



Article

Tribological Performance of MoS₂ Coatings in Various Environments

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Abstract: Molybdenum disulfide (MoS₂) is a well-known solid lubricant for tribosystems running in vacuum or dry gases. Problems arise due to its sensitivity to humidity, which is a drawback for its application under ambient conditions. However, by using a physical vapor deposition (PVD) process, deposition parameters can be optimized not only to gain a coatings structure with favorable frictional properties but also to minimize the sensitivity to attack by water molecules. Therefore, an improved tribological behavior even under moist conditions can be achieved. MoS₂ coatings are also candidates for being applied at cryogenic temperatures. They already have proven their suitability, e.g., for sliding support elements between superconducting magnets of the nuclear fusion-experiment Wendelstein 7-X. However, these coatings were exclusively produced for this particular application and the utilization for more common tribosystems may be precluded due to cost considerations. In view of a wider range of applications, pure and Cr containing PVD-MoS₂ coatings with an optimized structure were tested under varying environments including hydrogen gas and cryogenic temperatures. Results of the most promising variant are presented in this paper.

Keywords: solid lubrication; MoS₂; extreme environments; hydrogen; cryogenic environment

1. Introduction—Lubricating Properties of MoS₂

For vacuum and inert gaseous environment, molybdenum disulfide (MoS₂) is a well suited and widely applied solid lubricant [1]. In vacuum, solid lubricants of this type show minimum friction coefficients as low as 0.03, and, under certain conditions, even lower. However, a major drawback for the application of MoS₂ is its sensitivity to the presence of water vapor. In normal humid air, friction coefficients between 0.15 and 0.30, accompanied by high wear, are observed.

For low temperature environment some authors report a minimum temperature of 88 K for special liquid lubricants [2]. However, commercially available oils and greases are only applicable at temperatures above 173 K. Furthermore, the cryogenic liquids themselves are not able to build up hydrodynamic lubrication [3]. Therefore, for components such as bearings, liners, moving seals, and valves, dry sliding, dry rolling or non-contacting systems must be employed.

There are some special applications that require smooth sliding surfaces in vacuum environment at cryogenic temperatures. One example is the support structure of large superconducting magnets for nuclear fusion devices [4]. These magnets are cooled by liquid helium, which has a boiling temperature of 4.2 K. During cool down and ramping up of the magnetic field, the superconducting coils experience large forces due to thermal shrinkage of the materials and an increasing electrical current in the magnetic field. This results in a very slow tangential movement of several mm at the supports of the individual coils. Because frictional heat and mechanical shocks are able to initiate a normal conducting zone, a lubricant must be employed that guarantees low friction sliding without stick-slip at these

supports. Tests have shown that only MoS₂ coatings without any additions are able to meet such requirements [5].

Similar to graphite, MoS₂ crystallizes in a lamellar structure. The Mo atoms form hexagonal planes with covalently bonded S atoms at both sides. The spacing between the Mo and S atoms is 0.154 nm, between the S atoms to the nearest MoS₂ plane 0.308 nm. Between the planes only weak Van-der-Waals forces are acting, resulting in easy shearing tangential to the planes, which is responsible for the good lubricating properties. Molecular dynamic simulations show that during sliding a tribofilm is formed with basal orientation of the lattice planes [6]. These model calculations also show that, with perfectly aligned layers, a minimum friction coefficient of 0.006 is possible, due to the repulsive Coulomb forces between the sulfur atoms.

MoS₂ is applied by burnishing, physical or chemical vapor deposition (PVD, CVD), or with binders as anti-friction (AF) coatings. It can also serve as friction reducing component in polymer composites [1].

Anti-friction (AF) coatings consist of one or more solid lubricants and, in most cases, an organic binder. Some variants, in particular those containing MoS₂ or polytetrafluoroethylene (PTFE), are suitable for low temperature operation.

Many AF coatings need a certain running in for achieving the desired properties. One extreme case is shown in Figure 1. It is a sprayed MoS₂ coating with polycarbamide binder, which was specially designed for space applications [7]. Figure 1a shows a scanning electron microscopy (SEM) image of the virgin coating, which consists of only loosely adhered particles. During loading and sliding the particles are compacted and get a preferred order, resulting in a very smooth film (Figure 1b). In Figure 2, the friction coefficients of this coating against 100Cr6 steel balls (AISI 52100) are shown for several cryogenic environments [8]. It can be seen that it provides effective lubrication in all of the investigated media. In cryogenic environment, friction is considerably lower than at room temperature. The lowest friction coefficient is 0.02 at 77 K in vacuum. In most cases, higher loads and velocities give lower friction, which is a common feature of MoS₂ coatings.

