Combined atomic force microscope and acoustic wave devices: application to electrodeposition.

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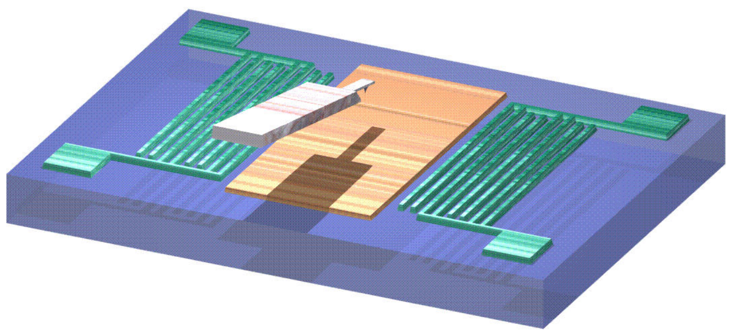
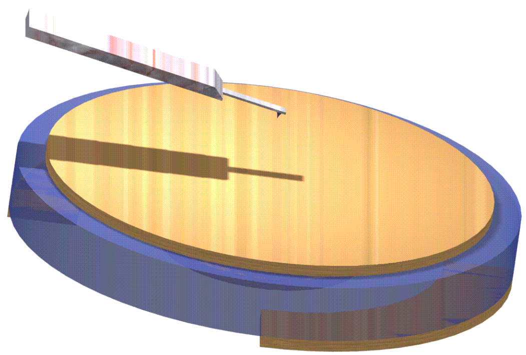
Aim

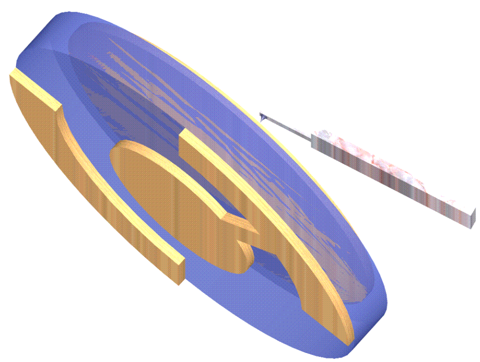
Development of biosensors based on acoustic wave devices: requires to understand the sensing mechanism in liquid medium.

Biological reactions are too slow: use of electrodeposition (fast, reversible and more reproducible).

Look at the nm scale what is happening and relate the observations to the frequency variations.

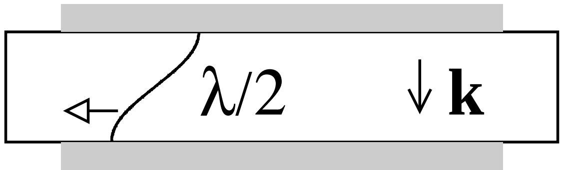


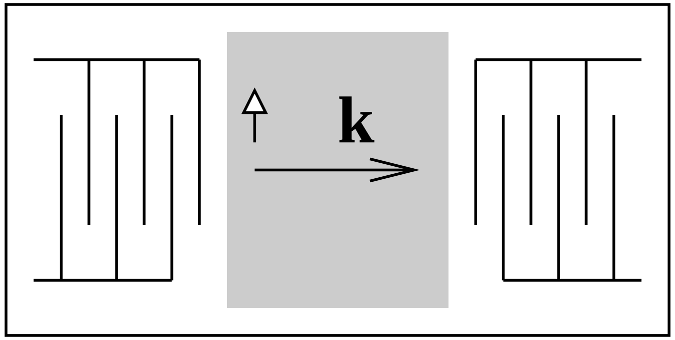




Principle

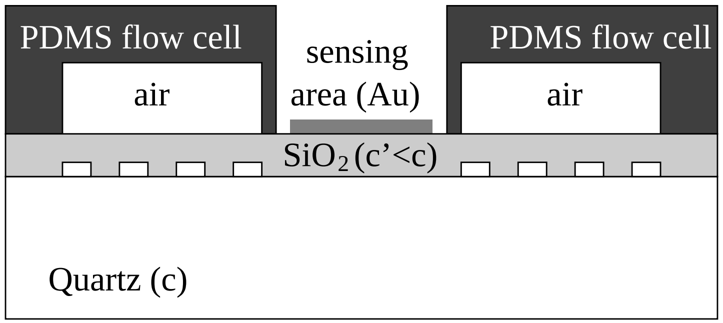
QCM (c=constant) SAW device (λ=constant)





