



Structural Health Monitoring with Carbon Nanotube and Graphene Sensors

Nondestructive Evaluation Sciences Branch

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ASNT Research Symposium 2020

3/25/2020



Overview

- Background
- Sensory Capabilities of Carbon Nanostructures (*CNT* and Graphene)
- Structural CNT composites with Strength to Mass ratio competitive with SOA carbon fiber composites
- Direct Printing of Graphene and CNT sensors on Additively Manufactured components
- Preliminary Test Results
- Future Directions
- Summary



Carbon Nanotubes and Graphene

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Potential

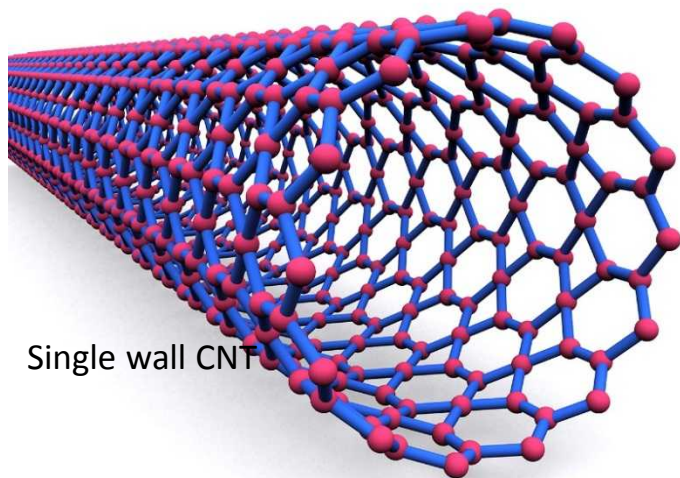
- Superior mechanical properties at the nanoscale
- Multifunctionality: e.g., electrical and thermal conductivity

Challenges

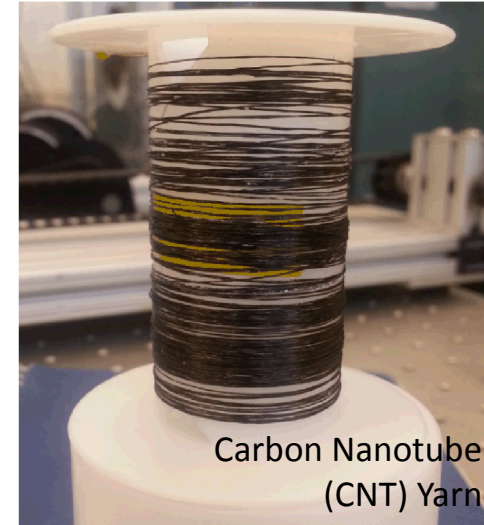
- Scaling up while retaining material properties
- Handling difficulties

Advantage

- Can be used in 3D printable forms



Single wall CNT



Carbon Nanotube
(CNT) Yarn



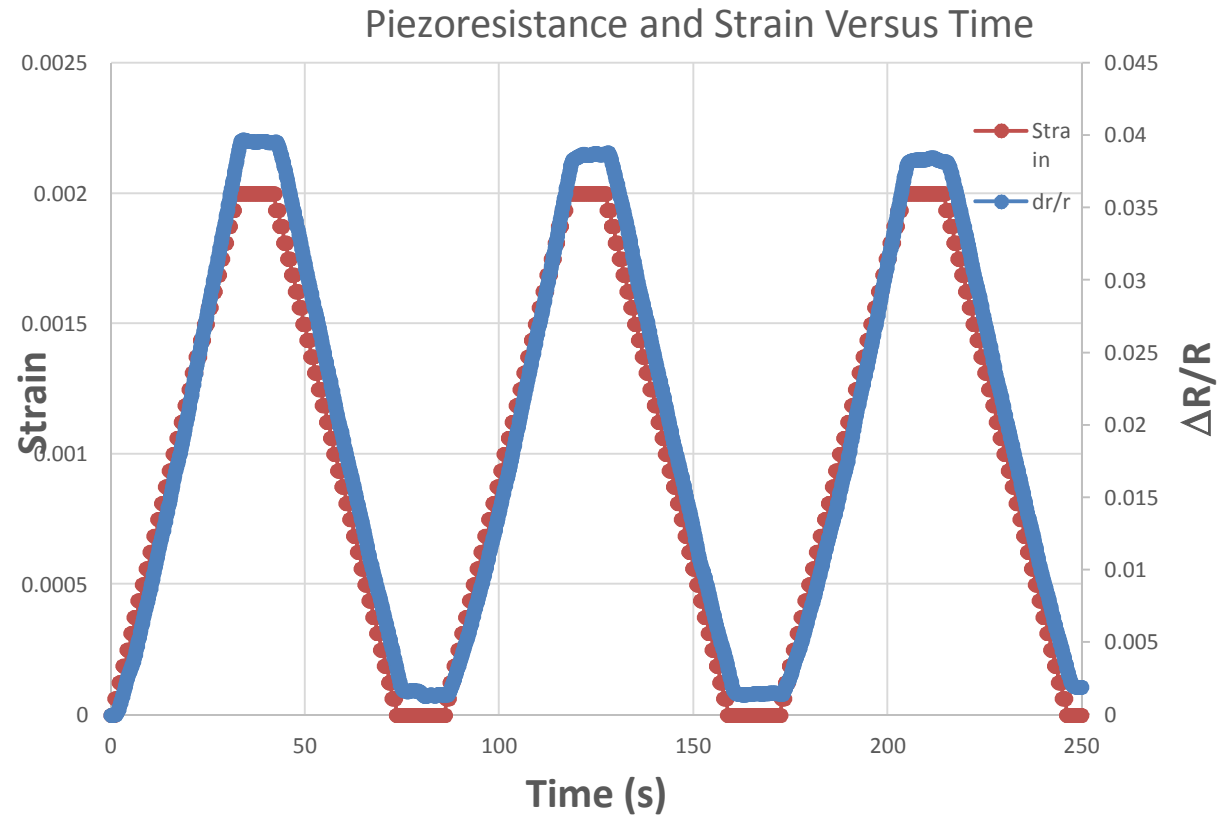
Graphene Oxide
(GO)



