

Novel Design of Multi-Layered Resonator Sensor For Detection of Atomic Oxygen

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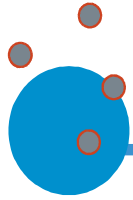
M.Sc. thesis project



***Technical University of Denmark
Department of Micro and Nanotechnology***

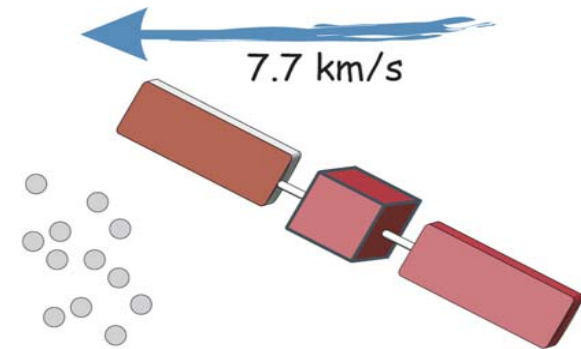


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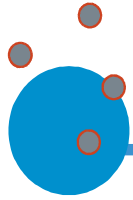
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- AO Sensors
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 - Oxidation
- Questions

Why is it important to measure Atomic Oxygen?



- Due to its **atomic form** and its **impact velocity**, oxygen is responsible for significant **erosion and degradation** of satellites.
- Acquire a better understanding of the **atmospheric chemical reactions** in the mesosphere and lower thermosphere.

Goal: achieve continuous monitoring of Atomic Oxygen



How has this been done until now?

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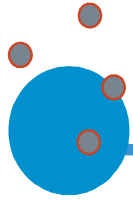
Testing

Characterization

Oxidation

Questions

- *Witness samples*
 - *Put an object in orbit and retrieve it after some time*
- *Mass spectrometers*
 - *Measure bended trajectory according to mass/charge ratio*
- *Quartz Crystal Microbalances*
 - *Measure change in mass as a sample oxidizes*
- *Actinometers*
 - *Measure change in resistance as a sample oxidizes*
- *etc.*

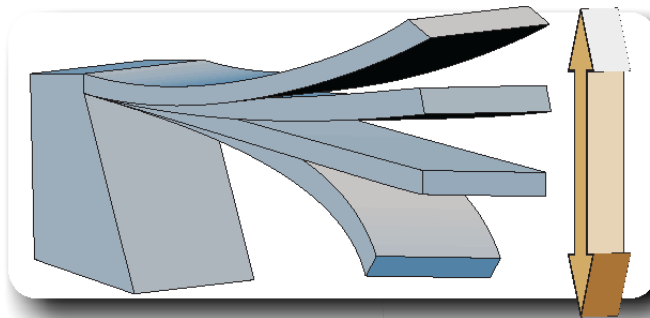
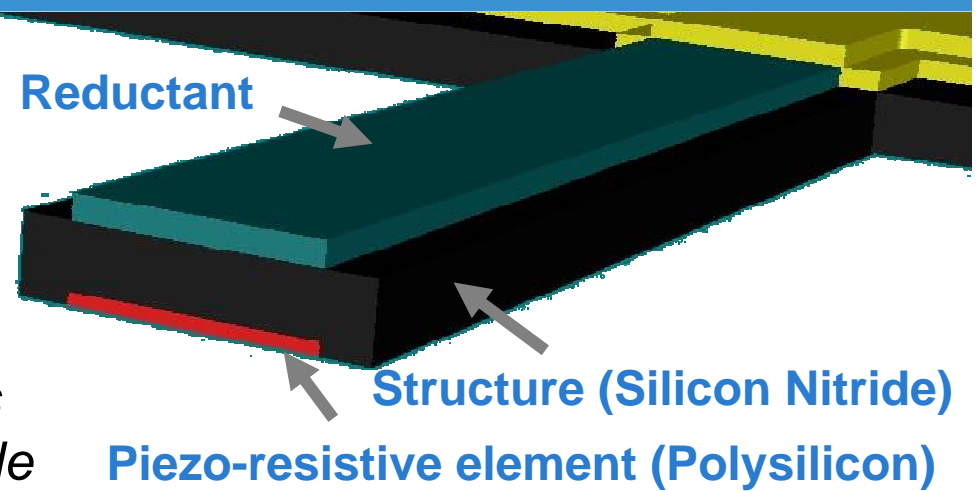


Design.

