Wireless Surface Acoustic Wave CO_2 Sensor for Carbon Storage Sites Monitoring

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Developing the Technologies and Building the
Infrastructure for CO_2 Storage

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Presentation Outline

- Technical Background
- Surface Acoustic Wave (SAW) Sensor Testing
- Carbon Nanotube (CNT) – Polyimide (PI) composite performance characterization
- CNT- Polyethylenimine (PEI) composite performance characterization
- SAW CO_2 sensor characterization
- Wireless module characterization

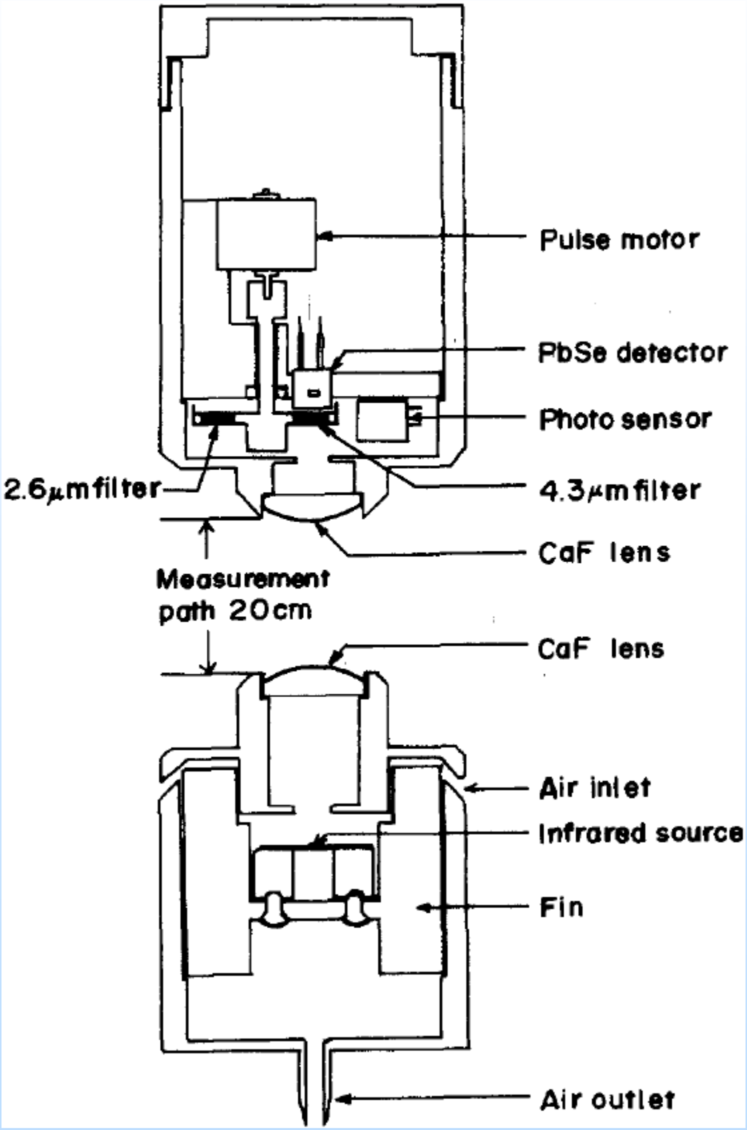
Benefit to the Program

- Program goals being addressed.
  - Conduct field tests through 2030 to support the development of BPMs for site selection, characterization, site operations, and closure practices.
- Project benefits statement.
  - The research project is developing a wireless CO_2 sensing system monitoring CO_2 leakage around the injection wells and ensure timely notification to control center once leakage is detected. The technology, when successfully demonstrated, will provide an improvement over current CO_2 sensors in both wireless performance and power consumption. This technology contributes to the Carbon Storage Program's effort of ensuring 99 percent CO_2 storage permanence in the injection zone(s) (Goal).

Project Overview: Goals and Objectives

- Describe the goals and objectives in the Statement of Project Objectives.
  - The research project aims to develop a wireless CO_2 sensor based on SAW technology and CNT-polymer composite for carbon storage sites monitoring.
  - The research project will develop a sensing system that can respond to CO_2 concentration change in the air in a timely manner and communicate wirelessly with a control center regarding the detection results.

Technical Background



NDIR CO_2 Sensor

CO_2 Absorption on Light with Particular Wavelength

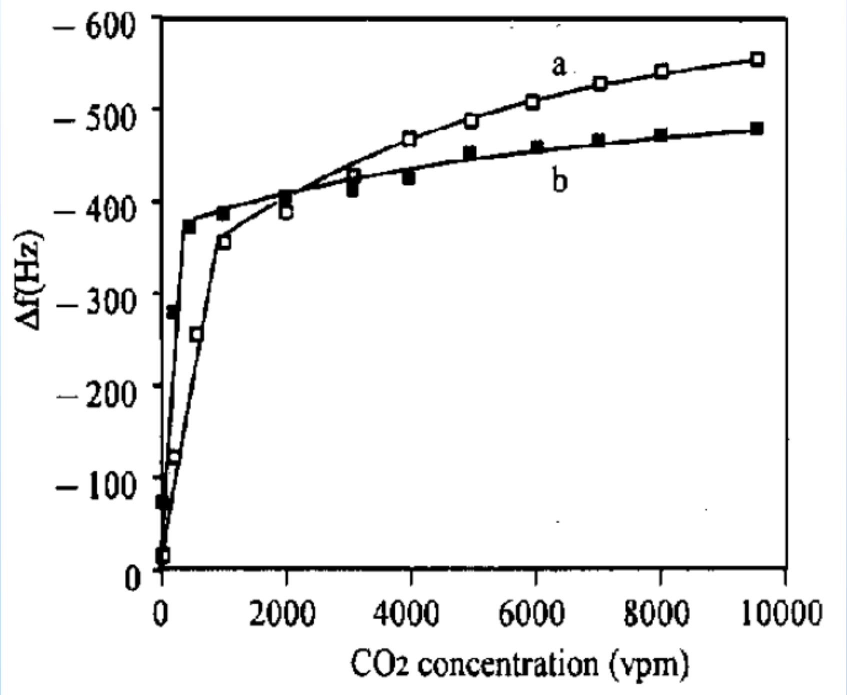
Active Light Source Power Consumption

Technical Background

Chemical CO_2 Sensor

Balance Type CO_2 Mass Sensor

$$\Delta f = -C_f (f_0^2 / A)\Delta m$$



A Electrode Area

C_f Mass Sensitivity Constant

f_0 Fundamental Frequency

Operated at 70℃

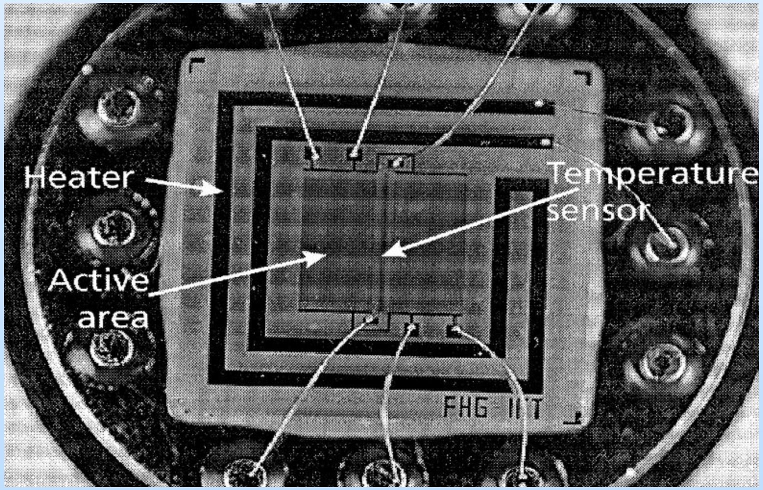
Technical Background

Chemical CO_2 Sensor

Capacitor Type CO_2 Sensor

70% Aminopropyltrimethoxysiloxane (AMO)

30% propy-ltrimethoxysilane (PTMS)



$-NH_2 + CO_2 + H_2O \leftrightarrow -NH_4HCO_3$

Require 120kJ/mol Energy

Operated above 60℃

Technical Background

Chemical CO_2 Sensor

Resistance Type CO_2 Sensor

$BaTiO_3$-CuO

$BaCO_3$

Ag_2SO_4

Na_2CO_3

Some Operated at elevated Temperature

Technical Background

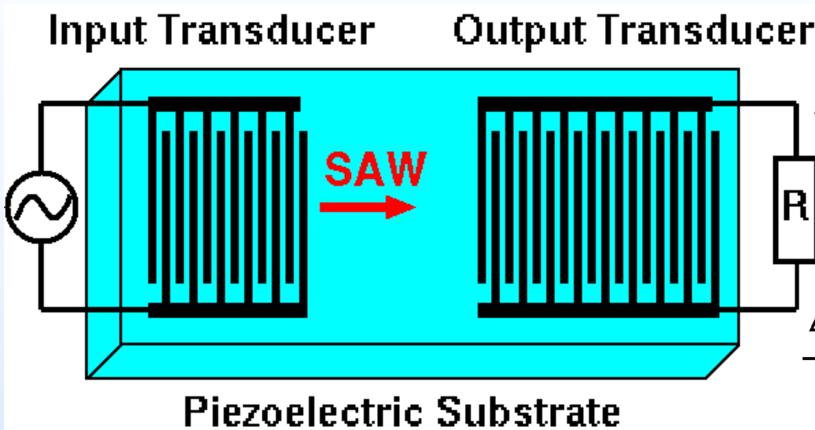
CO_2 Sensor

- *Non Dispersive Infrared (NDIR) CO_2 Sensor*
- Chemical CO_2 Sensor
  - *Aqueous and others*
  - **Solid State**

Wireless Passive Sensing → Surface Acoustic Wave (SAW) CO_2 Sensor

Technical Background

Surface Acoustic Wave (SAW) Sensor



$$\frac{\Delta\phi}{\phi} = \kappa \left[\underbrace{c_m f_0 \Delta\rho_s}_{mass} + \underbrace{\frac{K^2}{2} \Delta\left(\frac{\sigma_s^2}{\sigma_s^2 + v_0^2 C_s^2} \right)}_{conductivity} \right]$$

$$\frac{\Delta\alpha}{k} = \kappa \frac{K^2}{2} \Delta\left(\frac{v_0 C_s \sigma_s}{\sigma_s^2 + v_0^2 C_s^2} \right)$$

$\Delta\phi$ Phase Change

$\Delta\alpha$ Attenuation Change

K^2 Electromechanical Coupling Coefficient

σ_s Film Sheet Conductivity

C_s Capacitance per Length of Substrate

κ Fraction of Propagation Path Covered by Sensing Film

c_m Mass Sensitivity Coefficient

ρ_s Mass Per Unit Area

f_0 Frequency of Operation

k Wave Number

Technical Background

Common Used Piezoelectric Materials Properties

Substrate	Cut	Propagate Direction	Wave Speed(m/s)	Coupling Coeff K^2 (%)	Temperature Coefficient (ppm/℃)	Transmission Loss(dB/cm)
Quartz	Y	X	3159	0.23	-22	0.82
	ST	X	3158	0.16	0	0.95
$LiNbO_3$	Y	Z	3485	4.5	-85	0.31
	131°Y	X	4000	5.5	-74	0.26
	128° Y	X	4000	5.5	-72	
$LiTaO_3$	Y	Z	3230	0.74	-37	0.35
	X	112° Y	3295	0.64	-18	

ST cut Quartz → Zero Temperature Coefficient, High Transmission Loss

$LiNbO_3$ → Low Transmission Loss, High Temperature Coefficient

Technical Background

SAW Sensor

- Temperature/Pressure
- Mass Deposition
- Conductivity Change
- Elasticity Change

+

CO_2 Sensing Film

- Metal Oxide → (Heated) → *Conductivity Change*
- Polymer/Composite → **Conductivity Change**
- Ceramics
- Sol-Gel

Technical Background

CO_2 Sensing Film
Conductivity Change
→
Carbon Nanotube (CNT)
+
Polymer

CNT
- High Surface Volume Ratio
- Conductor
- Resistivity Change to CO_2 with help of Polymer

Polymer
↓
Polyimide (PI)
- Harsh Environment Capable
- Porous Structure for CO_2 Sensing

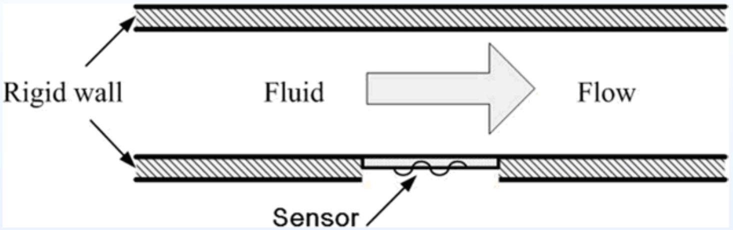
Technical Background

- SAW Sensor Fabrication and Characterization through Flow Sensor Development

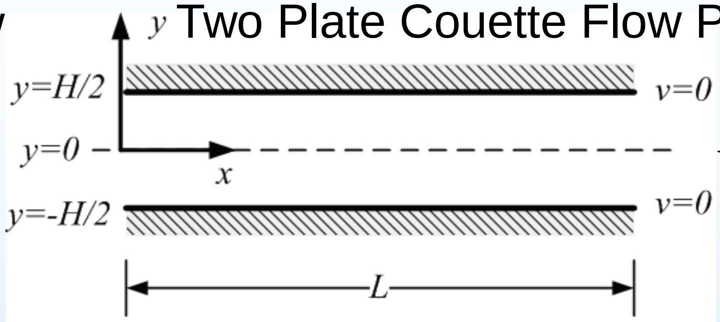
Purpose
- Study the Piezoelectric Materials Properties
- Investigate SAW Signal Characteristics
- Study the Impact of Different Parameters

SAW sensor testing-Flow sensor

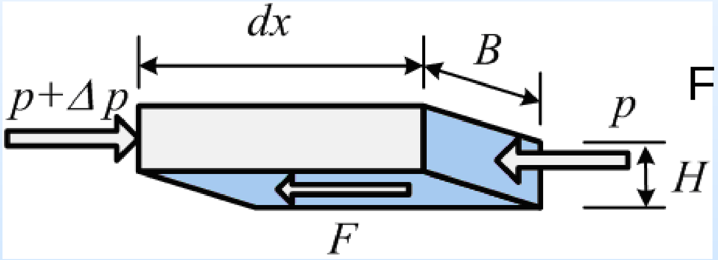
Pressure Driven Laminar Flow



Two Plate Couette Flow Problem



$$v = V\left(1 - \frac{4y^2}{H^2}\right)$$



F=f dx 2(B+H)= - dp A

$$V = -\frac{BH}{2\alpha(B+H)}\frac{dp}{dx} \approx -\frac{H}{2\alpha}\frac{dp}{dx}$$

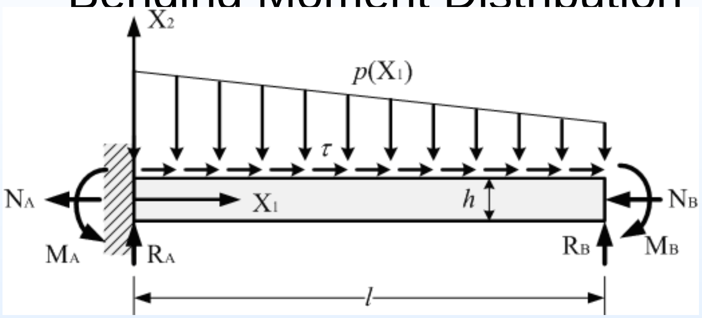
Flow Rate $Q = \frac{2VHB}{3} = \frac{p_0 H^3 B}{12\mu L}$

