U.S. DEPARTMENT OF ENERGY | Office of NUCLEAR ENERGY

Clean. Reliable. Nuclear.

Direct Digital Printing of Passive Wireless Sensors for Nuclear Energy Applications

Advanced Sensors & Instrumentation Annual Webinar

October 30, 2019

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Oak Ridge National Laboratory

Accomplishments

- Collaboration - Met with Southern Company to define technology targets – November 2018
  - Sensor designs to measure hydrogen, voltage and current
  - Toured remote monitoring facility in Birmingham, AL
  - Discussed how to integrate new sensors with monitoring system
- Milestone 1 – Develop hydrogen sensors for detecting leaks on turbine generators – Successfully completed on March 2019
- Milestone 2 – Sensors operate in a representative field environment, collecting data autonomously – successfully completed September 2019
- Outcome - Developing sensor printing capability reduces design revision turn-around time from 10 days to same day
- Collaboration - Second visit to Southern Company to demo 4-sensor system – November 2019

Technology Impact

Passive Wireless Sensors Technology (PWST) represents a new paradigm for process and equipment monitoring. This project is developing and will commercialize:

- Direct digital printing methods and designs for PWSTs;
- Sensors that do not require a power source and can be easily replaced in case of failure;
- Physical and chemical sensing devices (hydrogen, CO_2, temperature, strain, voltage, current etc.);
- Ultra-miniature and easy to deploy sensors;
- The capability to more completely observe process and equipment state-of-health; and
- Advanced sensor technology that impacts the DOE mission of resilient, reliable and cost effective energy supply for the nation.

Project Overview - Administrative

- Goal: Demonstrate passive wireless sensors for on-line monitoring of critical assets in a nuclear power generation facility
- Objectives: 1) Develop direct digital printing technology for manufacturing ultra-low cost hydrogen, voltage and current sensors; 2) Deploy sensors on a power generation turbine for on-line monitoring
- Participants: Oak Ridge National Laboratory, Southern Company, Nuclear Division, and EPRI
- Schedule: This project includes a 24 month development campaign ending with a commercial demonstration

Project Overview – Technology

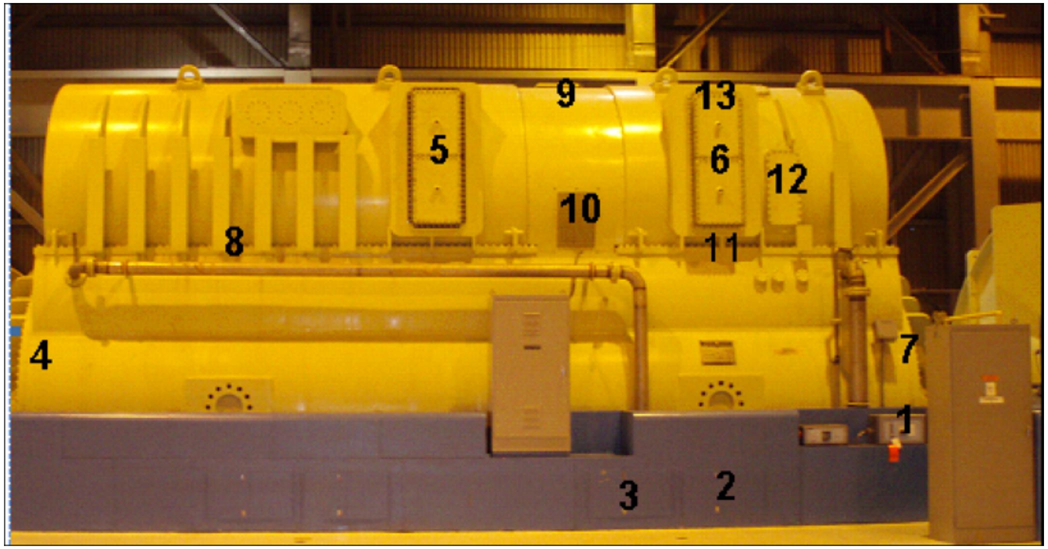
- Background
- Surface Acoustic Wave (SAW) sensor fabrication
- Orthogonal frequency coding (OFC); unique ID
- Antenna design & integration
- Interrogator hardware & software
- Signal processing
- Functionalizing coatings
- Sensor & interrogator packaging – Task not yet started
- The deployed sensor network – Performing initial demo at Southern Company, November

Passive Wireless Sensors

Background

- Commercial equipment exists for hydrogen detection but requires human operation.
- 4% hydrogen in air burns with deep blue to violet flame making it difficult to see.
- Accumulating hydrogen can be explosive (18% concentration in air), all it need is an ignition source (spark or flame).

GOAL
Direct digital printing of PWSTs that detect hydrogen, voltage and current



Steam turbine generator at utility power production facility. Numbered locations are inspected for H_2 leaks on a maintenance schedule.

BUT THAT IS NOT THE WHOLE STORY…
INVERTED FISH TANK???

Pushing the Limits of Additive Manufacturing

Background

The visionary premise is to print everything at the passive sensor.

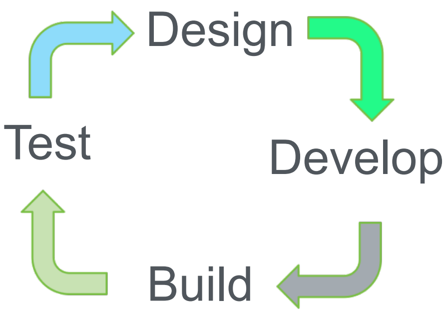
Some fundamental challenges to be overcome along the way, include:

- Printing the SAW sensor – spatial feature size
  - Then: 20μm feature size
  - Now: 1.0 to 5.0μm – need to go smaller
- Printing the antenna
  - Then: Only Redux of conventional antennas (10-20cm x 2cm)
  - Now: Dipole (10cm x 1cm); folded micro-patch (2.5cm x 0.5cm) @ 915MHz
- Printing the functional coatings
  - Then: None available
  - Now: Wide selection of solution, nano-particle and carbon nano-tube based inks chemical and physical parameters
- Printing the sensor package
  - Then: None available
  - Now: Concepts emerging – Sensors packaged in tradition AM printed structures, sensors embedded in components during manufacturing, or sensors printed on items (smart label), etc.

SAW Sensor Fabrication

It's not just Printing……..

