

# System Integration of Novel NOx Sensors for Emission Monitoring

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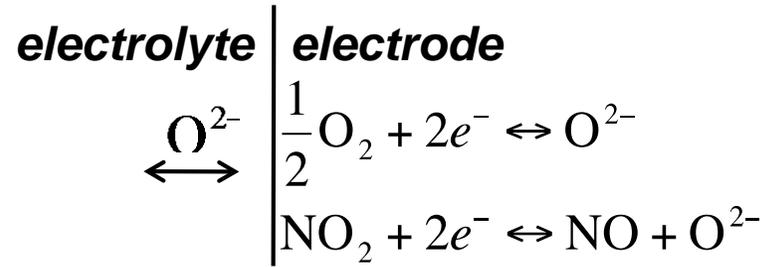
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# Previously reported solid-state electrochemical NO<sub>x</sub> sensor using novel alternating current (AC) method

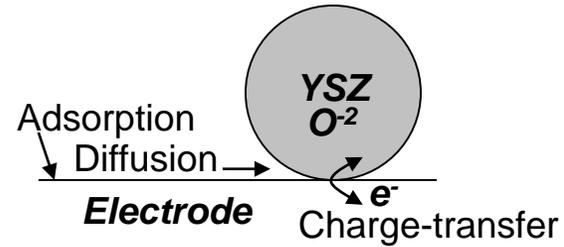
- Simple single cell design with two electrodes separated by oxygen-ion conducting ceramic yttria-stabilized zirconia (YSZ) – similar to O<sub>2</sub> sensor
- Applied oscillating signal with resulting wave distortion analyzed
- Potential as multigas sensor with lower cost and better performance than available amperometric (DC) sensors
- System integration for deployment in real-world applications – overview of some challenges in materials/ceramics and packaging



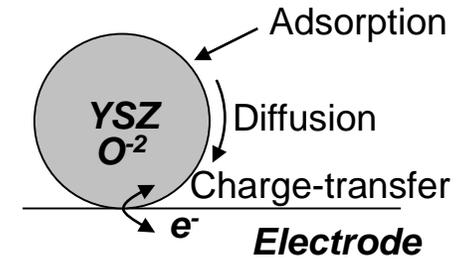
# Sensing relies on electrochemical reaction rates at the electrode/electrolyte interfaces



*Non equilibrium reactions*



Minimal NOx response when electrode surface dominates



Larger NOx response: controlling microstructure and composition is key

- Parallel reactions of O<sub>2</sub> and NO<sub>x</sub>: resolve ppm NO<sub>x</sub> in large 2-21% O<sub>2</sub> background by limiting O<sub>2</sub> reaction rate with porous YSZ electrolyte (conducts O<sup>2-</sup> > 600°C)
- Impedancemetric (AC) sensing: requires at least one sensing electrode; symmetric or asymmetric cells with design flexibility

Ohm's Law:  $V(t) = I(t) \cdot Z(\omega)$

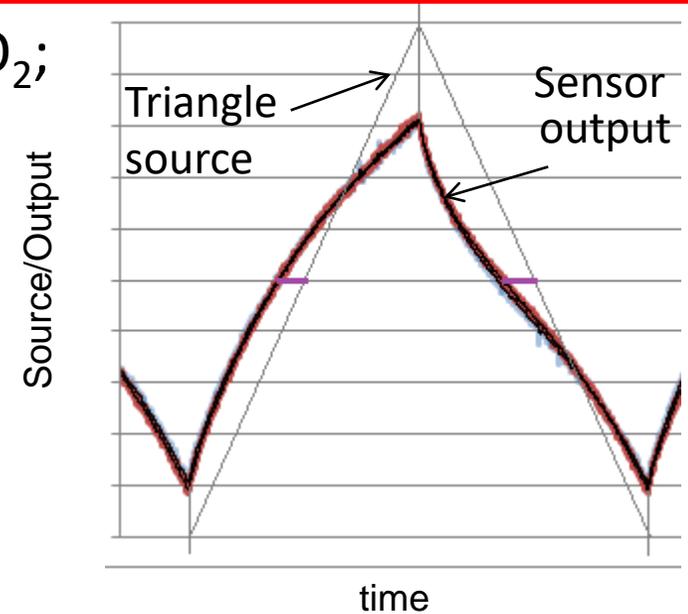
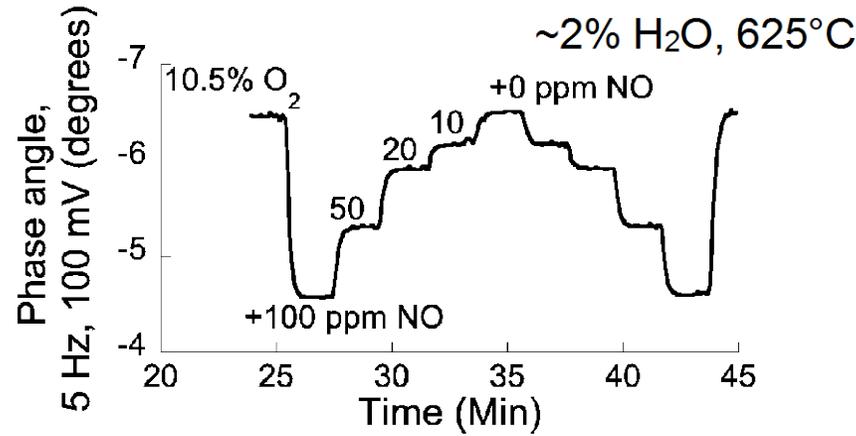
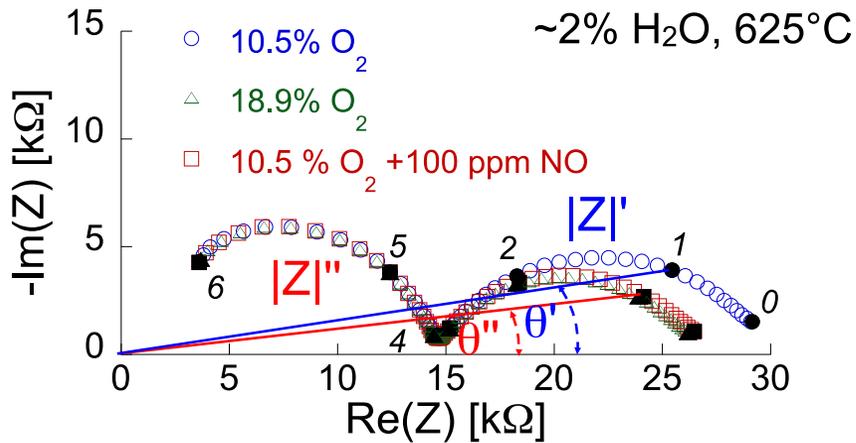
Frequency-dependent complex impedance

Phase angle  $\theta = \tan^{-1} \left( \frac{\text{Im}(Z)}{\text{Re}(Z)} \right)$

Magnitude  $|Z| = \sqrt{(\text{Re}(Z))^2 + (\text{Im}(Z))^2}$

# Impedancemetric sensing – phase angle ( $\theta$ ) signal with good stability and better sensitivity than magnitude $|Z|$

