

MoS₂ Coatings for the Narrow Support Elements of the W-7X Non Planar Coils

F. Koch*, R. Nocentini, B. Heinemann, S. Lindig, P. Junghanns, H. Bolt

Max-Planck-Institut für Plasmaphysik, Euratom Association,
Boltzmannstr. 2, D-85748 Garching, Germany

Abstract

WENDELSTEIN 7-X (W7-X) is a stellarator presently under construction and assembly in Greifswald, Germany. The superconducting magnet system of W7-X consists of planar and non planar coils. The electromagnetic forces on the superconducting magnet are supported by backing of all coils against a central ring and by wedging among coil casings through “Narrow Support Elements” (NSE) that have to satisfy very specific requirements.

Anti friction MoS₂ coatings have been developed and tested at room and cryogenic temperature (77K) to allow relative sliding of adjacent coils under load without stick-slip to avoid coil quenching. The influence of thickness on the coating endurance has been demonstrated. To prevent ageing both constructive methods and protective top coats have been investigated.

This paper describes the atomic sputter deposition technique, the reduced and full-scale tribological tests, ageing tests and arrangements to protect the coatings from humidity and oxidation which have been carried on so far. After deposition and after testing/exposure the coatings have been characterized in detail to obtain information for the industrial production of the coatings.

***Corresponding author:**

Tel.: +49 89 3299 2104

Fax: +49 89 3299 1212

e-mail: freimut.koch@ipp.mpg.de (Freimut Koch)

Keywords: Stellarator; Wendelstein 7-X; MoS₂; Solid lubricant coating, Cryogenic temperatures, Vacuum

1. Introduction

The WENDELSTEIN 7-X (W7-X) is a new steady state stellarator presently under construction at the Max-Planck-Institut für Plasmaphysik in Greifswald, Germany [1]. The superconducting magnet system of the W7-X consists of 50 non planar coils and 20 planar coils which are arranged as 5 identical modules in a pentagonal shape. Figure 1 shows a segment of W7-X with five non planar coils. The electromagnetic forces are supported by backing of all coils against a central ring and by wedging among coil casings through “Narrow Support Elements” (NSE) between the inner legs of two adjacent coils and “Lateral Support Elements” between the outer legs [2].

Three to seven NSEs (depending on type of coil pair) are placed between two adjacent coils in an area, where the coils come close to each other on the inner side (300 NSEs totally). Specific NSE blocks are cast or welded on both sides of the stainless steel coil casings (fig. 2). Between those blocks the NSEs are installed in a very limited space and have to satisfy the following requirements:

- to take high compression forces up to 1.5 MN (design value) at full magnetic field (3 T)
- to allow relative sliding of adjacent coils up to 5 mm each direction during energizing the coils
- to allow relative tilting between adjacent coils up to 1°
- smooth sliding and tilting to avoid a quench in the superconductor
- to assure the assembly of the coils with high accuracy
- to operate for the lifetime of the experiment (about 4000 magnetising cycles) without access after assembly
- to operate in high vacuum (10^{-4} Pa) and at cryogenic temperature (4 K)
- to allow 5 years storage at normal ambient conditions.

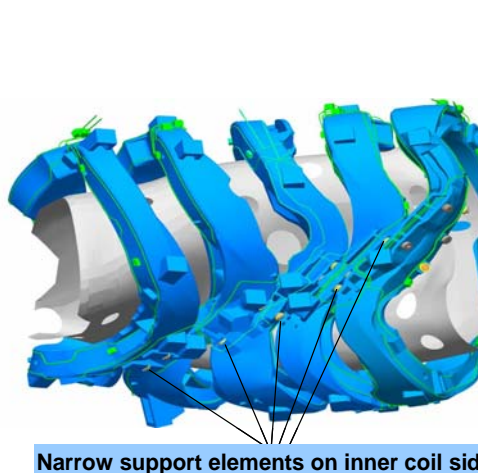


Figure 1. Segment of coil structure of W-7X showing the NSEs in between the coils