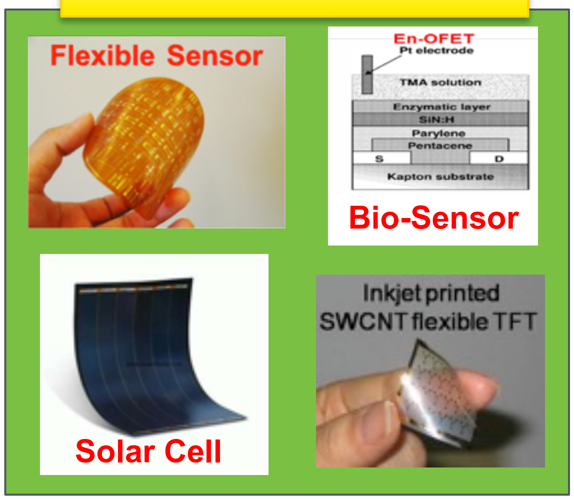
Multitude of Processing Tools Enable a Wide Palette for Functional Materials Additive Manufacturing (FM/AM)



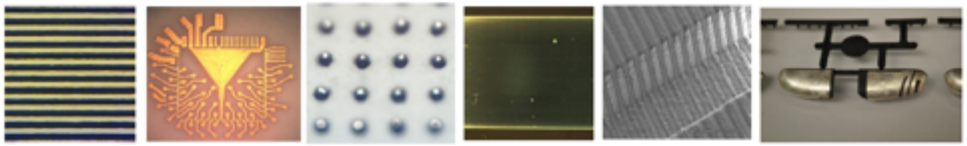
ORNL's R&D Platform

Materials	Printing Technology
Device Integration	Test & Measurements

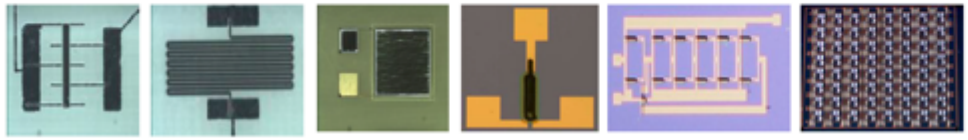
Advanced Devices



Fine Lines, Patterns, Bumping, Films, 2D/3D Surfaces



Building Blocks: Resistors, Capacitors, Inductors, Transistors, Memory

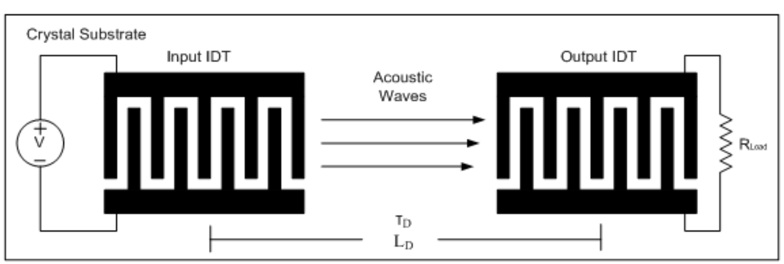


3D Micro-Structures (Developmental)

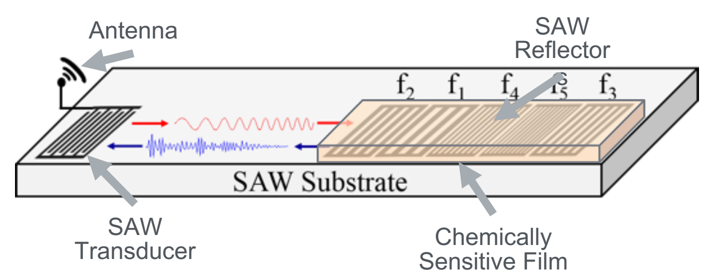
Rapid Thermal Processin

What is a SAW device?

- Solid state devices
  - Convert electrical energy into mechanical wave (and vice versa) on piezo-electric substrate
  - Very complex signal processing in small window <u>(Spatial mapping of time function)</u>
  - Acoustic Wave Velocity: ~3-4,000 m/s
- 4-5 Billion SAW devices produced each year (Probably More…)
  - Filters, Delay Lines, Resonators
  - **Sensors**, RFID
- One Port Device
  - Measure reflected signal (S_{11}); passive operation
  - Post-processing to determine how frequency, phase, delay of reflected signal is changing
  - "Cooperative RADAR target"
- 20MHz-3GHz Operation
  - Fabrication feature size determines sensor operating frequency
  - Fab capability also impacts antenna designs
- Variety of Device Embodiments
  - Resonator, delay line (narrow/wideband), reflective
  - Radiation hard



One port device, delay line



Figures Courtesy of Malocha et.al.

SAW Sensor Fabrication
continued

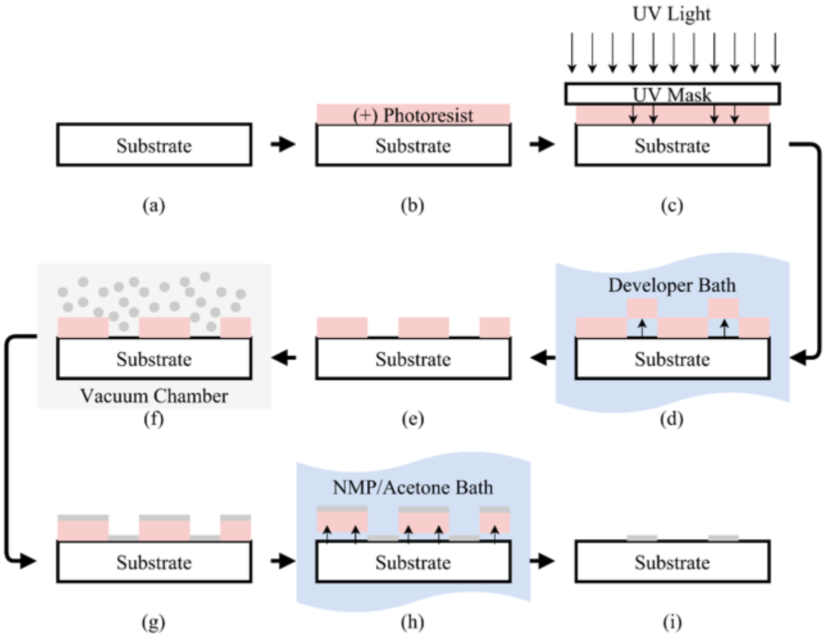
Started with the old fashioned way; photolithography

1. Create a ”gold standard” device platform against which direct digital printed sensors are compared.
2. Fabricate many identical sensor platforms used for functionalization development.

1. Dehydration Bake
   a. Hot Plate, >100°C
2. Spin Clean Wafer
   a. Acetone
   b. IPA
3. Spin On Photoresist
   a. Resist: 955CM-0.7
   b. Ramp: 15s @ 500 RPM
   c. Spin: 40s @ 4000 RPM
4. Soft Bake
   a. 90s @ 100°C
5. Expose
   a. Aligner Mode: Vacuum Contact
   b. Intensity: 20 mW/cm^2
   c. Time: 2.2s
6. Post Expose Bake
   a. 90s @ 110°C
7. Develop
   a. Developer: CD-26
   b. Time: 45s
8. Rinse
   a. DI Water
   b. N_2 Dry
9. Metallization
   a. Titanium (Adhesion Layer)
      6. Target: 100Å
   b. Aluminum
      6. Target: 800Å
10. Lift-Off
    a. Ultrasonic Acetone Bath

Observations:

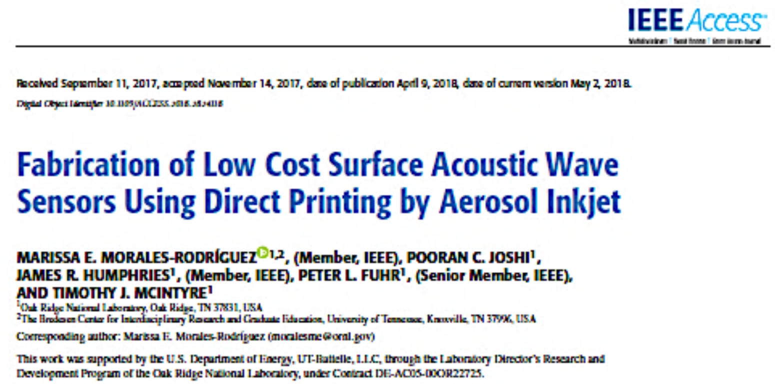
- Photolithography is <u>complex</u> multi-step process
- Many copies made <u>economically</u>
- A custom and <u>expensive shadow mask</u> must be fabricated
- <u>Multi-million dollar fab:</u> 100:1 vs. printing



SAW Sensor Fabrication
continued

Direct Digital Printing (DDP) of SAW sensors to characterize print quality and sensor performance

1. Print test structures
2. Examine print quality and repeatability of critical SAW sensor elements
3. Perform interrogations to evaluate response
4. Determine operational frequency limits
5. Develop testing hardware to characterize SAW sensor operation.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Fabrication of Low Cost Surface Acoustic Wave Sensors Using Direct Printing by Aerosol Inkjet

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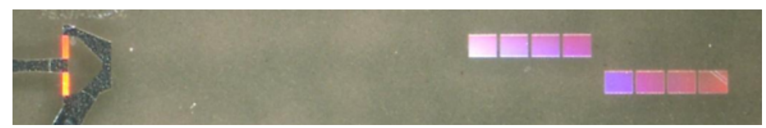
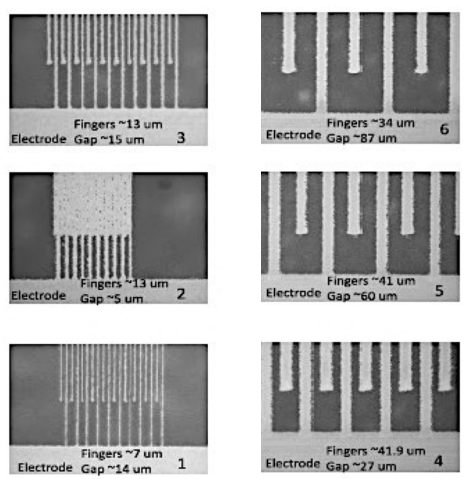


Our test structure employs (4) Bragg reflectors for unique ID

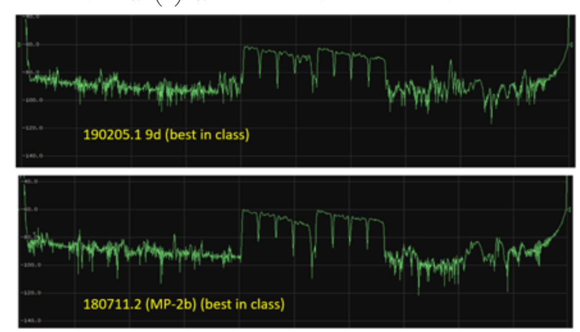
Observations:

- SAW sensors printed with <u>10µm feature size</u>
- Unlike photo-lithography, SAW sensor printed in <u>single step</u>
- Multiple sensor types printed with <u>no change in setup</u>
- Current printing capability produces 20-250MHz sensors



Simple structures to examine print quality.

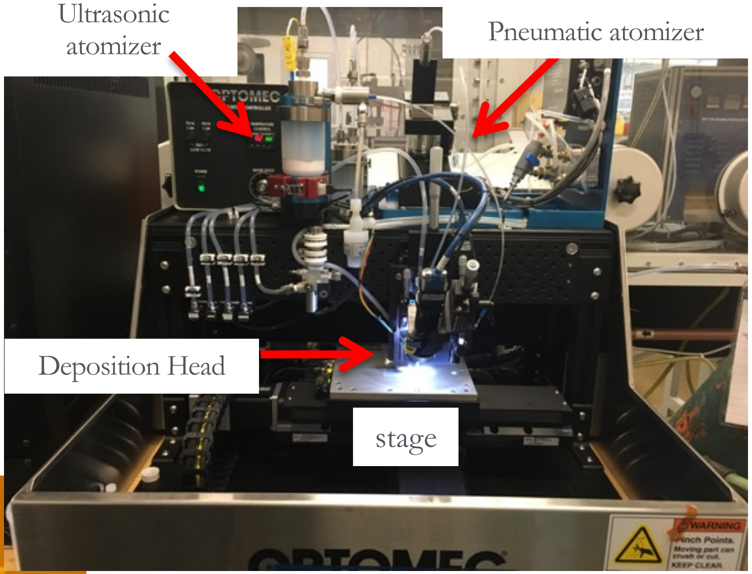
Data show (2) well formed reflections



SAW Sensor Fabrication
continued

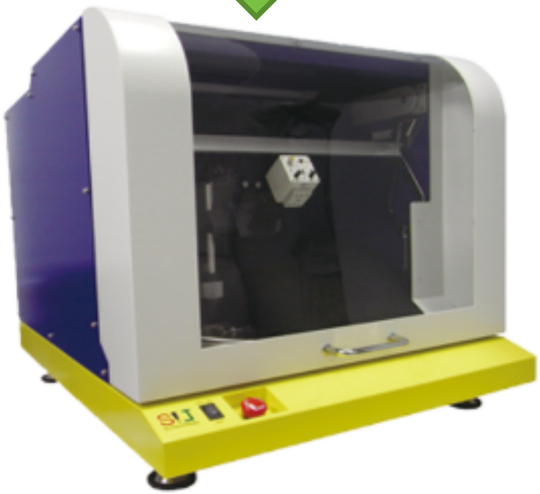
Our Workhorse

Optomec aerosol jet printer has ultrasonic and pneumatic modes
Feature size ~3-5µm



SIJ Technology, super ink jet printer.
Feature size limit ~1.0µm?

