Subsurface wireless chemical sensing strategy compatible with Ground Penetrating RADAR

Digital is good, analog is bad (TM): timing generator for RADAR systems

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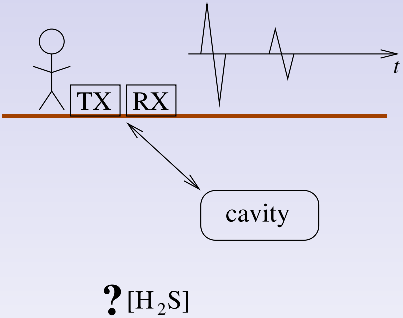
Outline

Subsurface chemical sensing system:



1. acoustic transducer acting as cooperative target (separate sensor echo from clutter)
2. transducer functionalization for chemical sensing: polymer formulation
3. sensor measurement using Groung Penetrating Radar (GPR)
4. sampling rate stability issue and solution for the Malå ProEx

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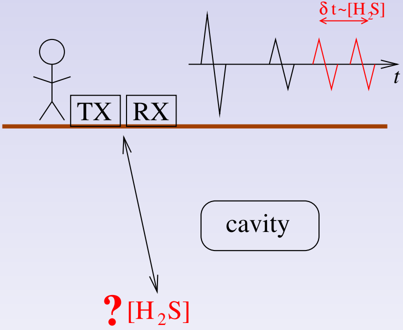

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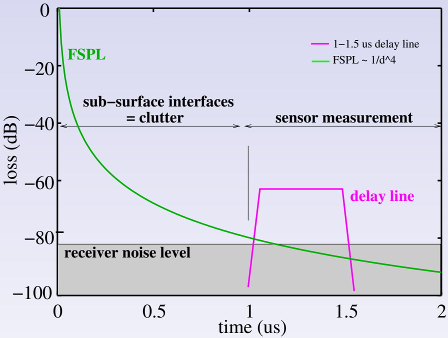
Surface Acoustic Wave transducers as cooperative targets

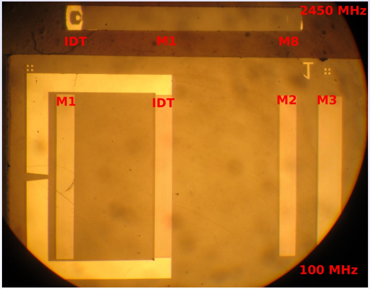
1. A RADAR emits an electromagnetic pulse,
2. the cooperative target delays the pulse beyond clutter delay for identifying the transducer response (**TDMA**)
3. the transducer becomes a sensor if the time delay is dependent on a known quantity (temperature, chemical compound concentration)



4. shrink delay line dimensions by converting the electromagnetic wave to a surface acoustic wave by using a **piezoelectric substrate** (acoustic velocity 10^5 times slower than electromagnetic velocity)

Chemical functionalization

- **Physical** transducer: conversion of adsorbed mass to velocity variation to time of flight variation ⇒ **no selectivity**
- **Chemical** sensor: adlayer selective to a single compound
- Amongst the quantities inducing acoustic velocity variation (temperature, stress), boundary conditions will define the acoustic velocity
- "Microbalance" application of acoustic transducers: the thicker the loading layer, the slower the wave – sensitivity S
  $S = \frac{\Delta f}{f}\frac{A}{\Delta m} = \frac{\Delta v}{v}\frac{A}{\Delta m} = \frac{\Delta v}{v}\frac{1}{\rho dt}$
  $S \simeq 200$ cm^2/g $\Rightarrow \frac{\Delta v}{v} \simeq 200$ ppm if $\frac{\Delta m}{A} = 1\ \mu$g/cm^2
- well known technique for biosensor applications, but can it be used for **subsurface wireless sensing** ?