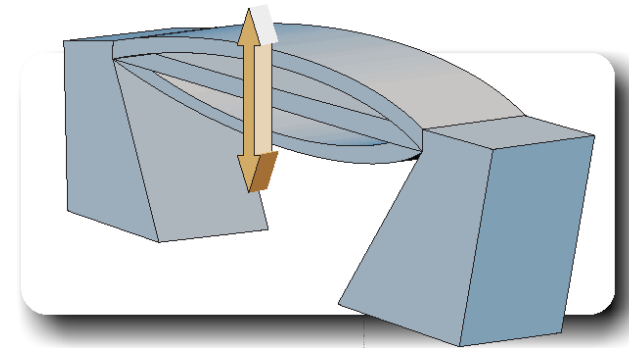
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Our design:

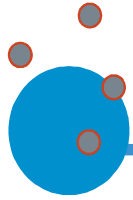
- Micro/nano resonators covered by an oxidizable reductant layer.



Cantilever



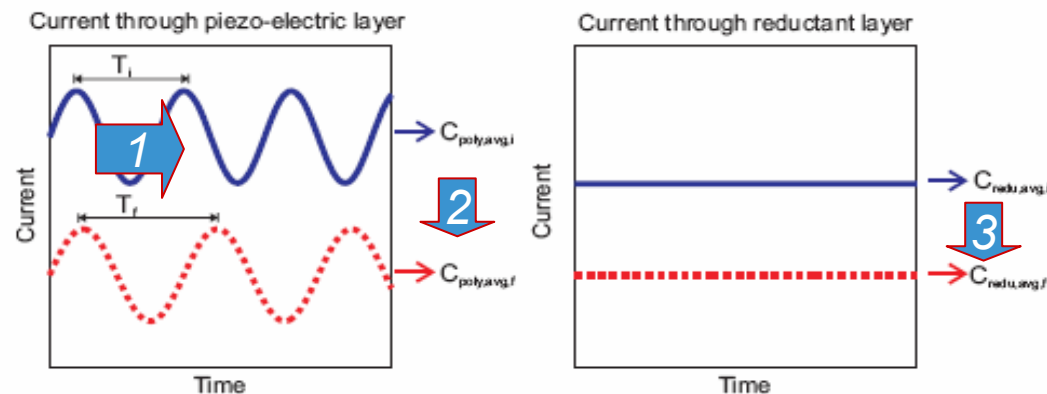
Bridge



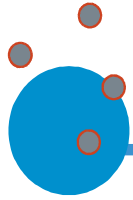
Three simultaneous detection methods:

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1. Dynamic osc. Change in mass and **elastic modulus** (due to oxidation) leads to a shift in oscillation frequency
2. Static bending Introduction of oxide layer leads to static bending
3. Actinometry Oxidation causes a change in the resistance of the reductant layer



Possibility of self
re-calibration



Resonant frequency of a cantilever:

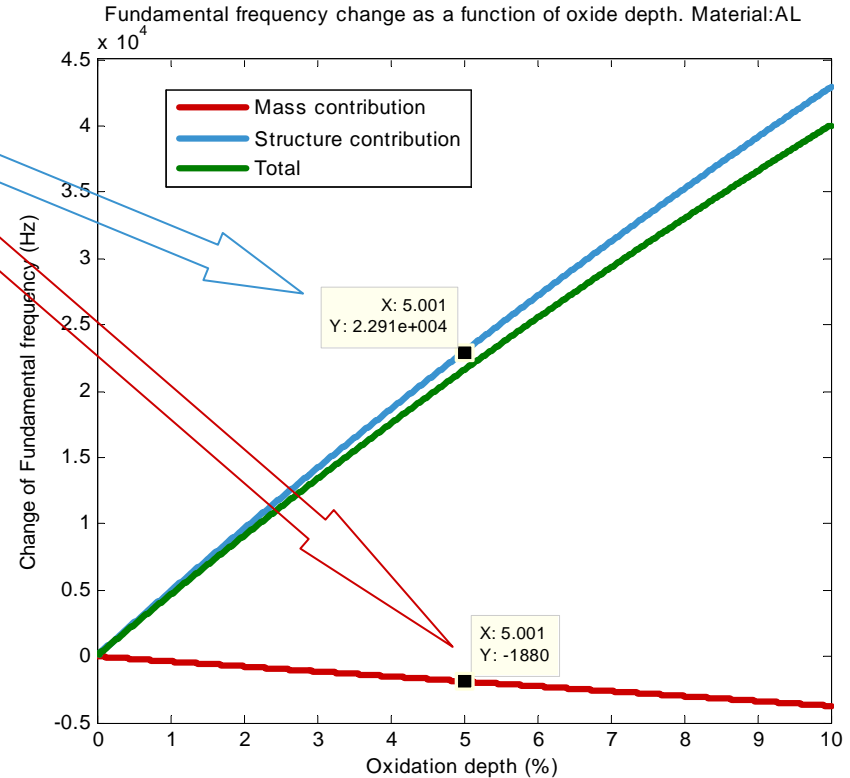
$$f_n = \frac{C_n^2}{4\sqrt{3}\pi} \sqrt{\frac{E}{\rho} \frac{t}{L^2}}$$

where:

- C_n : modal coefficients
- E : elastic modulus
- ρ : density
- t : thickness
- L : length

