2018 Annual Review Meeting for Crosscutting Research

INTEGRATED HARSH ENVIRONMENT GAS / TEMPERATURE WIRELESS MICROWAVE ACOUSTIC SENSOR SYSTEM FOR FOSSIL ENERGY APPLICATIONS

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OUTLINE

I. Introduction

- Motivation: Gas Sensor Need for Operation in HT / HE

II. Methodology:

Microwave Acoustics Technology for HT / Gas Sensors

- Technology accomplishments & Methodology for Gas Sensors

III. Project Objectives

IV. Recap: Last Year Reported Progress

V. Project Progress & Current Experiments

VI. Conclusions & Acknowledgements

I. Introduction

Motivation

➢ **High Temperature Gas Sensor FOSSIL FUEL: WHY?**

❖ **Better process control ⇒ ↓ maintenance ⇒ ↓ <u>POWER PLANT DOWNTIME</u>**

- Gas PP ⇒ Cost $11,000/h ⇒ $264,000/day (KCF Technologies)
- Average Outage (2007/11) Coal Units alone (NETL / Krulla 2014) →
  - ✓ Btwn 300 - 500 hours/unit-year ⇒ Over 40 M$ (coal units alone)

Motivation

➢ **High Temperature Gas Sensor FOSSIL FUEL: WHY?**

❖ ↑ **EFFICIENCY in fuel burning by controlling combustion**

- 1% Heat rate improvement (500MW) (NETL / Romanosky 2015) ⇒
  - ✓ $780,000/unit-year;
  - ✓ Entire coal-fired fleet $340 million/yr coal cost savings
- 1% increase in availability (500MW) ⇒
  - ✓ 44 Million kWh/yr added generation ⇒ ↑ 2.6 M$ /unit-year in sales
  - ✓ More than 2GW additional power / yr from the existing fleet

❖ **Emission / Pollution?**

- 1% Heat rate improvement

  Cool fleet alone ⇒

  ↓ 13.8 billion metric tons CO_2/yr

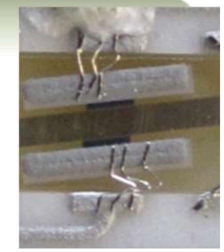
REQUIREMENTS / NEEDS

- GAS SENSORS capable of HTemperature Harsh-Environment oper.
  - Operate **RELIABLY** with very little or no wires
    - ✓ Wiring poses problem for reliability in harsh environments
    - ✓ Packaging restricts the use of several technologies
  - **NO MAINTENANCE** (inaccessible locations: no wires; no packaging deterioration; no replacement)
  - Sensor → **STABLE** in the environment over **LONG PERIODS**
  - **NO Battery**
    - ✓ Frequent maintenance
    - ✓ Limited to 500°C
    - ✓ Size restriction
    - ✓ Safety impediment for several applications
    - ✓ Compromise system operation and reliability

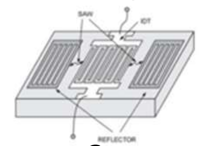
II. Methodology

Methodology

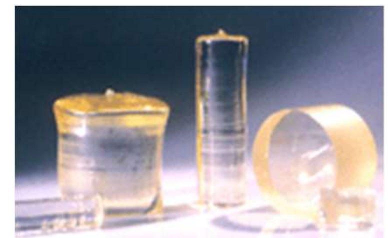
- μ~ acoustics → resilient platform for HT operation



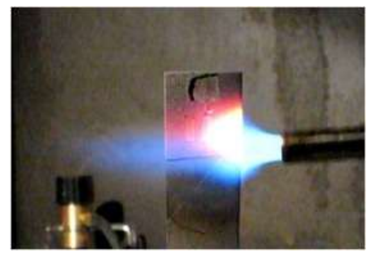
- Surface Acoustic Wave devices →



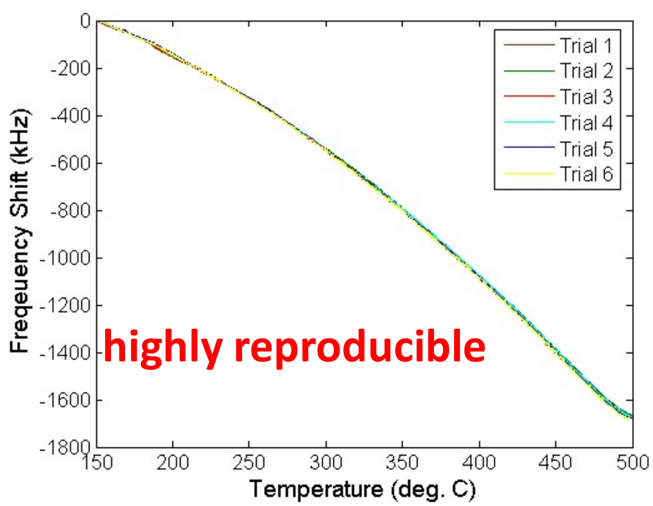
  - Platform developed & improved @ UMaine for over 17 yrs
  - Langasite $La_3Ga_5SiO_{14}$ Piezoelectric Crystal

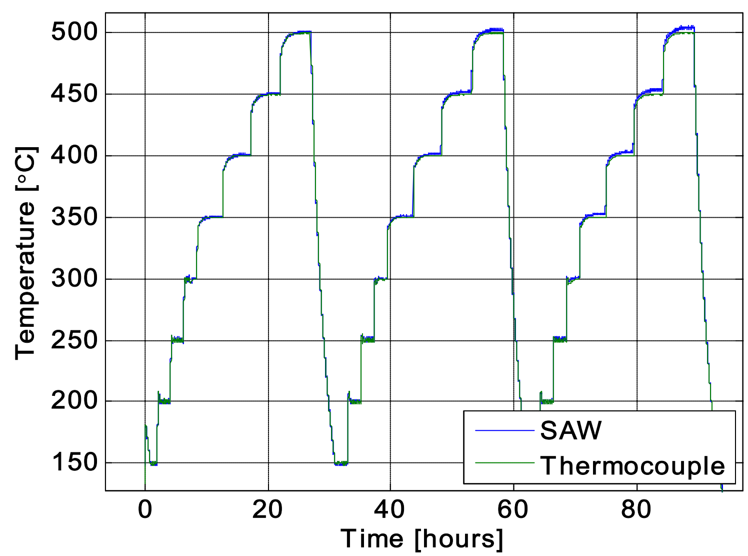


    - ✓ Stable up to 1400°C
    - ✓ Resistant to thermal shock

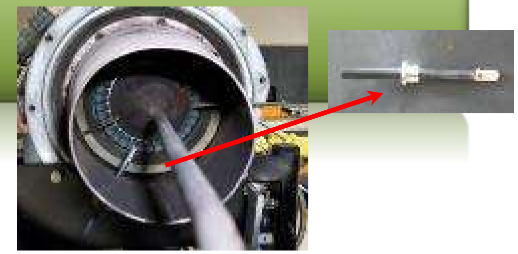


  - Stable / Repetitive operation
    - ✓ Tested over 5 ½ Mo @ 800°C

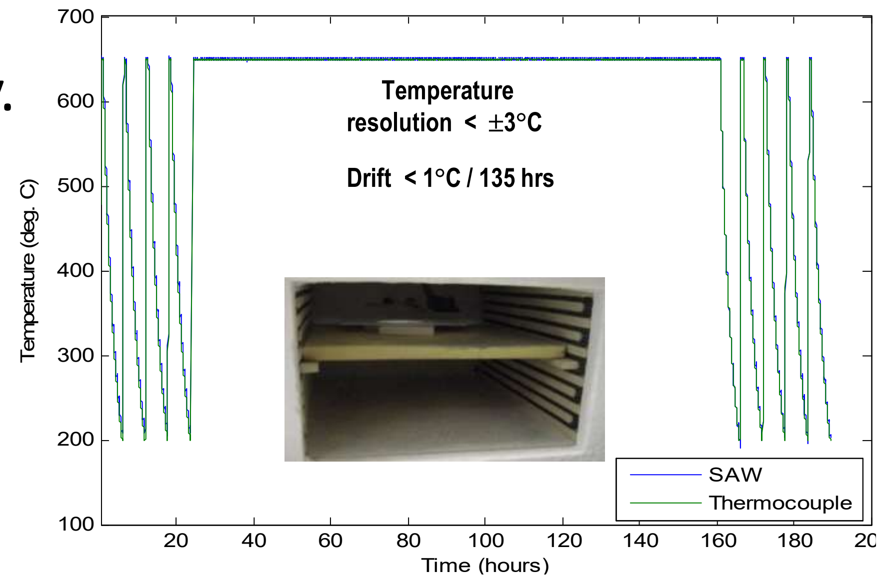


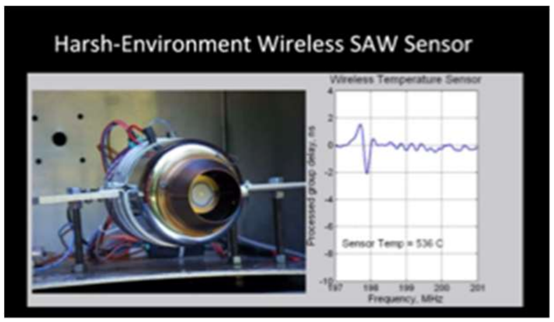


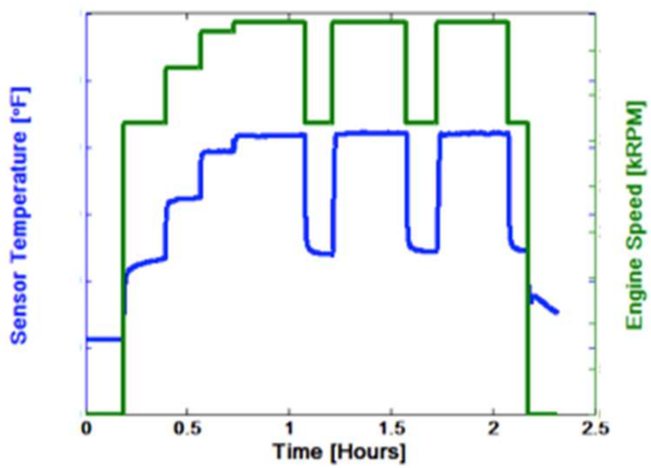
Methodology



- Surface Acoustic Wave T SENSORS →
  - Allow WIRELESS operation
  - Tested in multiple HT/Harsh Env.
    - ✓ **Sensor Turbines**



