

Príloha č. 1 k záverečnej monitorovacej správe projektu 09I03-0A-V04-00709

Technical report “Lubrication challenge for ultra-thin advanced 2D-TMDC in extreme conditions” for the period 07/2025 – 6/2026

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Work Package 1 (M1-M21): Processing. Synthesis of ultra-thin TMDCs layers with varied properties

(Task 1.1) Growth of ultra-thin PtSe₂, MoSe₂ and MoS₂ layers on the model Si/SiO₂ and Al₂O₃ substrates (scheduled as M1-M15).

While the majority of the planned experiments on synthesis development were completed during the first year of the project (as detailed in the Interim Progress Report and Deliverable D4 attachment 2), our efforts during the M12–M24 monitoring period focused on improving the structural and

morphological properties of the TMDC layers for tribological applications. To achieve this, we implemented several new approaches. In particular, we investigated MoSe₂ deposition in greater detail by varying the film thickness and utilizing different types of substrates. We optimized the deposition time to 15 minutes to yield optimal substrate coverage. Additionally, we utilized substrates with off-cut patterns to demonstrate that surface steps can act as nanoscale reservoirs for the 2D MoSe₂ layers, thereby prolonging tribological durability. To achieve this, we annealed the sapphire substrates at 1200 °C for 24 hours, which produced extended off-cut terraces with a width of 700 nm and a step height of 1.2 nm. The morphology of the substrate prior to deposition is shown in Figure 1.

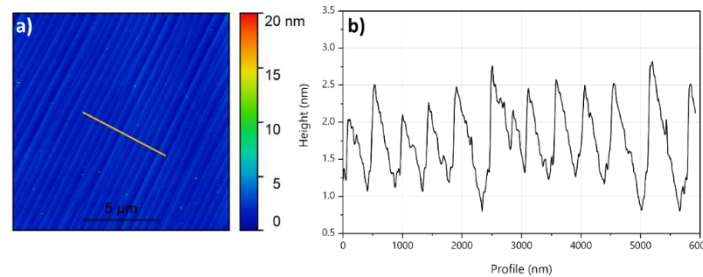


Figure 1. AFM image of the uncoated annealed sapphire substrate (a) and the corresponding line profile (b) taken along the selected line.

During this period, we also focused on fabricating TMDC-based multicomponent coatings. Taking into account the successful application of these technologies for hard coatings WN/TiSiN, where combining thin layers enhances overall toughness, wear resistance, and durability (see Attachment 7), we applied a similar structural architecture to 2D materials to improve their tribological performance. Accordingly, two types of hybrids were analyzed:

- a) Heterostructures of MoSe₂ and graphene oxide (GO). Ultrathin MoSe₂ layers, consisting of a highly crystalline base film with flower-like surface features, were deposited on sapphire substrates. A GO overlayer was subsequently applied to form the heterostructure. The weak interactions between the GO and MoSe₂ resulted in a significant improvement in the sample tribological properties (full details regarding the MoSe₂ and GO deposition methods are described in the prepared manuscript, Attachment 12).
- b) Transfer of MoSe₂ onto CVD diamond surfaces. Given the improvement in MoSe₂ friction behavior induced by the GO overlayer, the inverse approach was investigated by transferring a MoSe₂ layer onto a hard carbonaceous structure. For this purpose, pre-deposited MoSe₂ was transferred onto the diamond surface. This transfer was enabled by increasing the MoSe₂ deposition time to 1 hour, which allowed the growth of thicker MoSe₂ films consisting of discrete flakes with favorable delamination behavior. The preparation conditions for the diamond substrates are detailed in Attachment 11. In particular, we utilized a polymer-assisted seeding method for diamond growth with an SiO₂ interlayer. A general schematic of the mechanical contact transfer process is presented in Figure 2. This approach allowed us to investigate the interfacial interactions and transferability of MoSe₂ flakes onto a high-roughness diamond layer ($R_{\text{RMS}} \approx 15$ nm), using rotational shear forces to facilitate delamination and deposition of the MoSe₂.

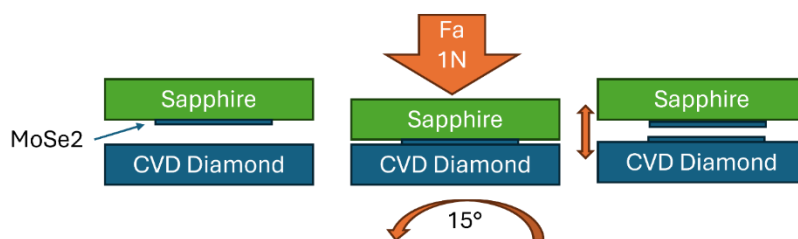


Figure 2. Transfer process of MoSe₂ onto the diamond surface.

The results of the work performed under this task contribute to the fulfillment of Milestone M1 and Deliverable D4. In particular, we have substantially expanded the parameter set for the synthesis and structural control of low-friction 2D materials.

(Task 1.2) Synthesis of ultra-thin coatings on the technologically relevant steel substrates (scheduled as M16-M21).

The primary objective of this task was to achieve sufficient adhesion between the synthesized layers and the substrates. To achieve these goals, it was decided to utilize pre-coated ALD oxide sublayers (HfO_2 , Al_2O_3 , and TiO_2). The details of the growth process and properties of the coating are presented in Attachment 3.

Work Package 2 (M1-M24): Properties. Structural, chemical and physical properties of ultra-thin TMDCs layers

(Task 2.1) Characterization of ultra-thin layers following the deposition (scheduled as M1-M21).

Comprehensive post-deposition characterization of the synthesized ultrathin layers and hybrid systems was conducted using a combination of AFM, FTIR, XPS, SEM, EDX, and Raman spectroscopy. The specific characterization suite was selected based on the sample configuration and substrate type. In the following section, we detail the structural and chemical parameters of layers synthesized during the second year (M12-M24) of project implementation.

1) MoSe_2 and MoSe_2/GO heterostructures.

To avoid redundancy across different parts of this report, the comprehensive characterization of MoSe_2 layers on sapphire substrates and MoSe_2/GO heterostructures is detailed in Attachment 12. Specifically, topographic and structural analyses are provided in Chapter 3.1; compositional and morphological analyses of MoSe_2 and MoSe_2/GO are in Section SI1; Raman spectra are detailed in Section SI2; and XPS analysis of the MoSe_2 and MoSe_2/GO samples is presented in Section SI3.

2) CVD Diamond with transferred MoSe_2 .

These hybrid systems were characterized using optical profilometry and Raman spectroscopy. The resulting optical image presented in Figure 3c confirms the successful mechanical transfer of both small and relatively large MoSe_2 agglomerates onto the CVD diamond surface. An optical image of the MoSe_2 sample on the sapphire substrate, with thicker discrete flakes prior to transfer, is shown in Figure 3a. Optical images of the CVD-grown diamond surface before MoSe_2 transfer are shown in Figure 3b.

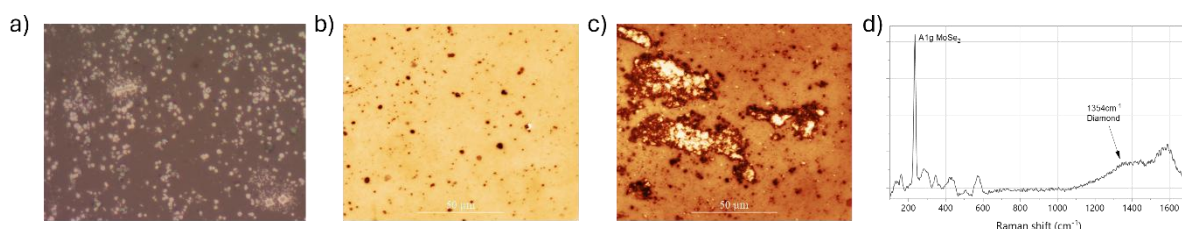


Figure 3. (a) Optical image of MoSe_2 samples featuring a high density of discrete, thick MoSe_2 flakes prior to delamination. The diamond surface before (a) and after (c) MoSe_2 transfer. (d) Raman spectra of transferred flakes on the diamond surface.

3) ALD buffer layers on Si substrates.

The as-deposited ALD layers (Al_2O_3 , HfO_2 , and TiO_2) with a thickness of 30 nm were first investigated on Si substrates using AFM (Figure 4a–c) and Raman spectroscopy (Figure 4d). AFM imaging demonstrated relatively low surface roughness (R_{RMS}) for the layers: 0.3 nm for Al_2O_3 , 2.3 nm for HfO_2 ,

and 1.8 nm for TiO₂, compared to 0.1 nm for the bare Si substrate. The morphological features observed in the AFM images suggest an amorphous structure for the Al₂O₃ and HfO₂ films, whereas the TiO₂ layer exhibited a distinct polycrystalline morphology. These observations were fully corroborated by Raman analysis, which showed characteristic TiO₂ peaks for the TiO₂ ALD samples, while no Raman active modes were detected for the amorphous Al₂O₃ and HfO₂ layers.