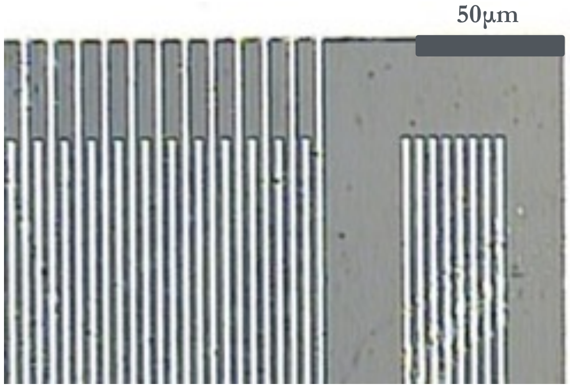
Heidelberg laser patterning system with feature size limit 0.6µm



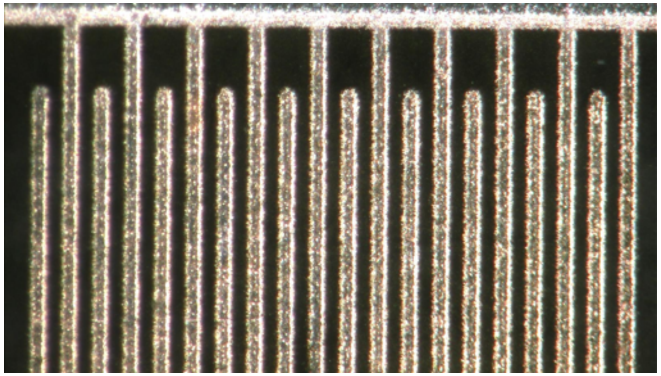
SAW Sensor Fabrication

continued

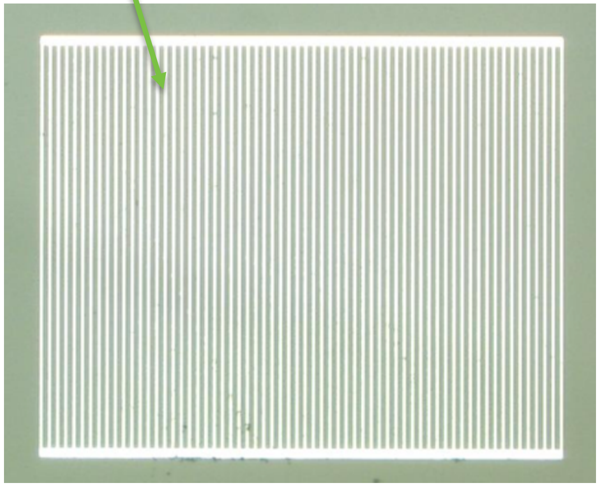
Benchmark device using photolithography



Aerosol jet printed Bragg reflector with ~3μm width/pitch



Super ink jet printed IDT with ~2μm features



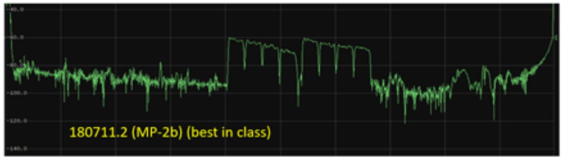
Approximately 0.8μm line width and pitch using photolithography.

Transitioning from photolithography, to mask-less laser scribe, and finally to direct digital printing (DDP) of components.

Orthogonal Frequency Coding Unique ID

What does Orthogonal Frequency Coding (OFC) require from a printing perspective?

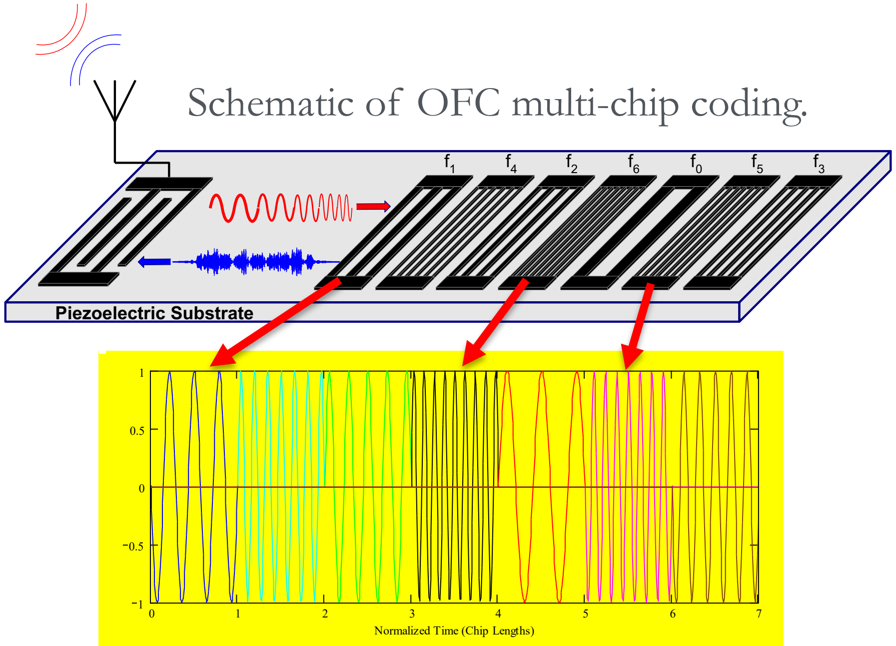
- Time diversity - printing structures (chips) with physical distance/geometry on device



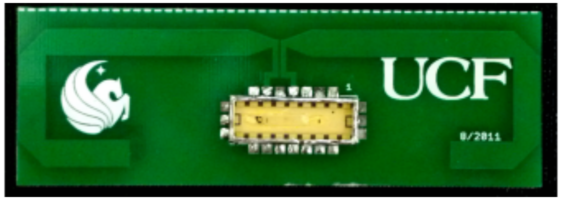
Dual channel device, 4 chips with time diversity, BUT each chip has a unique frequency also. Combining a series of unique frequencies creates an OFC ID.

- Frequency Diversity - chips can reflect anywhere within the 915MHz ISM bandwidth - 902MHz to 928MHz
- Wavelength change that occurs during sensing causes a related frequency shift
- OFC code chip frequencies require precise fabrication tolerances (<1µm @ 915MHz)
- Figure to the right shows train of chips with time diversity (physical distance delays the return signal) and frequency diversity (each chip has unique frequency)

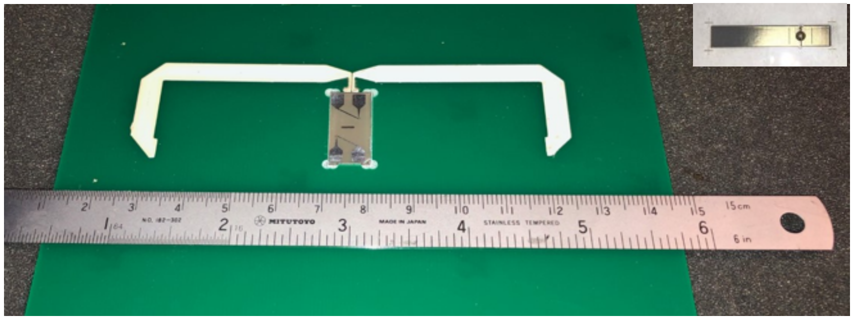
Schematic of OFC multi-chip coding.