$$\left.\begin{array}{r} \text{QCM: } \Delta m \rightarrow \Delta\lambda \rightarrow \Delta f \\ \text{SAW: } \Delta m \rightarrow \Delta c \rightarrow \Delta\Phi \rightarrow \Delta f \end{array}\right\} \text{what about additional viscosity effects ?}$$

Love mode device:



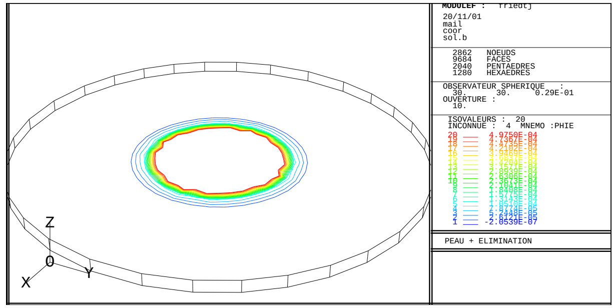
Advantages of SAW:

- higher sensitivity (when properly designed)
- open back side for adding more sensing techniques
- sensing electrode is not used/polarized

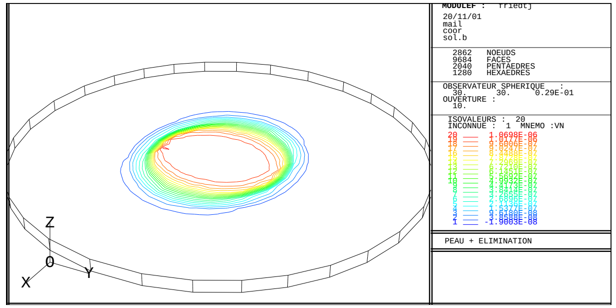
QCM/AFM combination

- static finite element analysis: out of plane displacement A is 0.1 pm
- $A_{dynamic} = A_{static} \times Q \Rightarrow$ out of plane displacement is 0.3 nm ($Q \simeq 3000$)
- in plane displacement is at most 3 nm, smaller than AFM pixel size
- standing wave pattern between QCM and cantilever holder only disturbs the resonance frequency during approach
- fundamental resonance frequency (5 MHz) is unstable and overtones of the QCM must be used

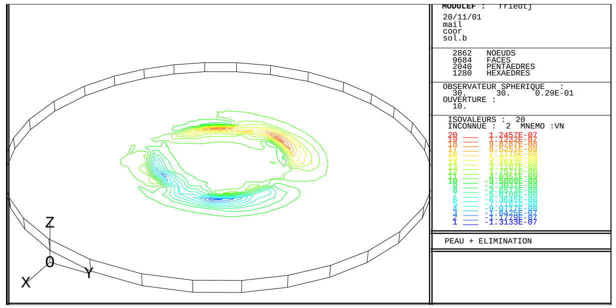
Use of commercial instruments:
- Q-Sense (Göteborg, Sweden) for QCM-D monitoring (overtones 1, 3, 5, 7+Q factor)
- Molecular Imaging (AZ, USA) AFM



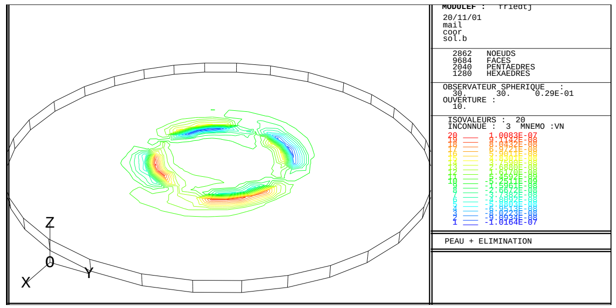
DC potential (0.5 V)



In-plane displacement (1 pm)

