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			5c. PROGRAM ELEMENT NUMBER 611102		
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14. ABSTRACT This report summarizes the design, fabrication, and characterization of thermal infrared (IR) imaging arrays operating at room temperature which are based on Y-cut-quartz bulk acoustic wave resonators. A novel method of tracking the resonance frequency based upon the measurement of impedance is presented. High-frequency (240-MHz) micromachined resonators from Y-cut-quartz crystal cuts were fabricated using heterogeneous integration techniques on a silicon wafer. A temperature sensitivity of 22.16 kHz/°C was experimentally					
15. SUBJECT TERMS Project Final Report					
16. SECURITY CLASSIFICATION OF:		17. LIMITATION OF ABSTRACT		15. NUMBER OF PAGES	19a. NAME OF RESPONSIBLE PERSON
a. REPORT UU	b. ABSTRACT UU	c. THIS PAGE UU	UU		Srinivas Tadigadapa
					19b. TELEPHONE NUMBER 814-865-2730

## Report Title

Monolithic Micromachined Quartz Resonator based Infrared Focal Plane Arrays  
FINAL REPORT

### ABSTRACT

This report summarizes the design, fabrication, and characterization of thermal infrared (IR) imaging arrays operating at room temperature which are based on Y-cut-quartz bulk acoustic wave resonators. A novel method of tracking the resonance frequency based upon the measurement of impedance is presented. High-frequency (240-MHz) micromachined resonators from Y-cut-quartz crystal cuts were fabricated using heterogeneous integration techniques on a silicon wafer. A temperature sensitivity of 22.16 kHz/°C was experimentally measured. IR measurements on the resonator pixel resulted in a noise equivalent power of 3.90 nW/Hz<sup>1/2</sup>, a detectivity D<sup>\*</sup> of 1 × 10<sup>5</sup> cmHz<sup>1/2</sup>/W, and a noise equivalent temperature difference of 4 mK in the 8 -14 μm wavelength range. The thermal frequency response of the resonator was determined to be faster than 33 Hz, demonstrating its applicability in video-rate uncooled IR imaging. This work represents the first comprehensive thermal characterization of micromachined Y-cut-quartz resonators and their IR sensing response. In addition the report also summarizes the work done on viscoelastic measurements performed using micromachined quartz resonators and quartz etching work undertaken as part of this work.

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**Enter List of papers submitted or published that acknowledge ARO support from the start of the project to the date of this printing. List the papers, including journal references, in the following categories:**

#### (a) Papers published in peer-reviewed journals (N/A for none)

<u>Received</u>	<u>Paper</u>
2012/05/08 1: 7	Ping Kao, Srinivas Tadigadapa. Micromachined quartz resonator based infrared detector array, Sensors and Actuators A: Physical, (02 2009): 0. doi: 10.1016/j.sna.2008.11.013
2012/05/08 1: 6	Ping Kao, David Allara, Srinivas Tadigadapa. Fabrication and performance characteristics of high-frequency micromachined bulk acoustic wave quartz resonator arrays, measurement science and technology, (12 2009): 0. doi: 10.1088/0957-0233/20/12/124007
2012/05/08 1: 5	S Tadigadapa, K Mateti. Piezoelectric MEMS sensors: state-of-the-art and perspectives, measurement science and technology, (09 2009): 0. doi: 10.1088/0957-0233/20/9/092001
2012/05/08 1: 4	Marcelo B. Pisani, Kailiang Ren, Ping Kao, Srinivas Tadigadapa. Application of Micromachined $\text{Y}$ -Cut-Quartz Bulk Acoustic Wave Resonator for Infrared Sensing, Journal of Microelectromechanical Systems, (02 2011): 0. doi: 10.1109/JMEMS.2010.2100030
2012/05/08 1: 3	Ping Kao, David L. Allara, Srinivas Tadigadapa. Study of Adsorption of Globular Proteins on Hydrophobic Surfaces, IEEE Sensors Journal, (11 2011): 0. doi: 10.1109/JSEN.2011.2157819
2012/05/08 1: 2	Kailiang Ren, Ping Kao, Marcelo B. Pisani, Srinivas Tadigadapa. Monitoring biochemical reactions using Y-cut quartz thermal sensors, The Analyst, (06 2011): 0. doi: 10.1039/c1an15153c
2012/05/08 1: 1	Ping Kao, Purnendu Parhi, Anandi Krishnan, Hyeran Noh, Waseem Haider, Srinivas Tadigadapa, David L. Allara, Erwin A. Vogler. Volumetric interpretation of protein adsorption: Interfacial packing of protein adsorbed to hydrophobic surfaces from surface-saturating solution concentrations, Biomaterials, (2 2011): 0. doi: 10.1016/j.biomaterials.2010.09.075

**TOTAL: 7**

**Number of Papers published in peer-reviewed journals:**

---

#### (b) Papers published in non-peer-reviewed journals (N/A for none)

<u>Received</u>	<u>Paper</u>
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**TOTAL:**

Number of Papers published in non peer-reviewed journals:

---

(c) Presentations

Number of Presentations: 0.00

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**Non Peer-Reviewed Conference Proceeding publications (other than abstracts):**

Received                  Paper

**TOTAL:**

Number of Non Peer-Reviewed Conference Proceeding publications (other than abstracts):

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**Peer-Reviewed Conference Proceeding publications (other than abstracts):**

Received                  Paper

- 2012/05/08 21 14 Ping Kao, Matthew P Chang, David Allara, Srinivas Tadigadapa. Investigation of spontaneously adsorbed globular protein films using high-frequency bulk acoustic wave resonators, 2010 Ninth IEEE Sensors Conference (SENSORS 2010). 2010/11/01 00:00:00, Kona, HI. : ,
- 2012/05/08 21 15 D. Allara, S. Tadigadapa, P. Kao. Label free piezoelectric DNA sensor array by selective immobilization via electrochemical method, 2011 IEEE 24th International Conference on Micro Electro Mechanical Systems (MEMS). 2011/01/23 00:00:00, Cancun, Mexico. : ,
- 2012/05/08 21 16 M.B. Pisani, P. Kao, S. Tadigadapa. Bulk acoustic wave resonators for infrared detection applications, TRANSDUCERS 2009 - 2009 International Solid-State Sensors, Actuators and Microsystems Conference. 2009/06/21 00:00:00, Denver, CO, USA. : ,
- 2012/05/08 21 12 Son Vu Hoang Lai, Ping Kao, Srinivas Tadigadapa. Thermal biosensors from micromachined bulk acoustic wave resonators, Eurosensors XXV Conference. 2011/09/04 00:00:00, . : ,
- 2012/05/08 21 11 Hwall Min, David Allara, Srinivas Tadigadapa. Investigation of the Viscoelastic Properties of Liquids Trapped in Nanoporous Cavities using Micromachined Acoustic Transducers, Eurosensors XXV Conference. 2011/09/04 00:00:00, . : ,
- 2012/05/08 21 10 Nichole Sullivan, Hwall Min, David Allara, Srinivas Tadigadapa. Nanoporous Gold: A High Sensitivity and Specificity Biosensing Substrate, Eurosensors XXV. 2011/09/04 00:00:00, . : ,
- 2012/05/08 21 9 Marcelo B. Pisani, Kailiang Ren, Ping Kao, Srinivas Tadigadapa. Room temperature infrared imaging array fabricated using heterogeneous integration methods, Eurosensors XXIV Conference. 2010/09/05 00:00:00, . : ,
- 2012/05/08 21 13 Ping Kao, Matthew P. Chang, David Allara, Srinivas Tadigadapa. Systematic studies on globular proteins using micromachined high frequency bulk acoustic wave resonators, Eurosensor XXIV Conference. 2010/09/05 00:00:00, . : ,
- 2012/05/08 11 8 Kailiang Ren, Marcelo Pisani, Ping Kao, and Srinivas Tadigadapa. Micromachined Quartz Resonator based High Performance Thermal Sensors, IEEE Sensors Conference. 2010/11/01 00:00:00, . : ,

**TOTAL: 9**

Number of Peer-Reviewed Conference Proceeding publications (other than abstracts):

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**(d) Manuscripts**

Received                  Paper

**TOTAL:**

**Number of Manuscripts:**

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**Books**

Received                  Paper

**TOTAL:**

**Patents Submitted**

Fabrication Method and Detection Enhancement Strategy for Ultra-Sensitive Temperature Detection Using Resonant  
~~Devices~~

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**Patents Awarded**

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**Awards**

Presented Paper selected one of the Best Papers presented at IEEE SENSORS 2010 in Hawaii and published as a paper in a  
Special Issue of IEEE Sensors Journal.

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**Graduate Students**

<u>NAME</u>	<u>PERCENT SUPPORTED</u>	Discipline
Ping Kao	0.10	
<b>FTE Equivalent:</b>	<b>0.10</b>	
<b>Total Number:</b>	<b>1</b>	

---

**Names of Post Doctorates**

<u>NAME</u>	<u>PERCENT SUPPORTED</u>
Marcelo Pisani	1.00
<b>FTE Equivalent:</b>	<b>1.00</b>
<b>Total Number:</b>	<b>1</b>

---

**Names of Faculty Supported**

<u>NAME</u>	<u>PERCENT SUPPORTED</u>	National Academy Member
Srinivas Tadigadapa	0.10	
<b>FTE Equivalent:</b>	<b>0.10</b>	
<b>Total Number:</b>	<b>1</b>	

---

**Names of Under Graduate students supported**

<u>NAME</u>	<u>PERCENT SUPPORTED</u>	Discipline
Matthew Chang	0.05	Electrical Engineering
<b>FTE Equivalent:</b>	<b>0.05</b>	
<b>Total Number:</b>	<b>1</b>	

**Student Metrics**

This section only applies to graduating undergraduates supported by this agreement in this reporting period

- The number of undergraduates funded by this agreement who graduated during this period: ..... 1.00
- The number of undergraduates funded by this agreement who graduated during this period with a degree in science, mathematics, engineering, or technology fields:..... 1.00
- The number of undergraduates funded by your agreement who graduated during this period and will continue to pursue a graduate or Ph.D. degree in science, mathematics, engineering, or technology fields:..... 1.00
- Number of graduating undergraduates who achieved a 3.5 GPA to 4.0 (4.0 max scale):..... 1.00
- Number of graduating undergraduates funded by a DoD funded Center of Excellence grant for Education, Research and Engineering:..... 0.00
- The number of undergraduates funded by your agreement who graduated during this period and intend to work for the Department of Defense ..... 0.00
- The number of undergraduates funded by your agreement who graduated during this period and will receive scholarships or fellowships for further studies in science, mathematics, engineering or technology fields: ..... 1.00

**Names of Personnel receiving masters degrees**

<u>NAME</u>
<b>Total Number:</b>

**Names of personnel receiving PHDs**

<u>NAME</u>
Ping Kao
<b>Total Number:</b>
<b>1</b>

**Names of other research staff**

<u>NAME</u>	<u>PERCENT SUPPORTED</u>
<b>FTE Equivalent:</b>	
<b>Total Number:</b>	

**Sub Contractors (DD882)**

## **Inventions (DD882)**

### **5 Fabrication Method and Detection Enhancement Strategy for Ultra-Sensitive Temperature Detection Using Resonant Devices**

Patent Filed in US? (5d-1) Y

Patent Filed in Foreign Countries? (5d-2) N

Was the assignment forwarded to the contracting officer? (5e) N

Foreign Countries of application (5g-2):

5a: Marcelo Pisani

5f-1a: The Pennsylvania State University

5f-c: 121 Electrical Engineering East Building

University Park PA 16802

5a: Srinivas Tadigadapa

5f-1a: The Pennsylvania State University

5f-c: 121 Electrical Engineering East Building

University Park PA 16802

## **Scientific Progress**

Please see the attached Presentation File

## **Technology Transfer**

# Project Title: Monolithic Micromachined Quartz Resonator based Infrared Focal Plane Arrays

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**Project PI: Srinivas Tadigadapa**

Department of Electrical Engineering

N-237 Millennium Science Center

Pennsylvania State University, University Park, PA 16802

Tel: 814 865 2730, Fax: 814 865 7065, e-mail: [sat10@psu.edu](mailto:sat10@psu.edu)

## **FINAL REPORT**

Grant # W911NF-07-1-0327

Project Start Date: June 2007

Period of Reporting: August 2010 – April 2012

**Program Manager: Dr. William Clark, Army Research Office**

# Main Goals of the Proposed Work

- In this proposal we aim to demonstrate IR staring arrays based on quartz crystal oscillators with a pitch of  $\sim 50 \mu\text{m}$ , NEDT of  $\sim 10 \text{ mK}$  and a response time of  $\sim 10 \text{ ms}$  eventually allowing for 100 frames/s video rate in the 8-14  $\mu\text{m}$  IR wavelength range.
- A \$25,000 addition to the proposal was added in November 2011 to explore high speed etching of glass using nitrogen trifluoride based gas.

# Personnel Working on the Project

- Dr. Srinivas Tadigadapa, Professor, Penn State University (Principal Investigator)
- Dr. Marcelo Pisani, Post Doctoral Research Associate (Project Technical Leader)
- Dr. Ping Kao (Graduated with Ph.D. partially supported by this grant)
- Mr. Mathew Chang, BS Student (Data Acquisition Program Development)

# Summary of Achievements

- The first prototypes of integrated micromachined IR detectors consisting of 200  $\mu\text{m}$  diameter QCM resonators, 241 MHz (6.9  $\mu\text{m}$ ) thick 5 x 5 resonator arrays from Y-cut Quartz have been successfully fabricated. These are the thinnest Y-cut resonators fabricated to date.
- Established process flow to fabricate arrays of small QCM devices.
- A novel measurement method for the real-time tracking of the resonance frequency based on impedance measurement around the resonance frequency has been established.
- A patent disclosure was filed on the findings and methods of this work.
- Nitrogen trifluoride based glass etching process was started as part of this project and is still in progress.
- One student obtained his Ph.D. partially supported by this project and one post-doctoral research associate was trained on the project.

# Report Overview

- The report is divided into the following main sections:
  - IR Detectors design, fabrication, and performance
  - Use of miniaturized quartz resonators for viscoelastic measurement applications (additional work done as part of this project)
  - Nitrogen trifluoride based etching of glass and quartz

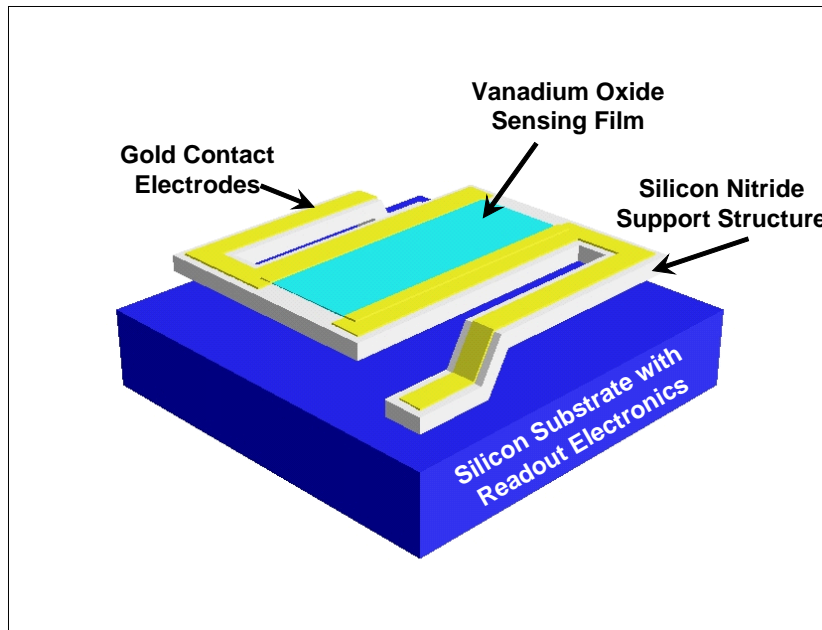


# **SUMMARY OF QUARTZ RESONATOR BASED ROOM TEMPERATURE INFRARED IMAGING ARRAY**

# Thermal IR Sensing

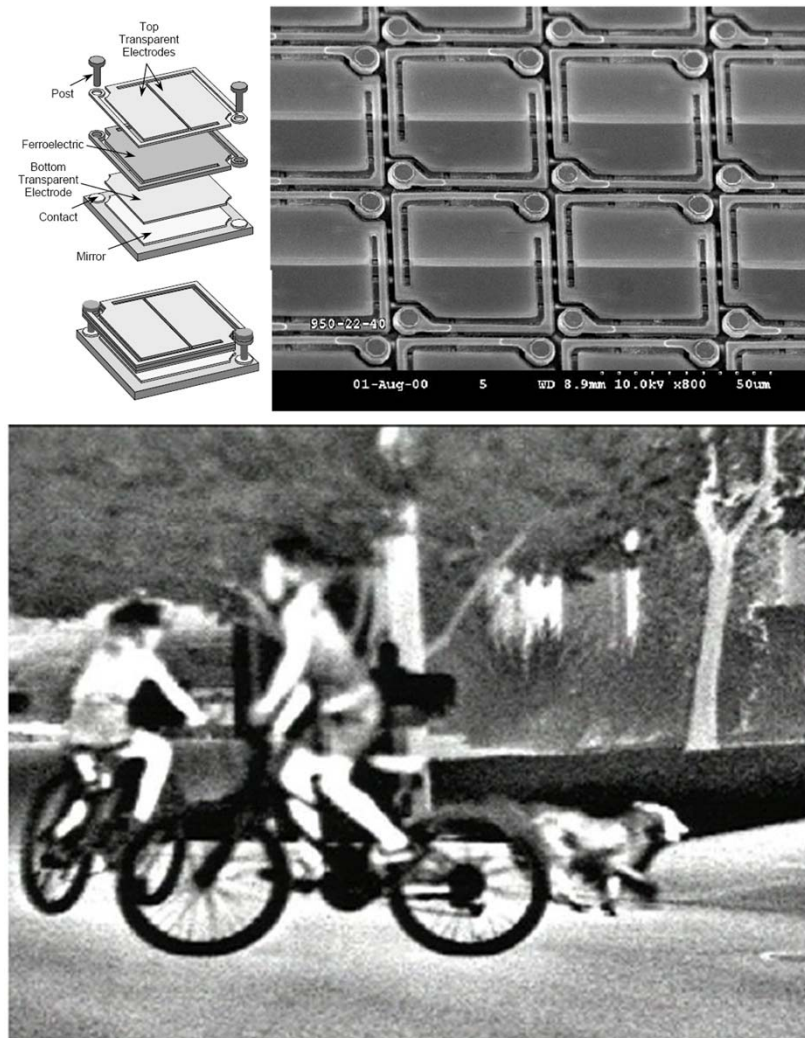
- Thermal infrared detectors are broadband detectors and can be operated at room temperature without cooling.
- They have lower detectivity and slower response time in comparison to cooled semiconductor photonic detectors.
- Micromachined thermal detectors owing to their small thermal mass and good thermal isolation demonstrate much improved performance.
- These detectors can be designed to operate near the room temperature thermodynamic noise limit arising from the thermal conductance fluctuation between the sensing element and the supporting substrate.

# Thermal Infrared Detectors: State of the Art



Vanadium Oxide based IR Detector configuration introduced by Honeywell in late 1990's & Perfected in 2000's.

Lanthanum-doped lead zirconate titanate based focal plane array – developed by TI.



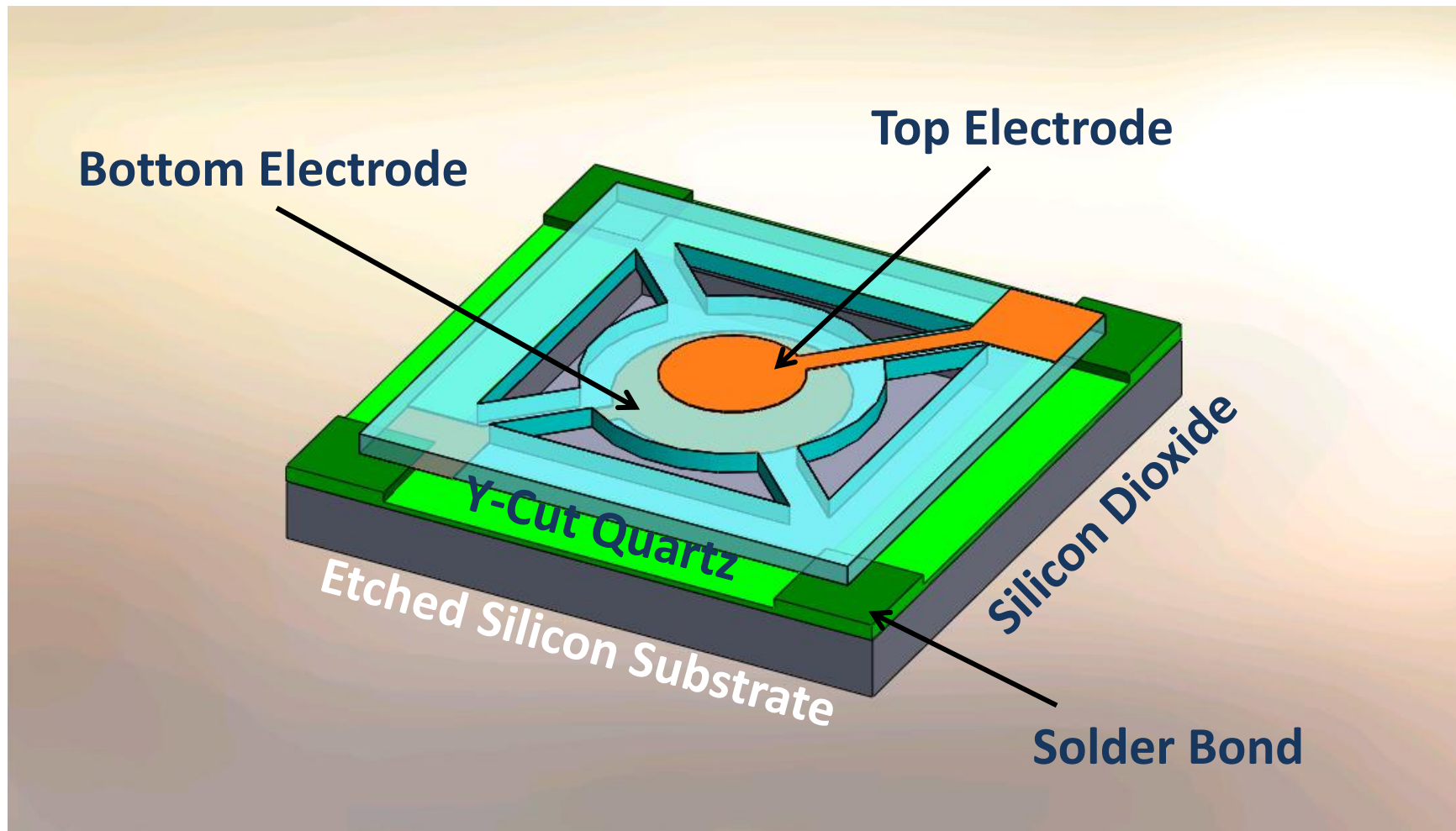
Wood R A, Proc. SPIE 2020 322–329, 1993

Hanson C M, Beratan H R, and Belcher J F, Proc. SPIE 4288 298–303, 2001

# Quartz IR Detector: Goals

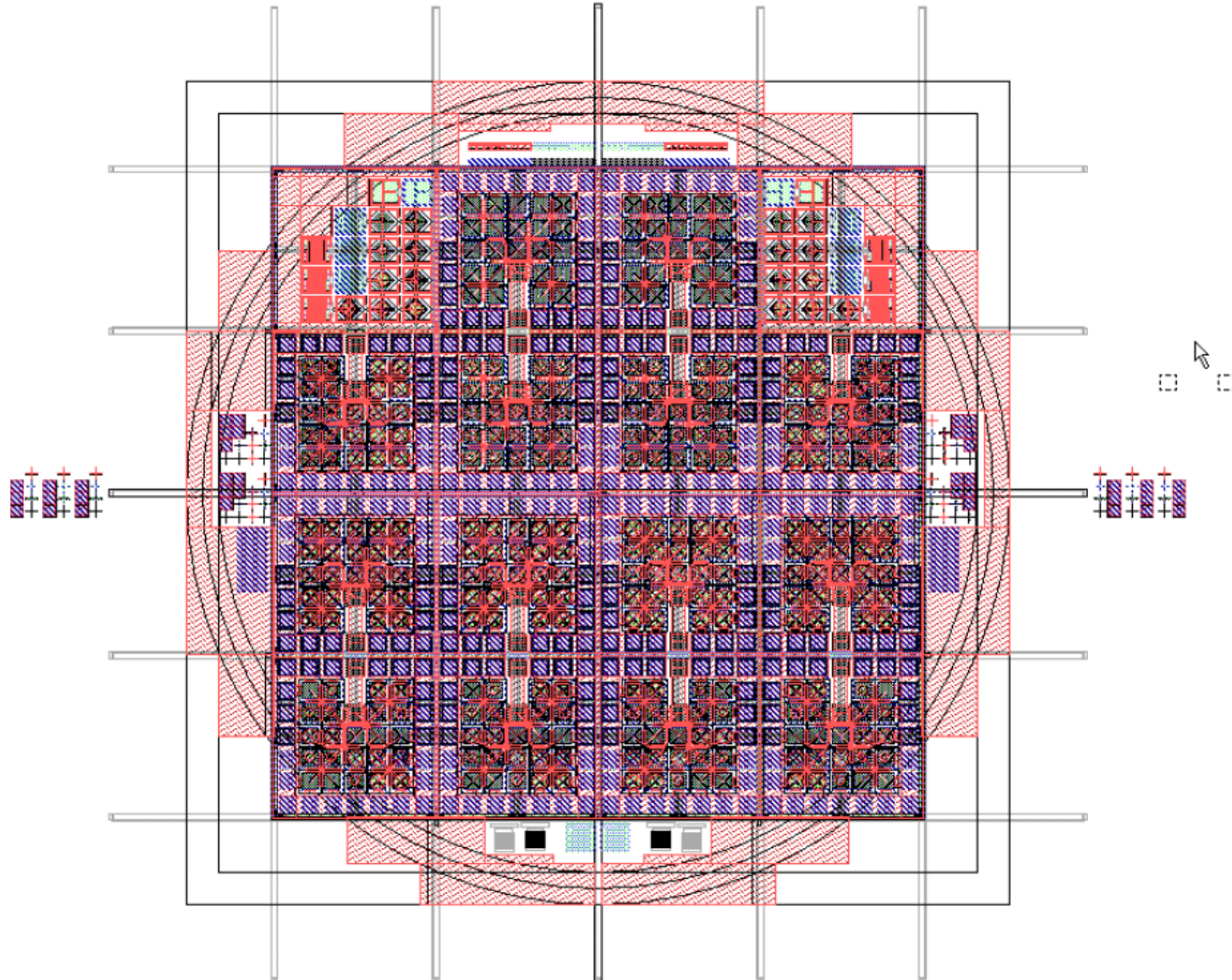
- Y-cut quartz is a sensitive temperature sensor capable of temperature resolution in  $\mu\text{K}$ .
- Develop micromachining process compatible for the production of  $10\ \mu\text{m}$  thick quartz resonators with minimal damping characteristics.
- This work demonstrates IR staring arrays based on quartz crystal oscillators:
  - Pitch of  $\sim 500\ \mu\text{m}$  (Oscillator size  $200\ \mu\text{m}$ )
  - NEDT of  $<10\ \text{mK}$
  - Response time of  $\sim 10\ \text{ms}$  allowing for 100 frames/s video rate in the  $8\text{-}14\ \mu\text{m}$  IR wavelength range.
- Develop thermal models to optimize the performance of the devices.