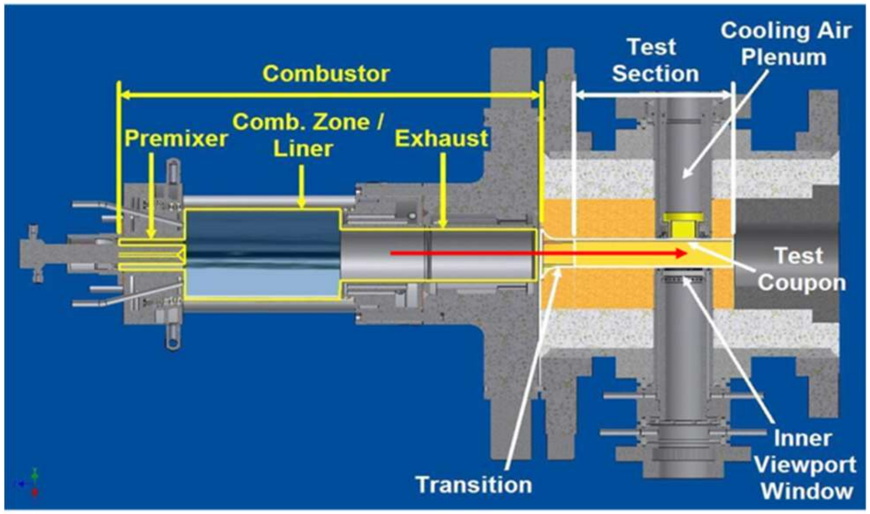




Methodology

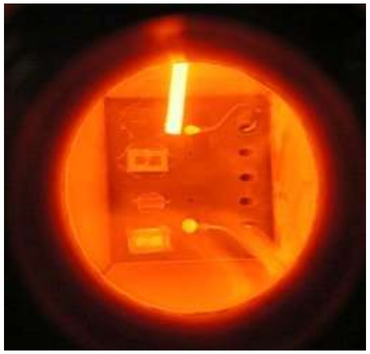
➢ Surface Acoustic Wave Temp. SENSORS (cont.) →

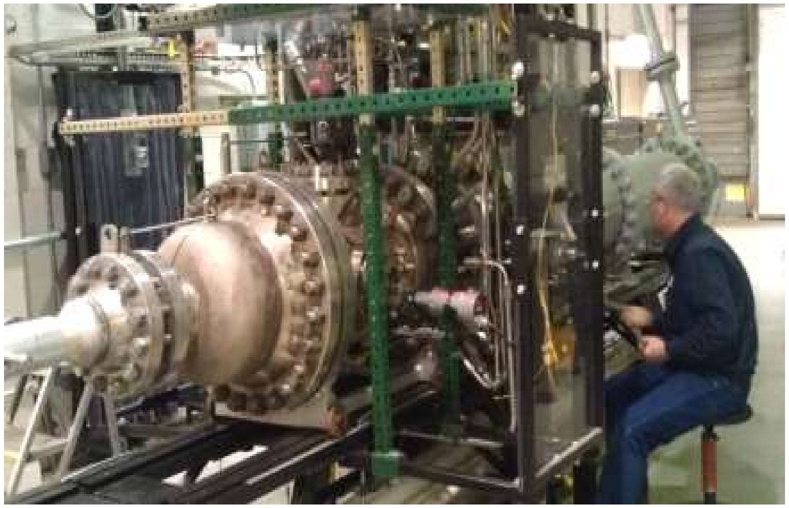
- WIRELESS operation
- Tested in multiple HT/Harsh Env.
  - ✓ **NETL Aerothermal Facility**



Sensor Performance Tests

- Sensor operation demonstrated in a combustor environment
- Multiple wired and wireless sensor designs tested up to 1100°C gas temp.
- All sensors survived entire test





Methodology

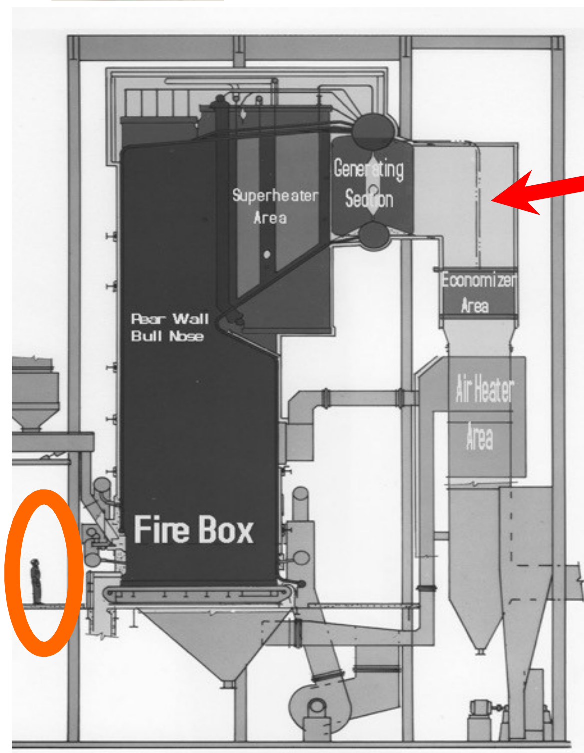
➢ Surface Acoustic Wave Temp. SENSORS (cont.) →

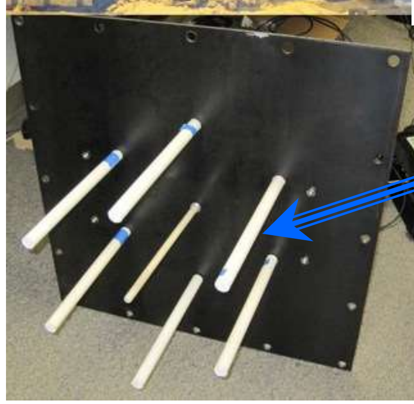
- WIRELESS operation → Tested in multiple HT/Harsh Env.

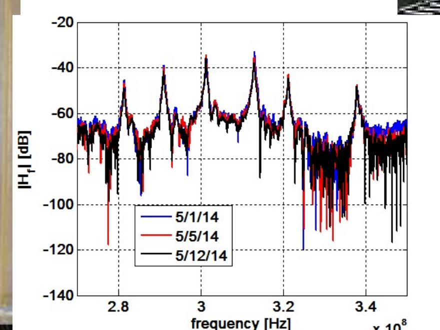
Penobscot Energy Recovery Company (PERC)

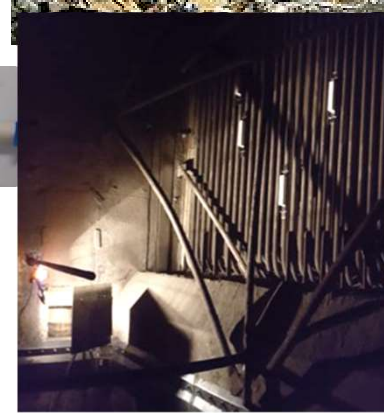
- Power plant: burns municipal **SOLID WASTE**

Installed in the boiler tubes → slag detection & removal









Methodology

- ➢ SAW → GAS SENSOR → **PLATFORM**
  - Provide **STABILITY** & **SENSITIVITY**
- ➢ For GAS detection:
  - **Selectivity**
  - **Retention** of gas in the sensor
- ➢ Selectivity:
  - For HT:
    - ✓ Addressed → arrays w/ ≠ films ⇒ Multi-dimensional signatures / sensor array training & learning
- ➢ Retention: To have a signature → Gas must be **detected**
  - At HT → gas @ ↑ energy level ⇒ film used to **RETAIN** the gas
  - In addition:
    - ✓ Other materials →used to **ATTRACT** the gas to sensor

III. PROJECT OBJECTIVES

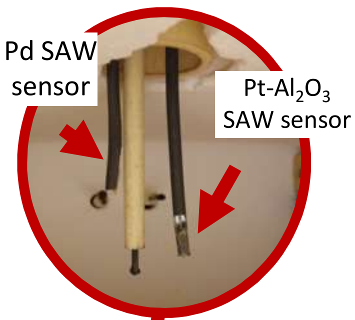
Project Objectives

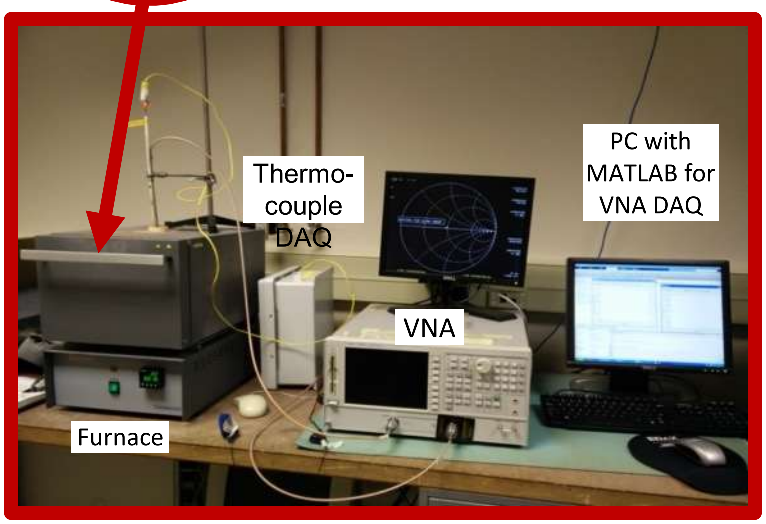
- Demonstrate → Performance μ~ acoustic sensor (SAW) for GAS SENSOR applications in power plant environments
  - Coal gasifiers, combustion turbines, solid oxide fuel cells, and advanced boiler systems
  - HT → in the range 350°C and 750°C
  - Passive operation
  - Targeting initially: detection of H_2 and O_2
- Major project targets:
  - Establish SAW gas sensor (platform + film) **STABILITY**
  - Establish adequate **RETENTION** for HT gas detection
- Thus functional sensor for long-term maintenance-free operation
  - @ power plant: ↑ **fuel burning efficiency**; ↓ **gaseous emissions**, and ↓ **maintenance costs & downtime** through condition-based monitoring

IV. RECAP: LAST YEAR REPORTED PROGRESS

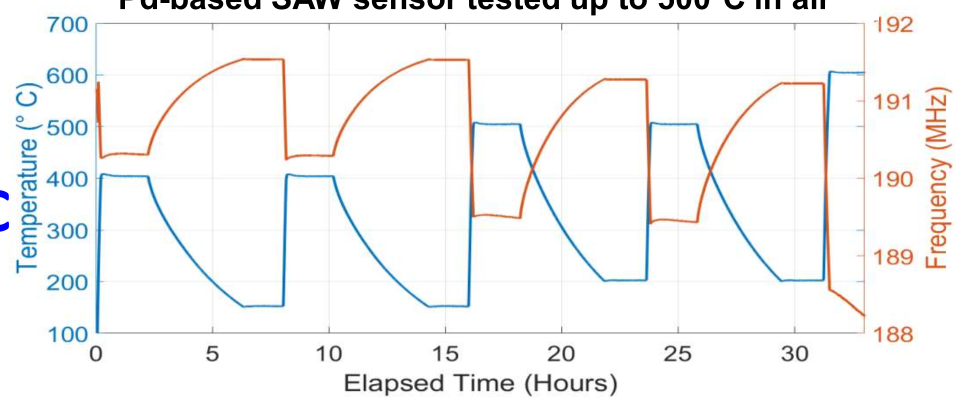
LAST YEAR REPORTED PROGRESS

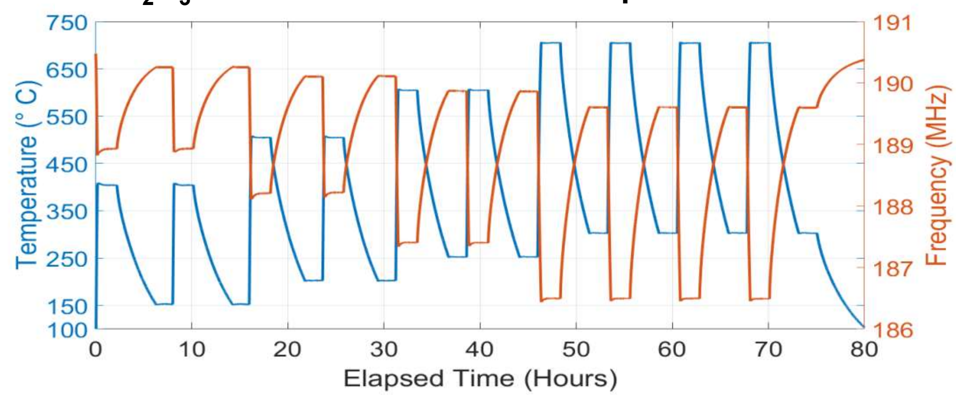
- SAWR platform development & testing for gas sensor
- Check stability of bare (no film) SAW sensor platform
  - LGS crystal with Pd & Pt-Al_2O_3 electrodes fabricated & tested





