Università degli Studi di
Modena e Reggio Emilia –
Dip. Scienze Fisiche
Informatiche
Matematiche

CNRNANO MODENA S3

SUP&RMAN
SUPerfici e Ricoprimenti per la
Meccanica Avanzata e la Nanomeccanica

RF magnetron sputtering prepared MoS2 coatings: frictional and wear performances in different environments

Elisabetta Serpini(1,2), A. Rota(3), A. Balestrazzi(1), D. Marchetto(1,2,3),
E. Gualtieri(3), S. Valeri(1,2,3)

(1) Dipartimento di Scienze Fisiche, Informatiche e Matematiche- Università di Modena e Reggio Emilia, Via Campi 213/A – 41125 Modena, Italy

(2) Istituto CNR-NANO S3, Via Campi 213/A – 41125 Modena, Italy

(3) Centro Interdipartimentale per la Ricerca Applicata e i Servizi nella Meccanica Avanzata e nella Motoristica Intermech-Mo.Re., Università di Modena e Reggio Emilia, Via Vignolese 905/b-41125 Modena, Italy

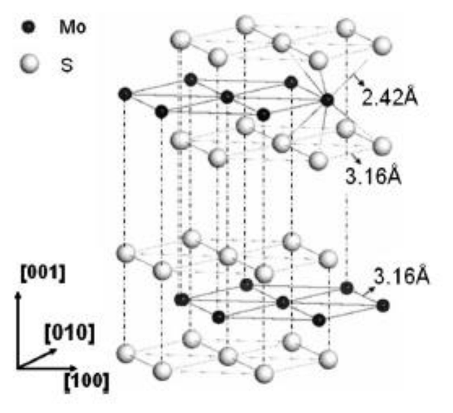
Introduction

MoS_2 is a <u>lamellar solid lubricant</u> formed by stacking S-Mo-S tri-layers weakly bonded by van der Waals forces.

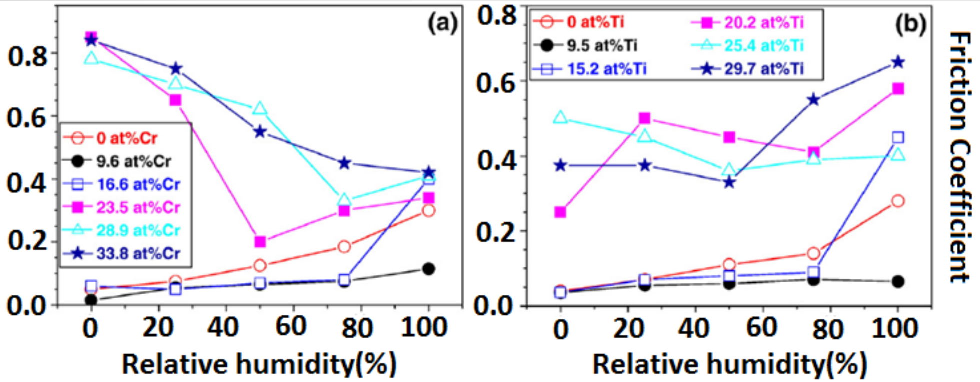
Optimal sliding conditions if:

- The **basal planes** (0001) of MoS_2 crystallites are parallel to the sliding direction.
- <u>Sliding occurs in **inert environment or vacuum conditions**</u>: **oxygen and water vapor introduce obstacles to easy shear between lamellae.**

→ <u>easy inter-lamellar shear</u> ONLY in aerospace conditions (COF < 10^{-2})



The most convenient way to use MoS_2 as a lubricant is in the form of a **<u>coating</u>**, in this way its lubricating <u>properties can be finely tuned</u>:

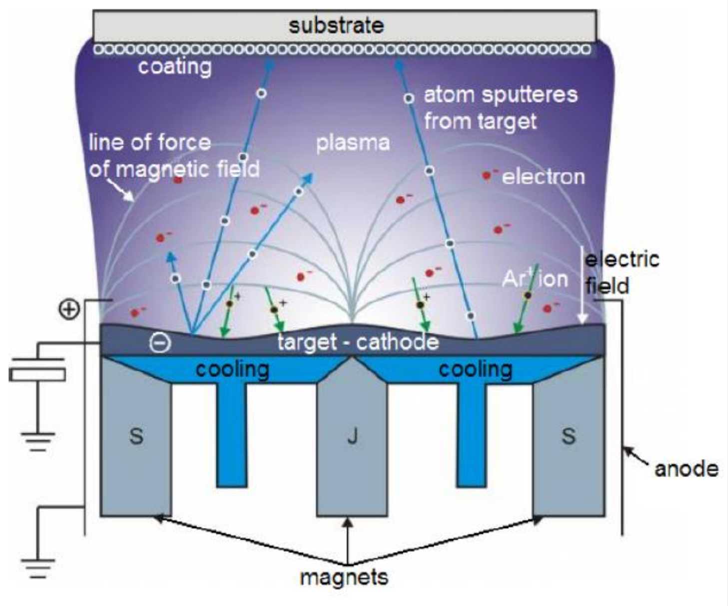


"Tribological properties of Cr- and Ti-doped MoS2 composite coatings under different humidity atmosphere», X. Ding Surf. Coat. Technol. 2010

PVD techniques: magnetron sputtering

Physical Vapor Depostion techniques allow a **fine tuning** of coating:

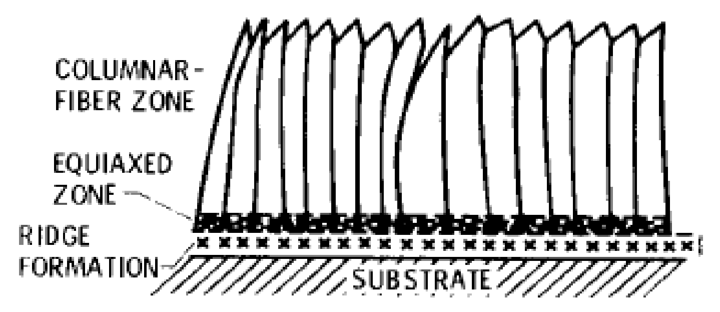
- mechanical (hardness, wear resistance, …),
- chemical (stoichiometry, doping, …),
- crystalline characteristics,
- substrate morphology replica.



RF Magnetron sputtering:
a **PVD** technique in which the plasma, generated by inert gas atoms ionization, is sustained and stabilized by a **B field**, which increases the sputtering rate. Target is pure MoS_2. RF impulses allow deposition of insulating species.

Morphology of MoS_2 PVD coatings (**3-zone model**):

- < 100 nm - dense and disordered zone
- < 200 nm - dense equiaxed zone
- > 200 nm - porous columnar zone



"A review of recent advances in solid film lubrication", T. Spalvins J. Vac. Sci&Technol A **5**, 212 (1987)

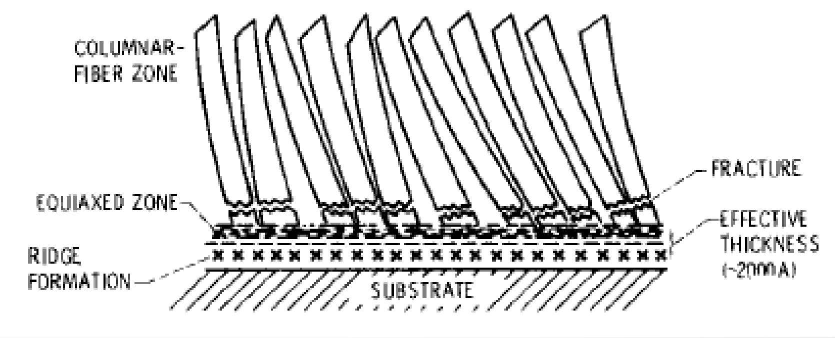
Coatings for MEMS applications

MEMS often require thin coatings of very controlled thickness: do thin films support lubrication well enough?

