Habilitation defense: acoustic sensors and associated embedded instruments

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Introduction

PhD obtained in 2000

1. 3 year postdoctoral position at IMEC in Belgium (biosensors)
2. 1 year postdoctoral position at Nuclear Microanalysis Laboratory (Besançon)
3. 1 year contract at FEMTO-ST (high frequency resonator)
4. since its creation in 2006: SENSeOR, hosted by the time & frequency department of FEMTO-ST (acoustic sensors)

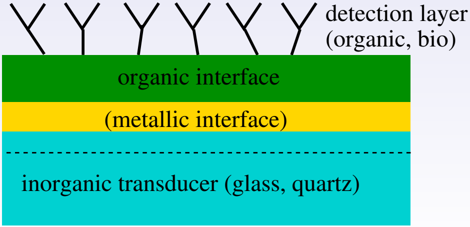
Main topics investigated during this period:

- radiofrequency acoustic sensors (QCM, Love mode SAW, tuning forks)
- evanescent field optical sensors (SPR)
- scanning probe microscopies (SECM, shear force, AFM in liquid)
- embedded instruments (hardware and software/operating systems)

Direct detection biosensors

- Direct detection: **time** resolved, no preparation ⇒ continuous **monitoring**
- **Piezoelectric** substrate: integrated conversion from electric to mechanical
- Love mode: **confine** energy close to the sensing area, reduce contribution of the bulk solution (improved SNR) = **sensitivity**
- **Selectivity** is solely provided by the surface chemistry

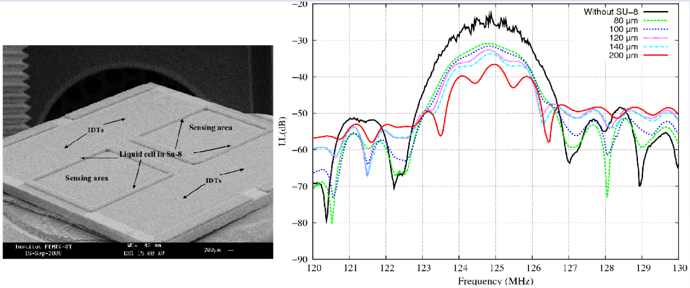
⇒ identify the **physical properties** of the adsorbed layer (modelling)
⇒ multiple informations on a given layer to **separate** contributions
⇒ provide a unique signature (**kinetics**)



Technology: packaging issue

Objective:

1. transmission delay line (S_{21})
2. **wafer scale** packaging preventing the **liquid** from reaching the IDTs
3. minimize acoustic **losses**

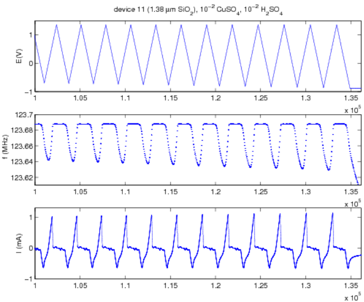


• L. Francis, J.-M Friedt, C. Bartic, A. Campitelli, *A SU8 cell for surface acoustic wave biosensors*, Proc. of SPIE **5455** (2004), 353-363

• L.El Fissi, J.-M Friedt, B. Belgacem, F. Chérioux, V. Luzet, S. Ballandras, *Fabrication and Packaging Technologies of Love-wave-based Microbalance for Fluid Analysis*, accepted Sensors & Actuators A (doi: 10.1016/j.sna.2010.01.027)

Electrodeposition for liquid phase sensitivity measurement

- Definition of mass sensitivity, assuming the gravimetric interaction (rigid layer) is the only cause of frequency shift $S = \frac{\Delta f}{f_0} \times \frac{A}{\Delta m}$
- Reversible for reproducibility and measurement at various masses
- Issue: is viscosity negligible ?



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

$N \times e = 96440$ C: 1 Faraday
$M_{Cu} = 63.5$ g/mol, $n_e = 2$
but
$\Delta m = m_{Cu} + m_{leak}$ with
$m_{leak} = \frac{I_{leak}}{N \times e} \times \frac{M_{Cu}}{n_e}$

Conclusion:

$$S_{observed} = \frac{\Delta f}{f_0} \times \frac{A}{\Delta m} = S_{th} \times \frac{m_{Cu}}{m_{Cu} + m_{leak}}$$

J.-M. Friedt, L. Francis, K.-H. Choi and A. Campitelli, *Combined Atomic Force Microscope and Acoustic Wave Devices: Application to Electrodeposition*, J. Vac. Sci. Tech. A **21** (4), 2003, 1500-1505

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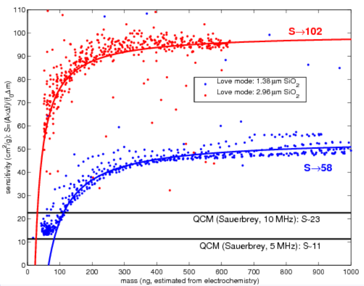
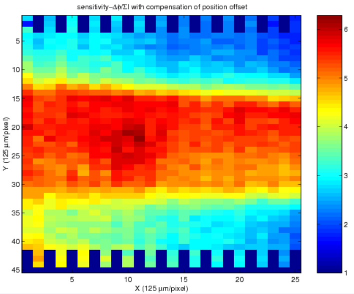