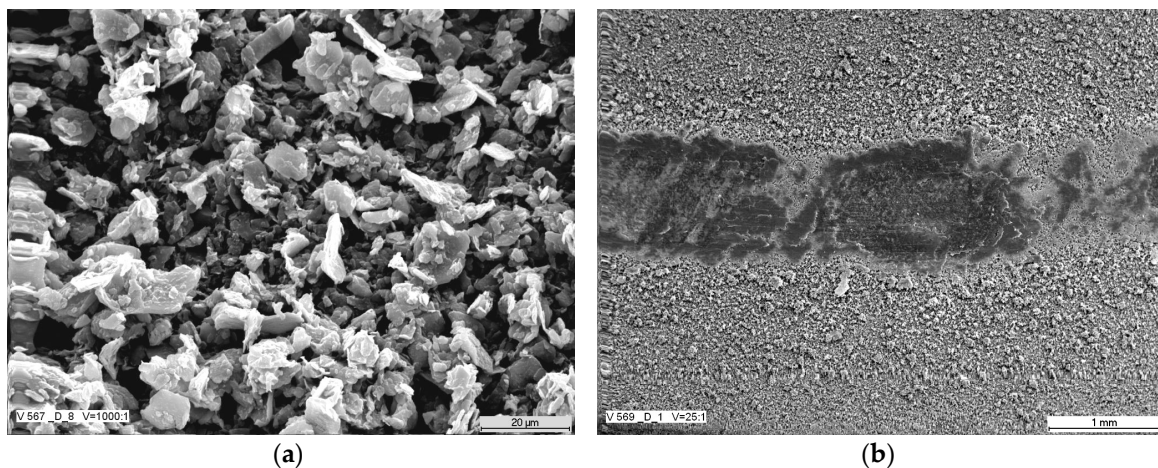


Figure 1. AF coating with polycarbamide binder [7]: (a) virgin coating; and (b) after sliding in liquid helium (LHe).

By using a PVD process, deposition parameters can be optimized to gain coatings with a more textured structure orienting the (002) basal planes parallel to the substrate surface [9]. A similar concept has been used by Koch et al. [5] for coatings developed for support elements in the superconducting magnet system of the WENDELSTEIN W7-X fusion experiment. They are able to withstand extreme operational conditions such as temperatures as low as 5 K in combination with high vacuum. Other authors report friction coefficients between 0.015 and 0.06 in liquid nitrogen (LN₂) and minimum wear on both sliding surfaces for Ti containing PVD-MoS₂ coatings [10,11].

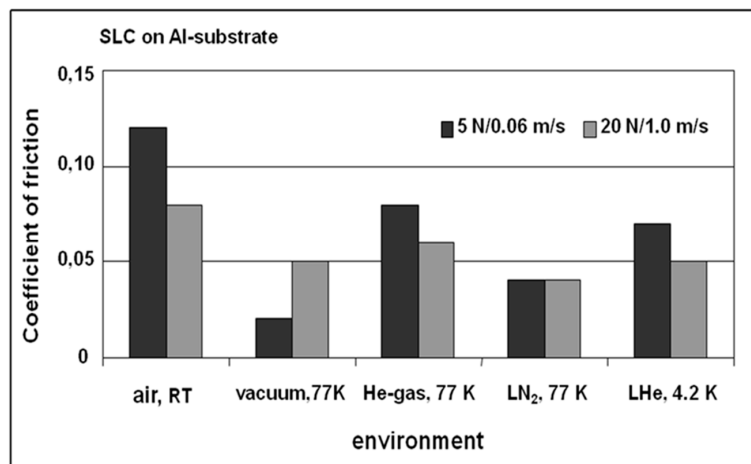


Figure 2. Coefficient of friction of a MoS₂-AF coating with polycarbamide binder in air and cryogenic media [8].

A general feature of MoS₂ coatings is that the friction coefficient decreases with increasing load, which makes them suitable for high-loaded friction contacts. The friction curve of a PVD coating for such an application in cryogenics is shown in Figure 3. This is the type of coating which is used for the narrow support elements in the WENDELSTEIN 7-X magnet system. It was chosen because it provides low friction without stick-slip effects and needs nearly no running-in [12]. Only the first cycle deviates noticeably from the following. During the next few cycles a stable frictional behavior is established with friction coefficients in the range between 0.03 and 0.06. Only very small static friction peaks occur and no stick-slip effects are visible. Static and dynamic friction have almost the same values. No coating failure occurred within the test duration, which was several hundred cycles for this particular application. A further result of this test was that the roughness of the substrate has only negligible influence on the steady state friction coefficient. Other authors report an optimum substrate roughness of $R_a = 0.1$ to $0.15 \mu\text{m}$ [10]. The sample with a roughness of $R_z = 2 \mu\text{m}$ is approximately within that range.

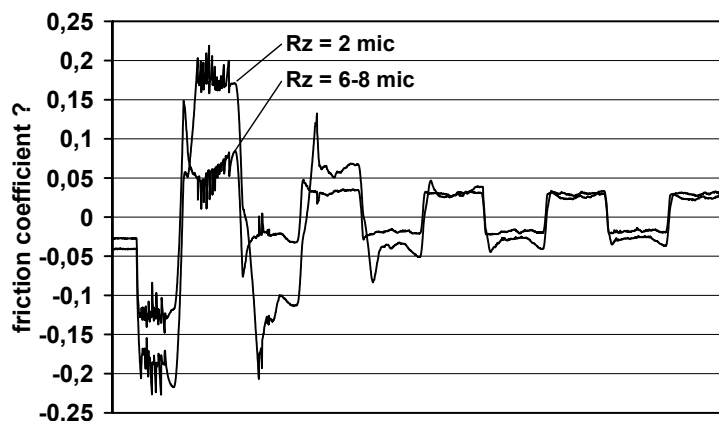


Figure 3. First friction cycles of a sputtered MoS₂ coating against Al-bronze in LHe environment; two samples with different substrate roughness; load: 250 N; average sliding velocity: $0.2 \text{ mm}\cdot\text{s}^{-1}$ (negative values indicate the reciprocating motion) [12].

Because the application of MoS₂ coatings in normal humid air is still a problem a test program for further optimization by a variation of deposition parameters for pure MoS₂ and doping by Cr atoms was conducted. The results of the best variants are shown in the following.