In this graph:

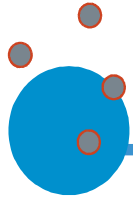
- cantilever
- aluminum reductant
- 10x20 μm
- Layers:
 - 200nm polysilicon
 - 500nm silicon nitride
 - 200nm aluminum



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Theoretical limit of sensitivity: $t_{ox} \sim 2 \times 10^{-10} \text{ m}$

Taking a measurement every minute: $\Delta(\text{Flux}) \sim 2 \times 10^9 \text{ atoms/cm}^2 \cdot \text{s}$

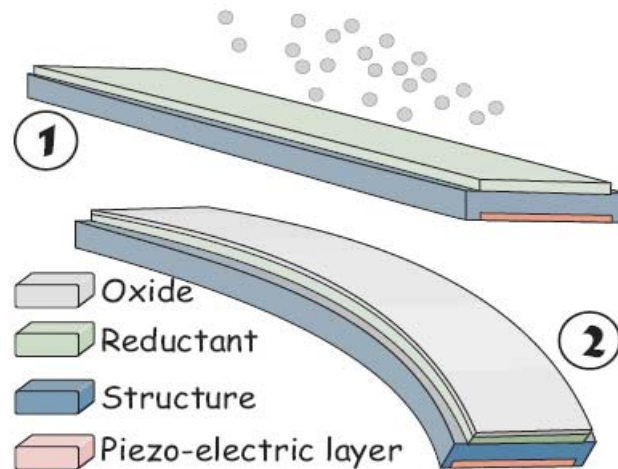


Expansion of reductant material as it oxidizes leads to bending

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Stoney's equation:

$$\sigma = \frac{Et^2}{6R(1 - \nu)t_{ox}}$$

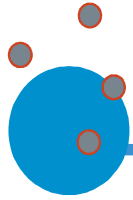


where:

- σ : stress (due to added layer)
- E : elastic modulus
- R : radius of curvature
- t : thickness
- t_{ox} : oxidation thickness
- ν : poisson ratio

Theoretical limit of sensitivity: $t_{ox} \sim 8 \times 10^{-12} \text{ m}$

Taking a measurement every minute: $\Delta(\text{Flux}) \sim 2 \times 10^5 \text{ atoms/cm}^2 \cdot \text{s}$



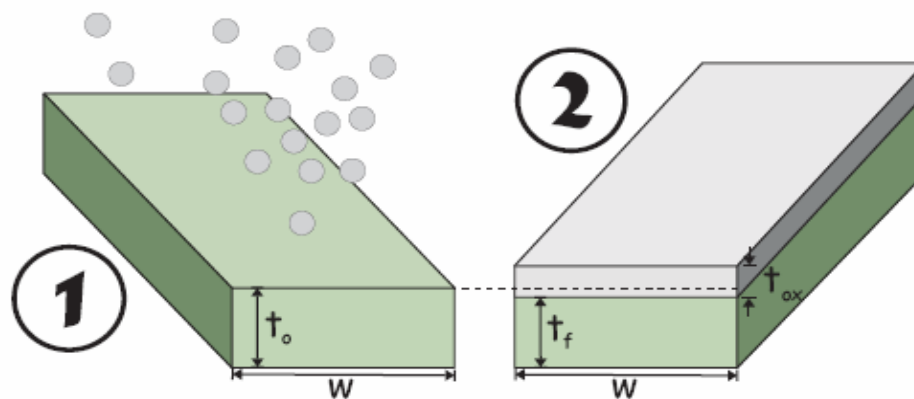
The resistance of the reductant increases as it is replaced by a non conductive oxide.

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$$t_{ox} = t_0 \left(1 - \frac{R_0}{R} \right) PBR$$

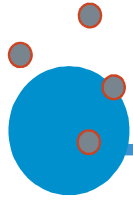
where:

- PBR: Pilling-Bedworth ratio
- R_0 : original resistance
- R : final resistance
- t_0 : original thickness
- t_{ox} : oxidation thickness