Pressure $\sigma_{xx} = \sigma_{yy} = -p(x) = -p_0(L - x)/L - p_1$

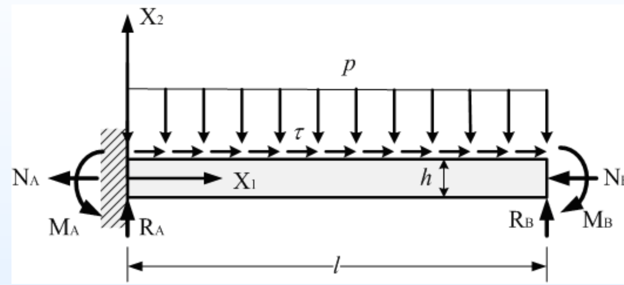
Shear Stress $\tau_{xy} = -\tau_0 y, \quad \tau_0 = \frac{p_0}{L}$

SAW sensor testing-Flow sensor

Bending Moment Distribution



Simplification



Extensional and Flexural Displacement

$$w_1^{(0)}(X_1) = \frac{\tau(l - X_1)X_1}{2\bar{c}_{11}A} = \frac{\tau(l - X_1)X_1}{2\bar{c}_{11}h},$$

$$w_{2,1}^{(0)}(X_1) = -\frac{pX_1(l - X_1)(l - 2X_1)}{12\bar{c}_{11}I} = -\frac{pX_1(l - X_1)(l - 2X_1)}{\bar{c}_{11}h^3},$$

$$w_2^{(0)}(X_1) = -\frac{pX_1^{\,2}(l - X_1)^2}{24\bar{c}_{11}I} = -\frac{pX_1^{\,2}(l - X_1)^2}{2\bar{c}_{11}h^3}$$

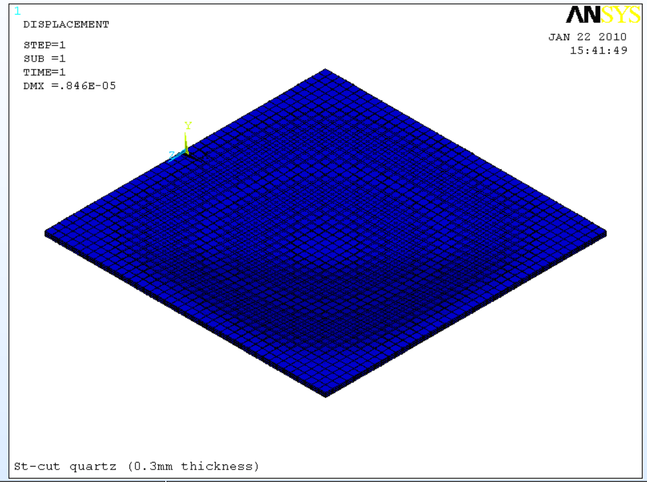
$$w_1(X_1) \cong w_1^{(0)}(X_1) - X_2 w_{2,1}^{(0)}(X_1),$$

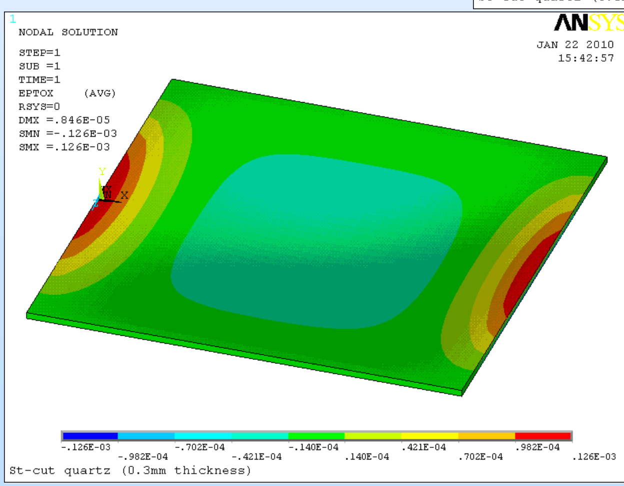
$$w_2(X_1) \cong w_2^{(0)}(X_1),$$

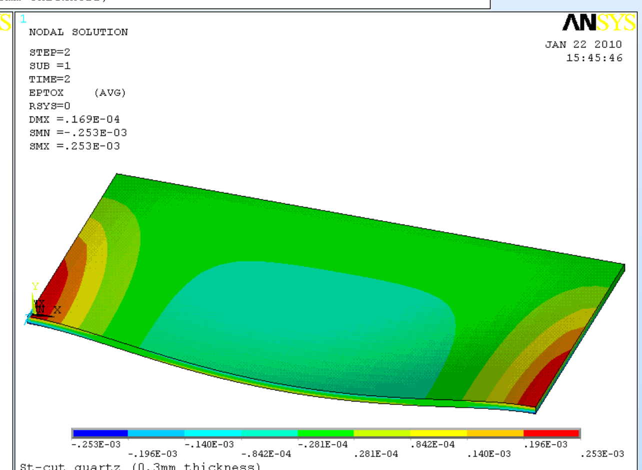
$$w_3(X_1) \cong 0.$$

SAW sensor testing-Flow sensor

Simulation

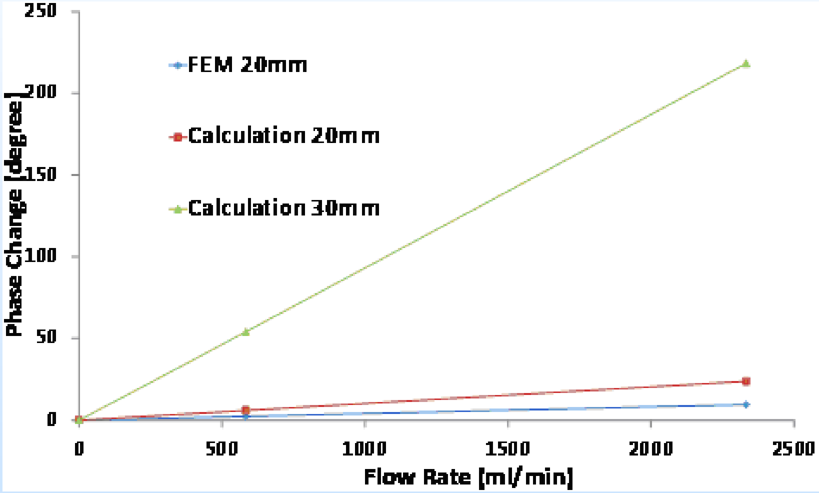


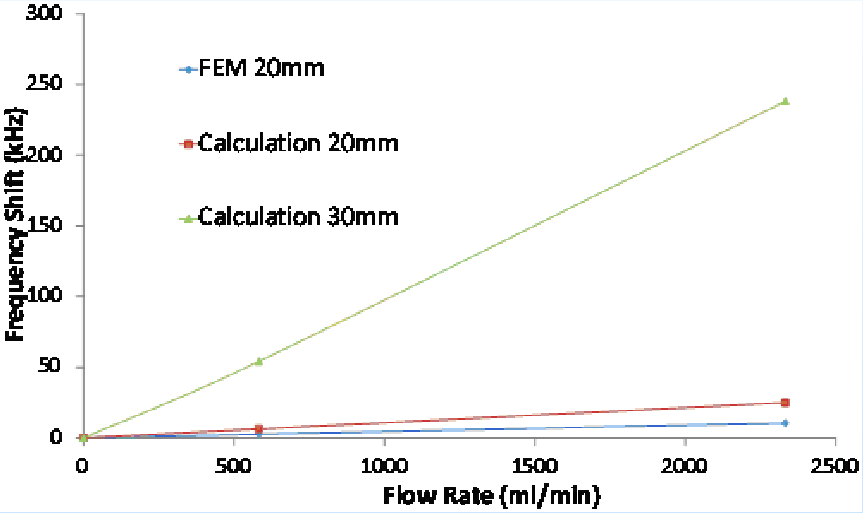




SAW sensor testing-Flow sensor

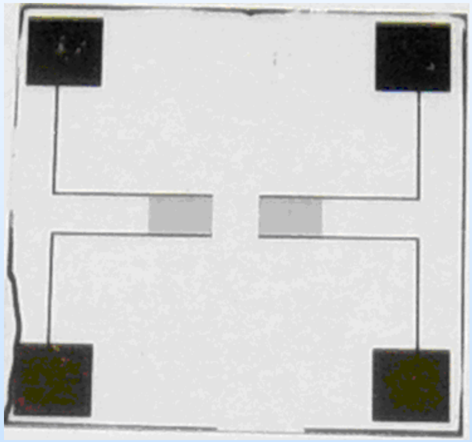
Simulation and Calculation Results

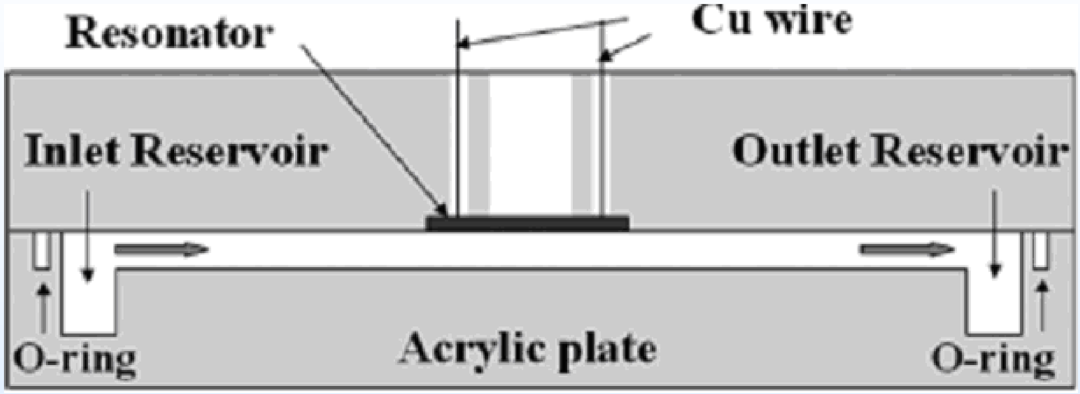


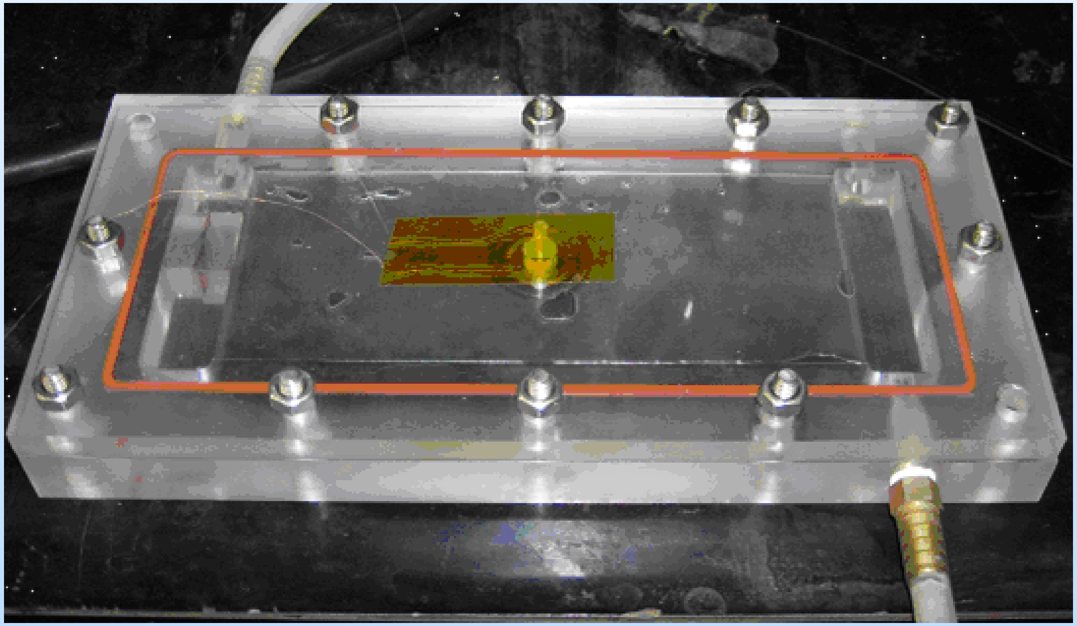


SAW sensor testing-Flow sensor

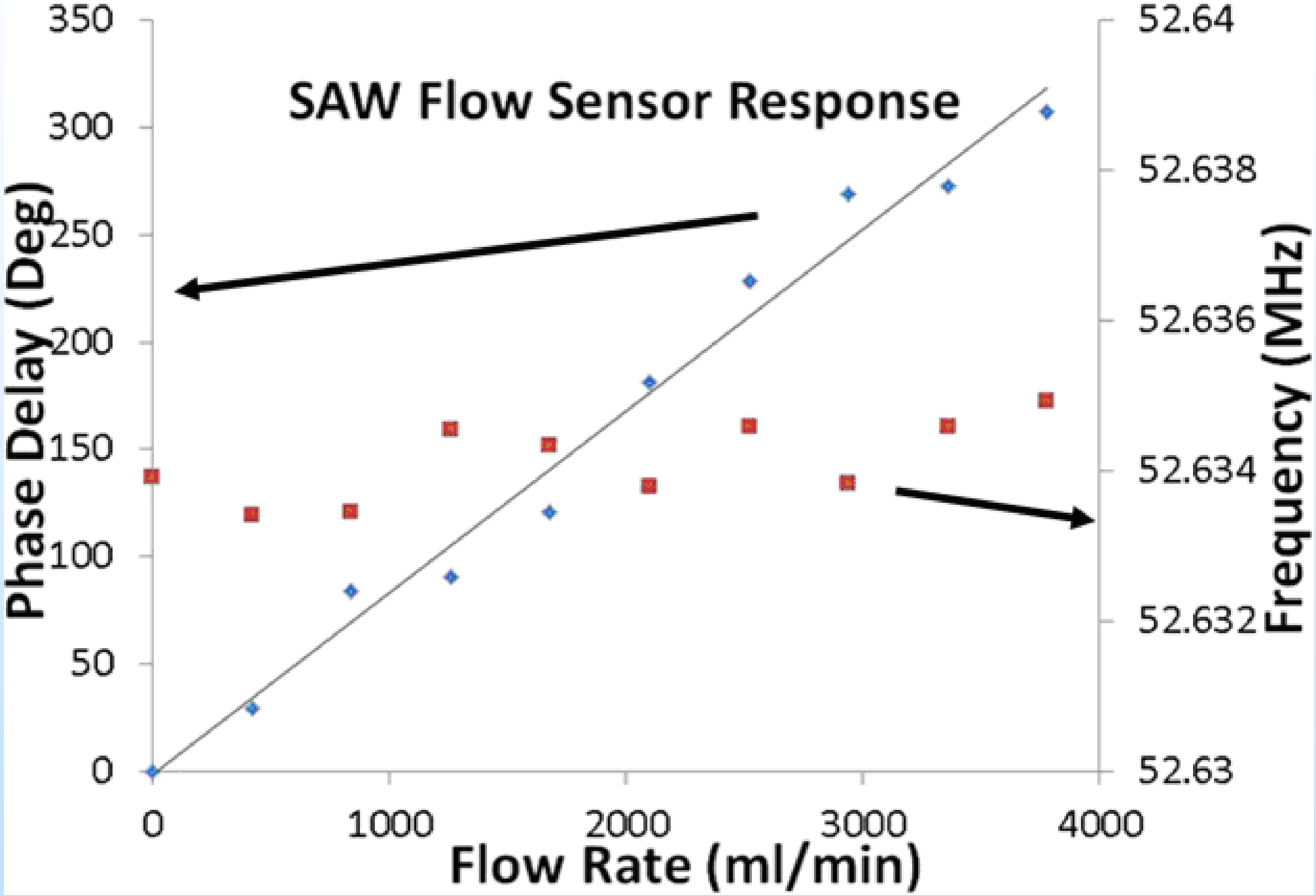
Sensor and Setup







SAW sensor testing-Flow sensor



Phase Delay → 1 Deg per 11.8ml/min

Frequency → Changes fall in fabrication error range

SAW sensor testing-Flow sensor

- Fabricated SAW Sensor for Flow Rate Measurement ✓
- Piezoelectric Materials Studied ✓
- SAW signal Characterized ✓
- Parameters Impact Studied ✓