2. Materials and Methods

2.1. Test Rig for Tribological Measurements at Temperatures below 120 K

Tribological investigations at cryogenic temperatures require special devices (cryotribometers) in respect of cooling system, thermal insulation, and instrumentation. Figure 4 shows an example of an apparatus for pin-on-disc tests in liquid helium (LHe) [13]. The liquid coolant is filled directly into the sample chamber and the friction couple is immersed completely into the liquid cryogen (bath cryostat operation). Therefore, the environmental temperature is equal to the boiling temperature T_b of the coolant (LN_2 : $T_b = 77.3 \text{ K}$; LH_2 : $T_b = 20.4 \text{ K}$; LHe : $T_b = 4.2 \text{ K}$). The advantage of this method is a very effective cooling of the sample. The frictional heat is not only removed by heat conduction and convection, but also by evaporation of the liquid. The cryostat shown in Figure 4 also allows cooling independently of the boiling temperature of the coolant. In this case, the coolant flows through a heat exchanger and the sample is surrounded by vacuum or contact gas. With additional heating, it is possible to adjust the temperature between 4.2 K (with LHe-cooling) and room temperature independently from the pressure in the sample chamber. The sample chamber can be evacuated to a residual pressure in the order of 10^{-5} mbar and therefore, the cryotribometer is also suitable for friction tests in high vacuum.



Figure 4. Cryotribometer CT3, suitable for cryogenic, vacuum and H_2 environment.

2.2. Materials and Sample Preparation

The disc-shaped specimens were coated by an unbalanced magnetron sputter process on a PVD coating plant H-O-T TT 300 at the Chair of Engineering Design, Friedrich-Alexander-Universität Erlangen-Nürnberg [14]. Samples were held in front of the target without additional rotation, thus enabling an adjustment of a well defined target/substrate distance. Coating thickness was kept constant at $1.8 \mu\text{m} \pm 0.3 \mu\text{m}$, adapting deposition time accordingly. To avoid unintended annealing effects during deposition, the substrate material consisted of secondary hardened high speed steel 1.3343 (S 6-5-2), which is tempering-resistant up to $450 \text{ }^\circ\text{C}$; hardness remained at approximately 62 HRC.

Five different coatings were tested, three variants of pure MoS_2 and two Cr containing types ($\text{MoS}_2\text{:Cr}$). One coating denoted as “standard” (STD) was identified by a preceding extensive variation

of deposition parameters, resulting in different mechanical and tribological properties. The methods and results of these tests are reported in detail in Reference [14].

Before the deposition, the substrates were polished and ultrasonically cleaned in acetone and isopropyl alcohol. Additionally, they were prepared by an argon-ion etching process at 2.3 Pa, applying a 600 V pulsed DC bias to the substrates for half an hour. Argon also served as working gas for sputtering.

The deposition parameters for the standard coating were a cathode voltage of 800 V, a deposition temperature of 150 °C, an argon pressure of 0.51 Pa and flow rate of 80 sccm, a distance of 90 mm between the sputter cathode and the sample and a resulting cathode current of 2.09 A. This type was further developed in order to produce coatings with higher intrinsic stress. The results of the two most promising variants are shown in the following. Both types, denoted as HS and LS, had higher intrinsic stresses than the standard coating (200 MPa), but to a different degree (HS: 370 MPa, LS: 300 MPa). For both variants the cathode voltage was 700 V and the temperature 50 °C. For the LS type, the cathode distance was reduced to 65 mm and the Ar flow rate increased to 115 sccm.

Additionally two coatings were doped with chromium at a high (120 A) and low (70 A) Cr cathode current, respectively, resulting in chromium contents of 10 and 5 at%. In the figures, these variants are referred to as CH and CL.

The tribological tests were carried out in ball-on-disc configuration. Hardened 100Cr6 steel balls (AISI 52100) with a diameter of 4 mm served as counter bodies. If not otherwise indicated in the figure captions, the applied load was 10 N (maximum Hertzian contact pressure: 1860.6 MPa) and the sliding velocity was 0.1 ms⁻¹.

The tests were carried out in high vacuum, in vacuum with H₂O vapor at a pressure of 1.4×10^3 Pa, in gaseous hydrogen at normal and low pressure (1×10^4 Pa) as well as in liquid hydrogen.

The wear coefficients were determined by stylus profilometry, measuring the wear track at four different positions (shifted by 90°). The coefficient of friction (COF) was averaged for a sliding distance of approximately 1000 m, excluding the running-in phase. In order to test the wear life, the tests were run until coating failure was detected (failure criterion: COF > 0.25).

3. Results

3.1. Vacuum Environment

Under vacuum conditions all variants showed friction coefficients below 0.1 and wear coefficients in the range of 1 to 3×10^{-7} mm³·N⁻¹·m⁻¹. As an example, Figure 5 shows a friction plot for a PVD-MoS₂ coating in high vacuum. The coating is similar to the one used in the Wendelstein 7-X fusion experiment [5]. The peaks to negative direction are caused by test stops and restarts. It can be seen that the friction coefficient is around 0.06 and the wear life more than 350,000 cycles.