8×6 mm^2 lithium niobate chip, 20 μm wavelength @ 200 MHz = 5 μm electrodes

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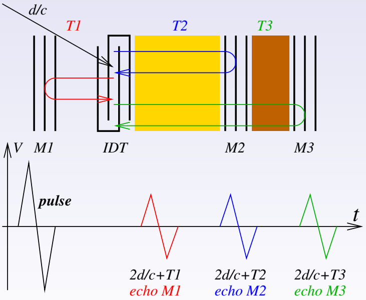

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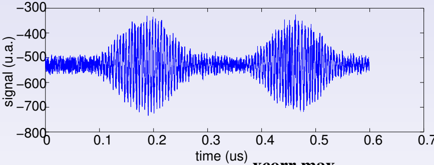
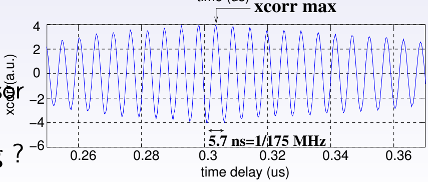
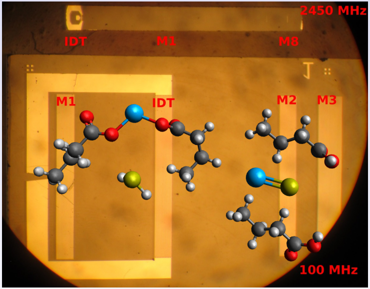




Chemical functionalization

Based on a well known chemical reaction, **design a polymer** whose formulation

1. allows for spreading a homogeneous layer with thicknesses of the order of the wavelength ($\simeq \mu$m)
1. ... with deposition technique compatible with wafer-scale processing (**cleanroom**),
2. includes as **many sensing sites** as possible (low weight polymer matrix),
3. is **selective** to the targeted compound and rejects interfering molecules.

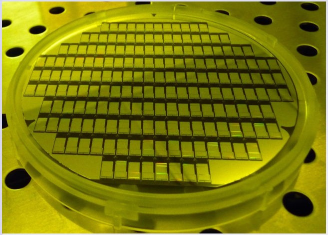


In this case, hydrogen sulfide (H_2S) looks like water (H_2O), but includes a sulfur reacting with heavy metals (thiolation)
$\Rightarrow$ introduce a **reactive heavy metal** ion in the polymer matrix

Sensor measurement

Chemical sensing induces a time delay variation of a few tens of ps:

$(R-COO)_2Pb + H_2S \rightarrow 2RCOOH + PbS$ R: functional alkyl chain



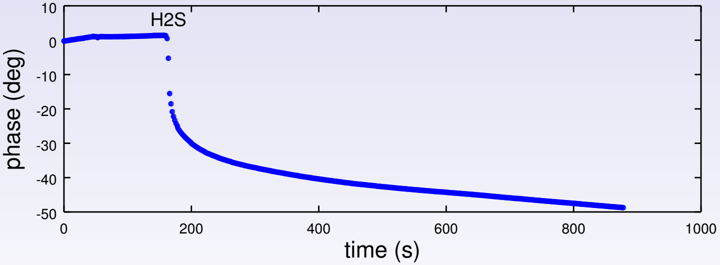
PbS nanoparticles (black): **visual** indicator of reaction

Wafer scale functionalization by spin-coating the synthesized polymer dedicated to H_2S detection: challenge of uniform spreading sub-μm thick polymer wafer.

- Sensitivity $S = \frac{df}{f}\frac{A}{dm} \simeq 200$ cm^2/g (wave property)
- $\rho_{polymer} \simeq 1$ g/cm^3 & $M_{polymer} = (131 \times 2 + 207)$ g/mol & $t \simeq 0.2~\mu$m $\Rightarrow R = \rho/M_{polymer} \cdot t$=43 nmol/cm^2 receptor density
- $M_{H2S} = 34$ g/mol$\Rightarrow$ **absorbed mass per unit area**: $R \times M_{H2S} = dm/A \simeq 1500$ ng/cm^2