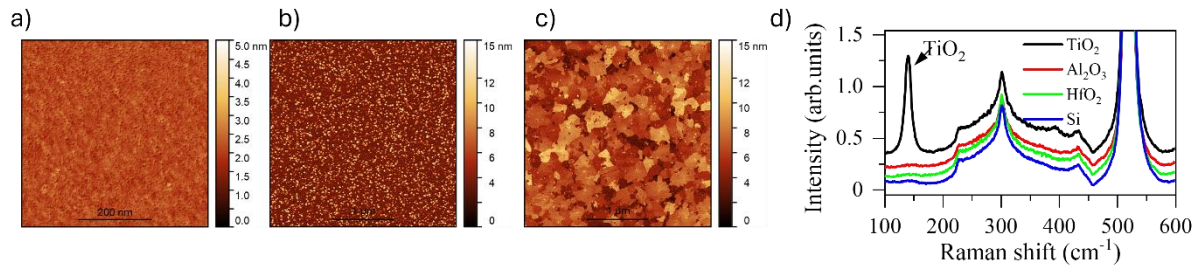


Figure 4. AFM topographical images of the 30 nm ALD buffer layers: (a) Al₂O₃, (b) HfO₂, and (c) TiO₂ on Si substrates. (d) Corresponding Raman spectra of the as-deposited ALD layers.

Topographical analysis of the samples after synthesis of the PtSe₂ layer (Figure 5a–d) revealed comparable surface roughness across all samples, with Ra values ranging from 1.5 to 1.9 nm. Furthermore, structural characterization via Raman spectroscopy (Figure 6) showed intense peaks corresponding to the characteristic Eg and A_{1g} modes of PtSe₂, confirming the formation of a high-quality polycrystalline phase on all evaluated ALD buffer layers.

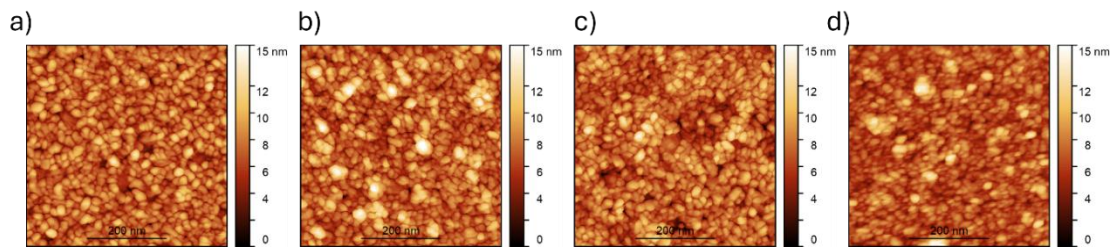


Figure 5. AFM topographical images of PtSe₂ layers synthesized on different ALD buffer layers: (a) Al₂O₃, (b) HfO₂, (c) TiO₂, and (d) PtSe₂ on a reference Si substrate.

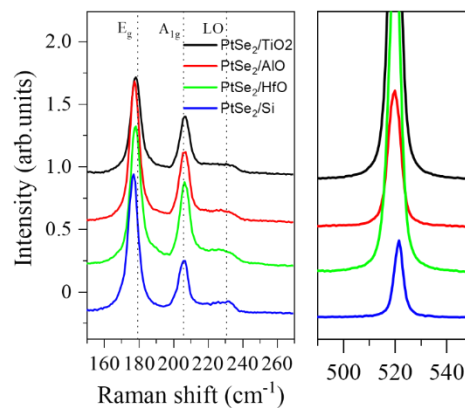


Figure 6. Raman spectra of the PtSe₂ layers synthesized on Al₂O₃, HfO₂, and TiO₂ ALD buffer layers compared to a reference PtSe₂ film grown on Si.

(Task 2.2) Characterization after long-term storage (scheduled as M13-M24).

The environmental stability of the synthesized layers was monitored over a 12-month period in ambient air and vacuum conditions (-0.08 MPa) using AFM (Figures 7 and 8) and Raman spectroscopy (Figure 9). To evaluate the changes, we focused on samples with different surface topographies. In the

case of PtSe₂, these were films with in-plane- and out-of-plane-oriented flakes and epitaxial layers. For MoSe₂, continuous thin layers as well as isolated pyramidal and flat flakes were investigated.

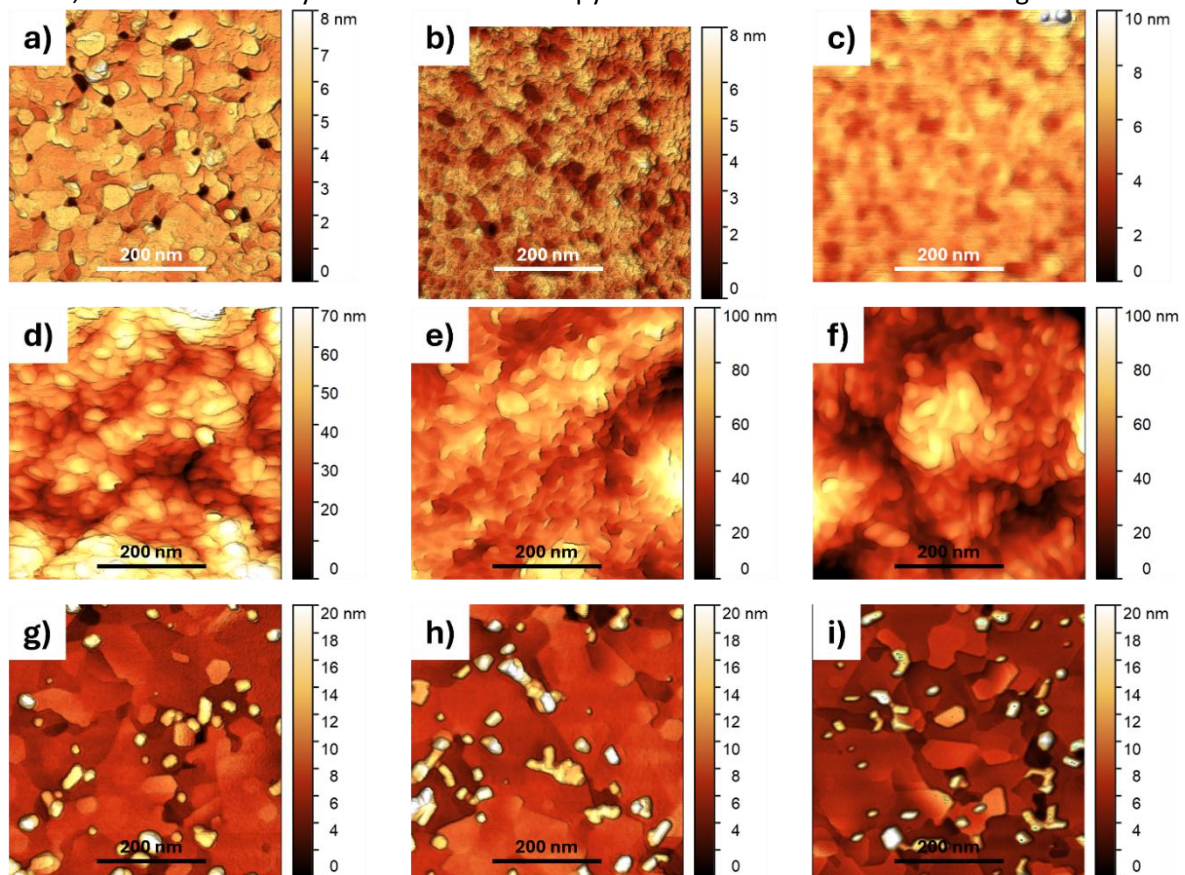


Figure 7. AFM images of as-deposited PtSe₂ layers and after 12-month storage in ambient air and vacuum. (a- c) - PtSe₂ layers with in-plane oriented flakes grown from a 1 nm Pt precursor. (d-f) - PtSe₂ layers with out-of-plane oriented flakes. (g-i) – epitaxial PtSe₂ films. (a, d, g) - as-deposited samples, (b, e, h) - samples after 12 months in ambient air, and (c, f, i) - samples maintained under vacuum for at least 12 months.