CNT-PI composite characterization

- Fabricate and Characterize Nanocomposite

CNT Concentration for Percolation Threshold

Temperature

Humidity – Can use Dessicant

Strain (deformation)

CNT-PI composite characterization

Resistance Measurement Device Fabrication

IDT-Regular Photolithography Process

- 50nm Cr layer deposited on Quartz
- 150nm Au layer sputtered on top
- AZ4210 spun-coated and Patterned
- Au/Cr Etching
- Photoresist Removal and Wafer Cleaning

→

- 60 Pairs Fingers
- 5mm Length
- 36um Width
- 44um Spacing
- 4.8mm Aperture

CNT-PI composite characterization

Nanocomposite Fabrication

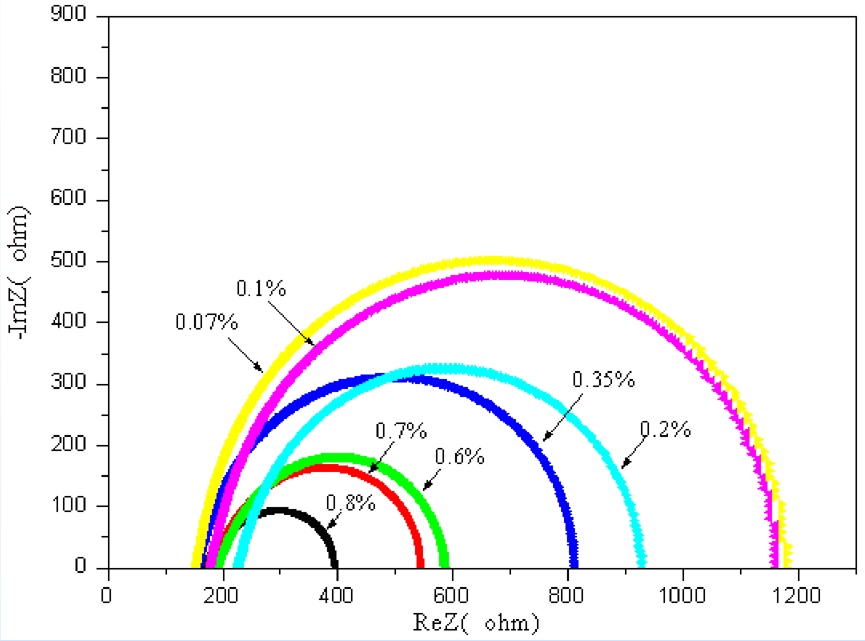
swCNTs → Aldrich, 2g, St. Louis, MO., USA, 1-2nm OD., 5-30um L, >90wt%

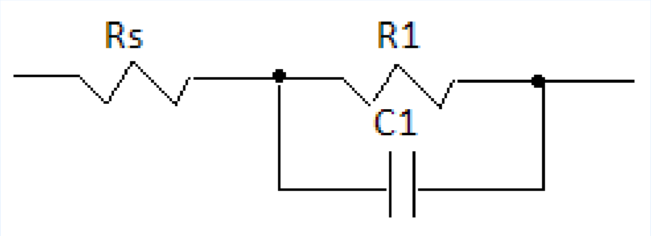
Polyimide → HD microsystem, 1 Gal., Parlin, NJ, USA

- CNT+Solvent
- Stir by Magnetic Stir Bar
- Ultrasonic until Uniform Texture
- Pour the mixture on IDT
- Baked at 350℃ in oven for 30 min