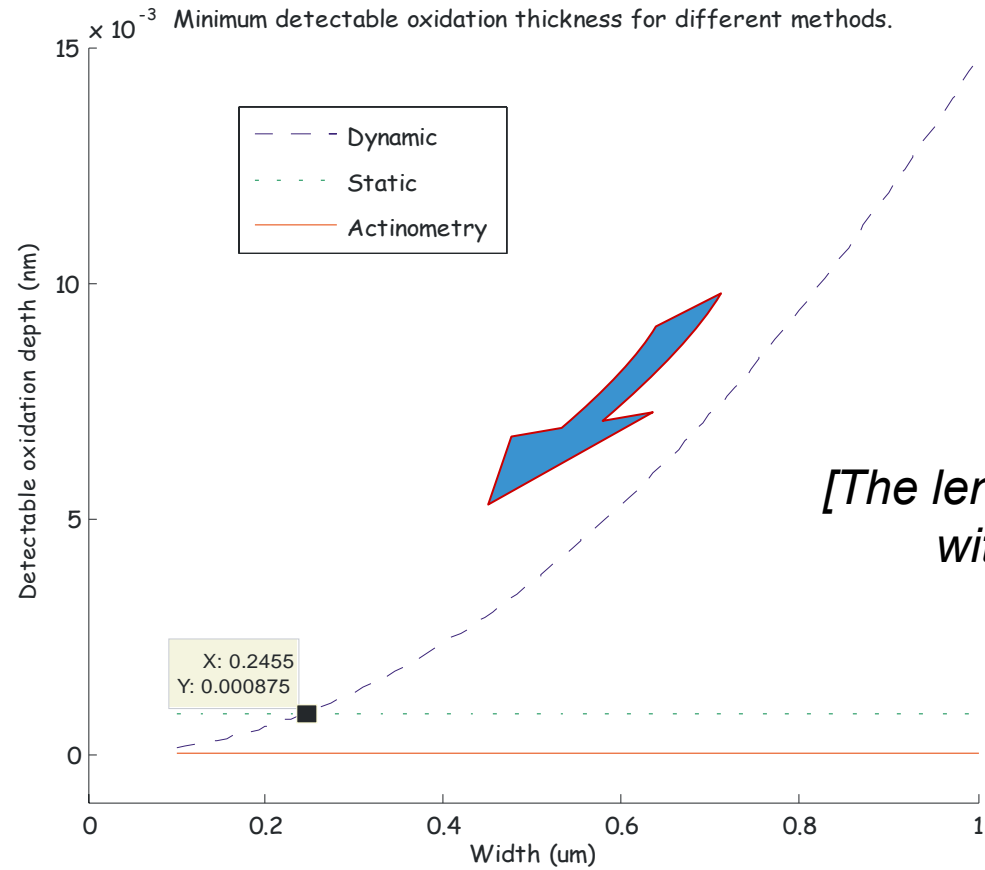


Theoretical limit of sensitivity: $t_{ox} \sim 2 \times 10^{-14} \text{ m}$

Taking a measurement every minute: $\Delta(\text{Flux}) \sim 20 \text{ atoms/cm}^2 \cdot \text{s}$

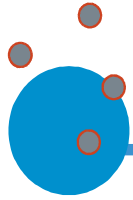


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[The length scales down with the width]

Dynamic sensitivity  as dimensions 



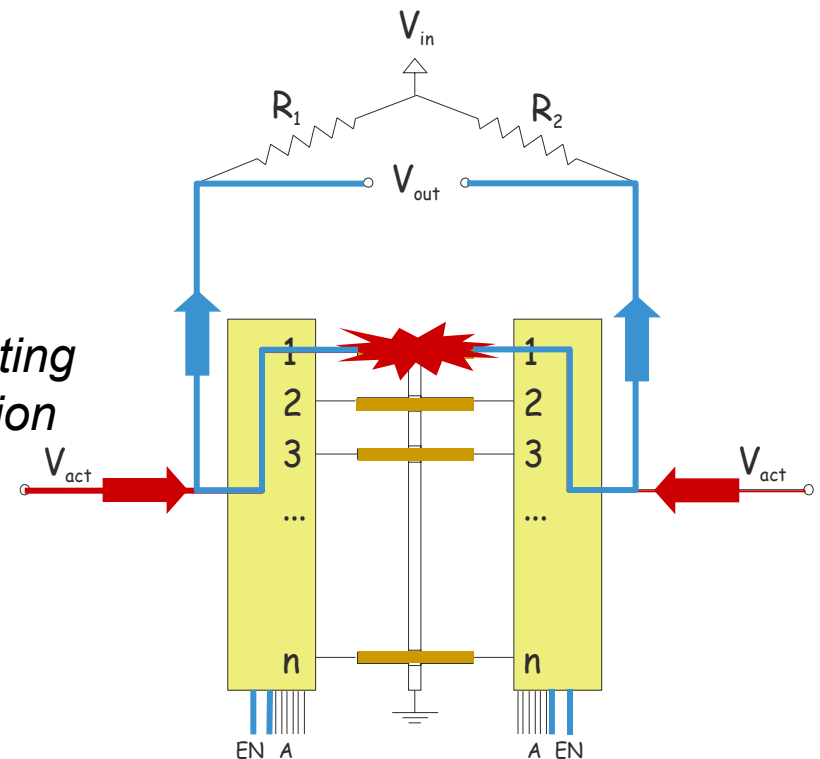
Large *array* of resonators

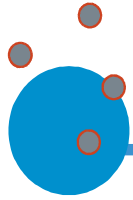
Activate devices as measurements are requested