# Conceptual Drawing of the Resonator

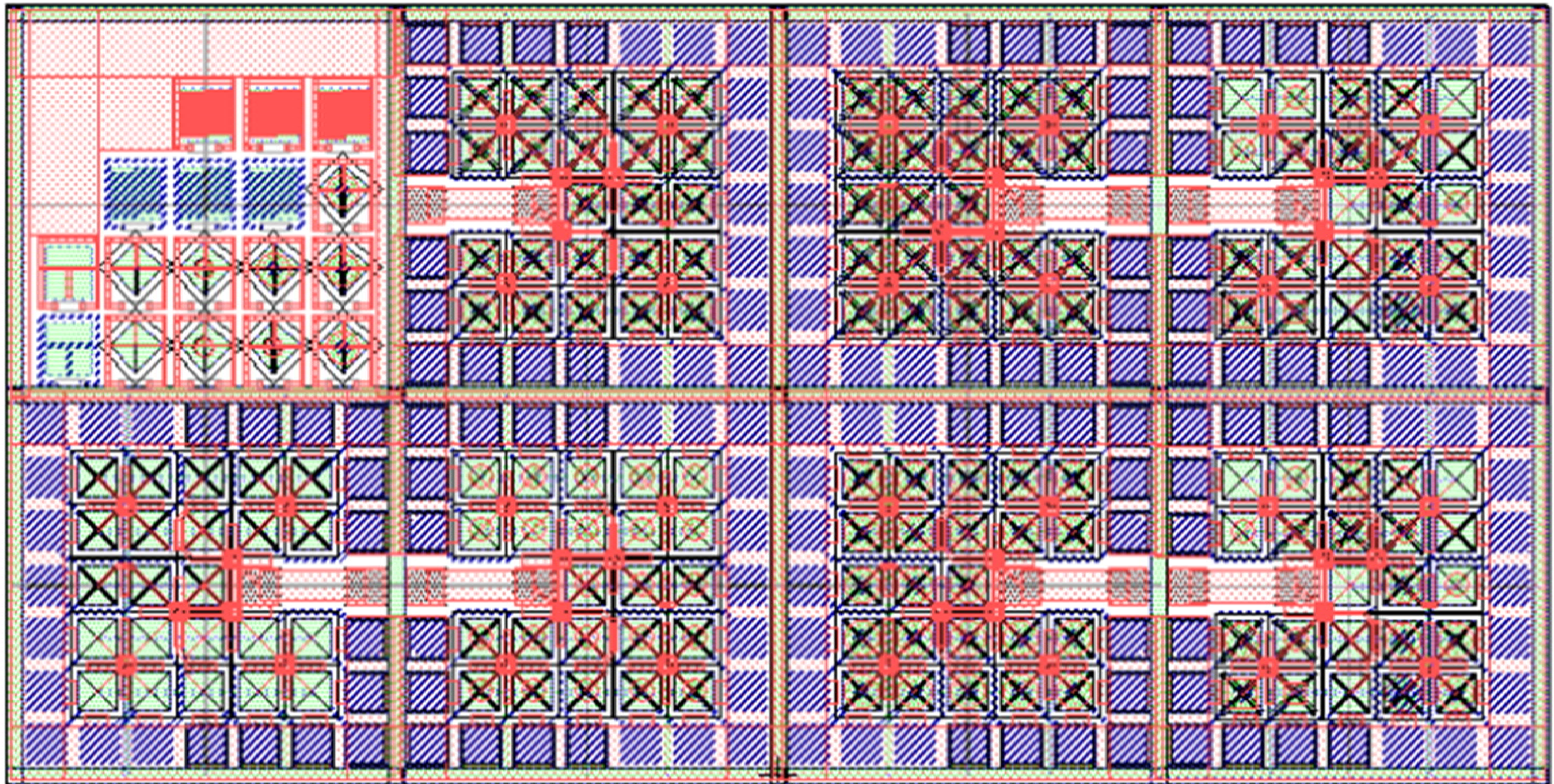


# Device Design and Mask Layout

# Overview of the Mask



# Chip Designs on the Wafer

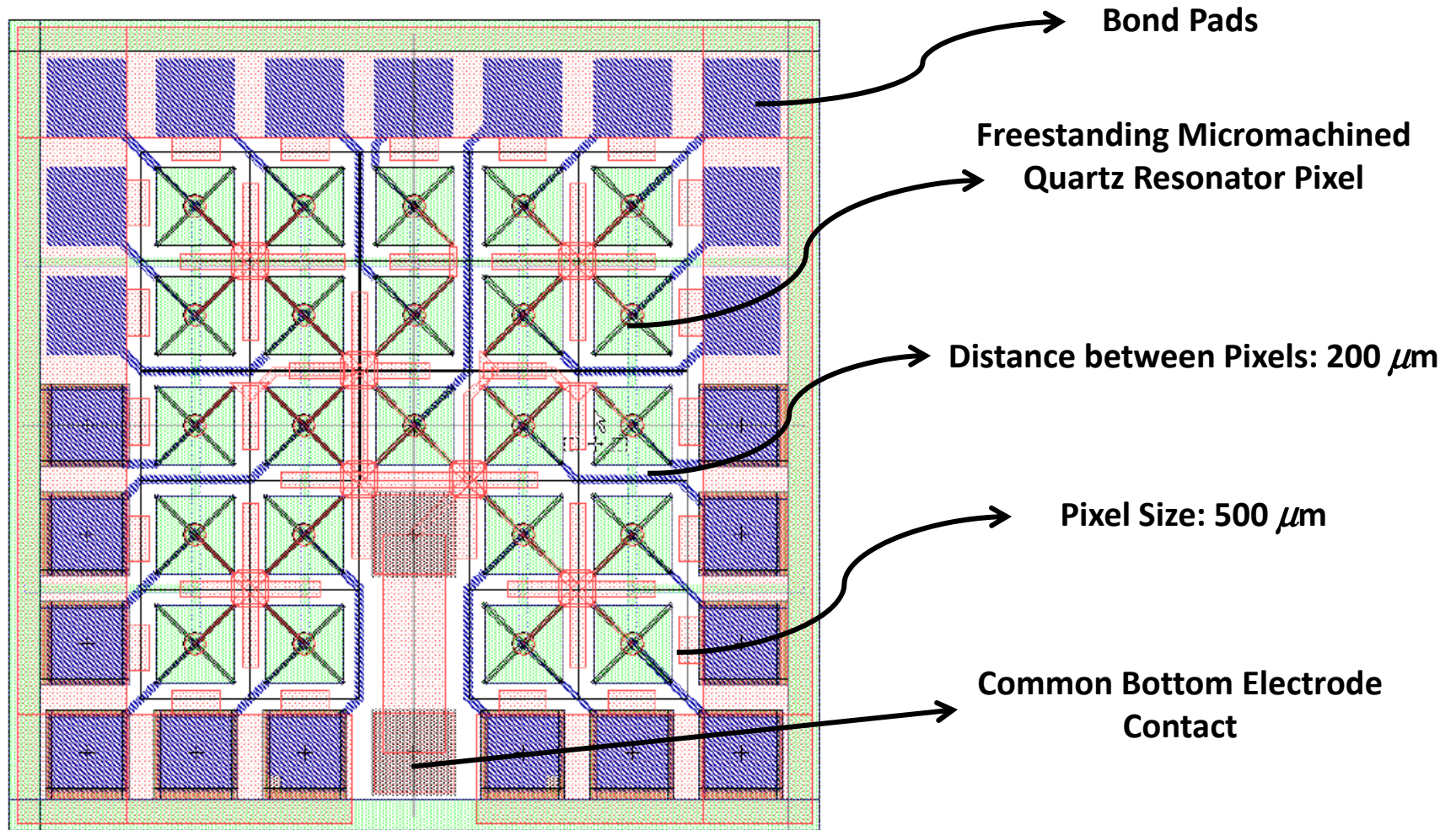


Resonator Size: 225  $\mu\text{m}$

Resonator Size: 150  $\mu\text{m}$

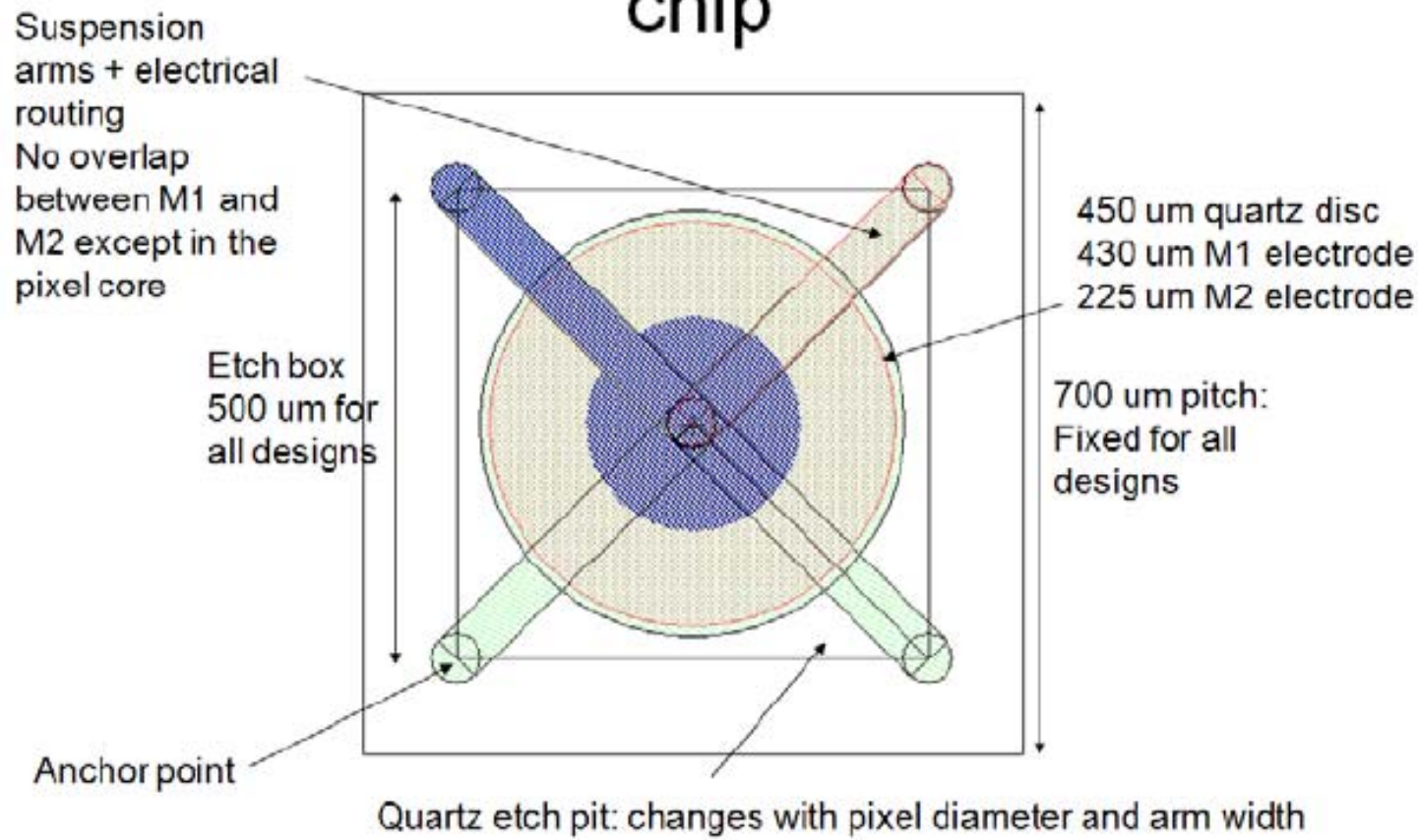
Resonator Size: 75  $\mu\text{m}$

# Zoom of One Chip

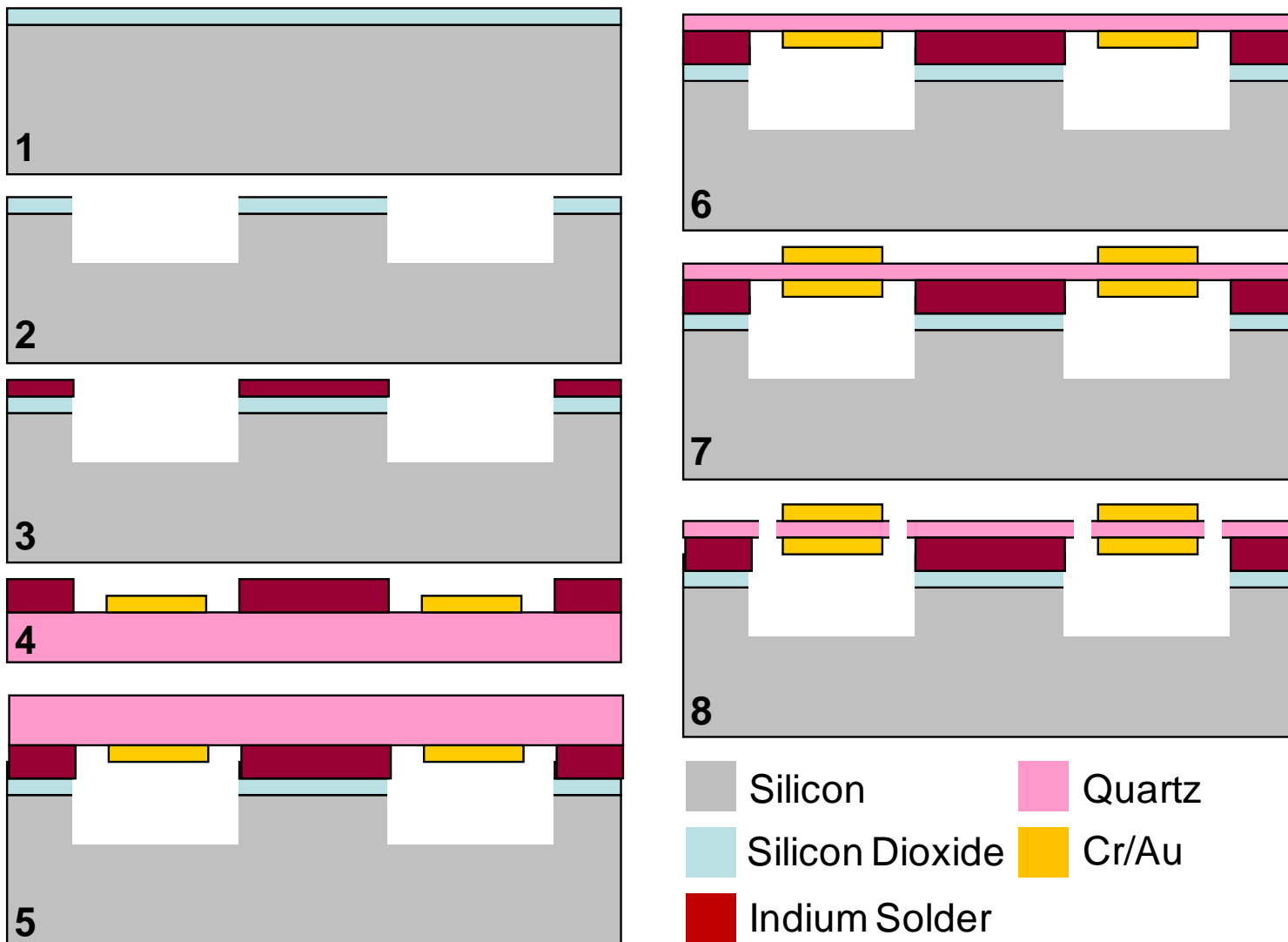


# Details of Individual Pixel

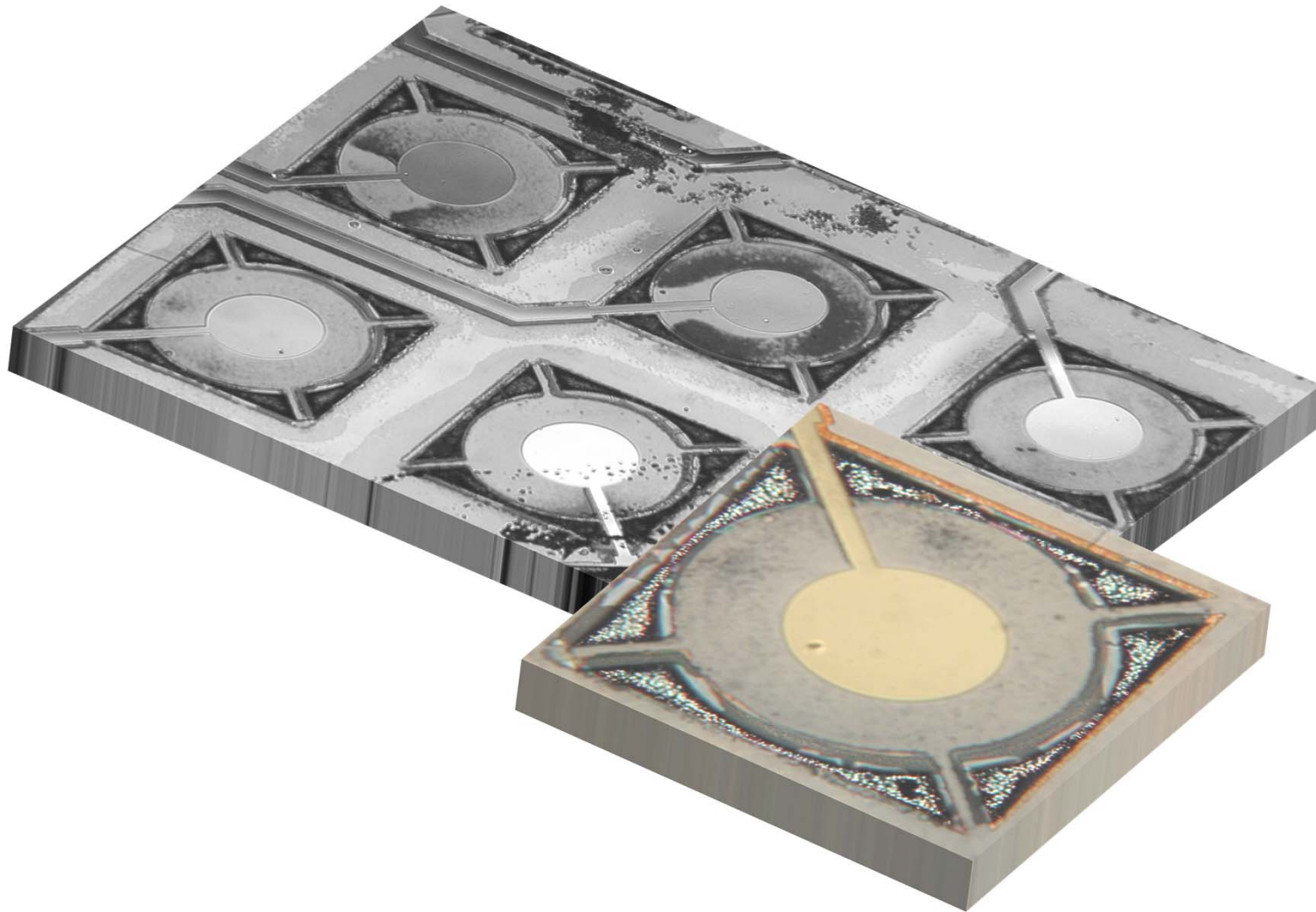
Pixel core: change from chip to chip



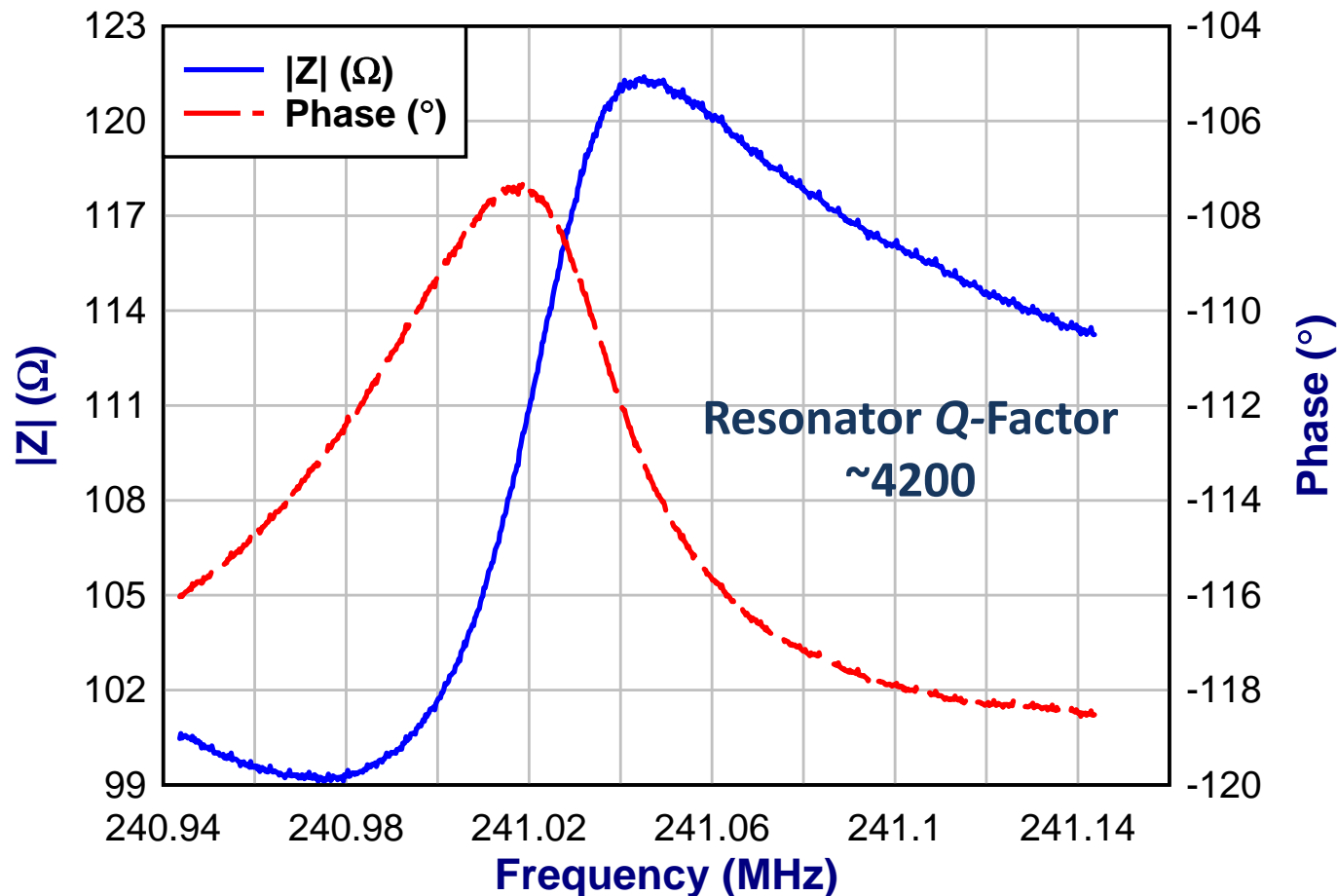
# Fabrication Process Flow



# Fabricated Device Photos



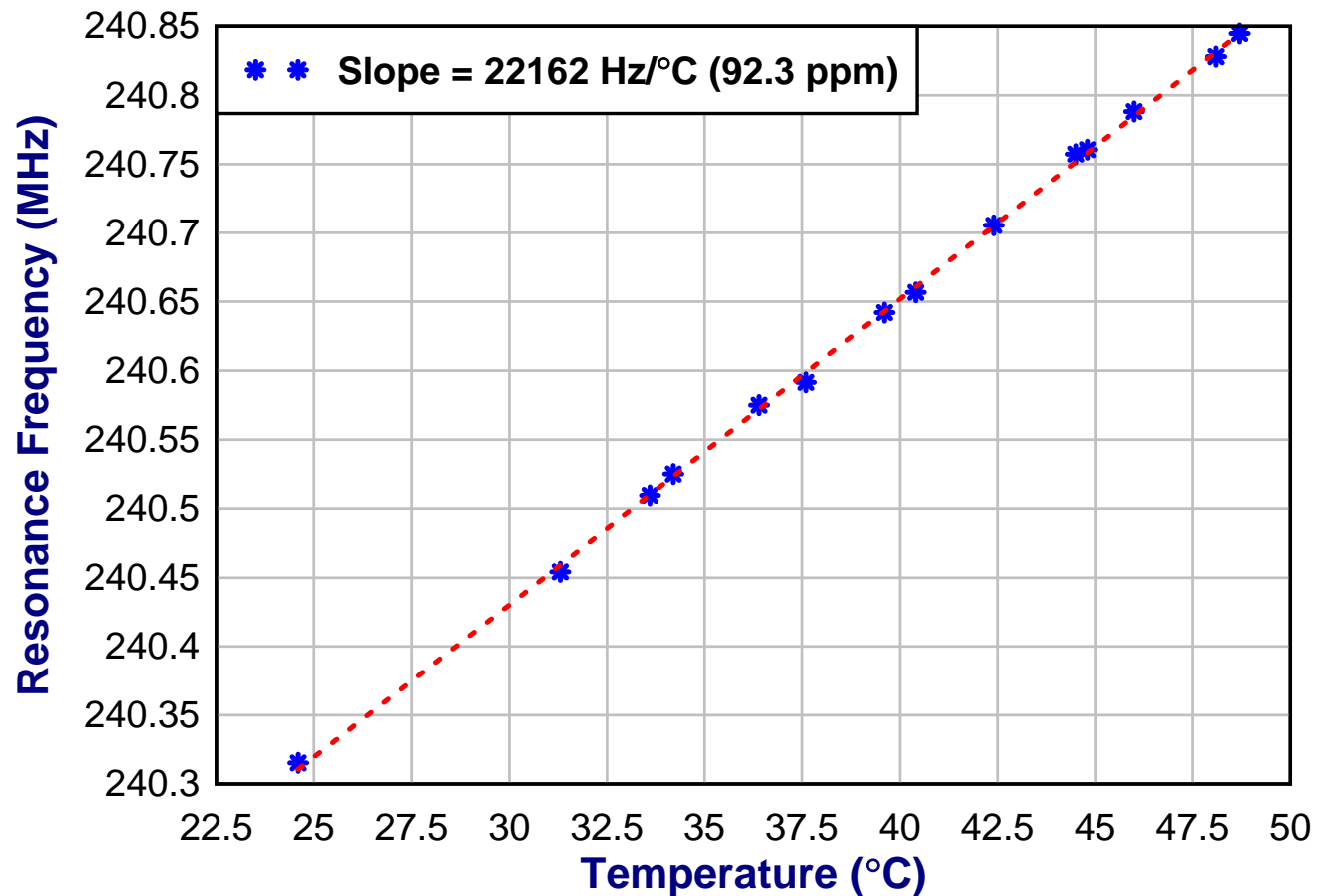
# Resonance Characteristics



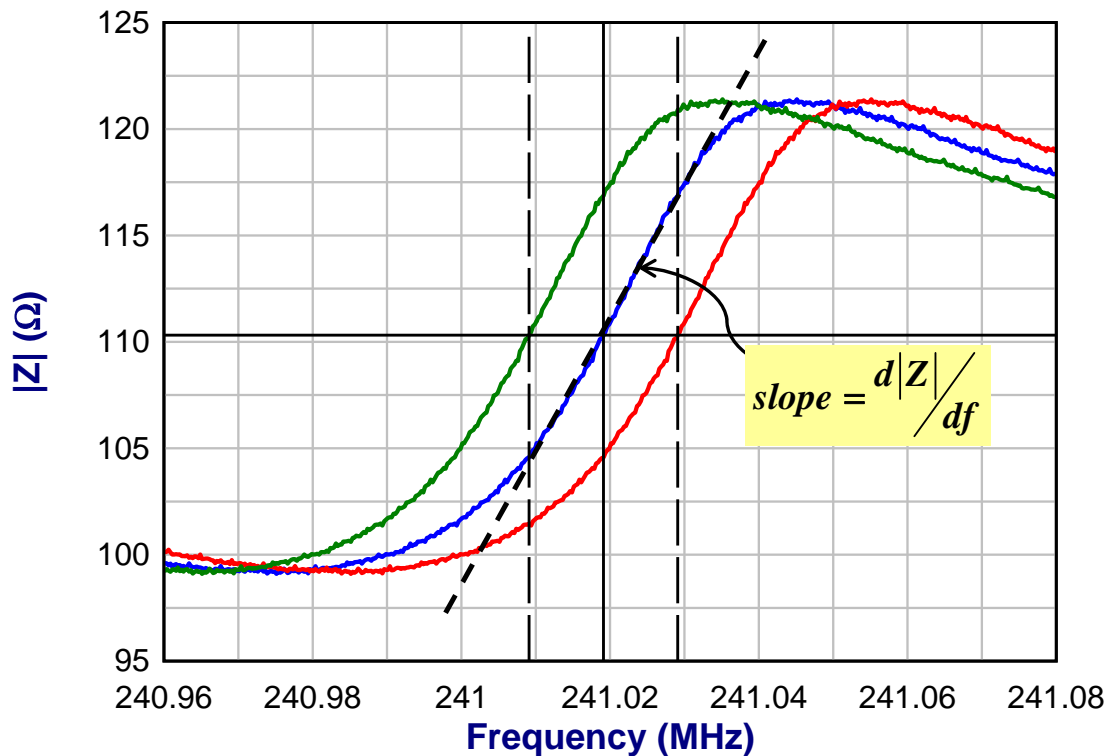
A resonance frequency of 241 MHz implies a resonator thickness of  $6.9 \mu\text{m}$ .

# Y-cut Quartz

## Temperature Sensitivity of Resonance



# Measurement of Resonance Frequency Shift in Impedance Domain



Normal sensing of the resonance curve involves measurement of the resonance frequency peak in real-time.

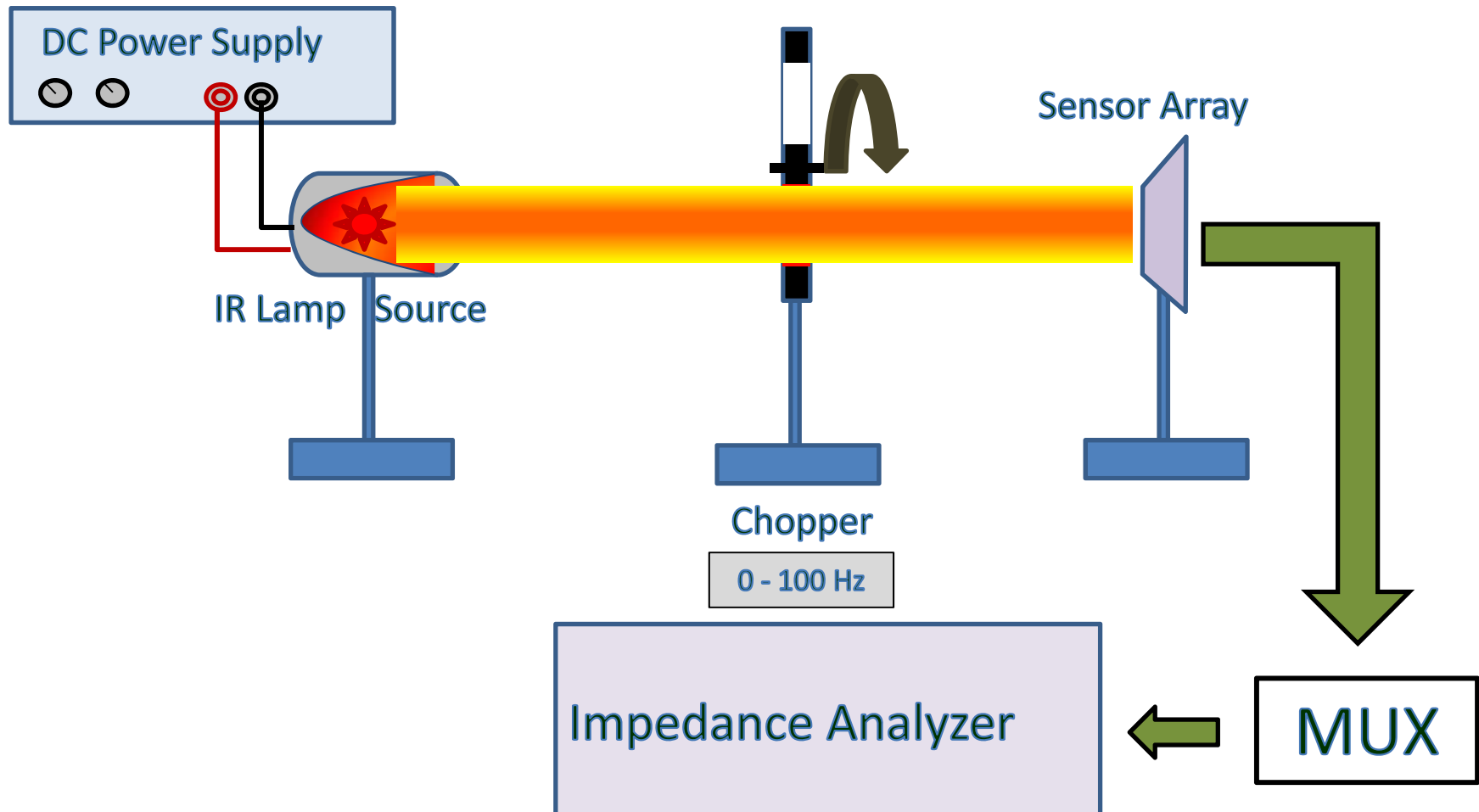
This temperature sensitivity of the measurement can be further enhanced as follows:

$$\frac{d|Z|}{dT} = \frac{d|Z|}{df_0} \cdot \frac{df_0}{dT} = Slope \cdot \frac{df_0}{dT}$$

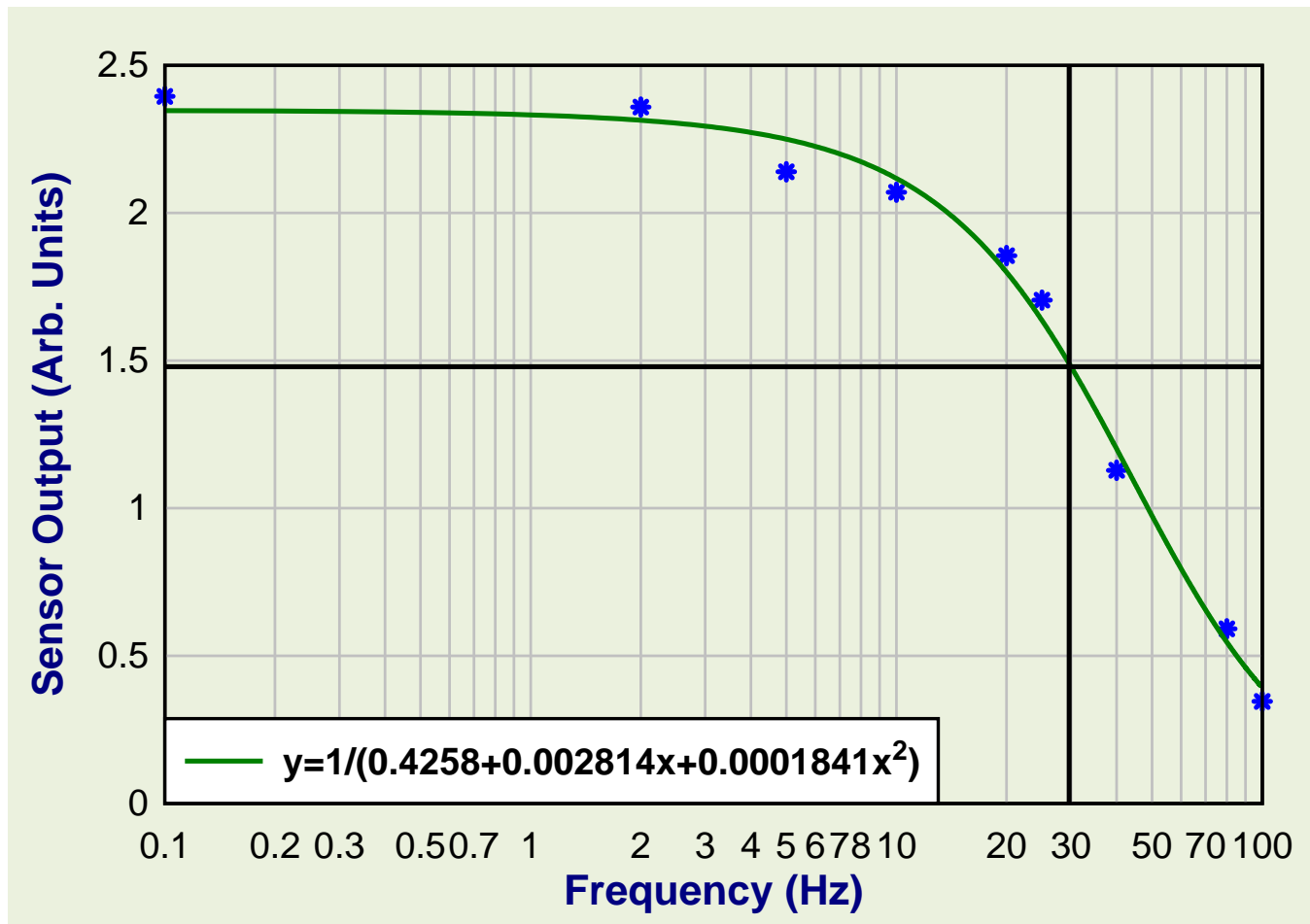
For small frequency changes, the slope shown in the graph results in an additional gain in the measurement.

Monitoring the magnitude of the impedance of the resonator near its resonance at a fixed frequency operating point as the incident infrared radiation is modulated results in a modulation of the impedance – the magnitude of which is calibrated against a reference sensor.

# Thermal and Infrared Characterization Measurement Set-up

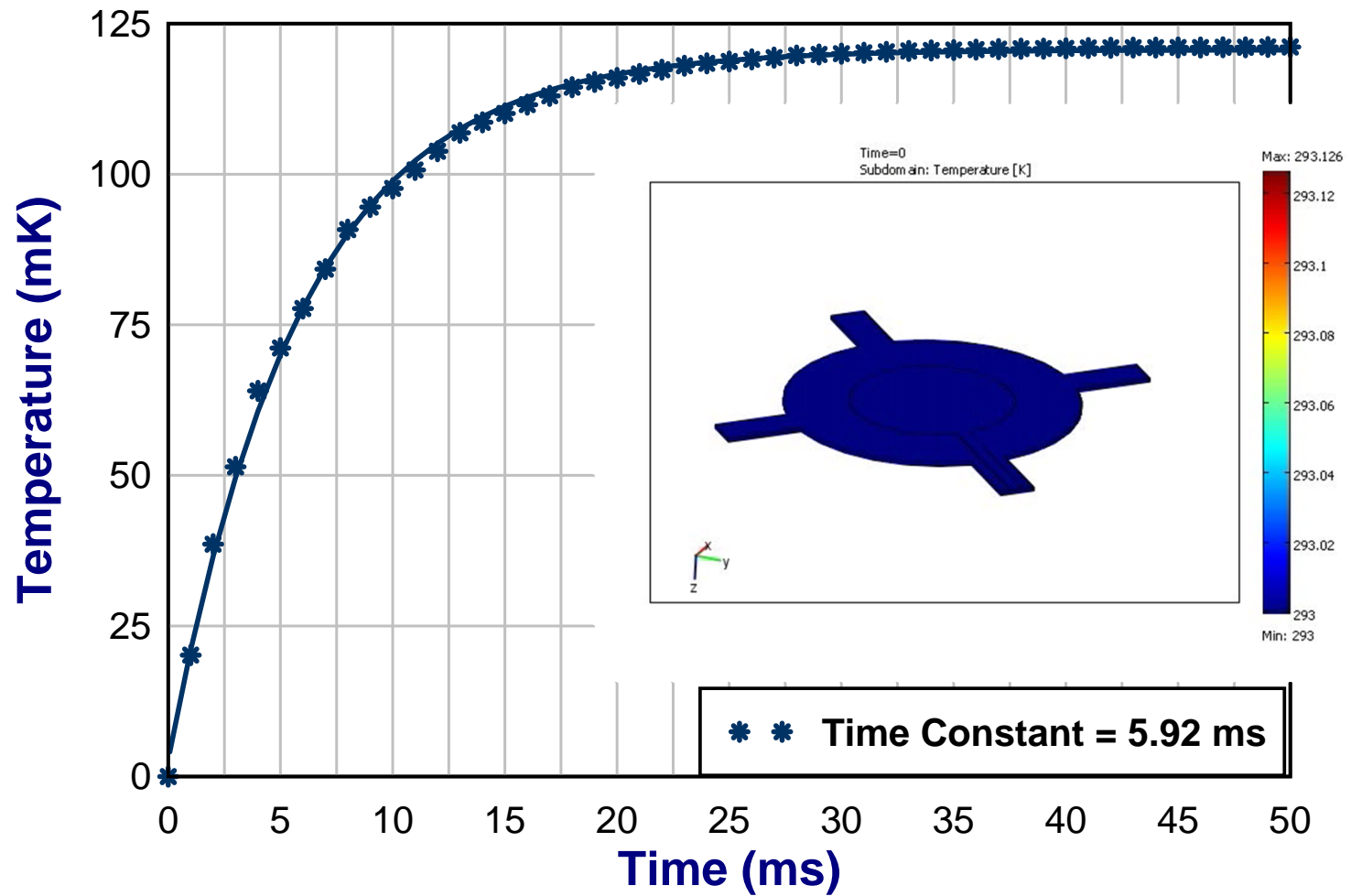