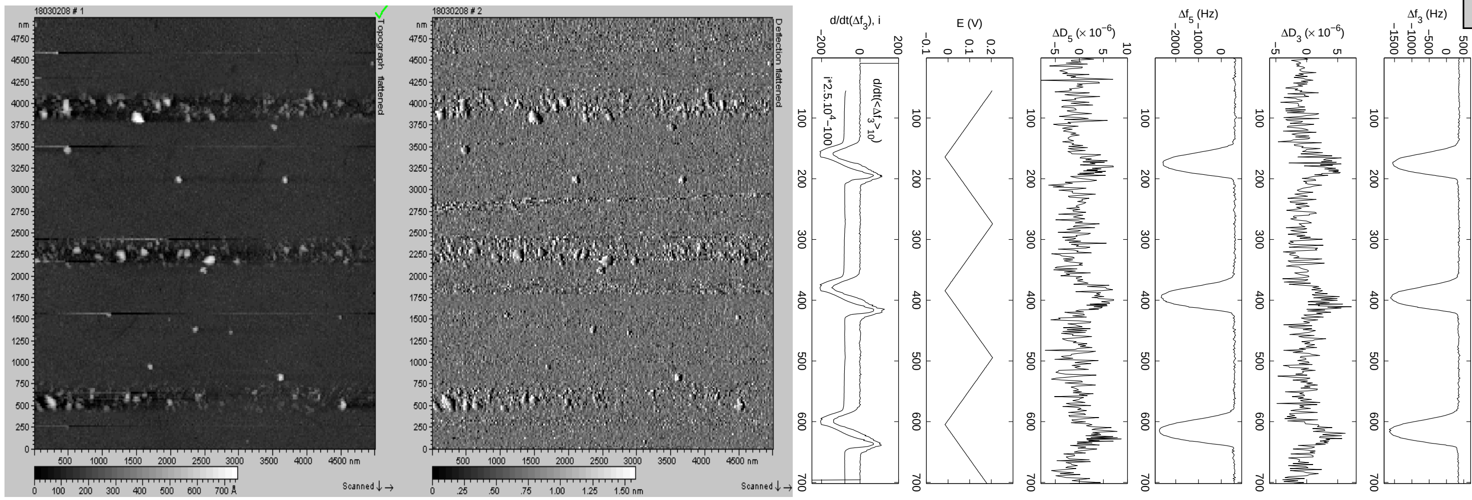


In-plane displacement (0.1 pm)