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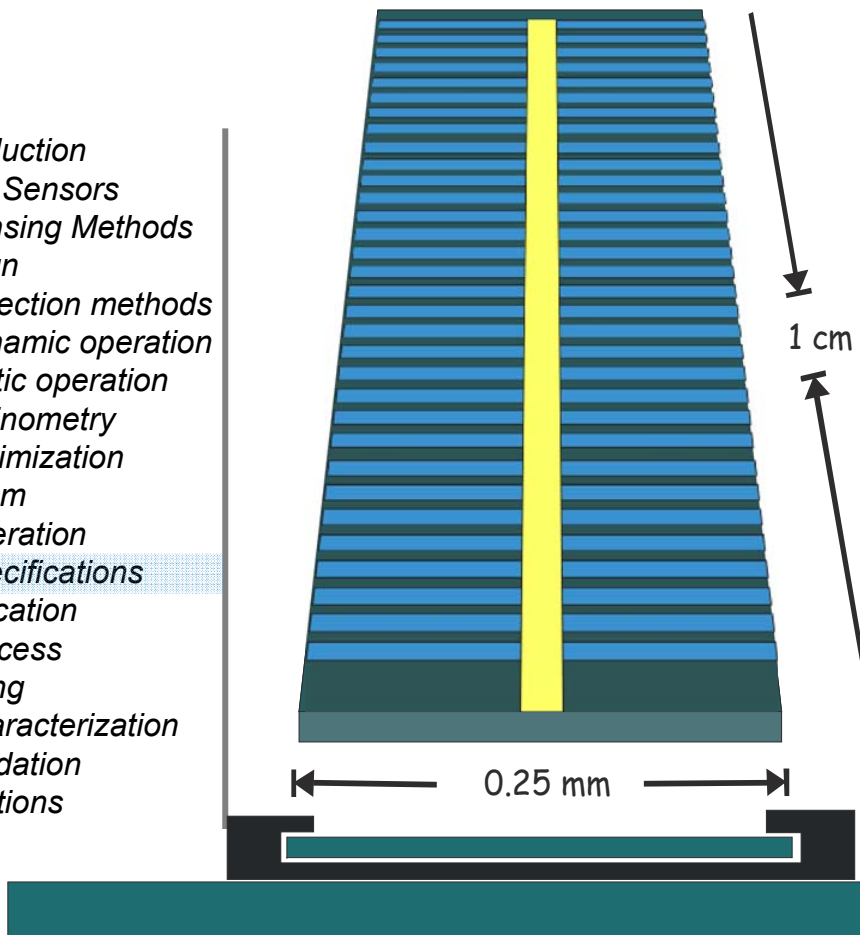
1. Enable circuitry
2. Activate a resonator by heating and removing oxide protection (gold)
3. Make measurement
4. Disable circuitry



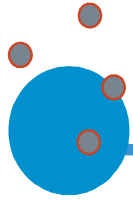


100 bridges

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- **size:**
 - **Device:** 2.5 mm²
 - **Circuitry:** ~10 cm²
- **weight:**
 - **Device:** 3 mg
 - **Circuitry:** 0.5 g
- **Power [shutdown]:**
 - **Device:** ~0.5 mW [50 μW]
 - **Circuitry:** ~10 mW [0.1 μW]



Fabrication. Process

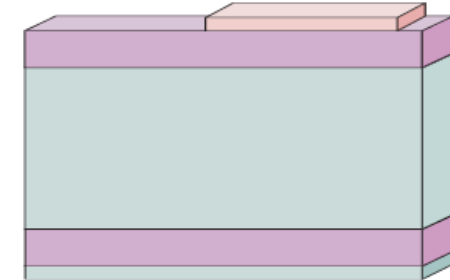
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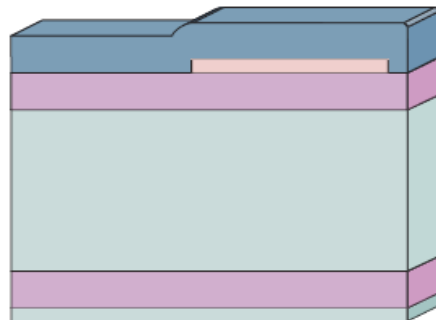
- Starting silicon wafer
- 500 nm oxide deposition
- 200nm LPCVD polysilicon



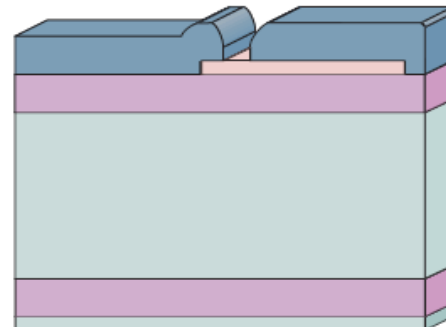
- $\sim 4 \times 10^{15}$ Boron doping in front