A.C. Hillier & M.D. Ward, *Scanning electrochemical mass sensitivity mapping of the quartz crystal microbalance in liquid media*, Anal. Chem. **64** (21), 1992, 2539-2554

Optics + acoustics

- Do optical and acoustic methods provide the same information ?
- We know that acoustic sensors are affected by viscosity. What about optics ?
- SPR: $n \times d$
- Love mode acoustics: $\rho \times d$, η

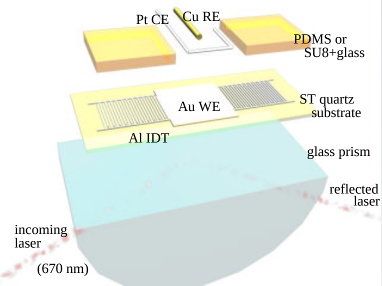
Assuming that ρ and n both depend on x the solvent fraction in the layer, then ρ, d and η should be identified with the measurement of θ_{SPR}, c_{Love} and IL_{Love}.

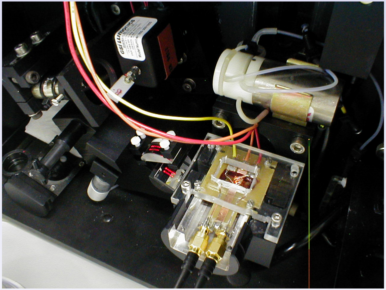
1. assume the layer is rigid: SAW provides $\rho \times d$ considering S is known
2. check SPR angle shift with d and assuming $n \propto \rho$
3. iterate until d and $\{\rho, n\}$ match experimental data

$\Rightarrow$ acoustic and optical 2D planar multilayer modelling capabilities

SAW/SPR combination

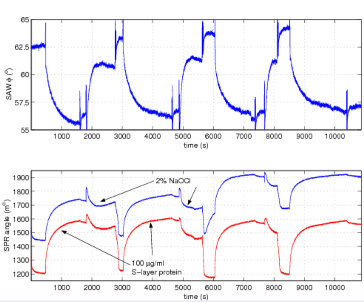
General strategy: replace the **passive** glass slide by an **active** SAW delay line (applicable to AFM, UV-VIS ...)

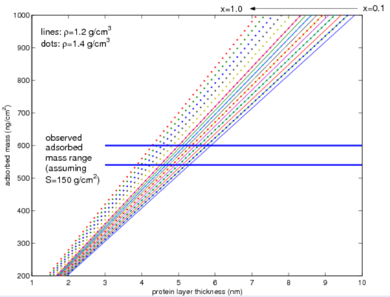




Apparatus and methods for simultaneous surface accoustic wave and surface plasmon resonance measurements, European Patent EP1636569, United States Patent 20060173636

From globular to fibrilar proteins



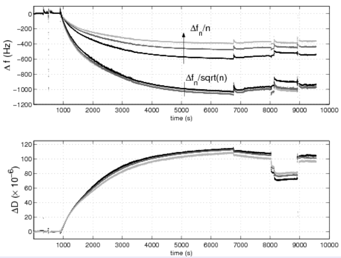


- **Globular proteins** acts mostly like a thin **rigid** film: valid approximations for both acoustic (mass effect) and optics ⇒ conditions applied during BIAcore's radiolabelling calibration

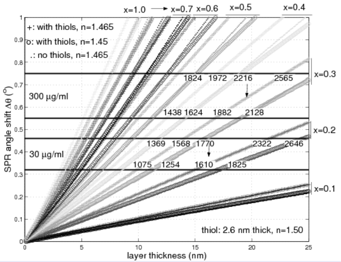
J.-M Friedt, L. Francis, G. Reekmans, R. De Palma, A. Campitelli and U.B. Sleytr, *Simultaneous surface acoustic wave and surface plasmon resonance measurements: electrodeposition and biological interactions monitoring*, J. of Appl. Phys., **95** (4), 2004, 1677-1680

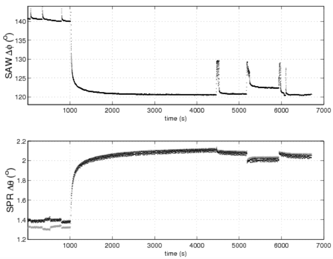
QCM and SAW/SPR analysis

- **Fibrilar proteins** extend deep in the buffer solution, equivalent to a thick layer full of solvent ⇒ strong contribution of **viscosity**

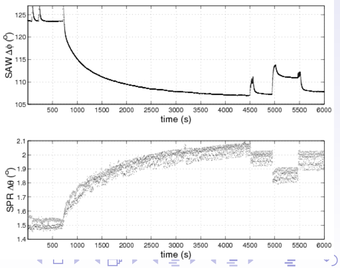


QCM



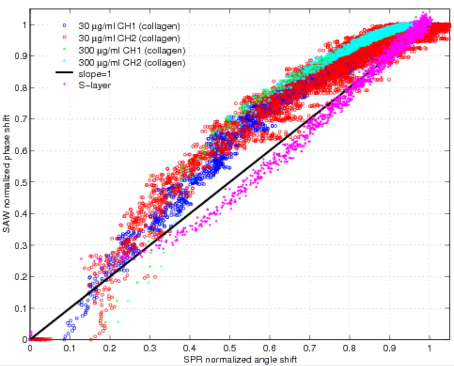


SAW/SPR



QCM and SAW/SPR analysis (2)

