



Article

Comparative Study of the Tribological Properties of MoSe₂ Coatings Under Dry and Oil-Lubricated Sliding Conditions

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Abstract

MoSe₂ is considered one of the most promising low-friction coatings for tribological applications due to its exceptionally low sensitivity to air humidity. However, knowledge of its tribological performance, especially in combination with oil lubrication, is still very limited. In this study, the tribological properties of MoSe₂ coatings deposited by magnetron sputtering were investigated using a reciprocating pin-on-flat tribometer against steel balls under both dry and PAO4-lubricated sliding conditions. The worn surfaces of the coatings and their counterparts were analyzed by profilometry, Raman spectroscopy, and scanning and transmission electron microscopy. Under dry lubrication, the coatings exhibited low friction (0.054), which was attributed to the combined effects of a lubricious transfer layer forming on the steel ball and a crystalline MoSe₂ tribolayer in the coating wear track, with MoSe₂ basal planes aligned parallel to the sliding direction. In contrast, under oil lubrication, the absence of a transfer layer on the ball and a crystalline tribolayer in the coating wear track resulted in higher friction (0.101). This high friction was accompanied by a 27% reduction in the wear rate due to the presence of PAO4 at the sliding contact, which served as a sealant and protected the coating from oxidation.

Keywords: MoSe₂; coating; oil lubrication; dry lubrication; PVD; friction; wear

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1. Introduction

Transition metal dichalcogenides (TMDs) such as molybdenum disulfide (MoS₂), tungsten disulfide (WS₂), and molybdenum diselenide (MoSe₂) constitute a group of materials well-known for their self-lubricating properties [1–5]. Their excellent tribological performance stems from their highly anisotropic crystalline structure, originating from strong intraplanar covalent forces within each monolayer and weak interplanar Van der Waals forces between adjacent layers [6–8]. These weak interplanar Van der Waals forces are easily overcome during sliding, allowing the layers to slide over each other with relatively low shear strength, resulting in a low coefficient of friction [7–11]. While this mechanism involves material detachment from the coating, the detached material can form a transfer layer on the sliding counterpart. This transfer layer acts as a protective barrier, reducing direct contact and friction between the coating and the counterpart, thereby significantly minimizing wear and prolonging the coating's lifespan [12–14].

There are two ways in which TMDs are utilized in tribological applications: either as surface coatings [1,15–17] or as nanoparticles incorporated into oil lubricants [18–21]. For

the former approach, MoS₂ is the most extensively investigated material among the TMD family and has found its way into several space applications as a solid lubricant coating due to its extremely low coefficient of friction of 0.002 [17,22–24]. Its most notable early application was on the Apollo Lunar Module's extendible legs in 1969 [17]. Despite its mature application in the space industry, the attempts to apply MoS₂ coating in humid air have largely been unsuccessful. This is because of its high susceptibility to oxidation in humid air, resulting in the degradation of its tribological properties with increasing coefficients of friction and wear [1,15]. To overcome this drawback, the coating has been doped with other elements, such as Ti [25,26], Al [27], Pb [28], C [1,29], Cr [30], and N [31,32], to act as oxygen sinks. Although this approach generally leads to an improvement in the mechanical properties of the coating, the detrimental effect of air humidity on its frictional behaviour has still not been overcome and remains a challenge.

While MoS₂ coating has received extensive research, only a small amount of attention has been directed toward MoSe₂ coating. Nonetheless, studies with MoSe₂ coating have demonstrated that it can be a good alternative to MoS₂ coating, with the advantage of being more resistant to the adverse effect of air humidity and having higher thermal stability [1,33–35]. Kubart et al. [33] compared the tribological properties of MoS₂ and MoSe₂ coatings under different levels of air humidity. MoSe₂ coating was found to outperform MoS₂ coating with lower coefficients of friction and better wear resistance. Additionally, the coefficient of friction of MoSe₂ coating was observed to be almost independent of air humidity.

Although TMD nanoparticles have demonstrated excellent tribological properties for oil-lubricated contacts, several limitations restrict their use across a wider range of applications, one of which is their low solubility and tendency to agglomerate in base oils [18,36,37]. To address this agglomeration, they must be dispersed with chemicals known as dispersant agents. However, the majority of these dispersants are corrosive and can damage the surfaces they come into contact with [38,39]. A further complication is that even if a dispersant is safe for the surface, its compatibility with other oil additives must also be guaranteed. Moreover, concerns have been raised about the safety of these nanoparticles for human health and the environment [40–42]. In this context, the introduction of TMDs into oil-lubricated contacts as surface coatings is a potential alternative to the use of their nanoparticles.

Surprisingly, there have been very few attempts to introduce TMDs into oil-lubricated contacts as surface coatings. Rodrigues et al. [43] deposited F-doped WS₂ coating by magnetron sputtering and evaluated its tribological performance in dry and PAO8-lubricated sliding. The coefficient of friction measured in PAO8-lubricated sliding was 0.07, which was higher than the 0.03 measured in dry sliding. It was reported that this higher coefficient of friction was caused by the uneven spreading of the oil on the sliding surfaces. In another study, Hovsepian et al. [44] investigated the tribological performance of Mo-W-doped carbon-based coating under oil-lubricated sliding conditions. The coating showed a low coefficient of friction of 0.03. Although this low friction was attributed to the in situ formation of a tribolayer containing particles of WS₂ and MoS₂ on sliding surfaces, the lubricating oil used was a fully formulated engine oil, making it difficult to determine whether the coating itself formed the tribolayer independently without any synergistic or antagonistic influence of oil additives. Despite the great promise of MoSe₂ coating for tribological applications, only one study has been carried out so far to examine its tribological performance in lubricated sliding. This work was conducted by Gustavsson et al. [45], who compared the tribological performance of different coatings based on diamond-like carbon (DLC), C-doped MoSe₂, and Ti-doped MoS₂ in five commercially available fuels. C-doped MoSe₂ coating showed very promising friction results in several fuels such as diesel. How-

ever, the tests were conducted with fuels intended for combustion engines, rather than for the lubrication of tribological surfaces. Apart from that, the underlying mechanisms contributing to these friction results were not elucidated. For the actual assessment of the potential of MoSe₂ coating for tribological applications, a thorough investigation and understanding of its tribological performance with oil lubrication are needed since most mechanical components are routinely used under oil-lubricated conditions.

The aim of this work was to investigate the influence of oil lubrication on the tribological properties of MoSe₂ coating. The coating was deposited by magnetron sputtering, and its tribological performance was evaluated using a reciprocating pin-on-flat tribometer under PAO4-lubricated sliding conditions. For comparison, its tribological behaviour was also assessed under dry sliding conditions. To the best of our knowledge, this is the first study to report on the tribological performance of MoSe₂ coatings under oil-lubricated sliding conditions.

2. Materials and Methods

The experimental procedures and methodology employed in this study were adapted from the foundational work presented in Dr. Saad Alshammari's PhD thesis [46]. MoSe₂ coatings were deposited on 100Cr6 steel discs with hardness of 8 GPa, a diameter of 50 mm, and a thickness of 5 mm. Additionally, a wafer of polished borosilicate glass was coated for coating thickness measurement. The discs were ground and polished to a surface roughness (R_a) of 0.03 ± 0.004 μm . They were then plasma etched with oxygen using a microwave plasma etching system (Tepla 300, PVA Tepla, Wetztenberg, Germany) to remove any carbon residue. The deposition took 22 h in a radio frequency (RF) magnetron sputtering system (Nano 38, Kurt J. Lesker, PA, USA) with a background pressure of 3 mTorr. The substrates were initially kept at room temperature with less than 10 °C increase observed during deposition. The coatings were fabricated from a MoSe₂ sputtering target with 99.99% purity. The power applied to the target was kept constant at 80 W, and the distance between the target and the substrates was kept at 150 mm. Argon (99.99% purity) was used as the sputtering gas at a constant gas flow of 20 sccm. The thickness of the deposited coatings was 2 ± 0.18 μm measured by a 3D optical profilometer (Infinite Focus, Alicona, Raaba, Austria). Energy Dispersive X-ray spectroscopy (EDS, Aztec, Oxford Instruments, Oxfordshire, UK) was used to determine the chemical composition of the coatings. The mechanical properties of the coatings (i.e., hardness (H) and reduced elastic modulus (E_r)) were evaluated using a depth-sensing indentation technique. This was performed with a nanoindenter (NanoTest Vantage system, Micro Materials) equipped with a diamond Berkovich tip having a radius of 5 μm . Indentations were conducted in a depth-controlled mode, with a maximum depth of 100 nm to ensure it remained below 10% of the coating thickness. Adjacent indents were spaced 10 μm apart to prevent any overlapping of the indented areas. There was a total of 25 indents, and the average values of H and E_r were calculated based on the method proposed by Oliver and Pharr [47]. The crystal structure of the coatings was analyzed using X-ray diffraction (XRD, SmartLab, Rigaku, Tokyo, Japan) with Cu K α radiation ($\lambda = 1.5418$ Å) at a grazing incidence angle of 2°.

The tribological properties of the coatings were assessed using a reciprocating pin-on-flat tribometer (TE77, Phoenix Tribology, Hampshire, UK). AISI 52100 steel balls with a diameter of 6 mm and hardness of 8 GPa were used as sliding counterparts. Prior to tribological testing, the coatings and the steel balls were ultrasonically cleaned in acetone and allowed to dry naturally at room temperature. The tests were carried out under dry sliding conditions at room temperature with air humidity ranging from 40% to 50%. A constant applied load of 5 N was used, corresponding to a maximum Hertzian contact pressure of 1.11 GPa. The reciprocating frequency was kept at 10 Hz over a stroke length of

10 mm, producing an average sliding speed of 0.2 m/s. The test duration and total sliding distance were 5 min and 60 m, respectively. A minimum of 3 tests were performed on each sample.