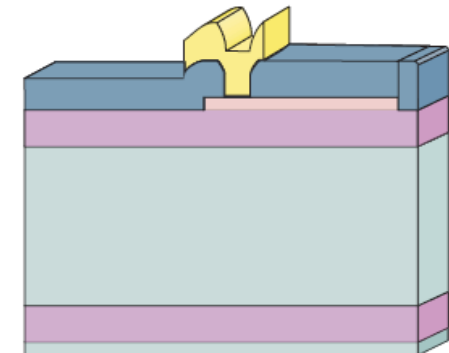
- Piezo-define (RIE)



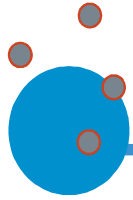
- 500 nm LPCVD nitride



- Nitride define (RIE)

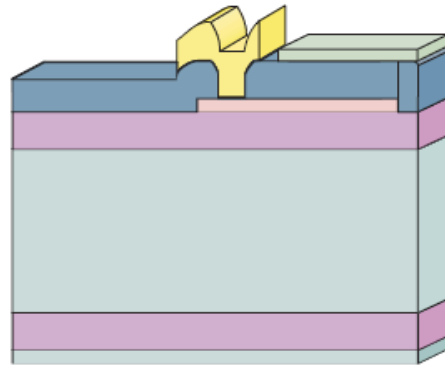


- 500 nm metal deposition (Gold contacts)
- Lift-off

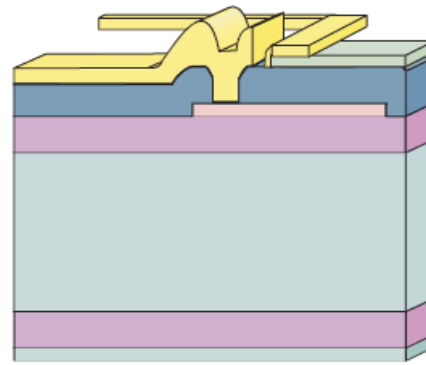


Fabrication. Process

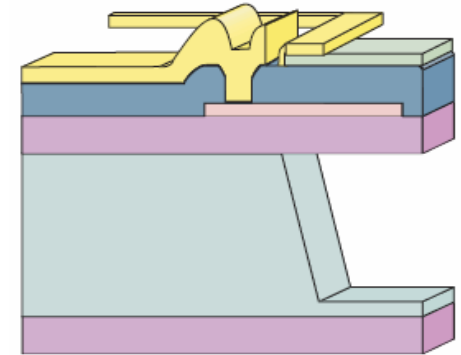
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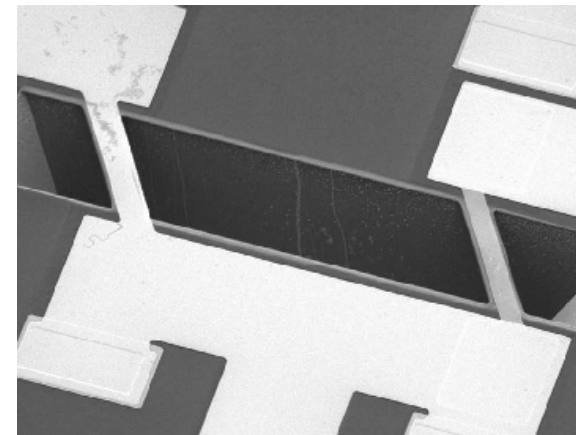
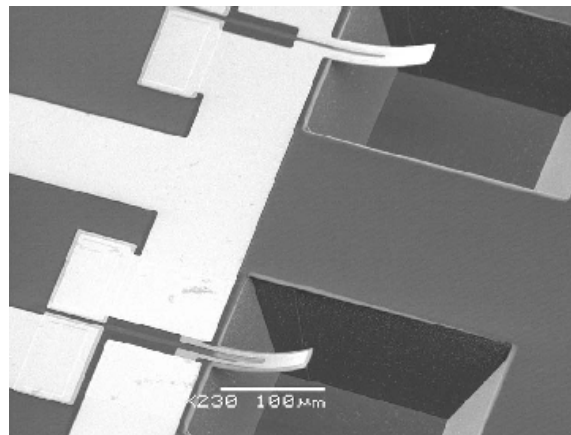
- 200 nm metal deposition (Redu)
- Lift-off