Stable platforms
Pd @ 500°C
Pt-Al_2O_3 @ 750°C





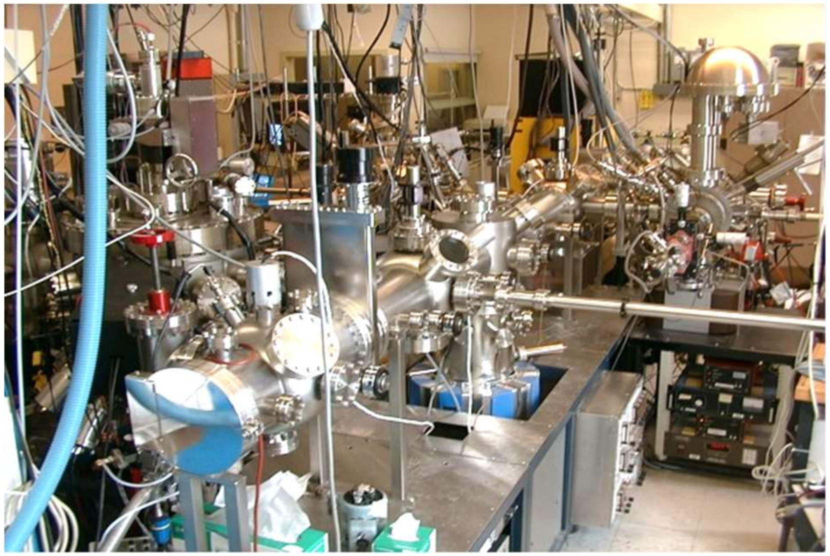
LAST YEAR REPORTED PROGRESS

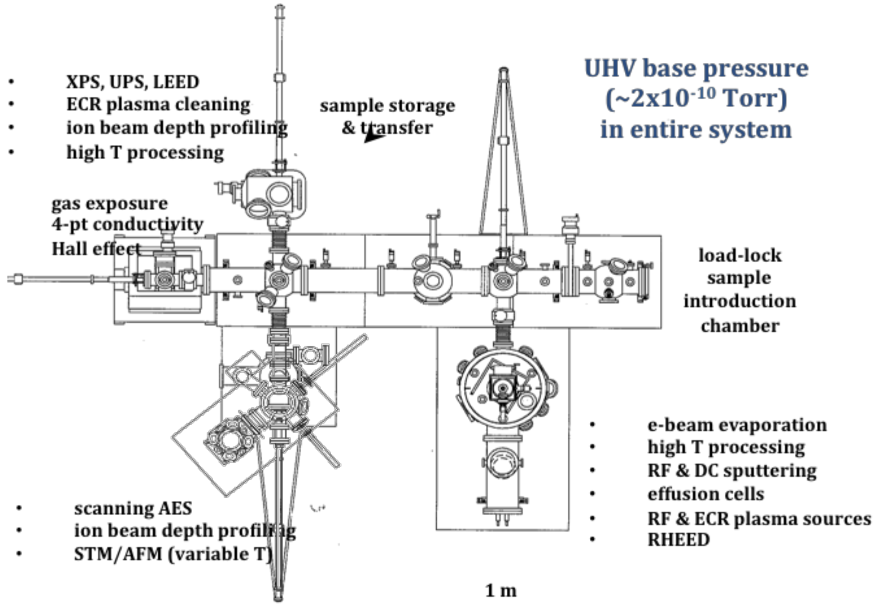
➢ In order to achieve the required gas RETENTION @ HT

- YSZ (Yttrium stabilized Zirconia) →
  - ✓ IYSZ film deposited initially on sapphire and then transitioned to LGS
- 15 to 30nm (reactive magnetron sputter deposition)

Photo & schematic:

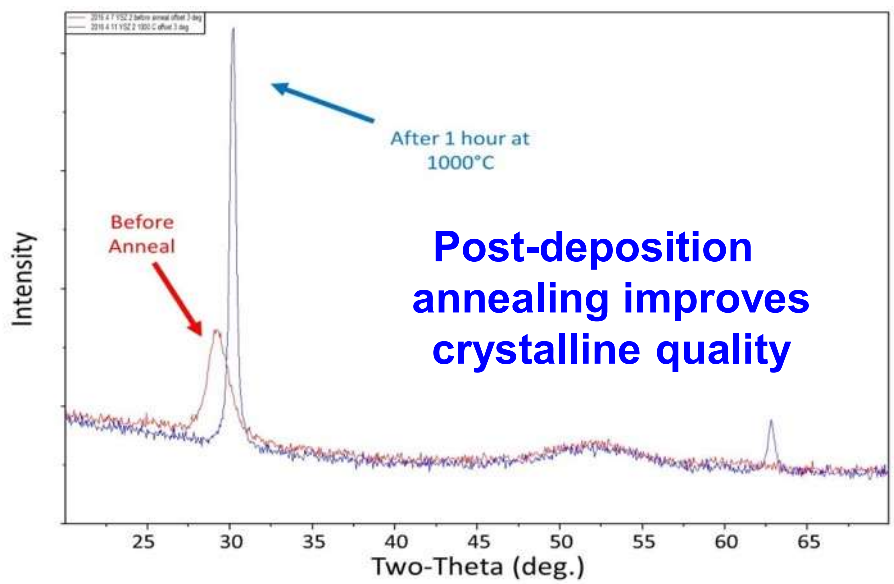
Thin Film Deposition, Processing, and Characterization Facility at the UMaine used to synthesize and analyze thin film materials for the SAW sensor devices

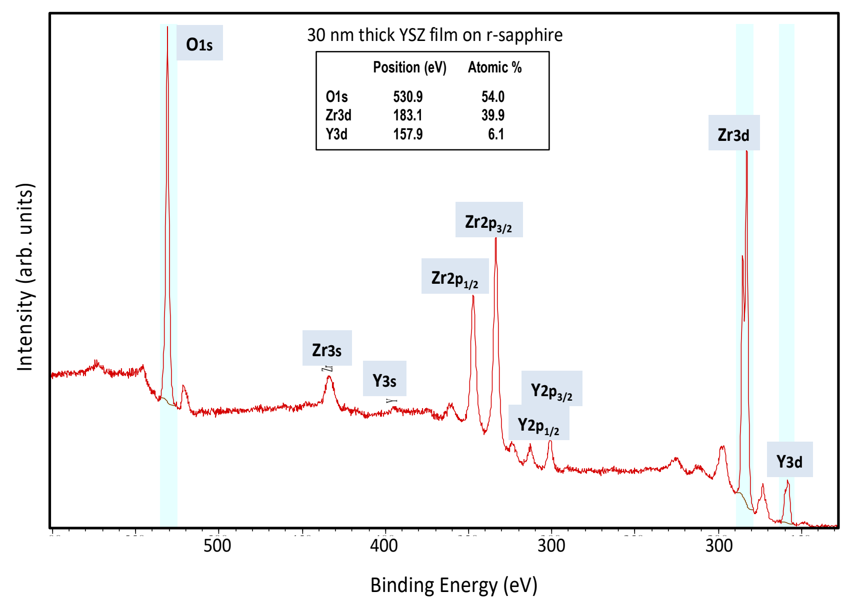




LAST YEAR REPORTED PROGRESS

- X-ray diffraction(XRD) & X-ray photoelectron spectroscopy (XPS)
  - ✓ 8%Y_2O_3-92%ZrO_2 film stoichiometry: film 65.9% O, 29.0% Zr, and 5.1% Y
  - ✓ Anneal 1000°C / 1h ⇒ ↑ crystalline quality





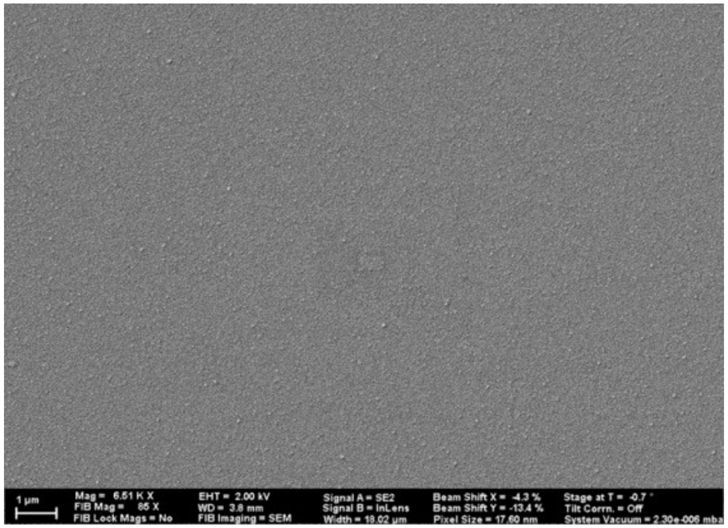
LAST YEAR REPORTED PROGRESS

- Stoichiometry: before & after 850°C 1hr
  - No detectable ≠ in stoichiometry

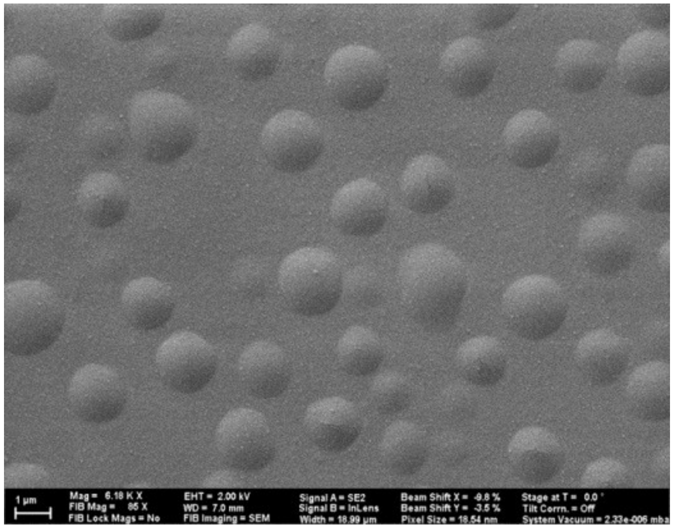
Sample	O %	Y %	Zr %
YSZ Unheated / LGS	53.8	6.3	39.9
YSZ Heated / LGS	53.2	6.4	40.4