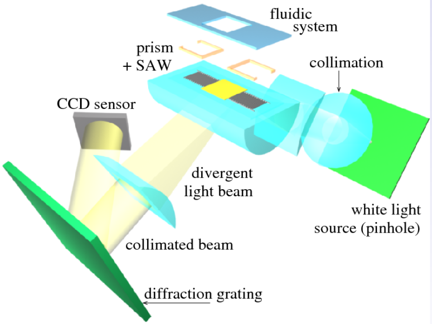
⇒ SPR **underestimates** the adsorbed mass (lower optical index than expected)

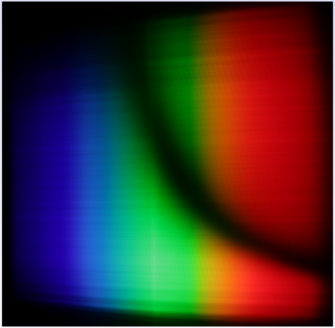


Analyte (bulk concentration, μg/ml)	surface density (ng/cm^2)	d (nm) SAW/SPR	x (%) SAW/SPR	$\Delta f_n/\sqrt{n}$ (Hz) QCM	$\Delta f_n/n$ (Hz) QCM	ΔD ($\times 10^{-6}$) QCM
Cu		2 − 12	?	?-1000	NO	50
S-layer	560±20	4.7 ± 0.7	75 ± 15	NO	45=900	3-5
CTAB	135±15	1.0 ± 0.1	100	NO	8=160	0.2-0.5
collagen (30μg/ml)	1750±150	16.0 ± 3.0	25 ± 15	1000	NO	100
collagen (300μg/ml)	2100±200	19.0 ± 3.0	35 ± 10	1200	NO	>120
fibrinogen (46μg/ml)	750±100	6.0 ± 1.5	50 ± 10	110±5	55±5≃ 1110	4-10
fibrinogen (460μg/ml)	1500±500	13.0 ± 2.0	50 ± 10	NO	100=1700	8-10

Future work: white light SPR

- Complement single wavelength SPR with full **dispersion relation**
- **No moving** part: compatible with SPM and RF connections to SAW
- Subsecond **time resolution**



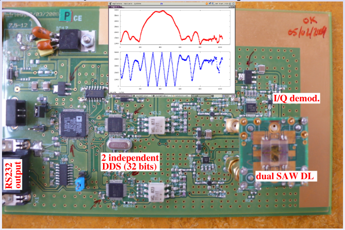


B. Sadani & J. Salvi, optics department, FEMTO-ST

Delay line or resonator ?

So far, acoustic delay lines were selected for

- ease of design: no need for simulation tool
- easy to fabricate: no strong constraint on lithography
- easy to interpret: insertion loss=dissipation, phase=velocity
- **structure free** sensing area (cf SPR, AFM)
- **robust** interrogation in an openloop configuration + failure diagnostics



However, are acoustic delay lines the most sensitive transducers ?

- beyond the influence on the acoustic velocity, the organic layer might affect the **reflection efficiency/phase** on the mirrors
- improved sensitivity expected, but results more difficult to **interpret**
- **gas sensors**: low insertion losses, reliable working conditions, but requires increased sensitivity

Resonator for wireless physical quantity measurement

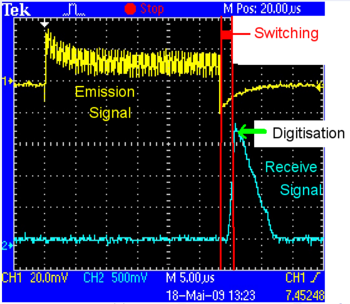
Battery-less sensors for **wireless** interrogation.

Compliance with European ISM regulations:

- 434 & 2450 MHz bands
- 2450 MHz is busy with digital communication modes + challenge for sensor manufacturing
- 434 MHz is narrow ⇒ narrowband device = **resonator**

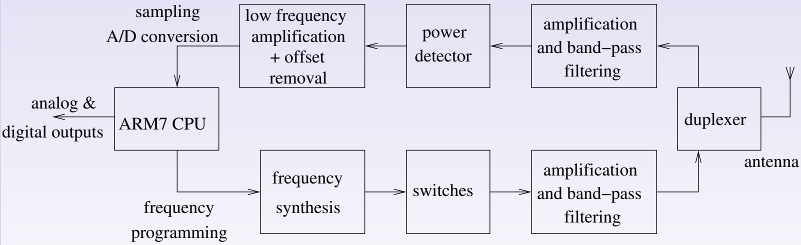
Dedicated, flexible electronics:

- switching mode S_{11} frequency-sweep network analyzer,
- uses **pulse-mode** RADAR for improved isolation between emission and reception,
- generate a short pulse at a given frequency
- listen for the returned power (time constant $Q/(\pi \times f)$ ~6 μs)



Dedicated hardware platform

- Quartz is linear (**no threshold**): known emitted frequency, listen over the whole frequency range at the returned power (**single measurement**, opposite of Fourier transform strategy) +
- All timings & emitted frequency are software defined (flexibility)
- Battery/USB bus powered, 1.5 W consumption



J.-M Friedt, C. Droit, G. Martin and S. Ballandras, *A wireless interrogation system exploiting narrowband acoustic resonator for remote physical quantity measurement*, Rev. Sci. Instrum. **81** (2010) 014701