# Thermal Response Time

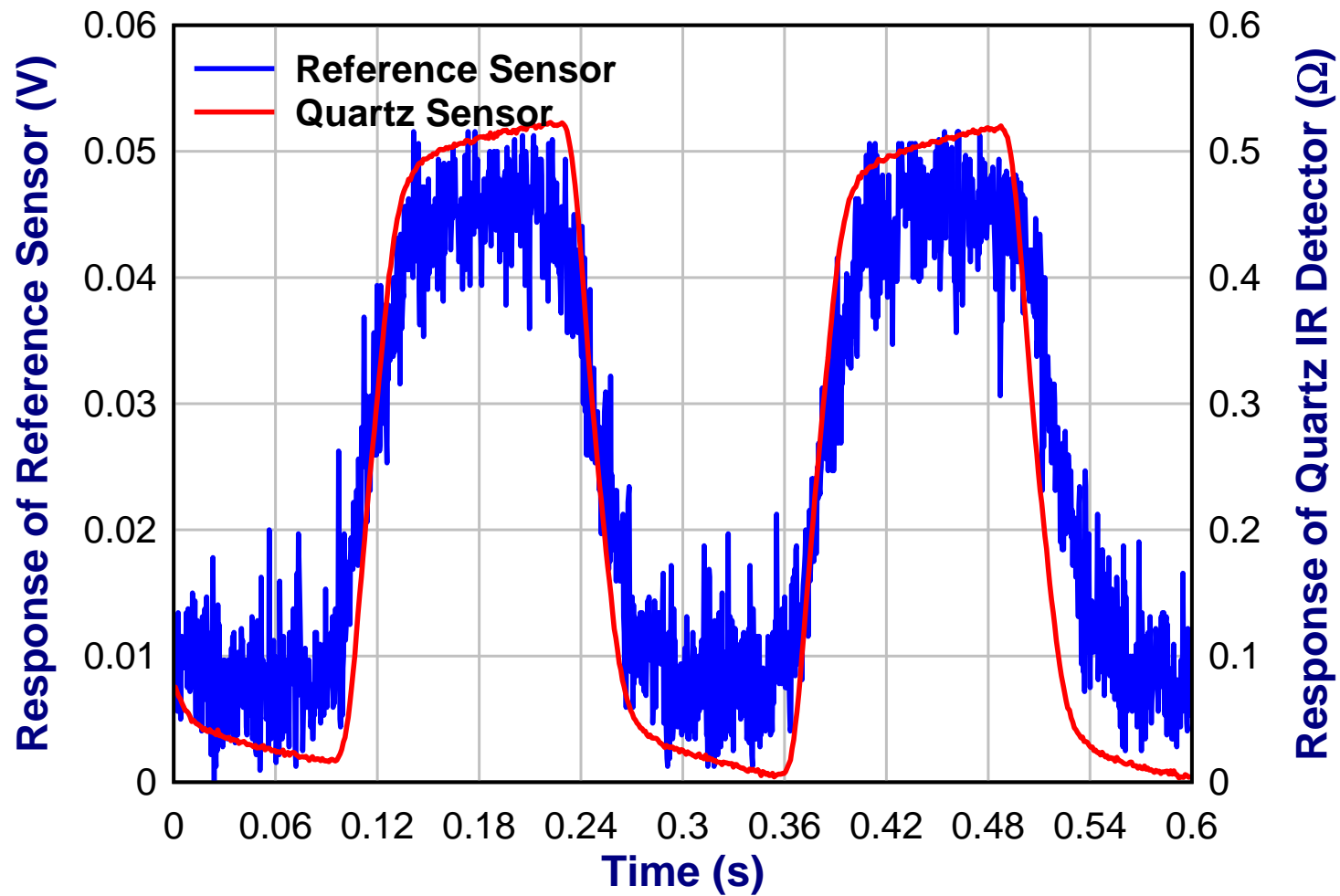


Sensor output at a given frequency divided by the output at DC for constant Lamp intensity is used to estimate the time-constant of the device.

# Thermal Simulation



# IR Response of the Quartz Resonator



# Summary

- At this point the 89 MHz and 241 MHz micromachined quartz resonators have been successfully fabricated and characterized.
- We have obtained good control over quartz etch rate and are able to successfully thin 100  $\mu\text{m}$  thick quartz to 5  $\mu\text{m}$ .
- The quality factor of the 241 MHz resonators fabricated using the new method is around 4200 and is largely affected by the parasitic coupling to the substrate silicon.
- The final through etch process to release the resonators has been improved to result in a high yield process.
- A new method to real-time track the resonance frequency of the Y-cut quartz sensors has been established.
- Initial testing of the IR detectors show very competitive performance in relation to the existing technologies. Quartz resonator based IR detectors are in principle capable of achieving background noise limited performance but need further research and development.
- A full patent based on the work in this project was filed through Penn State Intellectual Property Office.

# Conclusions

- Micromachined Y-cut quartz resonators have been successfully fabricated with a fundamental resonance frequency of 240 MHz (6  $\mu\text{m}$  in thickness)
- Temperature sensitivity of the sensors has been measured to 22.16 kHz/K!
- Fabricated resonators were tested for:
  - Responsivity: 7.32  $\text{k}\Omega/\text{W}$
  - Noise Equivalent Power: 3.9 nW/ $\sqrt{\text{Hz}}$
  - Response Time: < 30 ms
  - Noise Equivalent Temperature Difference: 4 mK
- Thermal models to explain the performance have been set-up.