For oil-lubricated testing, the base oil used was synthetic polyalphaolefin of viscosity grade 4 (PAO4), supplied by ExxonMobil. The oil had a density of 0.81 g/cm³ and viscosity of 29.67 mm²/s at 25 °C. Prior to each test, a small amount of PAO4 was manually applied to the surface of the coated disc, ensuring that the lubricant adhered to the ball via surface tension and maintained the contact area fully immersed in oil throughout testing. No additional oil was supplied during the test, as the initial application was sufficient to maintain lubrication for the test duration. The dimensionless lambda ratio was calculated using the well-known Hamrock–Dowson equation [48]. The calculated value was 1.3 (see Figure A1 in Appendix A), indicating that the operating lubrication regime was within the mixed lubrication range. All the tests were repeated twice for verification and reproducibility of the results. Upon completion, the discs and steel balls tested under dry sliding conditions were ultrasonically cleaned in acetone for 5 min, while those tested under PAO4-lubricated conditions were ultrasonically cleaned in petroleum ether for 5 min to remove residual oil.

The 3D optical profilometer was used to determine the wear volumes. The wear volume of the coating was calculated based on the cross-sectional area and the length of the wear track, whereas the wear volume of the steel ball (V_b) was determined using Equation (1):

$$V_b = \frac{\pi d^4}{64R} \quad (1)$$

where R and d are the radius of steel ball and the diameter of wear scar, respectively. After determining the wear volumes of the coatings and steel balls, their specific wear rates were calculated using Archard wear equation as stated in Equation (2):

$$k = \frac{V}{F \cdot s} \quad (2)$$

where k represents the specific wear rate, F is the normal load, V is the wear volume, and s is the total sliding distance.

The morphology of the worn surfaces was examined by scanning electron microscopy (SEM, FEI Quanta 200, FEI, Hillsboro, OR, USA). Raman spectroscopy (Renishaw inVia, Horiba, Kyoto, Japan) with a green laser of 532 nm wavelength was used to study tribo-induced structural changes in the worn surfaces. A laser spot diameter of 1.6 µm and a laser power of 2 mW were used for all acquisitions, with a total scan time of 60 s for each spectrum. Direct observation of cross-section samples, selected from the as-deposited coating and wear tracks, was performed using high-resolution transition electron microscopy (HR-TEM, Titan Themis Cubed X-FEG, FIE) operating at an accelerating voltage of 300 kV. The cross-section samples were prepared using a focused ion beam (FIB, FEI Helios G4 CX, Thermo Fisher Scientific, Waltham, MA, USA) equipped with a gallium ion source and operated at an accelerating voltage of 30 kV. Prior to ion milling, a protective platinum (Pt) layer was deposited to protect the region of interest from ion beam exposure during the milling process. Final thinning was performed at a reduced accelerating voltage of 5 kV to minimize sample damage.

3. Results

3.1. Crystalline Structure

XRD was used to characterize the crystalline structure of the deposited coating, and the corresponding diffraction pattern is shown in Figure 1. The diffraction peaks observed

at 32.15° , 37.83° , 47.45° , 57.81° , and 67.26° are well corresponding to the crystal planes of (1 0 0), (1 0 3), (1 0 5), (1 1 2), and (2 0 2), respectively, which are consistent with the hexagonal MoSe_2 crystal structure (ICDD card No. 00-077-1715). The diffraction peak associated with the (0 0 2) plane shifts slightly to a lower diffraction angle, from 13.69° to 12.65° . This shift indicates a lattice elongation caused by defects such as vacancies, impurities, and dislocations, as reported by Dunn et al. [49]. The broad peak at approximately 37.83° is highly asymmetric with a long tail toward higher angles, which is a typical feature of sputtered TMD coatings [5,50–55]. According to Weise et al. [56], this broad and asymmetric peak corresponds to a turbostratic stacking of (10 L) planes (where $L = 0, 1, 2, 3, \dots$), indicating that the deposited coating has a mixed-phase structure. This structure contains both amorphous regions and disordered crystalline (semi-crystalline) MoSe_2 nanocrystals.

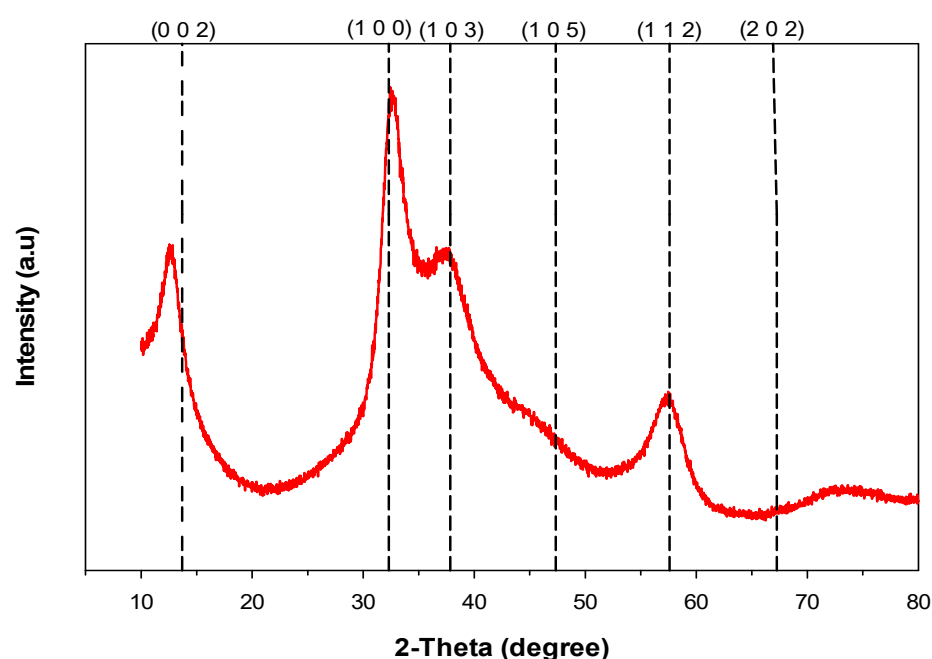


Figure 1. X-ray diffraction pattern of MoSe_2 coating and standard diffraction lines from (ICDD card No. 00-077-1715).

3.2. Chemical Composition, Mechanical Properties, and Cross-Sectional Morphology

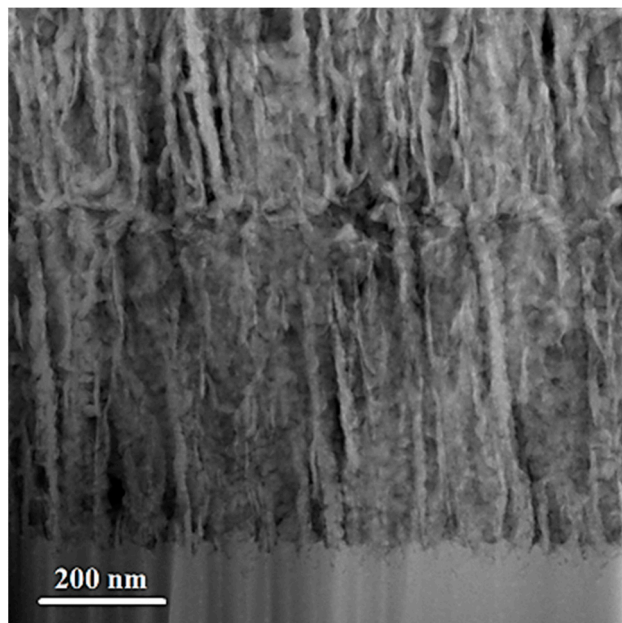
The chemical composition obtained from EDS analysis and the mechanical properties of MoSe_2 coating are summarized in Table 1. A small percentage of oxygen was detected in the coating, likely due to residual oxygen in the deposition chamber and/or contamination from the porous MoSe_2 target [49,50]. The fabricated coating exhibited a hardness value of 0.83 ± 0.07 GPa, which is reasonable considering its porous and columnar structure, see Figure 2 TMD coatings in their pure sputtered form are known to exhibit low compactness, high porosity, and a columnar cross-sectional morphology [1,50–52,56]. These structural characteristics result in low hardness, typically ranging from 0.3 to 2 GPa, depending on the deposition parameters used and the resulting surface morphology [1].

Table 1. Chemical composition and mechanical properties of MoSe_2 coating.

Chemical Composition (at.%)	
Mo	32.51 ± 0.2
Se	65.08 ± 0.1
O	2.41 ± 0.2

Table 1. *Cont.*

Mechanical Properties (GPa)	
Hardness (H)	0.83 ± 0.07
Reduced elastic modulus (E_r)	27.86 ± 1.7

**Figure 2.** Cross-sectional TEM image reveals the porous and columnar structure of MoSe₂ coating.

3.3. Tribological Behaviour

3.3.1. Friction Behaviour

Under both sliding conditions, the friction curves exhibited similar features, rapidly increasing to their highest values at the start of the tests before stabilizing at steady levels, see Figure 3. In the lubricated test, the coating displayed typical unadditized behaviour, with a coefficient of friction averaging around 0.1 and fluctuating between 0.08 and 0.14. In contrast, dry sliding showed different frictional behaviour, with the coating exhibiting a low coefficient of friction of 0.054 and very small fluctuations in the friction curve. The average coefficient of friction measured in PAO4-lubricated sliding was 0.101, which was higher than the 0.054 measured in dry sliding. In other studies, coefficients of friction ranging from 0.05 to 0.06 were reported for pure MoSe₂ coatings tested at the same load under dry sliding conditions [33,50,57]. In comparison to MoS₂ coating, the coefficient of friction of 0.054 measured here for MoSe₂ coating is significantly less than the 0.14 reported for pure MoS₂ coating evaluated under dry sliding conditions at the same load [33]. This behaviour is expected, as MoSe₂ coating is less sensitive to air humidity than MoS₂ coating, as reported by several studies [33,57–59].