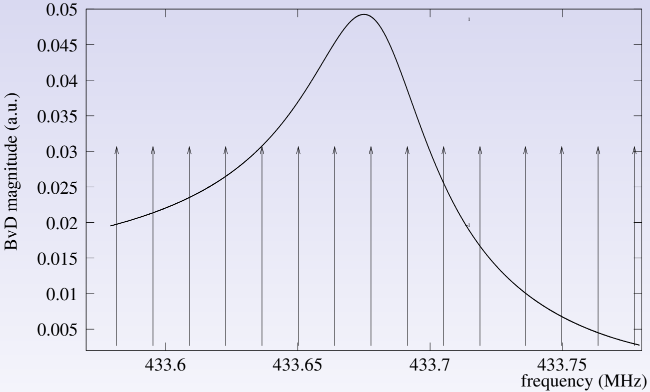
Various interrogation strategies applicable thanks to the **full software** control of the DDS (more about this later):

1. probe the resonator with a **fixed frequency comb**, and look for maximum returned power
2. focus the probe on the identified resonance (**closed loop frequency control**): faster, removes bias source, but requires constant vision of the sensor
3. **FM strategy**: the resonator acts as an FM to AM converter. Best accuracy but slow (each FM period must appear quasi-static from the resonator point of view)

→ In all cases, additional **signal processing** for resonance fitting and resonance frequency accuracy improvement.

Fixed comb strategy

- $\Delta f = f_0/(3 \times Q)$: three measurements within resonator bandbass
- pulse **duration** $> Q/f_0$ (spectral width defined by resonator)



- **Robust** but slow (typically 128 points and 16 averages =7.7 ms/sweep)
- **poor resolution** ($f_0/(3 \times Q) \sim 15$ kHz=6 K at 2500 Hz/K)

Signal processing

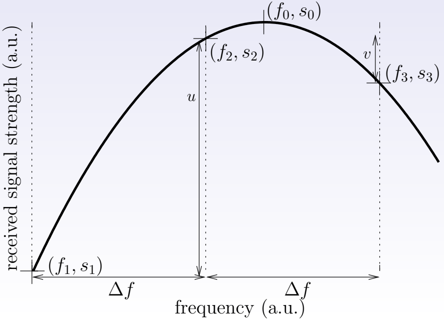
Target resolution: sub-500 Hz (0.2 K) $\Rightarrow$ high sensitivity to magnitude noise & too many frequency steps (slow)

$\Rightarrow$ **polynomial fit** of the resonance shape (local 2nd order polynomial fit approximation)

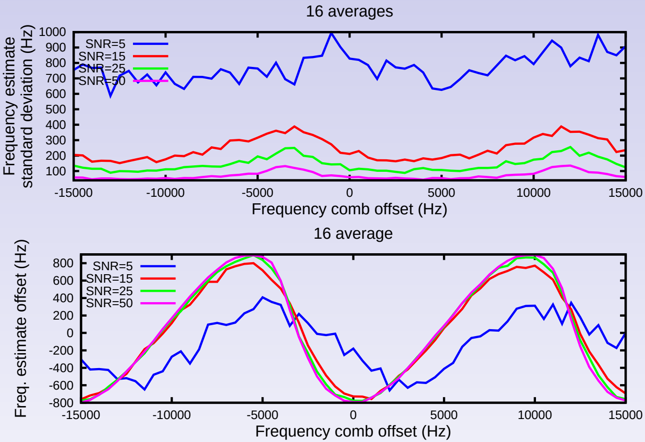
Computational **efficiency**: assuming constant frequency steps Δf, then

$$f_0 = f_2 + \frac{\Delta f}{2} \times \frac{s_1 - s_3}{s_1 + s_3 - 2 \times s_2}$$

Typical signal to noise ratio: 100 $\Rightarrow d(f_0 - f_2) \simeq \frac{\delta s}{\Delta s} \times \Delta f$: optimize Δf to keep $\Delta s = \{s_3 - s_2, s_2 - s_1\}$ large



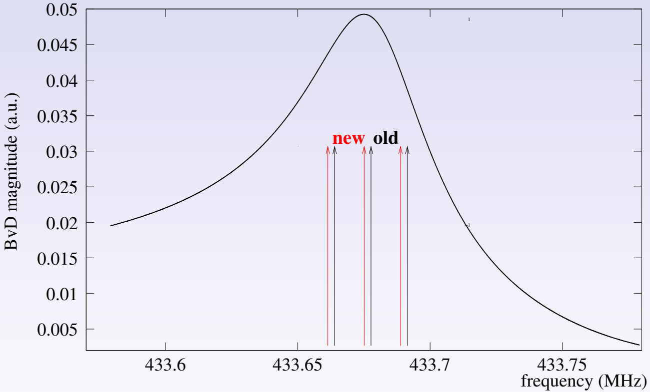
J.-M Friedt, *Method for querying a piezoelectric resonator and querying device implementing said querying method*, WO/2009/103769, `www.wipo.int/pctdb/en/wo.jsp?WO=2009103769`



- BvD resonance is **not symetric** (antiresonance) $\Rightarrow$ **bias**, depends on Q and signal to noise ratio ($\pm$800 Hz $\sim \pm$0.3 K)
- frequency estimate **standard deviation** is dependent on the position of the resonance with respect to the (fixed) frequency comb