# Papers, Patents & Presentations

- **The following papers have been published:**
  - Ren K., Kao P., Pisani M.B., and Tadigadapa S. Monitoring biochemical reactions using Y-cut quartz thermal sensors, *Analyst*, **136**, 2904 - 2911, **2011**.
  - Marcelo B. Pisani, Kailiang Ren, Ping Kao, and Srinivas Tadigadapa, Application of Micromachined Y-Cut Quartz Bulk Acoustic Wave Resonator for Infrared Sensing, *Journal of Microelectromechanical Systems*, **20(1)**, 288 - 296, **2011**.
  - Kao, Ping, Tadigadapa, Srinivas, Micromachined quartz resonator based infrared detector array, *Sensors and Actuators A: Physical*, Vol. **149(2)**, 189-192, **2009**
- **The following conference presentations have been made:**
  - Kailiang Ren, Marcelo Pisani, Ping Kao, and Srinivas Tadigadapa, Micromachined Quartz Resonator based High Performance Thermal Sensors, IEEE Sensors Conference, Waikoloa, Hawaii, November **2010**.
  - Marcelo B. Pisani, Kailiang Ren, Ping Kao, and Srinivas Tadigadapa, Room Temperature Infrared Imaging Array Fabricated Using Heterogeneous Integration Methods, Eurosensors XXIV, Linz, Austria, September **2010**.
  - Marcelo Pisani, Ping Kao, and Srinivas Tadigadapa, "Bulk acoustic wave resonators for infrared detection applications" Proceeding of IEEE Transducers, Denver, USA. **2009**.
- **The following invention disclosure has been made:**
  - Srinivas Tadigadapa and Marcelo Pisani, Fabrication Method and Detection Enhancement Strategy for Ultra-Sensitive Temperature Detection Using Resonant Devices, Invention Disclosure # P09582US01, submitted in **2011**.



# **Systematic Studies on Globular Proteins Using Micromachined High Frequency Bulk Acoustic Wave Resonators**

# Outline

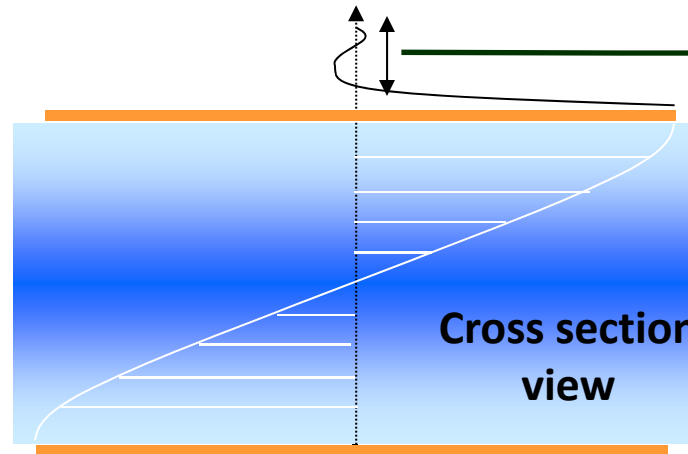
- Why Quartz?
- Why miniaturize QCM?
- Micromachining Quartz
- Evaluation of QCM MicroArrays
- Adsorption Experiments
  - Human Serum Albumin, IgG, and Human Fibrinogen Adsorption on HD-SAMS
- Conclusions

# Quartz Resonators: Motivation

$$\rho_q = 2648 \text{ kgm}^{-3}$$

$$C_{66} = 2.947 \times 10^{10} \text{ Pa}$$

$$f_0 = \frac{\sqrt{C_{66}/\rho_q}}{2t_q}$$



Decay Length

$$\delta = \sqrt{\left(\frac{\eta}{\pi\rho f_0}\right)}$$

Mass

Sauerbrey equation, 1959

$$\Delta f_s = -\left(2f_0^2 / \sqrt{\mu_q \rho_q}\right) \Delta m$$

$$= -S_f \Delta m$$

Thin, rigid, no slip condition

Viscous

Kanazawa and Gordon, 1985

$$\Delta f_s = -\frac{f_s^{3/2}}{N} \sqrt{\frac{\rho\eta}{\pi\rho_q C_{66}}}$$

Semi-Infinite Newtonian fluid

Viscoelastic

Martin, Kasemo,  
Hauptmann, 1996 - 2000

No Simple Equations  
Boundary Condition  
Dependent Solution

Viscoelastic Layer in Liquids

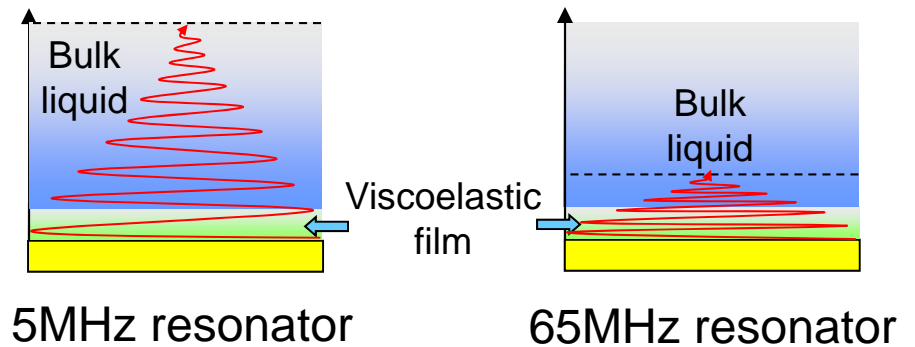
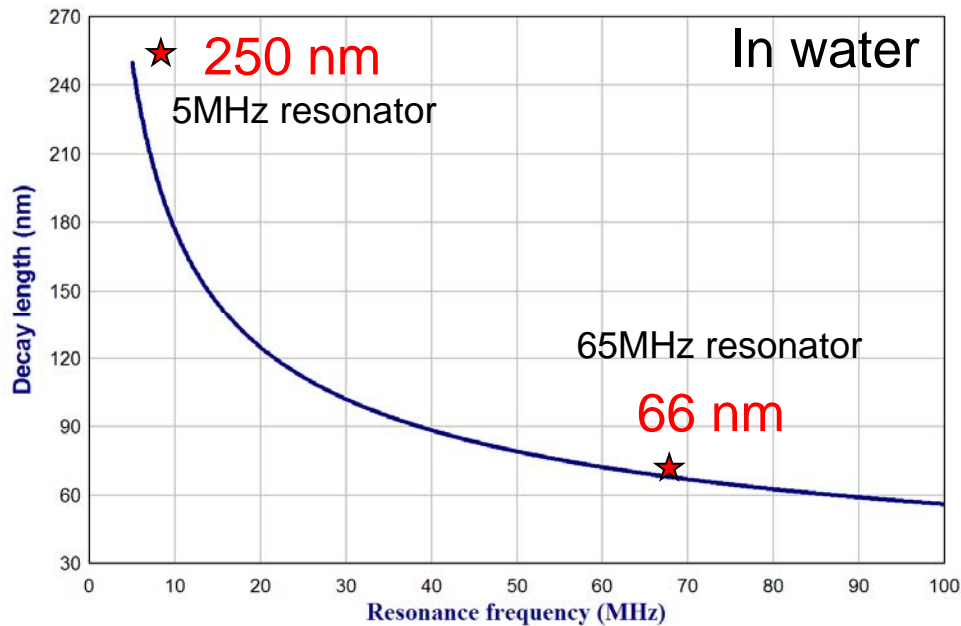
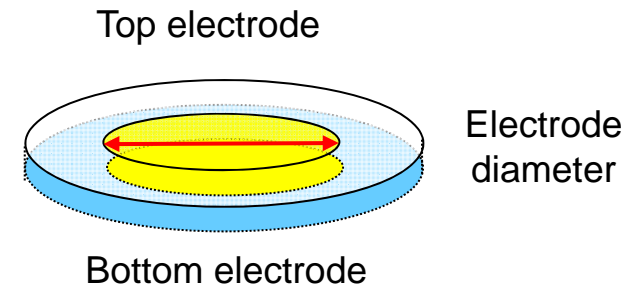
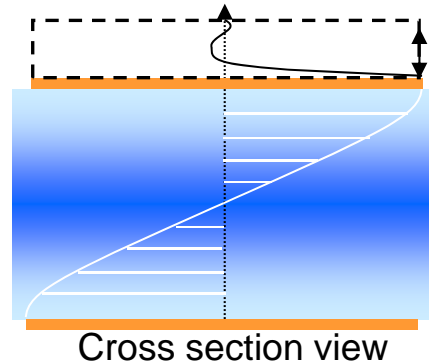
# Higher Sensitivity

## Viscoelastic film

Decay length

$$\delta = \left( \frac{\eta}{\pi \rho f_0} \right)^{1/2}$$

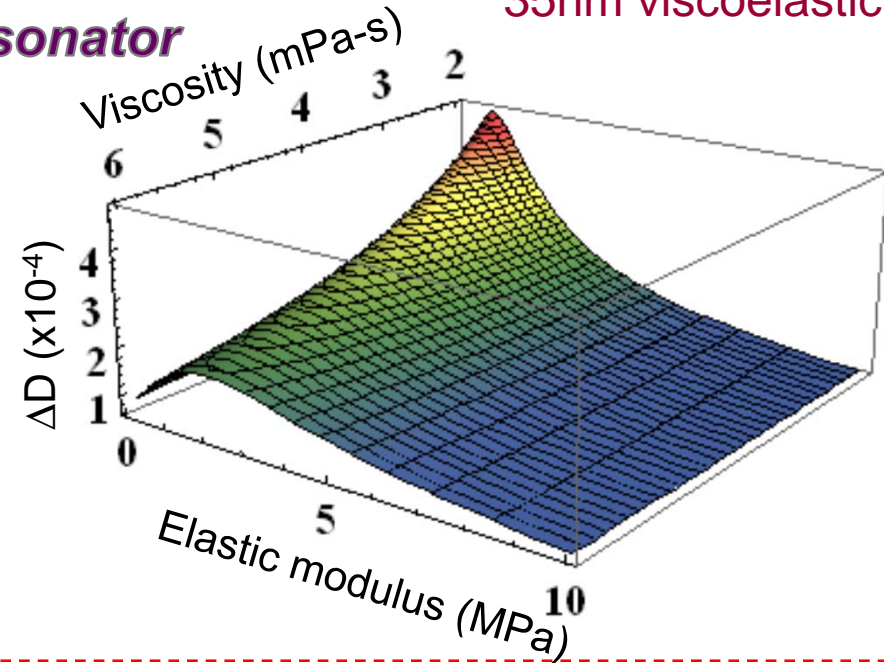
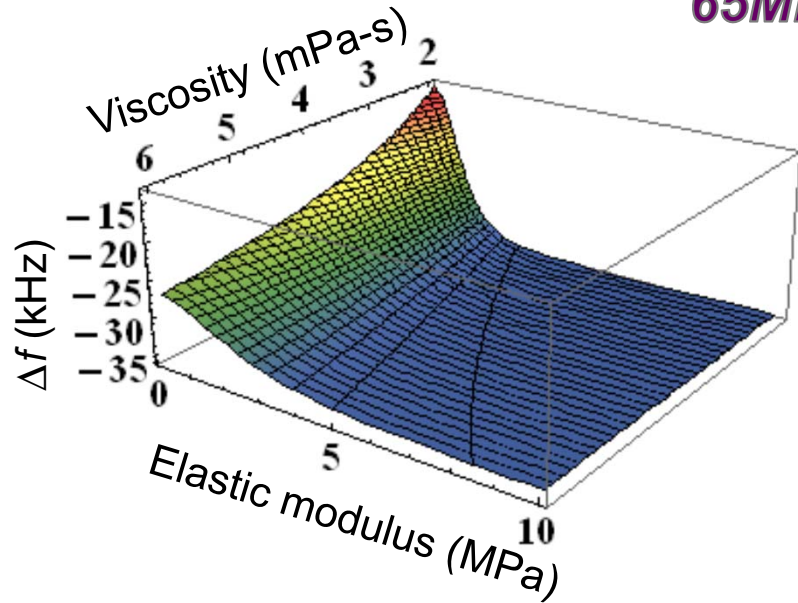
Liquid viscosity  $\eta$   
 Resonance frequency  $f_0$   
 Liquid density  $\rho$



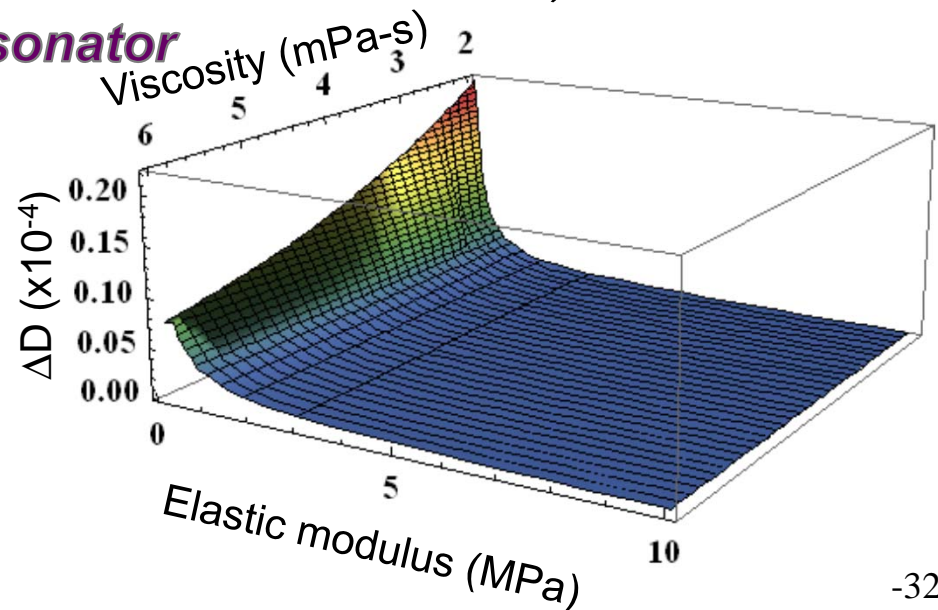
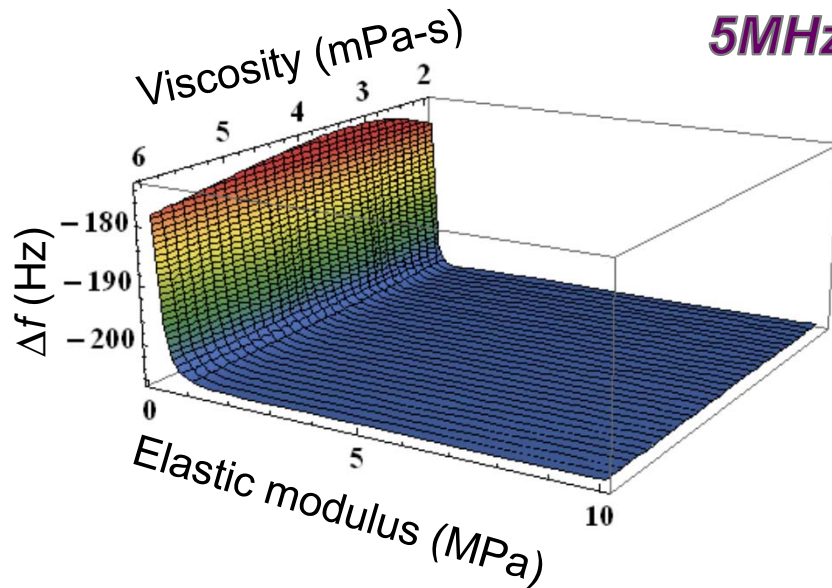
# Resonator Response

35nm viscoelastic film

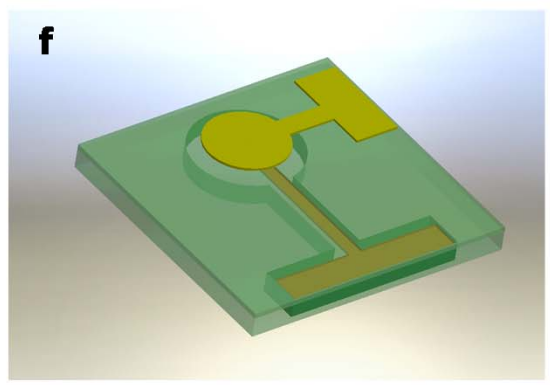
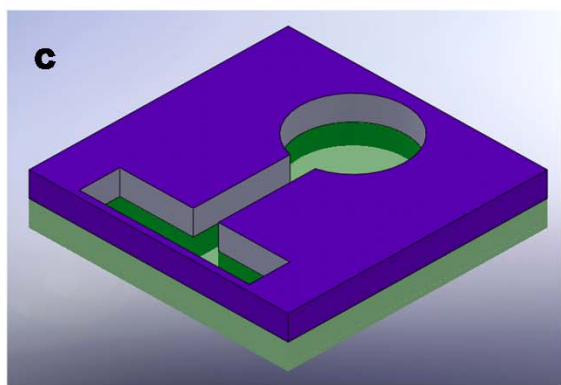
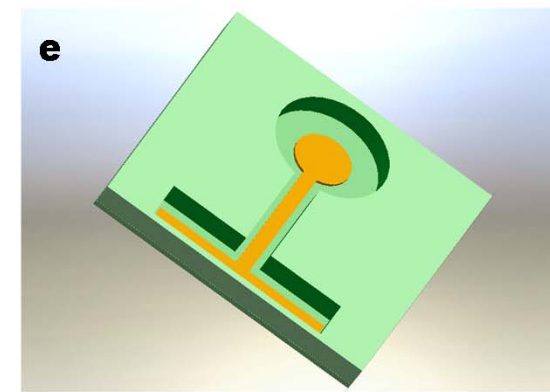
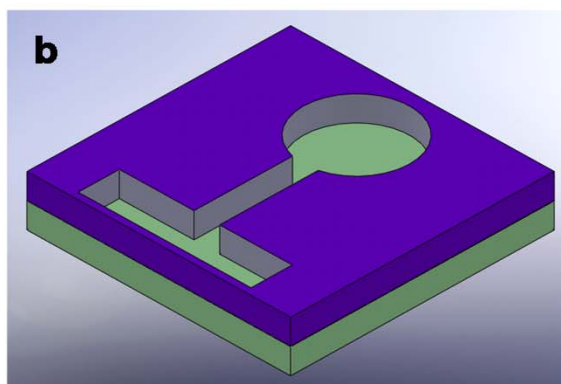
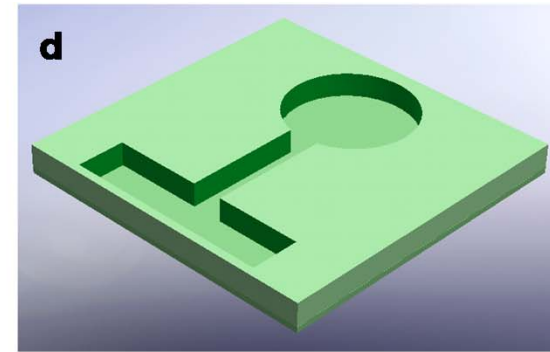
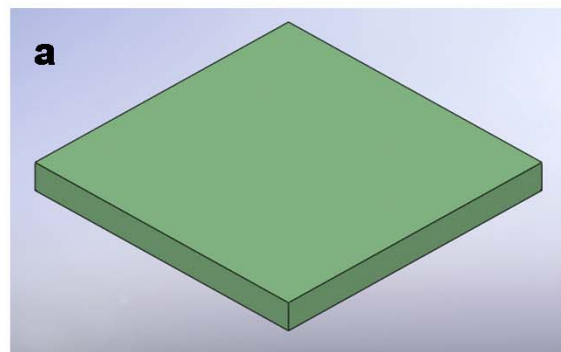
**65MHz resonator**