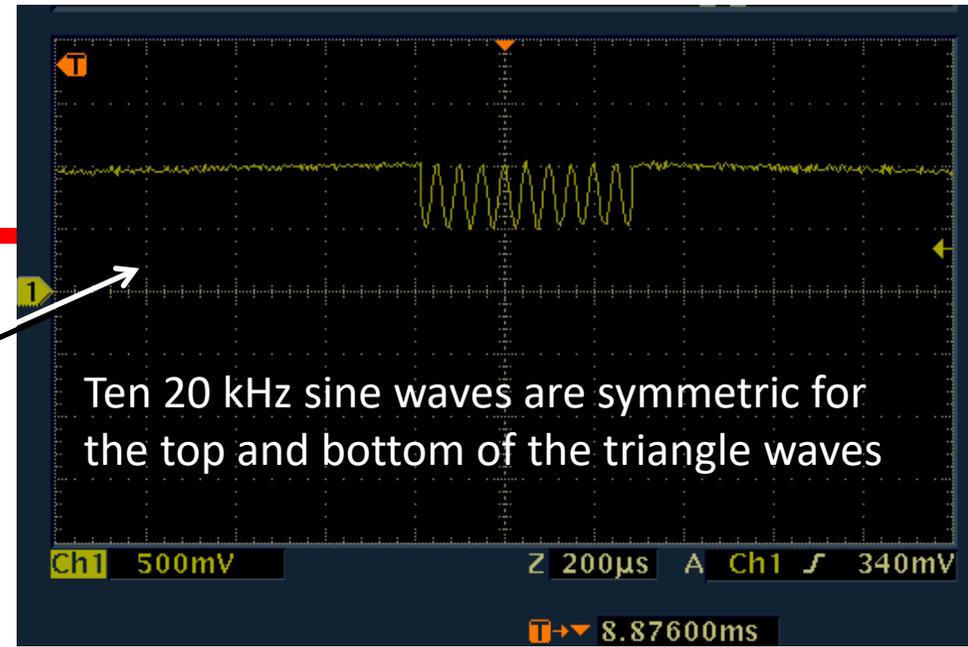
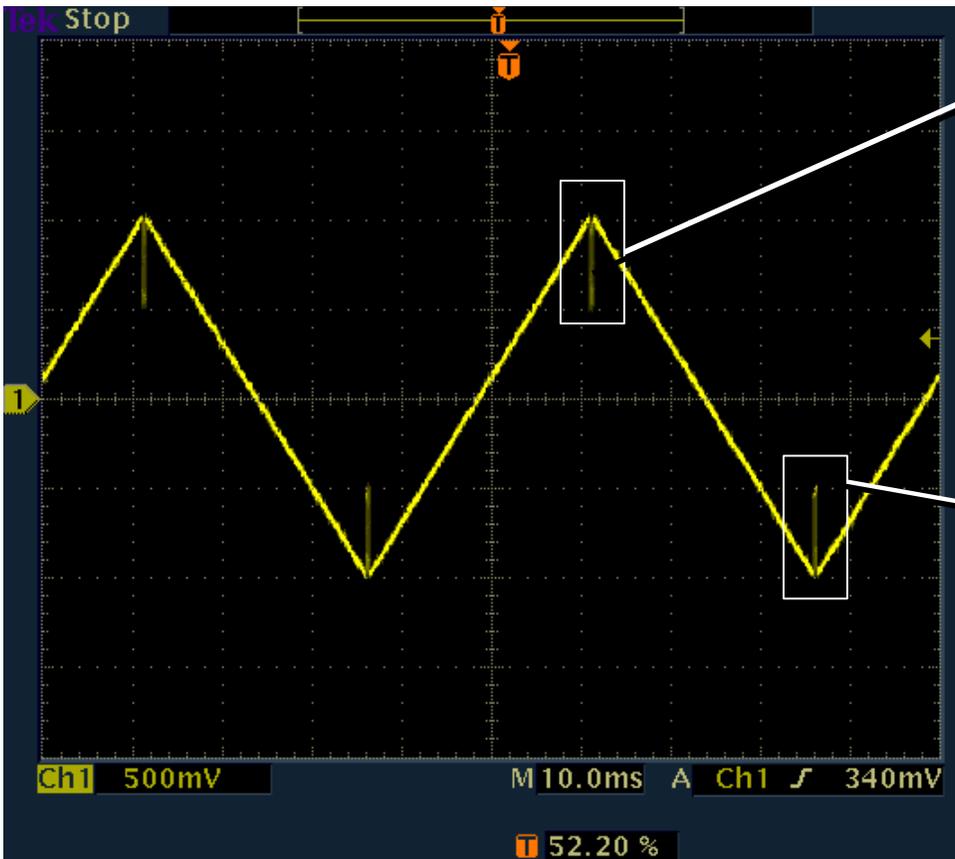
< 1 kHz: Impedance ( $\theta$  and  $|Z|$ ) decrease with increasing NOx and O<sub>2</sub>; typical operation 5-50 Hz and 100 mV excitation



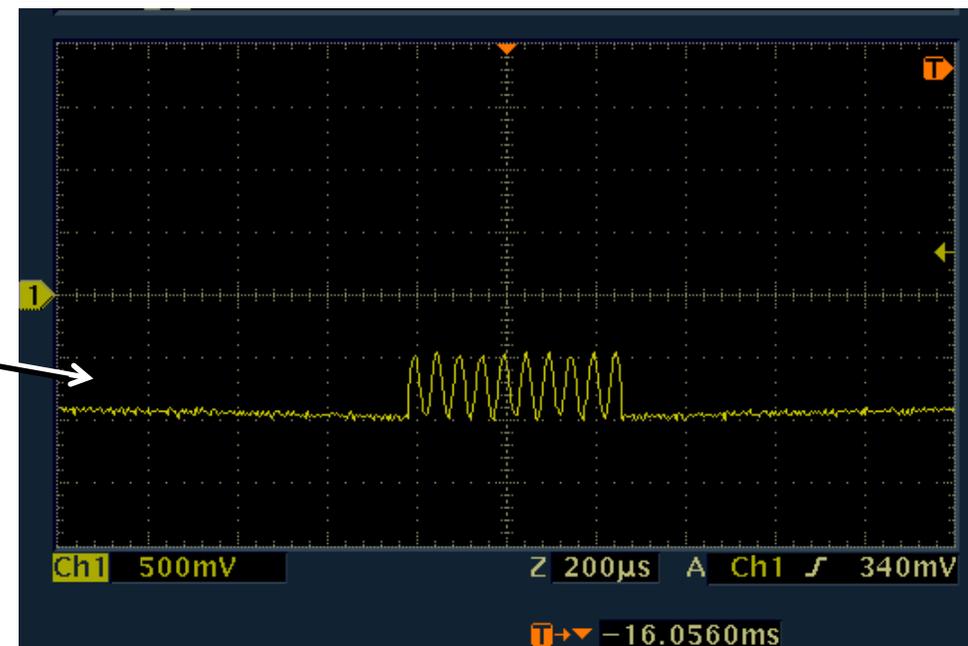
- Lab equipment (Solartron) costs ~\$50k – developed portable low-cost digital electronics using triangle wave form and zero-crossing detection where complex response showed peak relatively unchanged and no real phase shift
- Digital voltage-current time differential method using complex response – different influence of NOx and O<sub>2</sub> allows several ways to extract oxygen