3.3.2. Wear Behaviour

Figure 4 displays the cross-sectional profiles measured at three different positions on the wear tracks of the coatings. Because the coating had lower hardness and wear resistance than the steel ball, it suffered from severe wear damage under both sliding conditions, as evidenced by wide and deep wear tracks and a significant number of wear grooves. Despite the severity of wear, the wear track depth did not go beyond the thickness of the MoSe₂ layer (i.e., 2 μm) under both sliding conditions. In PAO4-lubricated sliding, the wear track depth was 1.75 μm , whereas dry sliding resulted in a wear track depth

of 1.98 μm , which was very close to the coating/substrate interface. Figure 5 depicts the volumetric wear rates of the coatings and their sliding counterparts. The coating evaluated in PAO4-lubricated sliding had a wear rate of $15.2 \times 10^{-6} \text{ mm}^3/\text{N.m}$, whereas a wear rate of $20.8 \times 10^{-6} \text{ mm}^3/\text{N.m}$ was measured for the one evaluated in dry sliding. It can be inferred that the addition of PAO4 resulted in a decrease in the wear rate of the coating by 27%, clearly demonstrating the advantage of PAO4 lubrication for improving the wear resistance of the coating. The wear rates measured for the steel balls were significantly lower than those of the coatings, suggesting that the steel balls were more resistant to wear. However, an opposite trend was observed for the steel balls: the one tested in dry sliding exhibited a lower wear rate compared to the one tested in PAO4-lubricated sliding.

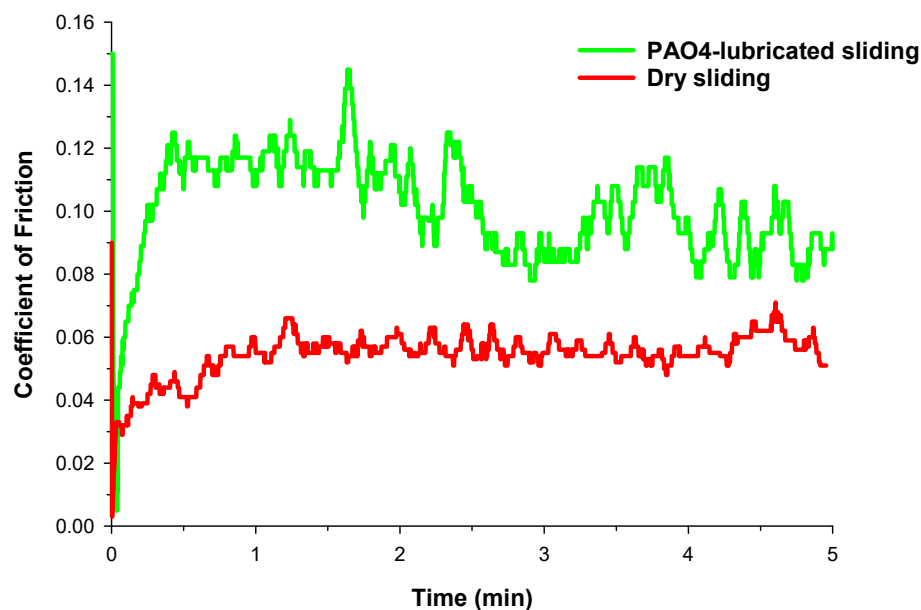


Figure 3. Coefficients of friction of MoSe₂ coatings as a function of sliding time tested under dry and PAO4-lubricated sliding conditions at a normal load of 5 N.

3.3.3. Analysis of Worn Surfaces

PAO4-Lubricated Sliding

To gain more insights into the underlying friction and wear mechanisms, the worn surfaces of the coatings and their sliding counterparts were analyzed using SEM, EDS, Raman spectroscopy, and TEM. Figure 6 shows SEM image of the wear track of the coating tested in PAO4-lubricated sliding. The wear track was approximately 435 μm in width and exhibited a non-uniform appearance. Severe coating delamination and chipping were observed at its edges, indicative of deteriorated wear resistance. Pure MoSe₂ coating exhibits typical brittle nature and low load-bearing capacity, attributed to its columnar and porous morphology [1,60,61]. Consequently, brittle chipping and delamination of the coating during sliding were expected. EDS analysis confirmed the wear damage observed in profilometry, revealing a significant difference in the chemical composition between the worn and unworn coating surfaces, see Figure 7. Analysis of the worn surface showed a dominant presence of iron and depletion of molybdenum and selenium. It should be emphasized that EDS is not a surface-sensitive technique and instead collects signals from a depth of a few microns into a tested sample. Therefore, this observed dominant presence of iron originates from the steel substrate beneath the remaining coating. EDS point analysis at the center of the wear track (point B), which was the area with the most severe damage, showed a small increase in the intensity of the peak belonging to oxygen compared to that of the as-deposited coating (point A), see Figure 8.

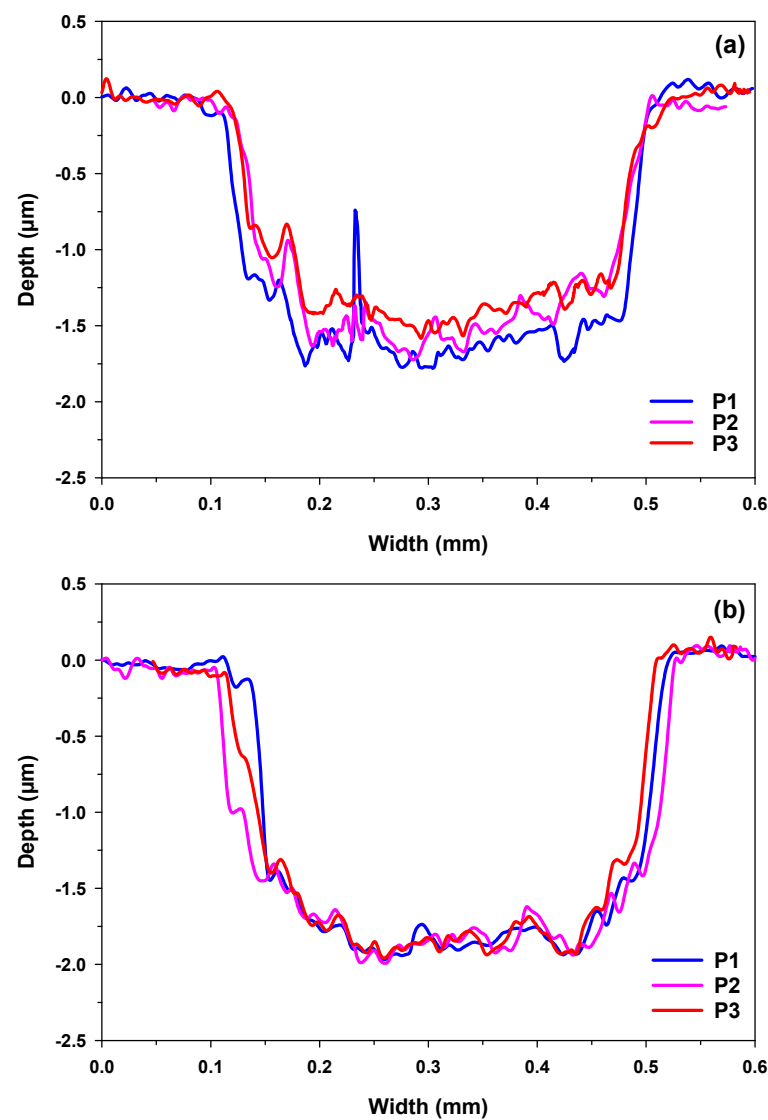


Figure 4. Cross-sectional wear track profiles of MoSe₂ coatings tested in: (a) PAO4-lubricated sliding and (b) dry sliding. P1 is measured at the center of the wear track, whereas P2 and P3 are measured at its edges.

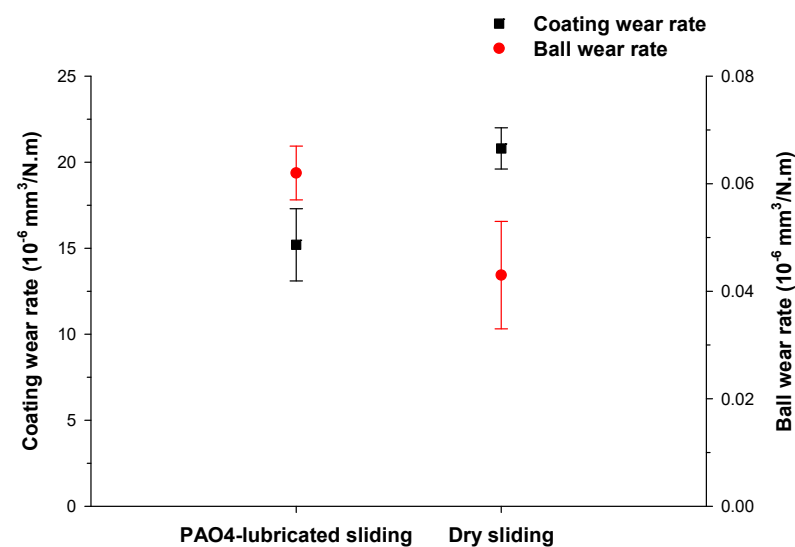


Figure 5. Wear rates of MoSe₂ coatings and their sliding counterparts tested under dry and PAO4-lubricated sliding conditions. The error lines correspond to the standard deviation of the mean value.

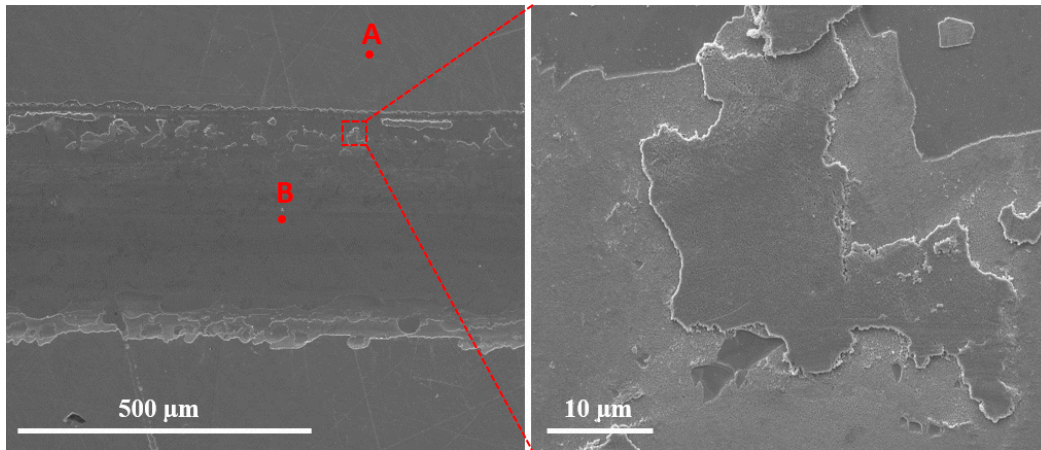


Figure 6. SEM image of the wear track of MoSe₂ coating tested in PAO4-lubricated sliding. The image on the right shows a close-up view of the top edge of the wear track.