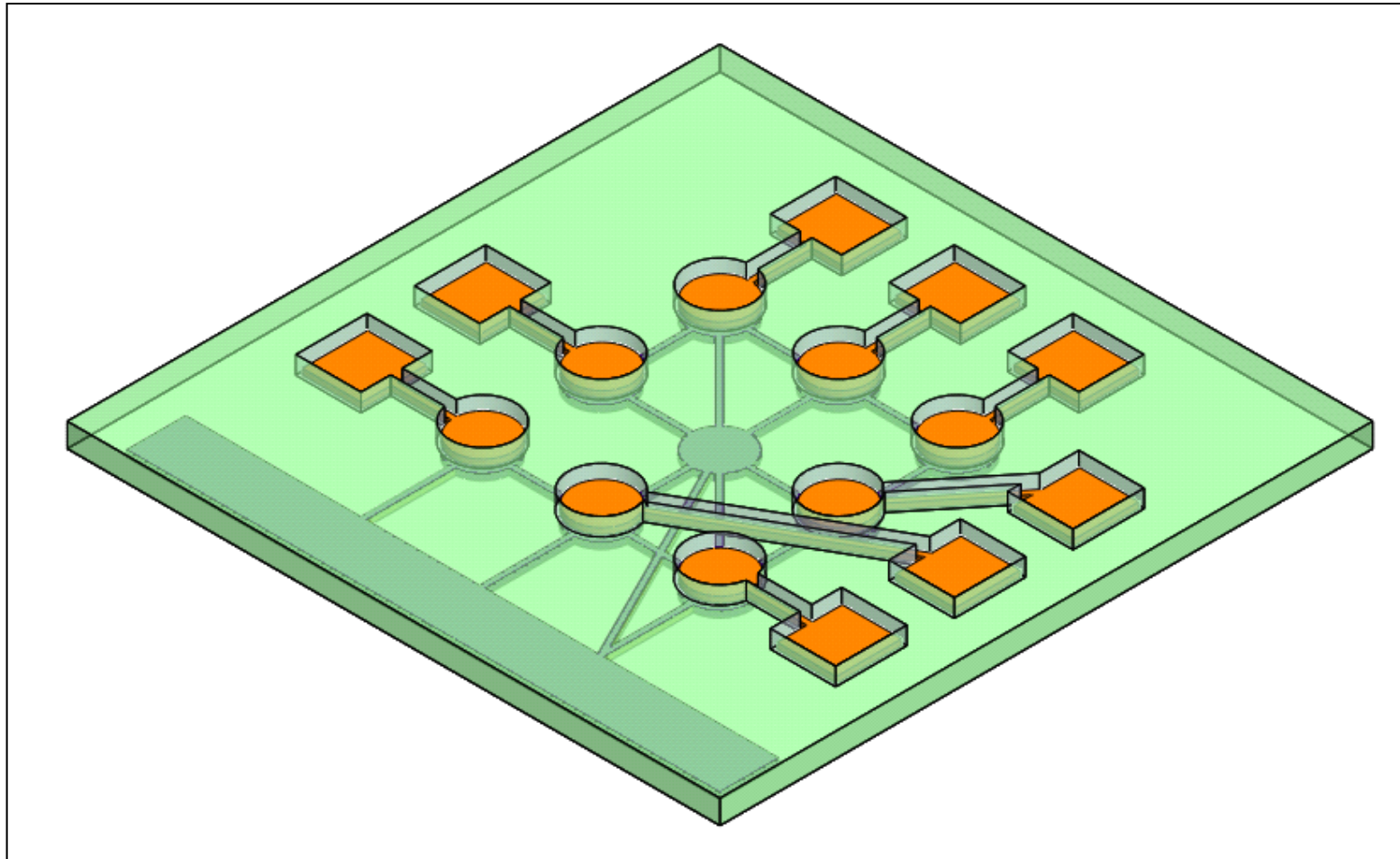
**5MHz resonator**



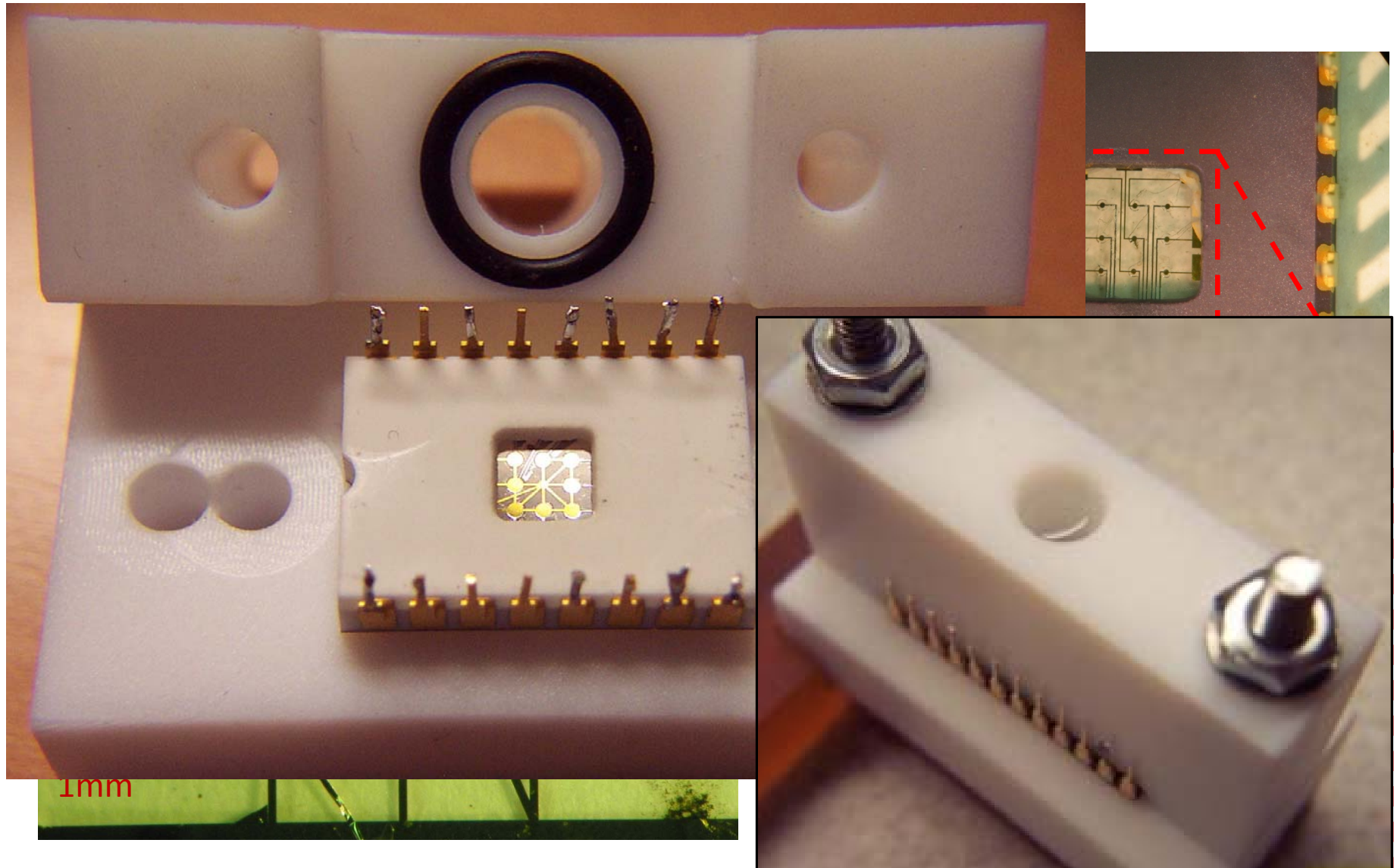
# Fabrication of QCM Microarrays



# Design of the QCM Array

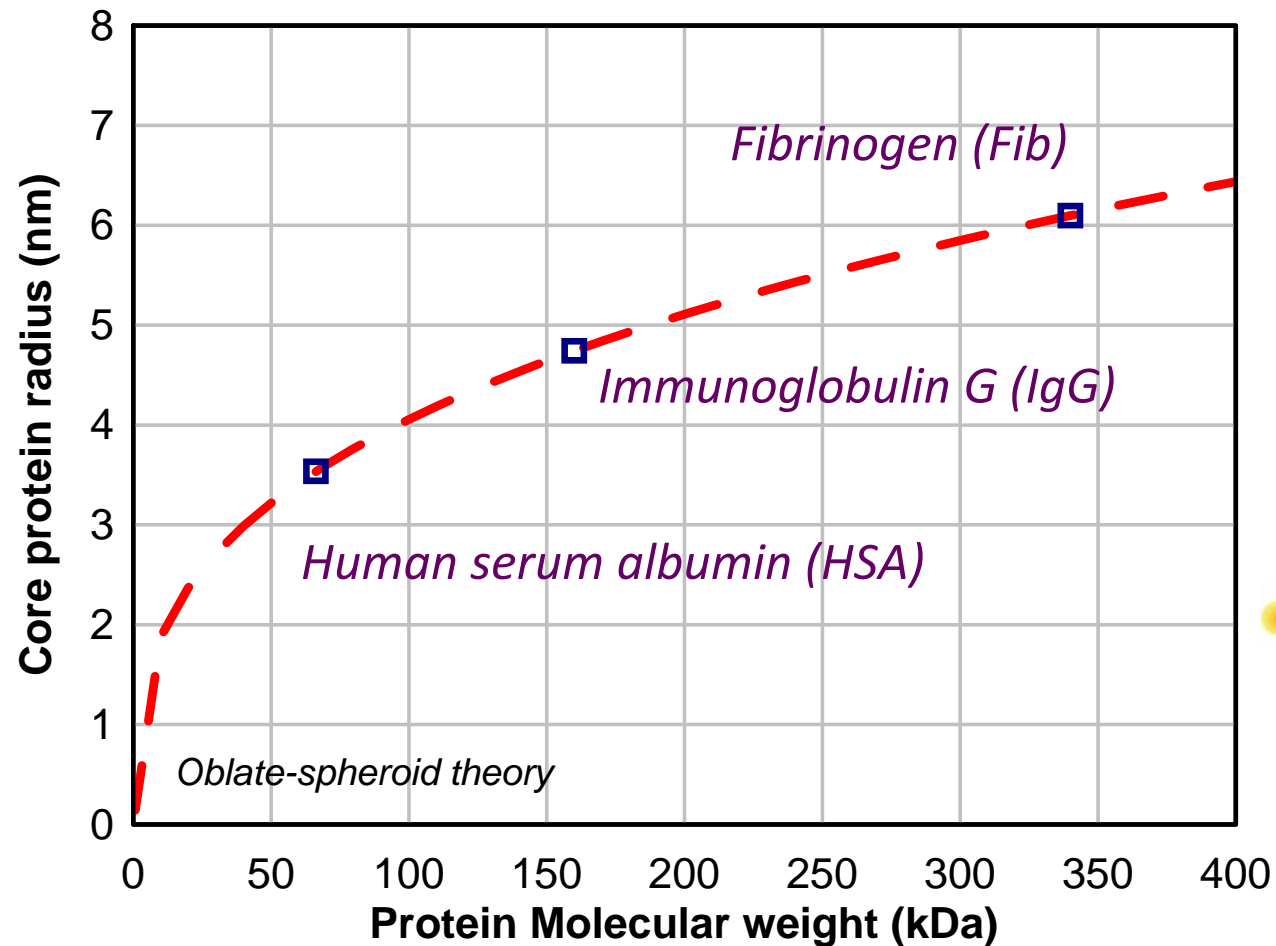


# Fabricated & Packaged Device

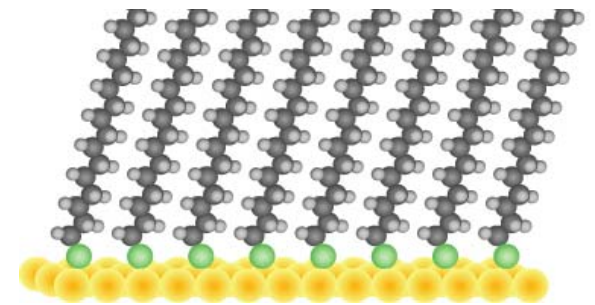


# Interfacial layers studies

## Globular proteins adsorption isotherm

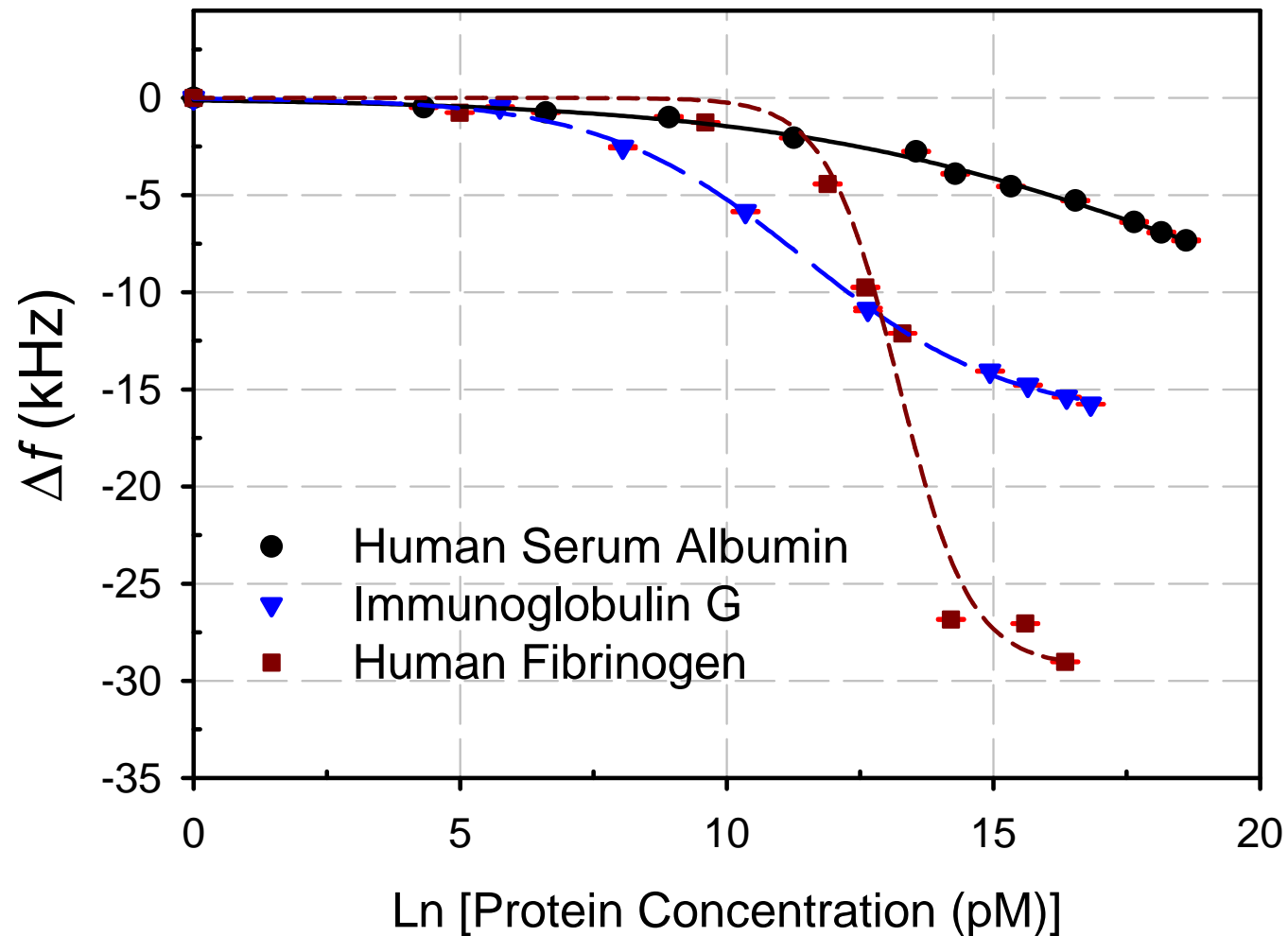


Artificial materials  
-Hydrophobic surface



Hexadecanethiol  
 $\text{CH}_3(\text{CH}_2)_{15}\text{SH}$

# Protein Adsorption Isotherm



# Langmuir Isotherms

The adsorption process between solution phase molecules, A, vacant surface sites, S, and the occupied surface sites, SA, can be represented by the equation



Assuming that there are fixed number of surface sites present on the surface, equilibrium coverage is independent of time and can be given by

$$\frac{\Gamma_{eq}}{\Gamma_{max}} \Rightarrow \frac{\Delta m}{\Delta m_{max}} \Rightarrow \frac{\Delta f}{\Delta f_{max}} = \frac{KC}{1 + KC}$$

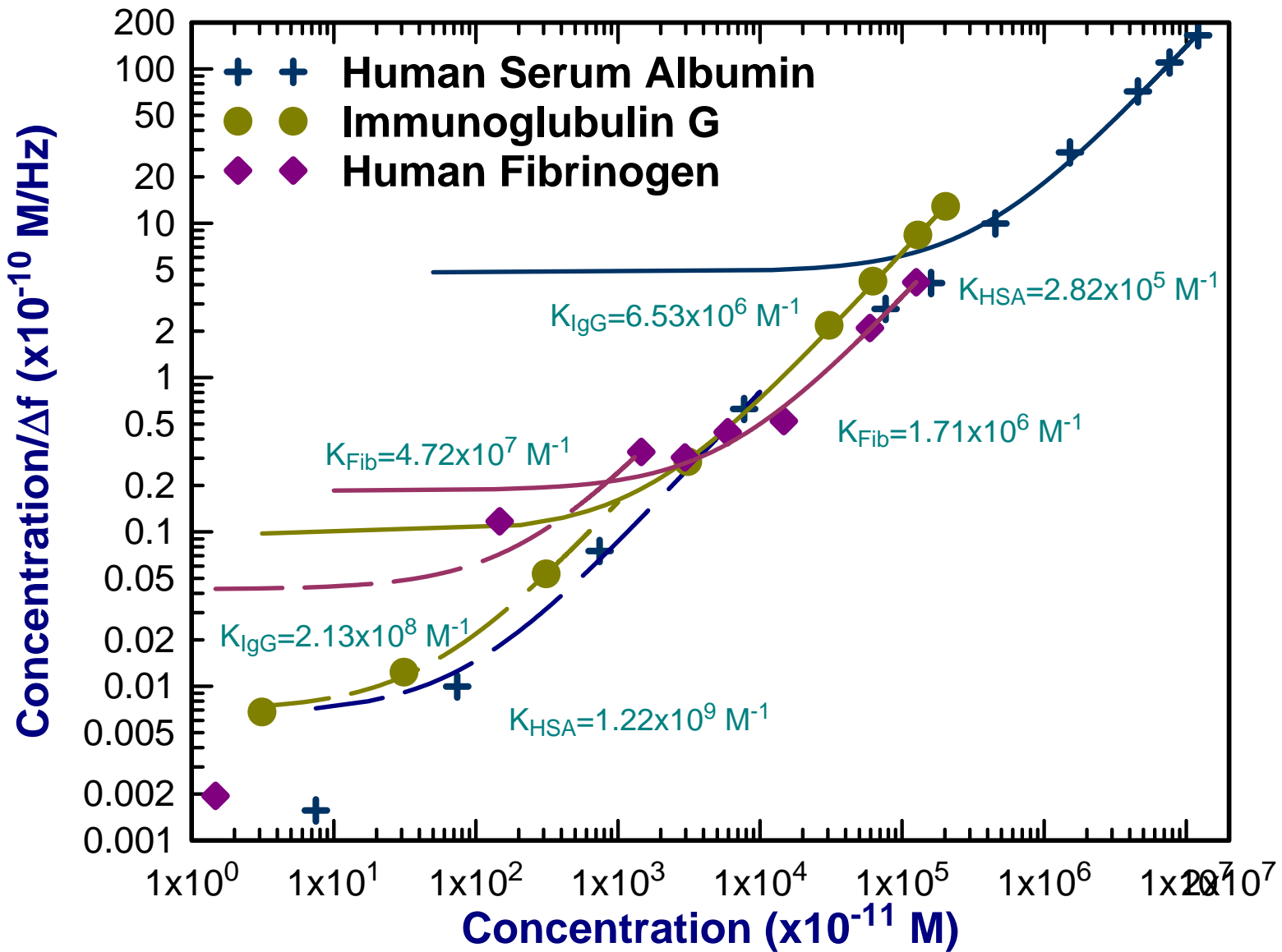
where  $K = k_+/k_-$  is known as the equilibrium adsorption constant., and  $C$  is the concentration of the adsorbate,  $\Gamma_{eq}$  and  $\Gamma_{max}$  are the equilibrium and maximum surface coverages respectively.

Rewriting:

$$\frac{C}{\Delta f} = \frac{C}{\Delta f_{max}} + \frac{1}{K\Delta f_{max}}$$

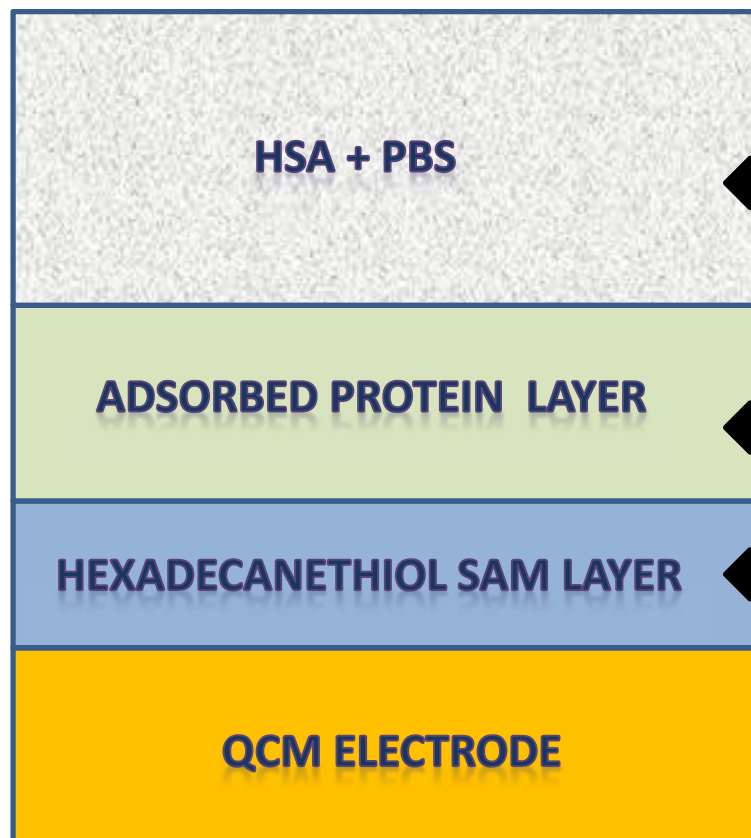
i.e. plotting  $C/\Delta f$  as a function of  $C$ , the equilibrium association constant can be obtained.

# Equilibrium Constant



# Modeling Protein Adsorption on QCM

AIR

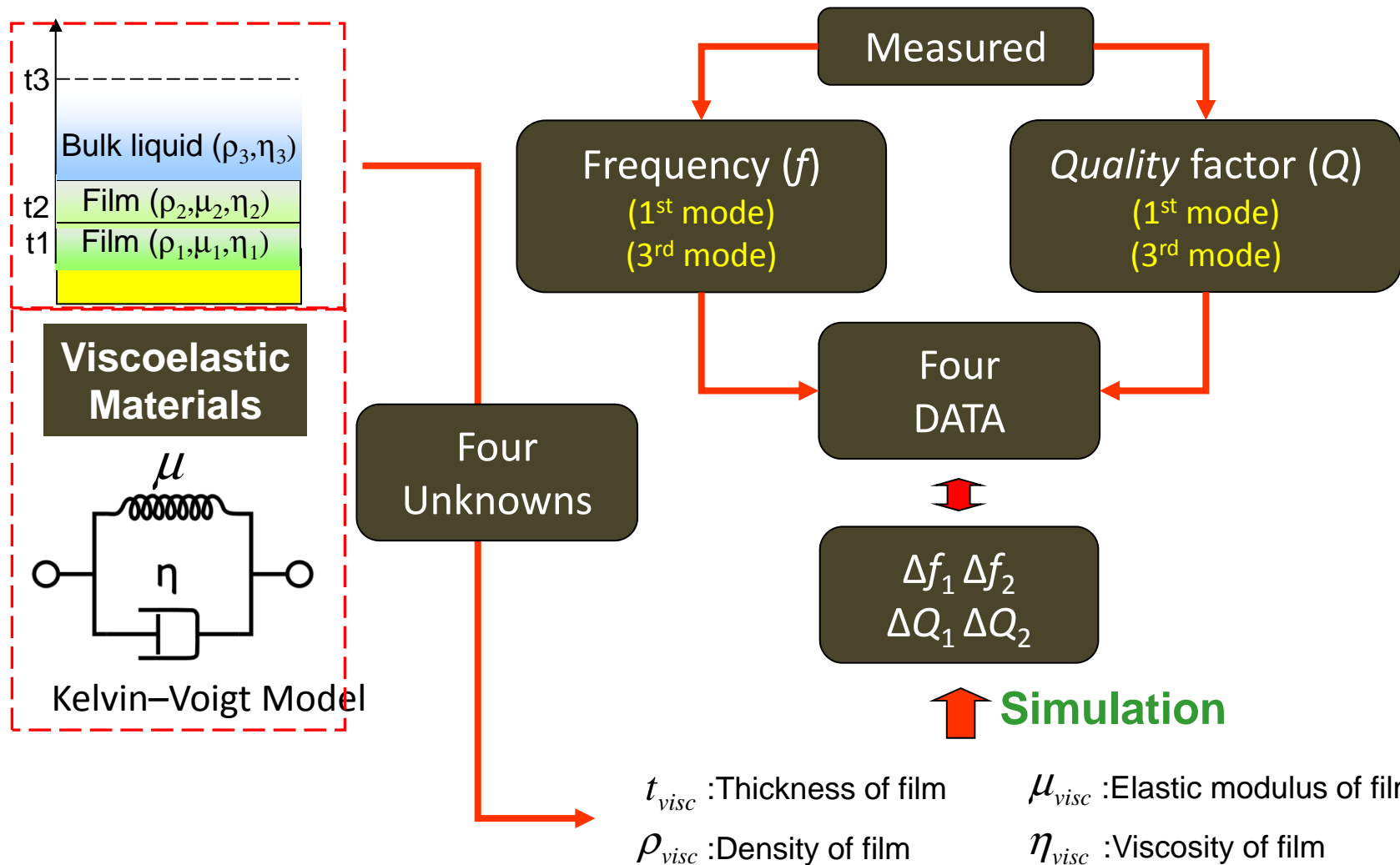


Modeled as a **Newtonian Fluid** characterized by viscosity  $\eta_L$  and density  $\rho_L$ .

Modeled as a **Viscoelastic Layer** characterized by a thickness  $t_F$ , density  $\rho_F$ , elastic modulus  $\mu_F$ , and viscosity  $\eta_F$ .

Modeled as an **Ideal Mass Layer** of uniform density  $\rho$

# Modeling the Viscoelastic Layer



# Modeling Protein Adsorption

- Using Continuum Mechanics Approach, the solution for viscoelastic layers adsorbed in a Newtonian liquid can be given by:

$$\Delta f \approx -\frac{1}{2\pi\rho_q t_q} \sum_{n=1,2,..} \left( t_n \rho_n \omega - 2t_n \left( \frac{\eta_{PBS}}{\delta_{PBS}} \right)^2 \frac{\eta_n \omega^2}{\mu_n^2 + \omega^2 \eta_n^2} \right)$$

$$\Delta Q \approx -2\pi f_0 \rho_q t_q \frac{\left[ \sum_{n=1,2,..} \left( 2t_n \frac{\mu_n \omega}{\mu_n^2 + \omega^2 \eta_n^2} \right) \right]}{\left[ 1 + \sum_{n=1,2,..} \left( 2t_n \left( \frac{\eta_{PBS}}{\delta_{PBS}} \right) \frac{\mu_n \omega}{\mu_n^2 + \omega^2 \eta_n^2} \right) \right]}$$

Subscripts  $n$  refer to the  $n^{th}$  layer, PBS refers to the supernatant liquid layer (Phosphate Buffer Solution) in which protein adsorption experiments.