Three-point strategy

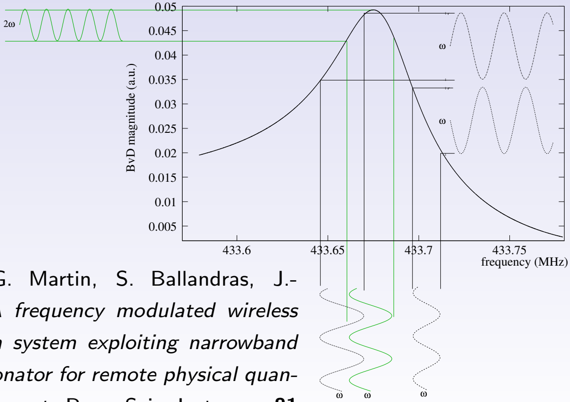
- **Feedback loop** keeps one comb frequency at last known resonance position ⇒ eliminates variance and bias dependence on resonance position
- parabola = **3 points**: no need to sweep whole ISM band



⇒ **fast**: 6×60 μs=360 μs/measurement or 2.6 kHz refresh rate + low memory consumption

FM method

- Resonator acts as an FM (ω_m) to AM converter
- Resonance is measured at frequency at which ω_m component is null (non-linearity generates $2\omega_m$ and cancels ω_m)
- closed loop feedback strategy: slow but **10-fold accuracy improvement**



C. Droit, G. Martin, S. Ballandras, J.-M Friedt, *A frequency modulated wireless interrogation system exploiting narrowband acoustic resonator for remote physical quantity measurement*, Rev. Sci. Instrum. **81** (5), 2010 (056103)

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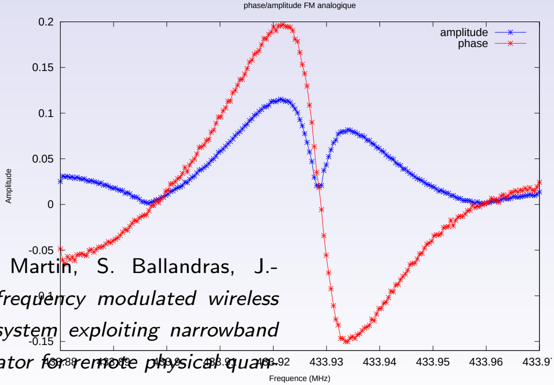
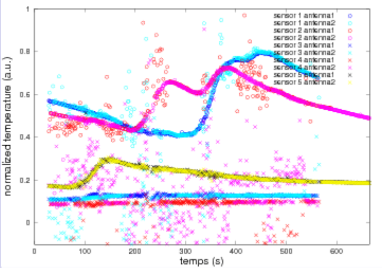


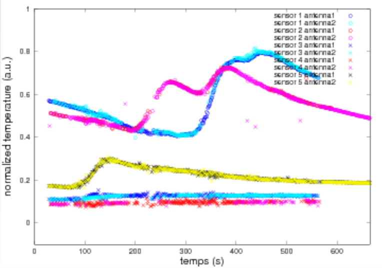
C. Droit, G. Martin, S. Ballandras, J.-M Friedt, *A frequency modulated wireless interrogation system exploiting narrowband acoustic resonator for remote physical quantity measurement*, Rev. Sci. Instrum. **81** (5), 2010 (056103)

Resonator measurement results

1. **Differential measurement** to reduce influence of local oscillator drift & sensor aging,
2. emitted power is **controled** by received power (feedback loop),
3. averaging ⇒ use variance as **quality of service** index,
4. usable with multiple sensors (**frequency multiplexing**)
5. interrogation **range**: 30-300 cm + antenna **multiplexing**

Demonstration of the working principle on **rotating** parts (>4000 rpm), strong magnetic fields (high current), high voltage, closed cavities, **embedded** in tires (>200 km/h) ...





Reflective delay lines for wireless physical quantity measurement

Resonator ⇒ 50% duty cycle.

1. Regulation: maximum power is 10 dBm (4 dBm average)
2. Requires a dedicated interrogation unit

But general purpose interrogation units already exist: RADARs.

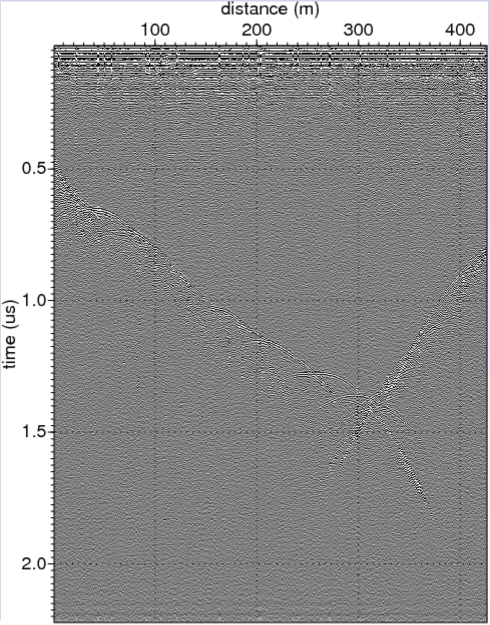
- 100 MHz RADAR (10 ns pulse) generated every 10 μs = 0.1% duty cycle
- Peak power=360 V=64 dBm in 50 Ω load (2.6 kW)

