

# MoS<sub>2</sub>-Metal Composite Layers

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## 1. Introduction

There is an ongoing industrial need to move to lighter weight, cheaper and more easily-machined bulk materials. However the mechanical and tribological properties of the presently-used materials must be kept, or better still, improved. Most frequently, the property of primary importance to be retained or increased is wear resistance. Additionally, in certain high-precision and contamination-critical applications, it would be attractive to add self-lubricating properties to the tribo-system.

Since no single surface treatment or coating can fulfill all of the requirements in all of the applications and on all of the bulk materials desired, a series of multilayer coatings are being developed. With a single multilayer system, or by combining different multilayer systems, the needed properties may be attained.

Figure 1 gives examples of how different layers and surface treatments could be assembled. Each layer may be itself a compound film or multilayer, and the counterpiece could be treated in the same way. The 3<sup>rd</sup> body in the tribocontact also presents the possibility to be an actively engineered material (e.g. oil with multiple additives). The work reported here covers one member of such a series of coatings.

## 2. Approach

Single layer films such as Cr or TiN have been described<sup>1</sup> as 1<sup>st</sup>

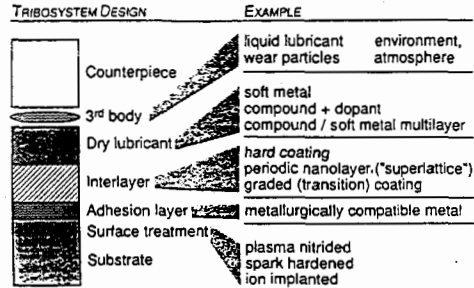


Figure 1: Diagram and examples of possible surface treatments and coating layers. The 3<sup>rd</sup> body can also be complex and the counterpiece can be similarly treated.

Generation coatings. combinations of 2 or 3 layers, for example TiC/TiN, and ternary compound coatings like TiAlN are considered to be 2<sup>nd</sup> Generation coatings. Multilayer and multi-component coatings are denoted as 3<sup>rd</sup> Generation, encompassing a wide range of engineered films such as superlattices, nanocrystalline coatings, gradient layers, composites, etc.

Molybdenum disulfide, (MoS<sub>2</sub>) has been known and studied as a solid lubricant for more than 60 years<sup>2</sup>. It is of continuing interest because of its extremely low friction coefficient in vacuum or dry conditions. However it rapidly loses performance in terms of both friction coefficient and lifetime, in the presence of humidity.

A consideration of the wear modes of MoS<sub>2</sub>, particularly in humid environments, leads to several possible routes to improvement.

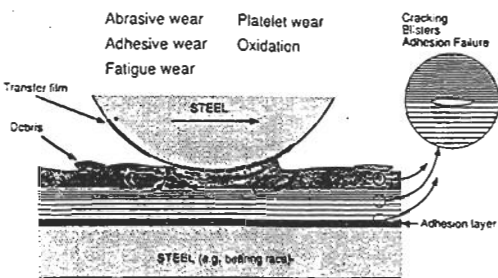


Figure 2: Schematic representation of the various wear modes observed in  $\text{MoS}_2$  thin films during rolling/sliding contacts in atmosphere.

$\text{MoS}_2$  in a typical rolling/sliding contact such as in a ball bearing (Fig. 2), displays all of the classical modes of abrasive, adhesive and fatigue or delamination wear<sup>3</sup>. In the presence of oxygen or humidity, oxidation plays an important role in the two latter cases. So-called platelet wear comes about from the reorientation, fracture and compacting typical of PVD  $\text{MoS}_2$  coatings under load. Blisters or voids have been shown to come about by both oxidation and localized stress concentrations.

Considering these wear mechanisms, there are several possible routes towards increasing  $\text{MoS}_2$  film lifetime in humid environments:

- Increase the grain size by using higher deposition temperatures or ion bombardment during the coating process.
- Reduce porosity in the film by producing a compact structure, e.g. by lower deposition pressures<sup>4</sup>, or using multilayers to force the film to renucleate before columnar structures develop.
- Neutralizing sites for oxygen or water attachment and blocking routes for diffusion by including metals such as Au or Pd.

- Adding materials that could preferentially oxidize or getter oxygen such as Ni, Pb or  $\text{Sb}_2\text{O}_3$ .

### 3. Experiments

Multilayers of  $\text{MoS}_2$  and Pb, Au or Ni have been prepared by magnetron sputtering using polished AISI 440C stainless steel substrates. Before coating, the substrates were ultrasonic cleaned in separate baths of cyclohexane, toluene and methanol.