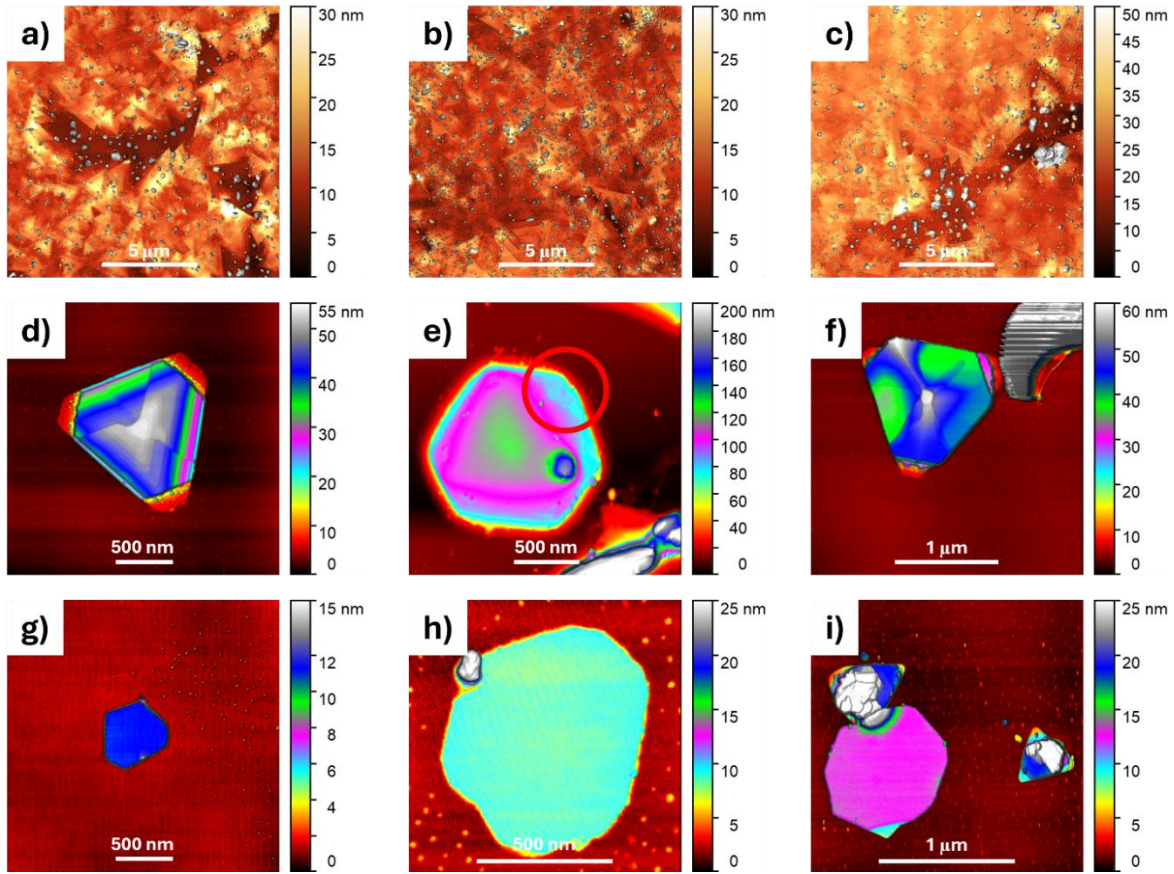


Figure 8. AFM images of as-deposited MoSe₂ layers and after 12-month storage in ambient air and vacuum. (a- c) - MoSe₂ continuous thin films. (d-f) - isolated MoSe₂ pyramidal flakes, and (g-i) - isolated MoSe₂ flat flakes. (a, d, g) - as-deposited samples, (b, e, h) - corresponding samples after 12 months in ambient air, and (c, f, i) - samples maintained under vacuum conditions for at least 12 months.

AFM measurements revealed notable topographical modifications in the PtSe₂ layers after one year of storage in ambient air, where the surface roughness increased from 0.71 nm to 0.8 nm for in-plane-oriented flakes (Figure 7a–b) and from 11.9 nm to 12.4 nm for out-of-plane-oriented samples (Figure 7d–e). At the same time, samples stored under vacuum for 12 months (Figure 7c, f) also demonstrated a slight increase in roughness to 0.88 nm and 14.6 nm, respectively. For the epitaxial films, the roughness increased from 2.5 nm to 3.11 nm in ambient air and to 3.0 nm in vacuum storage. Overall, the global surface roughness of the samples did not change dramatically, which is partly attributed to the high initial roughness created during deposition. However, the edges of the flakes became more blunted, especially for samples stored in ambient air, thereby highlighting the exceptional stability of the material in the absence of air and moisture.

In contrast to PtSe₂, continuous MoSe₂ layers exhibited high environmental stability, which can be attributed to their continuous morphology and a lower density of exposed unsaturated edges. It should be noted that the environmental degradation of ultrathin 2D materials, particularly at the monolayer and sub-nanometer scale, is a widely documented phenomenon [10.1021/acsnano.5b07677]. In order to confirm this, a detailed analysis of thin layers (Figure 8a-c) as well as isolated flakes of varying morphology, including tall pyramidal flakes (50–100 nm in height, Figure 8d-f) and small flat flakes (up to 20 nm in height, Figure 8g-i) was performed. This investigation revealed that morphology plays a critical role, as tall pyramidal flakes exhibited pronounced degradation localized along their edges compared to low-height, flat flakes. A comparison of flakes of different natures demonstrated that this degradation effect is significantly more pronounced in ambient air (Figure 8e) than in a vacuum (Figure

8f). This behavior can be attributed to the desorption of Se and subsequent passivation of Se vacancies by oxygen at defective edge sites, as further confirmed by Raman characterization (Figure 9).

The Raman spectra of the MoSe₂ samples stored in ambient air demonstrated a slight redshift of the A_{1g} mode from 242.4 cm⁻¹ to 241.7 cm⁻¹ (Figure 9b), which can indicate a minor loss of Se during the 12-month storage period [10.48550/arXiv.1904.09789]. In contrast, despite the observed morphological modifications, the PtSe₂ samples exhibited almost unchanged Raman spectra (Figure 9a), showing only a minor decrease in the intensity of the peak at 230.5 cm⁻¹, which confirms their excellent long-term chemical stability in air [10.1002/adma.201604230].

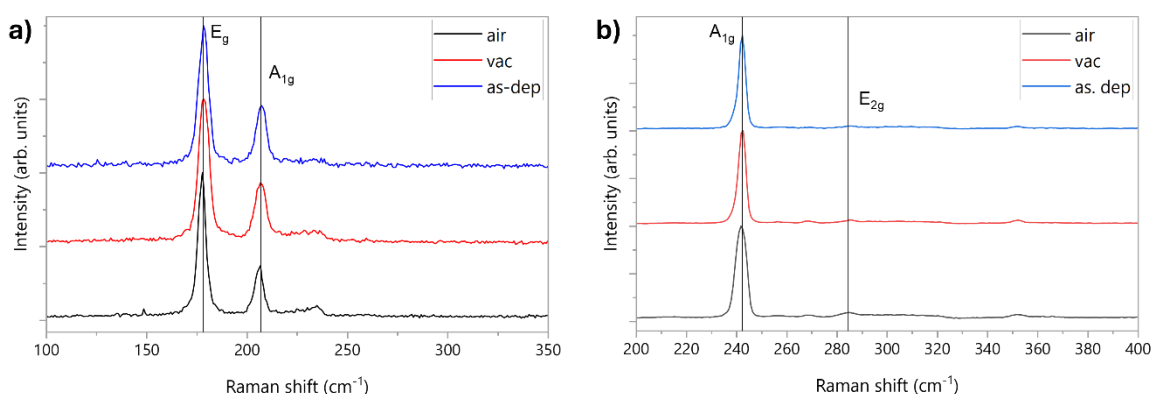


Figure 9. Raman spectra of as-deposited, 12 month vacuum, and ambient air stored (a) PtSe₂ and (b) MoSe₂ samples.

Work Package 3. Performance. Advanced tribological investigation and characterization

(Task 3.2.1) Macroscale friction analysis in ambient air and in vacuum

1) COF of MoSe₂ and MoSe₂/GO heterostructures.

Samples deposited on sapphire substrates were tested against a Si3M4 ball. The MoSe₂ layers exhibited a Coefficient of Friction (COF) of 0.15 in ambient air, which decreased to 0.10 in vacuum. The MoSe₂/GO heterostructures maintained a similar COF range in ambient air but showed a significant increase in wear life and a reduction of the COF to 0.03 in vacuum. These data clearly indicate that the chemical composition of the heterostructure directly influences the friction mechanism. A more detailed analysis of these results is provided in Attachment 12. Furthermore, we observed that the substrate morphology significantly impacts tribological performance. Testing on sapphire substrates with a patterned, off-cut surface morphology demonstrated a substantial enhancement in tribological durability,

2) Tribological performance of MoSe₂/diamond hybrid systems.

Previously, spin-coated GO layers demonstrated relatively low adhesion but tended to accumulate within the contact zone, providing a lubricant replenishment effect during sliding. We hypothesized that a similar 'third-body' replenishment mechanism would occur in the MoSe₂/diamond system. However, macroscale tribological evaluations revealed that the COF of the MoSe₂/diamond system did not significantly differ from that of the bare diamond surface (Figure 10). In ambient air, the COF for both samples was approximately 0.10. Under vacuum conditions, the COF increased significantly to 0.50 with a large ± 0.1 deviations.

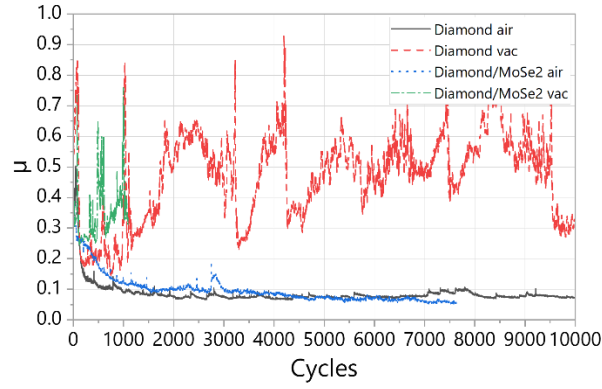


Figure 10. COF as a function of sliding cycles for CVD diamond and the MoSe₂/diamond hybrid system in both ambient air and vacuum conditions.