Sensor measurement

- Periodic signal delay measurement as a phase shift
- One period τ (5 ns @ 200 MHz) is one full phase rotation 360°
- $\frac{d\varphi}{\varphi} = \frac{df}{f} \Rightarrow d\varphi = \varphi \cdot S \cdot \frac{dm}{A} = 2\pi f \tau \cdot S \cdot \frac{dm}{A} = 20°$ or 280 ps @ 200 MHz (1.5 μg/cm^2, 1 μs)



Velocity variation = phase variation through $\varphi = 2\pi f d / c$ with c varying

Need to measure time delays with sub-100 ps long term accuracy

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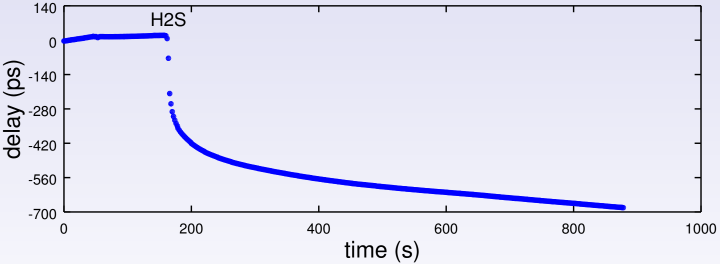

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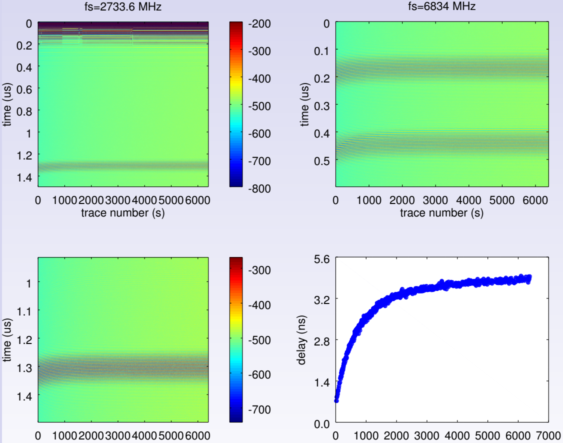
Timebase stability

Stroboscopic signal generation and cause of drift

Static environment, **controlled** temperature and **fixed** sensor – time delay as cross correlation between two echo signals:



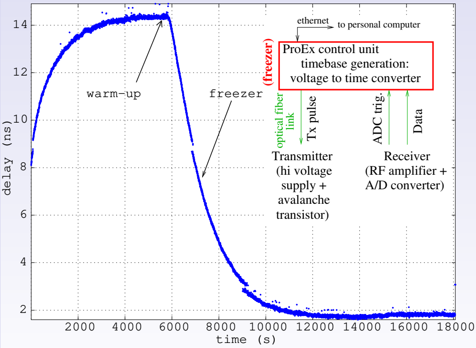
- Two echoes (differential measurement) separated by 300 ns, at 1.3 and 1.6 μs
- measure time delay (cross correlation) between echoes in a stable environment

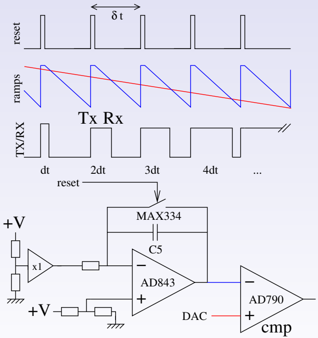
5 ns drift at 300 ns delay = 1.5 %=15000 ppm $\gg$ 200 ppm mass sensitivity or 70 ppm/K T sensitivity

Temperature dependence of the timebase

Stroboscpic signal generator [1] with an integrator of a constant voltage: voltage to time converter

$$f_s = 1/dt = \frac{B}{A}\frac{1}{\delta t} = \frac{V_{supply}}{\tau_{RC}} \cdot \frac{\tau_{slow}}{V_{slow}} \cdot \frac{1}{\delta t}$$





[1] B.A.T. Johansson, *Ground Penetrating RADAR array and timing circuit*, Patent US 6496137 (2002)