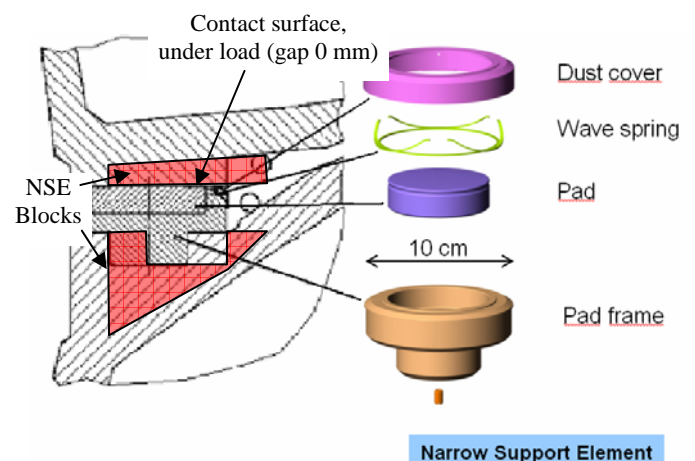


Figure 2. Cross section of two coils showing the NSEs in between under load

An intensive development program has been carried out on the narrow support elements. It was shown, that an additional anti friction coating is needed for the NSEs to assure a safe operation of the stellarator. Therefore MoS₂ coatings have been developed and tested at room and cryogenic temperature (77K). The influence of thickness on the coating endurance has been demonstrated. To prevent ageing both constructive methods and protective top coats of Cu, SiO₂, Au, Ti, and TiN have been investigated.

The atomic sputter deposition technique allows the deposition of dense coatings of several μm in thickness. Reduced and full-scale tribological tests up to 4000 cycles at liquid nitrogen temperature, ageing tests and arrangements to protect the coatings from humidity and oxidation have been carried on. After deposition and after testing/exposure the coatings have been characterized in detail in order to obtain information for the industrial production of the coatings.

2. Materials Selection

2.1 Narrow support elements

Al-bronze alloy has been selected as the pad material. It remains ductile and becomes stronger when deformed even at cryogenic condition. It has reasonable sliding properties, but not sufficient to avoid the stick-slip effect. Therefore all sliding surfaces have to be coated with a lubricant which is suited for cryo-vacuum and will survive during 5 years of assembly and 20 years of operation without maintenance.

2.2 Lubricant coating

MoS₂ is widely used under vacuum operating conditions in space industry and has shown the best behaviour during tests among all candidates. Thin films of molybdenum disulfide, produced by sputtering, yield exceptionally low friction in vacuum and at cryogenic temperature. Molybdenum disulfide has a layered crystal structure. Every layer consists of two sulphur planes with a molybdenum plane in between. Atoms are ordered hexagonally by ionic and covalent bondings in the triple layer. As in the in-plane carbon layers of graphite, there is no effective chemical bonds between the two sulphur layers, but only Van der Waals forces. The generally accepted lubrication mechanism for MoS₂ thin films is the movement of crystallites over one another through the sulphur layers, with friction determined by the interactive forces between the basal planes and wear by the possible loss of material during sliding.

Oxidation of molybdenum disulfide influences both, the friction coefficient and the lifetime of the coatings. Two ways for oxygen intake into MoS₂ have been described. Each one corresponds to different effects on the performance of MoS₂ as a lubricant [3]. The first mechanism takes place during the production of the coating. The process is very sensitive to water impurities that are always present in conventional coating devices, with base pressures in the range of 10^{-4} Pa. In this case a portion of sulphur will be substituted by oxygen and the ratio S/Mo will be lower than the theoretical value 2, depending on quality of vacuum in the device chamber. The presence of oxygen disturbs the planar structure of the sulphur layers, so that the coefficient of friction will be influenced.

The second mechanism plays a role during storing and use of MoS₂ anti friction films. The effects of air humidity on coefficient of friction and life time of MoS₂ coatings were investigated by D. Yu [4]. During tests in moist air a performance reduction was evident,

due to the local high temperature during the tribological load that leads to a greater oxidation rate. A drastic reduction in performance was also seen by storage in low relative humidity. The reaction of oxygen is more efficient at the layer edges, where molybdenum is exposed and can be attacked directly. On the contrary the sulphur planes are very stable against oxidation. This means that oxidation depends on dimension and orientation of crystallites. Large crystallites with smaller surface to volume ratio are preferable. Free grains, obtained for instance by spray techniques, are particularly sensitive in respect to oxidation. PVD processes offer the possibility to deposit compact layers to reduce the surface accessible to oxygen. Additives can be used in order to improve the structure of the films [4] or to make them chemically inert [5]. A further improvement consists in forcing the layer of MoS₂ to lie parallel to the surface. This was achieved by the production of intermediate layers during a complicated process with temperatures over 800 °C [5]. Nevertheless a columnar structure has to be avoided because of its high porosity and the risk of cracking during a load cycle.