3) Tribological evaluation of PtSe₂ with ALD sublayer.

To perform the tribological testing of PtSe₂ on steel substrates, ALD interlayers were utilized to enhance film adhesion. In this section, we firstly present the results regarding the influence of these ALD interlayers on the COF of PtSe₂ using reference Si substrates. This allows for assessment of the intrinsic tribological properties of the PtSe₂/ALD system on a well-defined, flat surface before evaluating the more complex steel-based samples.

In ambient air, the HfO₂/ PtSe₂ and TiO₂/ PtSe₂ systems demonstrated the greatest durability, maintaining stable COFs of approximately 0.2 and 0.15, respectively, over 800 cycles (Figure 11). Conversely, under vacuum conditions, the Al₂O₃/ PtSe₂ system exhibited superior performance with a COF of 0.08 for nearly 800 cycles, while the HfO₂/ PtSe₂ and TiO₂/ PtSe₂ systems showed significantly higher or more unstable friction levels.

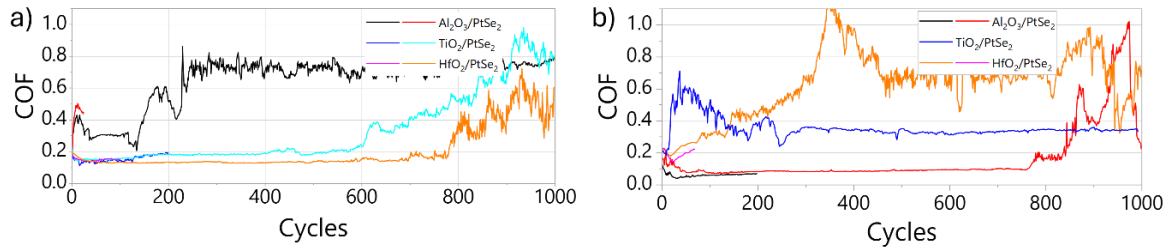


Figure 11. COF as a function of sliding cycles for PtSe₂ synthesized on Al₂O₃, HfO₂, and TiO₂ ALD buffer layers under (a) ambient air and (b) vacuum environments.

The PtSe₂ samples synthesized on steel substrates with Al₂O₃ and HfO₂ interlayers demonstrated promising performance during room-temperature evaluations. Testing with an Al₂O₃ ball revealed nearly identical COFs for both interlayers, with small deviations of around 0.3 (Figure 12). Under vacuum conditions, the Al₂O₃-buffered system reached approximately 3000 cycles before failure. However, in ambient air, tribological durability was significantly lower, with no more than 300 cycles. Notably, the use of an HfO₂ interlayer in vacuum provided a substantial increase in wear life, extending beyond 10000 cycles, whereas performance in ambient air remained limited to approximately 300 cycles.

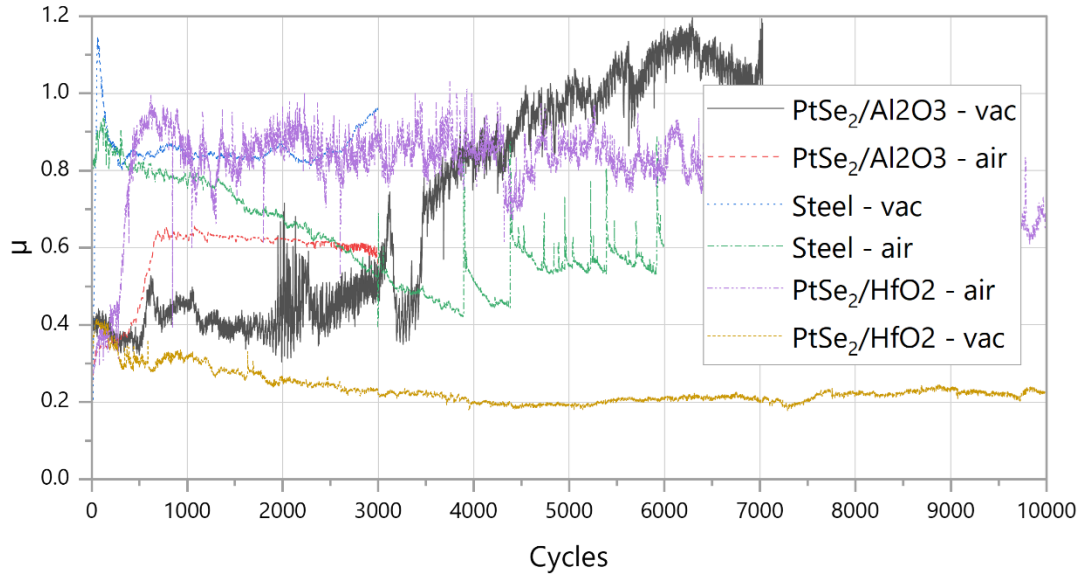


Figure 12. Comparison of the COF on the number of sliding cycles for PtSe₂ coatings on 16MnCr5 steel substrates using Al₂O₃ and HfO₂ interlayers, evaluated in ambient air and vacuum. Bare steel references are included for comparison.

Based on the structural and chemical analyses of the wear track and counterbody ball (Task 3.2.2 and Attachments 16, 17), it was determined that the chemical properties of the layers are independent of ambient conditions. Similar morphological and structural changes were observed in both vacuum and ambient air. However, in contrast to samples on silicon substrates, samples on steel substrates with an HfO₂ sublayer exhibited an abrasive wear mechanism. To further evaluate the durability of the coatings, additional tests were performed using various applied loads. Specifically, the applied load was reduced to 0.13, 0.05, and 0.025 N in ambient air (Figure 13a). Because initial vacuum data demonstrated high stability over 10000 cycles at an applied force of 0.25 N, further vacuum measurements were conducted at applied forces of 0.13, 0.25, 0.5, 1.0 and 3.0 N (Figure 13b). Concurrently, at the lower loads of 0.13, 0.05 and 0.025 N, the sliding velocity was reduced to 0.2 cm/s, as this study targets microscale devices.

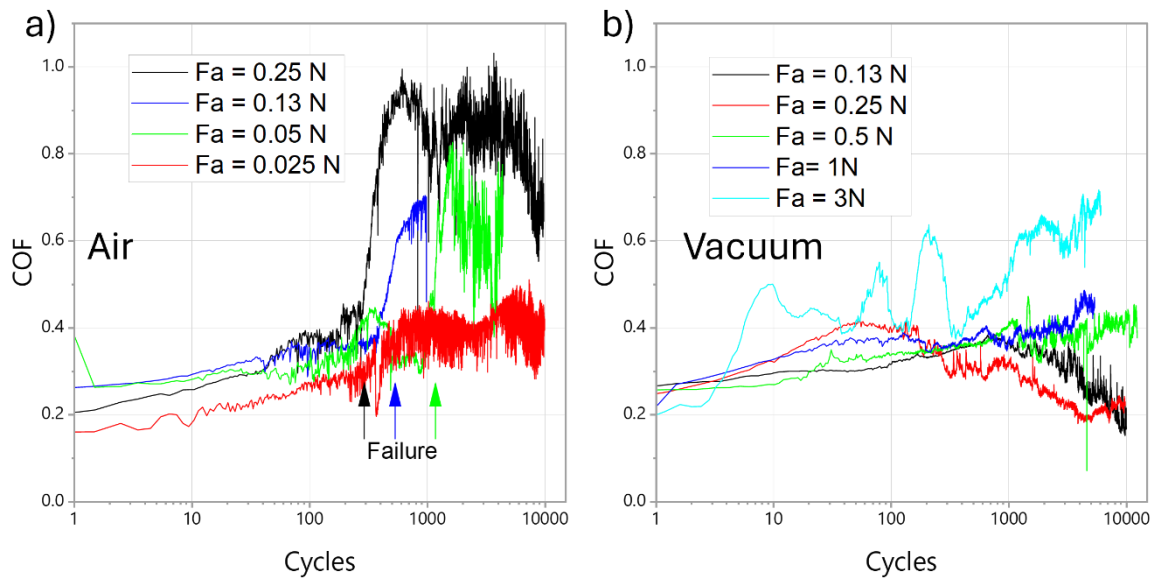


Figure 13. COF of PtSe₂ coatings as a function of sliding cycle number under various applied loads (a) in ambient air (0.025–0.25 N) and (b) in vacuum (0.13–3.0 N).

Ambient air measurements demonstrated that decreasing the load to 0.13 N increased the coating lifetime to 500 cycles. A further decrease to 0.05 N extended the lifetime to nearly 1050 cycles, and at 0.025 N, no complete failure was observed even after 10000 cycles. Under vacuum conditions, applied loads of 0.13 N, 0.25 N and 0.5 N maintained a stable COF over 10000 cycles. However, increasing the load to 3.0 N resulted in coating failure at 100 cycles. At the same time, at 100 cycles, the friction coefficient for this system reached 0.33 in ambient air and 0.31 in vacuum, corresponding to a COF_{air}/COF_{vac} ratio of 1.06. Under an applied load of $F_a = 0.25$ N, the coefficient of friction stably leveled off at approximately 0.3 in both ambient air and vacuum prior to the initiation of coating degradation, which occurred after 200 cycles in vacuum and within the first 50 cycles in air.