Ground Penetrating Radar (GPR) basics:

- generate a pulse at known wavelength by "quickly" emptying a capacitor in an antenna
- record at "high" sampling frequency the echos, for a duration up to $\simeq 5$ μs after pulse emission
- repeat at constant time or space interval

⇒ development of delay line tags compatible with GPR (Malå RAMAC, 100 and 200 MHz antenna)

GPR basics

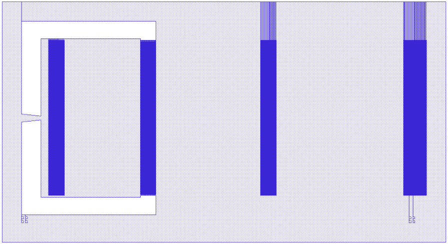


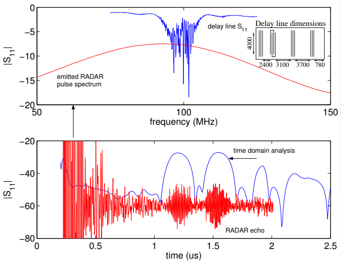
- permittivity and conductivity changes [1]
  $$v = \frac{1}{\sqrt{\frac{\mu\varepsilon}{2}}\left(\sqrt{1+\frac{\sigma^2}{\omega^2\varepsilon^2}}+1\right)^{1/2}}$$
- dielectric interface: Fresnel reflection coef.
  $$R = \left(\frac{\sqrt{\varepsilon_{ice}}-\sqrt{\varepsilon_{rock}}}{\sqrt{\varepsilon_{ice}}+\sqrt{\varepsilon_{rock}}}\right)^2 \simeq -19 \text{ dB}$$
- $v \simeq \frac{c}{\sqrt{\varepsilon_r}}$: 33 (water)-170 (ice) m/μs
- record 200 ns-5 μs
- $\simeq$1000 samples/trace

[1] G. Leucci, *Ground Penetrating Radar: the Electromagnetic Signal Attenuation and Maximum Penetration Depth*, Scholarly Research Exchange **2008**, doi: 10.3841/2008/926091

Reflective delay line design

- Few bits (3) since identification is not our purpose
- strongly coupled substrate ($LiNbO_3$),
- high temperature coefficient of frequency (TCF): sensor
- thick electrodes (1 μm, high reflection coefficient) $\Rightarrow$ **-35 dB**
- measurement principle: **time delay (phase)** between echos is a function of the temperature $\Rightarrow$ referenced measurement
- issue: works at constant wavelength, so that the **frequency depends on the soil permittivity**.





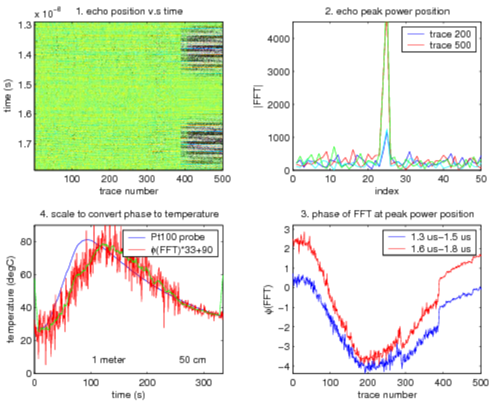
Delay line design by S. Alzuaga, manufacturing by T. Baron

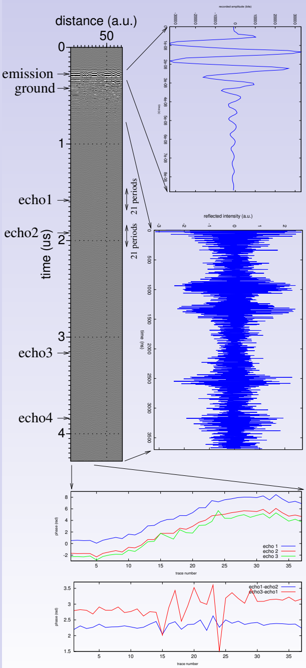
Acoustic delay lines for GPR as buried sensors

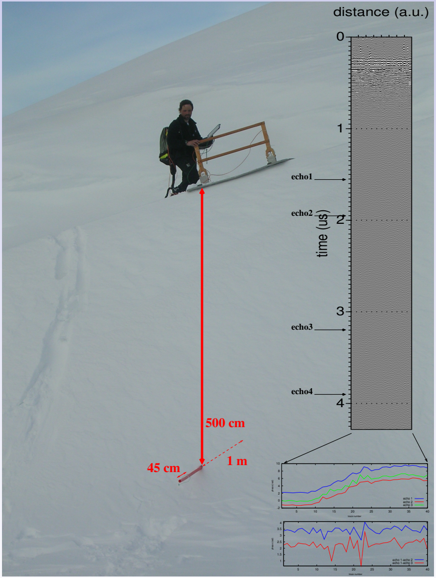
1. GPR = **bistatic low frequency** RADAR (50-1600 MHz), ideally one period but ringing ⇒ 3 or 4 periods emitted
2. record at 10 times the emitted frequency at least, using an equivalent time sampling strategy
3. signal processing to extract the **phase** information

