substrate during the rubbing contact with the alumina ball.

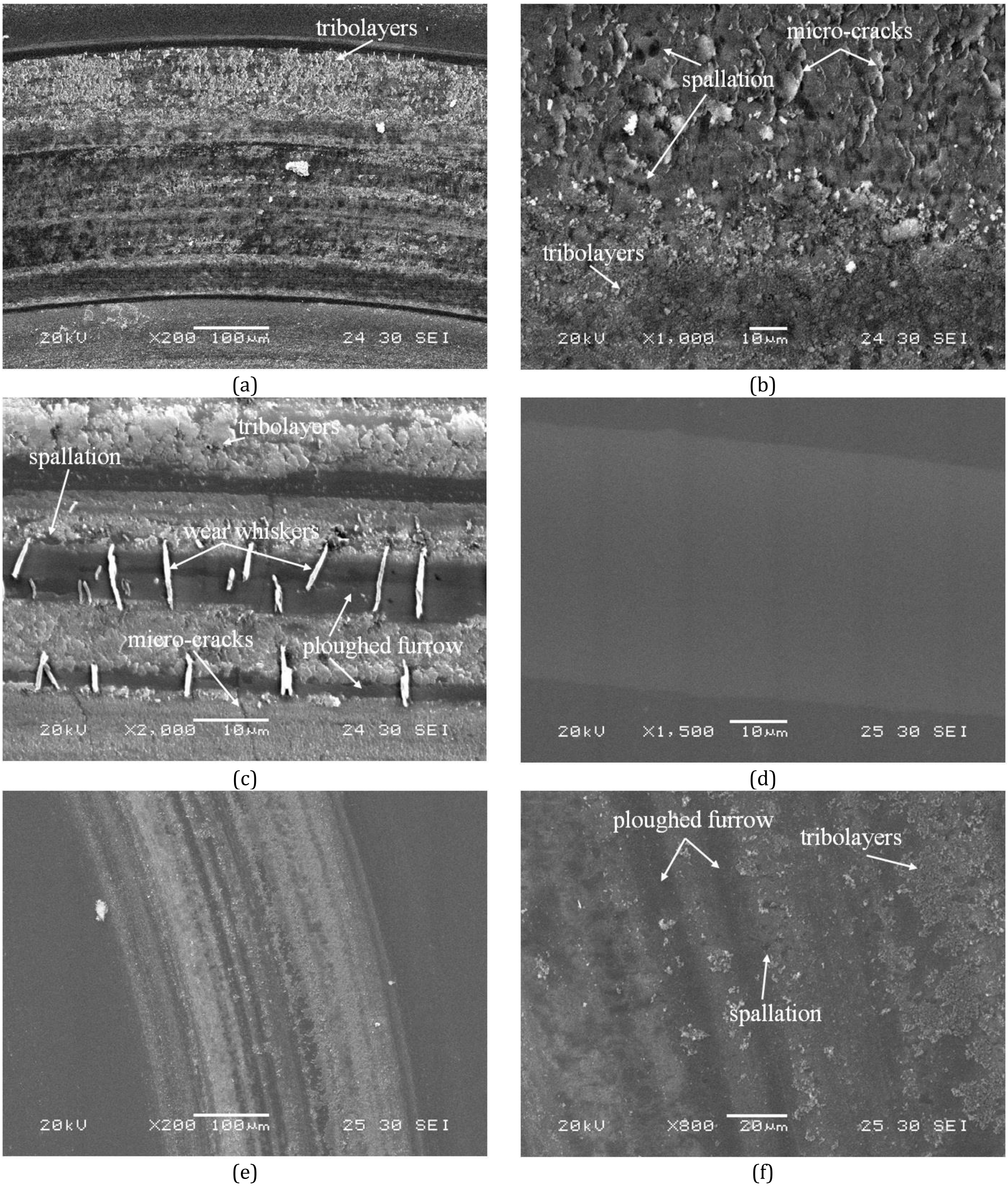


Fig. 7. Wear morphologies of (a, b and c) Si substrate and (d) NDLC and (e and f) PtRuNDLC-NC coatings observed at different magnifications.

The wear volume of the Si substrate is $116.2\pm13.3\times10^{-4}$ mm^3. The wear of the NDLC coating is not measurable, implying that it has an effective abrasive wear protective performance over its underlying Si substrate although its interacting track with about 45-60 µm width is found. However, the PtRuNDLC-NC coating has an apparent wear track with a wear volume of $31.6\pm8.03\times10^{-4}$ mm^3 that is 72.8% lower than that of the Si substrate. It is obvious that the PtRuNDLC-NC coating has the lower abrasive wear resistance than the NDLC coating, but certain wear protective performance over its underlying Si substrate. The reason is that the reduced hardness of the PtRuNDLC-NC coating with the incorporation of Pt and Ru is responsible for its lower abrasive wear resistance under the dry condition [32,36]. Under the applied normal load, the dry sliding of the alumina ball on the PtRuNDLC-NC coating surface results in an easier removal of coating surface materials by causing shear breaking between embedded Pt/Ru aggregates and DLC matrix [32]. Under the suppressed self-lubricating of the PtRuNDLC-NC coating, its higher sp^2 fraction via metal induced graphitization and Pt/Ru aggregates result in its lower abrasive wear resistance via degradation of its sp^3 bonded cross-linking structure [12,15].