# Proteins Film Properties

	Thickness	Density	Viscosity	Modulus
HSA	7.5 (4*)	1100	3.5	1.8
IgG	18 (7.2**)	1040	5.5	6.7
Fib	37.5 (10***)	1100	2.7	0.42

( ) is the thickness of single layer

\* Choi, E.J., et al., Langmuir, 2003. 19(13): p. 5464-5474. (Neutron reflectivity)

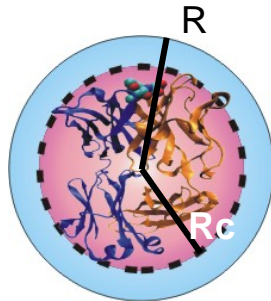
\*\* C. Zhou, et al., Langmuir, vol. 20, pp. 5870-5878, Jul 2004 (Surface plasmon resonance (SPR))

\*\*\* R. J. Green, J. Davies, M. C. Davies, C. J. Roberts, and S. J. B. Tendler, Biomaterials, vol. 18, pp. 405-413, 1997. (SPR)

# Theoretical Prediction

Proteins	Harmonic modes	Frequency Shift (kHz)		Q factor Change	
		Measured	Theory	Measured	Theory
HSA	1 <sup>st</sup> mode	7.33	7.33	27	26
	3 <sup>rd</sup> mode	14.04	17.02	100	91
IgG	1 <sup>st</sup> mode	15.75	16.48	54	54
	3 <sup>rd</sup> mode	43.39	47.20	254	258
Fib	1 <sup>st</sup> mode	28.85	26.93	168	193
	3 <sup>rd</sup> mode	56.85	57.43	490	469

# Proteins Packing



*Oblate-spheroid theory*

R is hydrated protein radius

Rc is the core protein radius

Proteins	MW (KDa)	Diameter of hydrated protein (nm) <i>Theory</i>	Thickness (nm) Measured	Assumed layer	Thickness per layer (nm) <i>Measured</i>
<b>HSA</b>	<b>66.3</b>	<b>7.1</b>	<b>7.5</b>	<b>1</b>	<b>7.5</b>
<b>IgG</b>	<b>160</b>	<b>9.5</b>	<b>18</b>	<b>2</b>	<b>9</b>
<b>Fib</b>	<b>340</b>	<b>12.2</b>	<b>37.5</b>	<b>3</b>	<b>12.5</b>

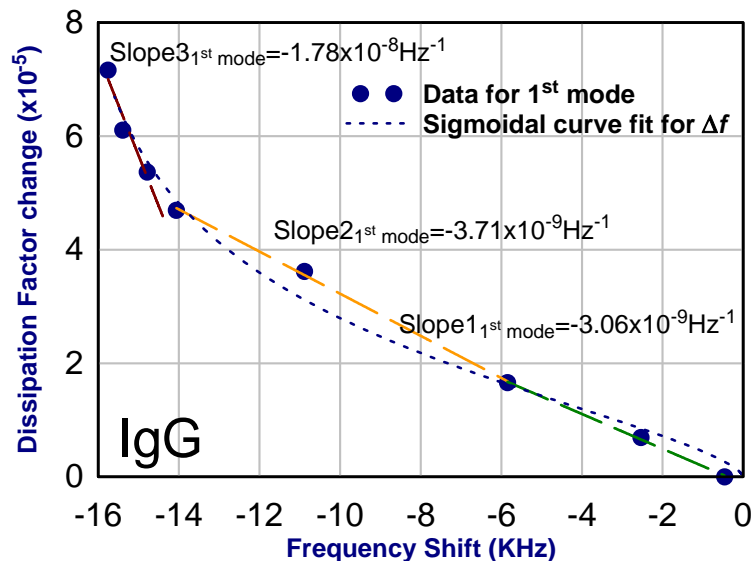
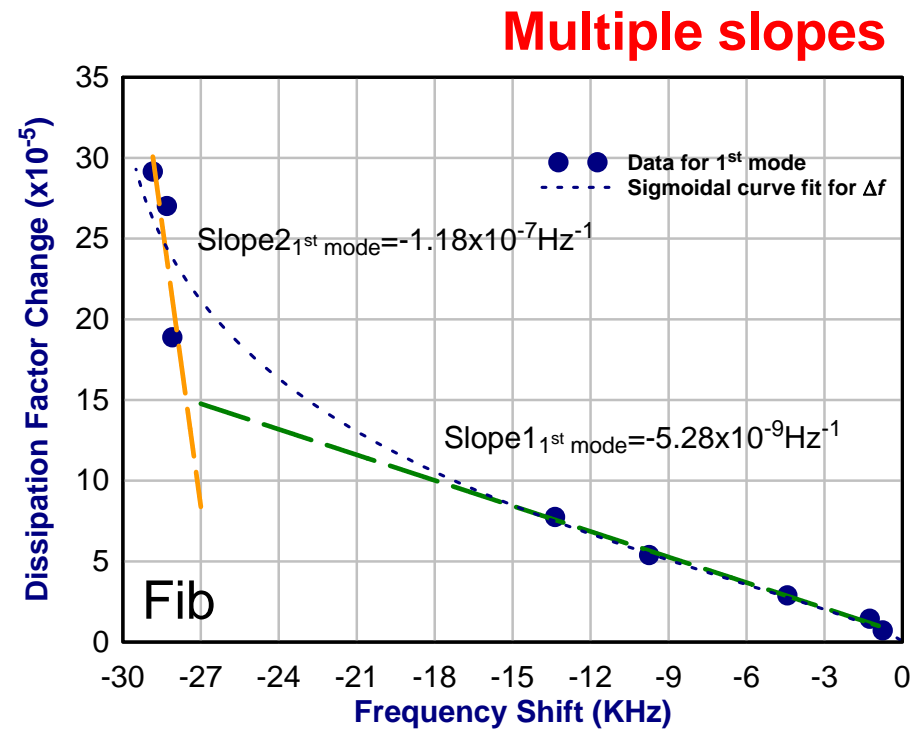
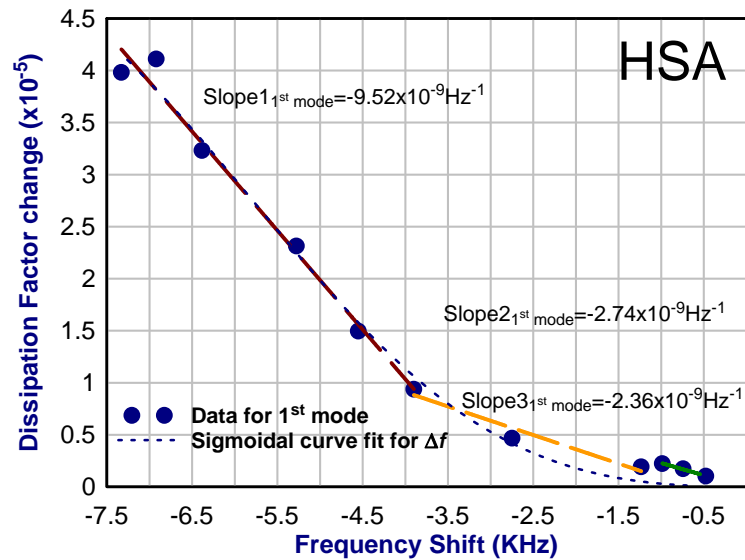
C. Tanford, *Protein Science*, vol. 6, pp. 1358-1366, 1997.

H. Durchschlag and P. Zipper, *Biophysical Chemistry*, vol. 93, pp. 141-157, 2001.

A. Krishnan, C. A. Siedlecki, and E. A. Vogler, *Langmuir*, vol. 19, pp. 10342-10352, 2003.

# Dissipation factor

Dissipation factor =  $1/Q$



( $10^{-9}$ )	1 <sup>st</sup> slope	2 <sup>nd</sup> slope	3 <sup>rd</sup> slope	High/low
HSA	2.36	2.74	9.52	<b>4</b>
IgG	3.06	3.71	17.79	<b>6</b>
Fib	5.27	117.5	N/A	<b>22</b>

Reorientation, Spreading, Conformation change

# Sampling Volume

Single cell  
Single resonator

Multi cells  
Multi resonators

Single cell  
Multi resonators **(Array)**

Maxtek

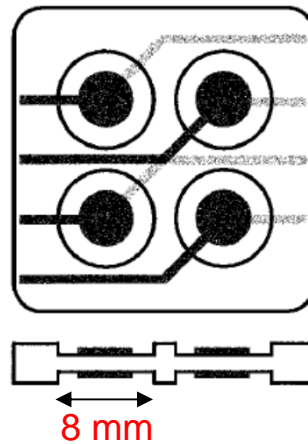
Q-Sense



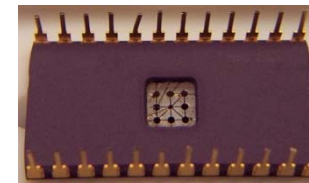
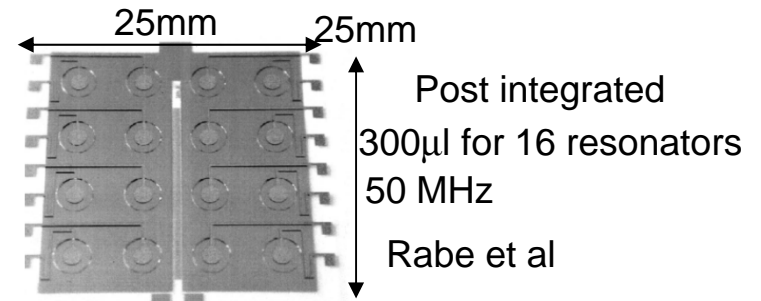
5MHz/ Diameter ~**7mm**

Minimum sample volume  
~ 100µl for one resonator

**\$4800 USD (same experiment)**



10 MHz  
Diameter ~**4.5mm**



**Our**  
9 mm x 9 mm

65~92 MHz/ Diameter ~**500µm**

Minimum sample volume  
~ 20 µl for 8 resonators

**\$ 300 USD** protein adsorption

[http://q-sense.com/dbfiles/Q-Sense\\_E4%20Auto.pdf](http://q-sense.com/dbfiles/Q-Sense_E4%20Auto.pdf)

<http://www.inficonthinfilmdposition.com/en/maxtekrqcm.html>

T. Tatsuma, Y. Watanabe, N. Oyama, K. Kitakizaki, and M. Haba, *Analytical Chemistry*, vol. 71, pp. 3632-3636, 1999.

J. Rabe, S. Buttgenbach, J. Schroder, and P. Hauptmann, *IEEE Sensors Journal*, vol. 3, pp. 361-368, 2003.

# Conclusions

- Analysis of physical and viscoelastic properties

- Thickness
- Density
- Viscosity
- Elastic Modulus

- Sensitivity

- Globular proteins

- Sampling volume

Thickness nm	Density kg/m <sup>3</sup>	Viscosity mPa-s	Modulus (MPa)
<0.1nm	1.98	0.05	0.05

- 8 resonators array type design (16 times reduction)

**Quartz Crystal Resonator is not just a Microbalance!**

# Papers & Presentations

- **The following papers have been published:**
  - Kao P., Allara D., Tadigadapa S. Study of Adsorption of Globular Proteins on Hydrophobic Surfaces, IEEE Sensors Journal; **11(11)**, 2723 - 2731, 2011.
  - Ping Kao, Purnendu Parhi, Anandi Krishnan, Hyeran Noh, Waseem Haider, Srinivas Tadigadapa, David L. Allara, Erwin A. Vogler, Volumetric interpretation of protein adsorption: Interfacial packing of protein adsorbed to hydrophobic surfaces from surface-saturating solution concentrations, Biomaterials **32**, 969-978, 2011.
  - Ping Kao, David Allara, and Srinivas Tadigadapa, Fabrication and Performance Characteristics of High-Frequency Micromachined Bulk Acoustic Wave Quartz Resonator Arrays, Measurement Science and Technology, **20(12)**, 9pp, 2009.
  - Srinivas Tadigadapa and Kiron Mateti, Piezoelectric MEMS sensors: state-of-the-art and perspectives, Measurement Science and Technology, **20(10)**, 1-30, 2009.
- **The following conference presentations have been made:**
  - Hwall Min, Nichole Sullivan, David Allara, Srinivas Tadigadapa, Nanoporous Gold: A High Sensitivity and Specificity Biosensing Substrate, Eurosensors XXV, Athens, Greece, September 2011.
  - Hwall Min, David Allara, Srinivas Tadigadapa, Investigation of the Viscoelastic Properties of Liquids Trapped in Nanoporous Cavities using Micromachined Acoustic Transducers, Eurosensors XXV, Athens, Greece, September 2011.
  - Son Vu Hoang Lai, Ping Kao, Srinivas Tadigadapa, Thermal biosensors from micromachined bulk acoustic wave resonators, Eurosensors XXV, Athens, Greece, September 2011.
  - Ping Kao, David Allara, Srinivas Tadigadapa, Label free Piezoelectric DNA Sensor Arrays Using novel selective immobilization techniques, Proc. IEEE MEMS Conference, Cancun, Mexico, January 2011.
  - Ping Kao, Matthew P. Chang, David Allara, and Srinivas Tadigadapa, Investigation of Spontaneously Adsorbed Globular Protein Films using High-Frequency Bulk Acoustic Wave Resonators, IEEE Sensors Conference, Waikoloa, Hawaii, November 2010.
  - Ping Kao, Matthew P. Chang, David Allara, and Srinivas Tadigadapa, Systematic studies on globular proteins using micromachined high frequency bulk acoustic wave resonators, Eurosensors XXIV, Linz, Austria, September 2010.



# High Speed Anisotropic Reactive Ion Etching (RIE) of Quartz and Pyrex Glass

# Introduction

- Glass Etching: State of the Art
  - Background
  - Results from ICP-RIE Etching: Effect of various Process Parameters on Etch Characteristics
- Glass Etching: Issues
  - Masking Materials
  - Sidewall and Etch Pit Roughness
  - Wafer Mounting and Cooling
- Glass Etching: Future Directions

# Requirements

- High Etch Rate
- High Anisotropy
- Planarity of Etched Surface
- Surface roughness control
- Optimum Properties of sidewalls
- High Selectivity

# State of the Art

- Glass Etching/Shaping can be accomplished using
  - Wet Etching
  - Plasma Etching
  - Femtosecond Laser Micromachining
  - Other Creative Processing Methods
- TYPICAL COMPOSITION:
- PYREX
  - $\text{SiO}_2$  (80.6%),  $\text{B}_2\text{O}_3$  (13.0%),  $\text{Na}_2\text{O}$  (4%),  $\text{Al}_2\text{O}_3$  (2.3%), and  $\text{K}_2\text{O}$  (0.04%)
- BOROFLOAT
  - $\text{SiO}_2$  (81%) ,  $\text{B}_2\text{O}_3$  (13%) ,  $\text{Na}_2\text{O}/\text{K}_2\text{O}$  (4%) , and  $\text{Al}_2\text{O}_3$  (2%).

# Wet Etching of Glass

Etchant	Etch Rate	Issues
HF (49%) <sup>1</sup>	1.32 – 3.2 $\mu\text{m}/\text{min}$	For Thermal Oxide, Masking
HF/HCl (10:1) <sup>2</sup>	7.86 $\mu\text{m}/\text{min}$	For Pyrex: Improved Roughness: 6nm

Typically in Pyrex Etching: Oxides, such as CaO, MgO or Al<sub>2</sub>O<sub>3</sub>, give insoluble products in HF solution: CaF<sub>2</sub> MgF<sub>2</sub> AlF<sub>3</sub>

## MASKING FOR WET ETCHING:

Photoresist: AZ 9260 – 3-4 minutes

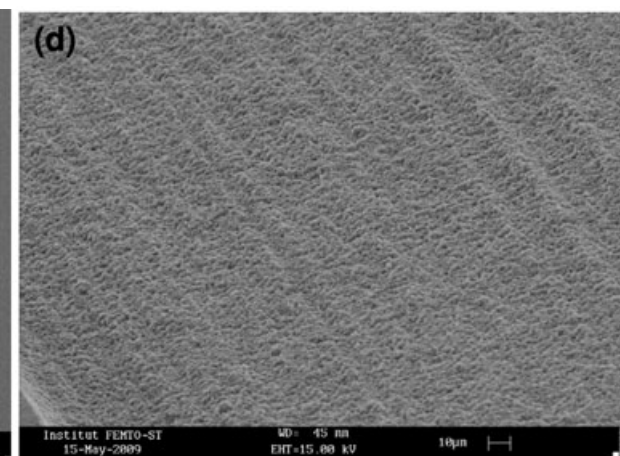
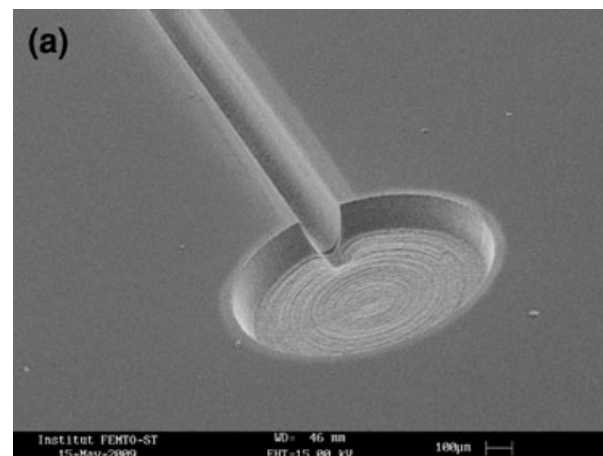
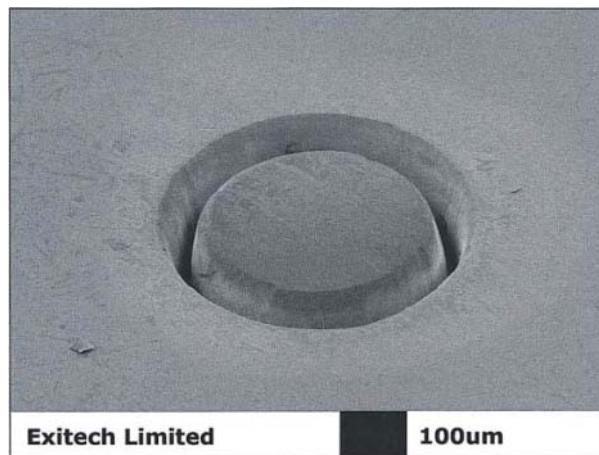
Au/Cr: 10 – 14 minutes

PECVD Polysilicon (Annealed @ 400 C: over 30 minutes

1. K.R. Williams, R.S. Muller: Etch rates for micromachining processing, J. Micromech. Syst. 5(4), 256–269 (1996)
2. Ciprian Iliescu, Ji Jing, Francis E.H. Taya, Jianmin Miao, Tietun Sund: Characterization of masking layers for deep wet etching of glass in an improved HF/HCl solution, Surface & Coatings Technology 198 (2005) 314– 318

# Laser Micromachining of Glass

Laser	Etch Rate	Issues
Excimer Laser	Channels of 100 $\mu\text{m}$ width, 140 $\mu\text{m}$ height and 4 mm length were written in 3 min using a spot size of 10 $\mu\text{m}$ with an average power of 160 mW at 5 kHz, that is with a pulse energy of 0.032 mJ and a power of 0.27 GW (pulse duration of 120 fs).	Tapering Profile, Cracking, Serial Process



Typical Femtosecond Laser Characteristics:  $\lambda_0 \sim 800 \text{ nm}$ ,  $\Delta\tau \sim 110 \text{ fs}$ ,  $E \sim 1 \text{ mJ/pulse}$ , Rep Rate  $\sim 3\text{--}5 \text{ kHz}$

S. Queste, R. Salut, S. Clatot, J.-Y. Rauch, Chantal G, Khan Malek, Manufacture of microfluidic glass chips by deep plasma etching, femtosecond laser ablation, and anodic bonding, *Microsyst Technol* (2010) 16:1485–1493

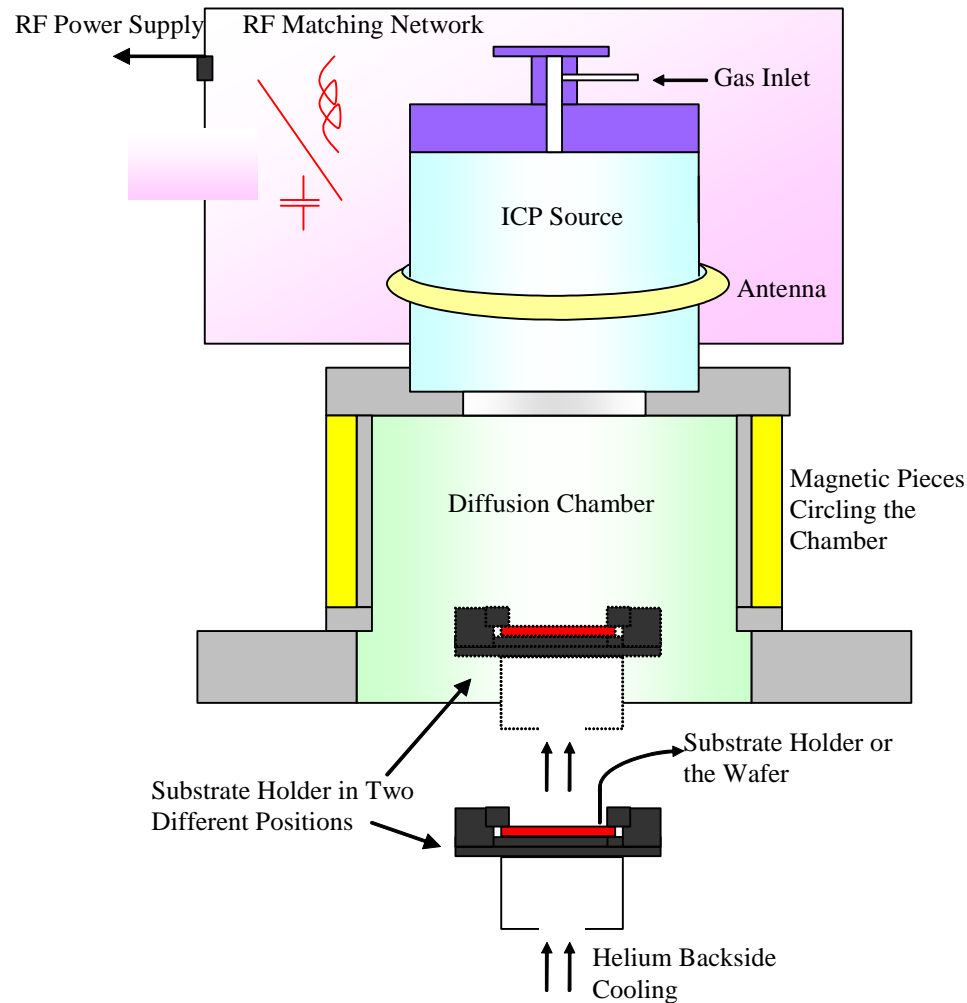
Rafael R. Gattass and Eric Mazur, Femtosecond laser micromachining in transparent materials, *nature photonics* (2008) 2: 219-225

# Quick Summary of Deep RIE of Glass

Reference	Mask type	Etching chemistry	Etching depth ( $\mu\text{m}$ )	Etching rate ( $\mu\text{m}/\text{min}$ )	Profile	Roughness Ra (nm)	Aspect ratio
Our work	Ni 8 $\mu\text{m}$	$\text{C}_4\text{F}_8/\text{O}_2$	120	1	$83^\circ\text{--}88^\circ$	2–10	40
Akashi and Yoshimura (2006)	Si wafer 200 $\mu\text{m}$	$\text{C}_4\text{F}_8$	430		$80^\circ$		
Goyal et al. (2006)	Ni 5 $\mu\text{m}$	$\text{SF}_6/\text{Ar}$	20	0.536		1.97	
Ichiki et al. (2003)	Cr	$\text{SF}_6$	<20	1.2	$88^\circ$	Very high	
Kolari et al. (2008)	Si wafer 400 $\mu\text{m}$	$\text{C}_4\text{F}_8/\text{He}/\text{O}_2$	250	0.5	$80^\circ\text{--}86^\circ$		<3
Kolari et al. (2008)	Si wafer 400 $\mu\text{m}$	$\text{C}_4\text{F}_8/\text{He}/\text{O}_2$	300	0.35			3
Kolari et al. (2008)	Ni 5 $\mu\text{m}$	$\text{C}_4\text{F}_8/\text{O}_2$	80	0.7	$80^\circ\text{--}86^\circ$		3.5
Li et al. (2001)	Ni	$\text{SF}_6$	200	0.6	$88^\circ$	4	10
Park et al. (2005)	Ni	$\text{SF}_6$	40	0.75	< $88^\circ$		
Park et al. (2005)	Ni	$\text{SF}_6/\text{Ar}$	27	0.54	$88^\circ$		
Queste et al. (2008)	Ni 6 $\mu\text{m}$	$\text{C}_4\text{F}_8/\text{O}_2$	120	0.8	$83^\circ\text{--}88^\circ$	2	6

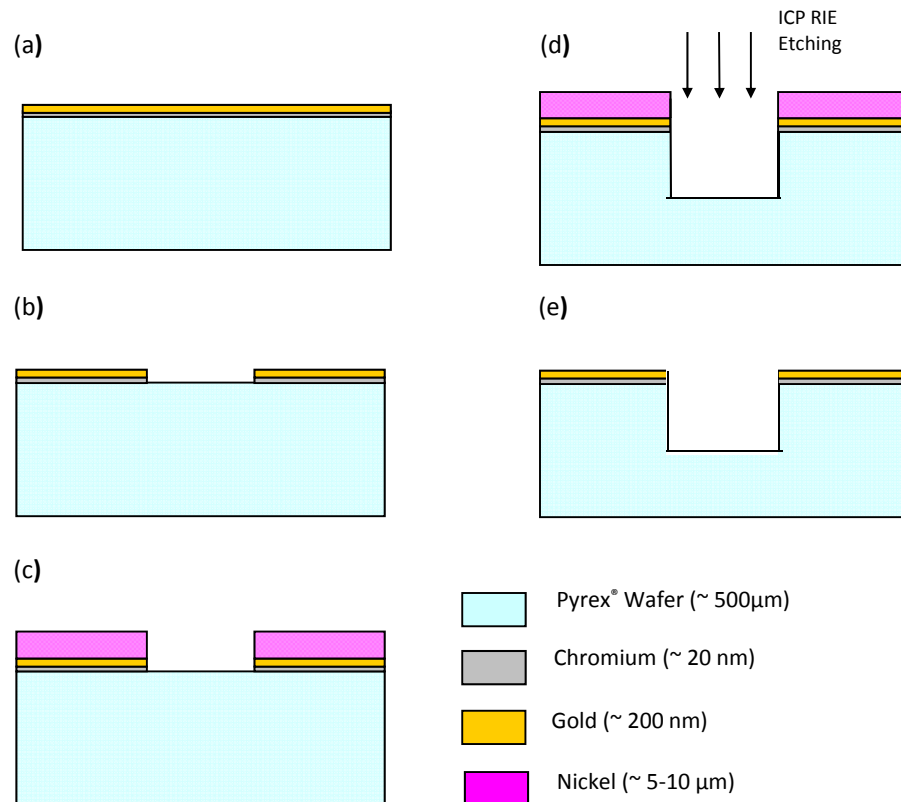
S. Queste, R. Salut, S. Clatot, J.-Y. Rauch, Chantal G, Khan Malek, Manufacture of microfluidic glass chips by deep plasma etching, femtosecond laser ablation, and anodic bonding, *Microsyst Technol* (2010) 16:1485–1493

# ICP-RIE Set-up used



- Chamber lined with magnetic pieces.
- The temperature of the substrate was controlled using back side He cooling and a dedicated chiller.
- The ICP power is decoupled from the substrate power.
- The position of the substrate holder with respect to the ICP source could be varied.
- The RF power supplied operated at 13.56 MHz.