# Measurement principle

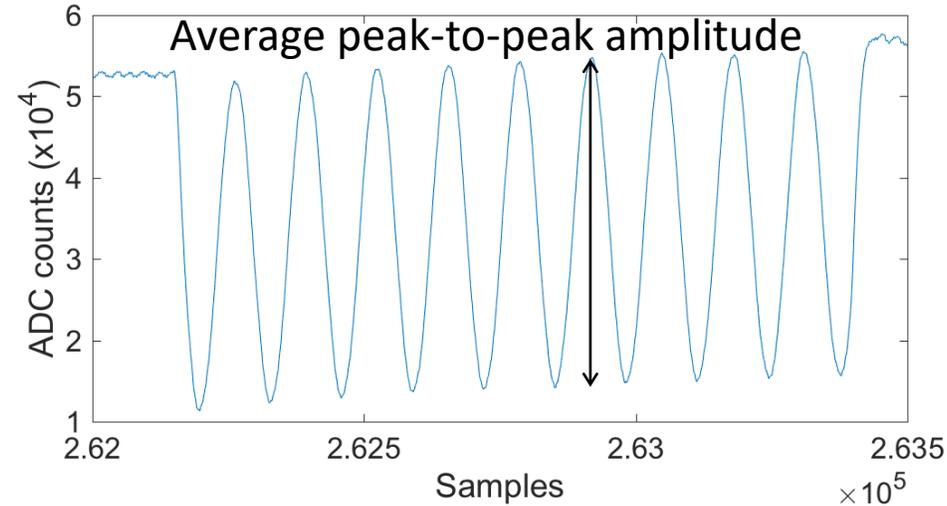
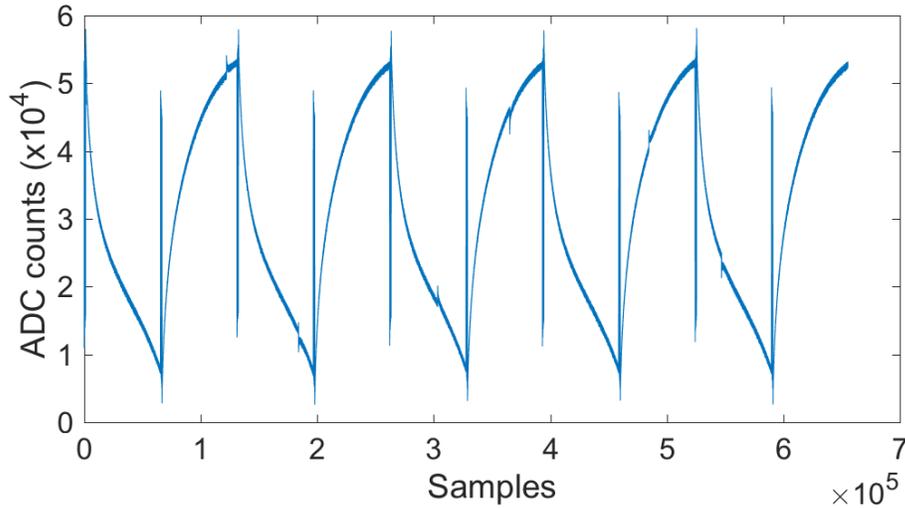
Excitation with 20 Hz triangle wave with bursts of 20 kHz sine waves at top and bottom of triangle waves



Ten 20 kHz sine waves are symmetric for the top and bottom of the triangle waves

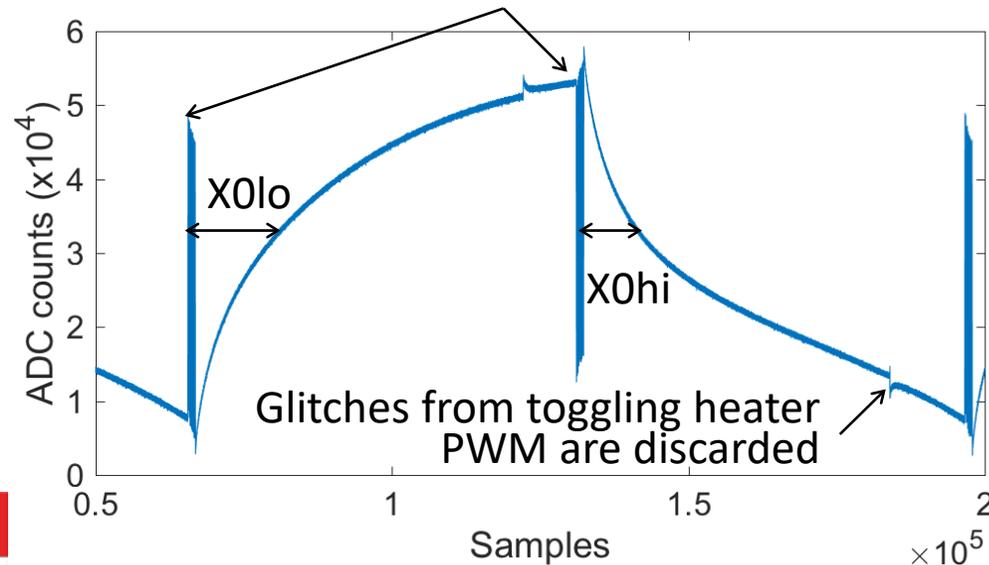


# Captured current – two gas measurements at zero current condition on either side of 20 Hz wave



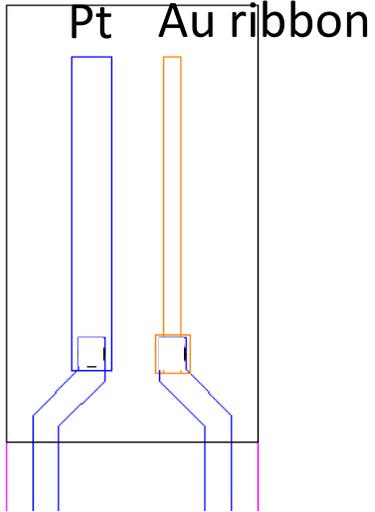
20 kHz sine waves – calculate real impedance to monitor and control temperature

Distortion from 20 kHz sine waves

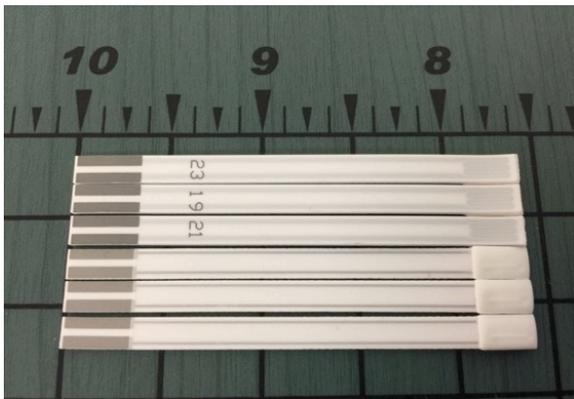


- Sampled time from start of half wave
  - X0lo: dominated by O<sub>2</sub> and NO
  - X0hi: dominated primarily by O<sub>2</sub>
- 20 kHz sine wave distortions do not interfere with gas measurement

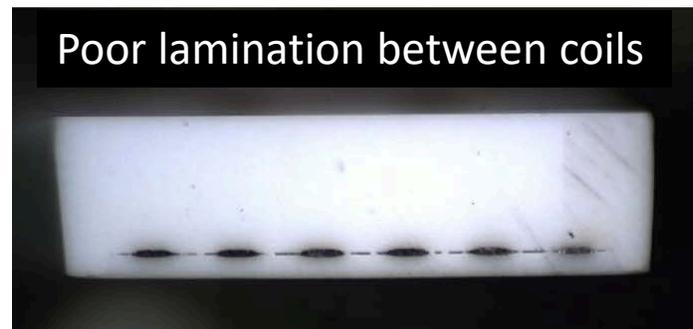
# Materials/Ceramics – 1450°C cofired active sensor components and substrate with platinum (Pt) heater coil