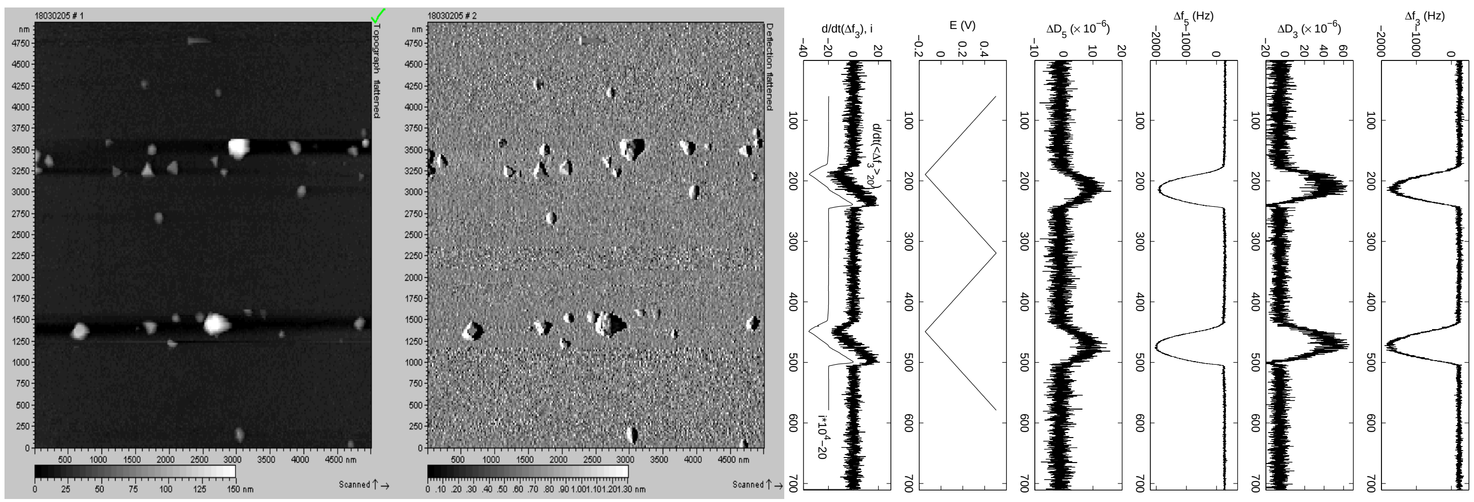


Out-of-plane displacements (0.1 pm)