This behavior confirms an adhesive friction mechanism. The differences in wear life can be attributed to the varied interactions between wear debris and the testing environment. Humidity in the ambient air promotes the agglomeration of wear debris, facilitating its removal from the wear track. Conversely, under vacuum conditions and low loads, small debris particles remain trapped within the wear track and require more time to be ejected. A schematic images of the wear mechanisms are presented in Figure 14.

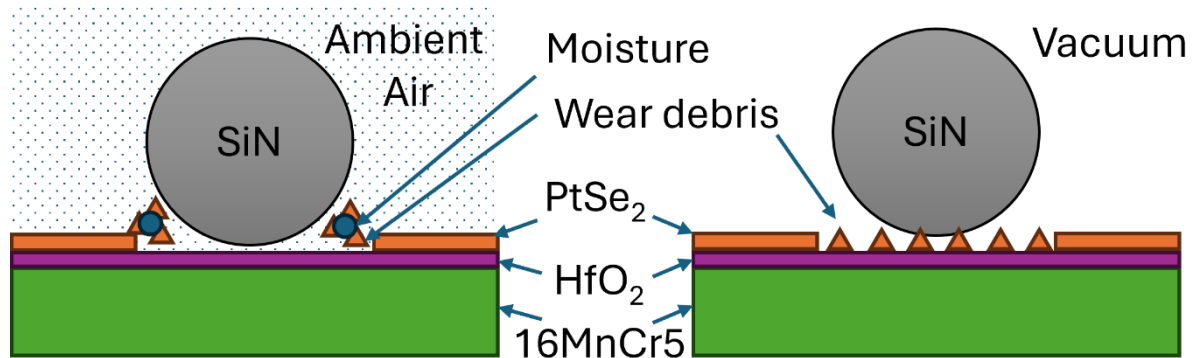


Figure 14. Schematic images of the wear mechanisms Of PtSe₂/ HfO₂/16MnCr5 structures in ambient air conditions and in vacuum.

(Task 3.2.2) Macroscale tribological analysis in ambient air and in vacuum at elevated temperatures (scheduled as M13-M24).

In this stage, the synthesized TMDC layers and TMDC-based heterostructures were systematically investigated at temperatures ranging from room temperature (RT) up to 400 °C. Testing was conducted on both sapphire and technologically relevant 16MnCr5 steel substrates to evaluate the thermomechanical stability of the solid lubricant films and to identify temperature-dependent tribochemical mechanisms at the sliding interface. Our investigation focused on two primary systems: (1) vertically aligned PtSe₂ flakes, which exhibited minimal variation in the COF between ambient air and vacuum, and (2) MoSe₂/GO heterostructures, which demonstrated high potential for friction control through interfacial tribochemical effects driven by the interaction between the two phases. For all vacuum experiments, the pressure in the chamber was $1.0\text{--}1.3 \times 10^{-5}$ mBar.

1) MoSe₂/GO heterostructures.

MoSe₂/GO heterostructures synthesized on sapphire substrates exhibited distinct tribological responses in vacuum versus ambient air when tested against Si₃N₄ balls. In the vacuum environment, the system showed stable, temperature-dependent friction behavior. The minimum COF decreased from 0.04 at room temperature to 0.02 at 400 °C (Figure 15a). In contrast, high-temperature testing in ambient air resulted in rapid film degradation within fewer than 100 cycles. Here, it should be noted

that the friction between the Sapphire substrate and the Si₃N₄ ball is also in the range of 0.03 (Figure 15b), but it is characterized by large splashes and rapidly increases after 3000 cycles.

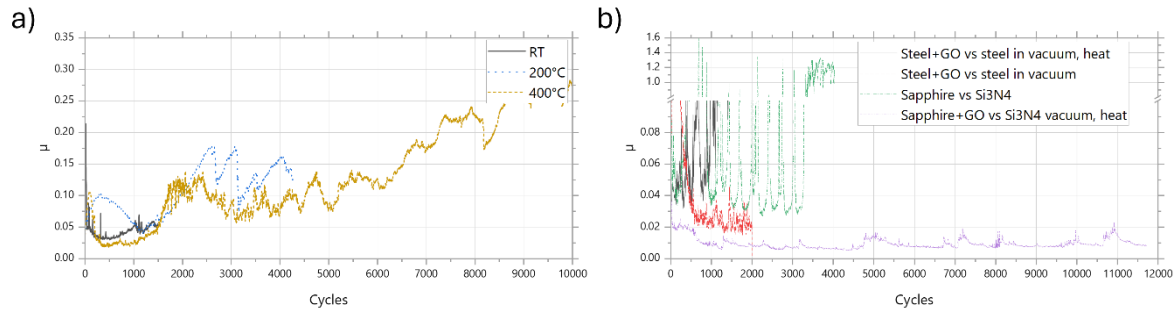


Figure 15. Influence of elevated temperatures on the vacuum tribological performance of TMDC/GO and GO-only systems. (a) COF as a function of sliding cycles for MoSe₂/GO on sapphire at various temperatures. (b) COF as a function of sliding cycles for GO samples on sapphire and 16MnCr5 steel substrates, tested against Si₃N₄ and 100Cr6 steel balls, respectively.

2) GO at elevated temperatures

The application of a GO layer on sapphire substrates in vacuum, compared with a Si₃N₄ counterpart at 400 °C, resulted in an ultra-low COF of 0.006 and extended tribological durability to 12000 cycles (Figure 15b). However, for GO-coated steel substrates under vacuum, Si₃N₄ balls typically led to rapid coating failure, whereas 100Cr6 balls proved more promising. When utilizing the steel ball, we observed that the COF for heated samples (400 °C) increased from 0.04 to 0.10 over the first 1000 cycles. In contrast, at room temperature, the COF remained stable at approximately 0.02 for over 2000 cycles (Figure 15b).

3) PtSe₂ layers at elevated temperatures.

Testing of PtSe₂ layers at 400 °C demonstrated rapid degradation in both vacuum (Figure 16a) and ambient air (Figure 16b). Our research focused specifically on samples synthesized on Si substrates with an Al₂O₃ interlayer. As shown in Figure 16a, despite using various sliding counterparts, the COF increased rapidly to values characteristic of the uncoated Si substrate under the same conditions. A similar behavior was observed in ambient air, where the PtSe₂/ Al₂O₃/Si system tested with a Si₃N₄ ball failed the film within the first 100 cycles. Based on these data, only preliminary testing was performed on 16MnCr5 steel substrates with Al₂O₃ ALD interlayers at elevated temperatures (Figure 16c). These tests also confirmed the rapid wear and insufficient thermal stability of the deposited coating

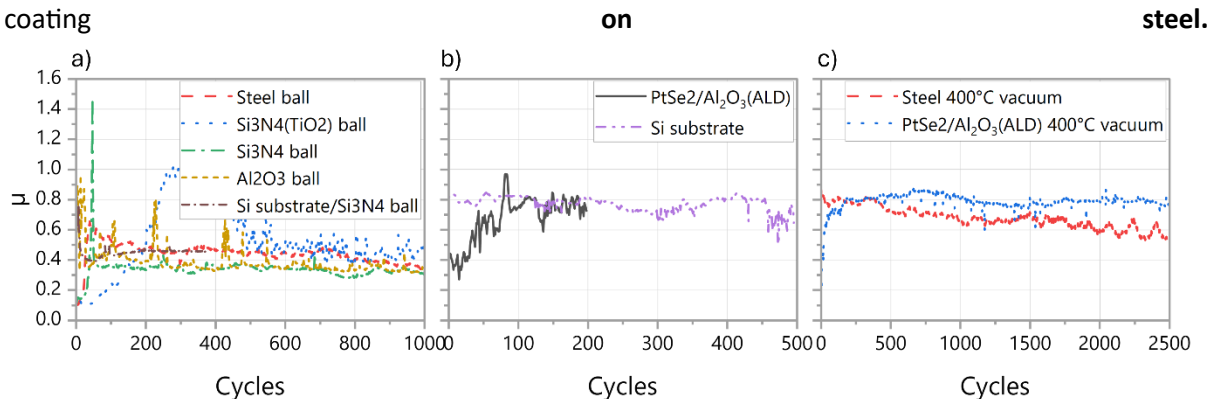


Figure 16. High-temperature (400 °C) tribological failure of PtSe₂-based systems. (a) Evolution of the COF for PtSe₂/ Al₂O₃/Si system in a vacuum against various counter-body materials. (b) Comparison of PtSe₂/ Al₂O₃/Si system and bare Si substrate in ambient air at 400°C. (c) Preliminary evaluation of the PtSe₂/ Al₂O₃ system on 16MnCr5 steel compared to a bare steel reference under vacuum conditions at 400 °C.

