Cooperative targets for Ground Penetrating or Spaceborne RADAR remote sensing

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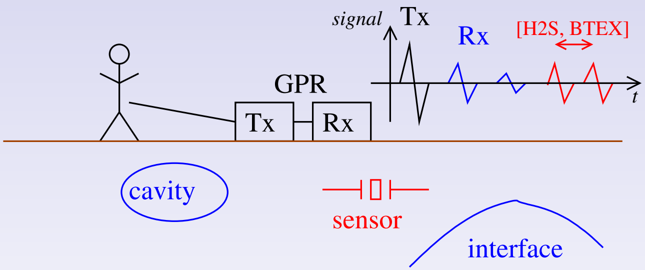
Context

Sub-surface pollution detection in gas and liquid phases using passive buried transducers probed from the surface.



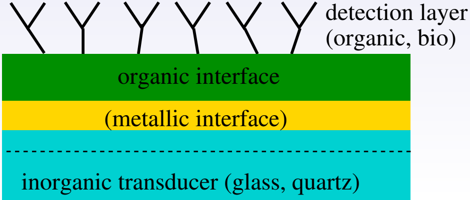
Ground Penetrating RADAR is used for detecting sub-surface interfaces and is readily usable for measuring sub-surface sensors [1] [2]

[1] J.-M Friedt *& al.*, *Surface Acoustic Wave Devices as Passive Buried Sensors*, J. Appl. Phys. **109** (3), p. 034905 (2011)

[2] J.-M Friedt, *& al.*, *High-overtone Bulk Acoustic Resonator as passive Ground Penetrating RADAR cooperative targets*, J. Appl. Phys. **113** (13), pp. 134904 (2013)

Direct detection (bio)sensors architecture

- No sample preparation: continuous monitoring + time resolution (kinetic)
- Surface immobilization of receptor molecules: multiple measurement steps are possible
- **Sensitivity** defined by mass to physical measurement conversion efficiency
- **Selectivity** defined by affinity of the surface functionalization to the targeted compound, rejecting unwanted interference (antibody)
- Improved signal to noise ratio by using evanescent wave (rejects bulk noise and keep only close-to-surface signal)
- **Detection limit** determined by system noise level (detection limit=noise/sensitivity) = complete system (electronics, fluidics ...)



Context

- Wireless passive sensor ...
- for direct detection for continuous monitoring compatibility
- of H_2S and sub-surface pollution by organic compounds.

Themes addressed:

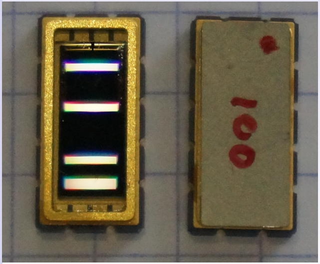
1. **acoustic** transducers for wireless measurement (**sensitivity**)
   1. lithium niobate substrate (Rayleigh wave – only compatible with gas)
   2. lithium tantalate substrate (shear wave, high permittivity)
2. measurement electronics exhibiting sufficient **stability** as a reference
   1. Malå ProEx GPR
   2. Sensors & Software GPR
   3. dedicated electronics

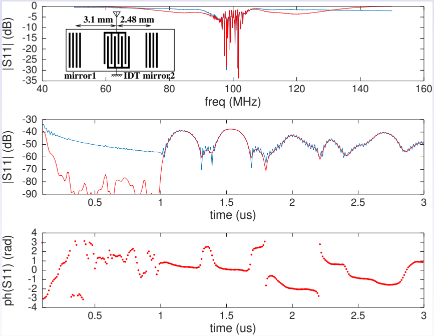
   } **electronics**
3. ability to react with a given chemical compound (**selectivity**)
   1. homogeneous thin film + **packaging**
   2. patterning of contact pads and temperature/RADAR range reference path
   3. **functional** layer

   } **chemistry**

Sensor

- A **surface acoustic wave delay line** converts the incoming electromagnetic pulse to an acoustic wave
- The acoustic wave velocity is dependent on boundary and environmental conditions [3]
- The returned echo delay is dependent on absorbed mass, temperature and distance to RADAR
- *differential* measurement to separate contributions





[3] J.-M. Friedt *& al.*, *Acoustic transducers as passive cooperative targets for wireless sensing the sub-surface world: challenges of probing with Ground Penetrating RADAR*, MDPI Sensors **18** (1), p.246 (2018)