- After heating 850°C 1hr ⇒ Bubbles (film unde stress)

→ ISSUE RESOLVED THIS YEAR



Heated 850°C
1h, vacuum
⇒

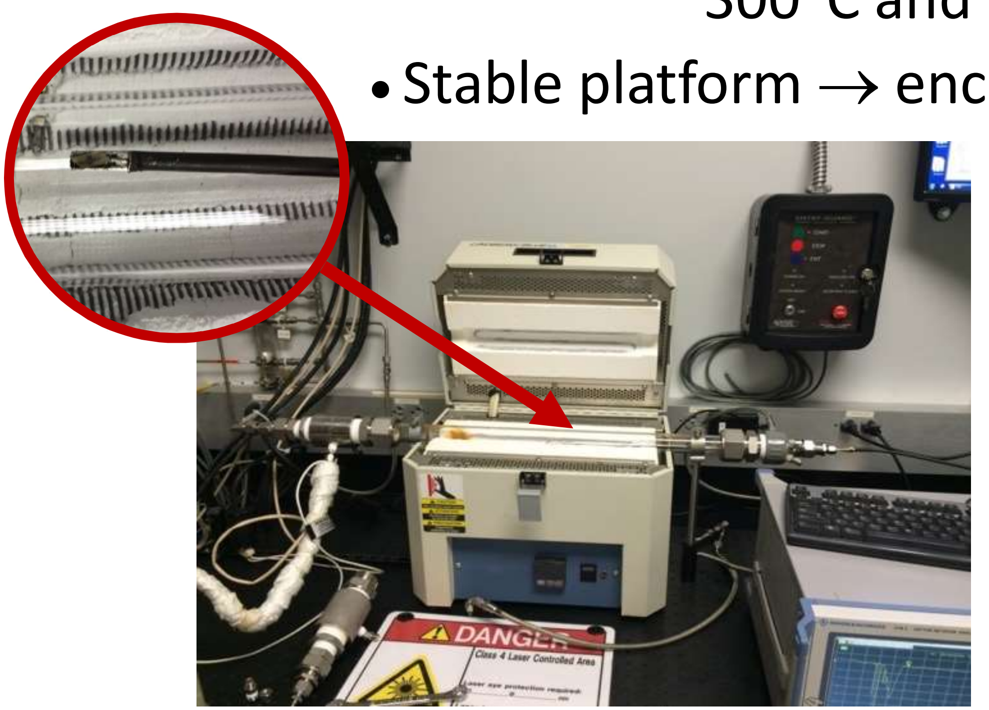


LAST YEAR REPORTED PROGRESS

- 2016 @ NETL/Pitts:

  1^{st} SAWRs platform test with 100% H_2

- Two days → Sensors exposed to:
  - 100% N_2, 5% H_2 in N_2, and 100% H_2
  - Room temperature, 300°C, and 500°C (Pd-based sensor) and 300°C and 700°C ($PtAl_2O_3$ - based sensor)
  - Stable platform → encouraging to develop gas detecting film



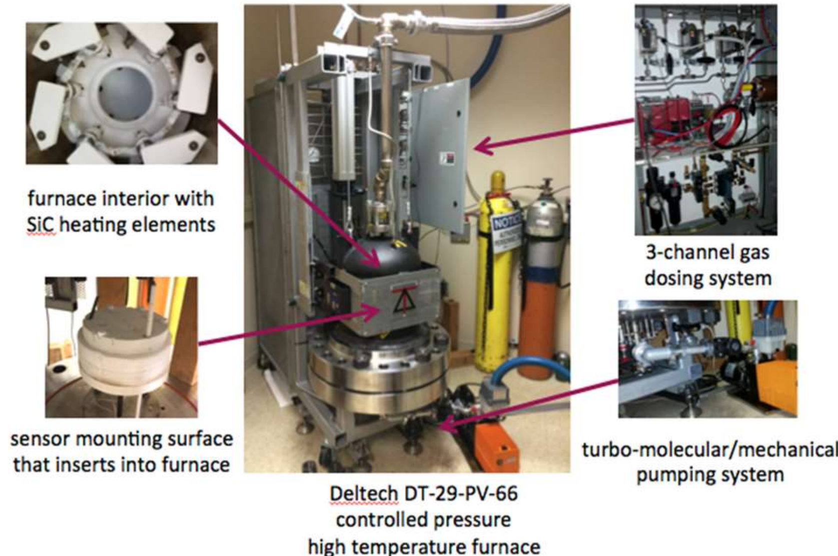
Test made in collaboration with: Paul Ohodnicki, Technical Portfolio Lead / Functional Materials Team & Robert Fryer, ORISE Postdoctoral Researcher

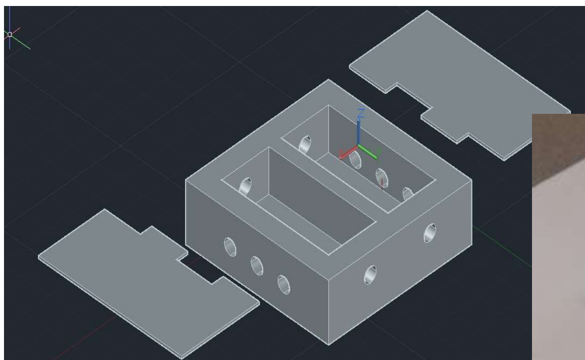
V. Project Progress & Current Experiments

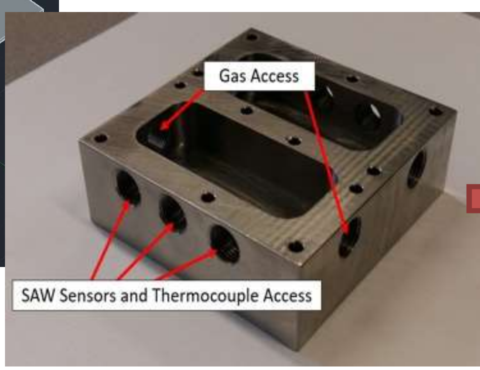
Project Progress & Current Experiments

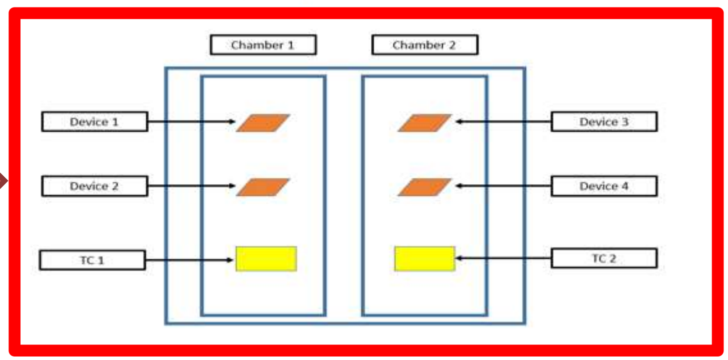
1) Gas Test System at UMaine

- High-Temp. High-Pressure DelTech DT-29-PV-66 Gas Furnace
  - ✓ Chamber: > 1 cubic feet ⇒ huge dead volume (time)
- Smaller chamber built
  - ✓ 2 chambers: (~1 in^3 ≅ 6.10^{-4} ft^3 each)
    - N_2 reference (temperature)
    - Gas sensing
    - Witness thermocouple access