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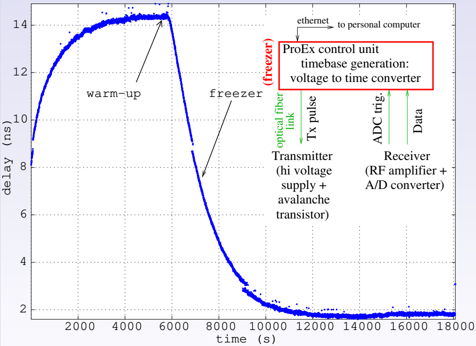

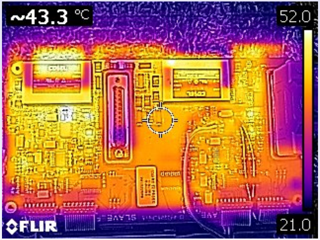

Operational amplifier rises to $>$ 50°C, inducing offset and drift in surrounding passive components

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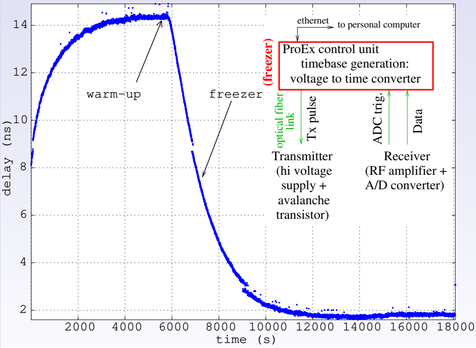

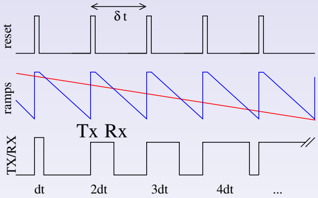

The (user controlled) slow ramp is generated by a Digital to Analog converter (voltage ref + **quartz** synchronized), little risk of drift

⇒ what about the **fast ramp** generation ?

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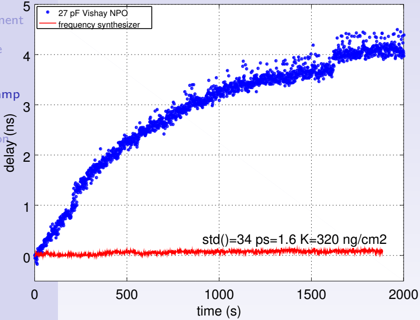
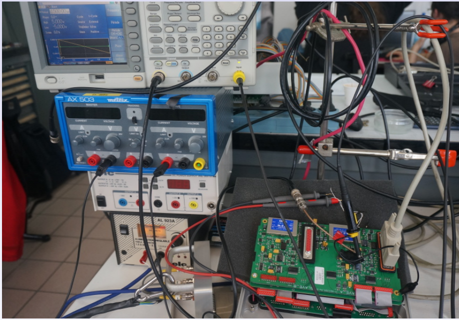
Solution: replace analog timebase with digital timebase

Solution: replace analog timing generator (drifting integrator capacitor) with digital ramp generator ⇒ 100-fold improvement (4 ns → 34 ps)



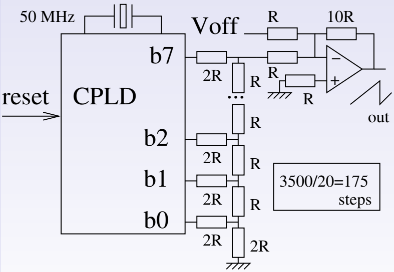


Tektronic arbitrary waveform generator configured for a ramp ranging ±5 V in 3.5 μs, triggered by integrator reset signal.

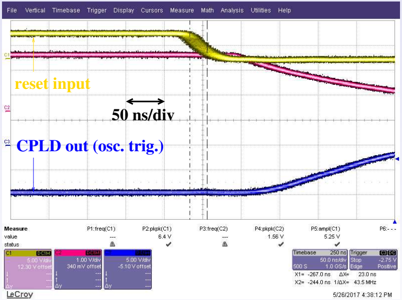
Lab-based, not compatible with field operation ⇒ embedded solution ?