- **Thick films**: grow columnar-like → get burnished with sliding → basal planes in the correct position for easy shear (Fleischauer)

… but: columns may break and be extruded from wear track (Spalvins)

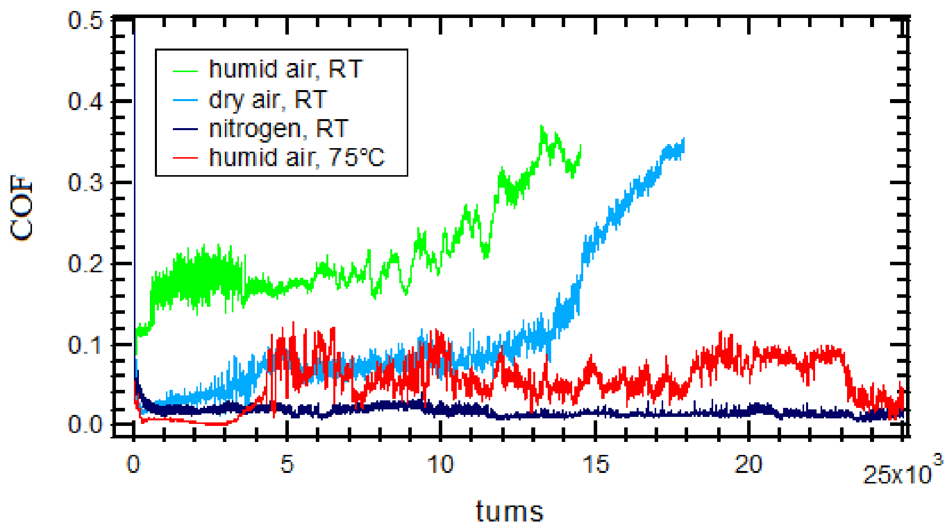
- **Thin films:** support lubrication if columns break?



"A review of recent advances in solid film lubrication", T. Spalvins J. Vac. Sci&Technol A **5**, 212 (1987)

Ball-on-disc friction tests on thin films (200 nm) deposited on Si (111) substrates without doping:

Counterpart: 4mm 100Cr6 steel (Ra = 30 μm)
Normal force: 0.4 N (Hertz contact pressure = 0.4 GPa)
Velocity: 0.1 m/s
Room Temperature → 75°C
Humid air/dry air/nitrogen/oxygen