The wear morphologies of the Si substrate and NDLC and PtRuNDLC-NC coatings are presented in Figure 7. The severe abrasive wear of the Si substrate is found in Figure 7a. The repeated dry sliding of the alumina ball initiates minute cracks on the surface and in the subsurface, propagates them perpendicular to the sliding direction into the subsurface and parallel to a free surface for some extent, respectively [37-39]. Eventually, minute cracks grown into the subsurface region connect each other to form micro-cracks as well as to remove surface materials as platelets [37-39]. Therefore, micro-cracks and micro-pits are found on the wear track of the Si substrate as shown in Figure 7b, which are indicative of its fatigue wear [37-39]. Ploughed furrows on the wear track of the Si substrate result from its abrasive wear caused by the repeated dry sliding of the alumina ball (Figures 7a-c) [40]. It is clear that the wear of the Si substrate is attributed to the abrasive and fatigue wear. Wear whiskers rolled during the dry sliding are found on the wear track of the Si substrate in addition to tribolayers (Figure 7c). Therefore, tribolayers and wear whiskers with a free-rolling effect are responsible for the decreased friction of the Si substrate during the up and down variation in its friction. In Figure 7c, the relatively large micro-cracks propagated perpendicular to the sliding direction into the subsurface region are found in the middle of the wear track of the brittle Si substrate where the contact pressure in front of the alumina ball is highest during the dry sliding as its significant fatigue damages [37-39].

In Figure 7d, the smooth wear track of the NDLC coating can be seen without any apparent wear damage, confirming that it can effectively protect its underlying Si substrate from wear. However, apparent wear damages on the wear track of the PtRuNDLC-NC coating (Figures 7e and f) confirm its lowered wear resistance associated with the incorporation of Pt and Ru. Together with tribolayers, ploughed furrows and micro-pits are found in the middle of the wear track of the Si substrate (Figure 7f) where the PtRuNDLC-NC coating is completely worn out. Rolled wear whiskers are not found on the exposed surface of the underlying Si substrate probably due to its greatly reduced abrasive wear with the PtRuNDLC-NC coating. No observation of apparent micro-cracks on the exposed Si substrate surface implies that the PtRuNDLC-NC coating apparently prevents the surface fatigue wear of its Si substrate. The wear morphologies confirm that the PtRuNDLC-NC coating cannot give rise to the better wear protective performance during the dry sliding against an alumina ball than the NDLC coating.

4. CONCLUSION

The structure, surface roughness, surface wettability, adhesion strength, hardness, friction and wear protective performance of the NDLC and PtRuNDLC-NC coatings were comparatively investigated.

- The PtRuNDLC-NC coating had the higher sp^2 fraction via metal-induced-graphitization compared to the NDLC coating.
- The surface roughness of the PtRuNDLC-NC coating was 45.7% higher than that of the NDLC coating due to the surface Pt/Ru aggregates and metal-induced graphitization.

- The surface of the PtRuNDLC-NC coating was more hydrophobic with a 15.4% larger water contact angle compared to that of the NDLC coating due to the reduced polar C-N bonds associated with the incorporation of Pt and Ru and the chemical inertness of surface Pt and Ru.
- The PtRuNDLC-NC coating had the 6.6% lower hardness and 3.6% lower reduced elastic modulus than the NDLC coating because of the incorporation of softer Pt and Ru and metal-induced-graphitization.
- The adhesion strength of the PtRuNDLC-NC coating was higher with a 9.5% larger critical load than that of the NDLC coating because the embedded Pt/Ru aggregates and metal-induced-graphitization degraded its rigid sp^3 bonded cross-linking structure and thereby released its residual stress. The both NDLC and PtRuNDLC-NC coatings had relatively strong cohesive strength to withstand scratch induced surface damages during the scratching.
- The NDLC coating exhibited relatively low friction and unmeasurable wear in this study. The PtRuNDLC-NC coating had the lower wear protective performance under a dry condition than the NDLC coating because the incorporation of Pt and Ru lowered its abrasive wear resistance by degrading its sp^3 bonded cross-linking structure, reducing its hardness, and eliminating its surface graphitization. However, the wear of the PtRuNDLC-NC coated Si sample was apparently 72.8% lower than that of the uncoated Si sample, which meant that the PtRuNDLC-NC coating still could effectively prevent its underlying Si substrate from severe wear. The SEM observation confirmed that the PtRuNDLC-NC coating could effectively suppress the fatigue wear of its underlying Si substrate.
- It could be concluded that the co-incorporation of Pt and Ru did not give rise to the higher mechanical strength and better tribological performance of the PtRuNDLC-NC coating than those of the NDLC coating.

Acknowledgement

This work was supported by the research grant (EWI-0601-IRIS-035-00) from the Environment and Water Industry Development Council (EWI), Singapore.

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