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#### Title

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Preparation by pulsed vacuum arc deposition and characterization of DLC/MoS<sub>2</sub> nanocomposite thin films

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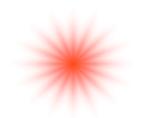


### Outline

- Why DLC + MoS<sub>2</sub> nanocomposites
- Set-ups
- Thermodynamics
- C + Mo + CS<sub>2</sub>: bonding
- Mo + S:
  - Bonding and structure
  - Ball-on-disk tests under air and dry nitrogen
- Mo + S + C:
  - Bonding and structure
  - Ball-on-disk tests under air and dry nitrogen
- Comparison with other systems and conclusions



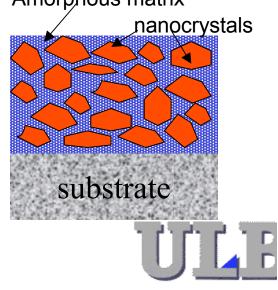




# Why DLC + MoS<sub>2</sub> nanocomposites

- Lubrication performances of non-hydrogenated DLC are limited in absence of moisture but good hardness and thoughness
- $MoS_2 = good lubricant under dry conditions or under vacuum but low hardness, and oxydation in presence of O<sub>2</sub> or H<sub>2</sub>O Amorphous matrix$

=> idea: incorporate clusters of MoS<sub>2</sub> in a matrix of DLC to get a film with good performances under wet and dry conditions.

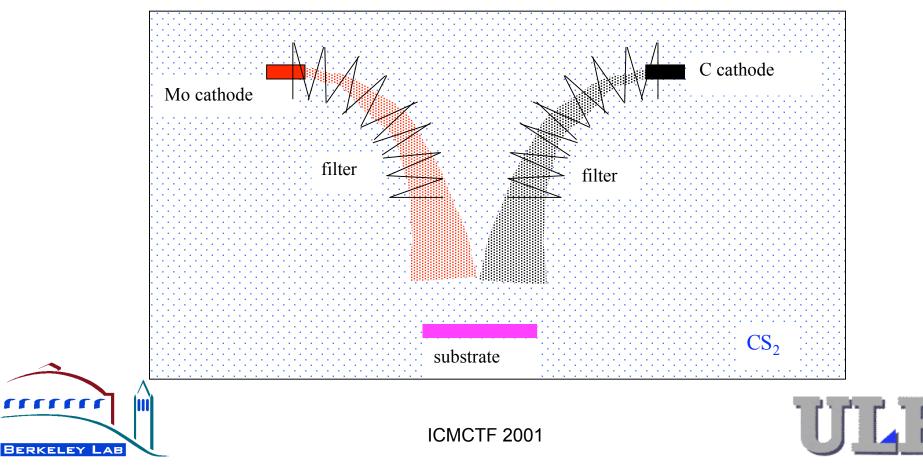






### Set-up: gaseous source of Sulfur

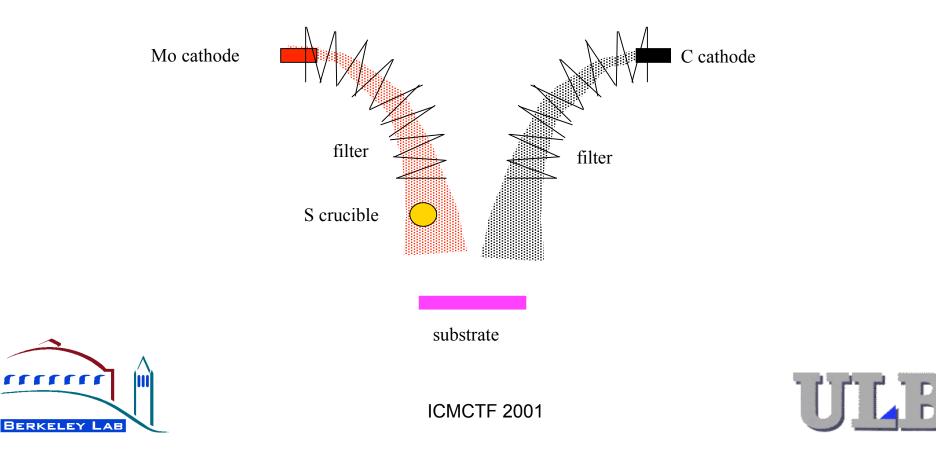
• Gaz source for  $S = CS_2$ 





### Set-up : solid source of Sulfur

• Evaporation of solid sulfur in a crucible





### Thermodynamics

CompoundEnthalpy of formation (at 298 K)MoS <sup>2</sup> solid-276.010 k		







#### In absence of S source

- C pulse 4 ms at 270A, Mo pulse 6 ms at 100A
- => formation of molybdenum carbide



ULF



## Films prepared with CS<sub>2</sub>

- C pulse 4 ms at 270A, Mo pulse 6 ms at 100A
   1 Pa of CS<sub>2</sub>
- => very low incorporation of sulfur
  - difficulty to control the Mo/C and S/C ratios
    -sensitivity to contamination







### Mo + S synthesis: bonding

- sulfur is incorporated exclusively if Mo is present
- a bias on the substrate reduces the incorporation of sulfur in the film. Two factors:
  - preferential sputtering of sulfur under Mo<sup>n+</sup> ion bombardment
  - the bias attracts the Mo<sup>n+</sup> ions but as the ionization of sulfur is low, the bias is not attracting the sulfur
- the structure is not well ordered: wide peaks in XPS





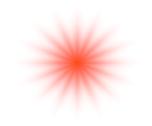


# MoS<sub>2</sub> synthesis: mechanical properties

- Ball-on-disk tests in dry  $N_2$ ,  $AI_2O_3$  ball
  - life time increased by a factor of 4 in comparison with air tests
  - failure by delamination and intensive cracking
- => very brittle films
- => not satisfactory as lubricants even under dry N<sub>2</sub>







### Mo + S + C: bonding

- S bonded mainly to Mo.
- No real proof of C-S bonds even in presence of an excess of S
- possible S-S bonds in presence of an excess of S
- in presence of an excess of Mo formation of MoO<sub>3</sub> at the surface and tendency to form Mo-C bonds in the bulk
- reduction of S incorporation in presence of bias







### Mo + S + C: mechanical properties

- Ball-on-disk tests with stainless steel and alumina balls in air
- Ball-on -disk tests with alumina balls in dry N<sub>2</sub>
- => best resistance to abrasion in air and dry N<sub>2</sub> for low concentration of MoS<sub>2</sub> (doping).
- => at low concentration of MoS<sub>2</sub> (doping), better resistance to abrasion with a steel ball than with Al<sub>2</sub>O<sub>3</sub> ball
- => performances are lower in air than pure DLC films
- => films with high MoS<sub>2</sub> concentration are brittle and don 't support the load: extensive cracking and delamination

NO improvement of the tribological performances of DLC by the addition of  $MoS_2$  in the deposition conditions investigated here.







# Comparison with other systems and conclusions

- The MoS<sub>2</sub> / DLC system has been compared with W doped DLC and Ti doped DLC films prepared in similar conditions:
- => W and Ti additions reduce the intrinsic compressive stress of the hard DLC films and made them thougher.

=> addition of MoS<sub>2</sub> does not improve the performances of doped DLC films, it increases the brittleness: could be related to the bonding in the films.