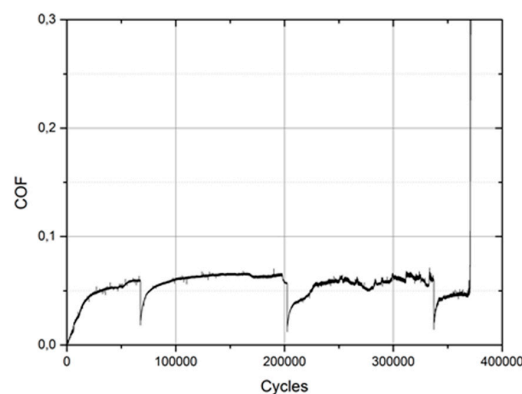


Figure 5. Coefficient of friction of a PVD-MoS₂ coating [5] under high vacuum conditions (residual pressure: 2×10^{-5} mbar); counterbody: 10-mm 100Cr6 ball, load: 40 N; sliding velocity: 0.5 ms⁻¹.

3.2. Air Environment with 50% Humidity

As expected, in normal air with a water vapor partial pressure of 1.4×10^3 Pa, corresponding to 50% relative humidity, the COF increased to values between 0.1 and 0.25 and the wear coefficient up to $10^{-5} \text{ mm}^3 \cdot \text{N}^{-1} \cdot \text{m}^{-1}$, which is more than one order of magnitude higher than under vacuum conditions. A typical example of the friction development is shown in Figure 6. However, best variants showed values in the order of $10^{-6} \text{ mm}^3 \cdot \text{N}^{-1} \cdot \text{m}^{-1}$, which is comparable to the wear of diamond-like carbon (DLC) coatings, developed for operation under high humidity [15]. As expected, coatings that were appropriate for ambient air conditions had a high degree of (002) basal plane orientation. This was detected by X-ray diffraction (XRD), which was carried out for the coating with the best performance in humid environment. The result is shown in Figure 7. Therefore, by means of a suitable adapted PVD-process MoS_2 coatings without further dopants can be developed that are able to sufficiently withstand adverse conditions such as high humidity. The results of the corresponding test program and the effect of the deposition parameters are reported in detail in Reference [14].

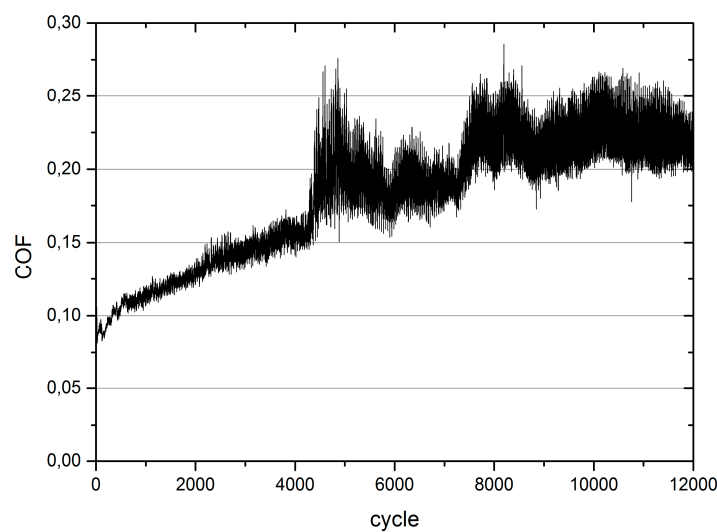


Figure 6. Example of the friction increase of a MoS_2 coating in air with 50% relative humidity.

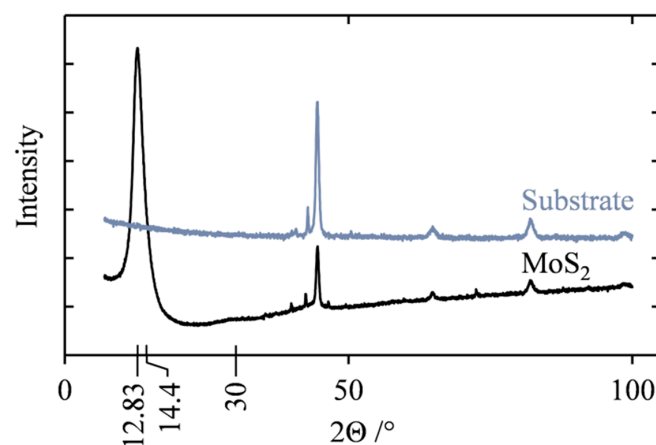


Figure 7. X-ray diffraction (XRD) patterns: Dominating (002) orientation of the coating with highest durability under moist conditions. Pattern of the uncoated sample for comparison [14]. Reproduced with permission from “Surface and Coatings Technology”; published by Elsevier, 2013.

Figure 8a shows a high resolution transmission electron microscopy (HR-TEM) image of a coating with higher internal stress. It still has a highly ordered layered structure which is visible in the

upper region. The selected area diffraction (SAED) image of Figure 8b clearly shows reflexes of the (002) planes.