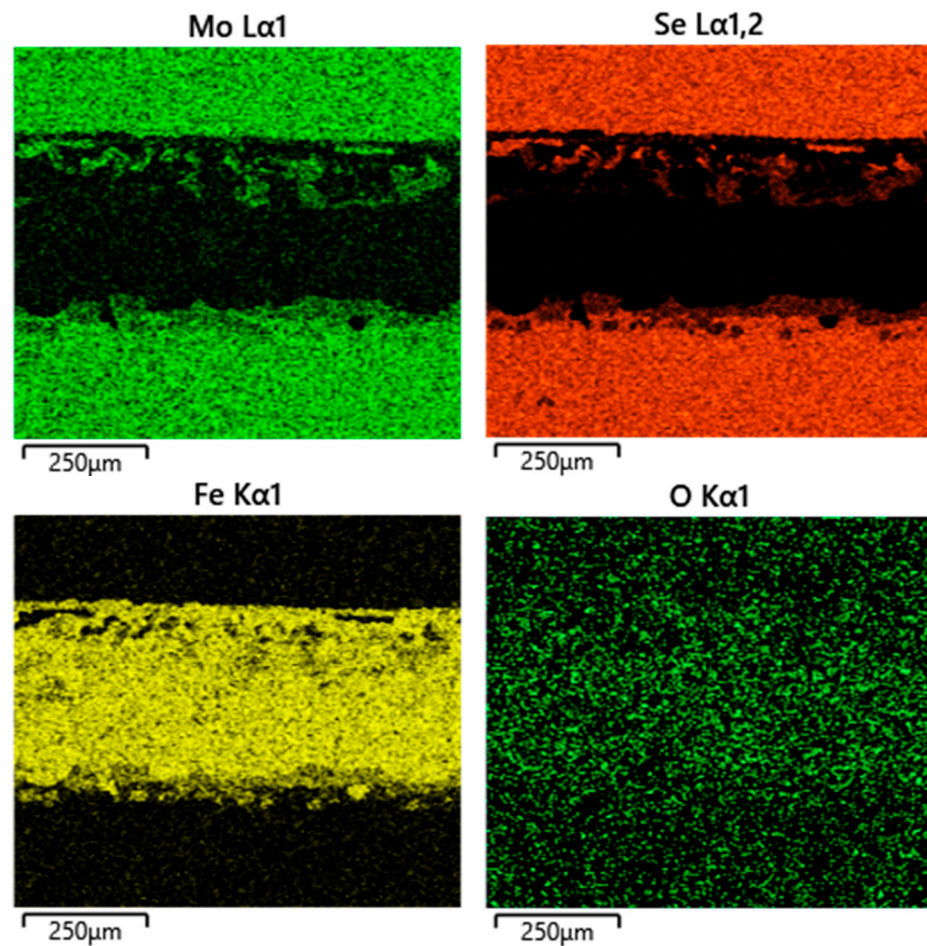


Figure 7. EDS mapping of the wear track of MoSe₂ coating tested in PAO4-lubricated sliding.

Raman spectroscopy, with its superficial detection depth and greater surface sensitivity compared to EDS, was used to investigate potential microstructural changes in the coating wear tracks and their sliding counterparts. The Raman spectrum of the as-deposited coating (reference) showed five peaks corresponding to MoSe₂ at wavenumbers 155 cm^{−1}, 235 cm^{−1}, 290 cm^{−1}, 443 cm^{−1}, and 596 cm^{−1} [54,62], see Figure 9. The Raman spectra acquired from different positions on the wear track showed that it was still largely covered with MoSe₂ despite the severity of wear. These spectra appeared similar to that of the

as-deposited coating, indicating that no tribo-induced structural changes in the crystallinity of MoSe₂ occurred within the wear track. This is consistent with other findings where Raman spectroscopy did not detect structural changes in TMD coatings within wear tracks [29,32,63].

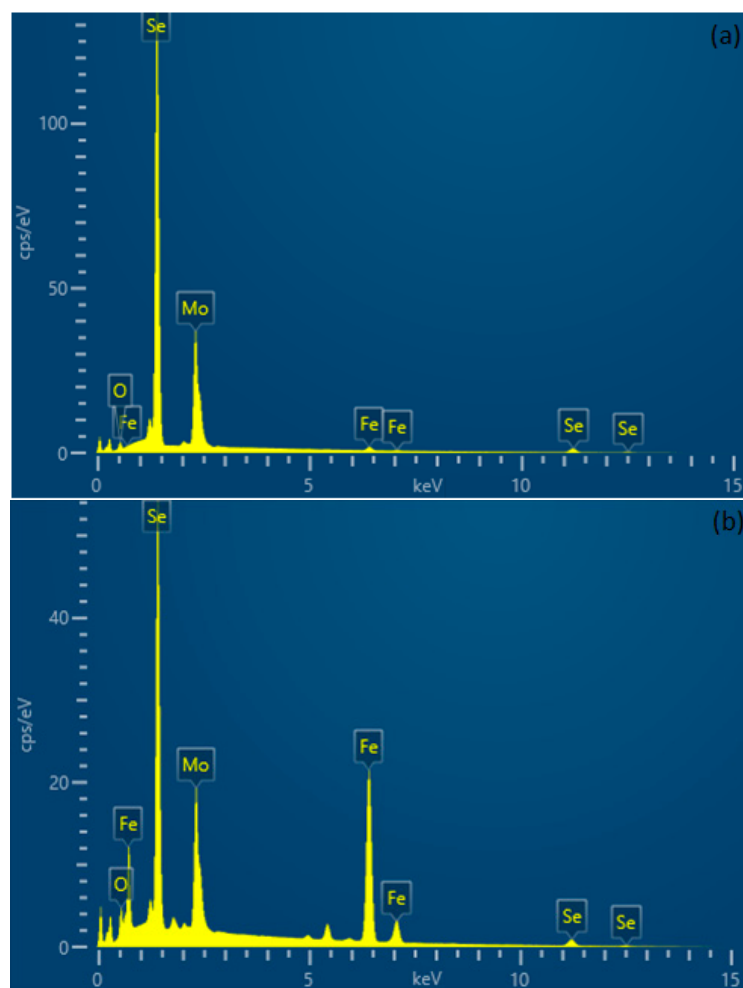


Figure 8. EDS point analysis of: (a) the as-deposited MoSe₂ coating (point A) and (b) the center of the wear track of the coating tested in PAO4-lubricated sliding (point B).

A thin lamella was extracted from the wear track for TEM analysis, see Figure 10. The image did not reveal any change in the crystallinity of MoSe₂ within the wear track, and consequently, a well-ordered crystalline MoSe₂ tribolayer did not form. The MoSe₂ basal planes in the topmost surface of the wear track were either perpendicular or randomly oriented relative to the sliding direction, preventing them from acting as easy slip planes, which are essential for minimizing friction in TMD coatings. This TEM result confirmed the observation of Raman spectroscopy and indicated that PAO4 hindered the formation of a crystalline MoSe₂ tribolayer.

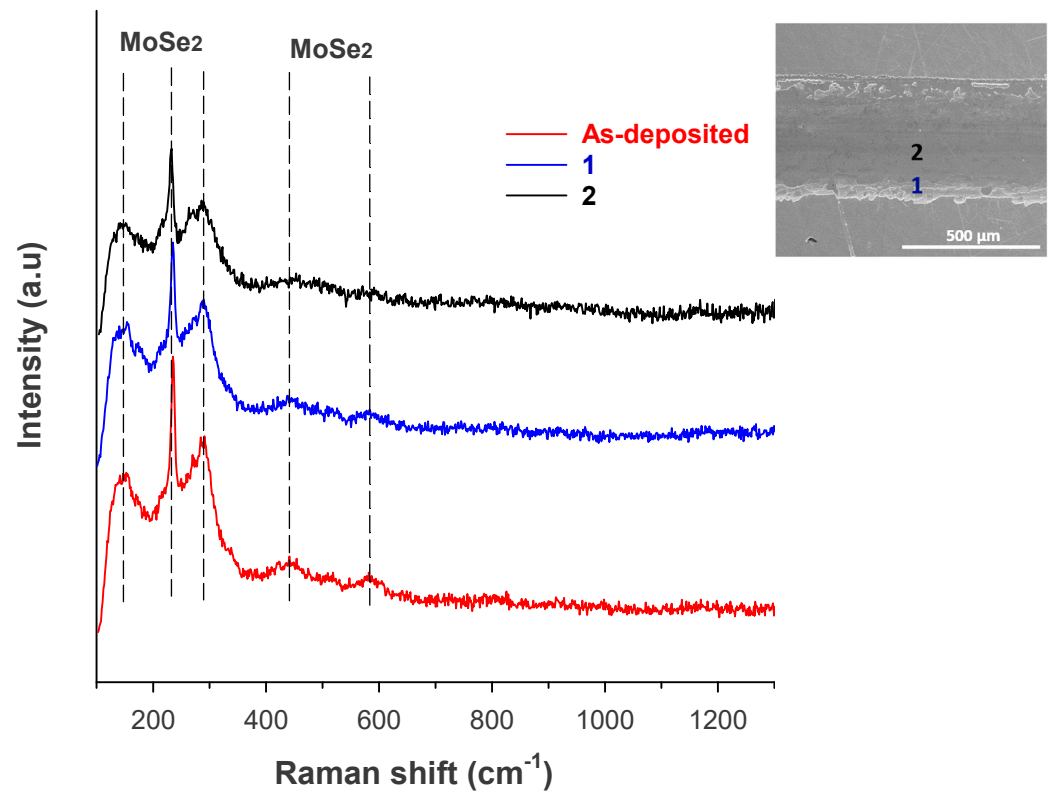


Figure 9. Raman spectra acquired from different positions on the wear track of MoSe_2 coating tested in PAO4-lubricated sliding.