CNT-PI composite characterization

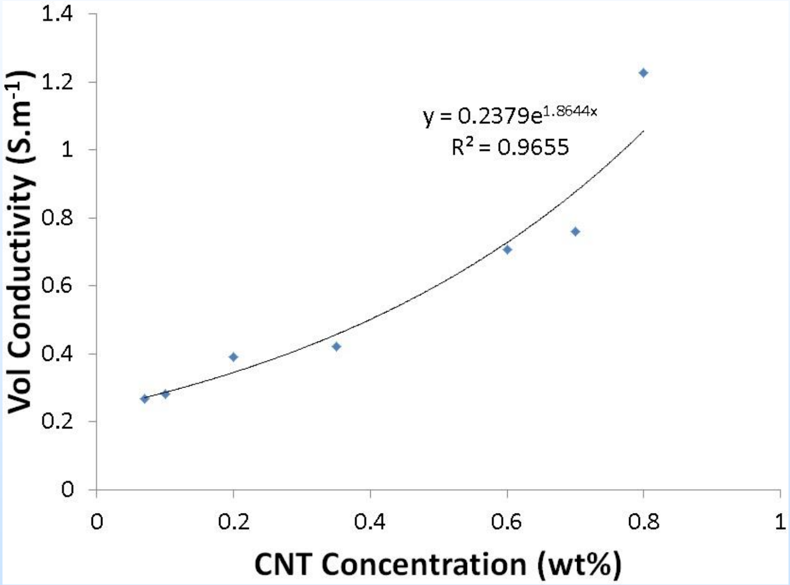
Impedance Analysis



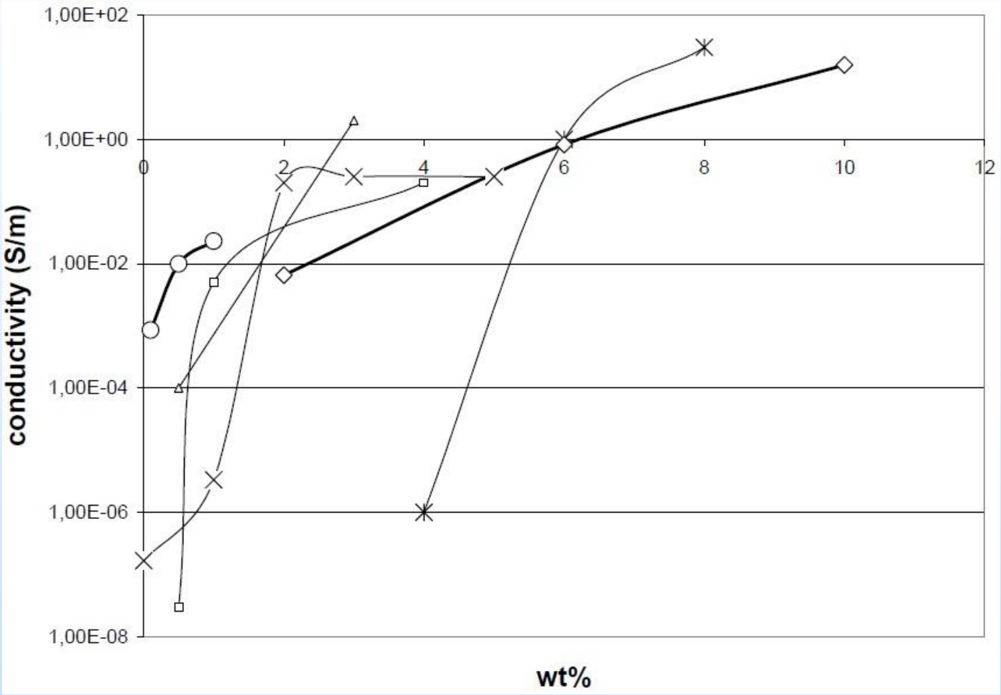


CNT-PI composite characterization

Percolation



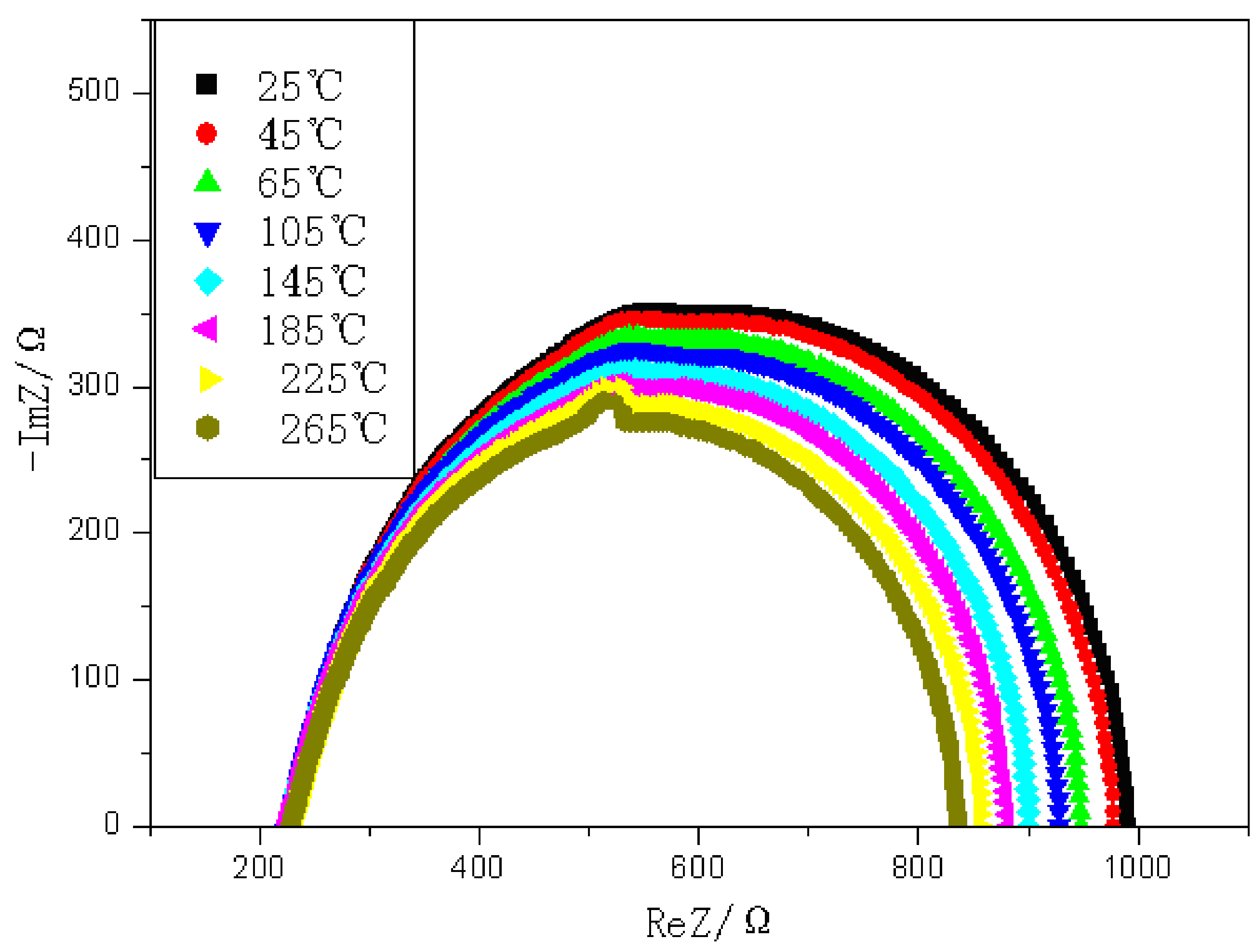
Reference



Threshold between 0.7% and 0.8%

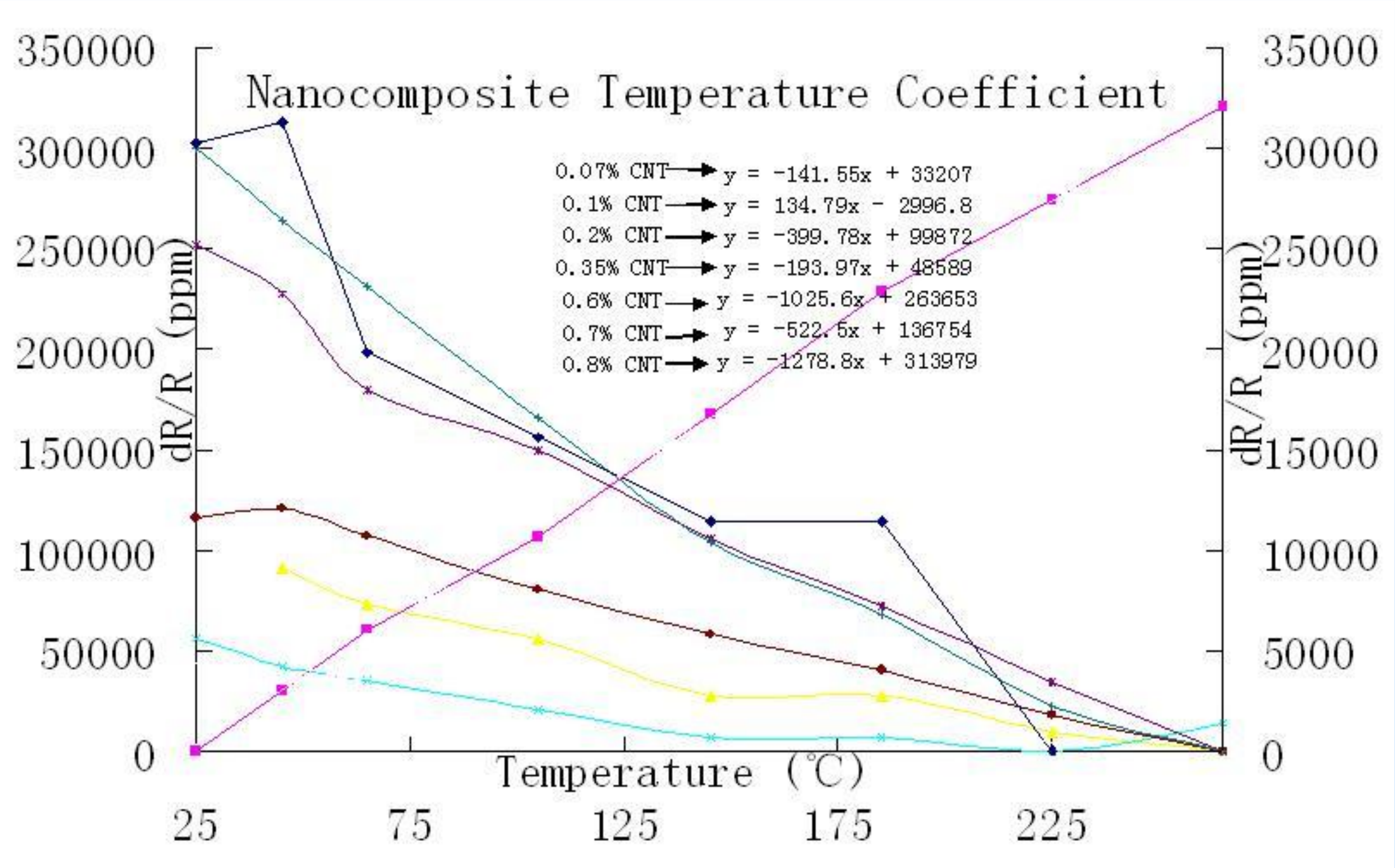
CNT-PI composite characterization

Resistivity Temperature Coefficient



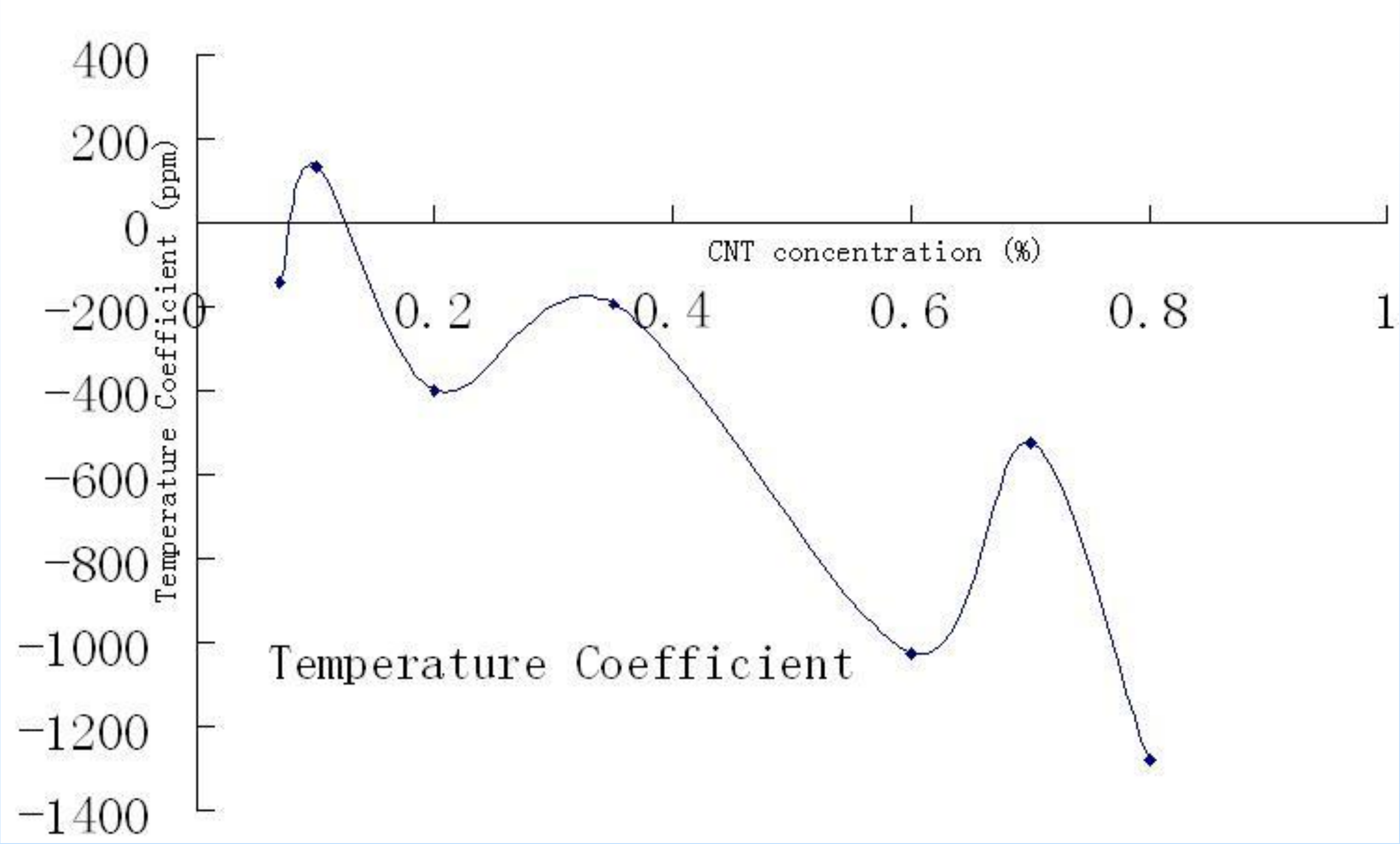
CNT-PI composite characterization

Resistivity Temperature Coefficient



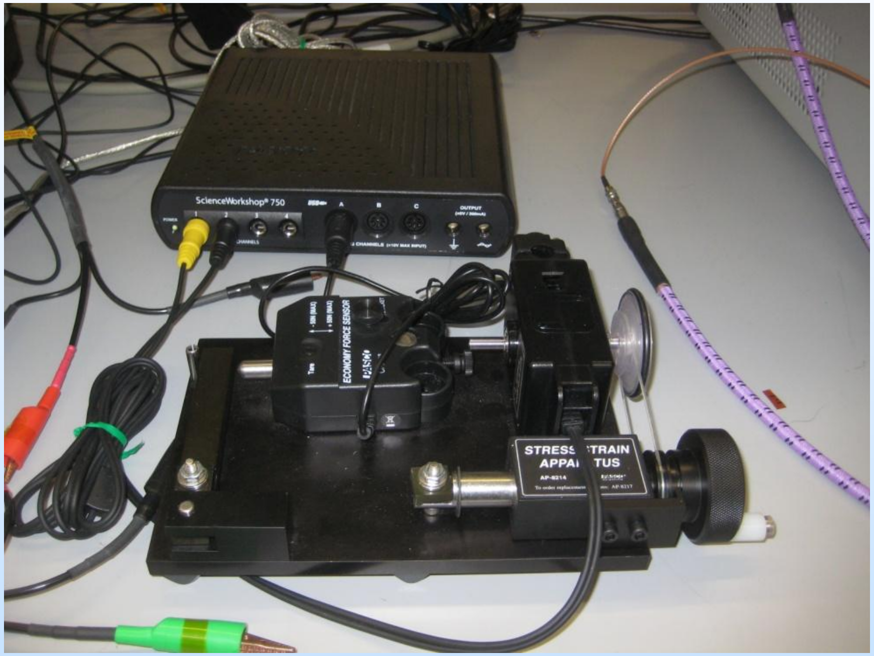
CNT-PI composite characterization

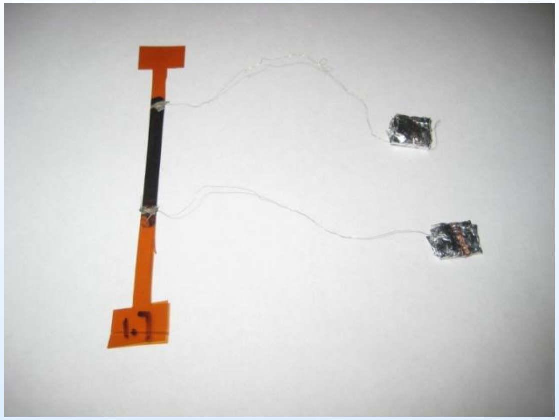
Resistivity Temperature Coefficient

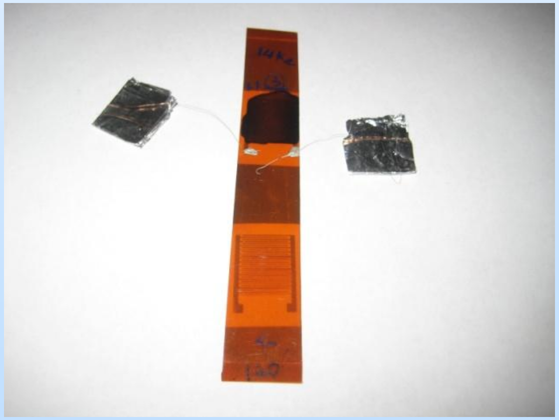


CNT-PI composite characterization

Resistivity Change with Strain

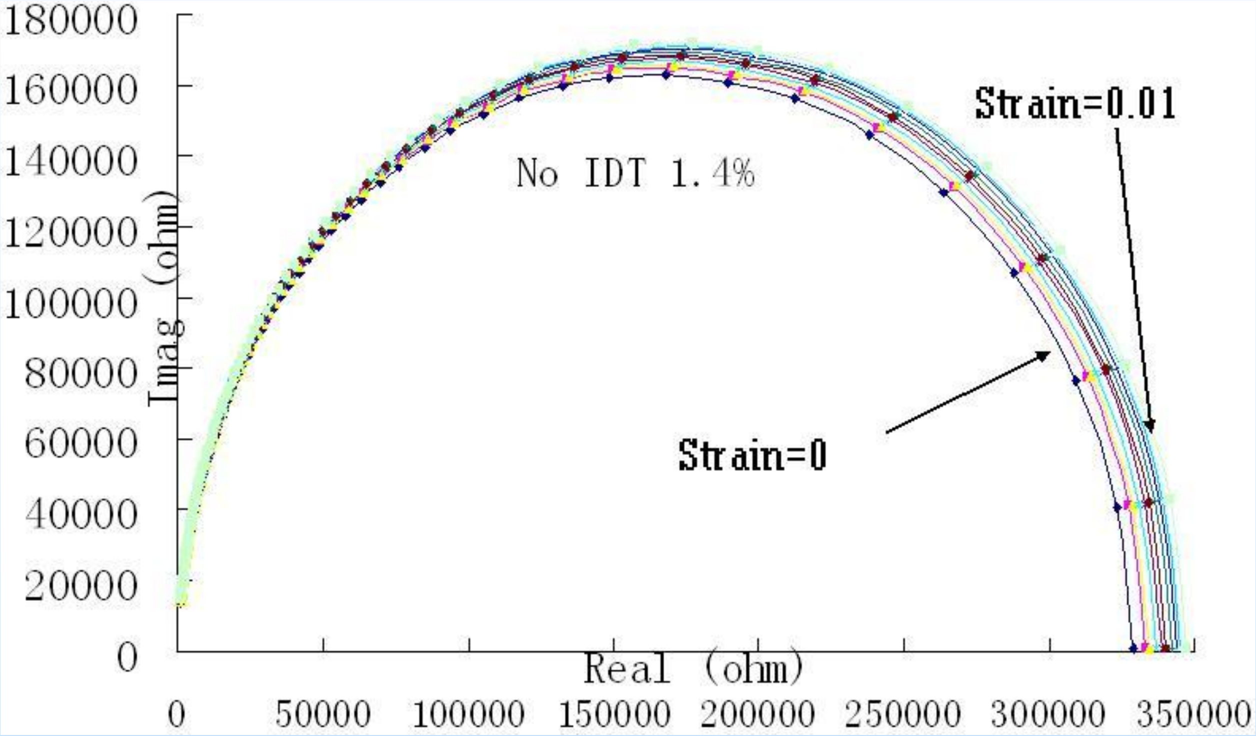






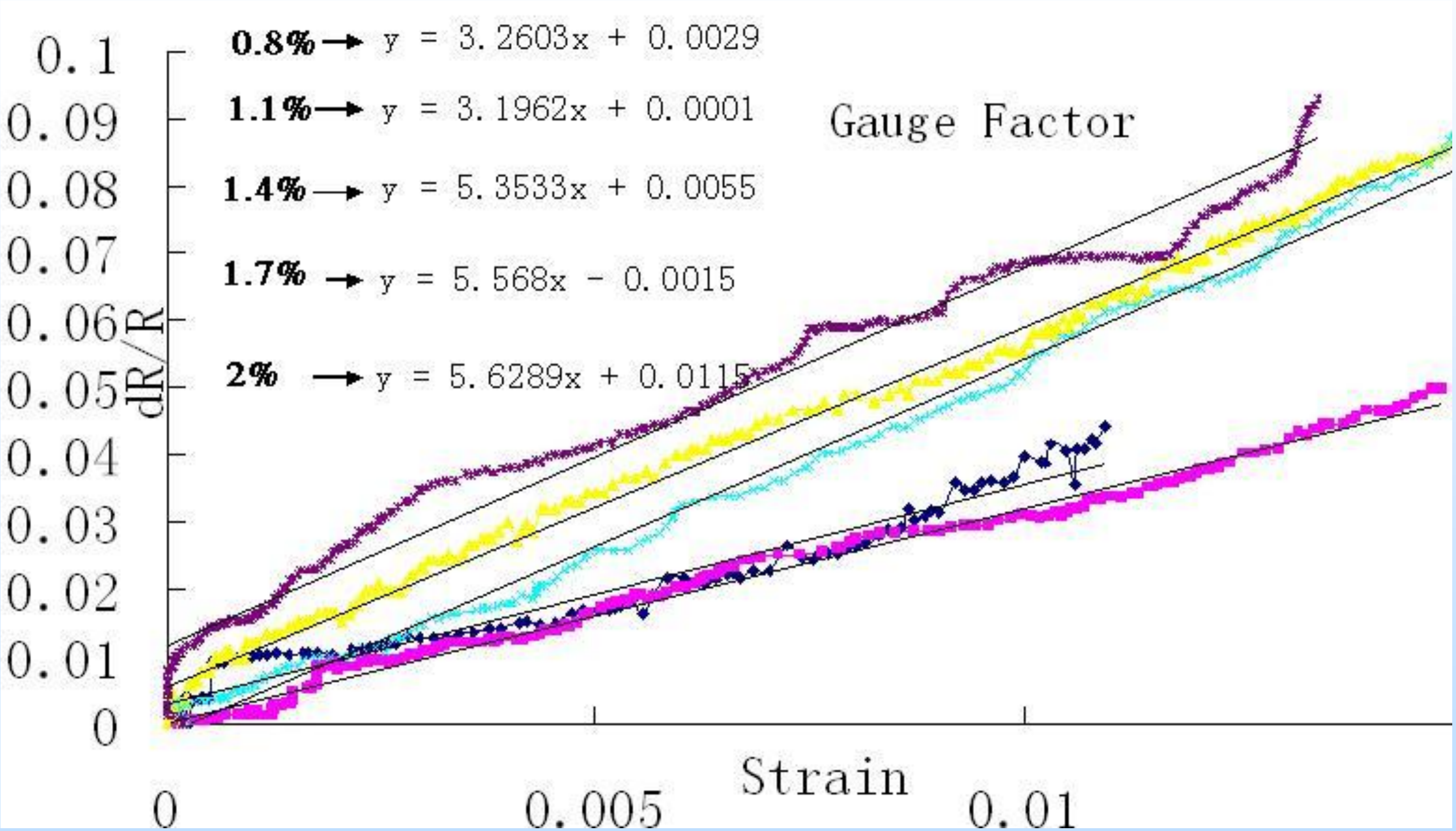
CNT-PI composite characterization

Resistivity Change with Strain



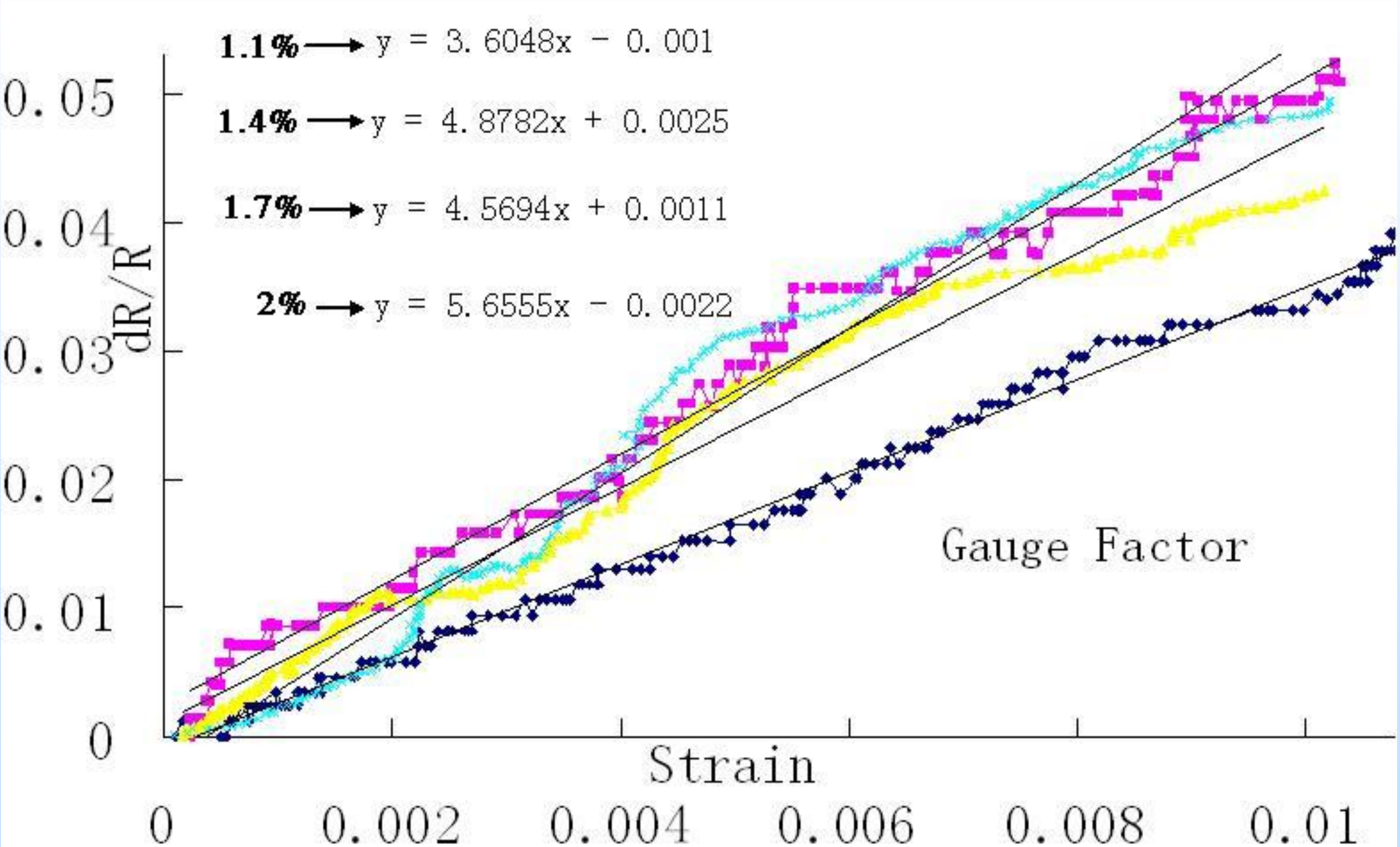
CNT-PI composite characterization

Resistivity Change with Strain for Test Sample without IDT



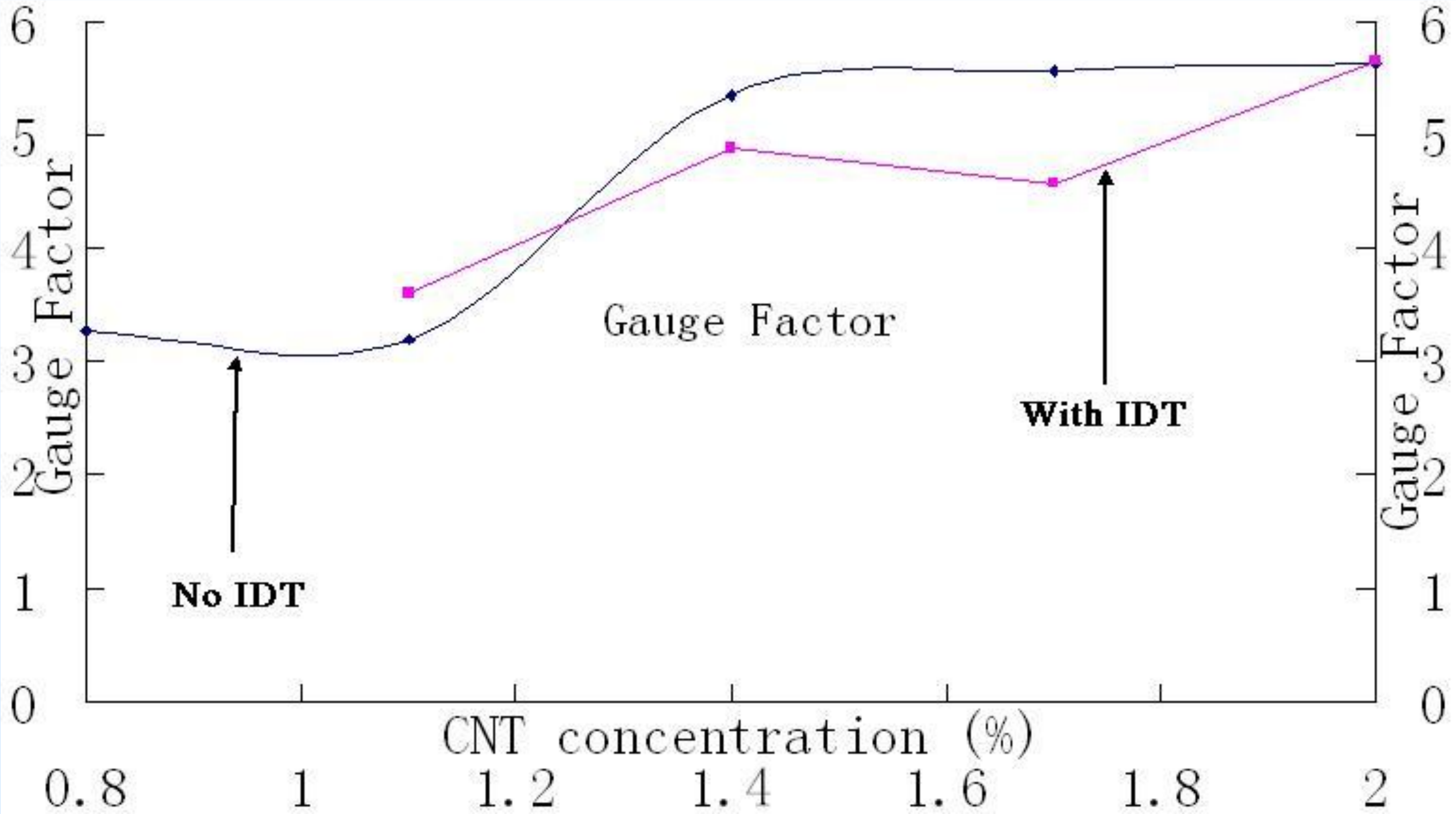
CNT-PI composite characterization

Resistivity Change with Strain for Test Sample with IDT



CNT-PI composite characterization

Gauge Factor



CNT-PI composite characterization