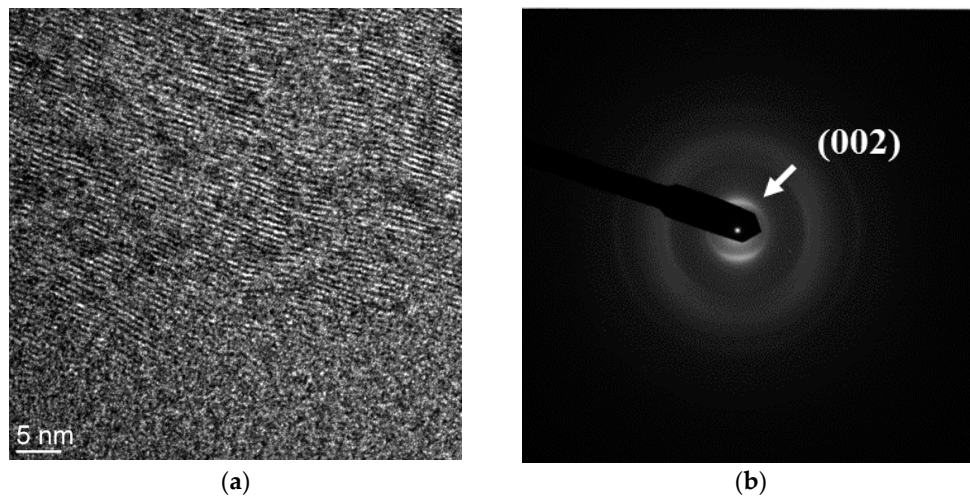


Figure 8. (a) High resolution transmission electron microscopy (HR-TEM) and (b) selected area diffraction (SAED) images of a MoS₂ coating with higher internal stress.

3.3. Low Pressure Water Vapor Environment

In order to test the influence of water vapor without the presence of oxygen, only H₂O gas was filled into the vacuum chamber until a pressure corresponding to 100% rH was reached. In Figure 9, it can be seen that, for a MoS₂:Cr variant with 5 at% Cr, the friction coefficient stabilized at about 0.17, which is considerably higher than in inert environment but still low for solid lubricants. This test clearly shows that oxidation to MoO₃ is not the only acting wear mechanism.

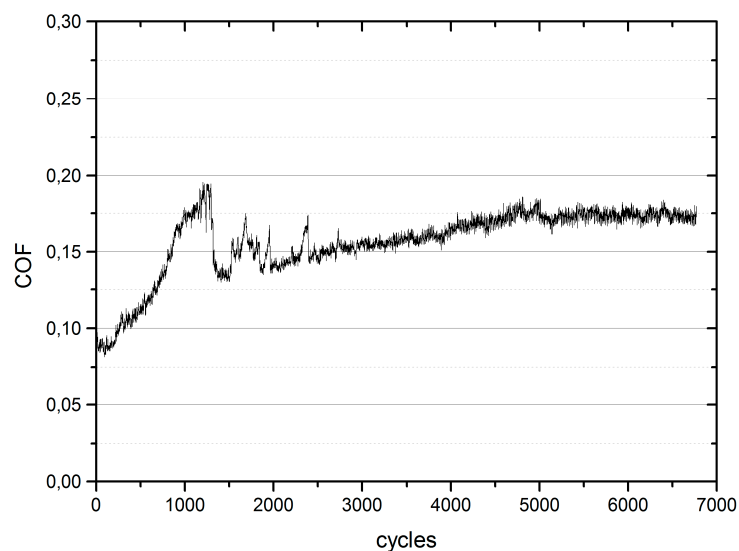


Figure 9. Friction of a MoS₂:Cr coating (5 at% Cr) in low pressure water vapor at room temperature.

Figure 10 shows the friction and wear data for all five variants in water vapor. The friction coefficients of the pure LS and HS coatings are about 0.13 and 0.17, respectively, for the Cr containing variants. For all optimized coatings, the wear coefficients are in the order of $10^{-6} \text{ mm}^3 \cdot \text{N}^{-1} \cdot \text{m}^{-1}$. For the standard coating both, friction and wear are considerably higher. This proves the lower sensitivity to humidity of these variants.

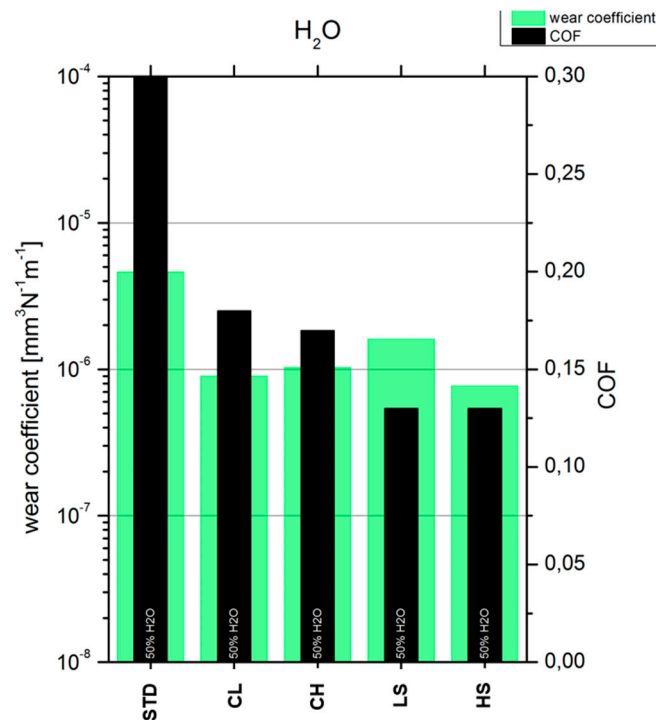


Figure 10. Friction and wear coefficients of optimized MoS₂ coatings vs. 100Cr6 in low pressure H₂O vapor; load: 10 N, sliding velocity: 0.1 ms⁻¹.

3.4. Hydrogen Environment

Tests in gaseous hydrogen were performed at 100 mbar and 1 bar environmental pressure. All variants showed a low COF, comparable to or even lower than the vacuum values. Figure 11 shows a friction plot for the reference coating in H₂ gas at 1 bar. Friction was very stable with a COF of 0.03 and the wear life with 340,000 cycles was satisfactory.