A completely different method for the reduction of oxidation of MoS₂ layers is to produce an additional oxidation-resistant protective film. This system can exercise its function only during the storage and can be destroyed during the first tribological load. As an alternative a bonded film would be applicable, that consists of MoS₂ embedded in synthetic resin or silicate into a matrix and protected against oxidation during storage and use. These lubricating bonded films are used particularly in air and space travel and are applicable according to manufacturer data down to -200 °C. However the lifetime of these films is limited compared with sputtered layers [3].

3. Coating production and humidity protection

3.1 Coatings by Magnetron Sputtering

Sputtering deposition has been performed with a Denton Discovery 18 device equipped with three 3" magnetrons, which can be used in DC or RF mode. The target diameter was 75 mm, the distance between centre of target to centre of substrate was 120 mm. The rotating substrate stage had a diameter of 150 mm. The base pressure of the chamber was 3·10⁻⁵ Pa. Before sputtering, RF etching has been performed for substrate cleaning. The deposition parameters are given in table 1.

Target	Gas	Pressure [Pa]	Power [W]
MoS ₂ , 99%	Ar	0.4	250 RF
Cu, 99%	Ar	0.4	300 DC
Au	Ar	0.4	20 DC
Ti, 99.6%	Ar	0.9	100 RF
Ti, 99.6%	Ar/N ₂	0.9 / 0.4	100 RF
Si, 99.999%	Ar/O ₂	0.3 / 0.2	300 RF

Table1: Magnetron deposition conditions

MoS₂ coatings with variable thickness up to 7 μm have been deposited on Al-bronze pads.

The MoS₂ anti-friction coating has been deposited without distinct columnar structure which is shown on the SEM cross section of a MoS₂ coating on silicon substrate (figure 3).

The layer composition was determined by Rutherford Backscattering with ⁴He ions of 4 MeV. It was calculated to be 65 at.% S and 35 at.% Mo, thus nearly stoichiometric.

Figure 4 shows a X-ray diffraction patterns of a MoS₂ film (d = 5 μm) on Al-bronze and of the uncoated Al-bronze. The film shows broadened maxima at the 002, 101 and 110 reflections. Using the Scherrer formula the broadening of the 110 reflection was used to estimate the crystallite size. A linear dimension in the order of 10 to 20 basal planes was roughly calculated. The value and also the XRD pattern itself corresponds well to the calculated patterns of G. Weise et al. [8].

Measurements of the 100 and 110 pole figures showed constant integral intensity which means there is a random orientation of the structural units without any texture. The lifetime of such nano-dispersive structure should be higher compared to layers containing MoS₂ columns.

The density of the deposited MoS₂ was calculated to be 4.5 g/cm³ using weight and volume of the layer. This is around 90 % of the theoretical value of 4.8 g/cm³.