- Furnace prototypes (no heaters) with calcined porous YSZ electrolyte and gold (Au) ribbon/Pt electrodes
- Found suitable ceramic interconnect material to allow electrical connection between Au and Pt
- Advanced prototypes with active components on improved alumina substrates with embedded Pt heater coil



Embedded Pt heater coils

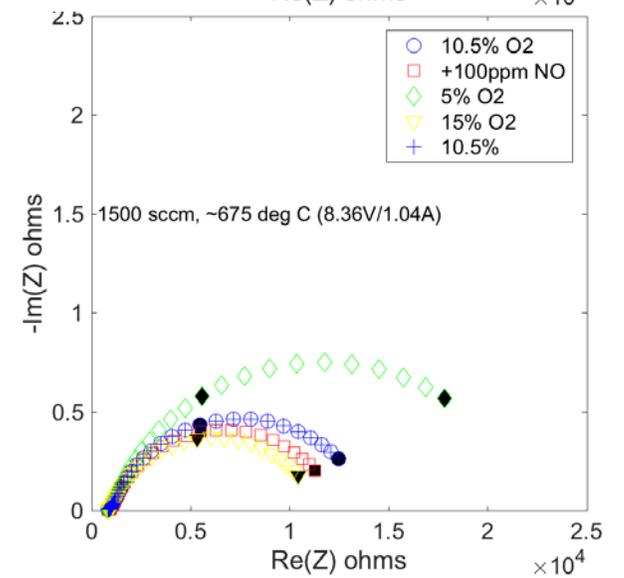
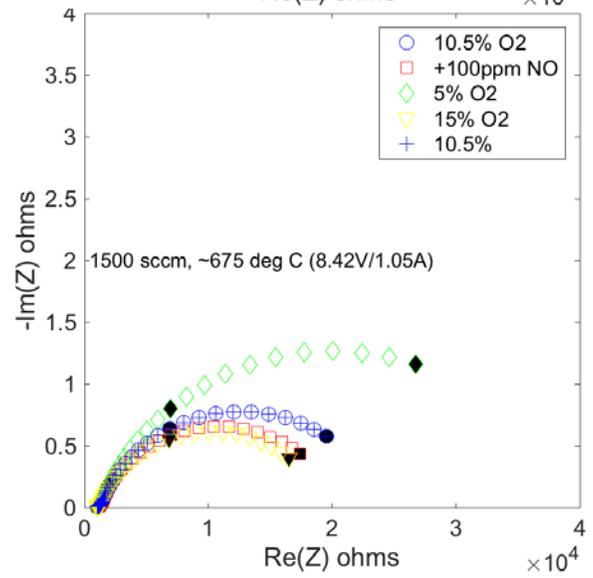
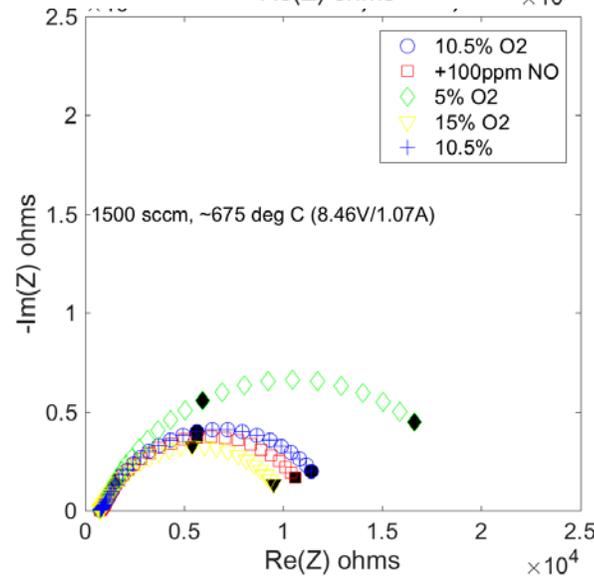
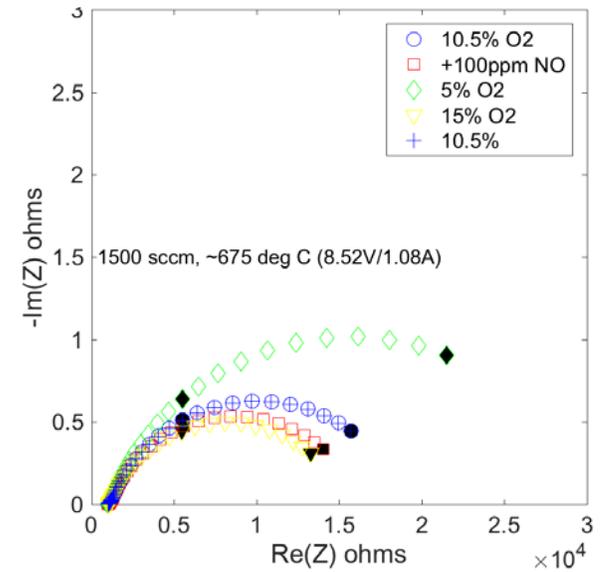
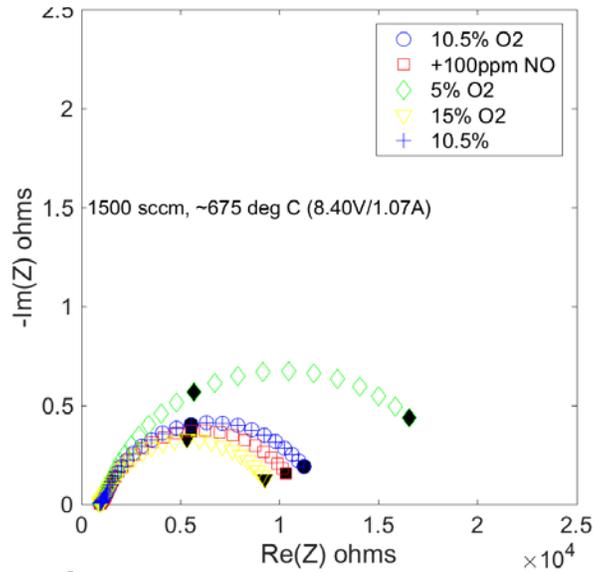
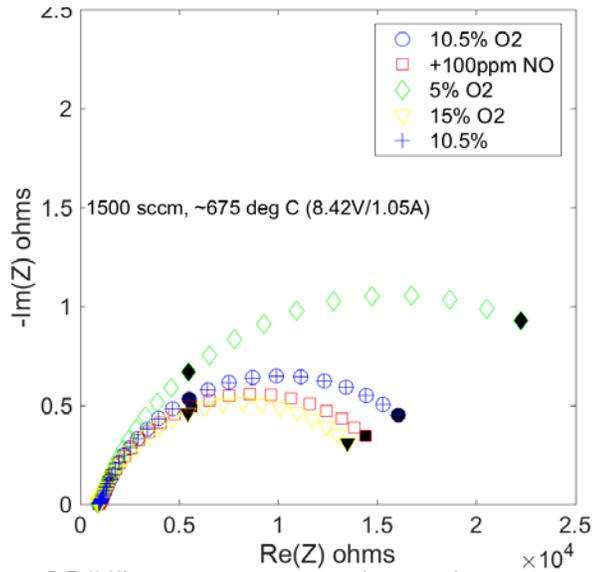