Results were similar for the Cr alloyed types (Figure 12), again showing friction and wear comparable to vacuum conditions. The wear life was longest in H₂ gas at 1 bar. After about 100 h, or 430,000 cycles, the test was stopped without showing coating failure.

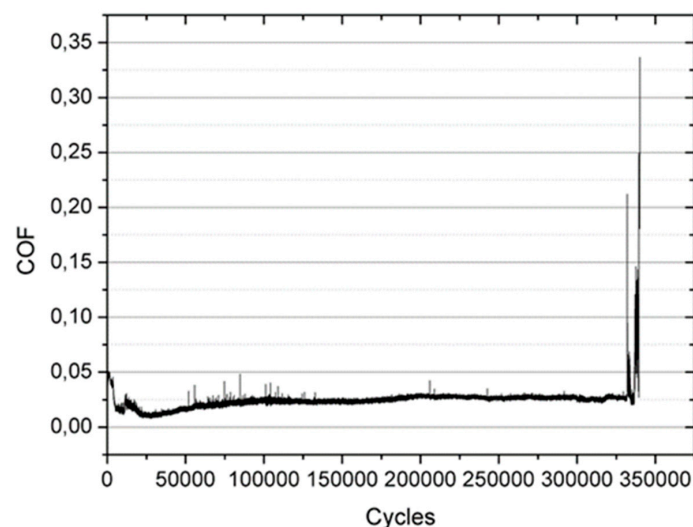


Figure 11. Friction of a MoS₂ coating (STD) vs. 100Cr6 in H₂ gas at room temperature.

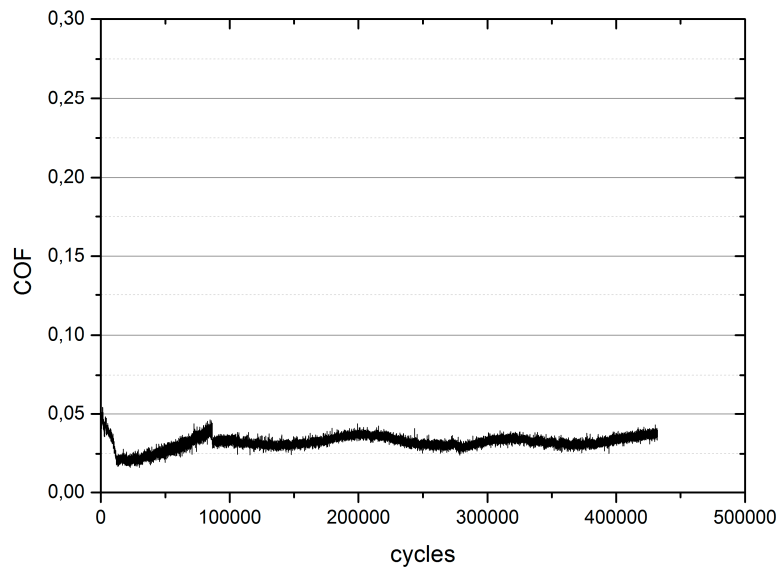


Figure 12. Friction of MoS₂:Cr (type CL) vs. 100Cr6 in H₂ gas at room temperature.

Figure 13 shows a comparison of the friction and wear behavior between high and low pressure hydrogen and vacuum environment for the four variants. Between vacuum and low pressure hydrogen, the differences are small. Only the pure coatings with optimized structure showed higher wear. In hydrogen gas at normal pressure all variants showed higher wear with the exception of the standard coating, which showed a similar wear rate and distinctively lower friction than in vacuum. In addition, the friction of the pure variant with lower intrinsic stress was as low as for the standard variant. However, these results are based on only two tests for each condition. Therefore, only the general tendency to low friction and wear in hydrogen environment is verified. The pressure dependence is less clear and needs to be proved by further tests.