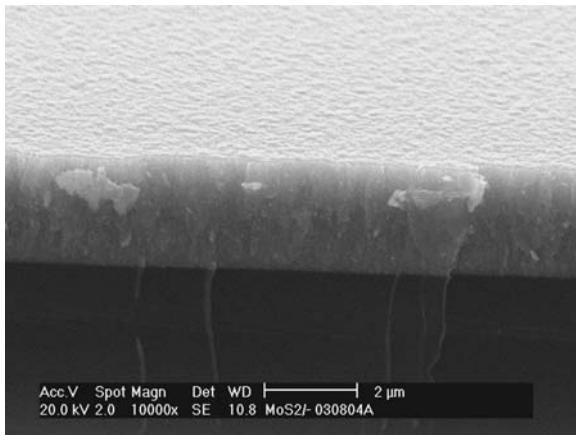


Figure 3. SEM image showing the non-columnar structure of the MoS₂ coating

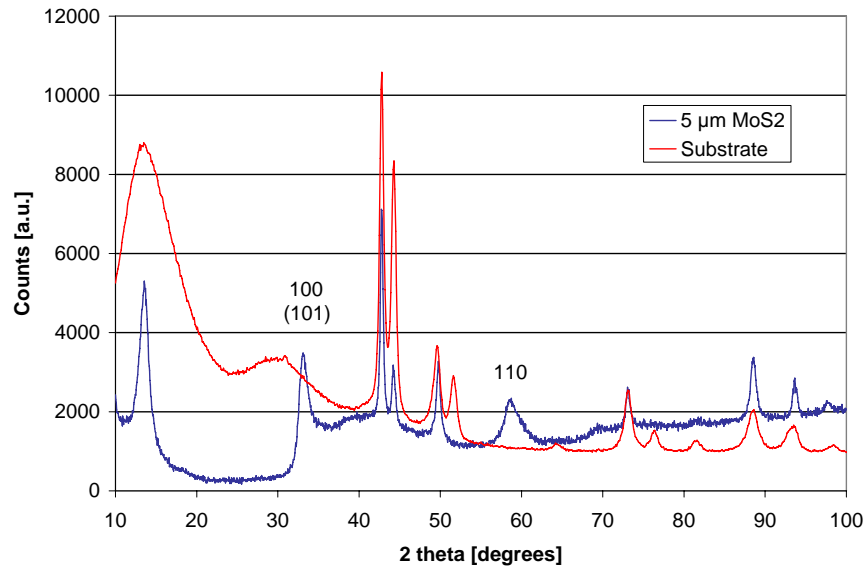


Figure 4. XRD analysis of MoS₂ coating and of Al-bronze

3.2 Humidity protection

To protect the MoS₂ coating against humidity during the 5 years of assembling of W-7X, thin top coats as diffusion barrier for water vapour have been tested. The top coat should have no negative influence to the anti friction properties of the MoS₂ basis layer. It means it should crack and be removed during the first load cycle of the NSE. At this time the MoS₂ coating will be always in vacuum conditions and do not need any further protection against humidity. Thin top coats of Cu, SiO₂, Au, Ti and TiN have been applied for humidity protection. An RF-bias of -50 V was used only for the reactive deposition of the top-coatings TiN and SiO₂. Very thin layers of brittle materials are preferred. The applicability of a material for a protective layer was tested by wear tests at high loads of a pad. If the pad showed comparable properties to systems without top coat, ageing tests at 100 % humidity at room temperature were performed.

Table 2 and figure 5 give an overview about the tested protection coatings.

uncoated substrate	strong oxidation was observed after 30 days
MoS ₂ / Cu: 5 μm/500 nm	Cu caused strong stick-slip effects
MoS ₂ /SiO ₂ : 5 μm/50 nm	oxidation at whole surface after 44 days
Au / MoS ₂ / Au: 10 nm/5 μm/50 nm	oxidation starting from the edges (fig. 8), after 48 days significant; weight gain 4.0 %, relating to coating system
MoS ₂ / Ti / TiN: 5 μm/15nm/50 nm	no visible oxidation, weight gain after 48 days 3.8 %, relating to coating system

Table2: Results of ageing tests with different protection coatings

Under given conditions none of the tested systems was stable against a relative humidity of 100 %. Supposably water adsorption was the reason for the mass gain of the samples. It was partially reversible, which was shown by evaporation under vacuum conditions, but 60 % of the weight gain was permanent.

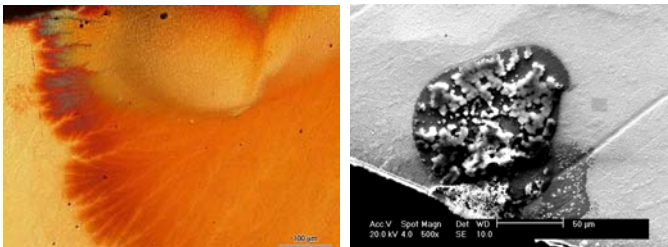
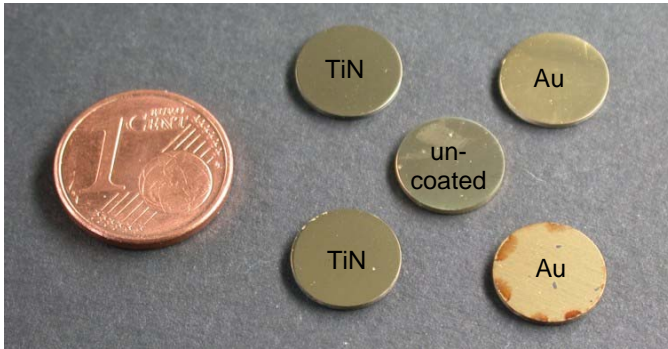


Figure 5. Ageing test: 100% humidity, 41 days. Detailed views: Au top coat

Simultaneously to the humidity protection by coatings the following constructive solutions were investigated:

- Since MoS_2 is relatively stable during storage at humidity below 50 % the whole assembling hall of W-7X could be hold at dry atmosphere.
- Also a constant flow of dry flushing gas inside the NSE volume via small copper pipes brazed to the dust covers of the NSEs would be possible.