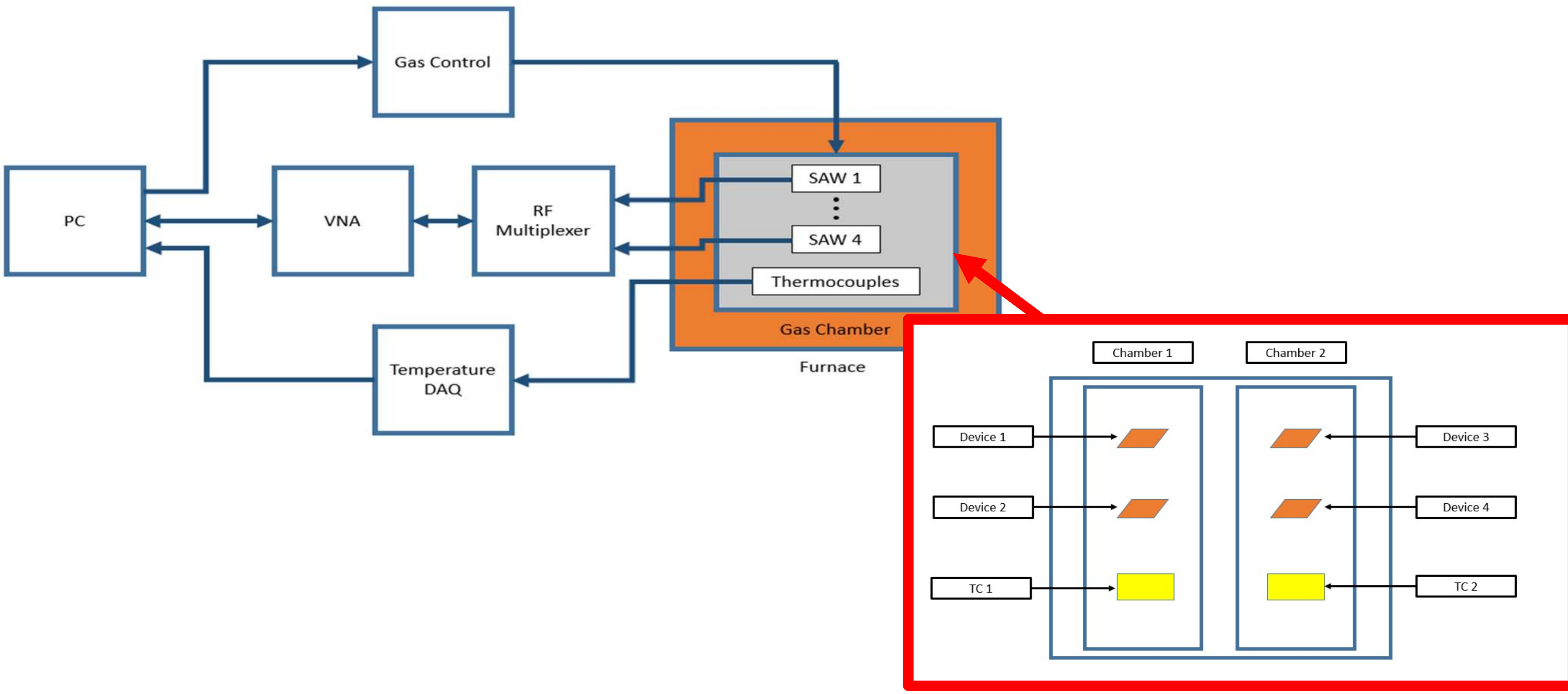






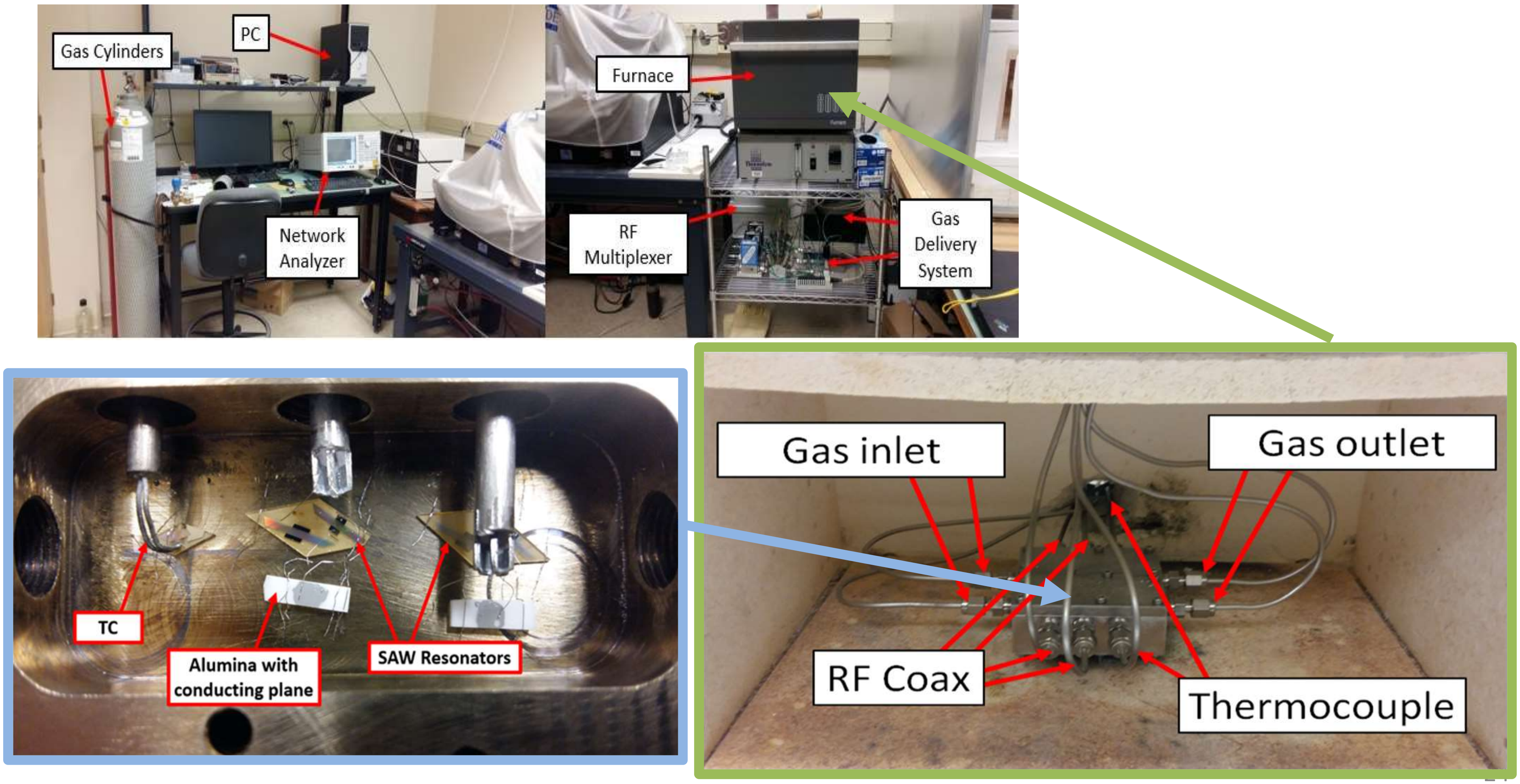
Project Progress & Current Experiments

- **HT Chamber → Sensors, gas delivery, & interrogation system**
- System developed:
  - ✓ Real-time interrogation of up to 4 sensors (two / chamber)
  - ✓ Two thermocouples (one / chamber)



Project Progress & Current Experiments

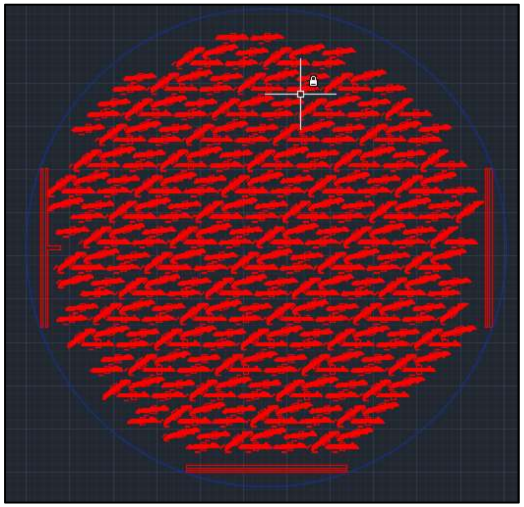
- Complete test setup: Chamber, devices mounted & respective equipment



Project Progress & Current Experiments

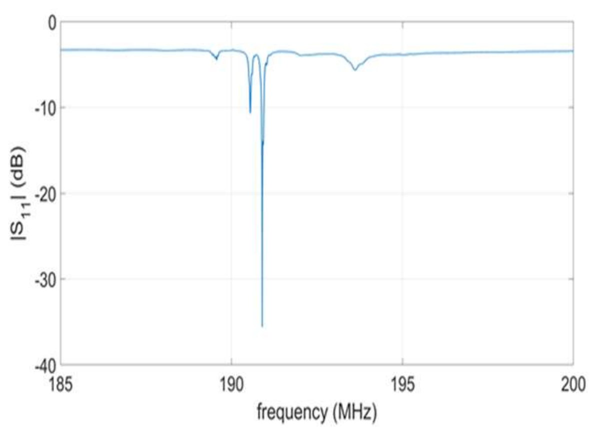
2) Design, fabrication, and experimental verification of alternate orientations on LGS plane

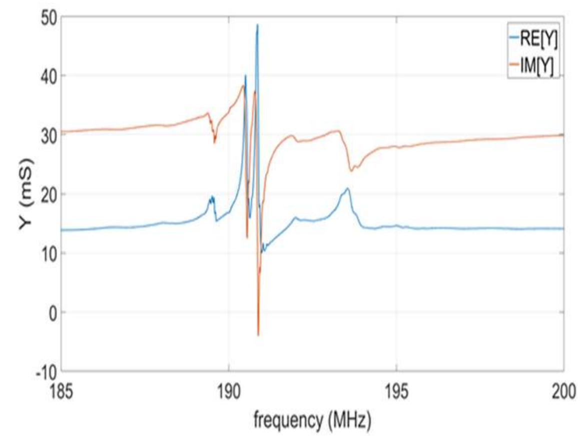
- ➢ Simulations carried out on commercial LGS wafer:
  - ✓ Two orientations identified: temperature compensation
    - 175°C & 300°C ⇒ insensitive to temp. ⇒ ↓ cross-sensitivity
    - Acceptable electromechanical coupling for SAWR sensor
    - Power flow angle addressed in the mask design
- ➢ Photomask generated & devices fabricated



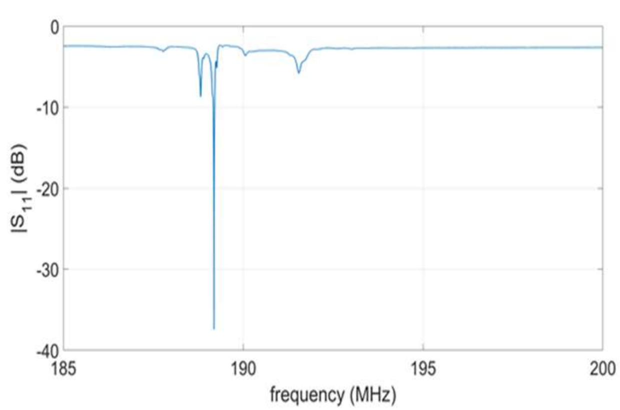
Project Progress & Current Experiments

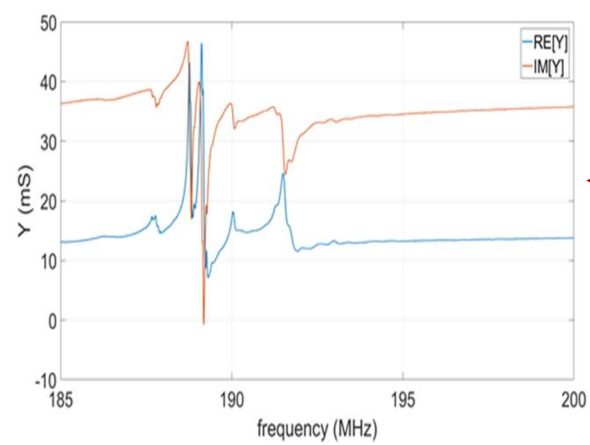
- Responses for the 175°C & the 300°C TCs SWRs
  - $|S_{11}|$ and admittance at room temp.