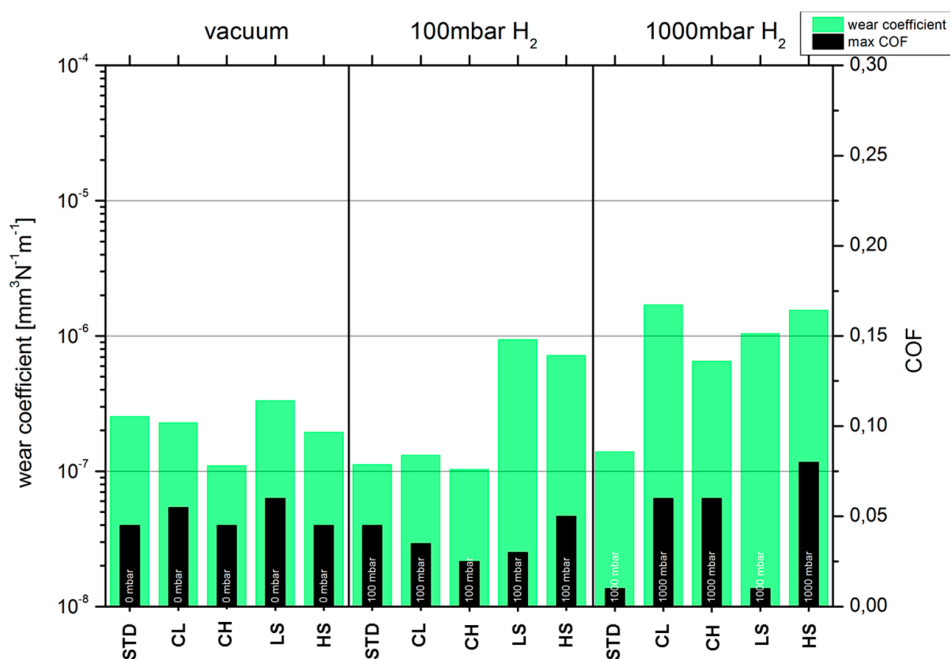


Figure 13. Friction and wear coefficients of optimized MoS₂ coatings vs. 100Cr6 in vacuum and H₂ environment; load: 10 N, sliding velocity: 0.1 ms⁻¹; STD: reference coating; MoS₂:Cr: CL: 5 at% Cr; CH: 10 at% Cr; pure coatings with higher internal stress: LS: 300 MPa; HS: 370 MPa.

3.5. Cryogenic Environment

A relatively long endurance was measured for MoS₂:Cr (5 at% Cr) at 77 K in liquid nitrogen (Figure 14). In contrast, in liquid helium, which is also an inert environment, the COF increased to 0.2 after only 3000 cycles (Figure 15). In addition, the large fluctuation indicated coating failure.

Additional experiments were conducted in liquid hydrogen (LH₂) at 20 K. In this environment, for most samples, the COF was initially as low as approximately 0.05, while the Cr-containing types yielded 0.06. However, as can be seen in the friction curve of Figure 16, wear life was short. The COF began to rise steeply beyond 0.1 after only 1000 and 3000 cycles for all variants. A maximum in durability was achieved at 4000 cycles before the test was aborted at COF > 0.25. Again, the variants with optimized structure showed no improvement in comparison to the standard coating.

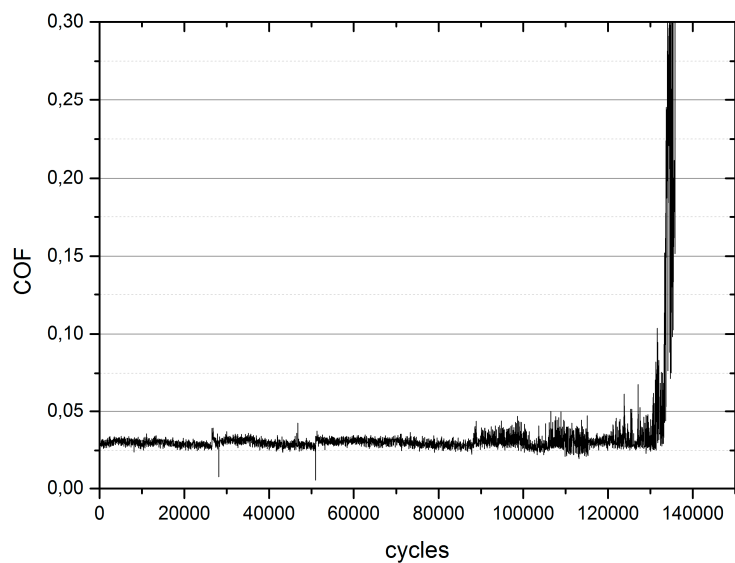


Figure 14. Friction of MoS₂:Cr (5 at% Cr) vs. 100Cr6 in LN₂, load: 10 N, sliding velocity: 0.1 ms⁻¹.

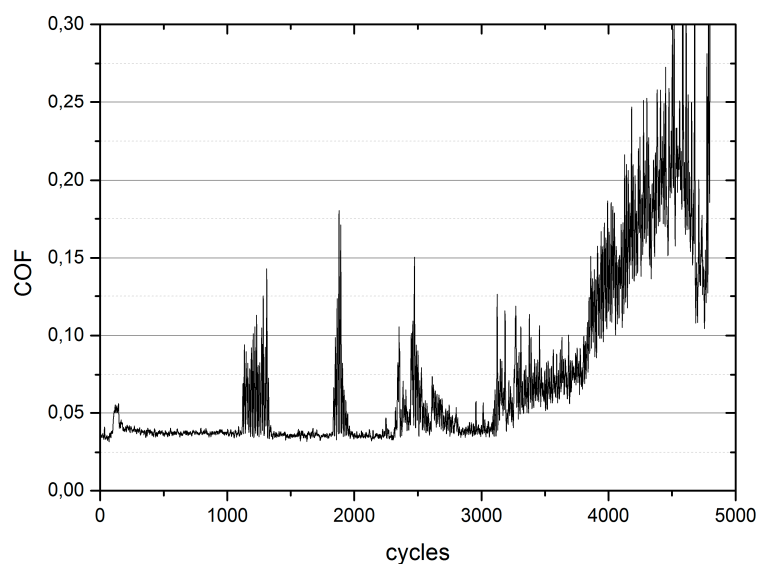


Figure 15. Friction of MoS₂:Cr (5 at% Cr) vs. 100Cr6 in LHe, load: 10 N, sliding velocity: 0.1 ms⁻¹.