Poor lamination between coils

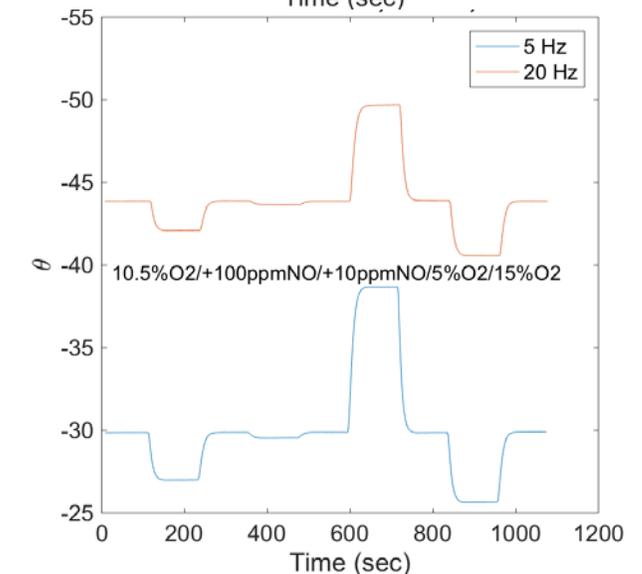
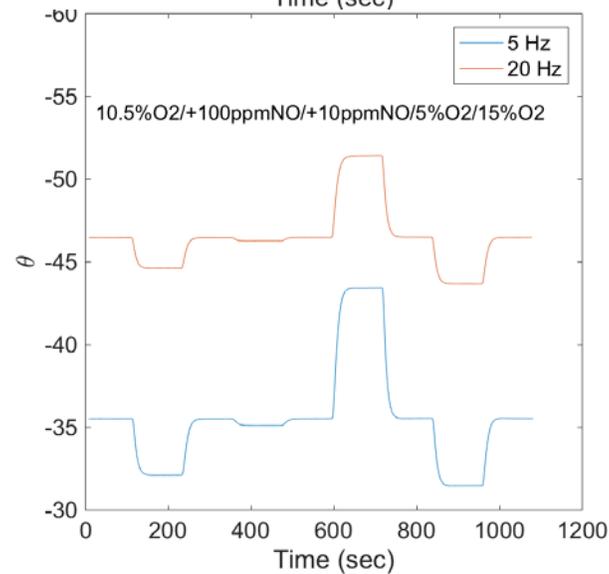
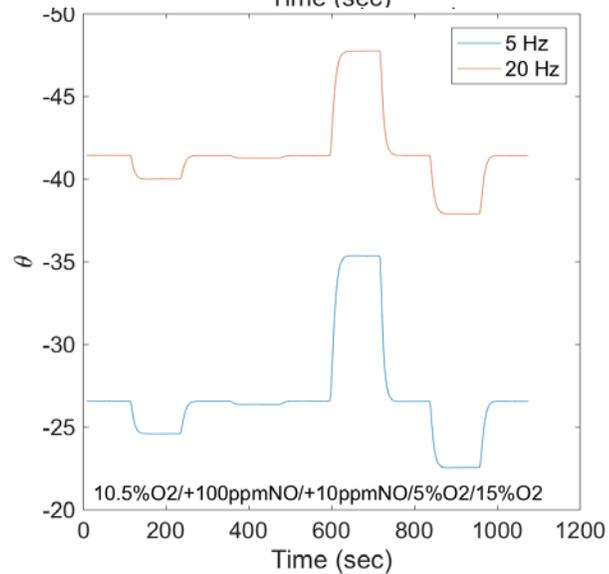
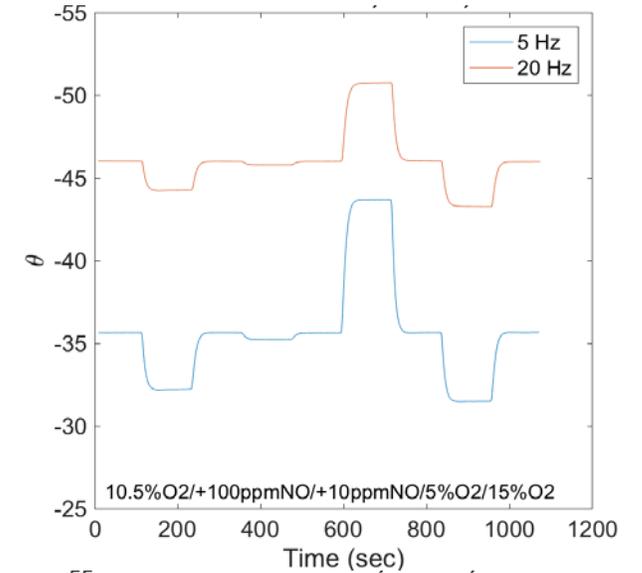
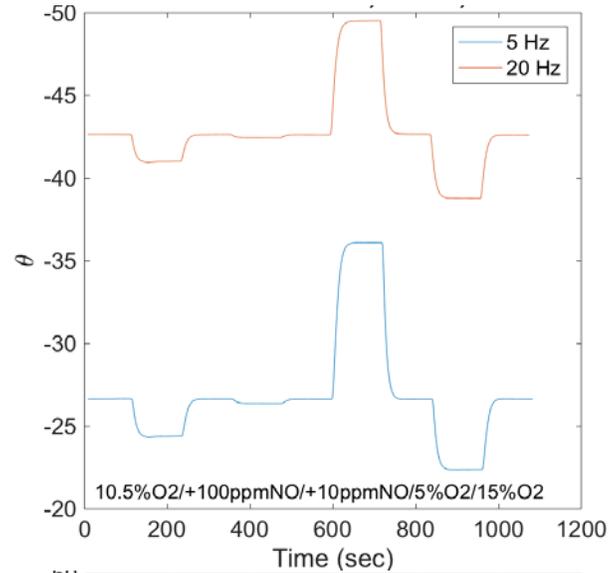
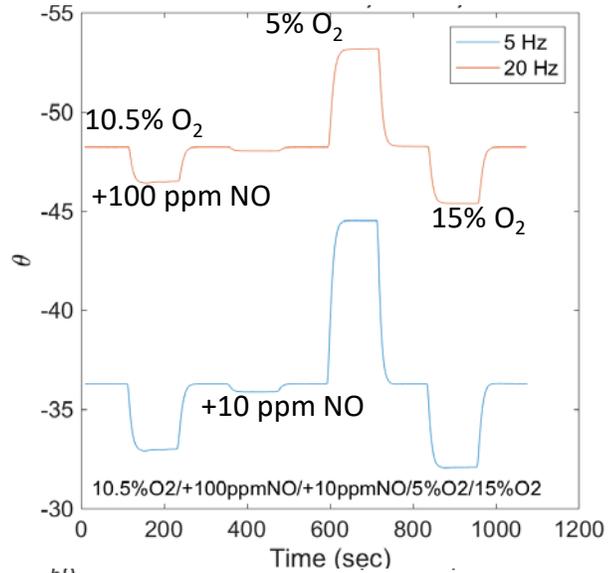