- Nanocomposite Fabricated ✓
- Percolation Threshold Studied ✓
- Temperature Impact Studied ✓
- Strain Impact Studied ✓

CNT-PI composite characterization

- Fabricate and Characterize CO_2 Sensor

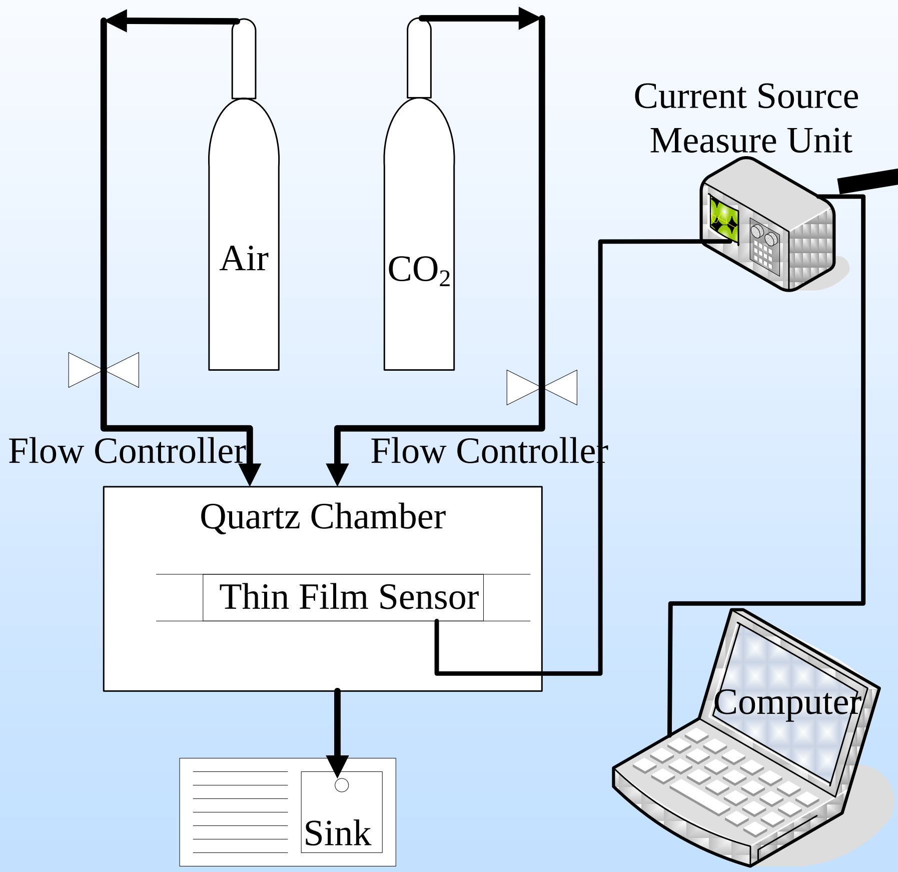
Testing System Construction

CO_2 Sensor Response Assessment

CNT-PI composite CO_2 Response

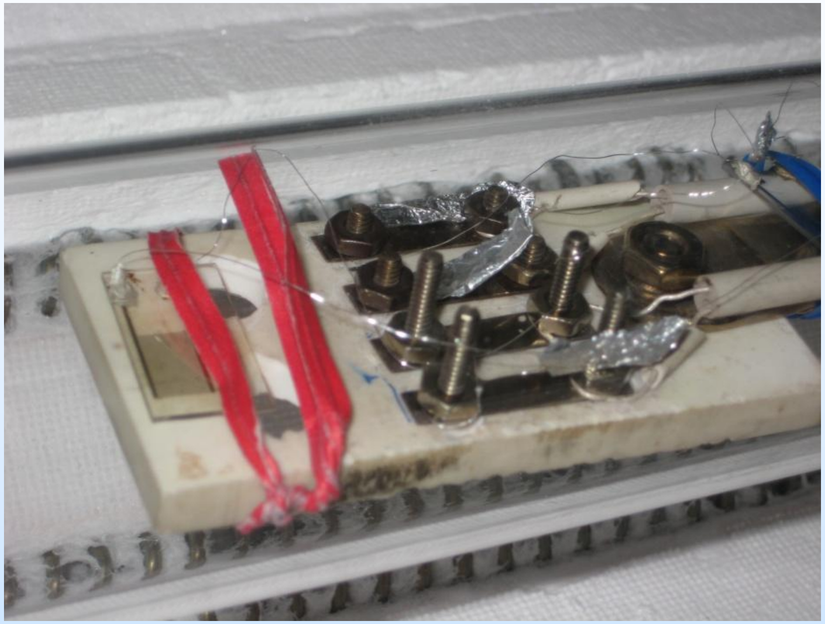
CNT-PI composite characterization

Testing System Construction



CNT-PI composite characterization

Testing System Construction



Tube
- ID 4.8cm
- L 96cm
- V 1.7L

Controller
- 100 sccm
- 50 sccm

→

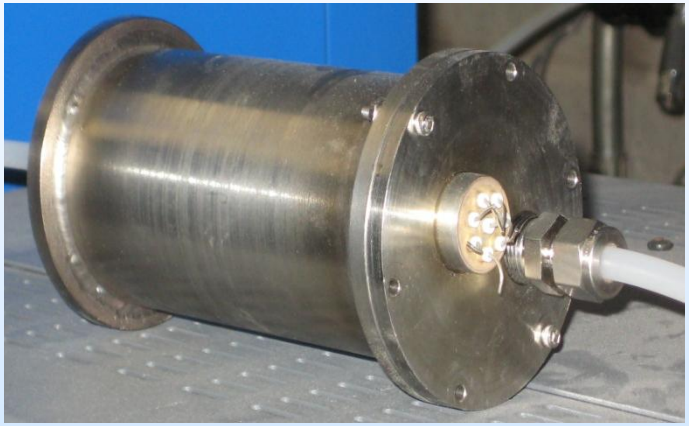
12 min to flush the system with air

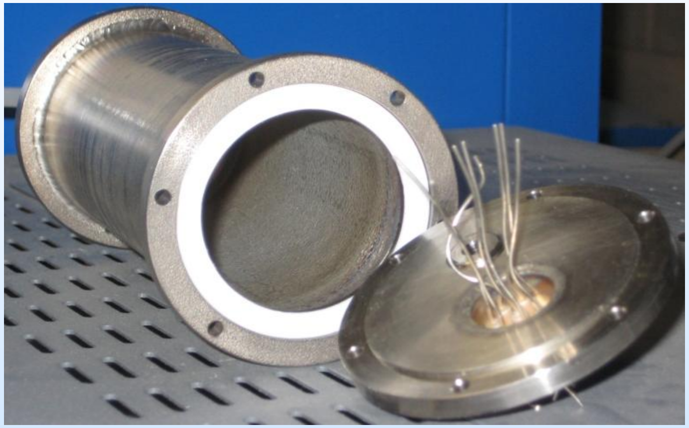
34 min to flush the system with CO_2

CNT-PI composite characterization

Testing System Improvement

1. Smaller Testing Chamber





2. Use Vacuum before every Flush and gas Concentration Change

CNT-PI composite characterization

Theoretical SAW Response

$$\frac{\Delta v}{v_0} \cong -\frac{\beta}{k} = -\frac{K^2}{2}\frac{(\sigma d)^2}{(\sigma d)^2 + {v_0}^2(\varepsilon_0 + \varepsilon_1)^2}$$

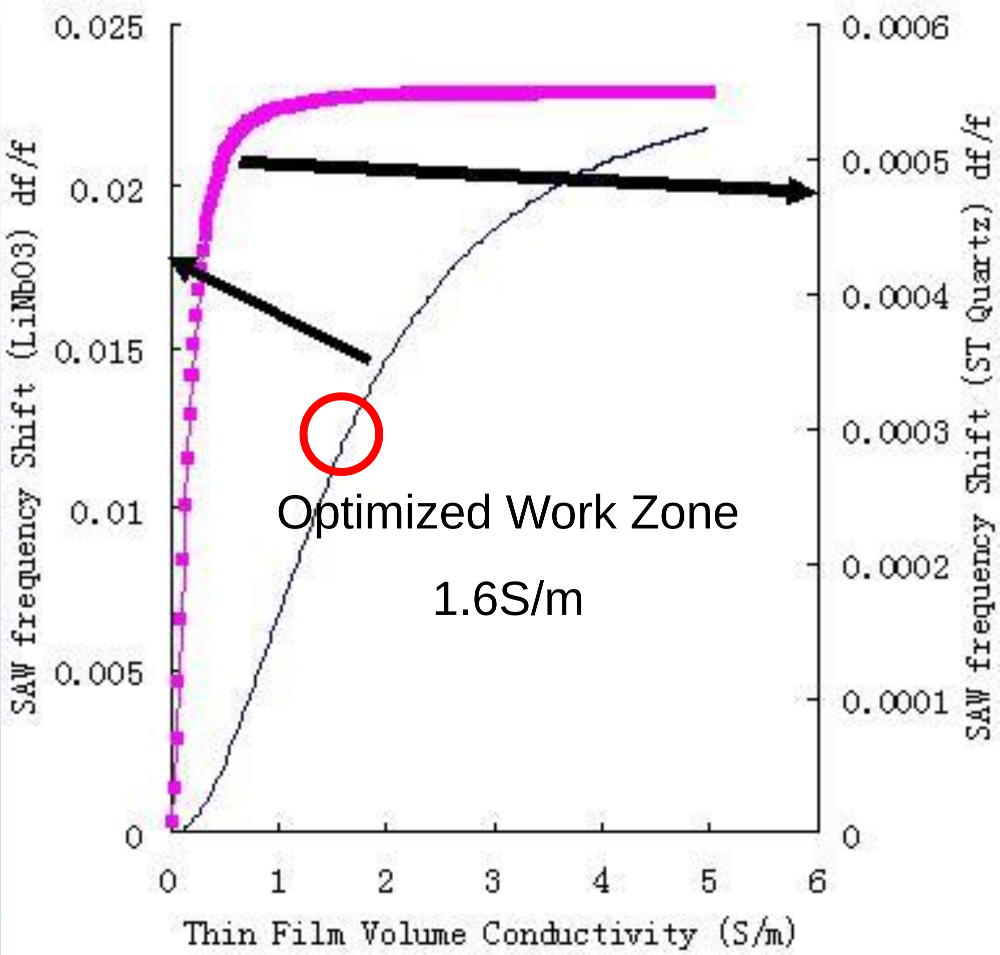
SAW Velocity Change

$$\frac{\alpha}{k} \cong \frac{K^2}{2}\frac{v_0(\varepsilon_0 + \varepsilon_1)\sigma d}{(\sigma d)^2 + {v_0}^2(\varepsilon_0 + \varepsilon_1)^2}$$