Antenna Design & Integration

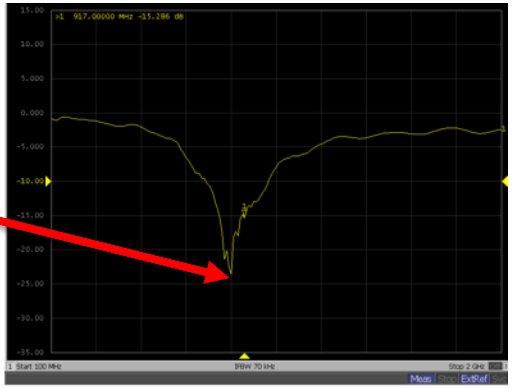


UCF 915MHz dipole antenna with integrated SAW device on FR4 board

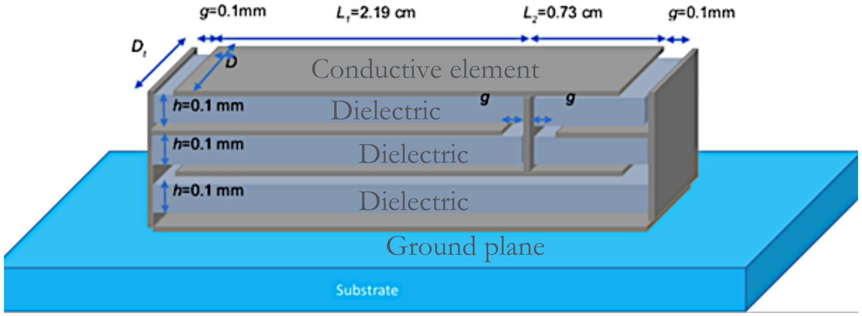


DDP quarter wave dipole antenna integrated with SAW sensor

~-20dB



Vector network analyzer frequency response sweep showing 99% efficient antenna



There are several feature size challenges to print this antenna, including:

- Conductive element thickness = 0.01mm
- Dielectric layer thickness = 0.1mm
- 7-later stack with multiple tolerances < 0.1mm and no lateral spillage (requires in-process curing).

Modeling Parameters for Folded Micro-patch Antenna

Folded Patch Antenna Dimensions	
Conductor thickness, t	0.01 mm
Dielectric height, h	0.1 mm
Gap, g (=h)	0.1 mm
Strip Width, D	5 mm
Ground Plane Width, D_t= (D+8h)	5.8 mm
Conductor length, L_1	2.25 cm
Conductor length, L_2	0.75 cm
λ=2x3(L_1+L_2)	18 cm
Dielectric constant, ε_r	4
Speed of light, c	3x10⁸ m/s

Antenna Frequency:

$f_0 = c/(\varepsilon_r^{1/2}\ \lambda)$

$= 3\text{x}10^8$ m/s /(2 x 0.18 m)

= 833 MHz

Antenna Frequency:

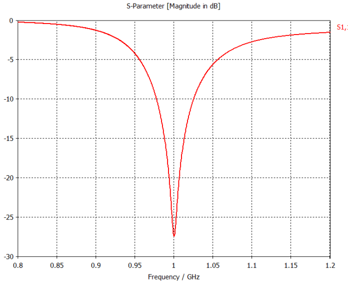
$f_0 = c/(\varepsilon_r^{1/2}\ \lambda)$

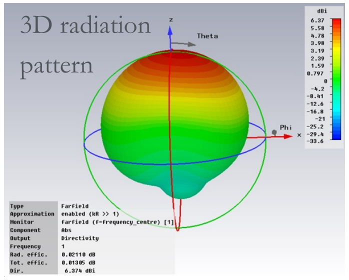
$= 3\text{x}10^8$ m/s /(1.87 x 0.1755 m)

= 914 MHz

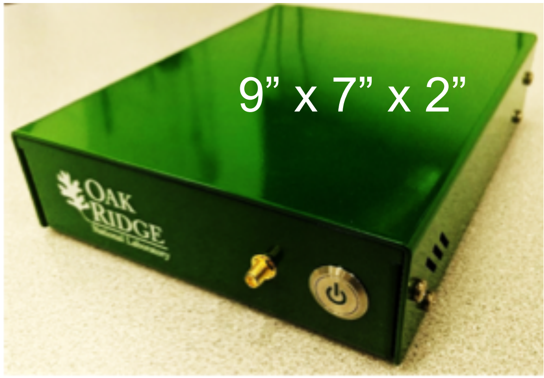
Folded Patch Antenna Dimensions	
Conductor thickness, t	0.01 mm
Dielectric height, h	0.1 mm
Gap, g (=h)	0.1 mm
Strip Width, D	5 mm
Ground Plane Width, Dt= (D+8h)	5.8 mm
Conductor length, $L1$	2.19 cm
length, $L2$	0.73 cm
+L2)	1.755 cm
constant, ε_r	3.5
ight, c	3x108 m/s

S1,1 parameter showing << 1% energy reflected





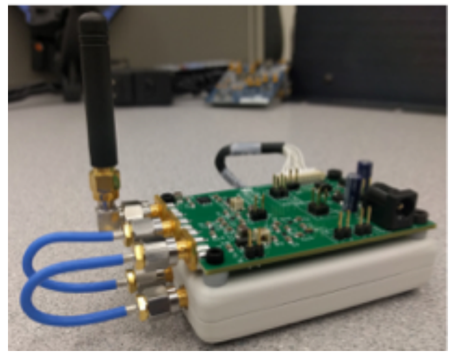
Interrogator Hardware & Software

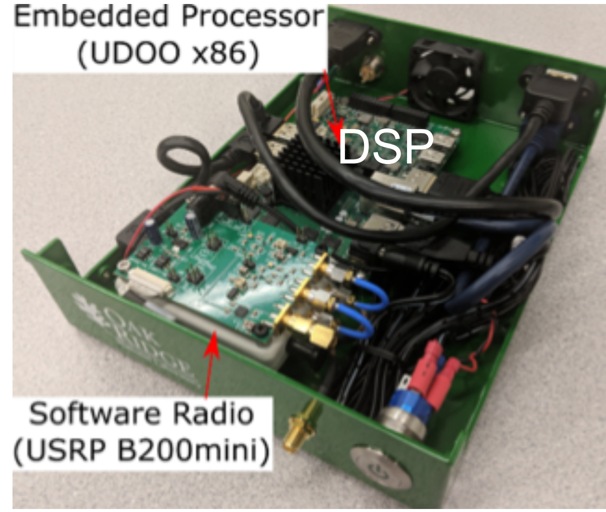


ORNL Custom Interrogator

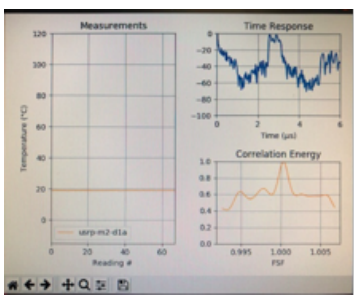
- Cost < $1k
- Portable/mobile
- Reconfigurable

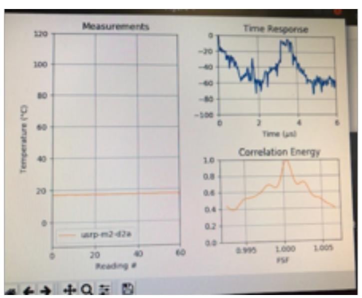
Software defined radio (SDR) interrogator