# Sample Preparation



- a) Deposition of Thin Layer of Au/Cr on top of Pyrex® 7740 wafer.
- b) Patterning Au/Cr layer using standard lithography and etching steps.
- c) Electroplating a thick (8-10 microns) layer of Nickel on the patterned seed layer.
- d) ICP RIE etching step with electroplated Ni acting as a hard mask.
- e) Removal of Nickel in a Piranha Clean solution.

# Process Parameters

Process Design Parameter	Units of measurement	Experimental Range
ICP Power	Watts	500-2000
Substrate Power	Watts	100-475
O <sub>2</sub> Flow Rate	sccm	5-100
SF <sub>6</sub> Flow Rate	sccm	5-50
C <sub>4</sub> F <sub>8</sub> Flow Rate	sccm	5-50
CH <sub>4</sub> Flow Rate	sccm	5-50
Ar Flow Rate	sccm	5-50
Operating Pressure	mTorr	1-20
Temperature of Substrate Holder	°C	5-30
Distance of Substrate Holder from ICP	mm	120-200

# Design of Experiment Results

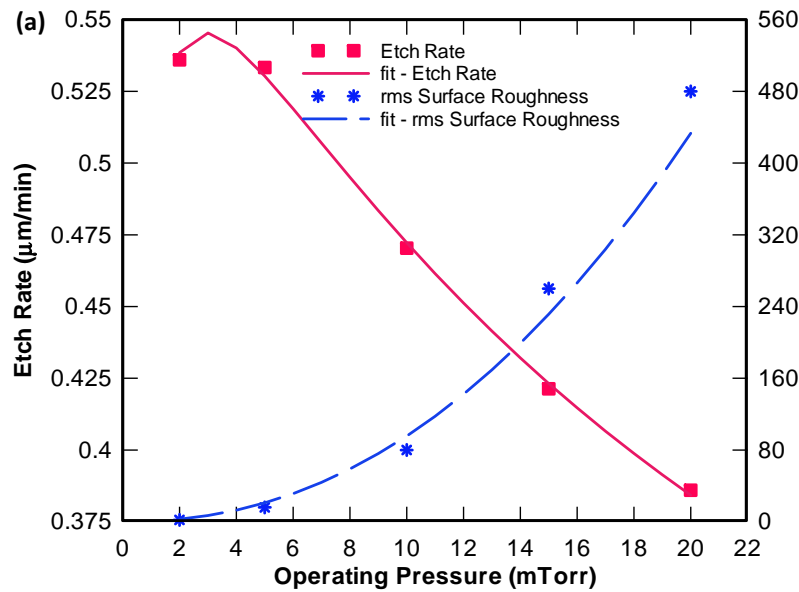
## Nominal Etching Condition for SF<sub>6</sub>/Ar chemistry

Process Parameter	Value	Units
ICP Power	2000	Watts
Substrate Power	475	Watts
Ar Flow Rate	50	sccm
SF <sub>6</sub> Flow Rate	5	sccm
Operating Pressure	<2	mTorr
Distance From Source	120	mm
Substrate Holder Temperature	20	°C

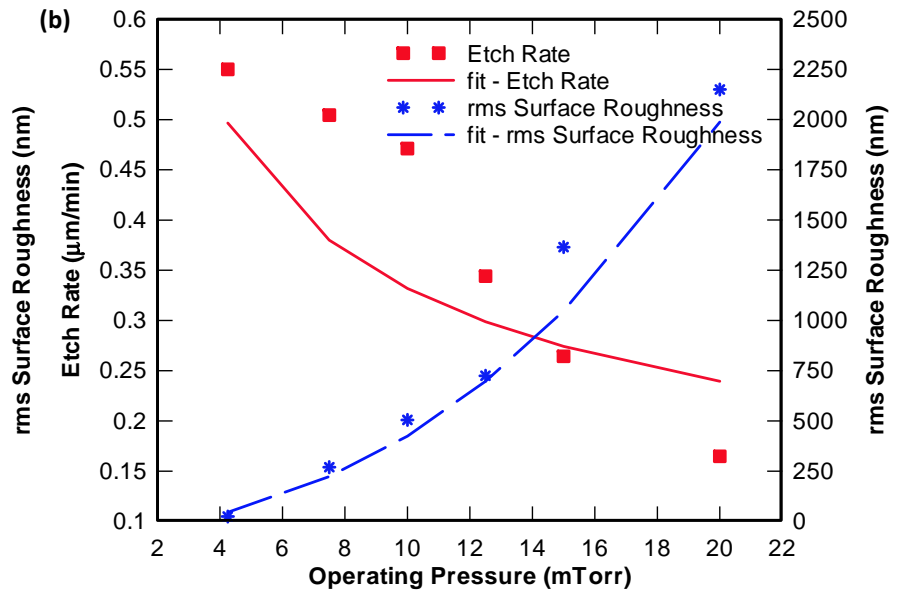
## Nominal Etching Conditions for the C<sub>4</sub>F<sub>8</sub>/SF<sub>6</sub>/Ar/O<sub>2</sub> based chemistry

Process Design Parameter	Units of measurement	Optimum Value
ICP Power	Watts	2000
Substrate Power	Watts	475
O <sub>2</sub> Flow Rate	sccm	50
SF <sub>6</sub> Flow Rate	sccm	5
C <sub>4</sub> F <sub>8</sub> Flow Rate	sccm	5
CH <sub>4</sub> Flow Rate	sccm	Not used
Ar Flow Rate	sccm	50
Operating Pressure	mTorr	Minimum possible
Temperature of Substrate Holder	°C	20
Distance of Substrate Holder from ICP	mm	120