Once inside the vacuum chamber, there was an additional Argon sputter etch. Adhesion layers of Cr or Ti were deposited, having a thickness of approximately 30 nm. This was followed by the lubricant film coating. The total solid lubricant thickness was kept at 300 nm, while a survey of different metals added to  $\text{MoS}_2$  was made. The thickness of individual  $\text{MoS}_2$  layers was on the order of 30 nm and the metal layers were in the range of 10 to 20 nm each.

Auger electron spectroscopy (AES) depth profiling and Rutherford backscattering (RBS) were used to analyze the film composition. Pin-on-disk tribometer tests were performed in both dry nitrogen and in 30% relative humidity (RH) air. Relatively severe sliding conditions of 10 cm/s and 1.1 GPa maximum Hertzian contact pressure were chosen to simulate the tribological conditions of the targeted industrial applications. The (stationary) ball was of 100Cr6 steel and had a diameter of 6 mm.

### 4. Preliminary Results

An AES profile is shown in Figure 3. The first 3 layers of Pb can be seen, but then the resolution is lost to cratering and ion mixing effects. Figure 4 shows an RBS spectrum for a multilayer made up of  $\text{MoS}_2$  and Au.

The first 4 Au layers can be clearly seen, along with several more that overlay the Mo signal.

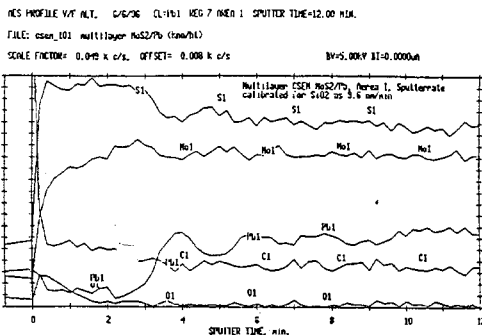


Figure 3: AES depth profile of an MoS<sub>2</sub>-Pb multilayer. Only the first 3 layers of Pb can be identified.

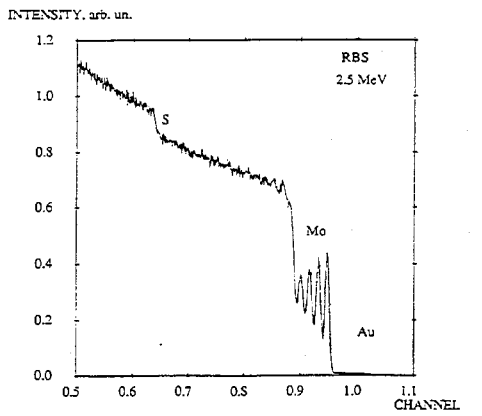


Figure 4: RBS spectrum of a MoS<sub>2</sub>-Au multilayer coating. The first 4 Au layers are clearly visible, with several more discernible on top of the Mo signal.

Table 1 gives relative sliding wear performances of various combinations of MoS<sub>2</sub> with different metals, and adhesion layers, with a standard, pure a pure MoS<sub>2</sub> coating directly on steel as a reference.

Multilayer	Relative Lifetime	$\mu$	Relative Lifetime
	0% RH	0% RH	30% RH
MoS <sub>2</sub> standard	1.0	0.005	0.3
MoS <sub>2</sub> + Cr adhesion	4.7	0.02	0.7
MoS <sub>2</sub> -Ni + Cr adhesion	10.9	0.02	0.7
MoS <sub>2</sub> -Pb + Ti adhesion	3.0	0.02	0.8
MoS <sub>2</sub> -Au + Ti adhesion	6.2	0.01	1.7

Table 1: Sliding lifetimes in 0% and 30% RH relative to pure MoS<sub>2</sub> on 440C stainless steel.

### 5. Next Steps

A detailed study of the run-in and wear mechanism of these film structures in humid air is underway. This may guide the optimization of metal percentage, which metal(s) to use, layer thicknesses, and whether co-deposited compound films instead of multilayers may be more promising.

### Acknowledgements

AES profiles and SEM photographs were made by L. Knoblauch and R. Hauert of EMPA, Dübendorf. RBS measurements and some of the coatings were made by S. Mikhailov, from the Institute of Physics at the University of Neuchâtel.

### References

- "Review of multicomponent and multilayer coatings for tribological applications" C. Subramanian and K.N. Strafford, *Wear*, 165 (1993) 85-95.
- "Molybdenum disulphide as a lubricant: a review of the fundamental knowledge" W.O. Winner, *Wear* 10 (1967) 422-452.
- "Wear behavior of triode-sputtered MoS<sub>2</sub> coatings in dry sliding contact with steel and ceramics", I.L. singer, S. Fayeulle, P.D. Ehni, *Wear* 195 (1966) 7-20.
- "Thick compact MoS<sub>2</sub> coatings" C. Müller, C. Menoud, M. Maillat, H.E. Hintermann, *surface & coatings Technology* 36 (1988) 351-359.