Task 3.2.3: Wear track and counterbody characterization (scheduled: M7–M24)

To elucidate the active friction and wear mechanisms at the sliding interface, the wear tracks and counterbodies (balls) were characterized using a comprehensive set of analytical techniques, including optical profilometry, Raman spectroscopy, AFM, SEM/EDS, and XPS. This systematic investigation, which began during the first year of the project and continued throughout the second year, aimed to correlate the observed tribological performance with interfacial chemical and structural changes. The detailed results obtained during the second year of project implementation are listed below:

1) MoSe₂ and MoSe₂/GO heterostructures. Patterned substrate effects.

A comprehensive report on the influence of patterned surface morphology on the COF of MoSe₂ and MoSe₂/GO heterostructures is provided in Attachment 12. Our findings demonstrate that in ambient air, the GO layer acts as a binder for oxidized MoSe₂ species, maintaining the integrity of the lubricating film. In vacuum conditions, the primary factor driving low friction is the separation of layers at the interface between the sliding counterparts.

2) Wear tracks of MoSe₂/diamond hybrids.

Optical profilometry (Figure 17) and Raman spectroscopy (Figure 18) of the sliding counterbodies and the resulting wear tracks confirmed that the transferred MoSe₂ layers delaminated rapidly from the CVD diamond surface. This premature failure was driven by low interfacial adhesion between the carbon (diamond) substrate and the MoSe₂ phases, which prevented the formation of a stable tribolayer (detailed in Attachment 12). More detailed Raman analysis and optical topography of the sliding Si₃N₄ ball showed that, in ambient air, abrasive wear occurs on the ball, without any inclusions from the wear track. In contrast, under vacuum conditions, the ball showed a significant amount of transferred carbonaceous species, suggesting degradation of the carbon-based layer and the formation of a transfer film on the counterbody. These results are in good agreement with observations for MoSe₂/GO heterostructures. In a vacuum, the Si₃N₄ ball is prone to the formation of a carbonaceous tribofilm. However, in ambient air, the carbon species tend to form wear debris that does not adhere to the ball. This is likely caused by surface passivation in air, which saturates dangling bonds and prevents the adhesion of a stable transfer film.

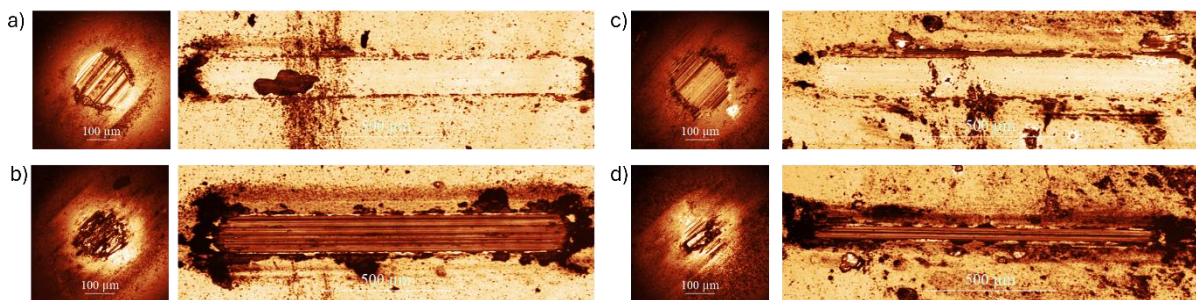


Figure 17. Optical images of the sliding balls and wear tracks for the bare diamond coating tested in (a) ambient air and (b) vacuum, and for the MoSe₂/diamond hybrid systems in (c) ambient air and (d) vacuum.

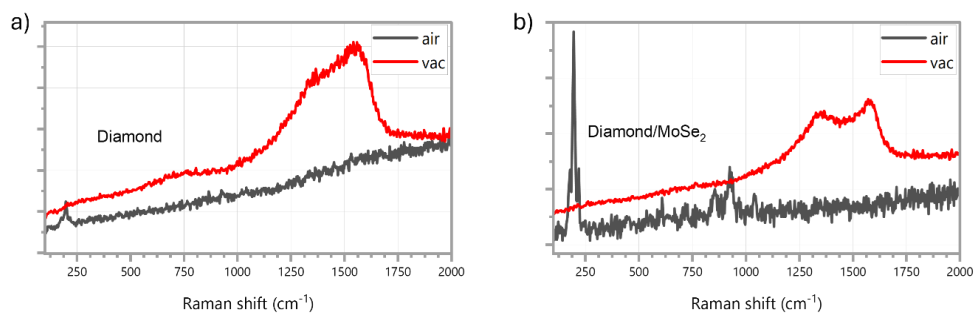


Figure 18. Raman spectra of the contact area on the sliding balls after testing in ambient air and vacuum for the bare diamond coating (a) and the MoSe₂/diamond hybrid systems (b).

3) Wear mechanism of MoSe₂/GO at elevated temperatures:

Topographical analysis of the wear tracks and sliding balls immediately after testing (Figure 19a–c) demonstrates that while the MoSe₂/GO film in the wear track is largely consumed, a prominent transfer layer is formed on the Si₃N₄ counterbody. Notably, the volume and coverage of this transfer layer tend to increase with rising temperature. Raman spectra (Figure 19d) confirm the formation of a composite tribofilm consisting of both MoSe₂ and carbonaceous species on the ball surface across all temperature ranges. However, a clear trend was observed. As the sliding temperature increased, the relative intensity of the carbon species decreased, while the MoSe₂ signal became more dominant. This behavior can indicate temperature-dependent control of the layer-separation effect.

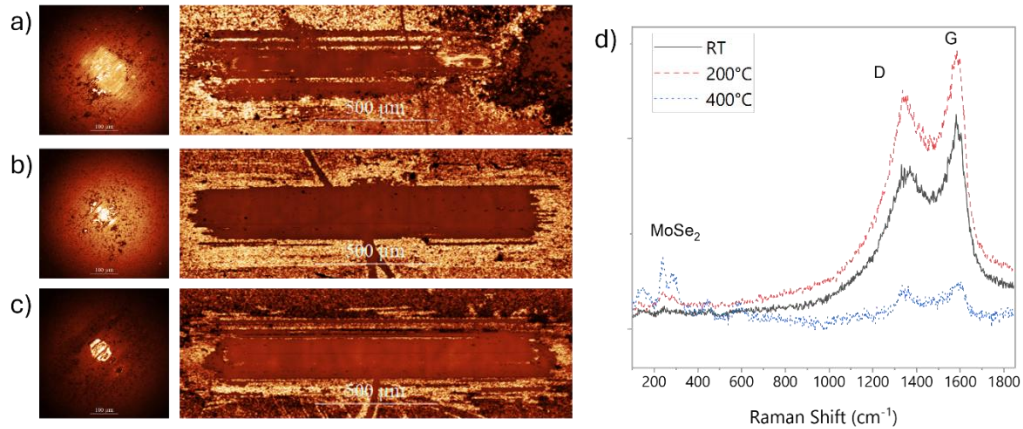


Figure 19. Optical images of the sliding balls and wear tracks for MoSe₂/GO heterostructures tested in vacuum at (a) room temperature, (b) 200 °C, and (c) 400 °C. (d) Raman spectra of the corresponding contact areas on the sliding Si₃N₄ balls after testing at these temperatures.

It is hypothesized that the thermally induced friction reduction is due to the thermally activated chemical reduction of GO. This process removes oxygen-containing functional groups and decreases total carbon content in the contact zone, thereby reducing the interfacial shear strength between the sliding counterparts and facilitating low friction.

4) High-temperature testing of GO.

High-temperature evaluations of GO were performed on sapphire substrates (against Si₃N₄ balls) and 16MnCr5 steel (against 100Cr6 steel balls). Analysis of the wear tracks on sapphire via optical profilometry and AFM (Figure 20 b,c) revealed that the coating was almost entirely depleted from the contact zone. Notably, unlike MoSe₂ or GO samples tested at room temperature, which typically exhibit debris agglomeration at the track edges, the high-temperature samples showed no accumulation of wear debris at the edges of the stroke. Raman spectroscopy (Figure 20d,e) of the wear track identified isolated 'carbon patches' which closely resembled the as-deposited coating. Consequently, while the ultra-low COF may be partially driven by the thermal desorption of surface adsorbates, the role of a sub-nanometer tribofilm or transfer layer on the ball (Figure 20a) remains a primary subject for further investigation. High-sensitivity XPS will be employed to resolve the interfacial chemistry and detect potential monolayer-level transfer films.