Process to improve lamination

# Sensors with improved sensor-to-sensor reproducibility

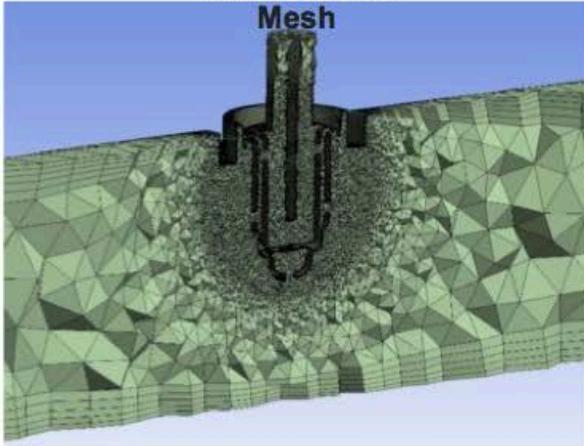


# Sensors with improved sensor-to-sensor reproducibility

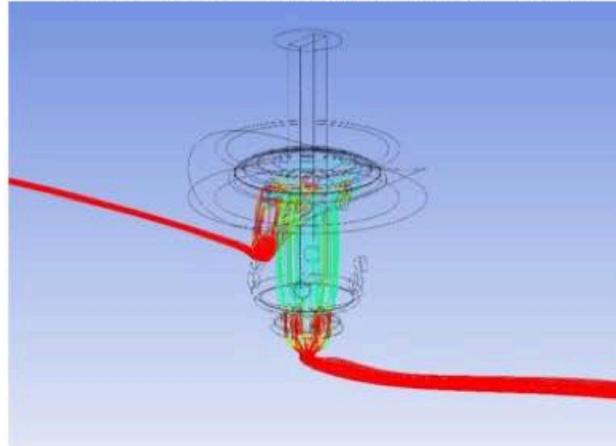


# Packaging and protection tube – Venturi modeling

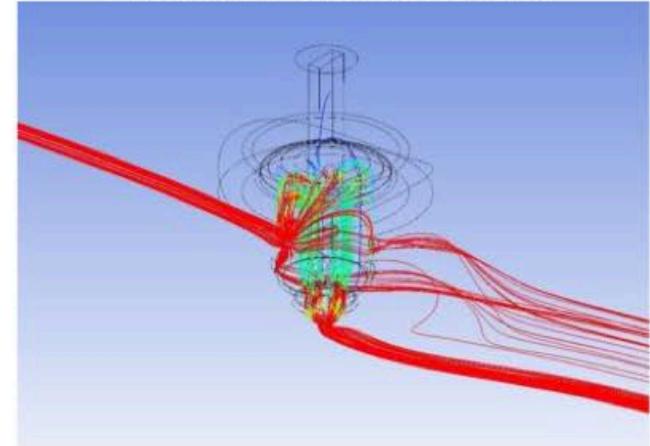
Fluid Volume  
Mesh



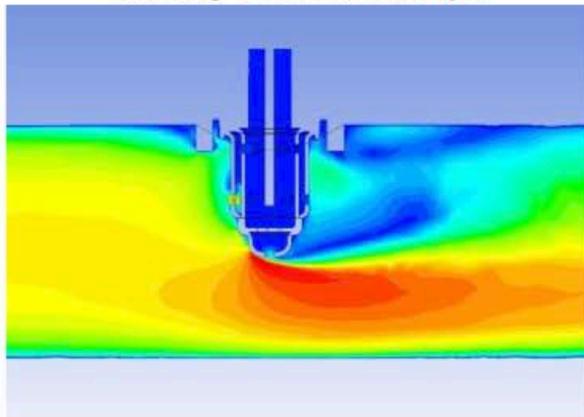
Streamlines Through Venturi Outlet



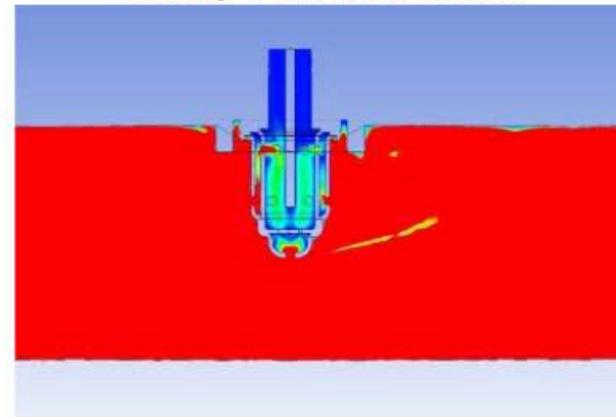
Streamlines Through Inlet Hole



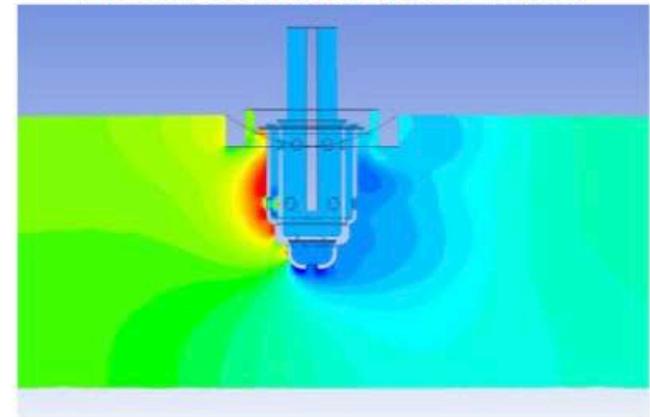
Velocity Contours in Pipe



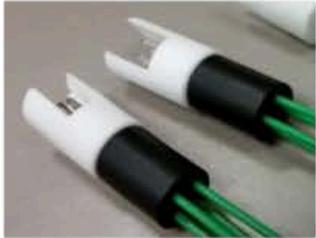
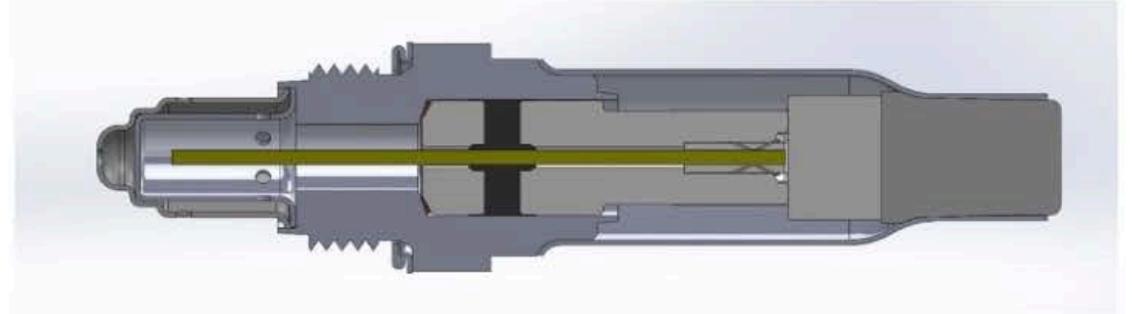
Velocity Contours in Sensor