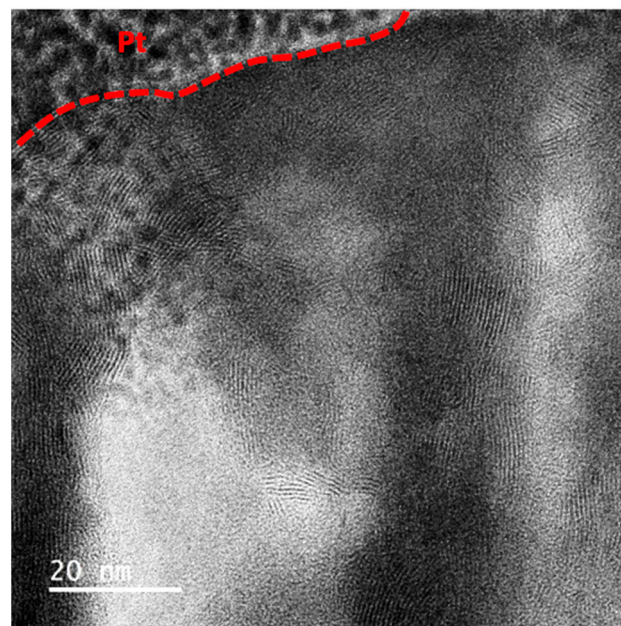


Figure 10. TEM image of the topmost surface of the wear track of MoSe_2 coating tested in PAO4-lubricated sliding shows no evidence of the formation of a crystalline MoSe_2 tribolayer. MoSe_2 basal planes are either perpendicular or randomly oriented relative to the sliding direction.

Figure 11 shows the surface morphology of the ball wear scar. The wear scar was circular in shape, exhibited a smooth appearance, and had a large diameter of 385 μm . Some wear particles were visible around the edges of the wear scar. As shown in Figure 12, EDS mapping revealed that these edges were rich in molybdenum and selenium, indicating the transfer and adherence of some MoSe_2 material to the ball during sliding. This transfer

layer was not uniformly distributed on the wear scar but rather adhered to its edges. This uneven distribution could be attributed to the presence of PAO4, which acted as a barrier between the sliding surfaces, preventing the uniform bonding of wear particles to the ball.

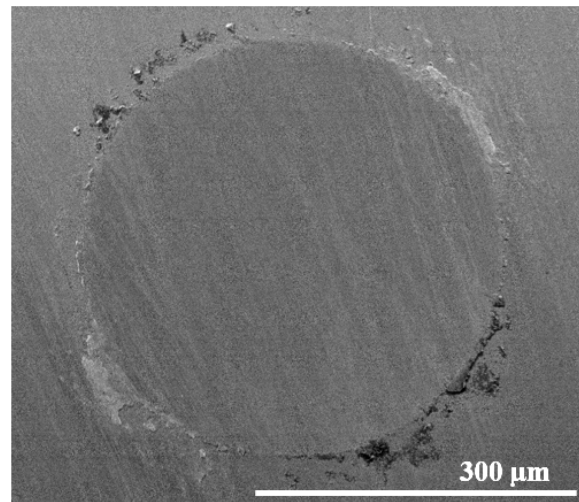


Figure 11. SEM image of the ball wear scar tested against MoSe₂ coating in PAO4-lubricated sliding.

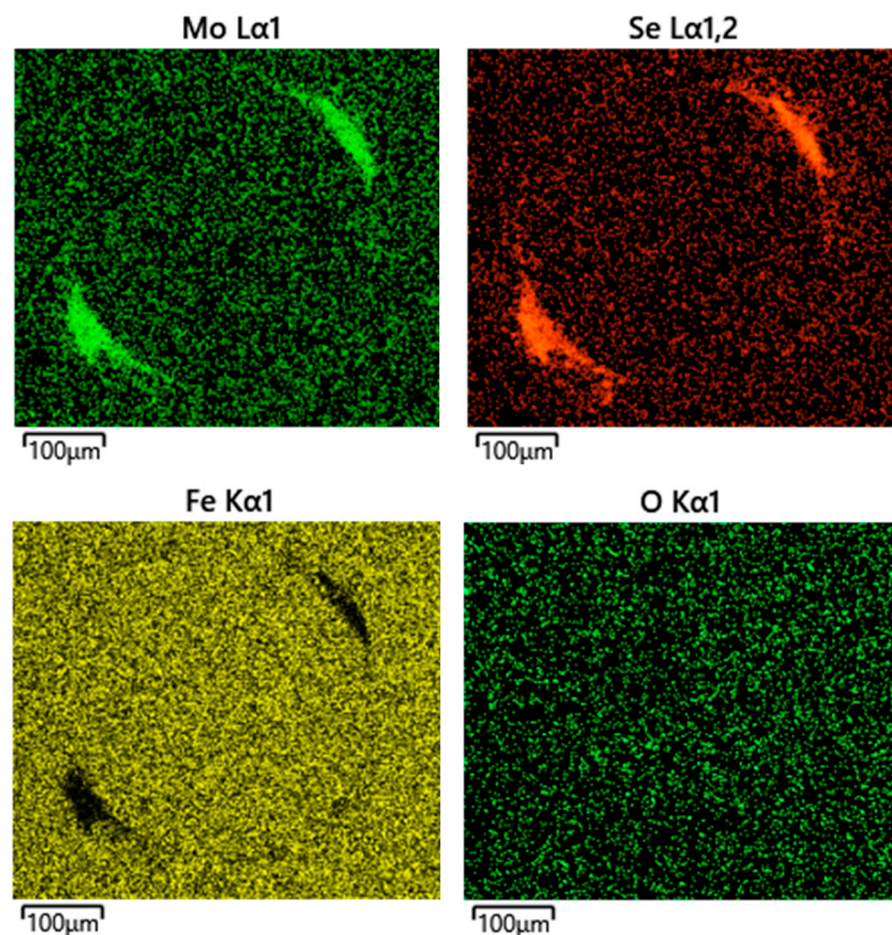


Figure 12. EDS mapping of the ball wear scar tested against MoSe₂ coating in PAO4-lubricated sliding.

Figure 13 shows the Raman spectra acquired from different positions on the ball wear scar. These spectra differed significantly from those acquired from the wear track shown in Figure 9. The Raman spectrum acquired from the center of the wear scar (position 3)

revealed very broad and not easily identifiable MoSe_2 peaks, suggesting that only a small amount of the coating material adhered to the center of the wear scar. Clear peaks of MoSe_2 with smaller intensities than those of the as-deposited coating were only observed at positions 1 and 2 on the wear scar. The Raman spectra acquired from the center and edge of the wear scar (positions 3 and 1) showed two broad peaks at wavenumbers 815 cm^{-1} and 940 cm^{-1} , attributable to MoO_3 [64,65]. In comparison to the intensities of MoSe_2 peaks in the as-deposited coating, the lower intensities of MoSe_2 peaks in the wear scar at all the analyzed positions clearly indicated that only a small amount of the coating material transferred and adhered to the ball during sliding, further supporting and confirming the results obtained from EDS.

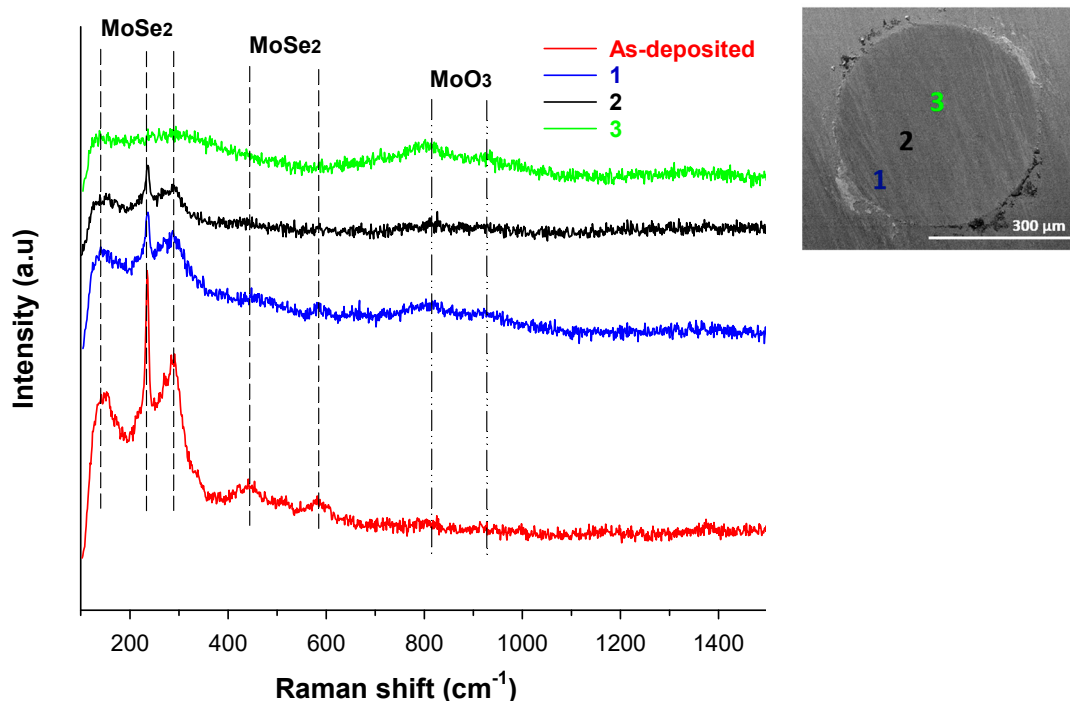


Figure 13. Raman spectra acquired from different positions on the ball wear scar tested against MoSe_2 coating in PAO4-lubricated sliding.

Dry Sliding

As shown in Figure 14, the wear track was $455\text{ }\mu\text{m}$ in width, exceeding the width of the PAO4-lubricated test. Similarly to the lubricated test, the dry sliding wear track also exhibited surface delamination and chipping around its edges. EDS mapping revealed a high concentration of iron within the wear track, and depletion of molybdenum and selenium compared to the untested coating surface, see Figure 15. It is important to emphasize again that EDS is not a surface-sensitive technique and instead collects signals from a depth of a few microns into a tested sample. Therefore, the observed dominant presence of iron originates from the steel substrate beneath the remaining coating, indicating significant wear and depletion of the MoSe_2 layer during dry sliding. The presence of oxygen was also detected within the wear track, particularly in scratches parallel to the sliding direction. EDS point analysis performed at the center of the wear track (point B), which was the area with the most severe damage, showed a significant increase in the intensity of the peak belonging to oxygen compared to that of the as-deposited coating (point A), see Figure 16. This was likely to be the reaction of the substrate with oxygen in the surrounding atmosphere, forming iron oxides.