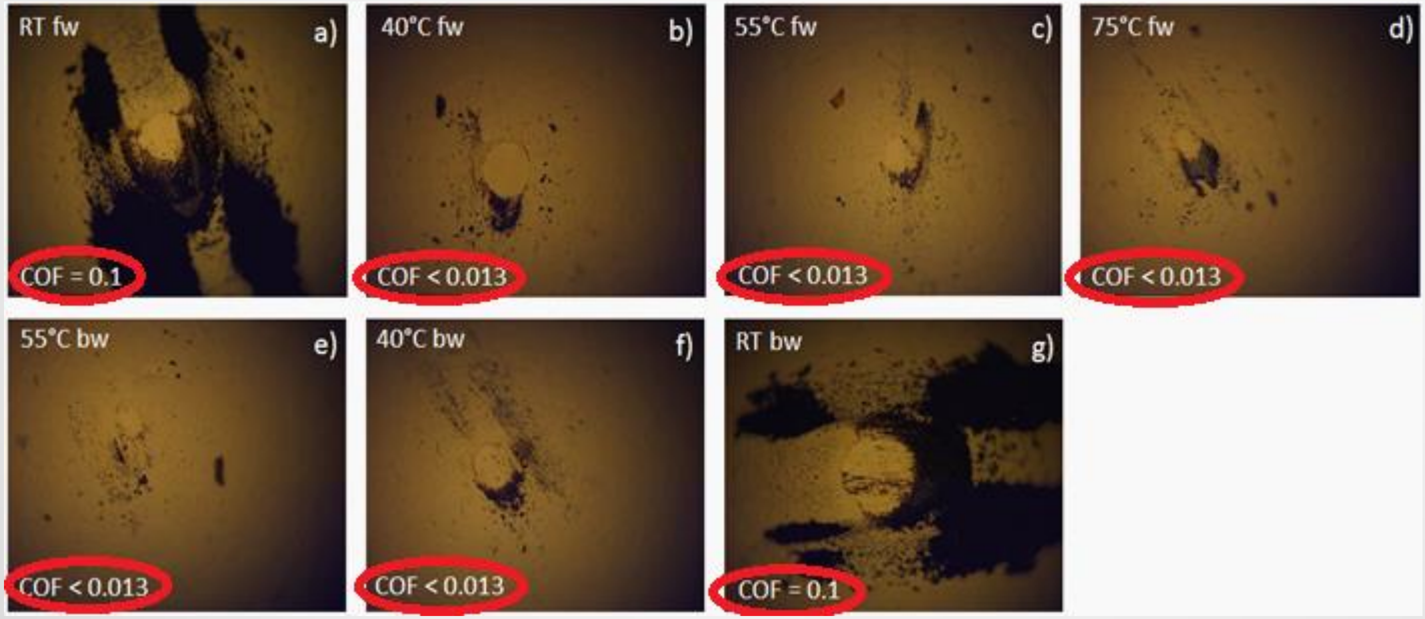


Compatible with results obtained for thick films!

"The role of humidity and oxygen onMoS2 thin films deposited by RF PVD magnetron sputterin", E. Serpini et sl , submitted

Heating to reduce friction (1)

Temperature ramp RT → 75°C → RT

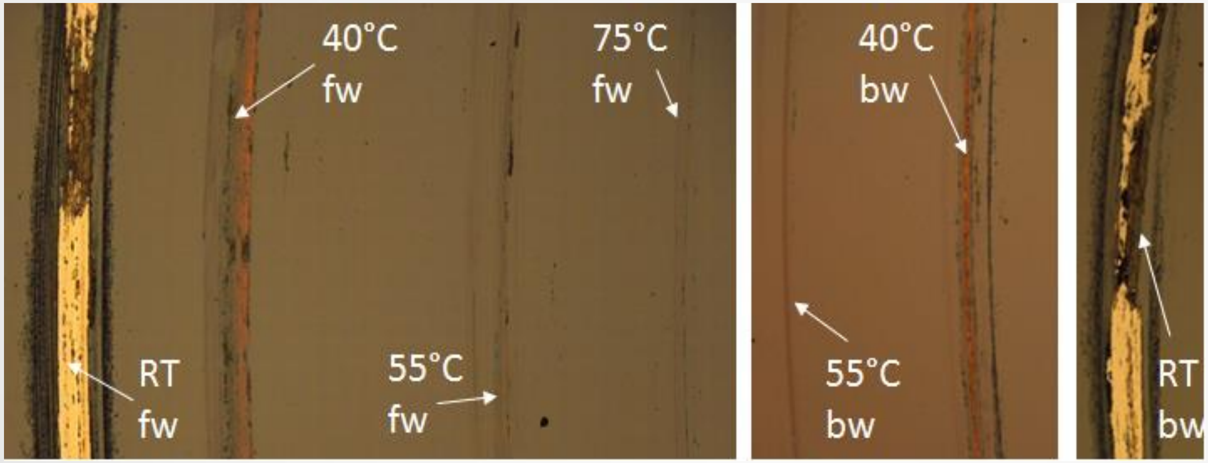


COF behavior:
- Decreases with T from 0.1 to 0.02
- No appreciable difference among Ts > 40°C

Counterpart appearance:
- Less material removed with increasing T
- Smaller wear scar with increasing T

“The role of humidity and oxygen onMoS2 thin films deposited by RF PVD magnetron sputterin”, E. Serpini et sl , submitted

Heating to reduce friction (2)



Track	O(%)	Mo(%)	S(%)
RT (fw)	13,8	26,9	52,4
40°C (fw)	9,5	26,8	58,3
55°C(fw)	8,7	26,6	61,4
75°C(fw)	8,7	27,7	58,6
55°C(bw)	8,9	27,8	56,8
40°C(bw)	12,8	27,1	48,7
RT (bw)	13,4	25,2	59,9

Wear track appearance:

- Less material removed with increasing T
- Thinner wear track with increasing T

Chemical analysis:

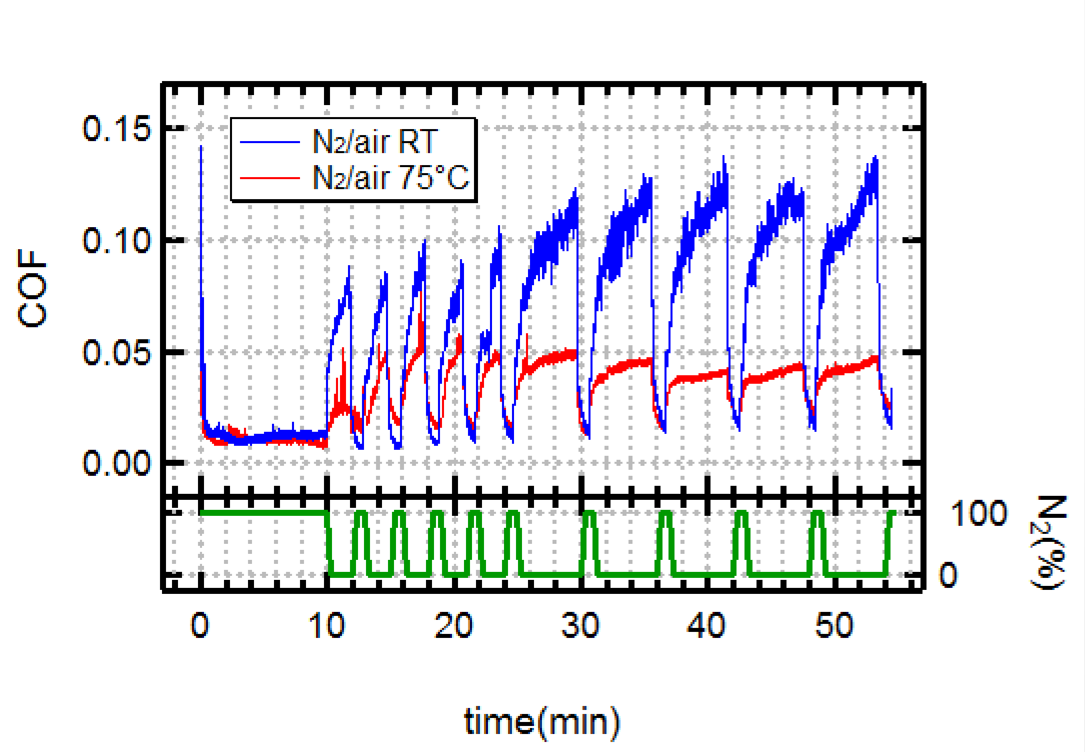
Auger analysis does NOT show appreciable differences in oxygen content (within the experimental error).

“The role of humidity and oxygen onMoS2 thin films deposited by RF PVD magnetron sputterin”, E. Serpini et sl , submitted

Inert environment to reduce friction: why does it work?

Pump and purge experiments:

- Initial stabilization in N_2
- Humid air IN (2’) – N_2 flux (1’) x5
- Humid air IN (5’) – N_2 flux (1’) x5
- Room T vs 75°C



“The role of humidity and oxygen onMoS2 thin films deposited by RF PVD magnetron sputterin”, E. Serpini et sl , submitted

→ extremely low COF in N_2, gradual growth when flux is suspended (humid air IN) – **REVERSIBLE**!

→COF is 3 times lower when substrate is heated (DESORPTION).

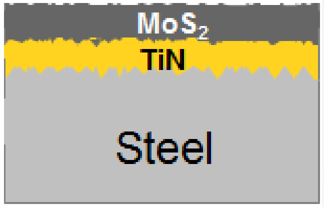
Below 100°C the <u>physisorbed</u> water drives the frictional behaviour of MoS_2.