SAW Attenuation Change

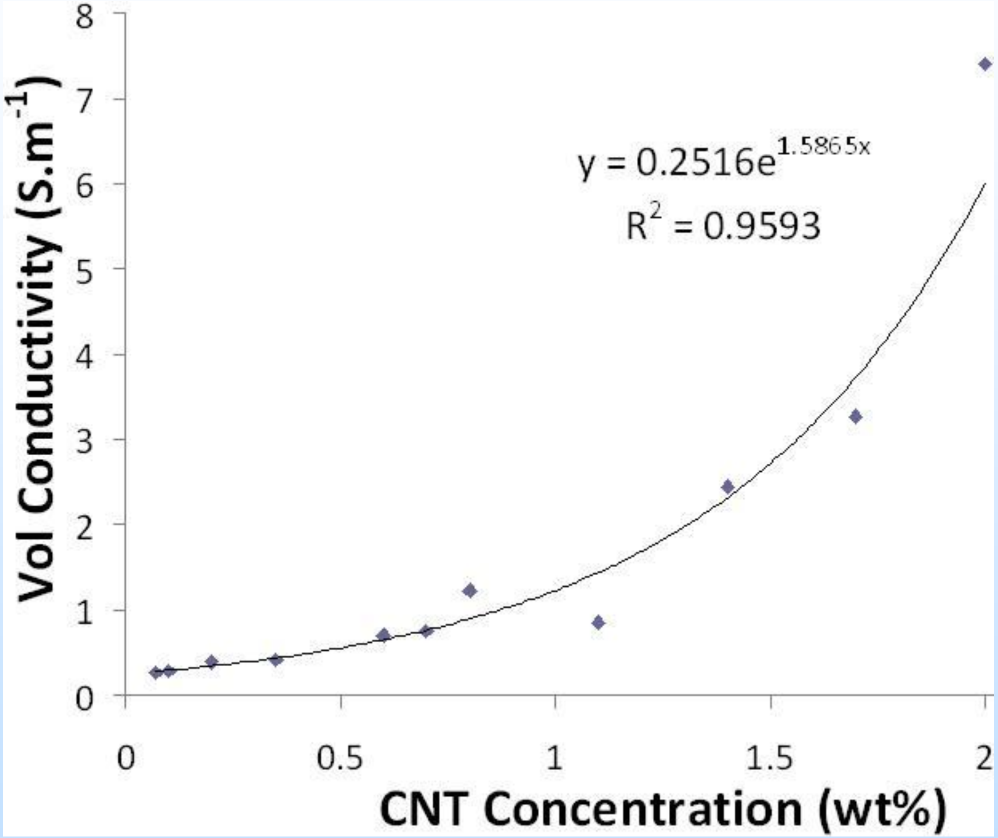
CNT-PI composite characterization

SAW Frequency Change induced by Film Conductivity Change



CNT-PI composite characterization

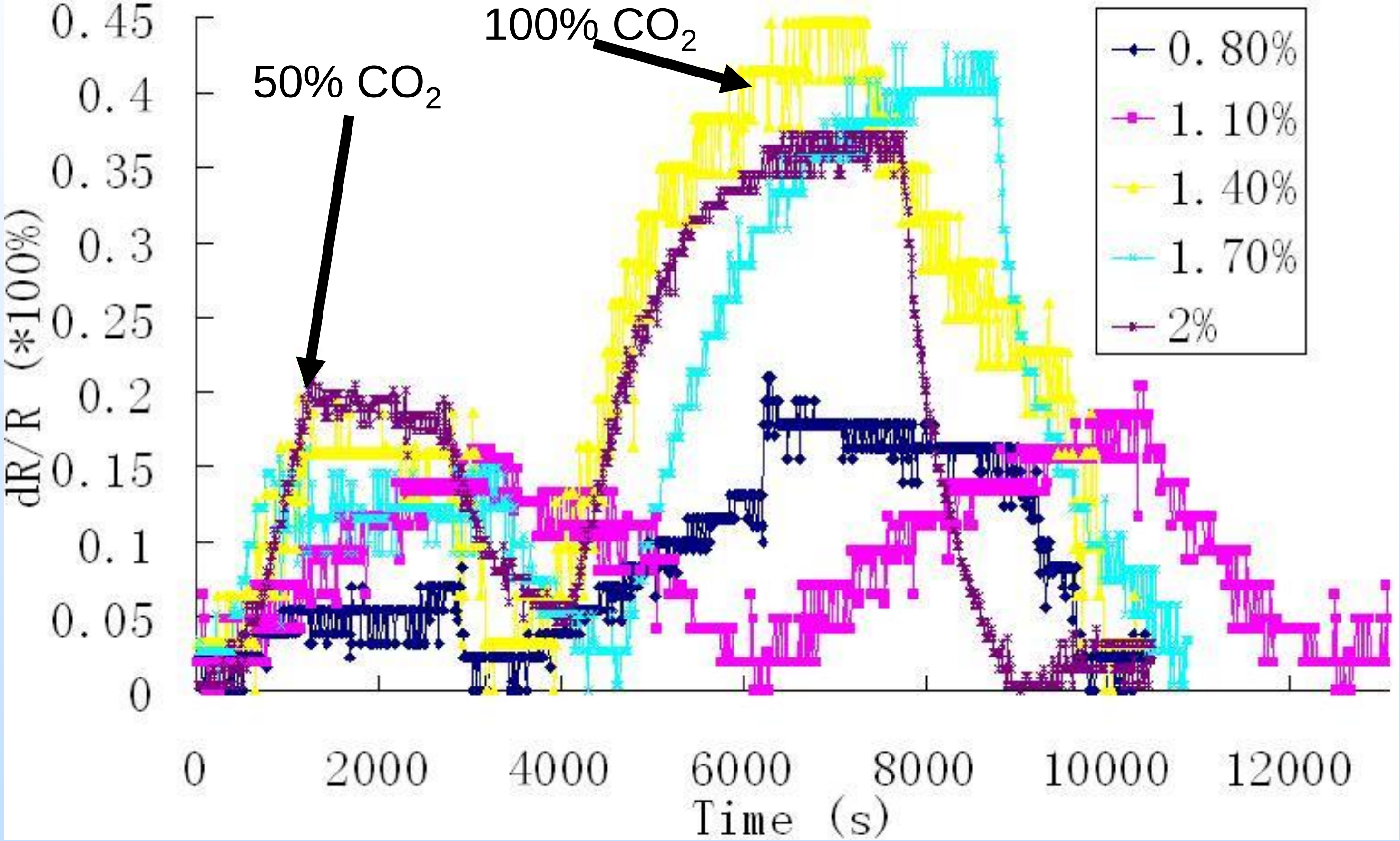
Optimum Film Conductivity = 1.6S/m



Optimum CNT concentration = 1.166wt%

CNT-PI composite characterization

CNT concentration resistance change



Estimated maximum frequency change 500Hz for a 52.63MHz System

CNT-PI composite characterization

- Fabricate and Characterize CO_2 Sensor

Testing System Construction ✓

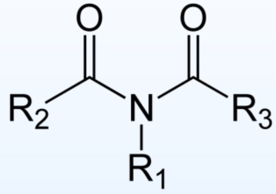
CO_2 Sensor Response Assessment ✓

CNT-PI composite CO_2 Response ✓

CNT-PEI composite characterization

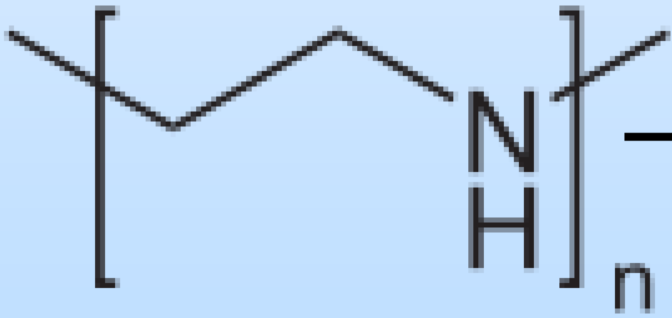
From Chemistry Point of View

- PI not ideal

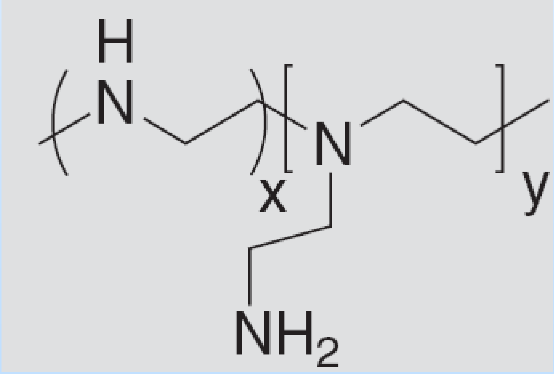


- Primary and Secondary Amines → Carbamates

Polyethylenimine (PEI)