4 Wear tests

In order to validate the designs and to optimize the roughness and lubricants of the sliding surfaces several test campaigns have been performed.

4.1 Pin on disk tests

Pin on disk tests of the coatings were performed by ESTL, UK using a ball diameter of 7.14 mm, load 20 N, Room Temperature, N_2 -gas environment, track radius 11.5 mm, 500 rpm. Outstanding performances could be reached, with no failure in the wear test within 2.8 million revolutions and a film wear rate of $8 \cdot 10^{-7}$ microns/rev.

4.2 Tests at room temperature (RT)

First screening tests were done at IABG in Munich with a facility able to apply simultaneously 1.5 MN of compression, 2 mm of sliding and 0.5° of tilting (Fig 6). The tests had the objective to investigate different pad materials, pad shapes and lubricants. To simulate the lifetime of W7-X, 500 cycles were performed using a compression force of 150 kN (conditioning phase), 3200 cycles at 1 MN and 400 cycles at 1.5 MN.

The lifetime of the MoS_2 coatings was increasing with the coating thickness. Layers of $1\ \mu\text{m}$ showed a low number of load cycles before the presence of stick-slip effects. Best results have been found for $6\ \mu\text{m}$ coatings on pads of a surface roughness $R_z = 2.5\ \mu\text{m}$. Stick-slip occurred at around 3800 cycles, the coefficient of friction was < 0.25 . Figure 8a shows the pad after the friction test.

Often it is described, that the lifetime of MoS_2 coatings will not increase with increasing thickness. But Müller et. al. [6] have shown, that MoS_2 films without columnar structure can be used effectively with thicknesses of several microns to ensure a long lifetime.

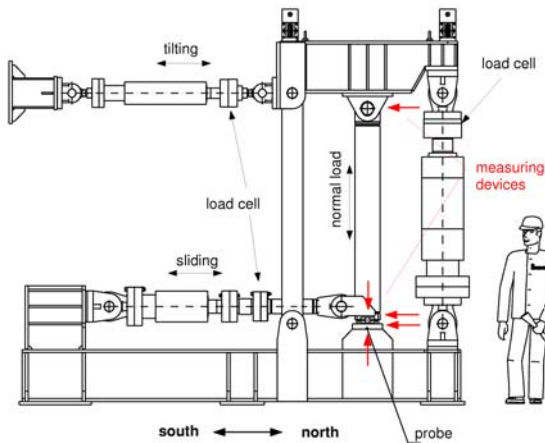


Figure 6. Schematic view of IABG test facility for the room temperature test

4.3 Tests under vacuum at 80 K

Further tests were performed at the KRP in Garching under vacuum and at 80 K. The facility uses a big tensile test machine and can apply 1.7 MN compression force, 5 mm sliding but no tilting (Fig 7). The main results compared to RT tests were a lower coefficient of friction (as low as 0.02) and a higher sensitivity to stick-slip.

Significant improvement has been obtained by polishing the sliding surfaces ($R_z < 1\ \mu\text{m}$) and by additional MoS_2 powder burnished onto the counter-sides.

The final pad design yielded more than 4300 cycles (3200 at 1 MN, 600 at 1.5 MN, 200 at 1.7 MN) without stick-slip and at a very low coefficient of friction $\mu < 0.08$. Figure 8b shows the pad after the friction test.

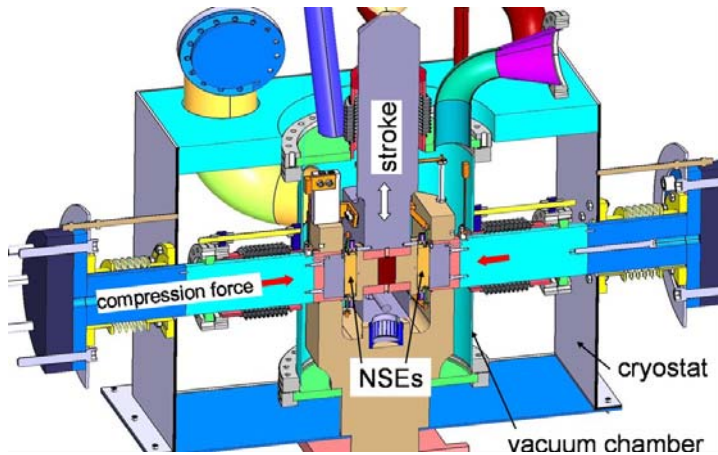


Figure 7. Test device at KRP, Garching, compressing two NSEs simultaneously against a vertical stroke piston