# Effect of Operating Pressure

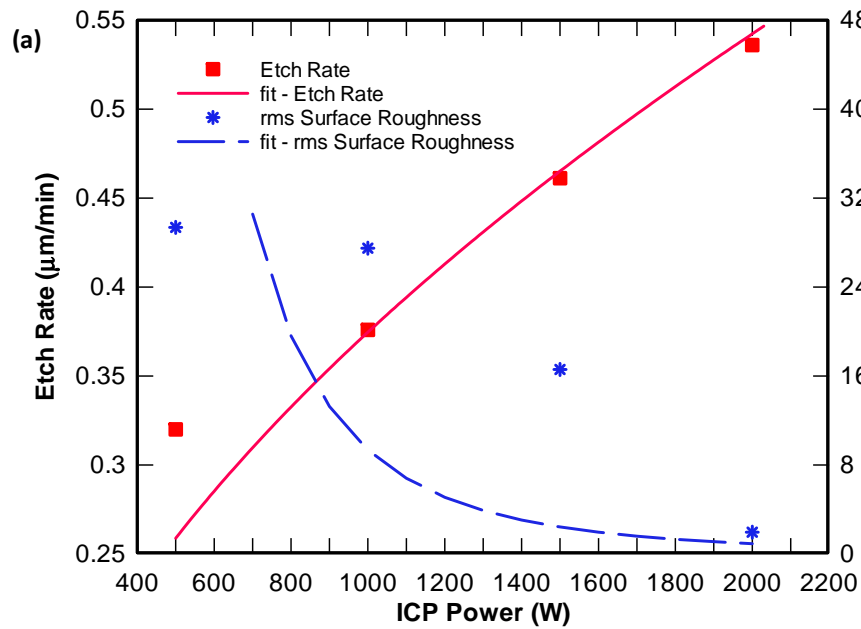


$\text{SF}_6/\text{Ar}$  based chemistry

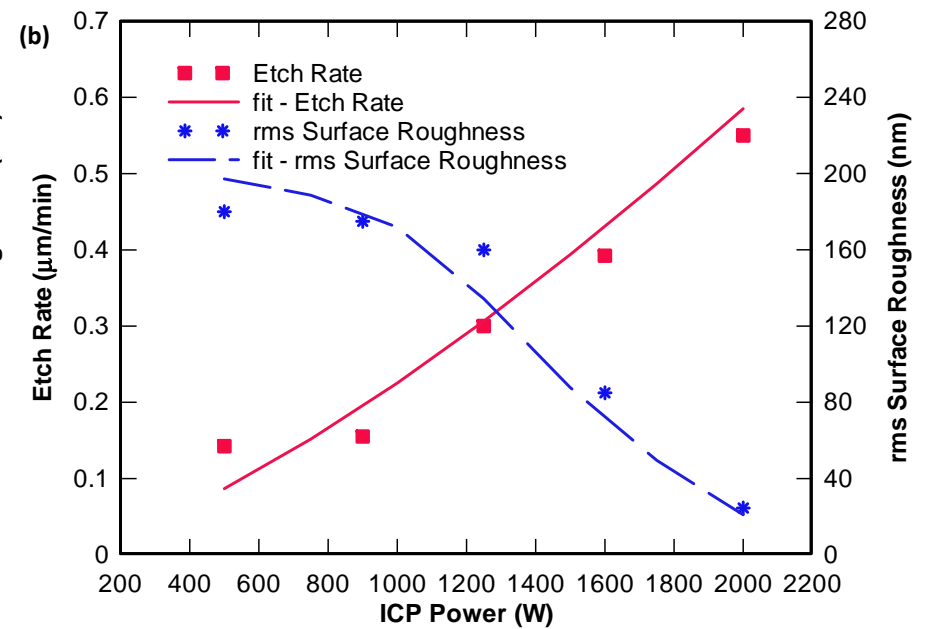


$\text{SF}_6/\text{C}_4\text{F}_8/\text{Ar}/\text{O}_2$  based Chemistry

# Effect of ICP Power

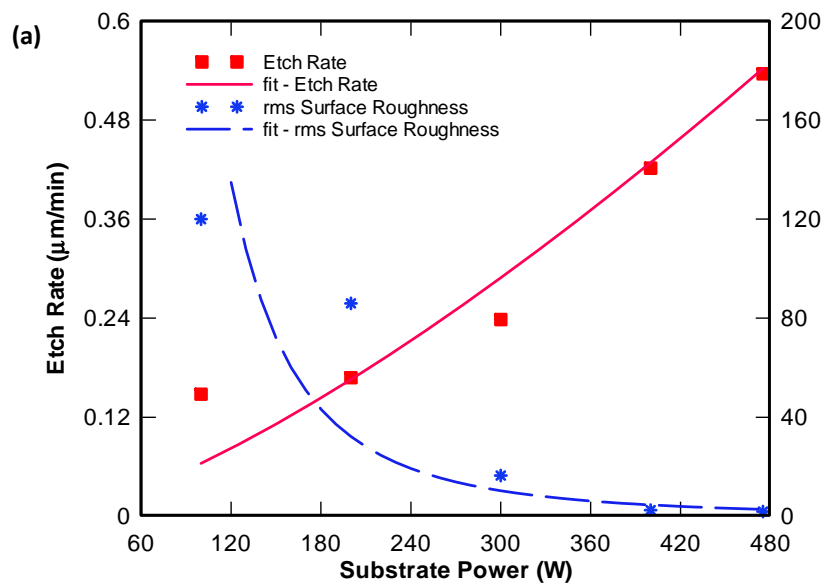


**SF<sub>6</sub>/Ar based chemistry**

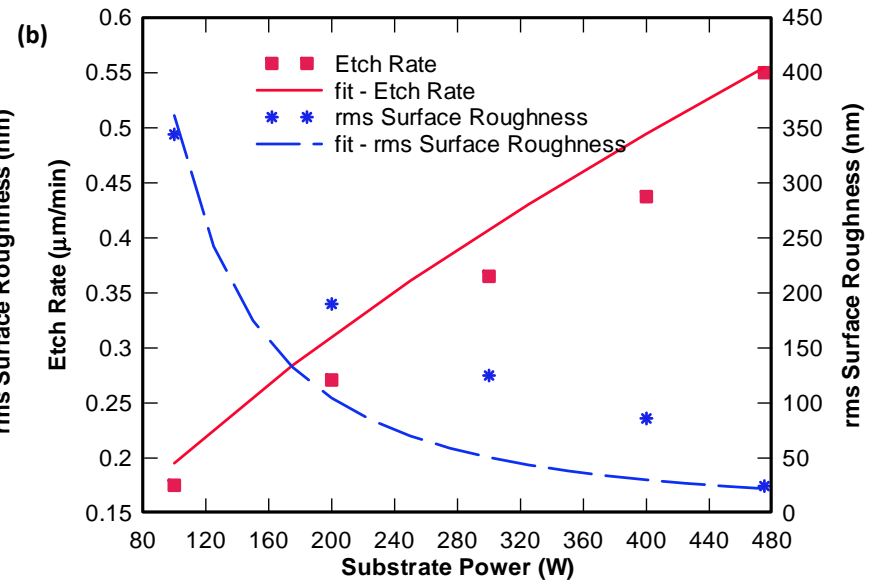


**SF<sub>6</sub>/C<sub>4</sub>F<sub>8</sub>/Ar/O<sub>2</sub> based Chemistry**

# Effect of Substrate Power

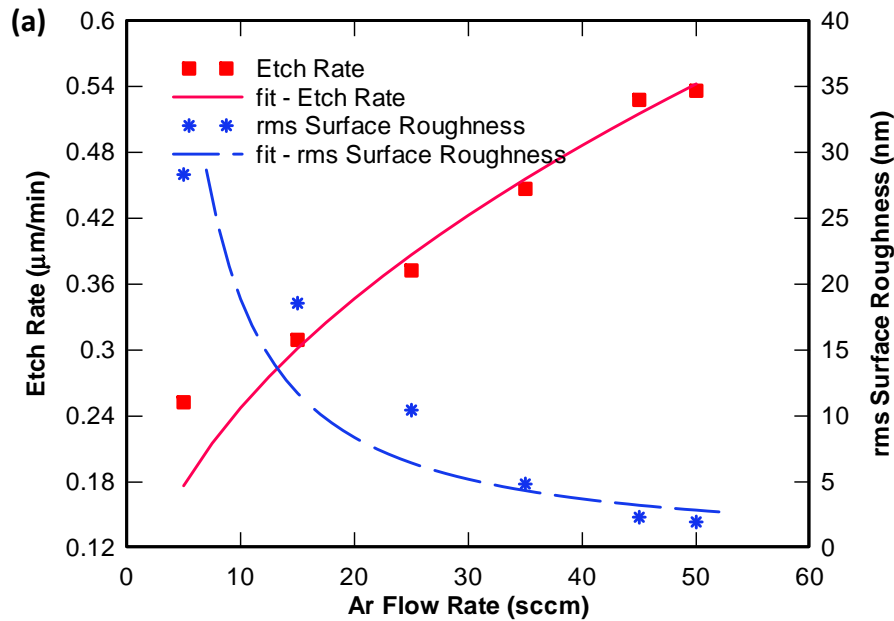


**SF<sub>6</sub>/Ar based chemistry**

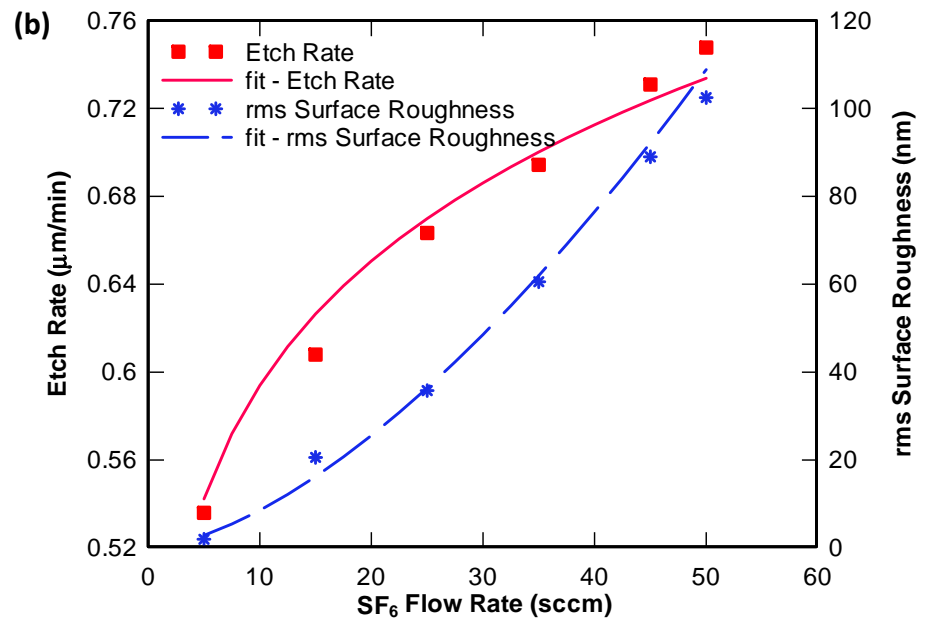


**SF<sub>6</sub>/C<sub>4</sub>F<sub>8</sub>/Ar/O<sub>2</sub> based Chemistry**

# Effect of Flow Rate of Gases

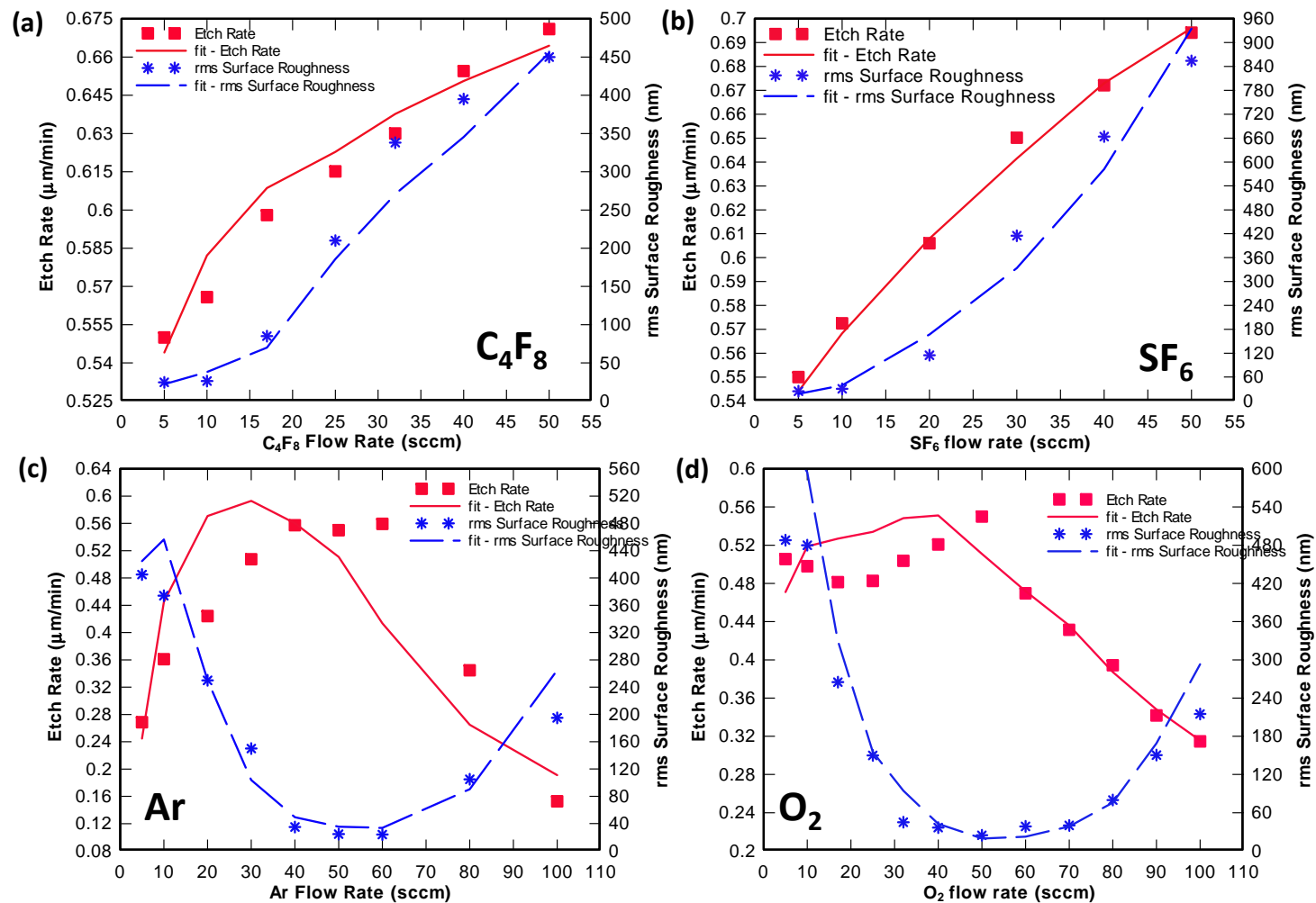


**SF<sub>6</sub>/Ar based chemistry**

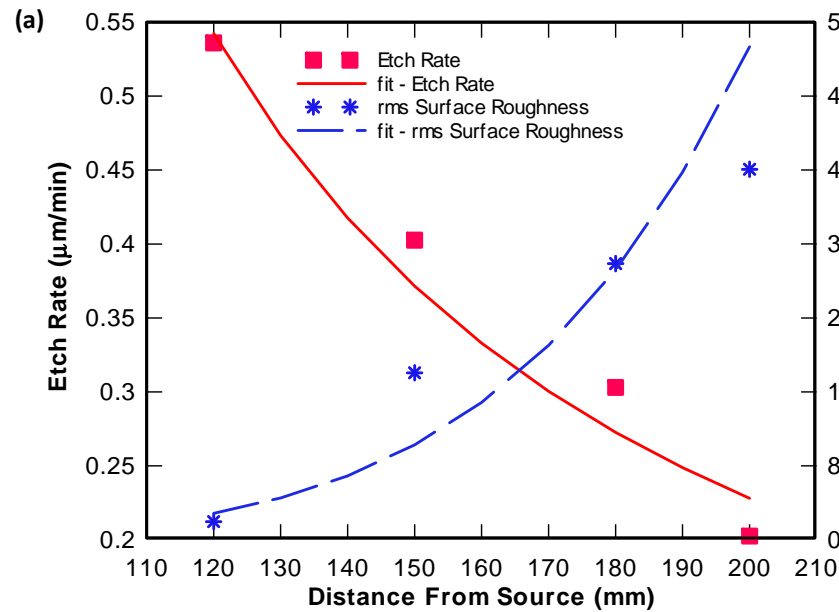


**SF<sub>6</sub>/C<sub>4</sub>F<sub>8</sub>/Ar/O<sub>2</sub> based Chemistry**

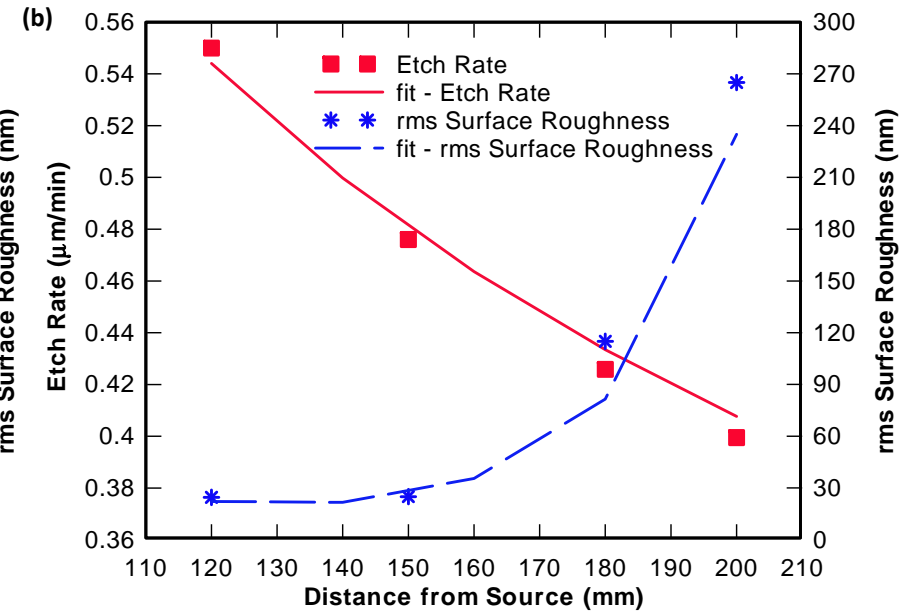
# Effect of Flow rate of Gases



# Effect of Distance from Source



$SF_6/Ar$  based chemistry



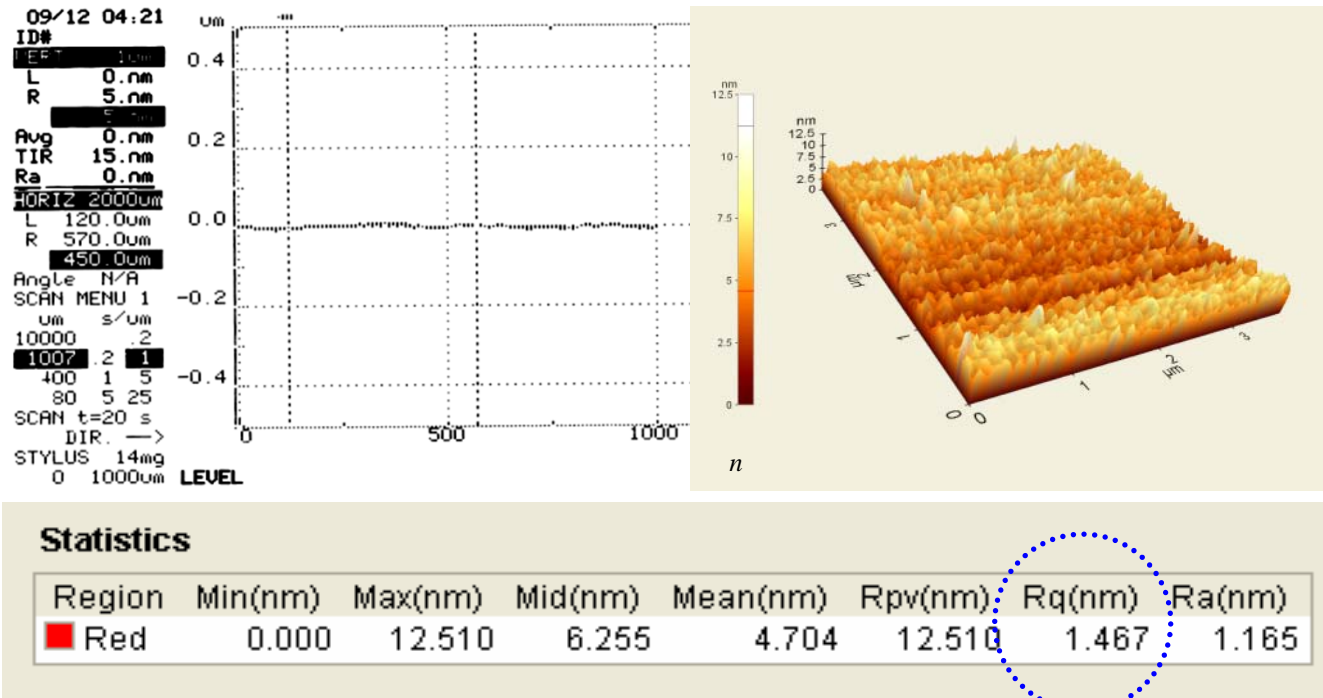
$SF_6/C_4F_8/Ar/O_2$  based Chemistry

# Effect of Temperature

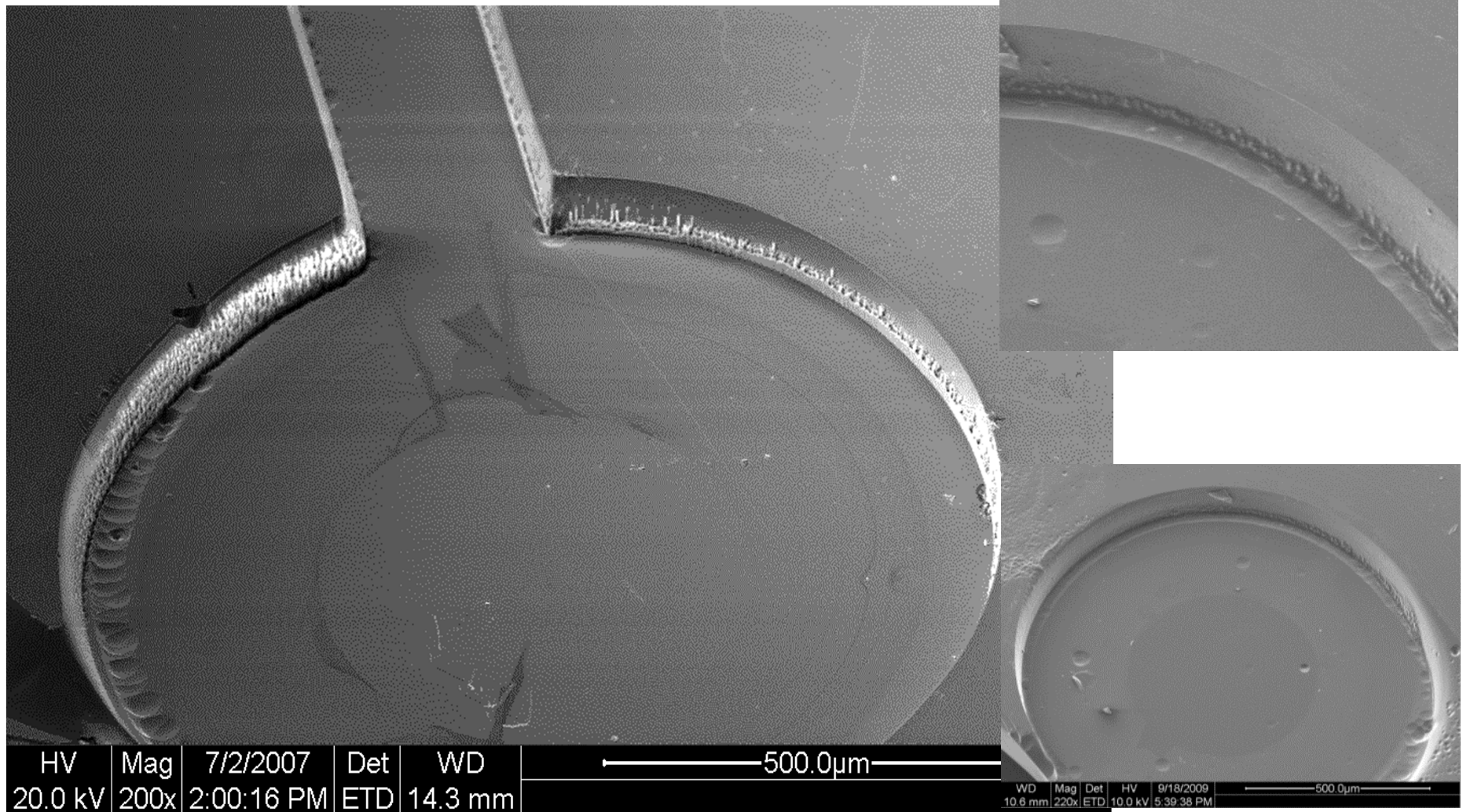
Temperature (°C)	SF <sub>6</sub> /Ar chemistry		SF <sub>6</sub> /C <sub>4</sub> F <sub>8</sub> /Ar/O <sub>2</sub> chemistry	
	Etch Rate (mm/min)	Rms Roughness (nm)	Etch Rate (mm/min)	RMS Roughness (nm)
5	0.498	2.1	0.5275	80
10	0.5	2	0.53	50
20	0.536	1.97	0.55	24.6
30	0.554	1.95	0.566	19.7

# Surface Profile after Etching

After etching to a depth of 75 microns



# Typical Etch Profiles



# Quantization of the Etch Process

- Very useful to define a quantitative metric to relate process parameters to etch characteristics.
- Etch characteristics which vary monotonically with the process parameters can be modeled as power law.
- However, for etch characteristics which do not vary monotonically, higher order terms are required in the quantization process.
- We arranged the data in a  $m$  by  $(n+1)$  matrix, where  $m$  are the number of runs and  $n$  is the number of process parameters being quantized. The last column is the etch characteristic being quantized.

## Quantization cont ....

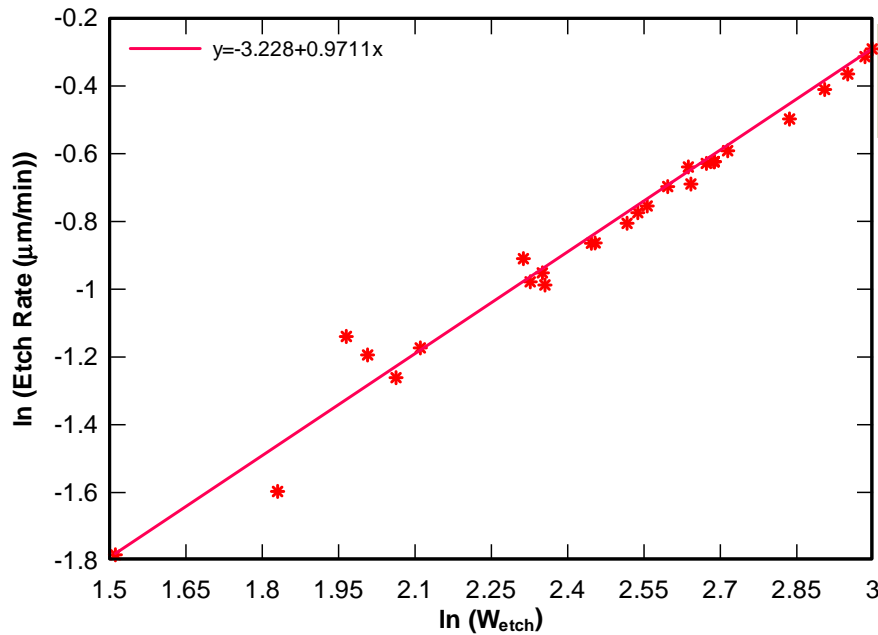
- We then used a commercial software to fit the equation in the form to the data matrix

$$\ln(W) = \ln(a_0) + \sum_{i=1}^n a_i \ln(PP_i) + \sum_{k=2}^3 \sum_{j=1}^n a_j \ln(PP_j^k)$$

where,  $W$  is the arbitrary number relating etch characteristics to the process parameters,  $a_i$  and  $a_j$  are the fitting parameters,  $PP_j$  are the process parameters whose effect on the etch characteristics are being quantized. Rewriting the equation by taking exponentials on both sides

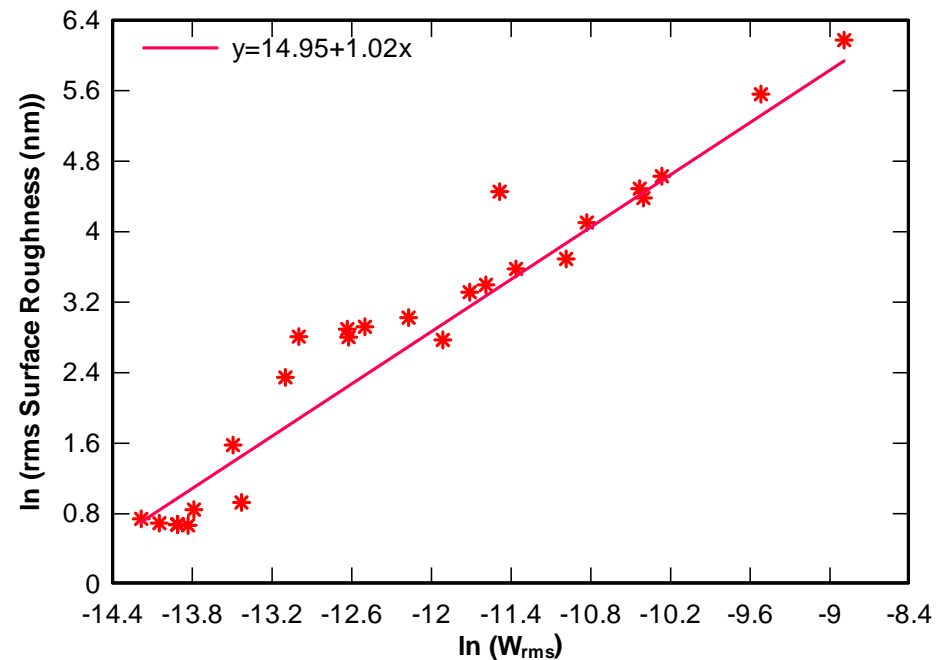
$$W = a_0 \left( \prod_{i=1}^n PP_i^{a_i} \right) \exp \left[ \sum_{k=2}^3 \sum_{j=1}^n a_j \ln(PP_j^k) \right]$$

# For SF<sub>6</sub>/Ar based Chemistry



$$W_{etch} = \frac{W_{ICP}^{0.52} W_{sub}^{1.36} Q_{SF_6}^{0.135} Q_{Ar}^{0.48} T_{sub}^{0.065} P_{process}^{0.2}}{D^{1.67}} \exp(-0.094(\ln P_{process})^2)$$

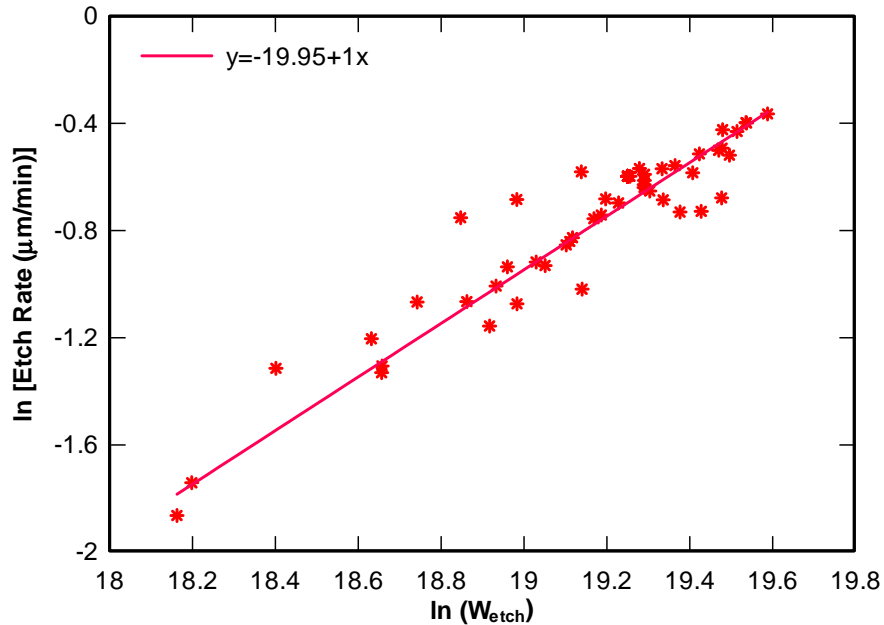
$$R_{etch} = aW_{etch}$$



$$W_{rough} = \frac{Q_{SF_6}^{1.58} P_{process}^{2.18} D^{5.726} T_{sub}^{0.197}}{W_{ICP}^{3.173} W_{sub}^{2.8} Q_{Ar}^{1.172}}$$

$$R_{rough} = bW_{rough}$$

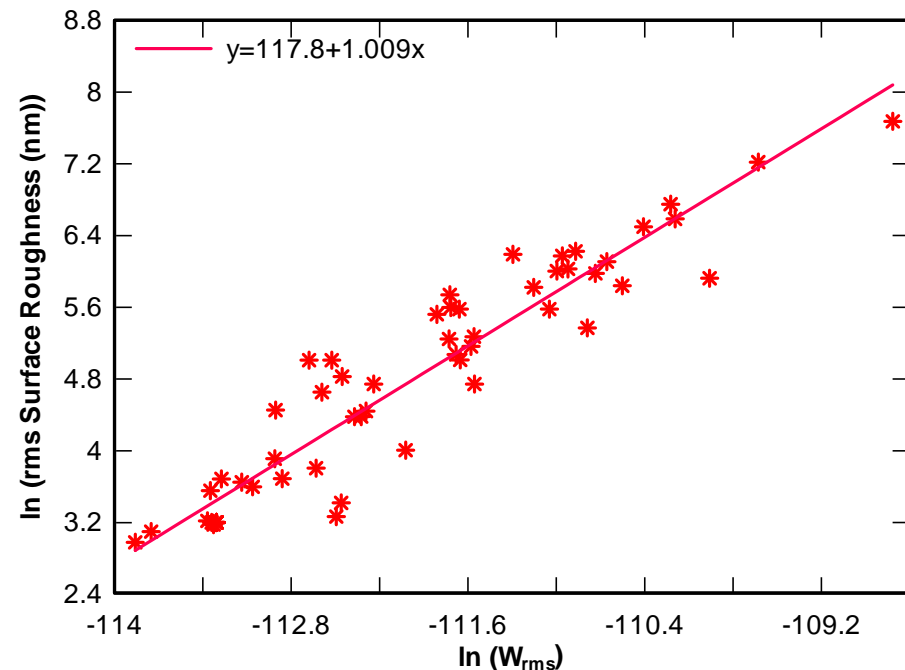
# For C<sub>4</sub>F<sub>8</sub>/SF<sub>6</sub>/Ar/O<sub>2</sub> based Chemistry



$$W_{etch} = \frac{W_{icp}^{1.38} W_{sub}^{0.68} Q_{Ar}^{2.78} Q_{O_2}^{1.32} Q_{C_4F_8}^{0.19} Q_{SF_6}^{0.23} T_{sub}^{0.000424}}{D^{0.465} P^{0.47}} \exp\{corr_1\}$$

$$R_{etch} = cW_{etch}$$

$$corr_1 = -0.42(\ln Q_{Ar})^2 - 0.18(\ln Q_{O_2})^2$$

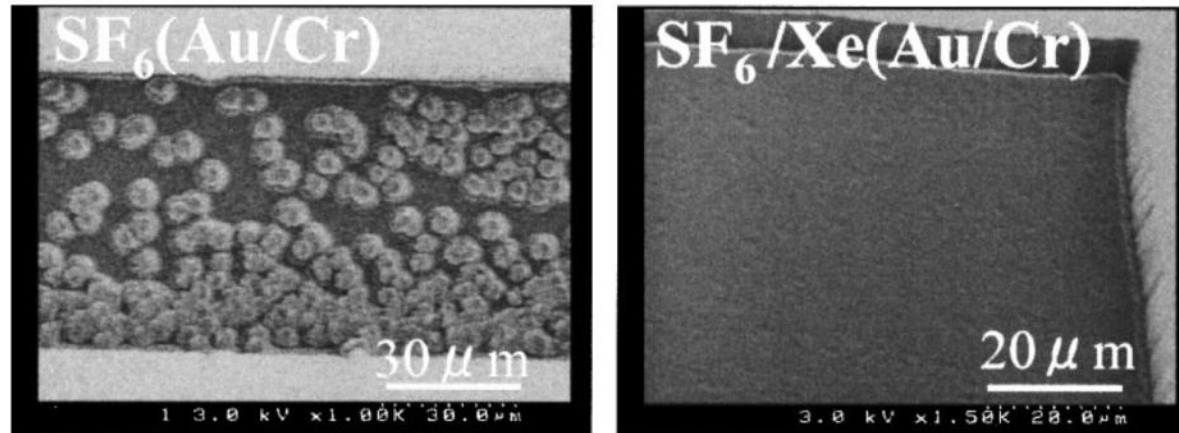


$$W_{rough} = \frac{W_{icp}^{66.39} Q_{Ar}^{30.13} Q_{C_4F_8}^{0.78} Q_{SF_6}^{0.83}}{W_{sub}^{1.79} Q_{O_2}^{50.5} D^{130.4} P^{2.23} T_{sub}^{0.67}} \exp\{corr_2\}$$

$$R_{rough} = dW_{rough}$$

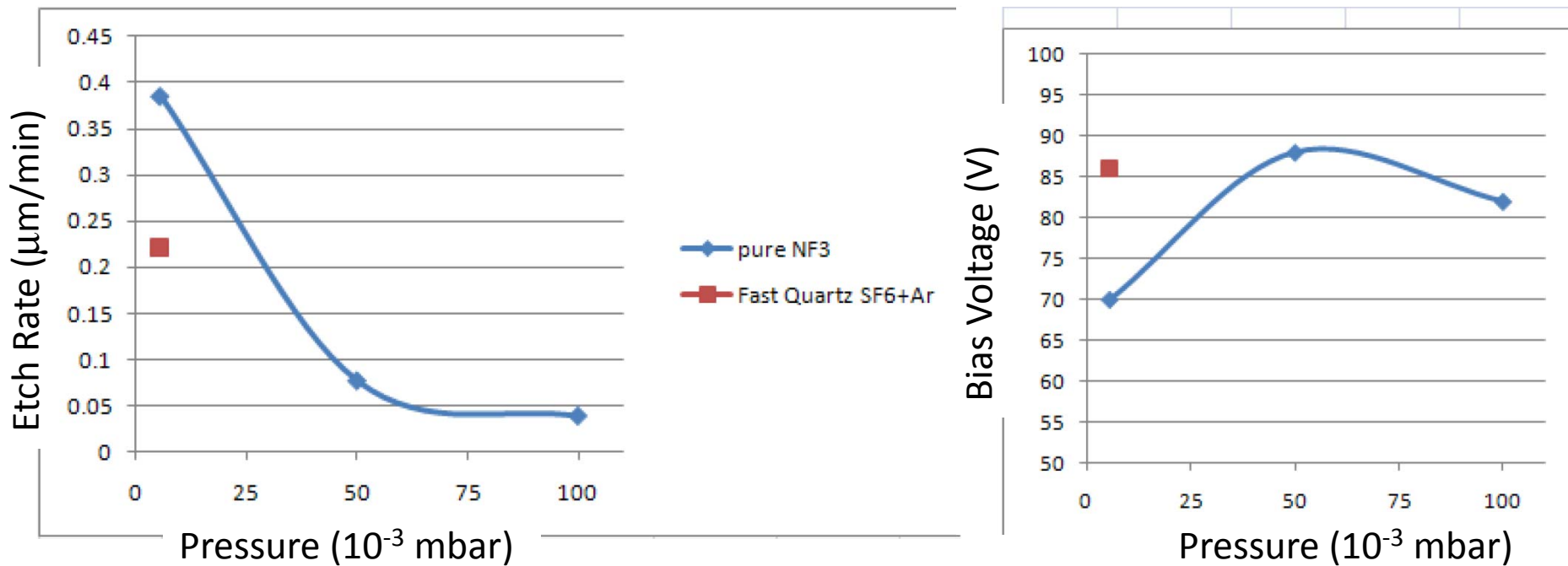
$$corr_2 = -11(\ln Q_{Ar})^2 + 1.2(\ln Q_{Ar})^3 + 13.4(\ln D)^2 + 27.6(\ln Q_{O_2})^2 - 6.8(\ln Q_{O_2})^3 + 0.6(\ln Q_{O_2})^4 + 0.2(\ln P)^2 - 0.04(\ln T)^2 - 4.8(\ln W_{icp})^2$$

# Etch Summary



- Effect of removal of Cr/Au mask before etching
- Sidewall roughness and etch angle is primarily determined by the shape and quality of the mask sidewalls.
- Electroplating with resist pattern in place helps.

# NF<sub>3</sub> Based Etching



- NF<sub>3</sub> based etching shows better etch rate – also leads to a smaller etch bias for similar substrate and ICP power conditions

# Etch Summary

- In ICP RIE process – low pressure improves etch rates since the kinetic energy of bombarding ions is higher due to the increased mean free path
- However, a trade off occurs since absolute number of ions/radicals impinging upon the etch surface decreases with pressure
- Fluorine improves the etch rate, but Ar is required to remove the non-volatile metal oxide residues.
- Thus an etch maximum in the 0.7 – 1.0  $\mu\text{m}/\text{min}$  range has been observed.
- Furthermore increased energy leads to the use of hard mask such as nickel which is not ideal.
- Possible strategies exist including the exploration of organosilane gases, nitrohalogens etc.
- Non-equilibrium plasma processes have been seen to provide higher etch rates – however these require high pressures which is typically not possible in ICP-RIE systems

# Overall Project Summary

The project was funded for \$325,000 for a period of 4½ -years and has been very productive in terms of research output. The following accomplishments summarize the overall results from this project support:

- The first micromachined IR detectors from quartz crystal resonators were successfully demonstrated. Even in their first prototype versions, these detectors have shown performance that rivals some of the more established materials.
- Micromachined quartz resonators were used to evaluate the viscoelastic behavior of various globular proteins. These studies have shown how the self assembly process in protein layers leads to changes in their conformation and rearrangement of the layers. A comprehensive study on various surface types has been undertaken.
- Through a \$25,000 supplement to the project, we have explored the use of nitrohalogen gas  $\text{NF}_3$  for etching of glass and found a small enhancement in the overall etch rate of glass. However, a complete study of this could not be completed due to the short duration of the additional funding.
- **A total of 7 Peer Reviewed Journal Publications and 9 International Conference Presentations and 1 Patent were published by the funds supported by this grant.**
- One Ph.D. student graduated partly supported by the grant, one post-doctoral research associate was trained on this project, and one undergraduate student was involved in the project in addition to the principal investigator.