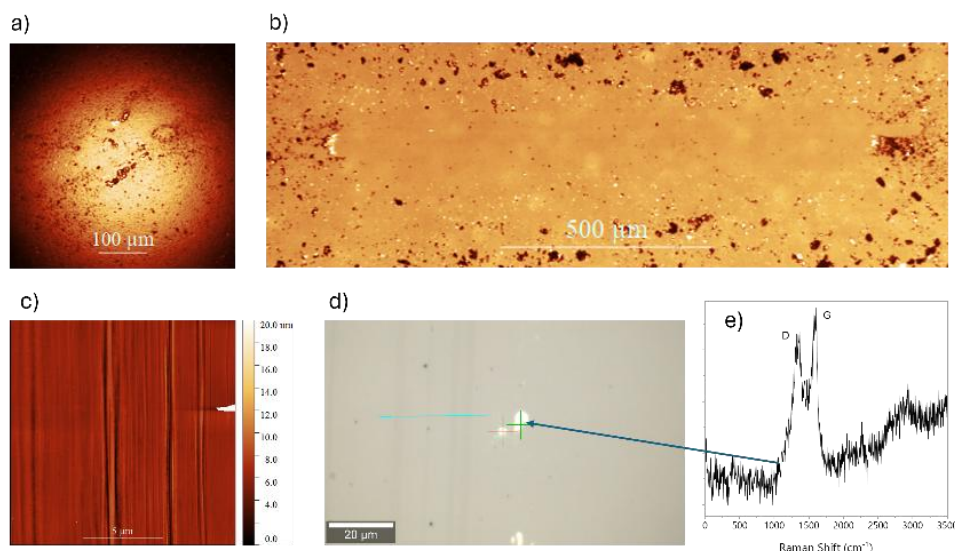


Figure 20. Post-test analysis of the GO/sapphire interface at 400 °C in vacuum. Optical images of the sliding ball (a) and wear track(b). AFM topography of the wear track taken from the middle of the stroke(c). Raman spectra of the contact area on the sliding ball after testing at elevated temperatures (d,e).

For the samples tested under vacuum conditions at RT and 400°C, we observed only slight wear on both the ball and the wear track (Figure 21 a,b). However, Raman spectroscopy of the worn area on the ball confirmed the presence of carbonaceous species (figure 21d). This indicates the formation of a transfer film that reduces direct contact between the sliding surfaces, thereby decreasing the overall friction.

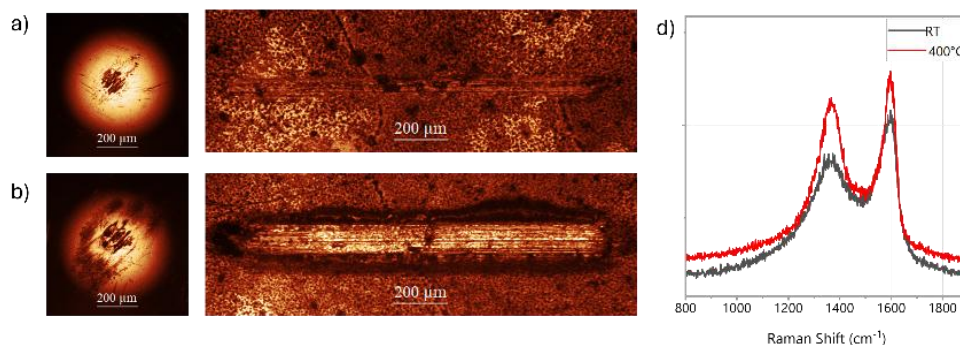


Figure 21. Tribological analysis of GO-coated 16MnCr5 steel substrates in vacuum. Optical images of the sliding balls and wear tracks tested at (a) room temperature and (b) 400 °C. (c) Raman spectra of the contact area on the sliding balls after testing at these respective temperatures.

A notable feature of the high-temperature Raman spectra is the narrowing of the D and G bands. Unlike room-temperature vacuum tests, where wear debris typically undergoes amorphization, elevated temperatures promote a more ordered transfer film, which is more effective at reducing friction and wear. While the GO coating on 16MnCr5 steel successfully reduced the COF to 0.03, it did not reach the ultra-low superlubricity values observed on sapphire substrates. This suggests that the sliding mechanism of GO on steel is primarily dominated by the physical separation of the sliding counterparts rather than the intrinsic interlayer shear of the GO layers themselves.

5) Testing of PtSe₂ layers: substrate and interlayer effects.

The development of PtSe₂ layers for steel substrates involves multiple stages. At each stage, the COF was evaluated to determine how the specific interlayer influences the system's tribological properties. Initially, we investigated model Si substrates with different ALD interlayers: Al₂O₃, HfO₂, and TiO₂.

Optical topography, AFM, and Raman characterization of the resulting wear tracks obtained at $F_a = 0.25\text{ N}$ (Figures 22 and 23) revealed distinct environmental behaviors. In ambient air, the Al_2O_3 interlayer resulted in severe abrasive wear. Conversely, HfO_2 and TiO_2 interlayers promoted a more beneficial adhesive wear mechanism, reducing the COF by forming a stable PtSe_2 transfer layer on the counterbody. In these cases, the COF is primarily determined by the sliding interface between the transferred PtSe_2 and the ALD interlayer.

Under vacuum conditions, a different mechanism was observed. The PtSe_2 layers tended to delaminate and adhere to the sliding contact, which significantly lowered the COF. Interestingly, the superior adhesion provided by the HfO_2 and TiO_2 interlayers resulted in a slight increase in the COF compared to more easily delaminated films, suggesting that interfacial shear is the dominant factor for vacuum lubrication.

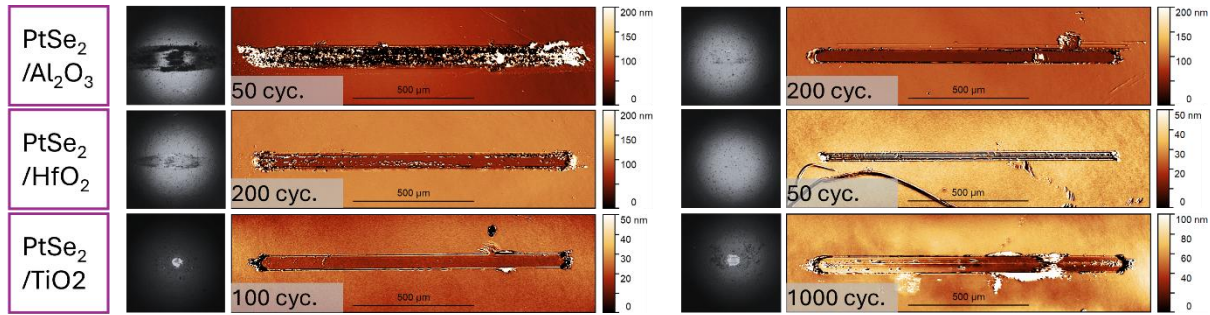


Figure 22. Interfacial wear analysis of PtSe_2/ALD systems. Topographical maps of wear tracks and optical images of sliding balls for PtSe_2 coatings on Al_2O_3 , HfO_2 , and TiO_2 interlayers. Results are shown for ambient air (left) and vacuum (right) conditions at various cycle counts.

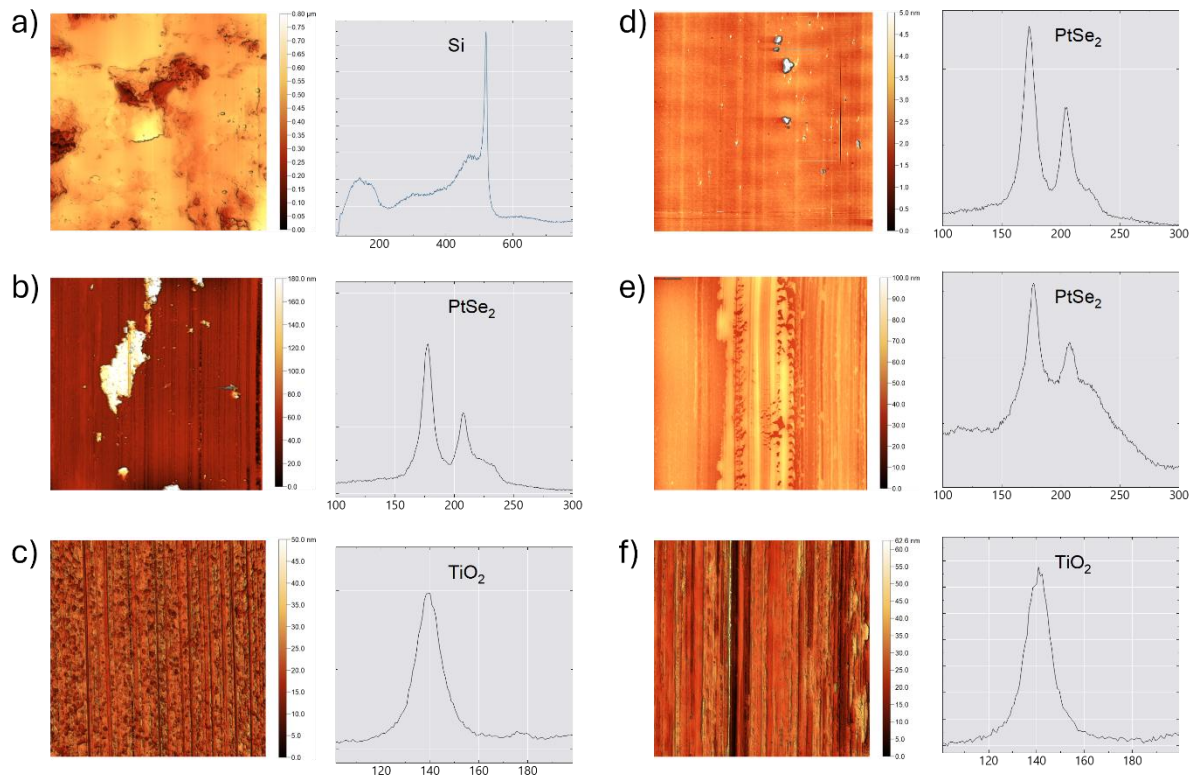


Figure 23. Morphological and structural analysis of the PtSe_2 wear tracks. AFM topographical images and corresponding Raman spectra were taken from the center of the wear tracks: (a, d) PtSe_2 with Al_2O_3 interlayer, (b, e) PtSe_2 with HfO_2 interlayer, and (c, f) PtSe_2 with TiO_2 interlayer. Panels (a–c) represent samples tested in ambient air, while (d–f) represent samples tested under vacuum conditions. Notation in Raman spectra represents the observed phase.