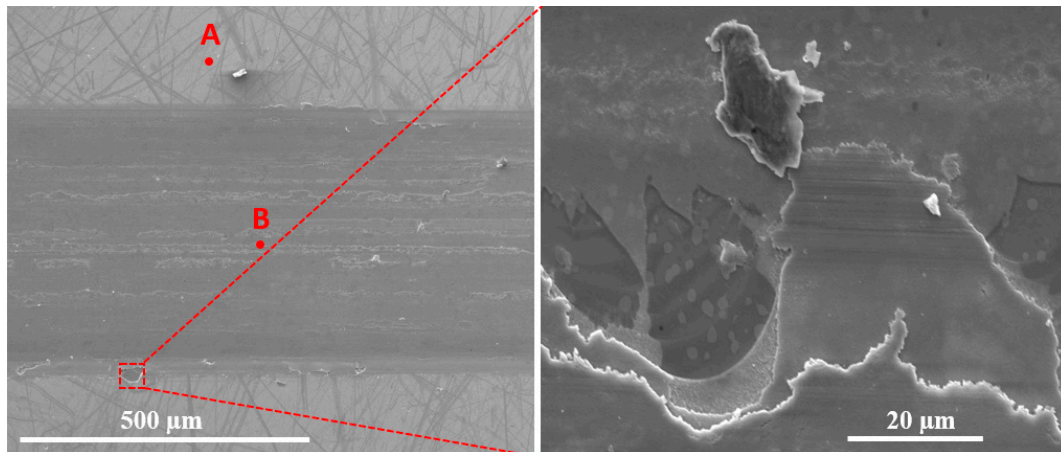


Figure 14. SEM image of the wear track of MoSe₂ coating tested in dry sliding. The image on the right shows a close-up view of the bottom edge of the wear track.

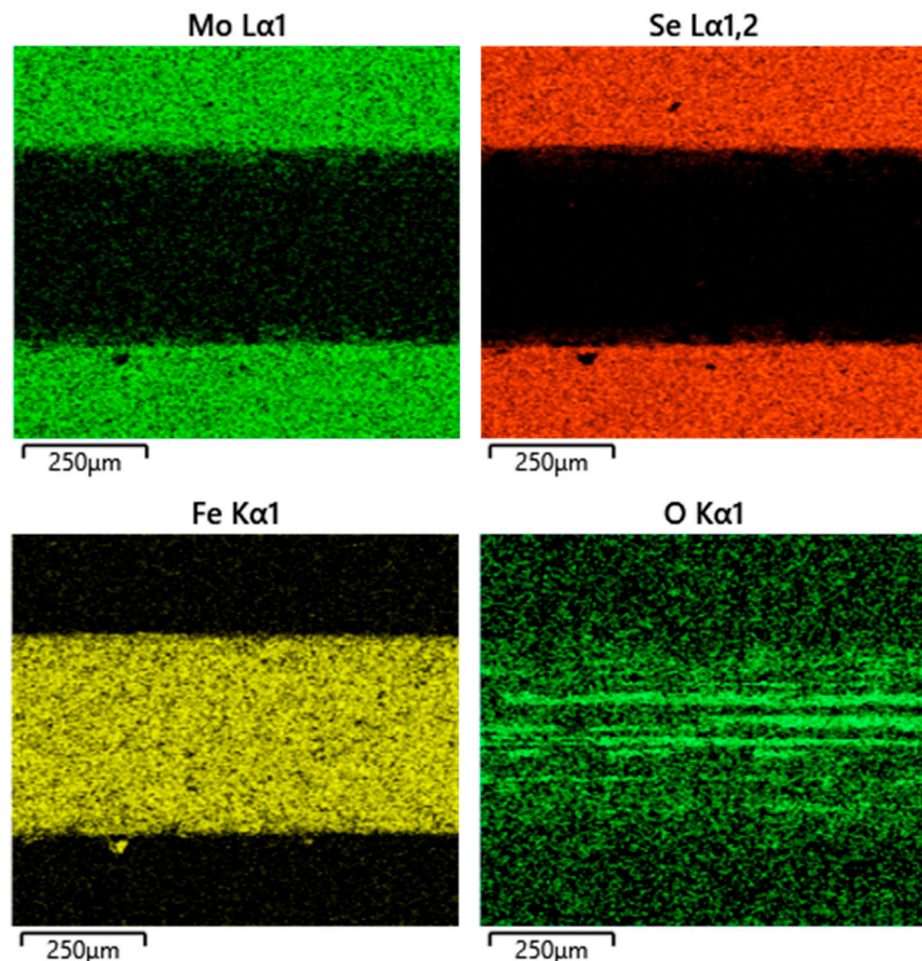


Figure 15. EDS mapping of the wear track of MoSe₂ coating tested in dry sliding.

Figure 17 shows the Raman spectrum of the as-deposited coating as a reference and the spectra acquired from different positions on the wear track of the coating. The Raman spectrum of the as-deposited coating showed five peaks corresponding to MoSe₂ at wavenumbers 155 cm⁻¹, 235 cm⁻¹, 290 cm⁻¹, 443 cm⁻¹, and 596 cm⁻¹ [54,62]. The Raman spectrum collected from the edge of the wear track (position 1) revealed that the MoSe₂ peak at wavenumber 235 cm⁻¹ was sharper and had a higher intensity than the one observed in the as-deposited coating. This was an indicator that a crystalline MoSe₂

tribolayer likely formed in the topmost surface of the wear track, which was consistent with the findings of earlier tribological studies of MoSe₂ coatings [54,66–69]. The presence of two broad peaks corresponding to MoO₃ at wavenumbers 815 cm^{−1} and 940 cm^{−1} at position 2 suggested that the coating experienced an oxidation reaction during dry sliding. This result was different from the wear track of the PAO4-lubricated sliding test shown in Figure 9, which did not reveal any peaks corresponding to MoO₃. This difference is a clear indication of the beneficial role of PAO4 in sealing the contact and protecting the coating from oxidation.

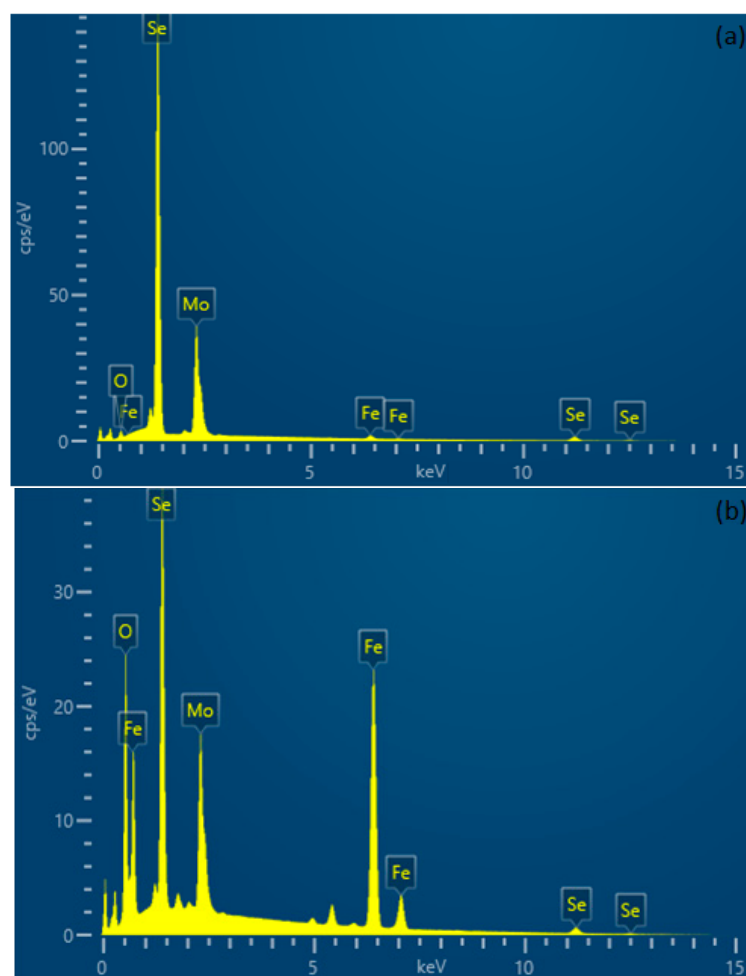


Figure 16. EDS point analysis of: (a) the as-deposited MoSe₂ coating (point A) and (b) the center of the wear track of the coating tested in dry sliding (point B).

The results of Raman spectroscopy indicated the likely formation of a crystalline MoSe₂ tribolayer in the topmost surface of the wear track during dry sliding. This was further investigated by subjecting a thin lamella extracted from the wear track to TEM imaging, see Figure 18. The image clearly revealed the formation of a crystalline MoSe₂ tribolayer in the topmost surface of the wear track, thus confirming the results of Raman spectroscopy. In contrast to the original coating's mostly amorphous structure with a small number of MoSe₂ nanocrystals, the formed tribolayer was approximately 10 nm thick and consisted of MoSe₂ basal planes aligned parallel to the sliding direction. This alignment resulted in low interfacial shear strength at the sliding interface, leading to a low coefficient of friction during dry sliding.