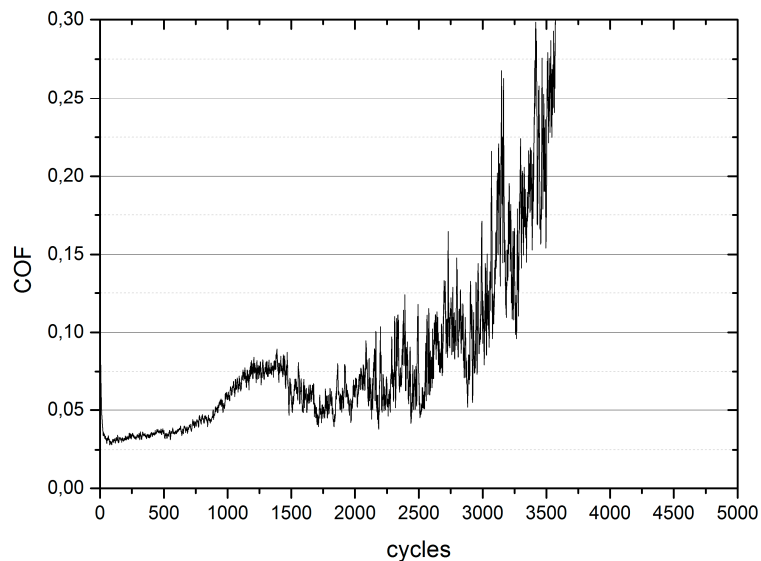


Figure 16. Friction of MoS₂:Cr (5 at% Cr) vs. 100Cr6 in liquid hydrogen (LH₂), load: 10 N, sliding velocity: 0.1 ms⁻¹.

4. Discussion

The results for the optimized variants show that already pure MoS₂ coatings with increased internal stress have a higher durability in humid environment. Doping with Cr does not result in lower friction and the wear is not influenced significantly. The reason for the lower sensitivity to humidity of the coatings with optimized structure may be the lower number of MoS₂ crystallite edges at the surface, which are postulated to be favorite areas for water adsorption [1]. Water molecules at the crystallite edges may be responsible for blocking crystallite alignment and shearing of the MoS₂-planes. This mechanism is active only at the surface and crystallite edges and may be an explanation for the fact that Cr-atoms in the bulk materials do not have a positive influence. Because, even for the best variants, numerous active sites are present at the surface, a low friction coefficient similar to the vacuum value cannot be achieved.

In contrast to H₂O, low pressure H₂ environment at room temperature enhances the friction properties of MoS₂. For all variants, friction is considerably lower than in humid environment. Depending on the coating type, wear is unchanged or up to one order of magnitude lower. In normal pressure hydrogen, all new variants show a slightly higher friction and a wear rate similar to humid environment. However, the wear life is also long in hydrogen gas at 1 bar. Because, for such coatings, most of the film thickness is removed during running in, wear rate is not directly related to the wear life. Contrary to humid environment, in hydrogen, a remaining thin layer seems to be stable for a long time.

The differences in the tribological behavior of all tested variants between the different environments show that oxidation is not the only mechanism responsible for increasing friction and wear of MoS₂. H₂O molecules seem to impede the alignment and shearing of the MoS₂ layers resulting in higher friction. In contrast, H₂ may act as termination of reactive edges of the MoS₂ planes, which is a mechanism similar to hydrogen in carbon coatings [15]. As a second effect, H₂ may trap residual oxygen in the environment.

There is no straight forward explanation for the very short wear life of all tested coatings in LHe and LH₂; in particular, because at 77 K in liquid nitrogen, more than 100,000 friction cycles with a COF not higher than 0.03 are still possible. However, in earlier tests with other MoS₂ variants, deviations in the tribological behavior between 77 and 4.2 K were also detected [16]. One reason for early failure in LHe and LH₂ may be thermal expansion mismatch between the coating and the substrate. The stress caused by this effect increases with decreasing temperature. For a verification of this effect, tests with coatings with different internal stresses are planned.

5. Conclusions

Under humid environmental conditions, MoS₂ usually exhibits high friction and short wear life. Improvements can be achieved by optimizing the deposition process in order to an orientation of the (002) basal plane parallel to the substrate surface or by doping with metal atoms, e.g., Cr.

Gaseous H environment at room temperature enhances the tribological properties of MoS₂ coatings. Friction and wear are low and comparable to the vacuum values under different hydrogen gas pressures at room temperature.

Early coating failure is observed in liquid hydrogen with a boiling temperature of 20 K. However, in liquid nitrogen at 77 K, friction is low and wear life is satisfactory. In liquid hydrogen as well as in liquid helium (4.2 K), friction is only low at the very beginning of sliding. After a few friction cycles, all variants showed early failure. Thus, in this extreme temperature range, thermal mismatch seems to dominate the tribological behavior and should be accounted for in future.

The initially low COF could motivate more elaborate testing in the future on this topic as well as on wear reduction. Operating components such as pumps and valves reliably in (liquid) hydrogen could become more important as hydrogen gains momentum as medium for energy storage and conversion.

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Author Contributions: Thomas Schneider designed and carried out the experiments in vacuum, inert, and hydrogen environment. Thomas Gradt wrote the paper.

Conflicts of Interest: The authors declare no conflict of interest.

Abbreviations

The following abbreviations are used in this manuscript:

COF	Coefficient of friction
LN ₂	Liquid nitrogen
LH ₂	Liquid hydrogen
LHe	Liquid helium
PVD	Physical vapor deposition
CVD	Chemical vapor deposition
HR-TEM	High resolution transmission electron microscope
SAED	Selected area electron diffraction
RT	Room temperature
STD	reference or standard coating
HS	coating variant with an internal stress of 370 MPa
LS	coating variant with an internal stress of 300 MPa
CH	MoS ₂ :Cr coating with 10 at% Cr
CL	MoS ₂ :Cr coating with 5 at% Cr

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