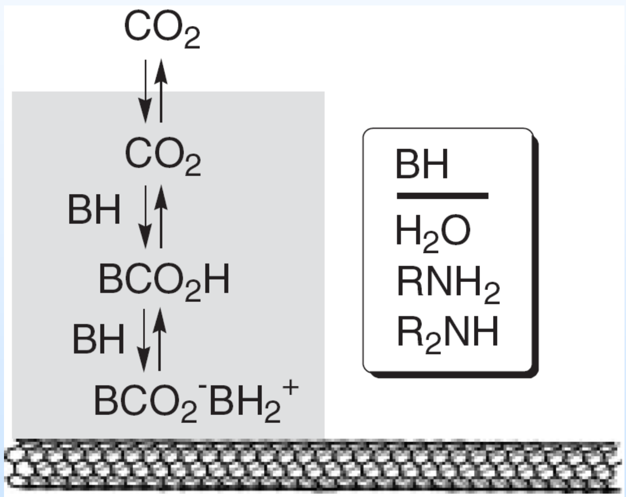


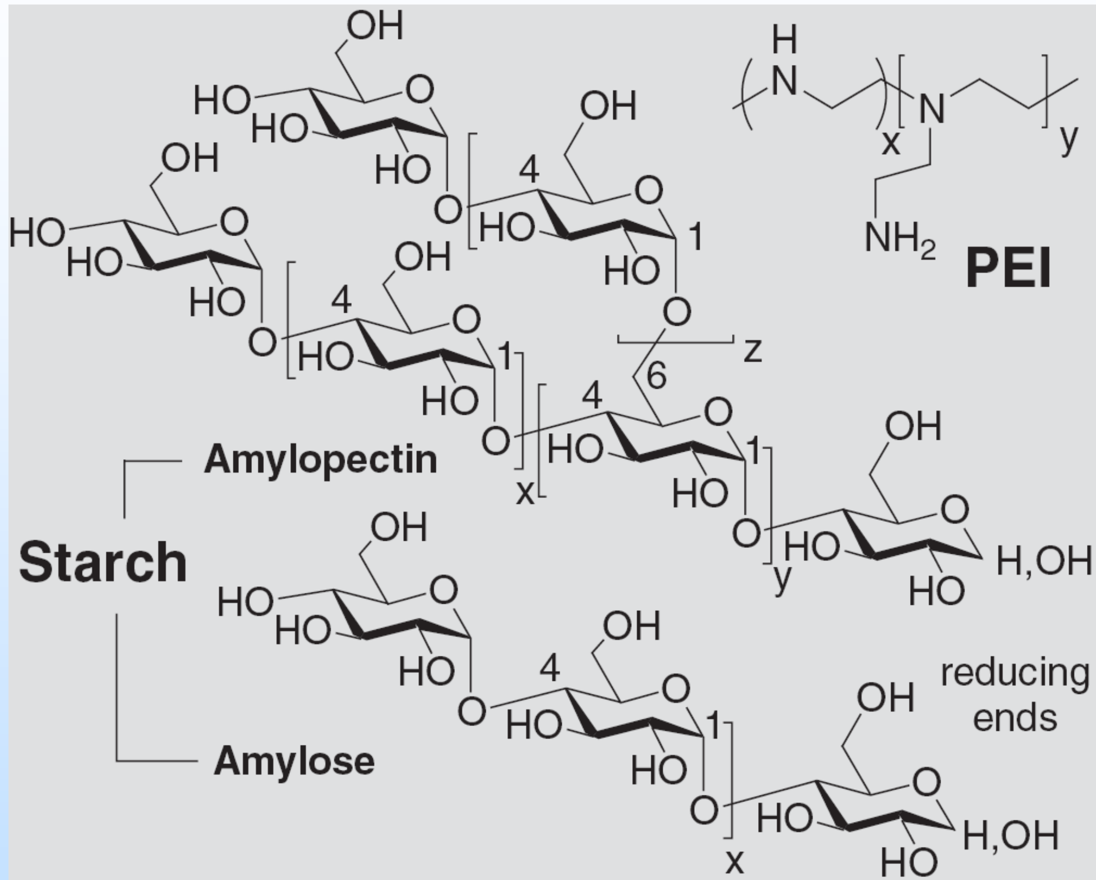
Branched PEI



CNT-PEI composite characterization

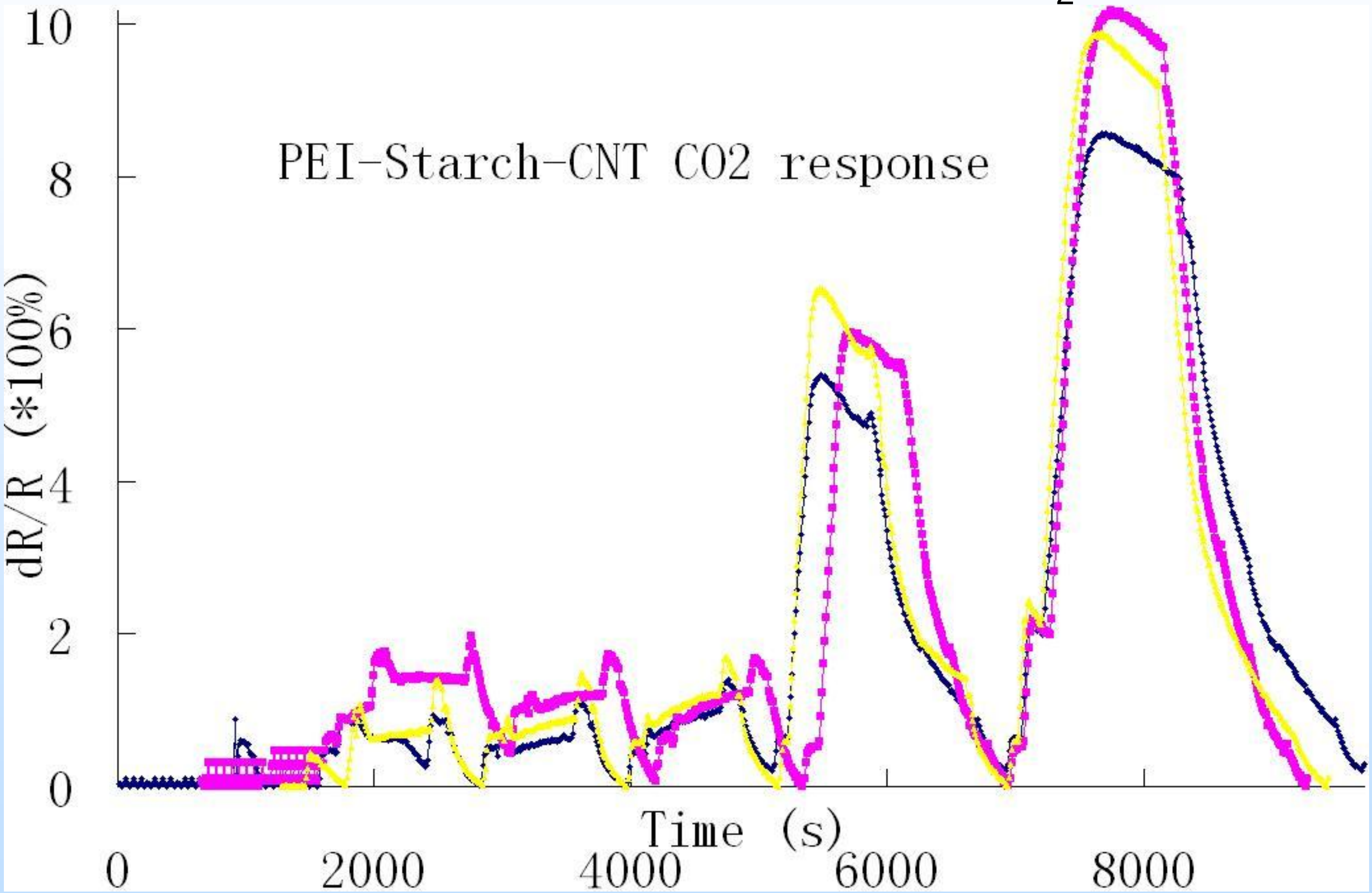
Mechanism





CNT-PEI composite characterization

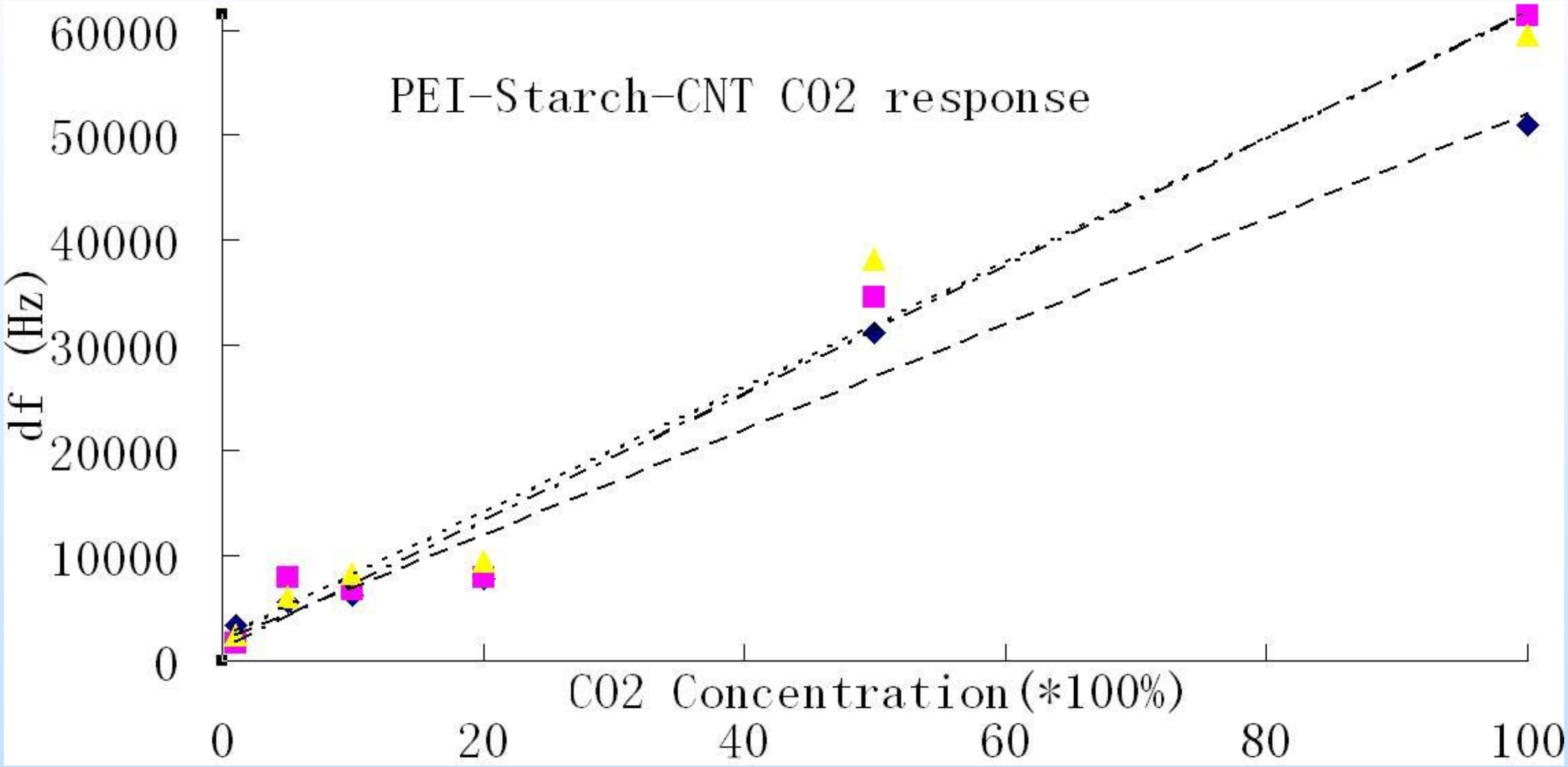
0%, 1%, 5%, 10%, 20%, 50%, 100% CO_2 in the air



20 times increase

CNT-PEI composite characterization

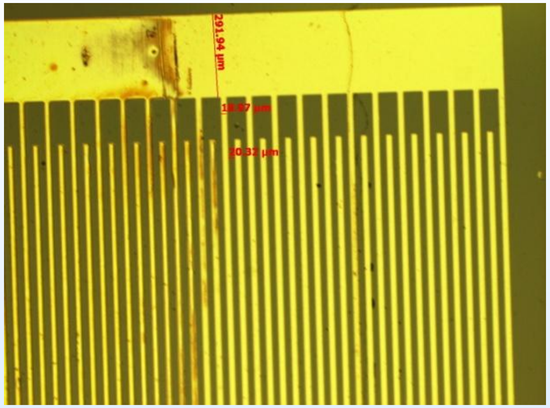
0%, 1%, 5%, 10%, 20%, 50%, 100% CO_2 in the air