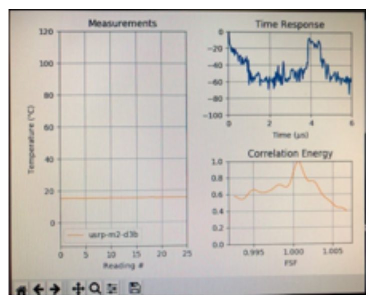




All commercial components + ORNL DSP (data-2-info) & communication to the enterprise network

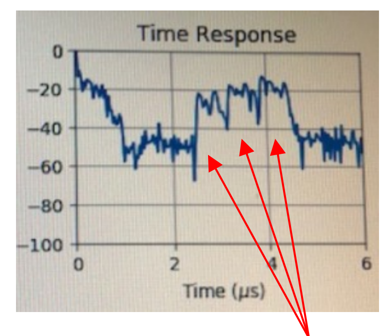






RF echo returning from (3) SAW sensors with time diversity

Outdoor testbed achieves 10m range with (4) sensors simultaneously; single sensor >20m. Currently operating at 5mW transmit power; can be increased >1W as needed for extended range and improved SNR.



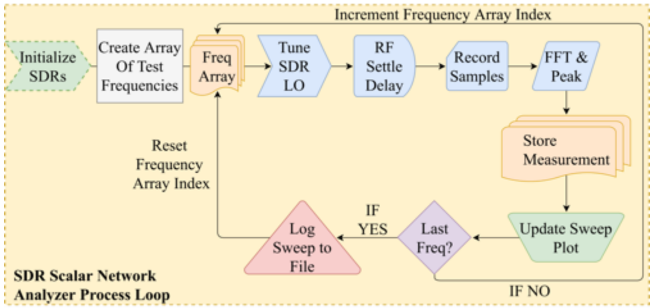
Simultaneous echoes from (3) sensors

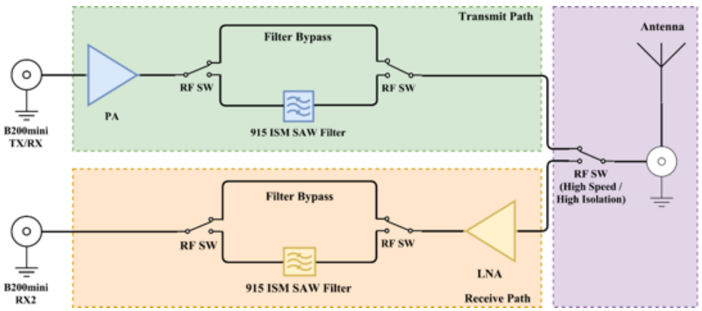
Interrogator Hardware & Software
continued

B200 Software Defined Radio (SDR)

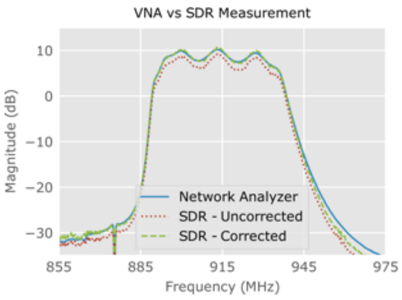
Single SDR for transmit (Tx) and Receive (Rx)

GNU Radio to generate tone and interact with radios via Python software

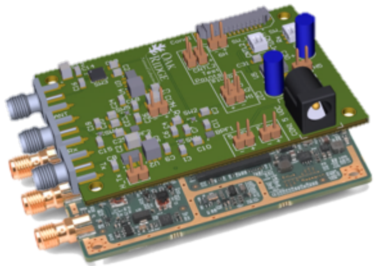




High-level block diagram of Tx/Rx



- For SAW sensing application, measurement range/precision driven by SNR
- B200mini Performance Can be Improved with External RF Hardware
  - Power amplifier → Increase output power
  - Low Noise Amplifier → Improve NF; amplify SAW response
  - TX/RX Switch → Single antenna; reduce self-jamming effects
  - Filters → TX harmonic suppression; RX out-of-band suppression



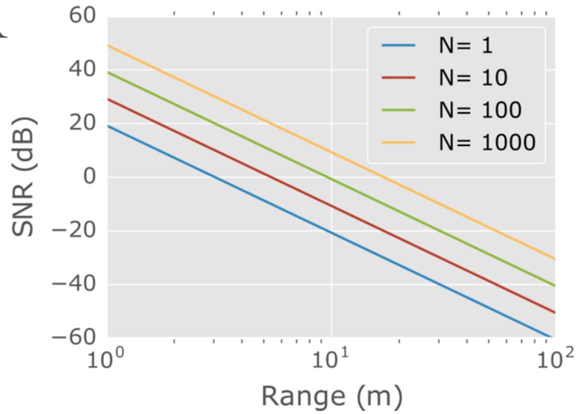
Ettus mother/daughter board SDR development platform

Signal Processing

PWSTs communication is often approached from a RADAR perspective, a pulse is transmitted, you listen for the echo.

Signal-to-Noise Ratio (SNR) is critical so you must exploit pulse compression gain, averaging, and others.

$$SNR = N\frac{P_t \cdot G_{SAW}^2 \cdot G_{INT}^2 \cdot \lambda^2 \cdot \sigma' \cdot \tau\beta}{(4\pi)^3 \cdot L_S \cdot F \cdot L_{SAW} \cdot R^4 \cdot kT_0B}$$



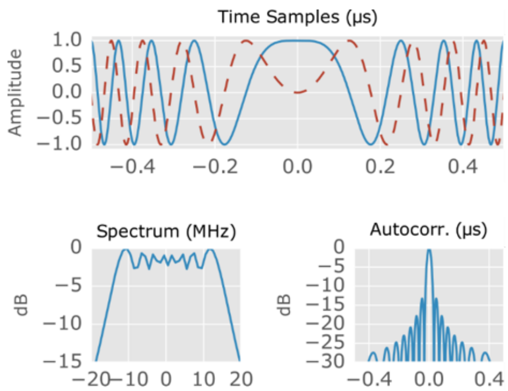
Chirp (Swept Frequency)

Advantages:

- Consistent frequency spectrum
- Many options for design (BW, windowing, chirp rate)

Disadvantages:

- Spreading of range bins in time
- High autocorrelation side-lobes



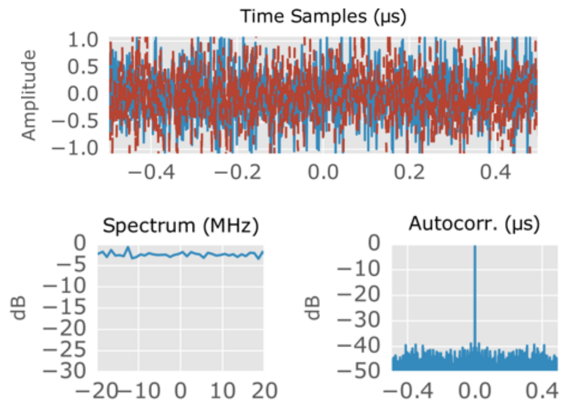
Noise (Random Modulation)