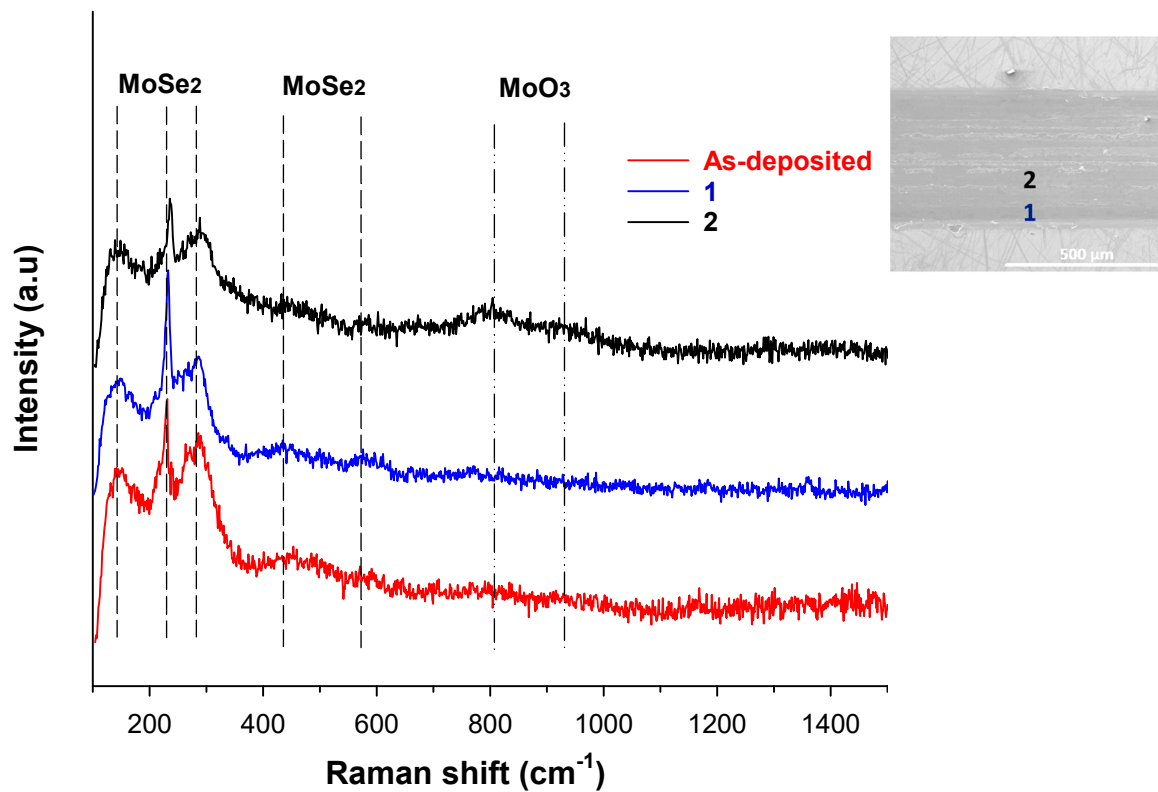


Figure 17. Raman spectra acquired from different positions of the wear track of MoSe_2 coating tested in dry sliding.

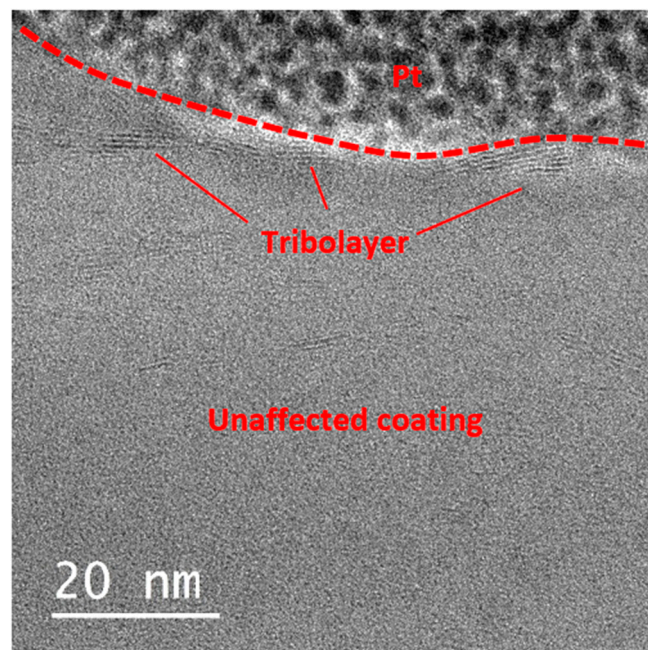


Figure 18. TEM image shows the formation a crystalline MoSe_2 tribolayer in the topmost surface of the wear track of MoSe_2 coating tested in dry sliding. This tribolayer is characterized by MoSe_2 basal planes aligned perfectly parallel to the sliding direction. The unaffected coating contains MoSe_2 nanocrystals embedded in an amorphous microstructure.

Figure 19 shows the ball wear scar was elliptical and exhibited a smooth surface with multiple scratches parallel to the sliding direction, indicating abrasive wear. EDS mapping (Figure 20) revealed a layer rich in molybdenum and selenium covering the wear scar. This

observation suggests the transfer and subsequent adherence of MoSe_2 material to the ball during sliding. Unlike the ball wear scar from the PAO4-lubricated sliding test shown in Figure 12, the transfer layer here was more evenly distributed and covered the majority of the wear scar. The presence of oxygen was also observed. Based on the EDS maps, where the oxygen map aligns closely with the molybdenum and selenium maps and not with the iron map, it is more likely that the oxygen is associated with the oxidation of the MoSe_2 coating rather than the steel ball.

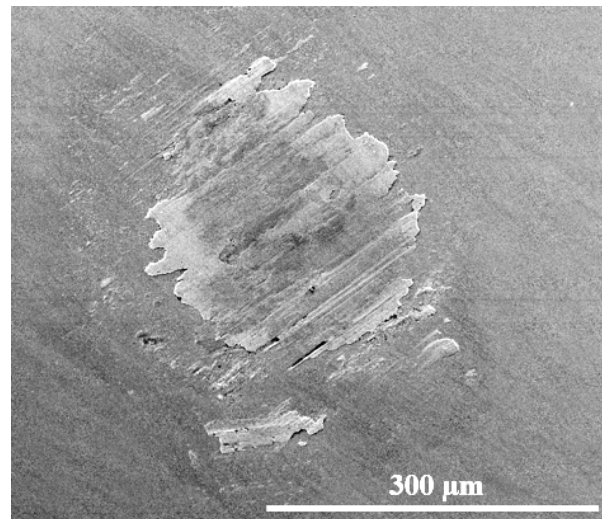


Figure 19. SEM image of the ball wear scar tested against MoSe_2 coating in dry sliding.

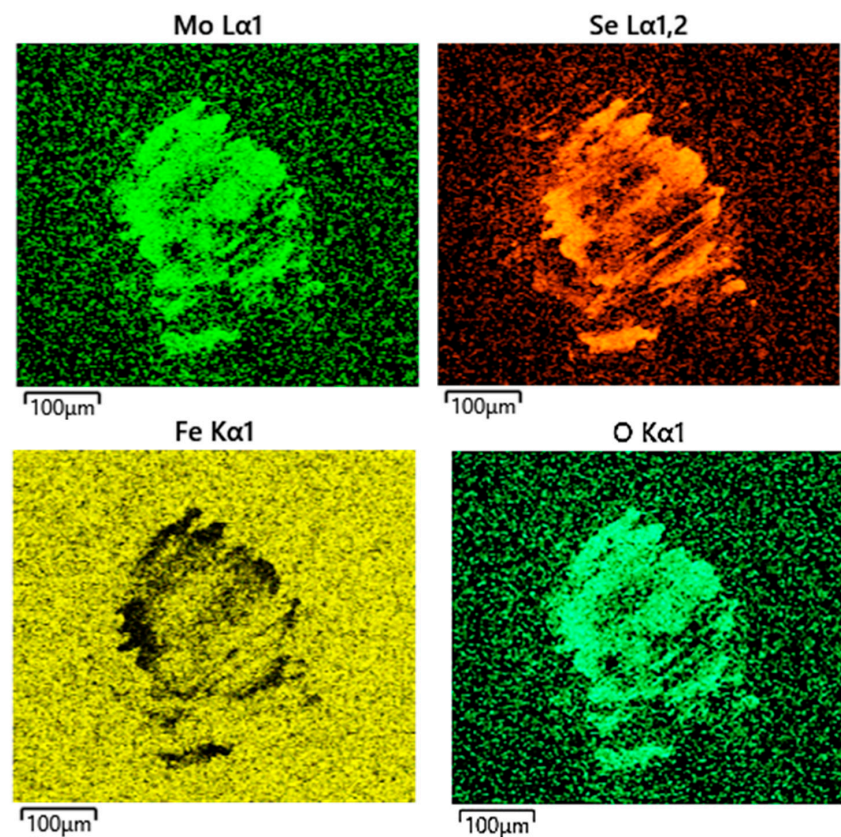


Figure 20. EDS mapping of the wear scar formed on the steel ball tested against MoSe_2 coating in dry sliding.

The Raman spectra acquired from different positions on the ball wear scar revealed the presence of a significant amount of adhered MoSe₂ material, see Figure 21. This result was different from the ball wear scar of the PAO4-lubricated sliding test, in which the presence of oil significantly impeded the transfer of the coating material. Peaks corresponding to MoO₃ at wavenumbers 815 cm⁻¹ and 940 cm⁻¹ were observed at all the analyzed positions on the wear scar [64,65]. However, the intensities of these peaks differed between the two tests, with the dry sliding test exhibiting more intense peaks compared to the PAO4-lubricated sliding test. This difference highlights the protective effect of PAO4 in preventing the oxidation of the coating during sliding.