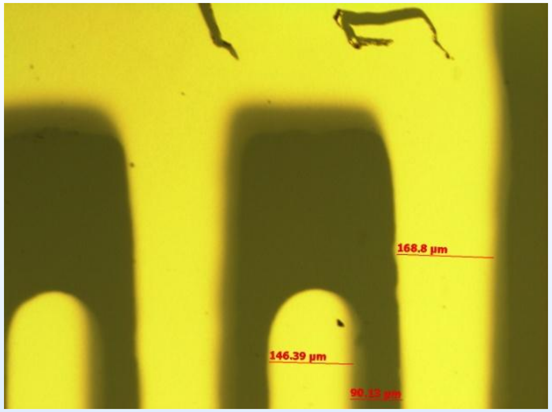
0.8% Frequency Change with 100% CO_2

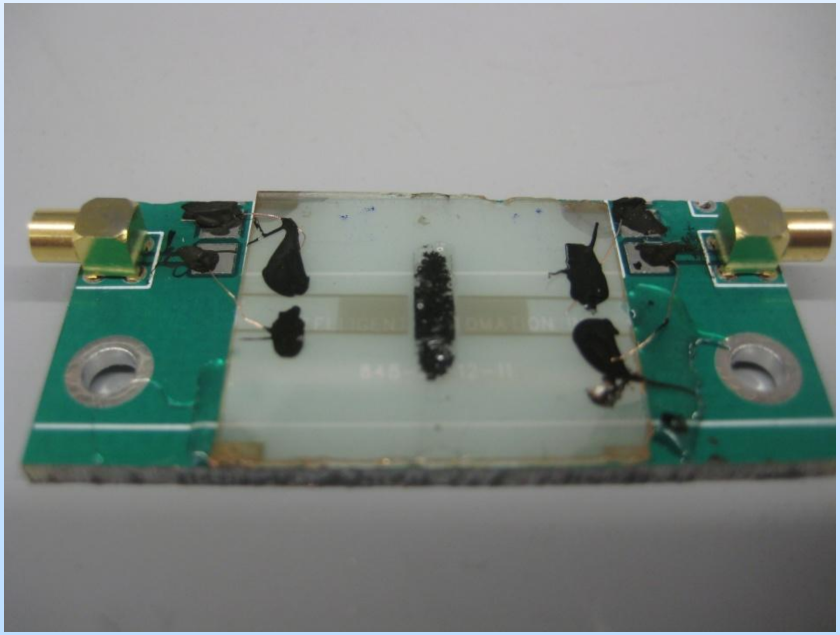
SAW CO_2 sensor characterization

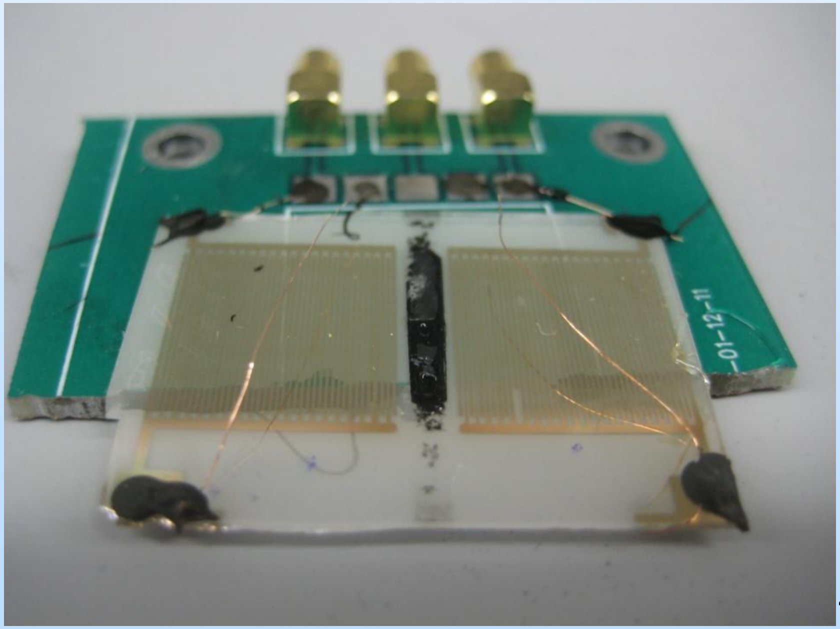
60.6MHz SAW by Lithography



6.45MHz SAW by Shadow Mask

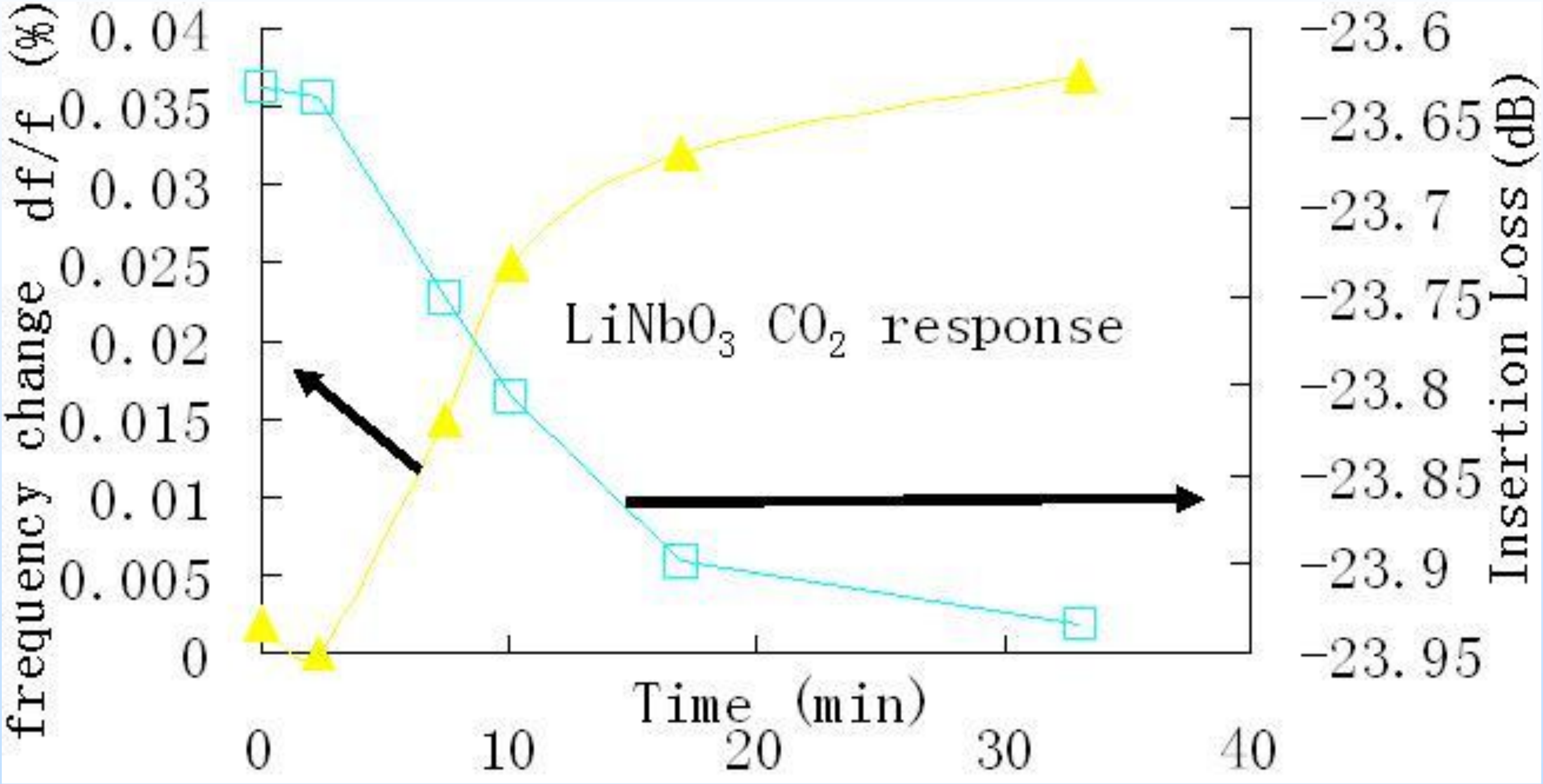






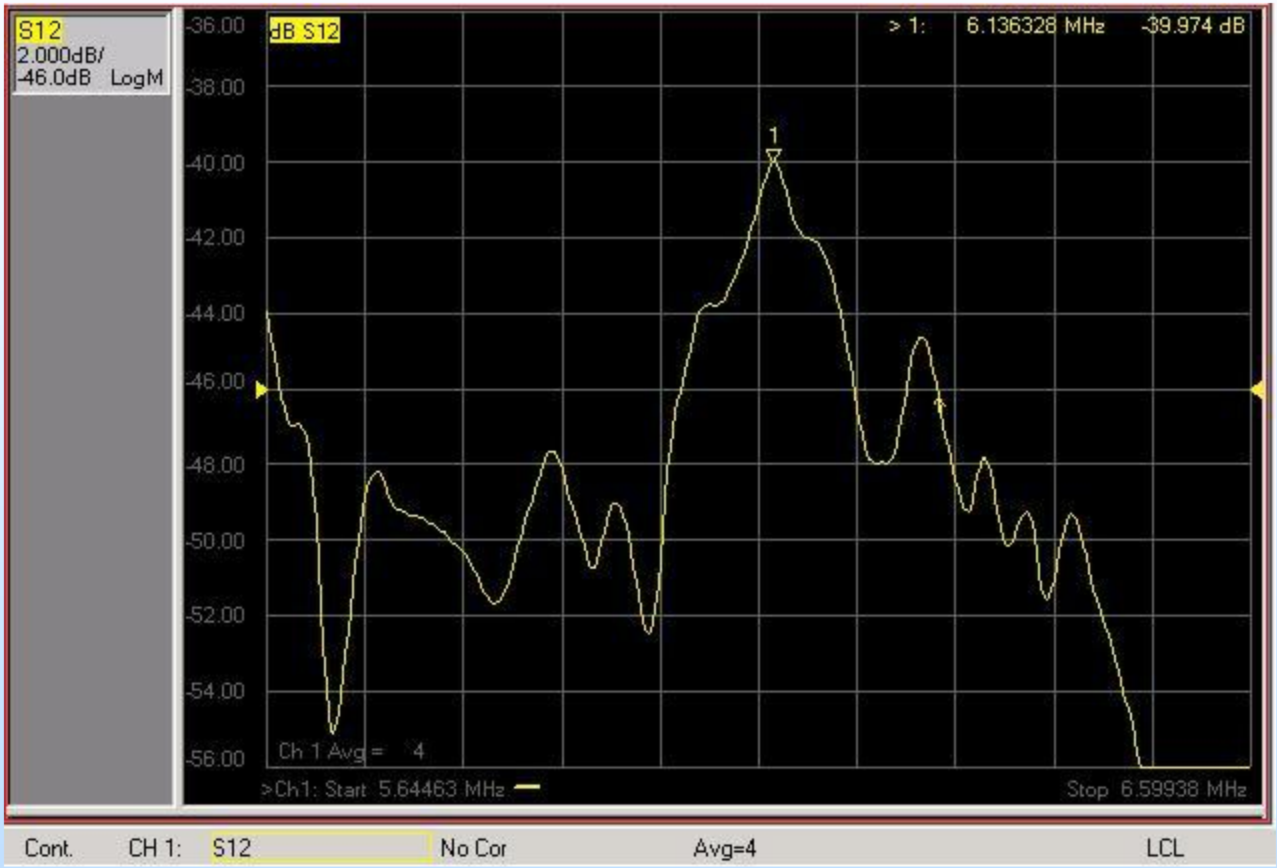
SAW CO_2 sensor characterization

SAW response for 100% CO_2



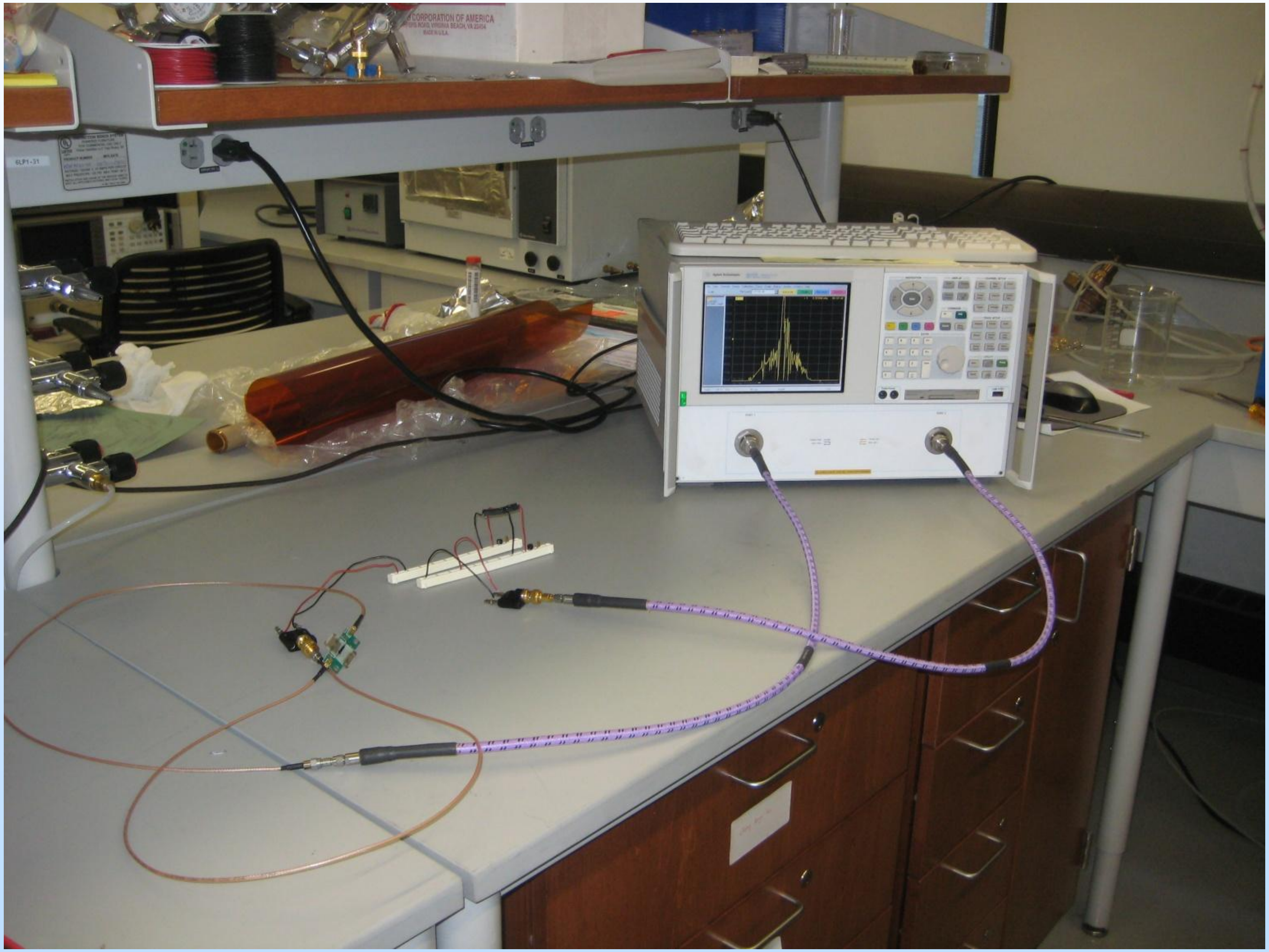
Wireless module characterization

Basic SAW response without antenna



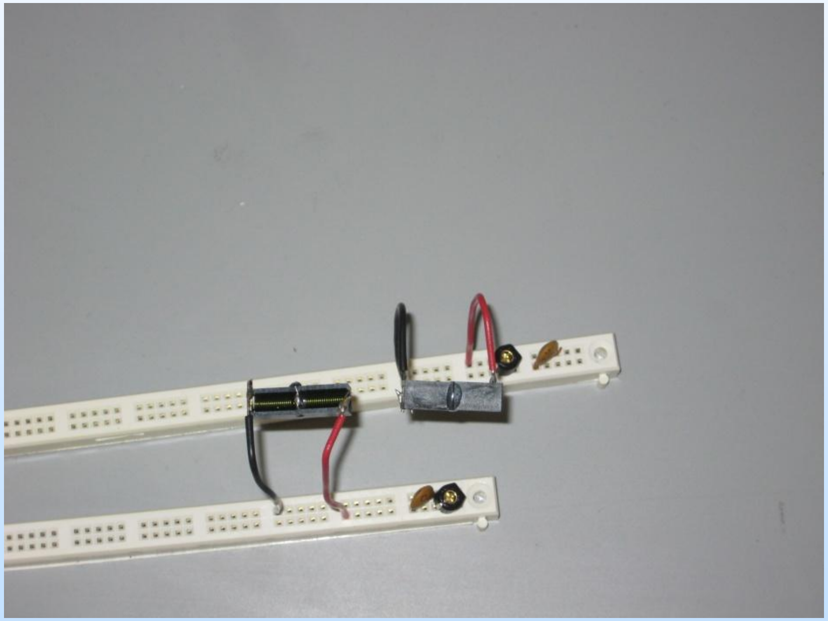
Wireless module characterization

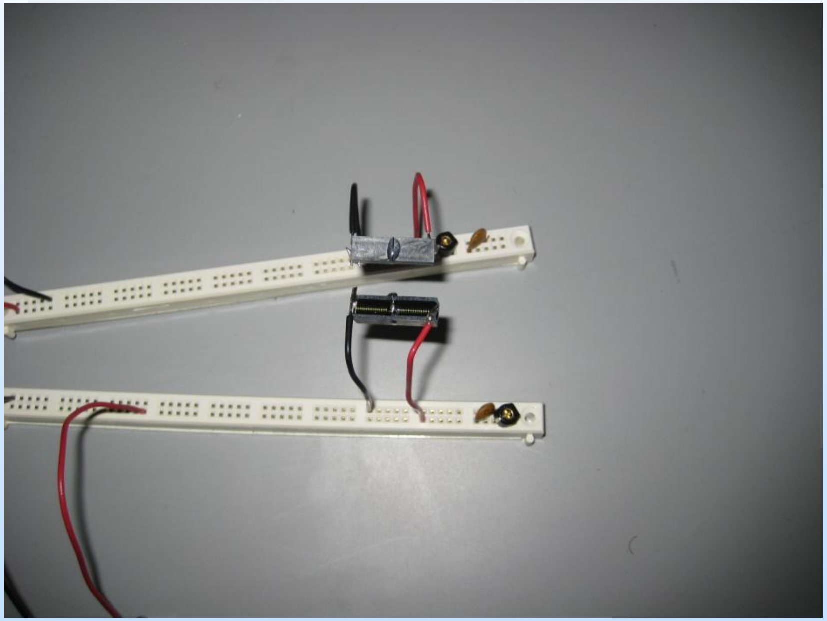
Testing setup for SAW sensor with antenna



Wireless module characterization

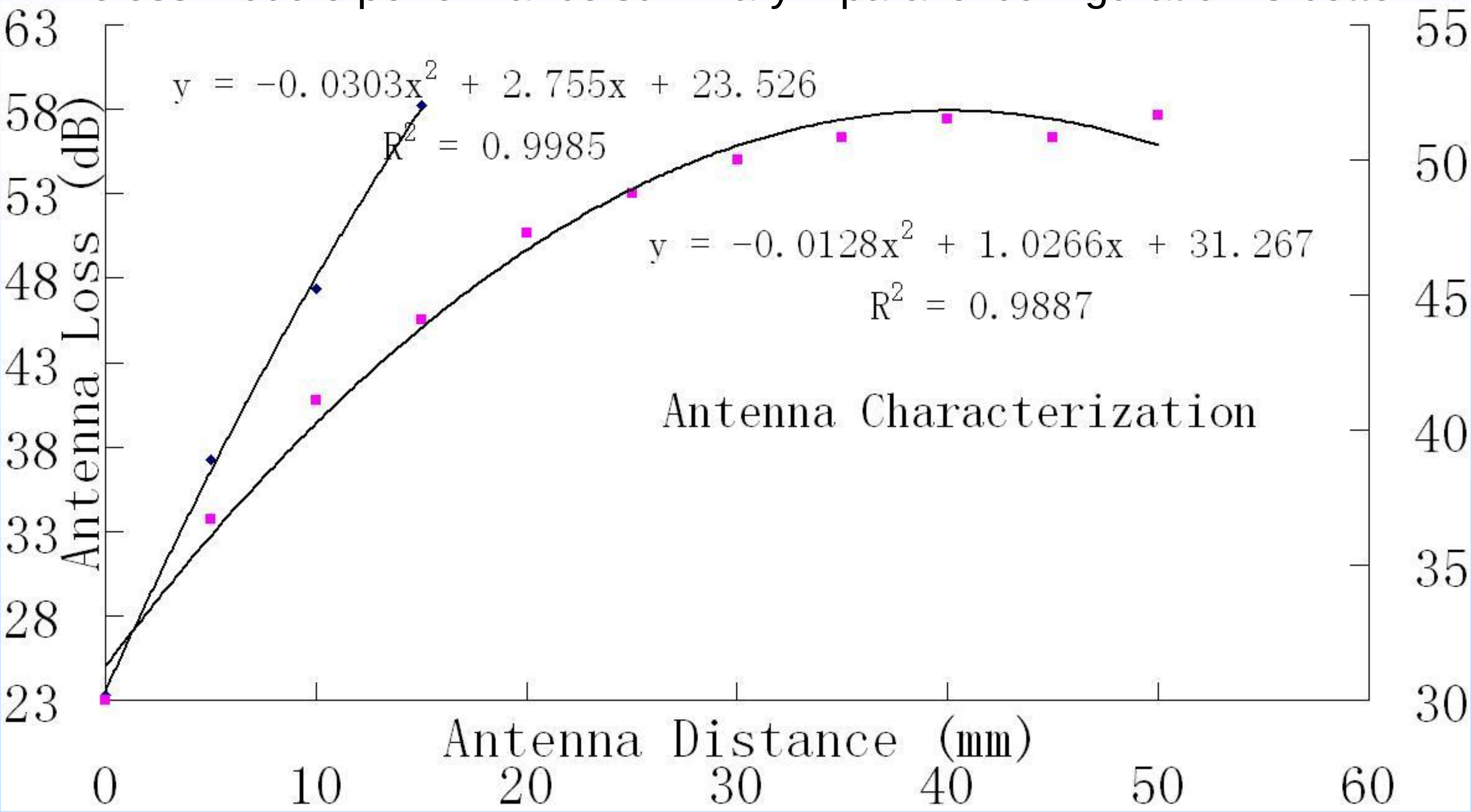
Close look for different wireless working pattern





Wireless module characterization

Wireless module performance summary – parallel configuration is better



SAW CO_2 Sensor Development and Improvement

Gas Response Improvement through CNT-PEI

CO_2 Sensor System Response

Wireless Module Characterization

Technical Status

- Focus the remaining slides, logically walking through the project. Focus on telling the story of your project and highlighting the key points as described in the Presentation Guidelines
- When providing graphs or a table of results from testing or systems analyses, also indicate the baseline or targets that need to be met in order to achieve the project and program goals.

Accomplishments to Date

- SAW sensor design, fabrication and testing process development completed
- Impact of temperature and strain on polymer composite study completed
- Polymer composite response to CO_2 investigation and improvement completed
- SAW CO_2 sensor performance assessment and analysis completed
- Wireless module development completed

Summary

– CNT-PEI composite is expected to produce ample room for detection improvement

– $LiNbO_3$ is the best choice as SAW substrate, not only for its potential in wireless sensing, but also for its highest yield in combination with current choice of polymer composite

– Wireless can be improved by increasing operation frequency and aligning antenna orientation

– Other parameters that can impact sensor performance, such as humidity and other gases like NH_3 and H_2, will be considered and evaluated

Appendix

Project Milestones

Milestone	Planned Completion Date	Actual Completion Date
Fundamental study of acoustic wave CO_2 gas sensors, including design, fabrication, characterization of acoustic wave sensors	6/30/2010	6/30/2010
Fabrication and characterization of highly CO_2 sensitive carbon nanotube-polymer thin coating layer	12/31/2010	12/31/2010
Design and fabrication of CO_2 sensor testing module	6/30/2011	2/18/2011
Evaluation of acoustic wave gas sensors using CO_2 selective and sensitive nanocomposite thin film interface	12/31/2011	12/31/2011
Fabrication and evaluation of passive wireless SAW CO_2 sensors	6/30/2012	6/30/2012
Development of sensor prototype and monitoring system for field monitoring CO_2 emission	12/31/2012	9/30/2012 (expected)

Organization Chart

- Department of Mechanical Engineering, University of Pittsburgh.
  - Qing-Ming Wang, PI
  - Minking K. Chyu, Co-PI
  - Yizhong Wang, Graduate Student
  - Lifeng Qin, Postdoctoral Researcher

Gantt Chart

	Year 1				Year 2				Year 3			
	Q1	Q2	Q3	Q4	Q1	Q2	Q3	Q4	Q1	Q2	Q3	Q4
Design, fabrication of acoustic wave sensors												
Development of CO_2 sensitive CNTs/polymer nanocomposites thin films												
Annual Report 1												
Design and fabrication of CO_2 gas sensor testing module												
Fabrication and evaluation of bulk acoustic wave CO_2 gas sensors												
Annual Report 2												
Fabrication and evaluation of passive wireless SAW CO_2 sensors												
Development of sensor prototype and monitoring system for field monitoring CO_2 emission at NETL												
Final Report												

Bibliography

- Journal, multiple authors:
  - Yizhong Wang, Zheng Li, Lifeng Qin, Minking K. Chyu and Qing-Ming Wang, 2012, Theoretical and Experimental Studies of a Surface Acoustic Wave Flow Sensor. IEEE Transactions on Ultrasonics, Ferroelectrics, and Frequency Control, v. 59, p. 481-489, available at: http://ieeexplore.ieee.org/stamp/stamp.jsp?arnumber=06174194.
  - Yizhong Wang, Ying Wang, Yongzheng Fang, Minking K. Chyu and Qing-Ming Wang, 2012, Fabrication and Characterization of Carbon Nanotube-Polyimide Compiste Based High Temperature Flexible Thin Film Piezoresistive Strain Sensor, Journal of Applied Physics, Accepted.
- Publication:
  - Yizhong Wang, Zheng Li, Lifeng Qin, Minking K. Chyu and Qing-Ming Wang, 2011, Surface Acoustic Wave Flow Sensor, 2011 Joint Conference of the IEEE International Frequency Control and the European Frequency and Time Forum (FCS), p. 1-4, available at http://ieeexplore.ieee.org/xpls/abs_all.jsp?arnumber=5977735.