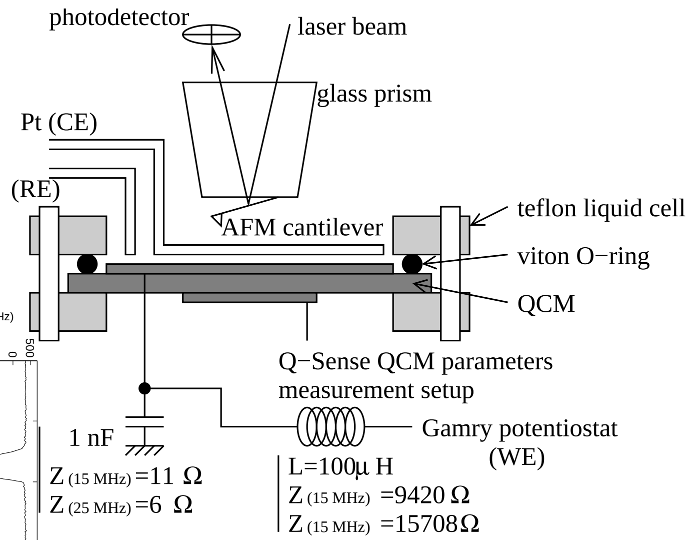
Measurements



Ag



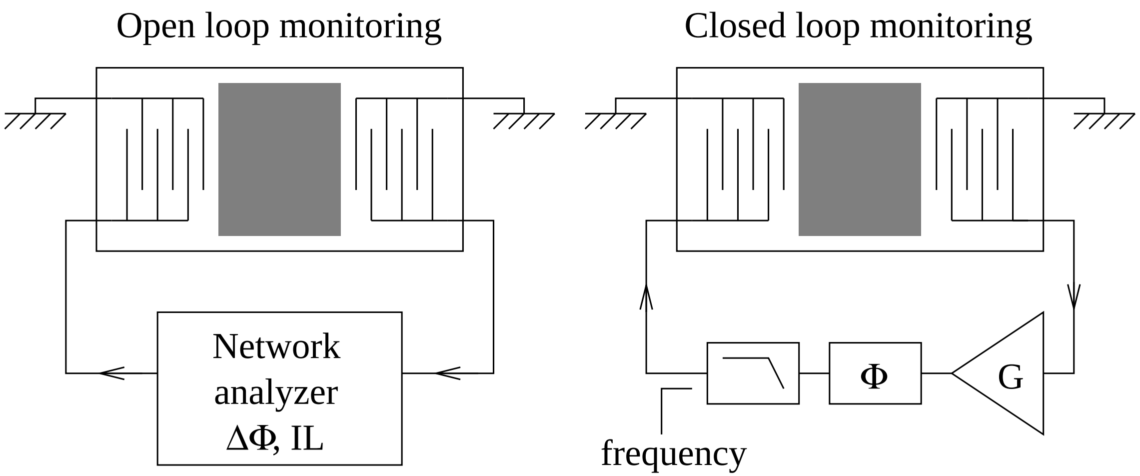
Cu

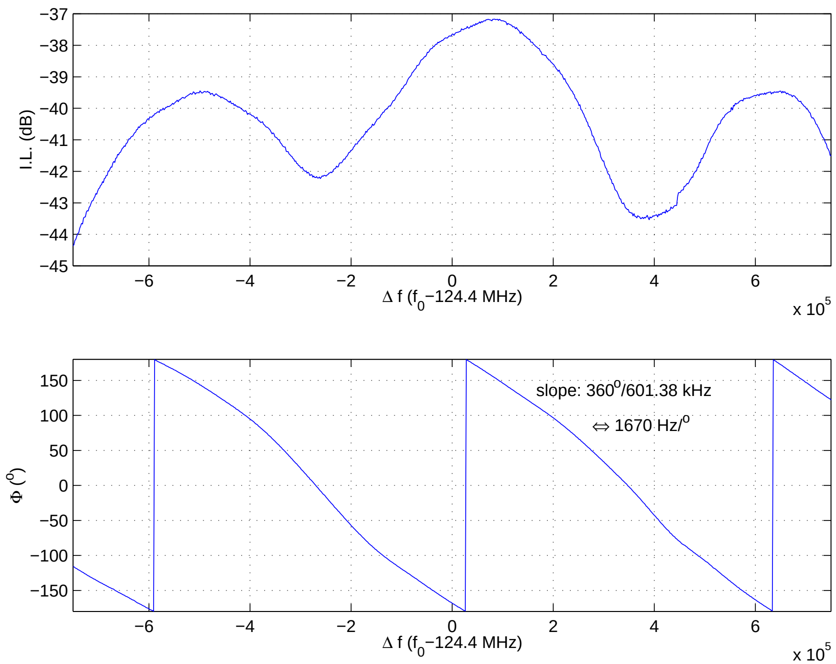


Rigid layer ⇒ $\Delta f_n/n \propto \Delta m_{layer}$ (low damping) with n: overtone number

Viscous layer ⇒ $\Delta f_n/\sqrt{n} \propto \{\Delta m_{liquid}, \Delta m_{layer}\}$ (large damping)

Experimental setup



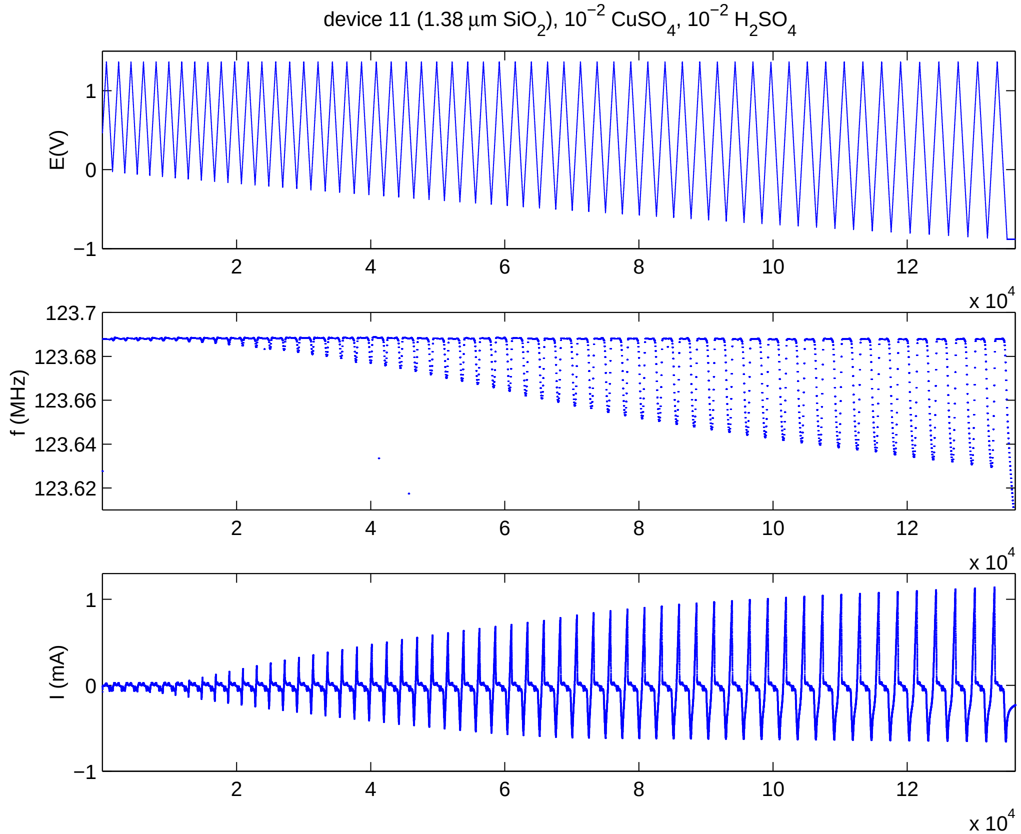


Open/closed loop operation: requires a 4-port network analyzer

PLL setup: compensate for energy losses (and phase shift)