Coming to the investigation of samples on steel substrates, the PtSe_2 layer was selected for its ability to maintain a consistent COF across different environments. This stability is attributed to the vertical alignment of the flakes and a dominant adhesive friction mechanism. This assumption was confirmed using an HfO_2 and Al_2O_3 interlayer, which yielded a stable COF of 0.3. While the wear life in ambient air was limited (less than 300 cycles), the system demonstrated significant durability of over 10,000 cycles under vacuum conditions, particularly for samples with an HfO_2 interlayer. Based on the model Si surfaces, we hypothesize that the extended lifetime of the HfO_2 -buffered samples is due to the superior adhesion of PtSe_2 to the interlayer. This performance is likely attributed to the high thermal stability and high dielectric constant of HfO_2 , which promote stronger interfacial bonding and provide a more effective diffusion barrier against the steel substrate. These properties prevent premature film depletion and maintain a stable sliding interface. The failure of the Al_2O_3 system appears to be driven by sublayer wear. This was confirmed by an interrupted sliding test at 3000 cycles (Figure 24). Optical analysis of the HfO_2 wear track revealed a significant amount of residual lubricant debris, whereas the Al_2O_3 wear track showed extensive scratching, indicating the onset of severe adhesive wear on the underlying steel substrate.

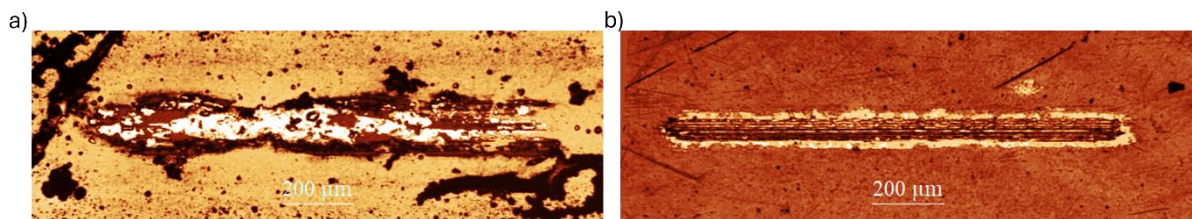


Figure 24. Optical micrographs of PtSe_2 wear tracks on 16MnCr5 steel substrates after an interrupted sliding test (3000 cycles) for (a) HfO_2 and (b) Al_2O_3 interlayers.

6) Testing of PtSe_2 layers: effect of temperature

"In vacuum at room temperature, PtSe_2 layers with Al_2O_3 interlayers exhibit rapid wear, though a low COF is temporarily sustained by the formation of a transfer layer and the movement of third-body wear debris. We suggest that the high shear forces encountered during sliding induce structural instability within the PtSe_2 layers. This leads to rapid thermal degradation and an increase in the COF.

Millstone achievements

The comprehensive analysis of wear tracks, interfaces, and sliding counterbodies conducted under WP3 provided the scientific foundation for fulfilling the project milestones. Specifically, the evaluation of surface topography and COF data enabled the successful completion of **Milestone 1 (M15)**: "Selection of the proper material/technology for deposition on steel substrates." PtSe_2 with a vertical flake orientation was selected as the primary technology for steel applications due to its ability to maintain a consistent COF across varying environmental conditions. In parallel, GO-based heterostructures were identified as the optimal choice for applications requiring extended sliding lifetimes and ultra-low friction.

The investigation of interfacial and transfer-layer dynamics led to the successful achievement of **Milestone 2 (M21)**: "Determination of friction mechanisms for optimization of sliding parameters." We established that MoSe_2/GO heterostructures offer the most effective route for COF control. The observed **interfacial chemical modifications and layer separation friction** mechanisms allow the COF to be tuned over a wide range by adjusting sliding parameters such as temperature. Additionally, ultra-low friction was achieved in **GO layers** through the **thermal desorption of surface adsorbates** at elevated temperatures. Finally, for **edge-oriented PtSe_2** , the **dominant adhesive sliding mechanism** was found to provide environmental stability, yielding nearly identical COF values in both ambient air. Wear life is dependent on the applied load and can exceed 10 000 cycles for samples tested in ambient air at $F_a \leq 0.025$ N and in vacuum at $F_a \leq 1$ N.

Report attachments

- 2) **D4.** Report on high-quality PtSe₂, MoSe₂ layers with different thicknesses, flake lateral size, and in-plane and out-of-plane orientation of the flakes with respect to the substrate surface normal (for PtSe₂) on the Si/SiO₂ and Al₂O₃ substrates.
- 3) **D5.** Report on PtSe₂, MoSe₂, and MoS₂ layers on the steel substrates.
- 4) **D6.** Report on Structure, morphology, and composition of the PtSe₂ and MoSe₂ layers

- 5) “Friction of the PtSe₂ layers with different grain sizes in ambient air and vacuum” at Tribology 2025, Albufeira, Algarve Region, Portugal 23 - 25 April 2025.
<https://setcor.org/userfiles/files/2025/AlBueira23to25April2025/Book-of-Abstracts/SICT-PlasmaTech-Tribology-2025-Book-of-bstracts.pdf>

- 6) “Tribological properties of Ti₃C₂T_x in vacuum and ambient air conditions” at “8th International Conference on Multifunctional, Hybrid and Nanomaterials 2025, Montpellier, France, 3-6 March 2025
<https://virtual.oxfordabstracts.com/event/public/44200/session/134733>
Príspevok - P1.079

- 7) “Tribological and micromechanical performance of hard yet tough TiSiN/ WN coatings: An experimental analysis of multilayer effects”, International Journal of Refractory Metals and Hard Materials 131 (2025) 107212
Link - <https://doi.org/10.1016/j.ijrmhm.2025.107212>

- 8) “The low-temperature photoluminescence of thin PECVD Si–C–N–H films: An effect of hydrogenation”, Optical Materials 160 (2025) 116751
Link - <https://doi.org/10.1016/j.optmat.2025.116751>

- 9) “Effect of polymer grafting on the tribological performance of graphene oxide under ambient air and vacuum”. *ACS Appl. Mater. Interfaces* (2025) 17 (32): 46172–46184.
Link - <https://doi.org/10.1021/acsami.5c09549>

- 10) “Investigating Ohmic contacts in PtSe₂-based electronics”, *FlatChem* 54 (2025) 100943
Link - <https://doi.org/10.1016/j.flatc.2025.100943>

- 11) “Heterogeneous integration of β-Ga₂O₃ and polycrystalline diamond via SiC and SiO₂ interlayers with optimized adhesion and thermal boundary resistance for power-electronic applications”. *Carbon* 260 (2026) 121921
Link - <https://doi.org/10.1016/j.carbon.2026.121921>

- 12) “Environment-dependent sliding interface of MoSe₂/GO heterostructures”
Submitted draft
- 13) “Nano- and Macroscale Tribological Properties of the PtSe₂ Layers with Different Morphology in Ambient Air and Vacuum Conditions” at 2025 IEEE 15th International Conference “Nanomaterials: Applications & Properties” Bratislava, Slovakia, Sept. 7-12, 2025
Link - https://ieeenap.org/data/Book_of_Abstracts_2025.pdf
Príspevok - 05mtfc-45

- 14) “Effect of the PECVD Plasma Power Density on the Structural and Friction Properties of the SiC-N Coatings” at 2025 IEEE 15th International Conference “Nanomaterials: Applications & Properties” Bratislava, Slovakia, Sept. 7-12, 2025
Link - https://ieeenap.org/data/Book_of_Abstracts_2025.pdf
Príspevok - 05mtfc-44

- 15) “Friction of GO/ MoSe₂ films in ambient air and vacuum conditions” at SICT 2026 / PLASMA TECH 2026 / TRIBOLOGY 2026 JOINT CONFERENCES 06 - 08 May 2026 | Prague - Czech Republic
<https://setcor.org/userfiles/files/2026/Prague/Book-of-Abstracts/SICT-PlasmaTech-Tribology-2026-Book-of-Abstracts.pdf>
- 16) “Effect of oxide sublayers on the tribological properties of PtSe₂ coating in different environment conditions” at NORDTRIB 2026 9-12 June 2026 Tampere University Tampere, Finland
<https://trepo.tuni.fi/bitstream/handle/10024/237862/978-952-03-4666-9.pdf?sequence=2&isAllowed=y>
- 17) “Effect of oxide buffer layers on the tribological properties of PtSe₂/oxide heterostructures in different environment conditions”
Submitted draft
- 18) **D7.** Annotation to the submitted project „Advanced graphene-based polymer hybrids for environment-adaptive lubrication“ (APVV-25-0739).