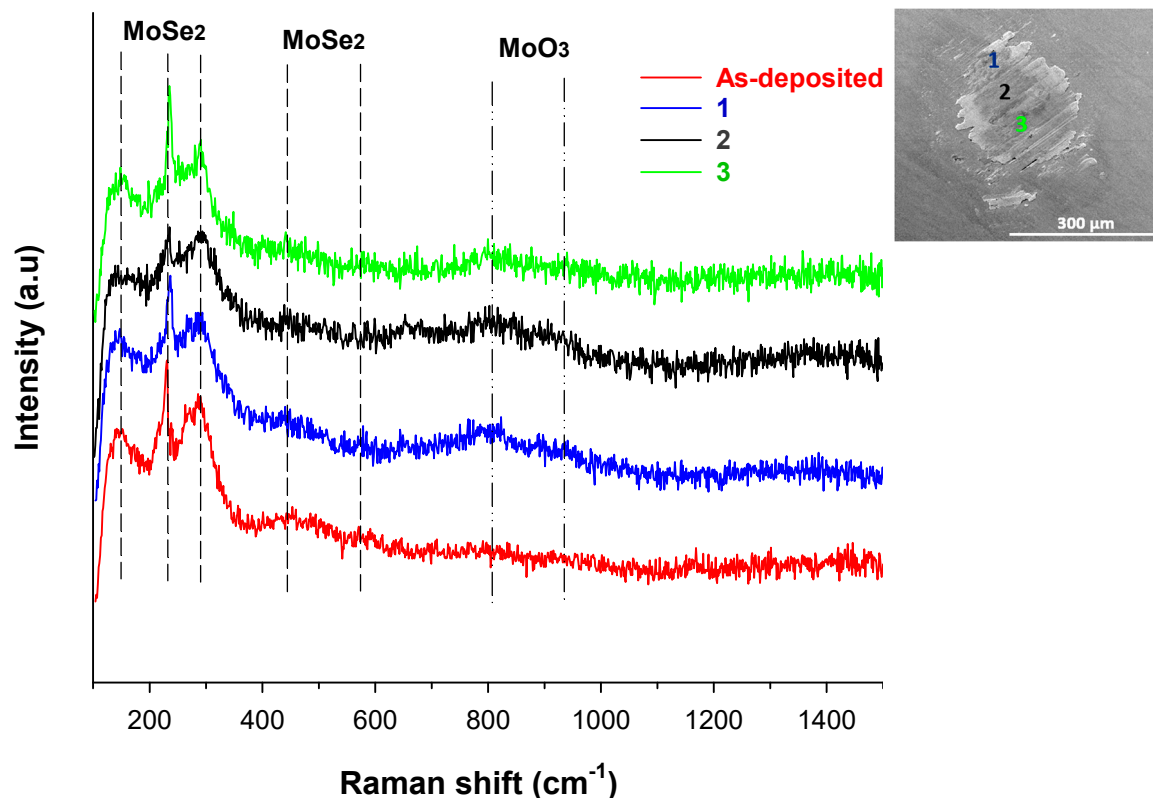


Figure 21. Raman spectra acquired from different positions on the ball wear scar tested against MoSe₂ coating in dry sliding.

4. Discussion

4.1. Friction Performance

The low-friction mechanism of TMD coatings is governed by two key factors: (a) the formation of a transfer layer on the coating's counterpart, resulting in a predominantly self-mated coating/coating contact with easy interfacial sliding, and (b) the formation of a well-ordered crystalline TMD tribolayer in the topmost surface of the coating wear track, with TMD basal planes aligned parallel to the sliding direction, facilitating interlamellar slip with minimal frictional resistance to sliding [1,15,50,54,57,66,69–71].

In PAO4-lubricated sliding, the formation of the above-mentioned tribologically beneficial transfer layer was significantly hindered. This was evidenced by EDS analysis and Raman spectroscopy, which showed that only a very small amount of MoSe₂ material transferred to the steel ball during sliding. This left the majority of the steel ball surface uncovered by a transfer layer required to impart minimal interfacial shear between the sliding surfaces. Such behaviour consequently resulted in a higher coefficient of friction, as well as a higher wear rate of the steel ball compared to dry sliding. It could also account

for the high instability observed in the friction curve of the PAO4-lubricated sliding test. Similarly, a well-ordered crystalline MoSe₂ tribolayer did not form in the topmost surface of the wear track. This was evidenced by TEM examination, which did not reveal the formation and alignment of MoSe₂ planes parallel to the sliding direction. Therefore, it can be inferred that PAO4 played a key role in the friction performance of MoSe₂ coating by significantly impeding the transfer of the coating to its sliding counterpart and preventing the formation and alignment of MoSe₂ planes parallel to the sliding direction. These two factors contributed to the higher coefficient of friction when compared to dry sliding.

In dry sliding, EDS analysis and Raman spectroscopy showed that the ball wear scar was fully covered by a relatively well-adherent and uniform layer transferred from the coating to its counterpart. The formation of this lubricious transfer layer prevented direct contact between the sliding surfaces and provided significant wear protection for the ball. TEM examination further revealed the formation of a crystalline MoSe₂ tribolayer in the topmost surface of the wear track, with MoSe₂ basal planes perfectly aligned parallel to the sliding direction. The formation of this low-friction tribolayer resulted from a sliding-induced crystallization of the initially amorphous MoSe₂ material. This amorphous-to-crystalline phase transformation has also been reported in other TMD coatings tested under dry sliding conditions [66,72–76]. Therefore, it can be inferred that the presence of easily sheared MoSe₂ basal planes in the topmost surface of the wear track and the formation of a uniform transfer layer covering the steel counterpart were all together responsible for the observed decrease in the coefficient of friction when compared to PAO4-lubricated sliding.

4.2. Wear Performance

During tribo-testing, TMD coatings can react with oxygen in the surrounding atmosphere, forming metal oxides such as MoO₃ and WO₃. The presence of these oxides at the sliding interface is very detrimental to the wear resistance of both the coatings and their counterparts. This is because these oxides act as abrasive particles, causing severe surface deterioration and ploughing [17,77–81].

In dry sliding, the characterization of the wear track of the coating by EDS revealed a significant presence of oxygen. This was further supported and confirmed by Raman spectroscopy, which revealed several peaks corresponding to MoO₃. The newly formed MoO₃ particles, being harder than MoSe₂, led to severe abrasion and ploughing of the coating, consequently increasing its wear rate. This was not the case with PAO4-lubricated sliding. EDS analysis did not detect any oxygen in the wear track. Similarly, Raman spectroscopy did not detect any peaks that could be identified as those of MoO₃ in the wear track. This protection from oxidation provided by PAO4 led to a reduction in the wear rate of the coating by 27% when compared to dry sliding. From these results, it can be deduced that PAO4 can play a crucial role in achieving low wear of the coating by functioning as a sealant and protecting it from oxidation.

With regard to the observed wear mechanisms, SEM and EDS analysis of the wear tracks and ball wear scars of both sliding conditions revealed the occurrence of abrasive wear, as evidenced by the presence of multiple scratches and wear grooves, and adhesive wear, as evidenced by the detachment and adherence of the coating material to the sliding counterparts. Delamination wear was another detected wear mechanism at the edges of the wear tracks.

5. Conclusions

MoSe₂ coatings deposited by magnetron sputtering were tribologically evaluated against steel balls under dry and PAO4-lubricated sliding conditions. The effect of oil

lubrication on their tribological performance was investigated. The following conclusions can be drawn from the experimental results.

- Evaluation of the coating during PAO4-lubricated sliding revealed a coefficient of friction that was double the value recorded in dry sliding.
- Characterization of the worn surfaces from the PAO4-lubricated sliding test showed that a crystalline MoSe₂ tribolayer, with MoSe₂ basal planes perfectly aligned parallel to the sliding direction, did not form in the coating wear track. Similarly, no beneficial transfer layer formed on the coating's sliding counterpart. The combined effects of these two factors contributed to the higher coefficient of friction when compared to dry sliding.
- Despite the higher coefficient of friction, the coating exhibited enhanced wear resistance in PAO4-lubricated sliding, with a wear rate 27% lower than that observed in dry sliding. This improvement in the wear resistance of the coating was due to the presence of PAO4 at the sliding interface, which acted as a sealant and protected the coating from the formation of abrasive metal oxides.
- The coating demonstrated better friction performance in dry sliding due to two major factors. The first was the formation of a uniform and compact transfer layer on the counterpart surface, which effectively prevented direct contact between sliding surfaces. The second was the presence of a crystalline MoSe₂ tribolayer in the topmost surface of the wear track with perfect alignment of the basal planes parallel to the sliding direction, which led to low interfacial shear strength at the sliding interface.
- The coating inevitably oxidized during dry sliding, resulting in the formation of MoO₃ particles. These newly formed particles with abrasive properties deteriorated the wear resistance of the coating and led to the higher coating wear rate when compared to PAO4-lubricated sliding.
- Under both sliding conditions, the coatings exhibited a combination of three wear mechanisms: abrasion, adhesion, and delamination.

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Abbreviations

The following abbreviations are used in this manuscript:

DLC	Diamond-like Carbon
EDS	Energy Dispersive X-ray Spectroscopy
PAO	Polyalphaolefin
RF	Radio Frequency
SEM	Scanning Electron Microscopy
TEM	Transition Electron Microscopy
TMD	Transition Metal Dichalcogenide

Appendix A

Configuration		Ball Coating Lubricant		STEEL MoSe ₂ PAO4
Oil lubricant	Temperature	T	°C	25
	Density	ρ_{15}	g/cm ³	0.817
	Kinematic Viscosity	η_{40}	mm ² /s	16.8
		η_{100}	mm ² /s	3.9
Materials	Elastic modulus	E1 (Ball)	Pa	2.07×10^{11}
		E2 (Disc)	Pa	2.1×10^{11}
	Poisson's ratios	V1 (Ball)		0.3
		V2 (Disc)		0.3
	Radii	R1x = R1y (Ball)	m	0.003
		R2x, R2y (Disc)	m	-
	ellipticity parameter	k (=R1y/R1x)		1.03
	Roughness	Ra (Ball)	nm	10
		Ra (Disc)	nm	13.53
	composite surface roughness	σ	nm	17
	Entrainment speed	U	m/s	0.2
		U1	m/s	0
		U2	m/s	0.4
	Load	W	N	5
Parameters	Pressure viscosity coefficient	α	m ² /N	1.83×10^{-8}
	Dynamic viscosity	η_0	Pa · s	2.41×10^{-2}
	Reduced Yong's modulus	E'	Pa	2.29×10^{11}
	Reduced Radii	R'x	m	0.003
		R'	m	0.0015
	Speed parameter	U		7×10^{-12}
	Materials parameter	G		4.18×10^3
	Load parameter	W		2.42×10^{-6}
Hertz theory	Contact radius	a	m	4.61×10^{-5}
			μm	46
	Maximum contact pressure	Pmax	Pa	1.12×10^9
			GPa	1.122
	Average contact pressure	Paverage	Pa	7.48×10^8
			GPa	0.75
Lubrication parameters	Minimum film thickness	<i>hmin</i>	m	2.18×10^{-8}
			nm	22
	Lambda ratio	λ		1.3

Figure A1. Calculation sheet for lambda ratio of MoSe₂ coating tested in PAO4-lubricated sliding.

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