   = acoustic delay

   P.Sandoz, J.-M Friedt, É. Carry, *In-plane rigid-body vibration mode characterization with a nanometer resolution by stoboscopic imaging of a microstructured pattern*, Rev. Sci. Instrum. **78** (2007), 023706
4. insensitive to sensor **distance to GPR** unit
5. practical demonstration in a low conductivity environment (ice/snow)







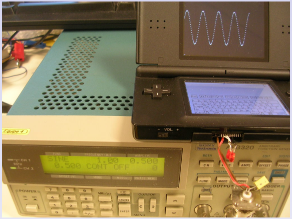
Experiment performed with É. Bernard, ThéMA/CNRS, Besançon

Embedded electronics developments

- Beyond the transducer: provide the **interrogation electronics** in addition to the transducer
- Either RF/digital electronics **development**, or **reverse engineering** to adapt commercial instruments to our needs
- 2005-2009: embedded **operating systems** (uClinux, followed by RTEMS). Provides useful functionalities (networking, filesystem, scheduler, memory management) but hard to keep up to date over long durations and usually functionalities are not needed.
- Low level embedded software for instrument control: **flexibility**, ability to adapt to various conditions (algorithm for filtering unwanted data based on spectral signature, band pass filter, parabolic fit, closed loop control of DDS)
- **Teaching experience** (20 h course + labs, 2 years): dedicated electronics & consumer electronics

Embedded electronics around us

- handheld **game consoles**, PDA, mobile phones



uClinux on Sony PSP | iPod | RTEMS on Nintendo DS

- **consumer** electronics as **teaching** tools
- from **powerful** computers to **low power** consumption embedded electronics: more abstract than embedded electronics (RTEMS & TinyOS executive environment), but reduced resources for computer science
- challenge: keep **up to date** with the technology !

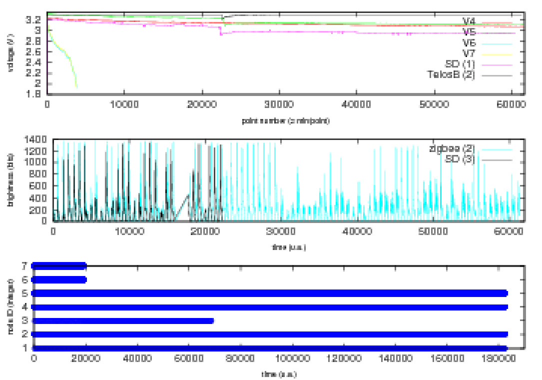
• J.-M Friedt and G. Goavec-Mérou, *Interfaces matérielles et OS libres pour Nintendo DS : DSLinux et RTEMS*, GNU/Linux Magazine France Hors Série **43** (2009)
• S. Guinot and J.-M Friedt, *GNU/Linux sur Playstation Portable*, GNU/Linux Magazine France, **114** (2009), 30-40
• G. Goavec-Merou, S. Guinot and J.-M Friedt, *Developing embedded devices using opensource tools: application to handheld game consoles*, Hacking at Random (2009)

Active sensors

- **Complement** to passive sensors: low power consumption active microcontrolers.
- **MSP430** power consumption: 25 μA ; battery storage: 3600 mA.h $\Rightarrow$ 1 year autonomy (TPMS claims 5 to 10 years ?)
- fancy power management strategies: save time and use an OS (TinyOS)
- yet, low level electronics still has a major contribution (pull up resistors, voltage regulators ...)
- data transmission or **local storage** ?

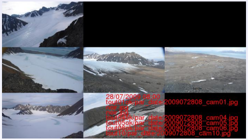
PhD of E. Paco-Chichi, under the direction of H. Guyennet (CS department)

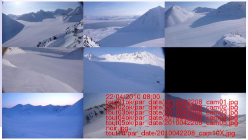
G. Goavec-Mérou, J.-M Friedt, *Étude d'un système d'exploitation pour microcontrôleur faible consommation (TI MSP430) : pilote pour le stockage de masse au format FAT sur carte SD*, GNU/Linux Magazine France **HS 47** (2010)



Polar glacier basin monitoring

- Average of 10 **automated digital cameras** around a polar glacier basin (10 km^2)
- 3 pictures/day since mid-2007
- Challenge of **protecting** the electronics from the environment, **reliability**, data management + **user interface**
- Objective: **complement scalar data** (qualitative), image processing for **quantitative** data extraction, reaching areas imposible to monitor otherwise (**slopes**), high **time and spatial resolution** (w.r.t satellite)





Sequel: new **ANR funding** for the next 4 years

Future embedded electronics developments

1. Processors are unable to guarantee sub-microsecond latencies
2. RF starts at 30 MHz and in our case, sensor start being usable above 100 MHz
3. Need to **complement general purpose CPU** with flexible yet fast electronics extending into the RF range: **FPGA**
4. Complementarity of FPGA approach (dedicated reconfigurable instruments) with general purpose processing architecture (CPU/operating system for data management, user interface, scheduling)

$\Rightarrow$ creating ties with a local company (PhD funded by Armadeus Systems, hosted by FEMTO-ST: implementing image processing algorithms for vision on FPGA)