Problem: liquid must *not* reach the IDTs

Measurements

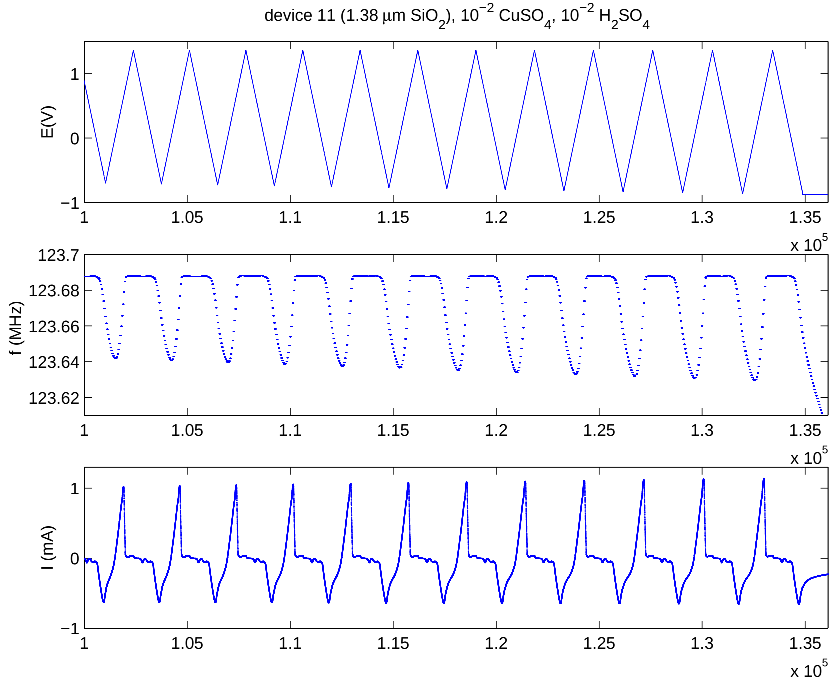


$\longrightarrow$ time (a.u.)

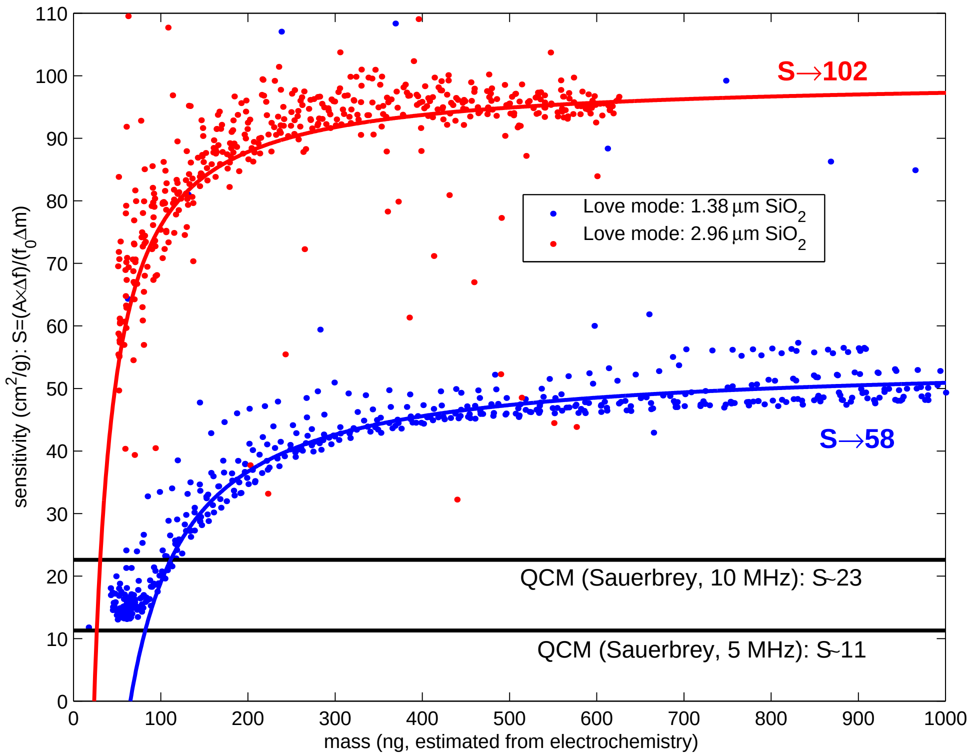
$$m_{Cu} = \frac{\sum I \times \delta t}{N \times e} \times \frac{M_{Cu}}{n_e}$$