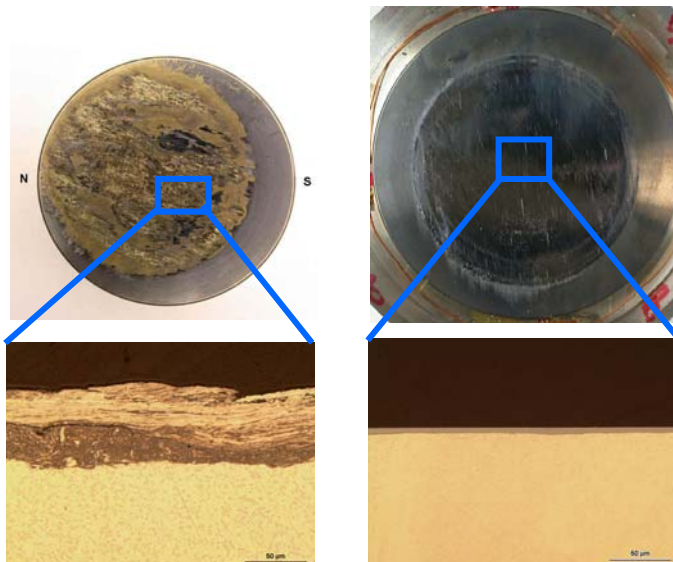


Figure 8. a): after test at RT in air, with Al-bronze 2.0978, material surface eroded and mixed with MoS₂. b): after test at 80 K in vacuum, with Al-bronze 2.0966, material surface still ok

5 Conclusion

The Narrow Support Elements between the inner sides of the non planar coils have been designed as flexible contact elements. They have to allow sliding up to 5 mm and tilting up to 1° under 1.5 MN compression force.

MoS₂ anti-friction coating without distinct columnar structure has been developed using magnetron sputtering to optimize the sliding behaviour, showing that the lifetime of the lubricant increases with increasing film thickness.

Several test campaigns at room temperature and in cryo-vacuum have been performed to validate the design. More than 4300 cycles at loads above 1.0 MN have been carried out in cryo-vacuum. This fulfils the lifetime requirements for WENDELSTEIN 7-X.

The best results were shown by pads with a very low roughness of the sliding surfaces ($R_z < 0.7$) and with MoS₂ as lubricant.

Application of top coats of 15 nm Ti and 50 nm TiN is the most promising humidity protection for long time storage. But it shows mass increase during ageing.

Therefore the humidity in the W7-X assembling hall will be controlled to be below 50 %.

NSEs for assembly of W7-X will be provided using in-house deposition devices.

References

- [1] M. Wanner et al., "Status of WENDELSTEIN 7-X Construction", Nuclear Fusion 43 (2003), pp. 1-9
- [2] M. Gasparotto et al., "The WENDELSTEIN 7-X Mechanical Structure Support Elements: Design and Tests", Proc. of the 23rd symposium on Fusion Technology (Venice 2004), to appear
- [3] P. Fleischauer, J. Lince, "A comparison of oxidation and oxygen substitution in MoS₂ solid film lubricants", Tribology International 32 (1999), pp. 627-636
- [4] De-Yang Yu, Jun-An Wang, Jin-Lin Ou Yang, "Variation of properties of the MoS₂-LaF₃ cosputtered and MoS₂-sputtered films after storage in moist air", Thin Solid Films 293 (1997), pp. 1-5.
- [5] S.R. Cohen et al., "The tribological behavior of type II textured MX₂ (M = Mo, W; X = S, Se) films", Thin Solid Films 324 (1998), pp. 190-197.
- [6] C. Müller, C. Menoud et al. "Thick compact MoS₂ Coatings", Surface and coatings technology 36 (1988), pp. 351-35
- [7] V. Buck "Microanalysis and modelling of tribological coatings", Surface and coatings technology 57 (1993), pp. 163-168
- [8] G. Weise, et al. "Preparation, structure and Properties of MoS_x films", Thin Solid Films 298 (1997), pp. 98-106