Advantages:

- Suppressed range ambiguity (multi-sensor environments)
- Reduced mutual interference (multi-interrogator environments)

Disadvantages:

- Many interrogation cycles for 'complete picture'
- Deconvolution of different TX signal for every cycle (slower processing)

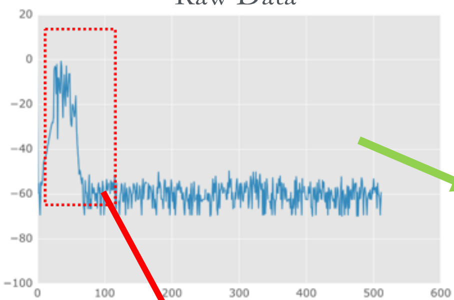


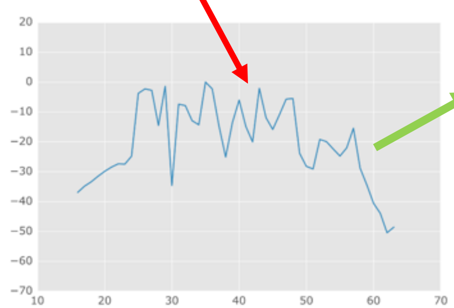
Signal Processing
continued

Complex Sequence of Signal Processing to Tease Out Echos

$$H_R(f) = [H_{TX}(f) \cdot H_{Tag}(f)] \cdot e^{-j2\pi f \tau_{EM}} + H_G(f) + H_J(f)$$

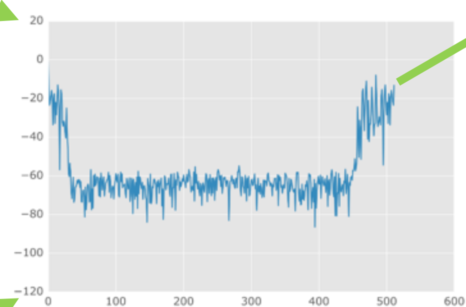
Raw Data





Gated TX Burst
(TX→RX Leakage)

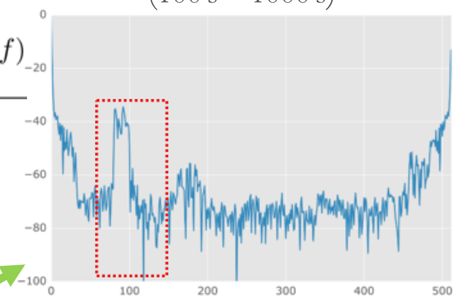
Deconvolve TX Signal

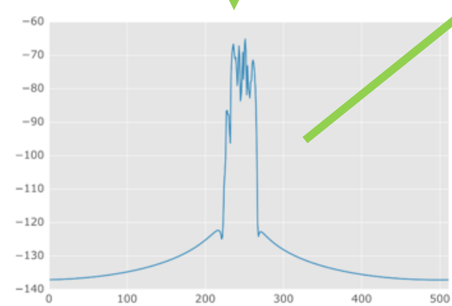


$$H_{dec} = H_R(f) \cdot H^*_{TX}(f)$$

$$H_{Ave}(f) = \frac{\sum_{i=0}^{N} H_{Rpp_i}(f)}{N}$$

Average Sweeps
(100's – 1000's)



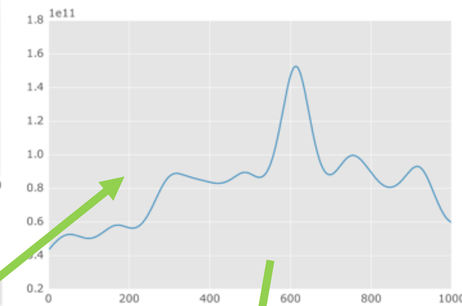


Apply Matched Filter Bank to Data

$$\begin{bmatrix} H_{R_{MF_1}}(f) \\ H_{R_{MF_2}}(f) \\ \cdot \\ \cdot \\ H_{R_{MF_N}}(f) \end{bmatrix} = H_{Ave}(f) \cdot \begin{bmatrix} H_{MF_1}(f) \\ H_{MF_2}(f) \\ \cdot \\ \cdot \\ H_{MF_N}(f) \end{bmatrix} \quad (2)$$

$$E_{Corr_i} = \int_{-BW/2}^{+BW/2} H_{R_{MF_i}}(f)df$$

Integrate and Find Closest Match

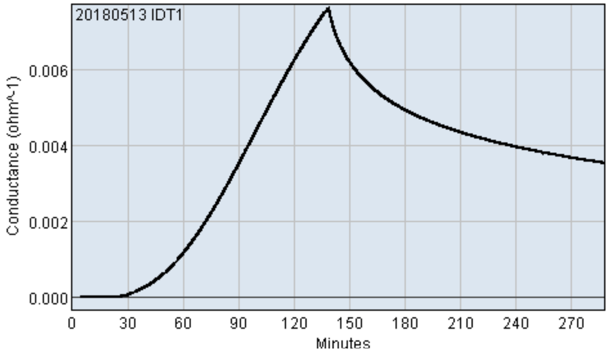


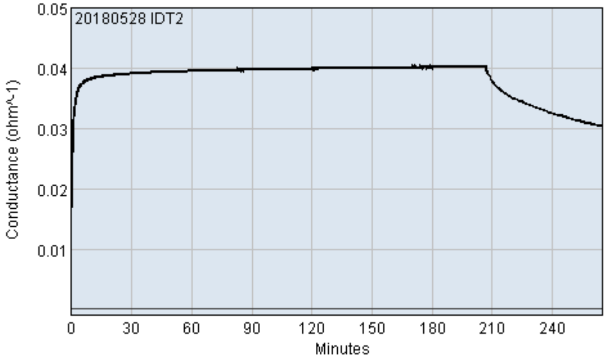
Relate Frequency/Time Shift to Sensing Measurement
Amplitude shift also works

Functionalization - Hydrogen Sensing

- **Recent Accomplishments**
  - Chemo-chromic (color indicating) material for hydrogen leaks tested
  - Hydrogen sensitivity - very strong response by resistance changes
    - > 10 MΩ without hydrogen
    - < 100Ω with 1% hydrogen (~10^6 change!)
- **Next Steps**
  - Determine calibration and response times for bare thin films
  - Test for cross-sensitivity with temperature, humidity, and other gases of interest
  - Further investigate time response and recovery