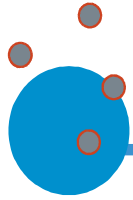


- 200 nm metal deposition (Gold)
- Lift-off



- KOH etch from the front

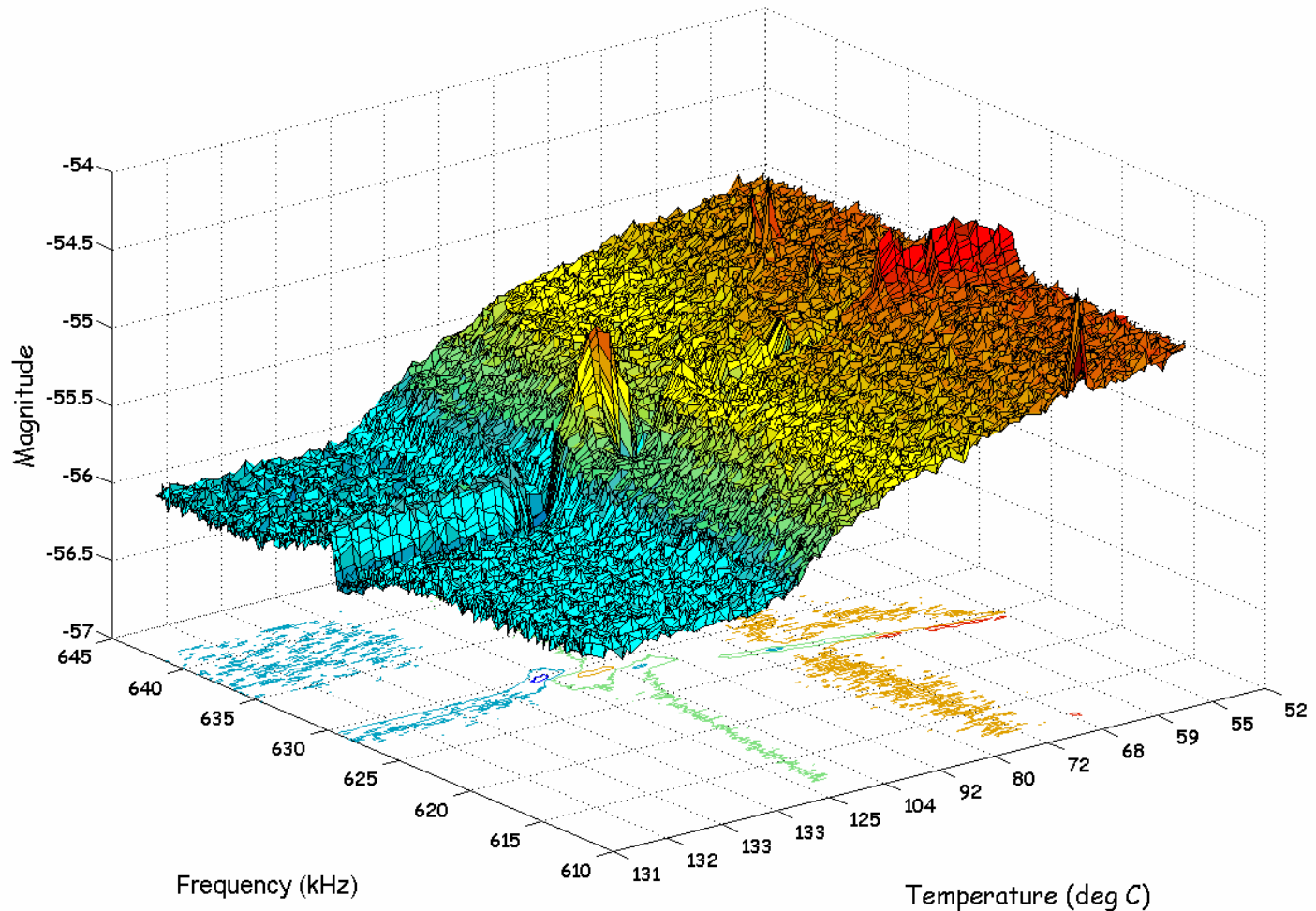




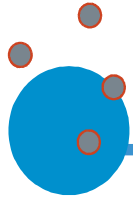
Testing. Characterization

Device: bridge 30x150 μm^2

Temperature response spectrum of resonator device



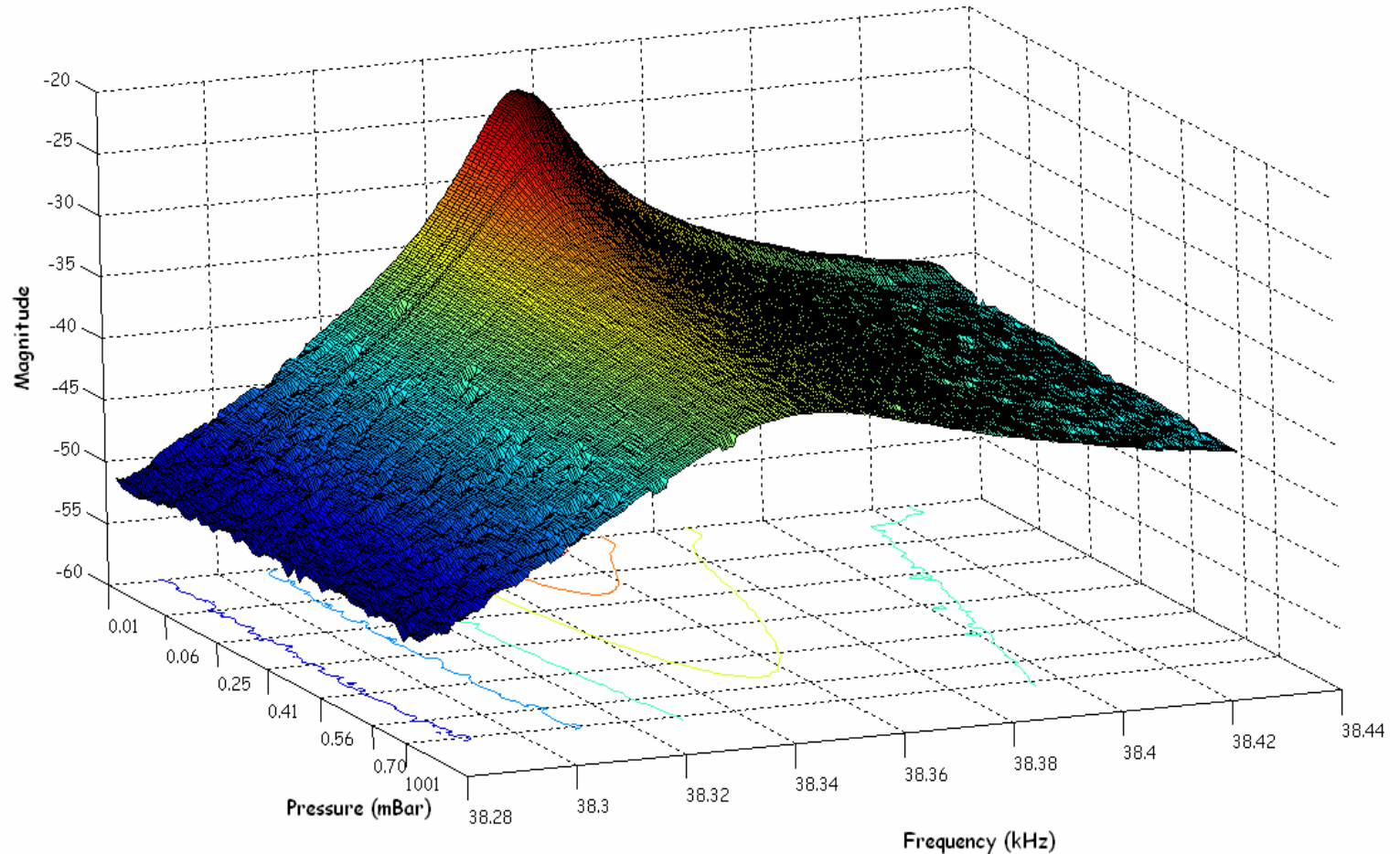
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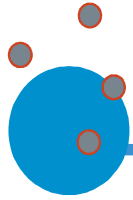
Testing. Characterization

Device: cantilever 30x150 μm

Pressure response spectrum of resonator device: cant 150x30

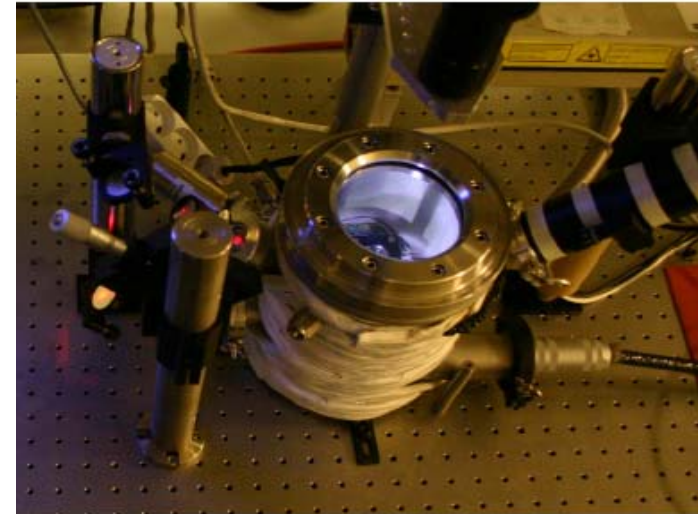


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Oxidization was attempted with temperature regulated vacuum chamber



- ➔ *800 mBar of pure oxygen introduced in the chamber and heated up to 120 °C. 8 hour soak.*
- ➔ *results cannot conclusively support that oxidization occurred*
- ➔ *A better experimental setup will be required to proceed*

The background consists of several overlapping, irregular geometric shapes in three colors: a medium blue, a reddish-brown, and a light grey. The shapes are layered, creating a sense of depth and movement. The word "Questions?" is centered over the composition.

Questions?