Temperature Compensated at 175°C

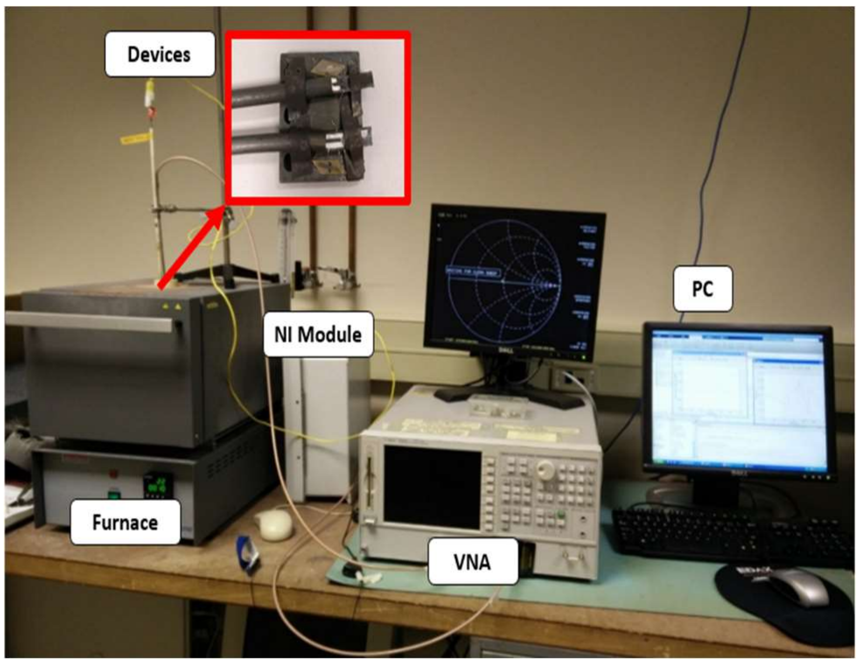


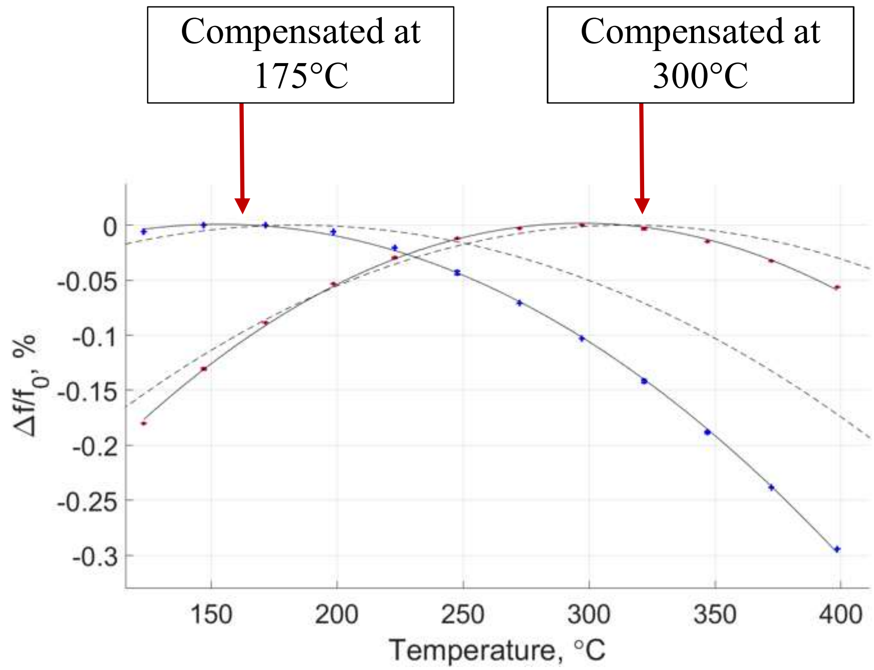


Temperature Compensated at 300°C

Project Progress & Current Experiments

- Experimental verification of temperature compensation at temperatures above 150°C
  - ✓ Two orientations compensated at higher temperatures
  - ✓ Publication on method of selection and verification of TC orientations on LGS at HT