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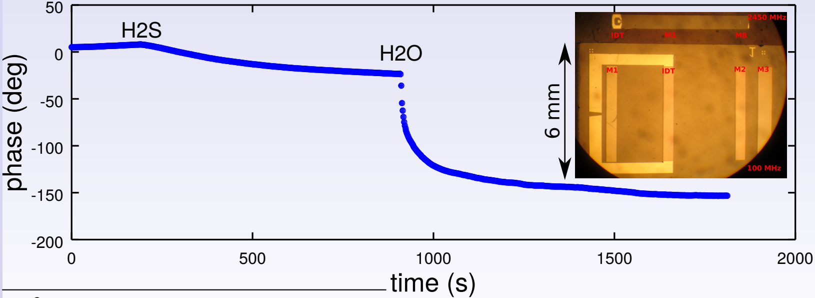
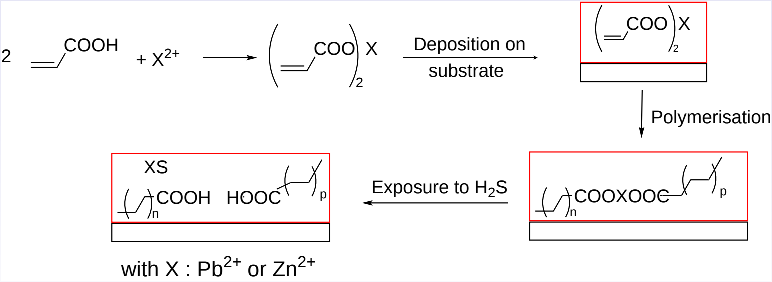


[3] J.-M. Friedt *& al.*, *Acoustic transducers as passive cooperative targets for wireless sensing the sub-surface world: challenges of probing with Ground Penetrating RADAR*, MDPI Sensors **18** (1), p.246 (2018)

Chemical functionalization

Chemical surface functionalization must provide

- selectivity to a given compound, based on **known** detection reaction
- stability over time & chemical environment
- compatibility with thin film deposition (spin coating, drop casting, spraying): **custom resist** using bulk functionalization ($\neq$ surface)
- $\Rightarrow$ enough reactive sites to generate a strong response
- replace thermo-polymerization with **photo**polymerization: patternable to open pads and selectively functionalize a given area (multi-parameter sensing)

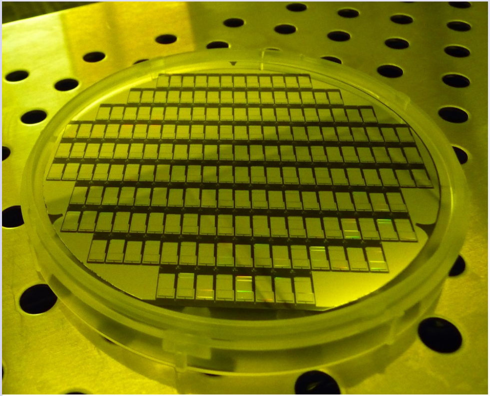


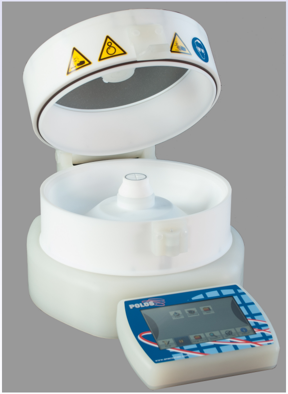
Monomer deposited on inorganic surface followed by polymerization before exposure to H_2S

Wafer scale coating

Wafer scale homogeneous coating (bottom left) **but**

- single chip coating would be well suited to prototyping (tabletop spin coater acquired – bottom right)
- pad opening still challenging (mechanical mask?)
- **chip packaging** for protecting the sensing layer from the environment while leaving the chemical compound reach the surface



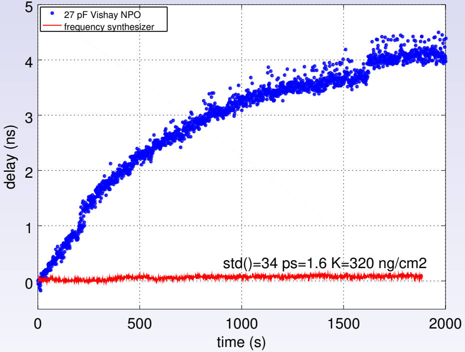


Malå ProEx GPR timebase

Electronics issue: the reference timebase must exhibit better stability than the varying time of flight associated with the measurement [4].

Excessive drift of the local timebase

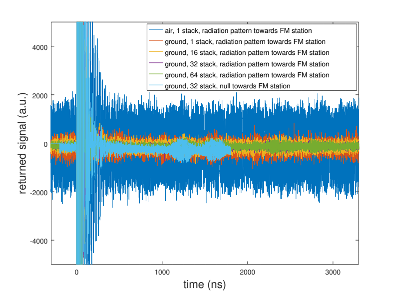
- analog timebase generation sensitive to temperature (warmup)
- replaced analog with digital solution
- excellent stability with laboratory instrument
- implemented an embedded solution: noise due to clock leakage