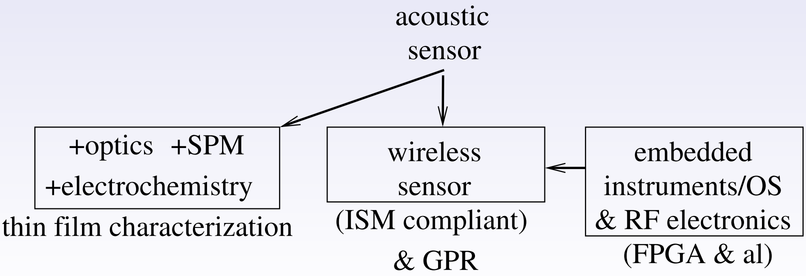
Conclusion

Common topic of all these activities: acoustic sensors

1. characterizing thin film **physical properties**
2. **direct detection** (bio)sensors
3. **wireless passive** sensors
4. associated **embedded electronics**

Additional topics tackled during these endeavours:

1. scanning probe microscopy
2. SPR, UV-Vis spectroscopy, electrochemistry



Perspectives

Future activities:

- **white light** SPR+SAW
- **GPR** for buried sensors (beyond temperature & ice)
- **FPGA** for software defined RF components & coprocessors (VHDL $\rightarrow$ ASIC)
- increased **working frequency** (beyond GHz): electronics & resonator
- sensor **identification** and improved **resolution** through signal processing, dedicated to each component kind
- SAW-based **active** solutions (oscillator + counter)

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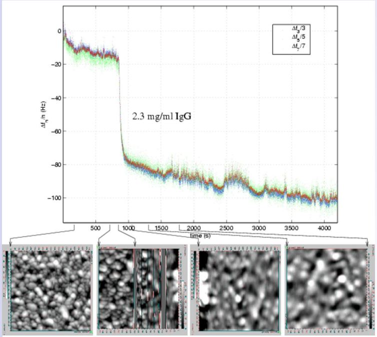
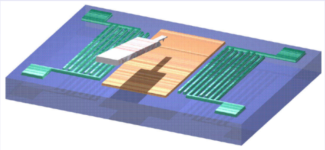
- FEMTO-ST: time & frequency department, optics department (Remo & al) ...
- Association Projet Aurore
- Hydro-Sensor-FLOWS team (& future Cryo-Sensor ANR program)
- Sim Guinot

AFM + acoustic

- Independent estimate of surface topography (adsorbed layer thickness) to deduce, from the mass, the layer density
- Result: poor resolution, poor surface flatness, difficult interpretation (contact/tapping)
- Identify longitudinal mode generation of QCM due to finite electrode dimensions
- Hard to use with organic samples, useful with electrochemical deposition





- K. H. Choi, J.-M Friedt, F. Frederix, A. Campitelli and G. Borghs, *Simultaneous Atomic Force Microscope and Quartz Crystal Microbalance Measurement*, Applied Physics Letters, **81** (7), 2002
- C. Zhou, J.-M. Friedt, A. Angelova, K.-H. Choi, W. Laureyn, F. Frederix, L. A. Francis, A. Campitelli, Y. Engelborghs and G. Borghs, *Human Immunoglobulin Adsorption Investigated by Means of Quartz Crystal Microbalance Dissipation, Atomic Force Microscopy, Surface Acoustic Wave, and Surface Plasmon Resonance Techniques*, Langmuir, **20** (14) 2004, 5870-5878

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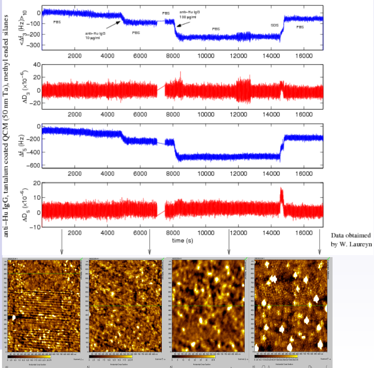
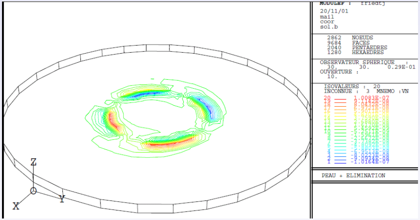




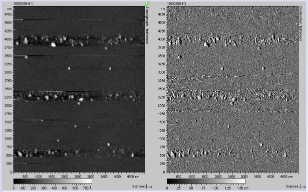
J.-M Friedt, K. H. Choi, L. Francis and A. Campitelli, *Simultaneous Atomic Force Microscope and Quartz Crystal Microbalance measurements: interactions and displacement field of a QCM*, Japanese J. Appl. Phys., **41** (6A), 2002, 3974–3977

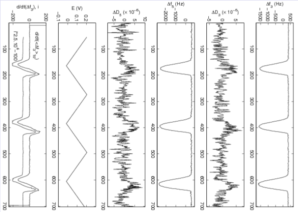
AFM + acoustic

Purpose: replace the passive glass slide with QCR

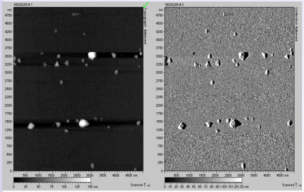
Technical challenge: size of the cantilever + holder, optical setup

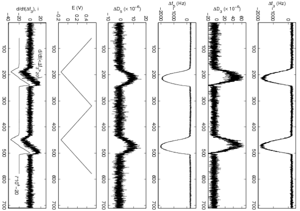
$\Rightarrow$ shear force microscopy (integrated sensor)





Ag





Cu

Rigid layer $\Rightarrow \Delta f_n/n \propto \Delta m_{layer}$ (low damping)

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