Patterning to reduce friction

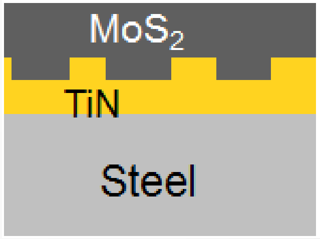
Random patterning (abrasive papers)

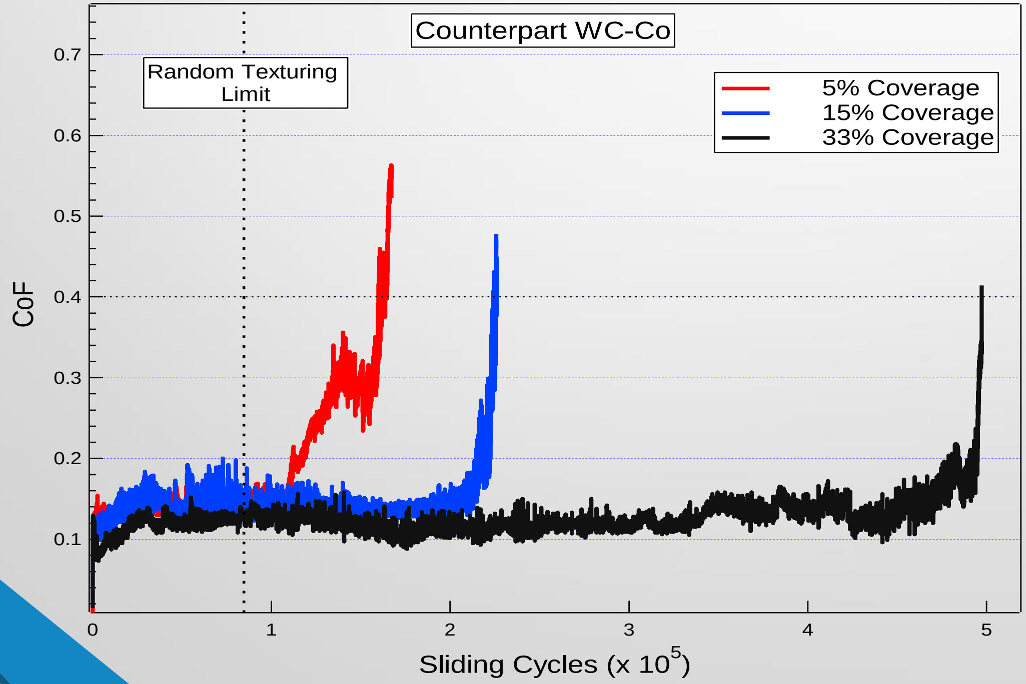
Regular patterning (laser texturing)

RMS < 1 µm



Dimples: depth=1 µm, diam=10 µm





Micro-scale regular texturing is more effective than the random sub-micro-scale one (both COF stability and lifetime)

Conclusions

MoS_2 is a solid lubricant which is usually employed as thick coatings in aerospace applications.

PVD techniques offer a variety of ways to tune the coatings' characteristics in terms of mechanical, chemical and crystalline properties.

We show that it is possible to achieve the same results in terms of friction reduction with thin quasi-amorphous coatings (<200 nm) as it was previously found for thick, crystalline films (> 1 µm).

To improve MoS_2 lubricant properties in humid environment, we show that heating the system up to only 40°C is sufficient, which can be useful for MEMS applications. Alternatively, the coating must be employed in dry environment: we show that below 100°C the physisorbed water drives the frictional behaviour of MoS_2.

Another way to improve MoS_2 CoF stability and lifetime is micro-scale regular texturing.

ACKNOWLEDGMENTS

People involved in the project:

Prof. S. Valeri
Dr. A. Rota
Dr. Diego Marchetto
A. Ballestrazzi
Dr. Enrico Gualtieri

This work has been supported by:

- Progetto FAR - FONDO DI ATENEO PER LA RICERCA ANNO 2014 - Meccanismi di attrito ed usura di superfici autolubrificanti alla multiscala - by Università di Modena e Reggio Emilia,
- **Progetto MetAGEAR** - Piattaforma integrata per la progettazione e la produzione avanzata di riduttori industriali, by **Centro Interdipartimentale INTERMECH – Unimore,**
- Progetto di Ricerca Applicata 2013/14 – Fondazione Cassa Risparmio Modena N. SIME: 2013.0650
- COST ACTION MP1303.

Author also acknowledges **Tecnopolo INTERMECH**, High-Technology Network of Regione Emilia Romagna (Italy).