Solution: replace analog timebase with digital timebase

- Replace laboratory equipment with embedded electronics: FPGA or microcontroller triggers on reset signal and generates ramp
- not so obvious ... 8-bit (256 steps) within 5 μs=50 MHz DAC
- R-2R network on FPGA output
- **different clocks** for FPGA and GPR $\Rightarrow$ jitter in reset detection



Analog is asynchronous, digital **must** be synchronous



$\pm$20 ns when FPGA is clocked with 50 MHz oscillator

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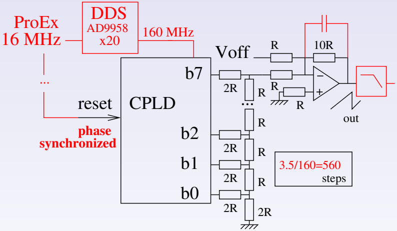
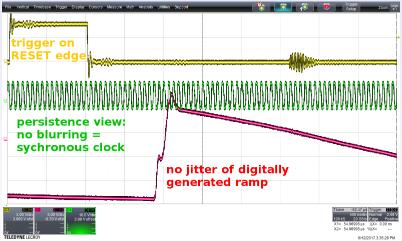
- clock synchronization on reset signal: use the GPR 16 MHz reference signal (quartz referenced = $\pm$ ppm/K)





Ramp synchronized with clock, but 160 MHz emission from DDS

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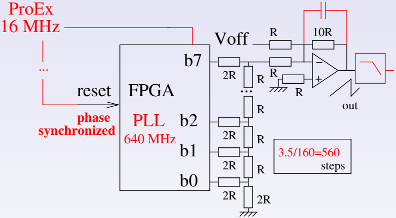
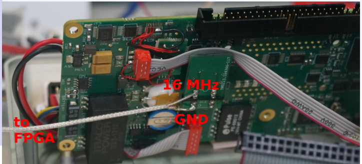
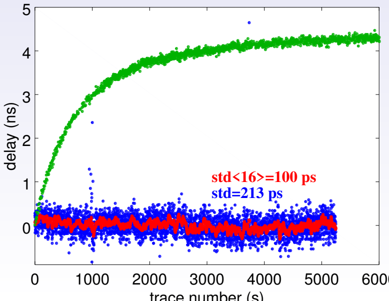


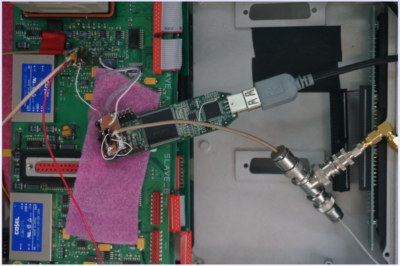




Conclusion and perspectives

We have demonstrated

- using surface acoustic wave (SAW) transducer as cooperative target and more broadly reference echo generator with $\geq \mu$s delay
- sensing capability of SAW transducers when functionalized with the appropriate polymer: H_2S detection yields X00 ps delay @ 200 MHz
- issue of **drift** of the sampling rate reference of the stroboscopic measurement ...
- ... solved by replacing the analog timing generator with a **quartz**-synchronized digital timebase.



Perspectives:

- Replace R-2R network with a "real" DAC with voltage reference
- investigate PLL jitter impact on sampling rate stability

Who else ? anyone using the phase, i.e. beam focusing/time reversal

Project repository: `sourceforge.net/p/proexgprcontrol/wiki/Home/`

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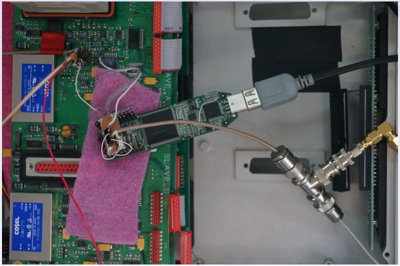

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