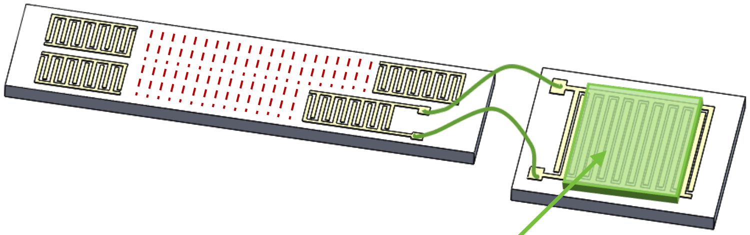
IDT Test Device



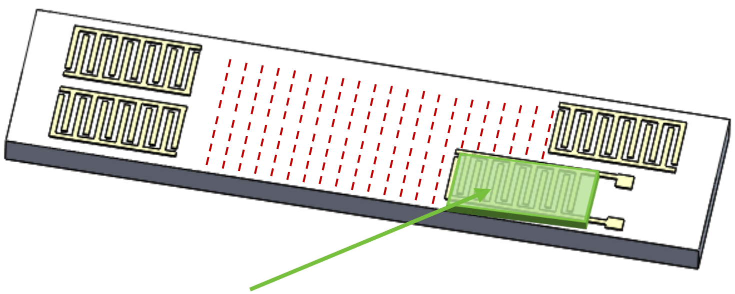


Functionalization - Hydrogen Sensing
continued

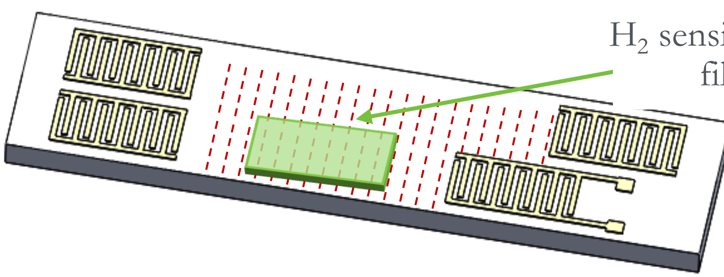
- **Next Steps, continued**
  - Step 1: Wire chemo-resistive H_2 sensor to SAW IDT reflector for electro-acoustic modulation
  - Step 2: Apply thin film directly to SAW reflector IDT for stress, mass and electro-acoustic modulation
  - Step 3: Apply thin film to delay line



H_2 sensitive thin film

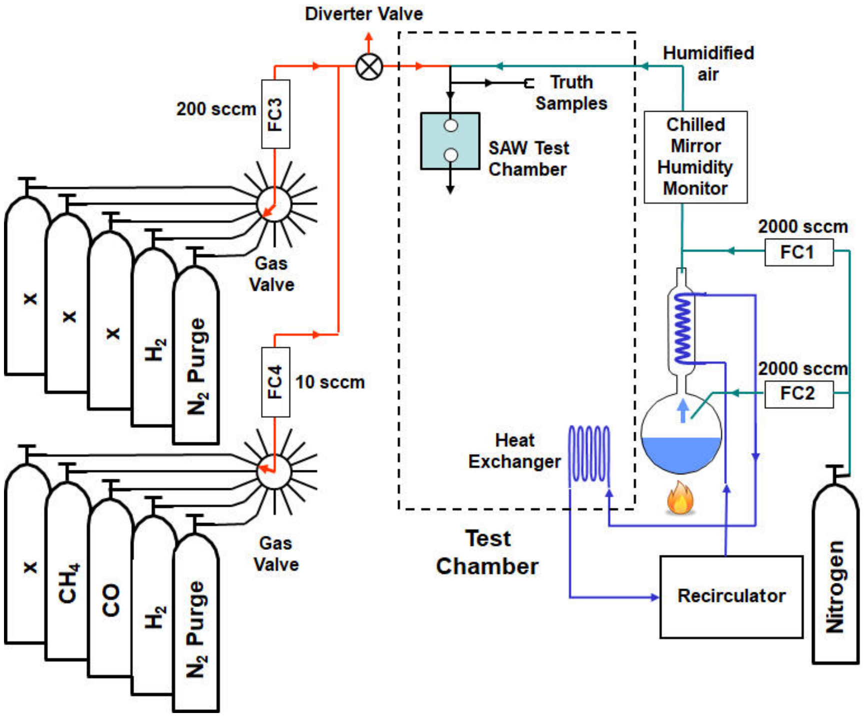


H_2 sensitive thin film

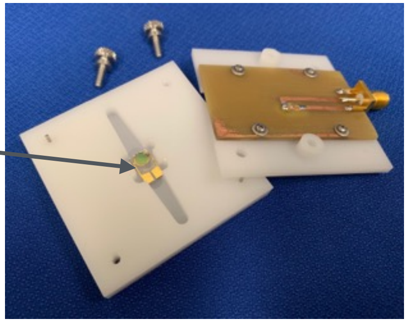


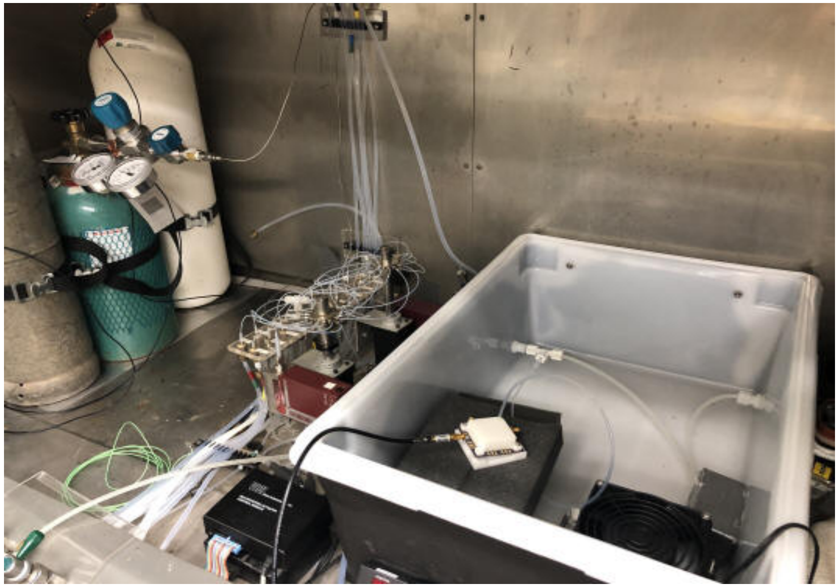
Functionalization - Hydrogen Sensing
continued

ORNL Test bench



Chemo-resistor chip





Automated testing 0.01% (100ppm) – 100% H_2
10°C – 40 °C
0% – 90% humidity

Summary

Advantages

- PWSTs (RF/SAW) sensors have no batteries;
- Can be economically produced by direct digital printing (aerosol jet additive manufacturing);
- Can be functionalized to sense many things (e.g. moisture, H_2, CH_4, CO, CO_2, C_2H_2, V, I, etc.);
- Can sense physical parameters without functionalization (e.g. temp., strain, pressure, etc.); and
- Also working on roll-2-roll printing for economic mass production

Limitations

- Limited range (100+ft.), so far, have not turned up the power yet
- Limited frequency due to existing commercial printing capabilities – rapidly evolving (~10µm feature size ≅ 40MHz)
- ORNL evaluating multiple fabrication approaches ≅ 1-5µm, functional up 433MHz - closing fast on 915MHz ≅ 0.8µm feature size

Clean. **Reliable.** **Nuclear.**