$N \times e = 96440$ C: 1 Faraday

$M_{Cu} = 63.5$ g/mol, $n_e = 2$



Sensitivity estimates



$$S = \frac{\Delta f}{f_0} \times \frac{A}{\Delta m} \text{ cm}^2/\text{g}$$

A: sensing area (3.2×3.5 mm^2)

f_0: center frequency (123.7 MHz)

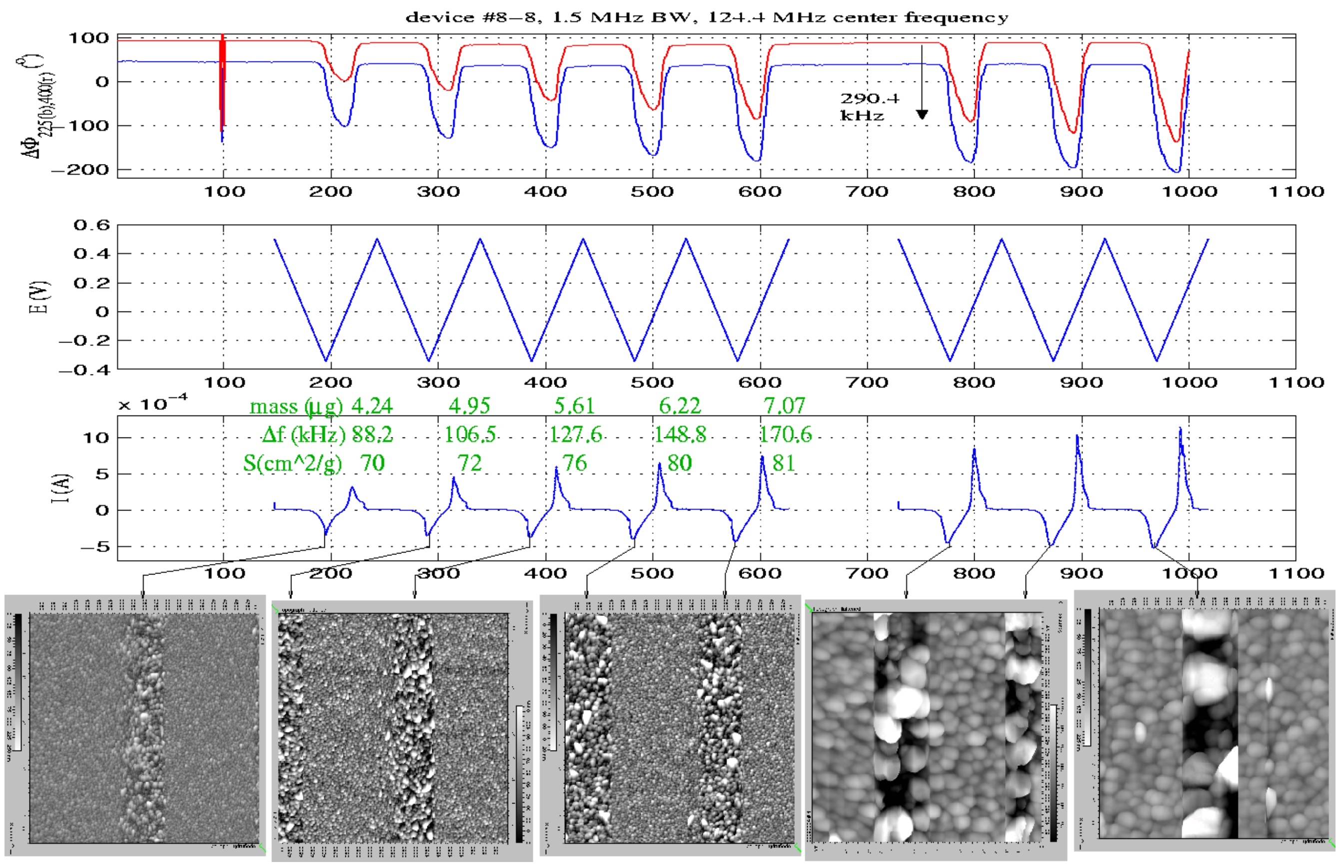
Δm: deposited mass (electrochemistry)