Pressure Contours Across Sensor



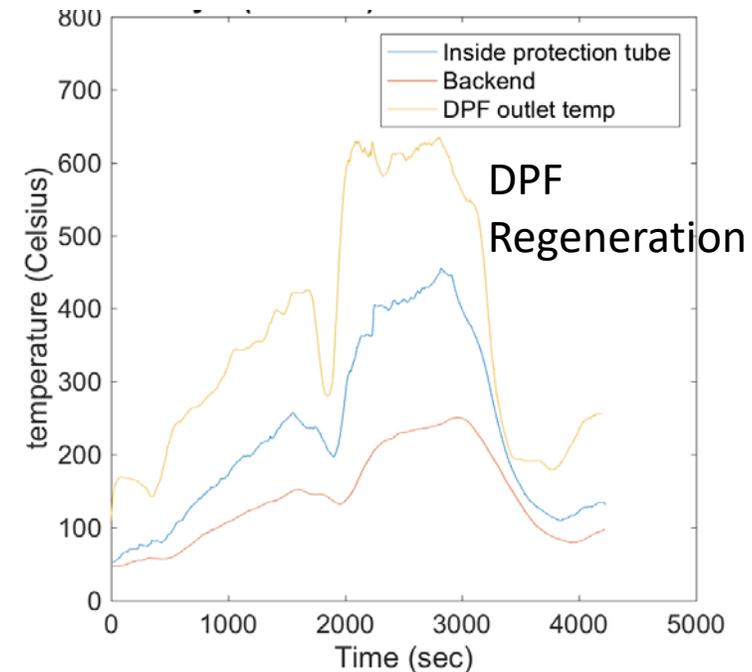
# Packaging – Venturi protection tube in full assembly



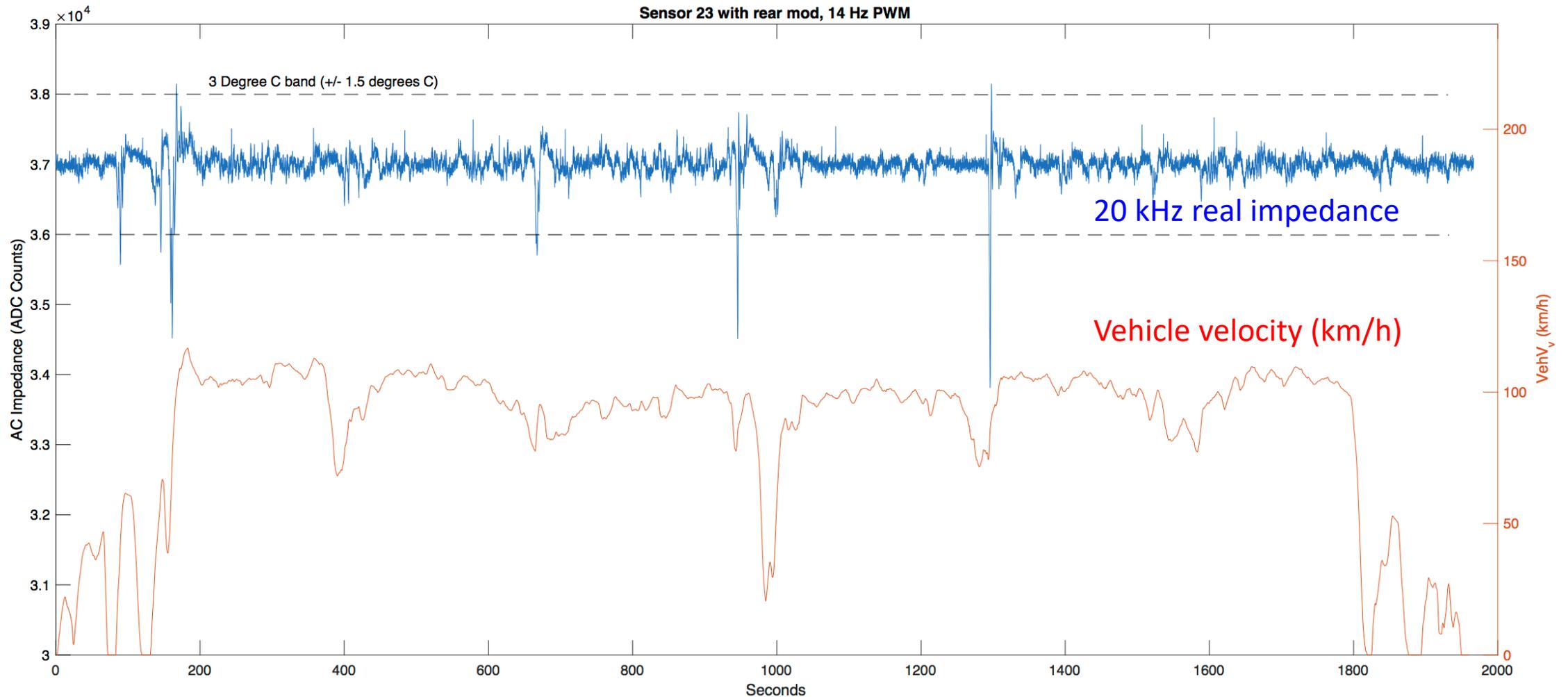
# Ford (F-250 Lariat) vehicle testing at Salt Lake City (SLC)

– demonstrate thermal control with heater strategy

- One test position in vehicle exhaust located next to commercial NOx sensor – added a temporary bracket to hold electronics
- Characterized drive cycle (along I-80 from SLC to Park City) using instrumented thermocouple in our packaging

