Capteurs micromécaniques : les couplages mécano-chimiques à l'oeil des mesures de champs

F. Amiot [1] F. Hild [2] J. P. Roger [3] F. Kanoufi [4]

[1]FEMTO-ST Institute, CNRS-UMR 6174 / UFC / ENSMM / UTBM

[2]LMT Cachan, ENS Cachan / CNRS-UMR 8535 / U. Paris 6 / PRES UniverSud Paris

[3]Langevin Institute, CNRS UMR 7587 / ESPCI ParisTech

[4]PECSA, CNRS-UMR 7195 / ESPCI ParisTech

Dynamic operation : TNT adsorption

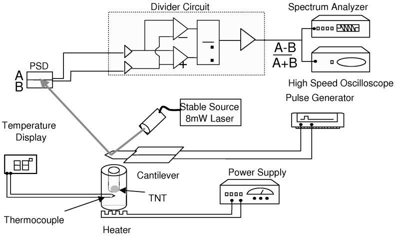


Fig. 1. Schematic of the setup used for the experimental measurements.

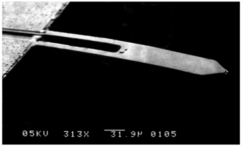


Fig. 2. SEM image of a typical piezoresistive cantilever used for the experiments.

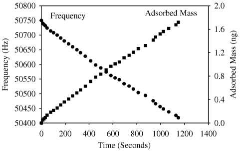
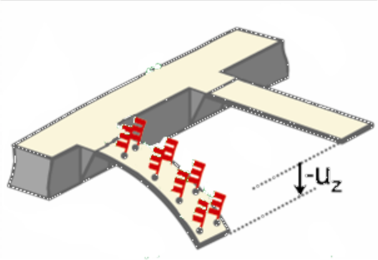


Fig. 3. Variation in the resonant frequency of the microcantilever and the adsorbed mass as a function of time during adsorption of TNT.

Initial mass : 130 ng, resolution $\simeq$ pg

Muralidharan *et al.*(2003)

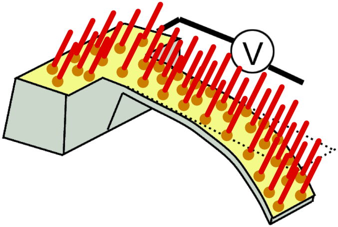
Quasi-static operation



- (alkane-)Thiol adsorption
- DNA : Fritz *et al.*(2001)
- Proteins (PSA) : Arntz *et al.*(2003)
- Reproducibility issue
- Drifts

Problem :
Improve the specificity and reproducibility

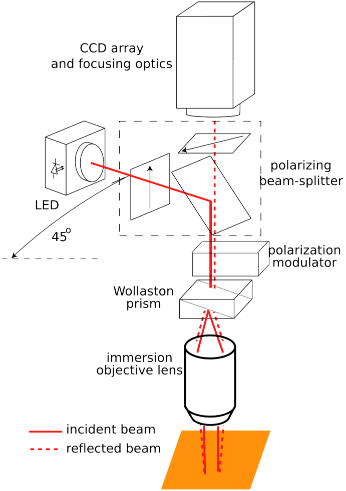
Electrochemically-actuated cantilevers



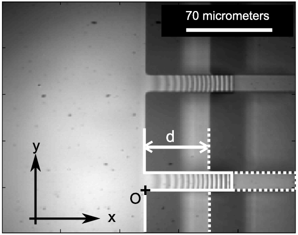
- Microcantilever AND Microelectrode
- To improve detection's specificity
- Query mechanism
- Tabard-Cossa *et al.*(2005,2007)
  Tian *et al.*(2005)
  Amiot *et al.*(2007)
  Fredlein and Bockris (1974)

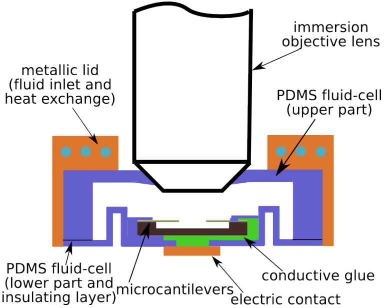
GOAL :
Investigate the way the coupling is altered upon adsorption