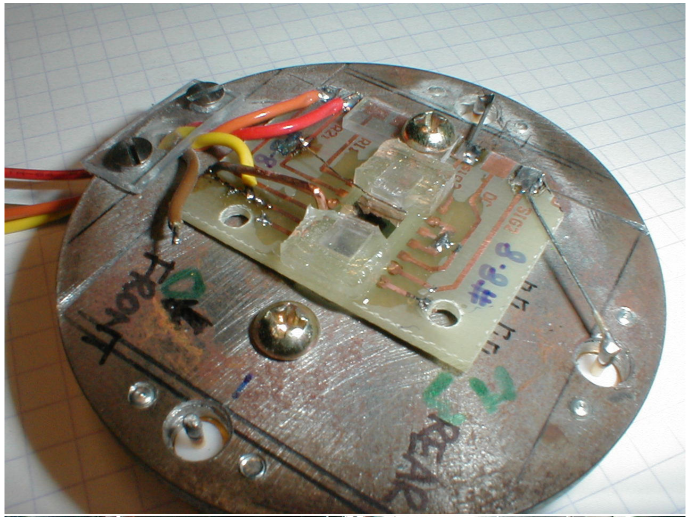
Δf: frequency variation

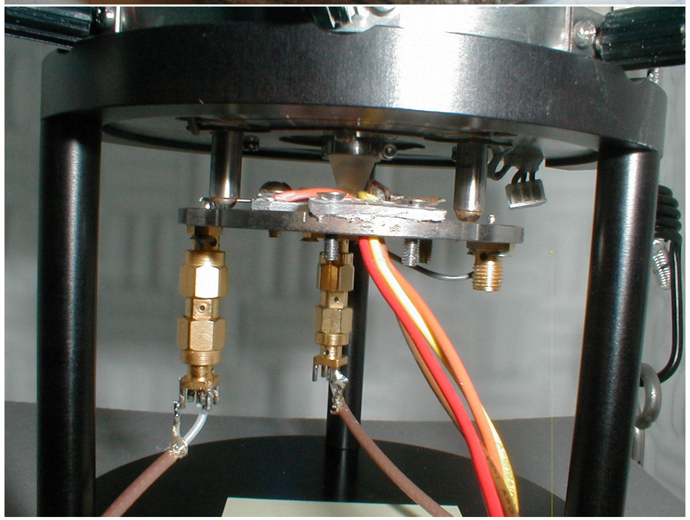
S assumed to be constant, $\forall \Delta m$. Good fit when including offset in the current measurement by the potentiostat $\Rightarrow$ rigid mass.

AFM combination

Monitoring simultaneously $\Delta\Phi \Rightarrow \Delta f$, Δm and topography at the nm scale $\Rightarrow S \simeq$ 70..80 for $\Delta m =$ 4.5..7.5 μg ($S_{QCM} \simeq$ 20 cm^2/g).







Conclusion and perspectives

- ability to independently measure deposited mass and frequency shift
- estimation of the sensitivity: constant over the mass range analyzed
- high sensitivity of Love mode device ($\sim$ 10 times more sensitive than QCM)

- fluidics setup (IDTs protection)
- application to biology: replace electrochemical cell by biochemical reactions
- combination with other detection methods (SPR)

Copies of the slides at `http://mmyotte.free.fr/chua`