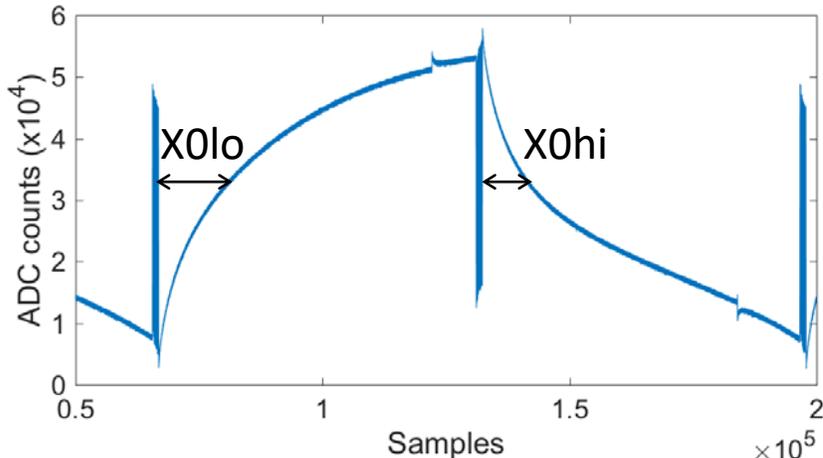


# Temperature control using real impedance from 20 kHz sine waves better than $\pm 1.5^\circ$ during drive cycle

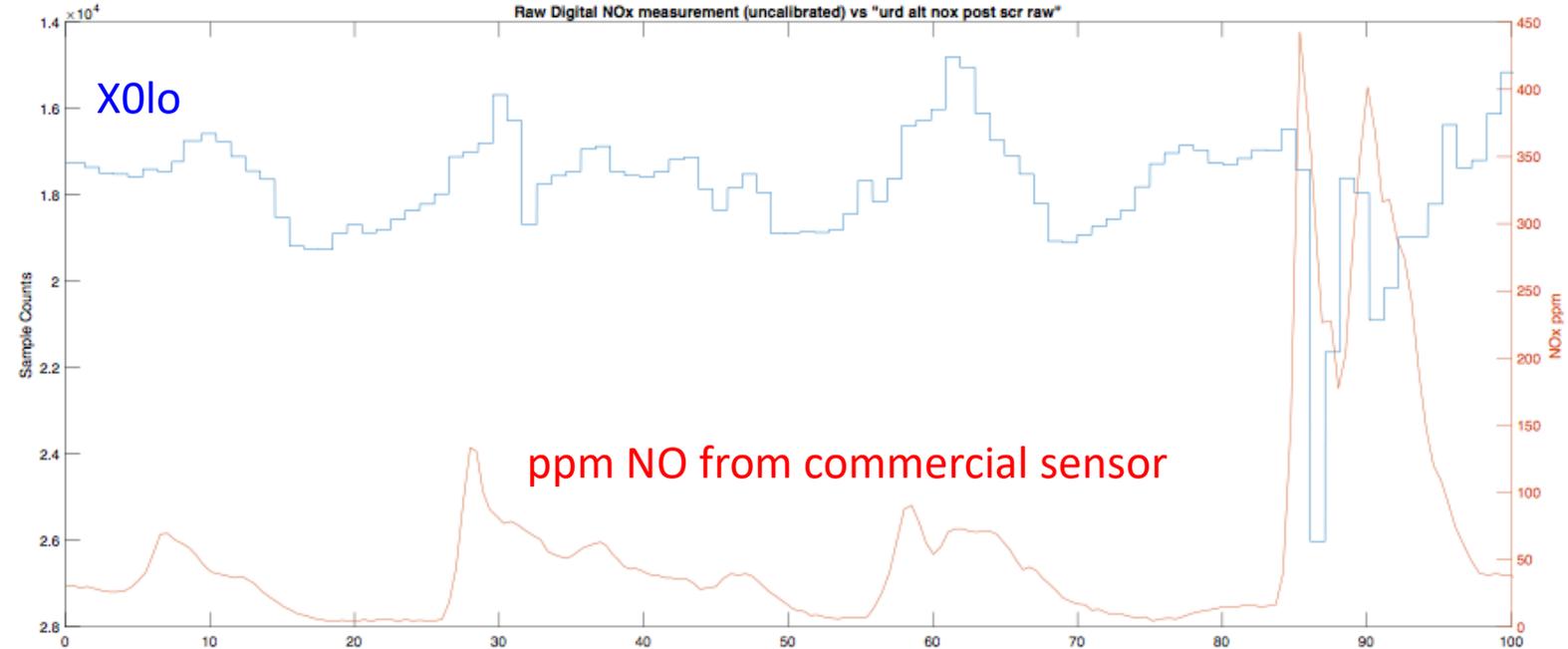
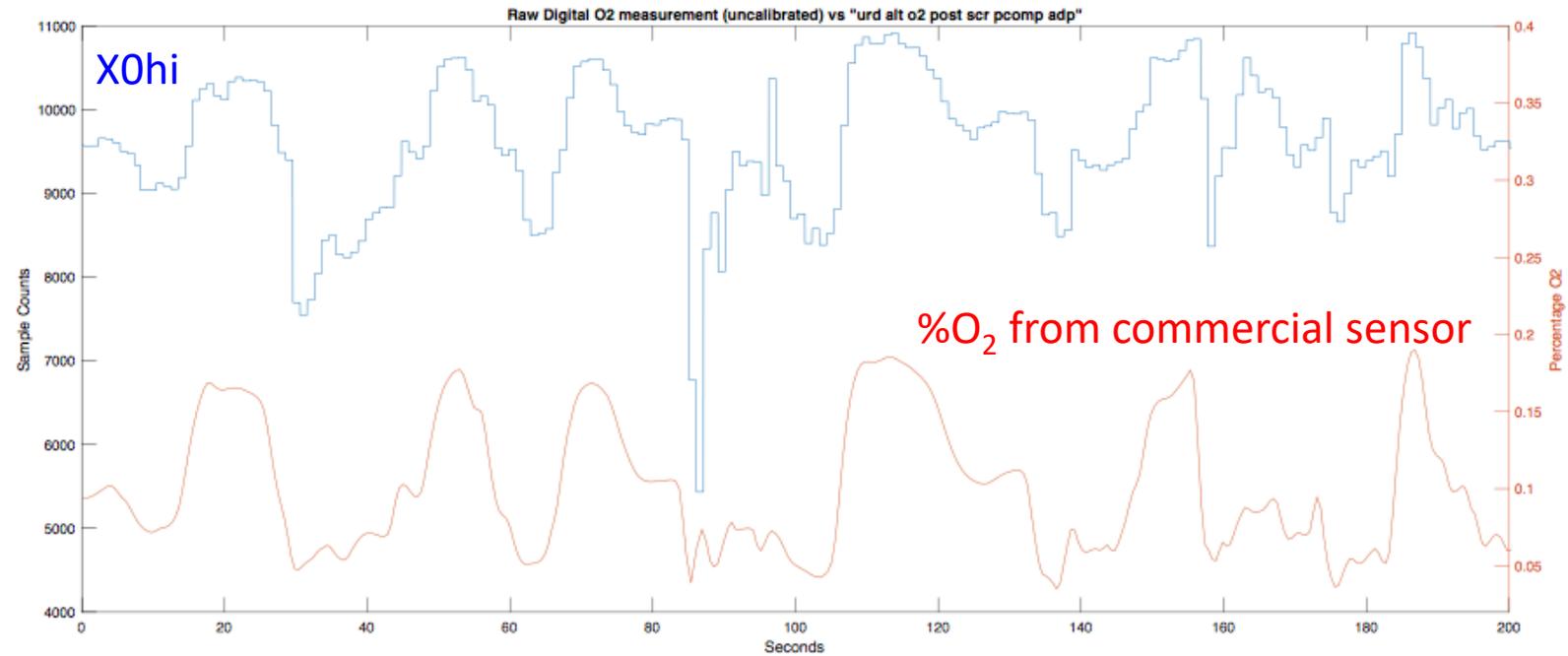


# Vehicle testing

- Gas measurements on either side of 20 Hz wave correlated with readings from commercial NOx sensor



- X0lo: O<sub>2</sub> and NO
- X0hi: mostly O<sub>2</sub>



# Ongoing activities in system integration

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- Multiple strategies for O<sub>2</sub> measurement including DC values from 20 kHz sine wave excitation – measurements to mitigate interference and cross-sensitivity to other components including H<sub>2</sub>O and NO<sub>2</sub>
- Improving stability of 20 kHz thermal measurement – better stability of furnace prototypes compared to advanced prototypes
- Demonstrating panelization and materials selection for mass fabrication – better understanding Au/Pt electrical connection and work on alternative interconnection strategies
- Packaging – components with improved electrical connection (vibration and thermal cycling) and water bath testing of water resistant treatments
- Planned vehicle testing at Ford scheduled in April

# Summary

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- Measurement principle of impedancemetric multigas/NO<sub>x</sub> sensor – digital voltage-current time differential method using complex response of YSZ-based electrochemical cell
- Materials/ceramics work for more advanced prototypes with better sensor-to-sensor reproducibility and packaging improvements demonstrated in vehicle testing – temperature control to  $\pm 1.5^\circ$
- Ongoing system integration activities include multiple strategies for detecting O<sub>2</sub> and other components, improving stability of thermal measurement, and demonstrating mass fabrication and robust packaging
- Planned vehicle testing at Ford scheduled in April

**Questions? Thank you for your attention! [lw@emisense.com](mailto:lw@emisense.com)**