Full-field measurements



[A and Roger, Appl. Opt. (2006)]



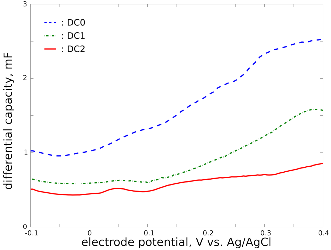


Electrochemical procedure

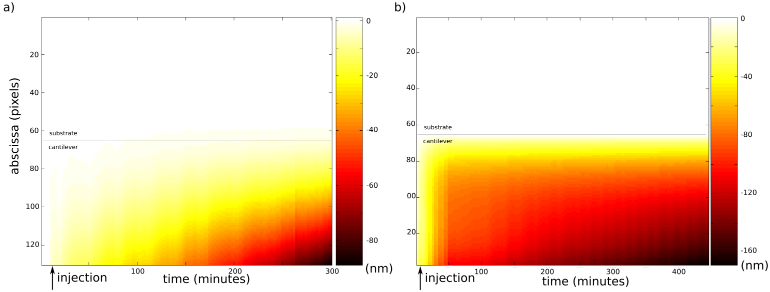
Outline
Cantilever-based sensors
Electrochemically-actuated cantilevers
Experimental
Results
Conclusion

$70 \times 20 \times 0.84\mu\mathrm{m}^3$ $SiO_2/Ti/Au$ microcantilevers in KCl 10^{-2}M.
Potential range : $[-0.1 \ldots 0.4]$ V vs. Ag/AgCl.

1. Electrochemical activation
2. Impedance measurement (DC0): 25mV pp. @ 20 Hz
3. Cyclic Voltammetry (CV0): 3 cycles, 2mV/s, 61 images.
4. Decanethiol adsorption : $7 \times 10^{-5}\mathrm{M}$, 5h, 1 image/ 2min.
5. Impedance measurement (DC1): 25mV pp. @ 20 Hz
6. Cyclic Voltammetry (CV1): 3 cycles, 2mV/s, 61 images.
7. Decanethiol adsorption : $7 \times 10^{-5}\mathrm{M}$, 7h30, 1 image/ 3min.
8. Impedance measurement (DC2): 25mV pp. @ 20 Hz
9. Cyclic Voltammetry (CV2): 3 cycles, 2mV/s, 61 images.

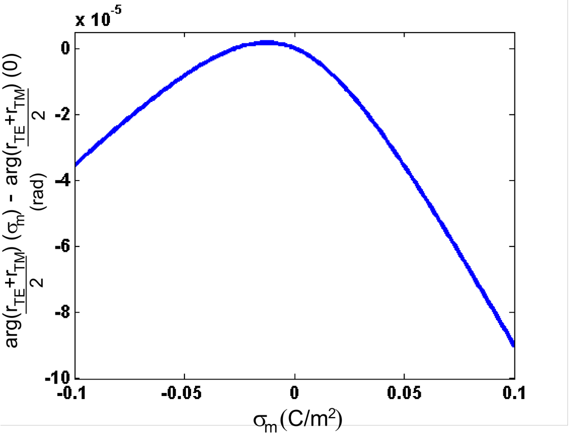


- Minimum differential capacity @ -0.05 V vs. Ag/AgCl
- Estimated coverage ratio: 0.71 for DC1
- Decanethiol layer capacitance: 0.8 $\mu\mathrm{F}/\mathrm{cm}^2$



Heterogeneous chemical reaction [A *et al.*, J. Phys. D (2007)] :

$$\phi_{mes} = \phi_{mec} + \phi_{ec}$$



Heterogeneous chemical reaction [A *et al.*, J. Phys. D (2007)] :

$$\phi_{mes} = \phi_{mec} + \phi_{ec}$$

to be decoupled...