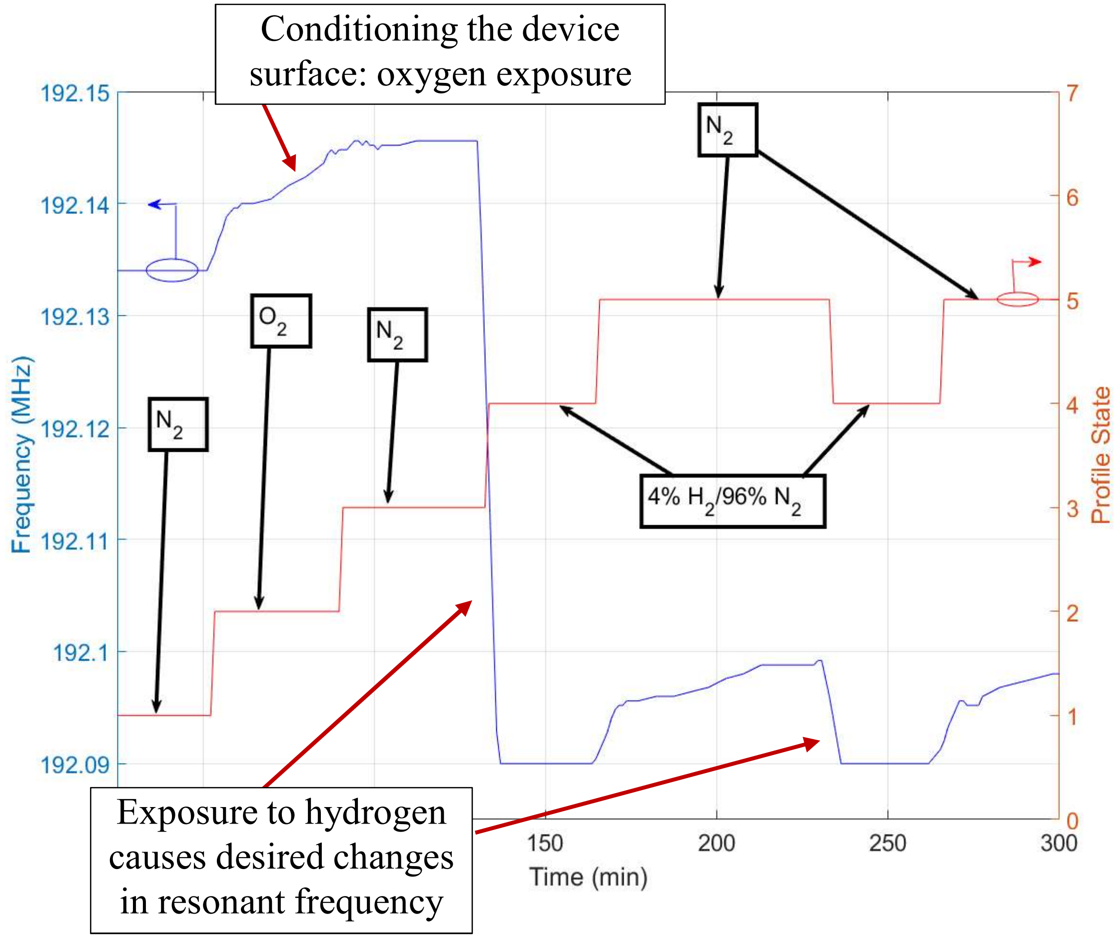


A. Ayes, A. Maskay and M. Pereira da Cunha, Elec. Lett., 2017.

Project Progress & Current Experiments

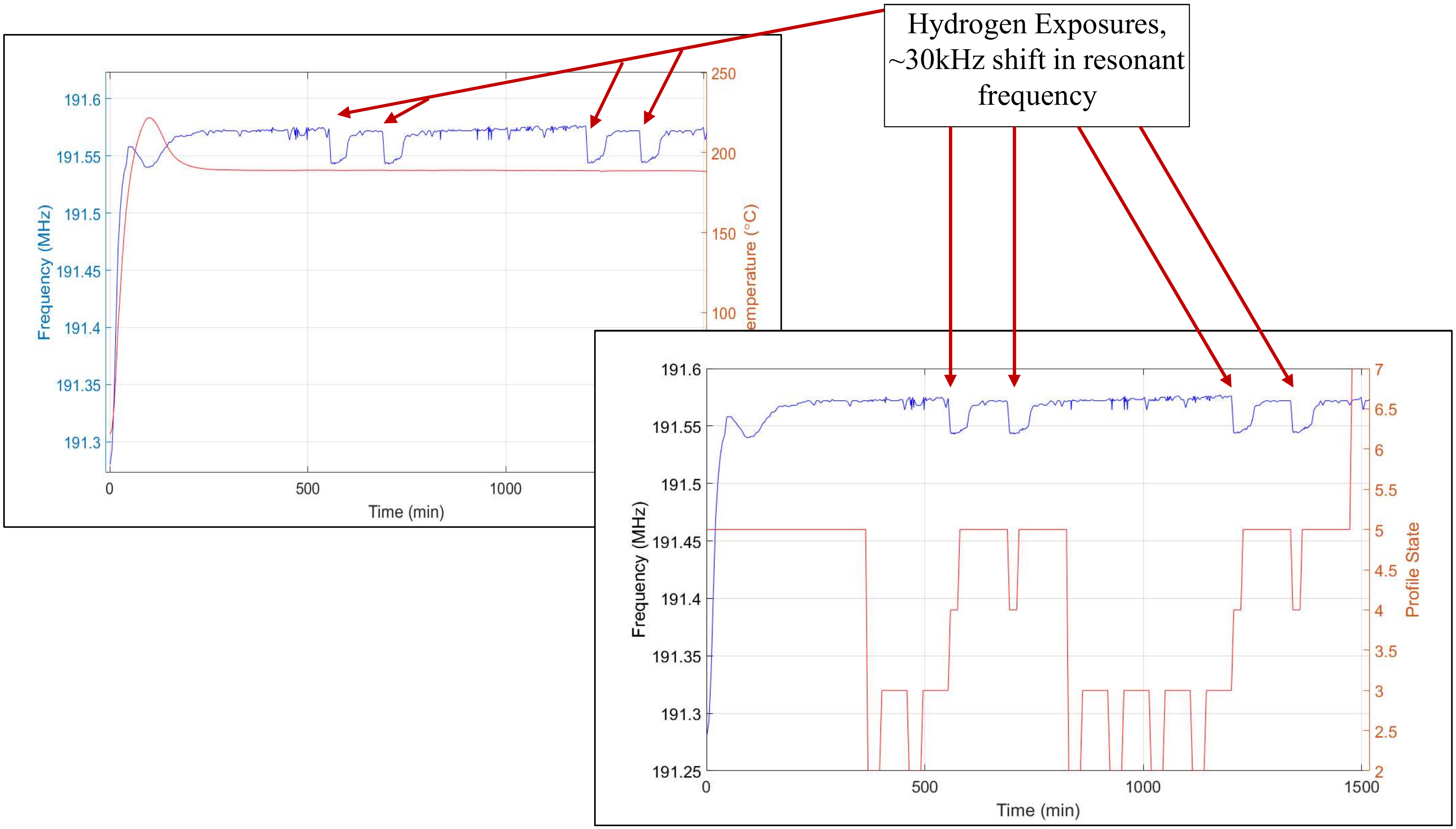
3) Gas Delivery Measurements

- TC SAWR 175°C → Gas response @ Room Temperature



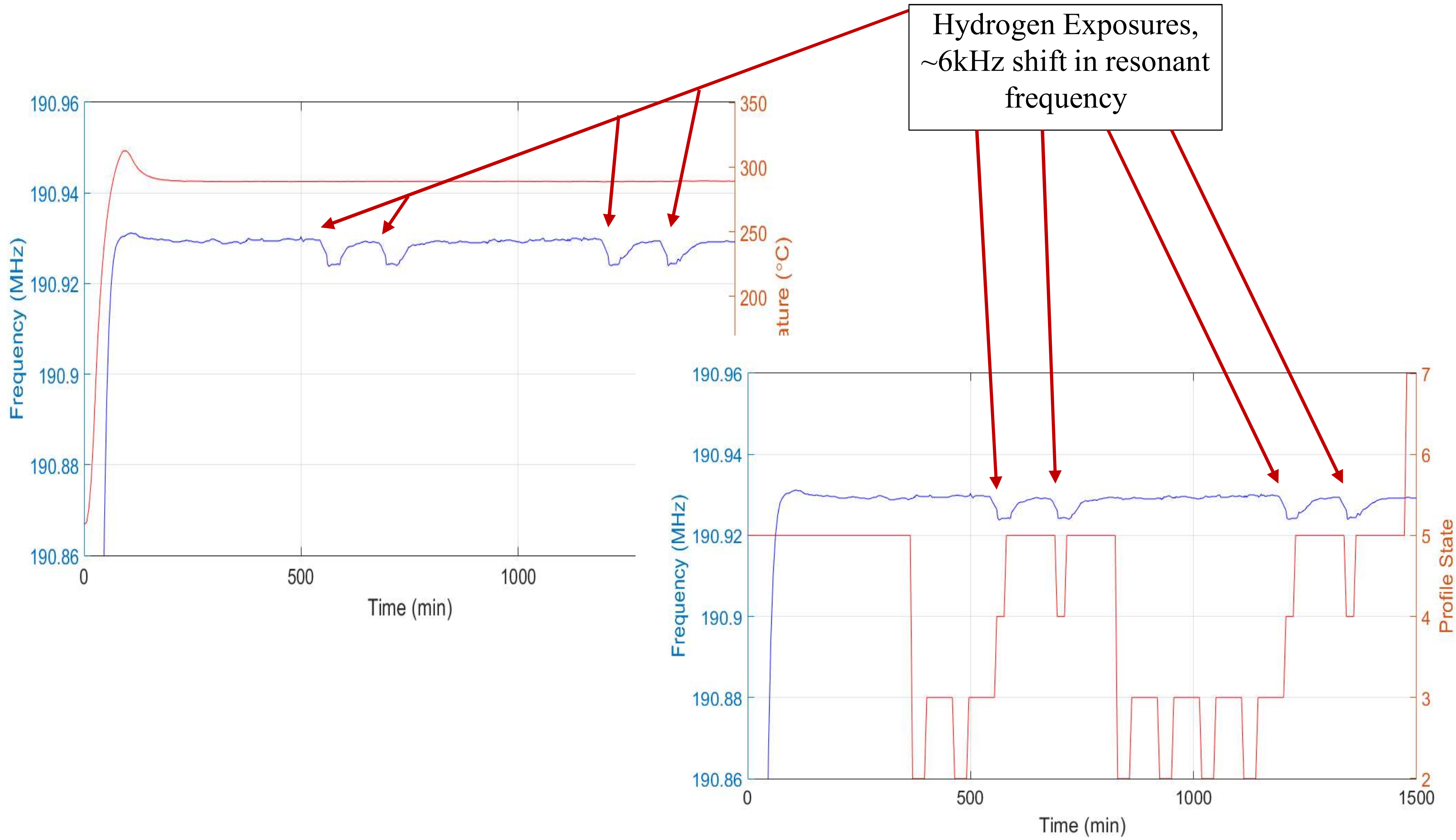
Project Progress & Current Experiments

- TC SAWR 175°C → Gas response @ 200°C



Project Progress & Current Experiments

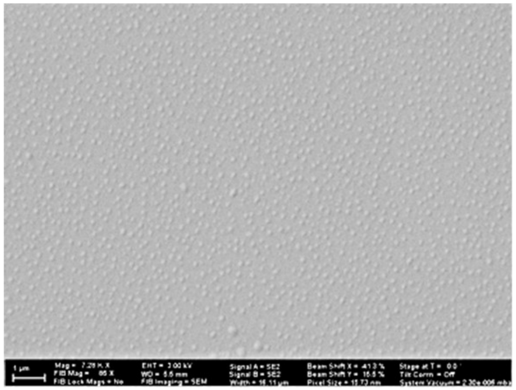
- TC SAWR 300°C → Gas response @ 300°C



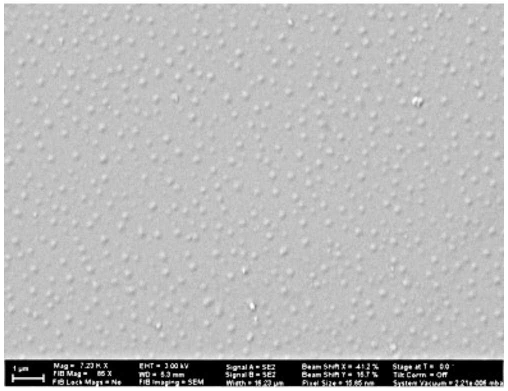
Materials Development

4) YSZ material development

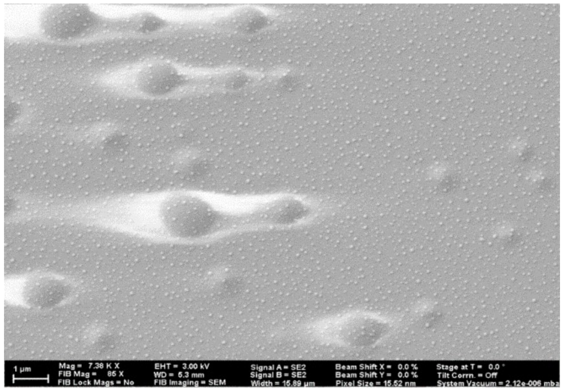
- YSZ deposited at room temperature
  - ✓ Bubbles after HT annealing (700°C) and cycling (750°C)
  - ✓ Issue more severe for thicker layers
  - ✓ Could affect sensor stability over time



25 nm YSZ



50 nm YSZ

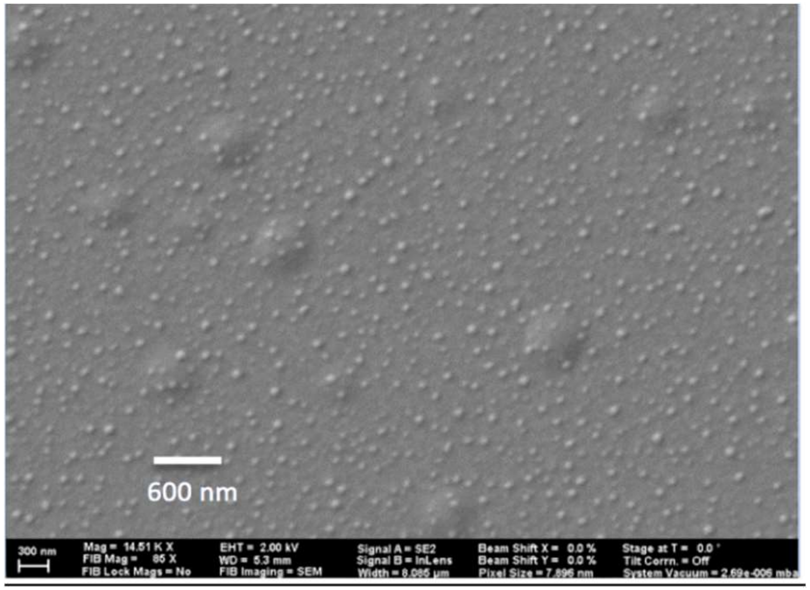


200 nm YSZ

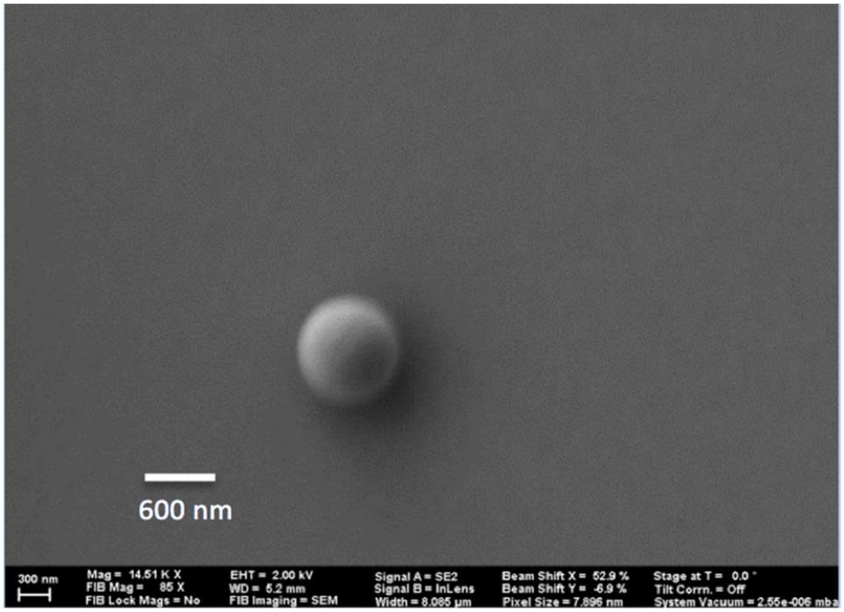
Materials Development

- ➢ High-temperature deposition of YSZ (25 nm @ 850°C)
  - ✓ Releases stress ⇒ PROBLEM SOLVED!!!

RT deposited YSZ, after HT cycling up to 750°C

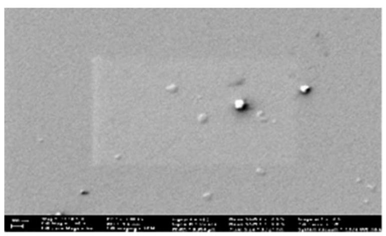


850°C deposited YSZ, after HT cycling up to 750°C

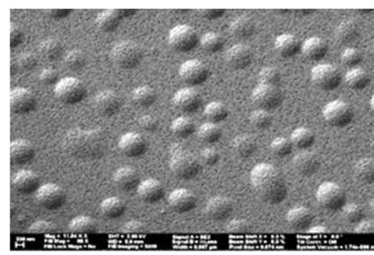


Materials Development

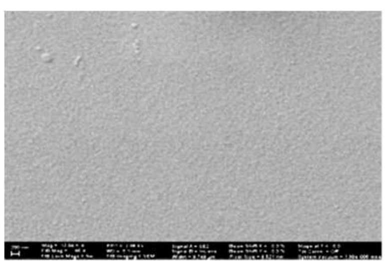
- $PtAl_2O_3$ – based electrode sensors
  - Develops stress hillocks much like YSZ
  - $PtAl_2O_3$ deposited @ ↓ temp. → photoresist (lift-off process)
  - Exploration of different interfacial layers to diminish stress