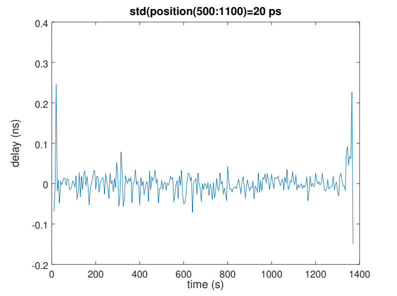


[4] J.-M Friedt, *Passive cooperative targets for subsurface physical and chemical measurements: a systems perspective*, IEEE Geoscience and Remote Sensing Letters **14** (6), pp.821-825 (2017)

Sensors & Software GPR

Alternative solution: another commercial GPR supplier



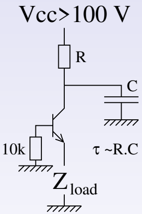


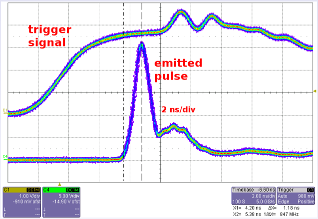
1. excellent long-term stability ($\leq$20 ps)
2. **BUT** closed hardware: sensor measurement as post-processing
3. unable to develop custom sensor reader software [5]
4. lower signal to noise ratio, possibly due to regulation compliance (lower emitted power with respect to Malå?)

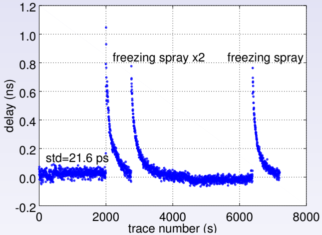
[5] `https://sourceforge.net/projects/proexgprcontrol`

Dedicated electronics

- One of the latest ST-Microelectronics microcontroller provides high resolution timer allowing for 5 GHz equivalent sampling time
- used for demonstrating a dual chip GPR solution[6]
- emitter fabrication has been investigated[7]
- Objective: stable enough hardware to be used on the field





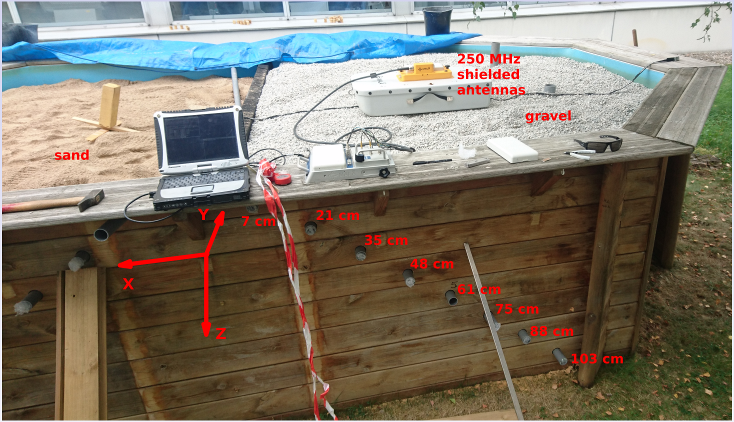


[6]F. Minary, D. Rabus, G. Martin, J.-M. Friedt, *Note: a dual-chip stroboscopic pulsed RADAR for probing passive sensors*, Rev. Sci. Instrum. **87**, p.096104 (2016)

[7]D. Rabus, F. Minary, G. Martin, J.-M Friedt, *A high-stability dual-chip GPR for cooperative target probing*, GPR2018 conference

Testbed

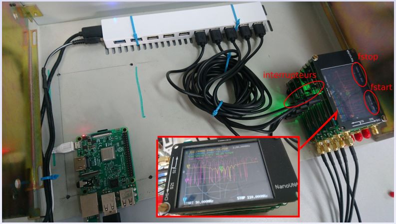
Buried sensor inserted in sandpit

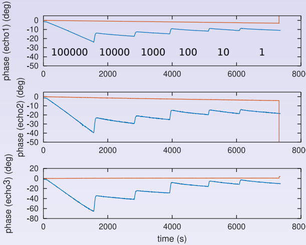


- Measurement demonstrated at 100 MHz (1.5 m long antenna) at a range of 1 m
- Use this testbed to demonstrate realistic measurement environments+develop an **antenna geometry** compatible with sensor insertion from the surface (borehole)

Conclusion

- Sub-surface SAW sensor measurement using COTS GPR
- Demonstrated H_2S detection using wafer-scale functionalization of sensors





Experiments performed at PERL, Total, Lacq

Work in progress:

- Extension to other chemicals (BTEX[8], **pesticides**? biosensors[9]?)
- Use spaceborne satellite signal (**Sentinel1**) for probing sensors?

[8] ANR grant UNDERGROUND @ `https://anr.fr/Project-ANR-17-CE24-0037`

[9] T. Leïchlé, L. Nicu & T. Alava, *MEMS Biosensors and COVID-19: Missed Opportunity* ACS Sensors **5** 3297–3305 (2020)