$$\sigma_m(x, Q) = D(x) \times Q$$

$$D(x) = \frac{c_q(x_0)}{(x_0 - x)^p}$$

$$\tau = \sum_{t=1}^{T} \delta_t \sigma_m^t$$

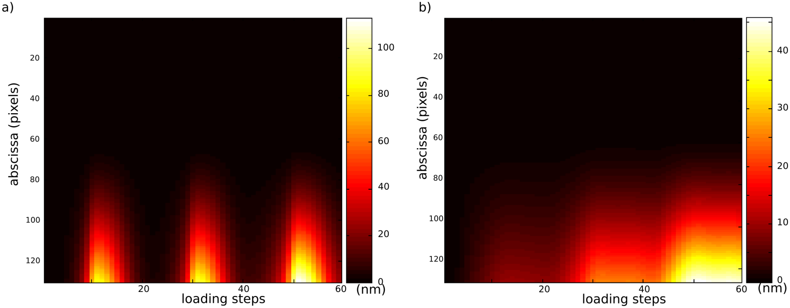
- Charge localization function
- Local coupling relation (stress-charge function)
- ϑ : mixing ratio
- To be identified

Objective function :

$$\eta^2 = \frac{1}{N_p} \sum_{x,y,n} \frac{1}{\mu(x,y,n)^2} (\phi_{test}(x,y,n) - \phi_{test}(x,y,n-1) - \phi_{mes}(x,y,n) + \phi_{mes}(x,y,n-1))^2$$

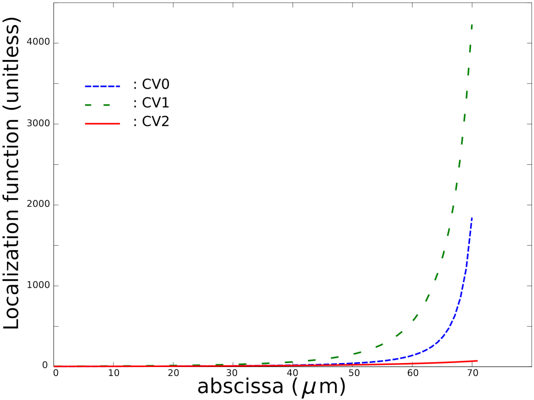
Electrochemically-induced cantilever bending

CV test	CV0	CV1	CV2
ϑ	7×10^{-2}	9×10^{-2}	$\sharp$
Γ_{ϑ}	0.32	0.19	10^{7}
residual η^2_{min}	6.3×10^{-2}	5.8×10^{-2}	8.5×10^{-2}



CV0 Max displ. amplitude : 100 nm

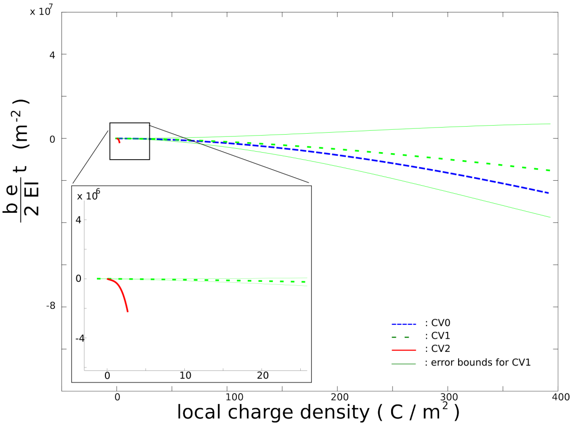
CV2 Max displ. amplitude : 45 nm



- High charge localization (clean surface)
- Weakened singularity upon adsorption

Charge redistribution upon adsorption !

Coupling relationship



- Reduced charge density range
- Increased dielectric constant [A, JoMMS (2007)]

Max. stress divided by 10 !

Conclusion

- Full-field method to monitor electrochemically-actuated cantilever sensors
- A dedicated identification method reveals that passivation results in :
  - Local effect (dielectric constant : specificity?)
  - Global effect : charge redistribution
- $\times 10$ on the (local) coupling efficiency but $\times 2$ on the displacement : Ambiguous results?
- Design issues to make the most of their potential
- Ongoing : new experimental set-up