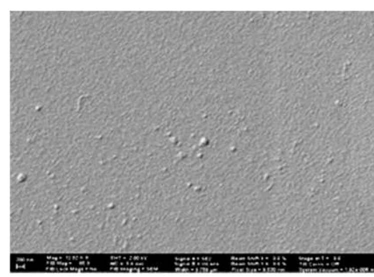
Heated 800°C
1h, air
⇒



ZrO_2 / Zr
interfacial layer



Heated 800°C
1h, air
⇒

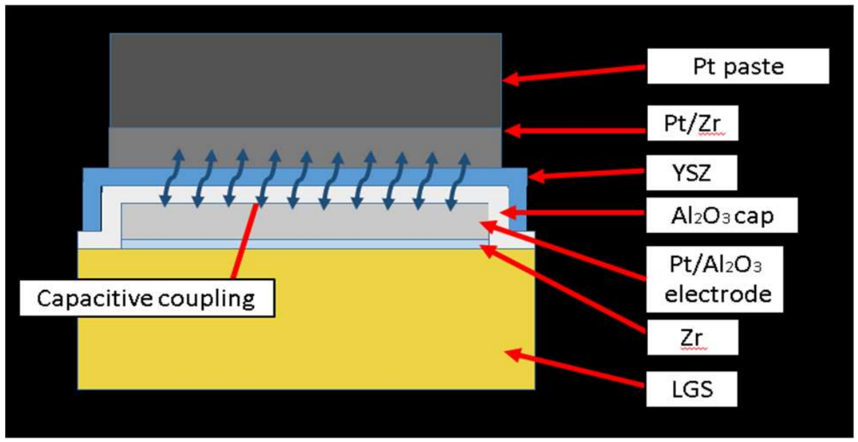


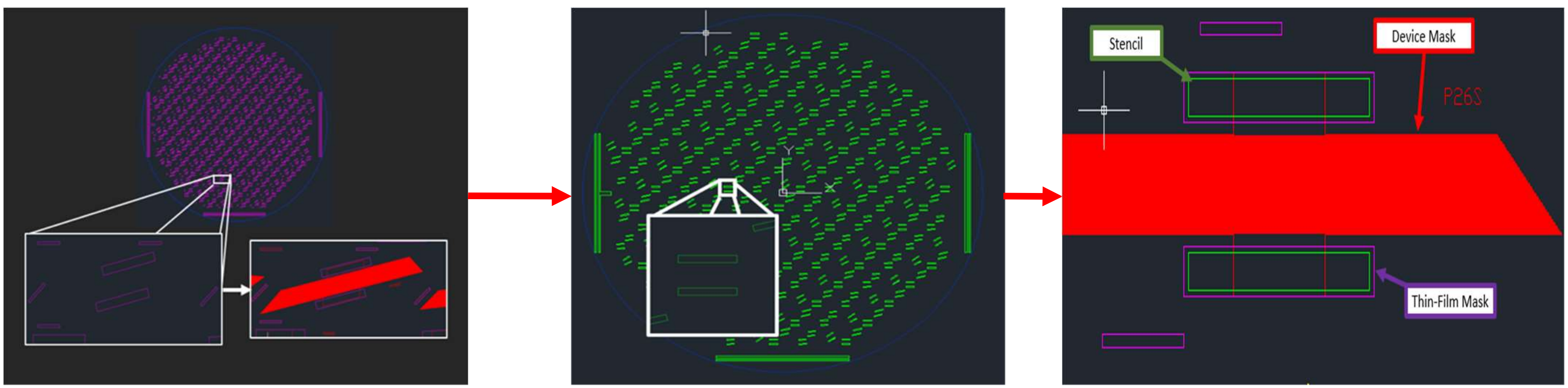
Zr / ZrO_2
interfacial layer

SAW Sensors with YSZ on Top

5) Capacitive coupling → YSZ deposited @ HT

➢ **No photoresist necessary to define contacts**



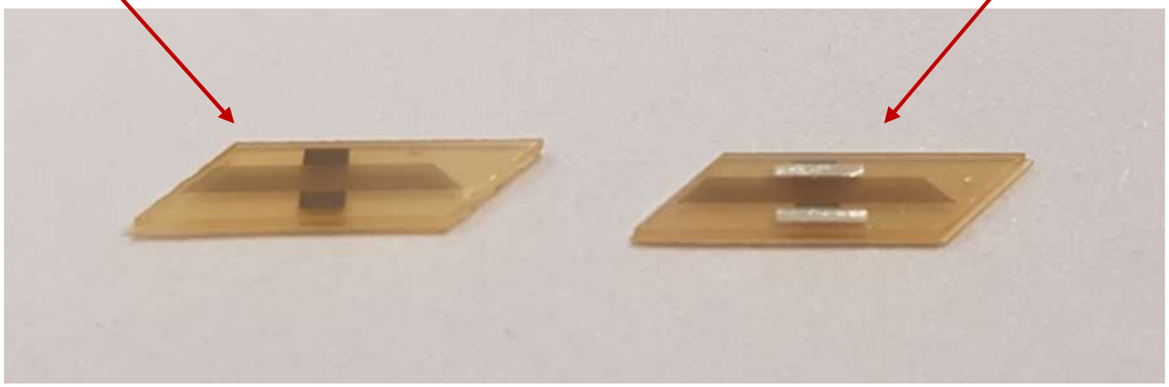


SAW Sensors with YSZ on Top

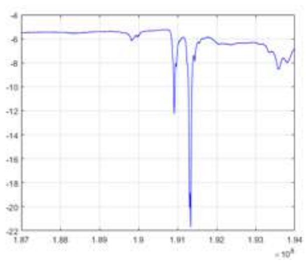
- Fabrication of SAW resonators with YSZ deposited at HT

Bare Device

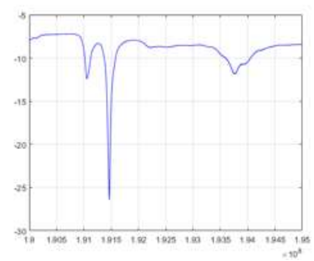
Device with 15 nm of YSZ



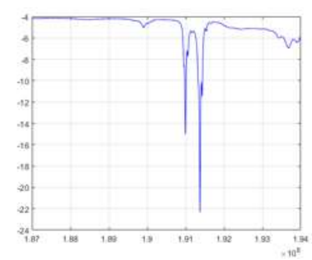
Bare SAWR TC 175°C



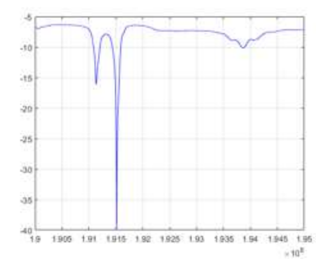
300°C
N_2
⇒



YSZ on Top SAWR TC 175°C



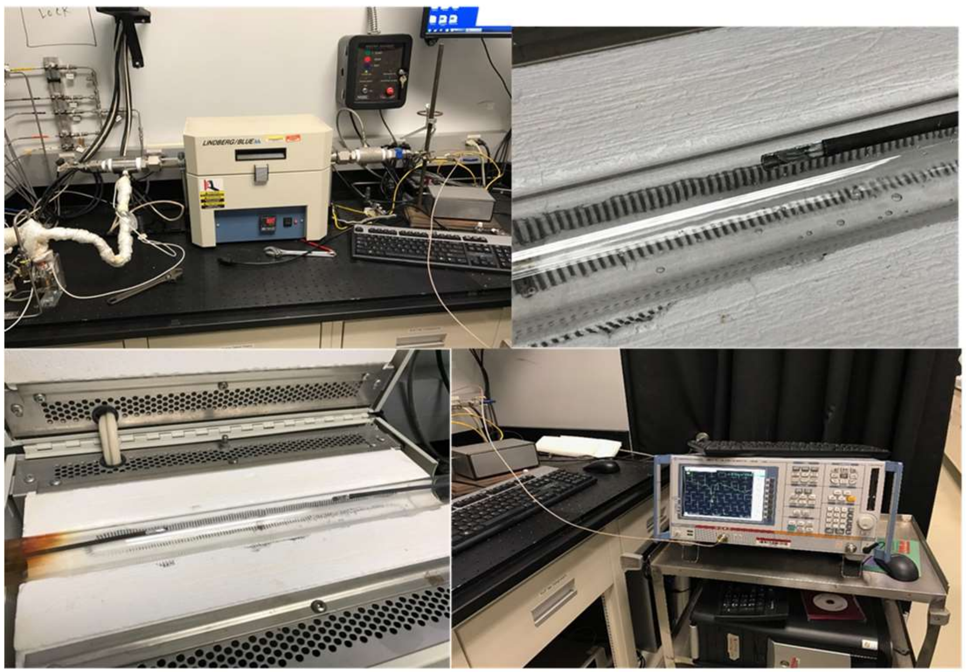
300°C
N_2
⇒

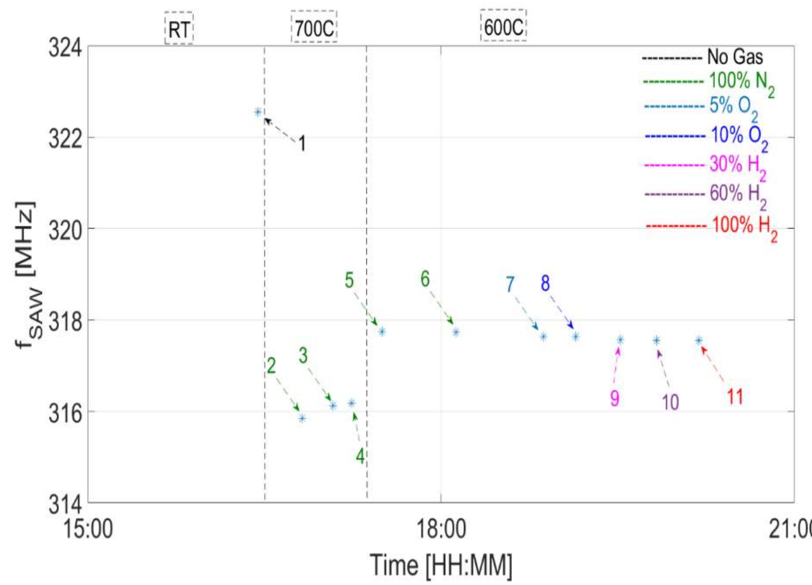


Collaborations

6) Collaborations with NETL/Pittsburgh

- ➢ Robert Fryer / Paul Ohodnicki
- ➢ Confirmation of SAWR platform stability (no detecting film)





Collaborations

6) Collaborations with NETL/Pittsburgh

- Investigation of stability of UMaine Pt-decorated YSZ film on LGS
  - ✓ GOAL: Verification of chemical composition and morphology of Pt-doped YSZ films deposited onto LGS substrates
  - ✓ Process A = Temp. cycling (750–300°C, 750°C dwell, 750–300°C); fixed gas (air)
  - ✓ Process B = Gas cycling (O_2, N_2, H_2, N_2, H_2, N_2, O_2, N_2, H_2); fixed temperature (700°C)
  - ✓ Planned measurements:
    - In situ 4-point electrical conductivity during annealing
    - XPS and SEM before and after annealing
    - XPS depth profiling as a control for the Pt-doped

VI. CONCLUSIONS & ACKNOWLEDGEMENTS

CONCLUSIONS

- Previous period activities & progress → HT μ~ SAW Gas Sensor
- The presentation started with the:
  - Motivations, Methodology, and Project Objectives
- Last year project developments → reviewed
- This year's activities & advances:
  1. Gas Test System at UMaine → new ↓ vol./↓ dead vol. ⇒ fast response
  2. Design, fab., and experim. verification → TC 175°C and 300°C LGS orientations
  3. Gas Delivery Measurem. (O_2 / H_2 / N_2): successful gas tests up to 500°C
  4. YSZ Pt-decorated film developed & fabricated: film stress → bubbles
     - ✓ Problem solved by HT deposition of YSZ
  5. Electrical access → **capacitive coupling** technique developed at UMaine
  6. Samples sent to NETL/Pitt. → Pt-YSZ / LGS → Stability → chem. comp. & morph
- Successful H_2 detection. Encouraging results wrt:
  - Sensor stability/endurance
  - Temperature compensation at 175°C and 300°C → explored with Pt-YSZ SAWR
  - Capability of detecting with H_2 tested up to 500°C

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NETL NATIONAL ENERGY TECHNOLOGY LABORATORY

U.S. DEPARTMENT OF ENERGY

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