Single Strand CNT Fiber, 21 Micron Diameter

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31.6 microamp bias current



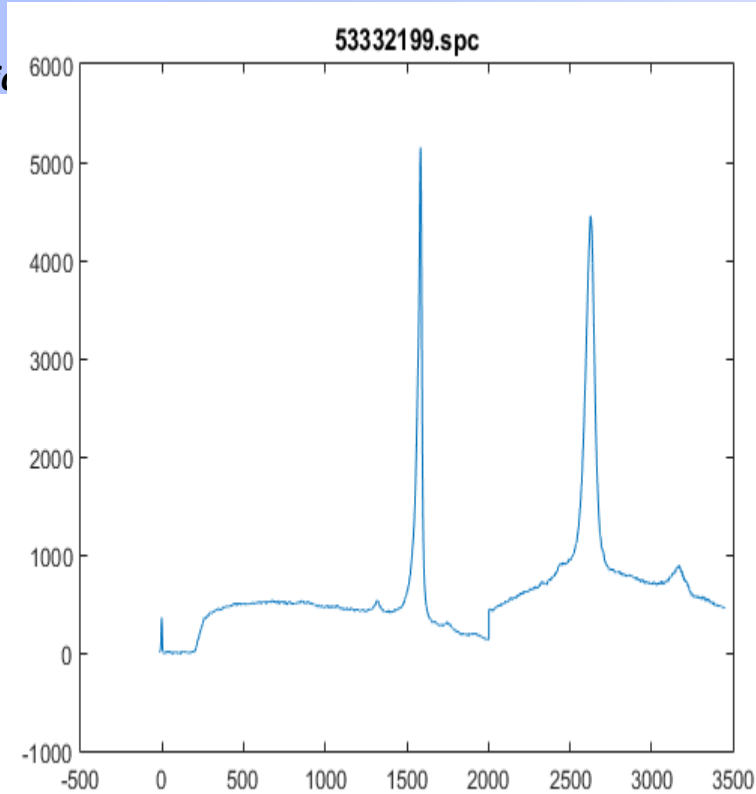
$R_0 = 66$ ohms, Gauge Length = 1"
Gauge Factor = 19 (at 2 millistrain)

Resistivity = 9×10^{-5} ohm-cm
Conductivity = 1.1×10^4 S/cm

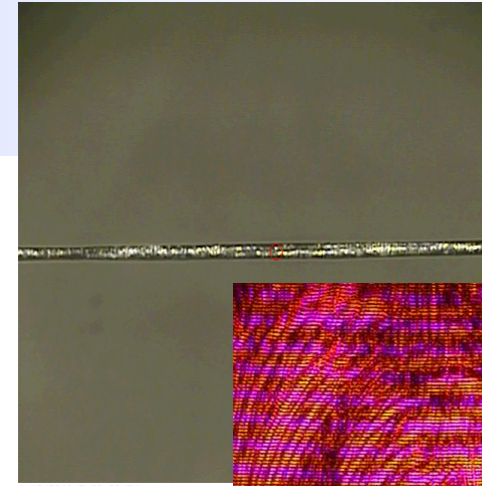


Raman Spectroscopy

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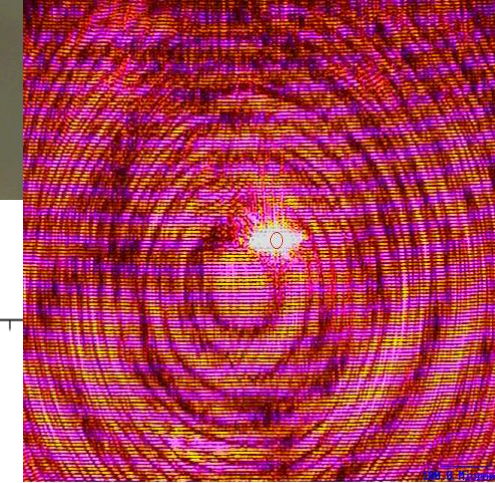
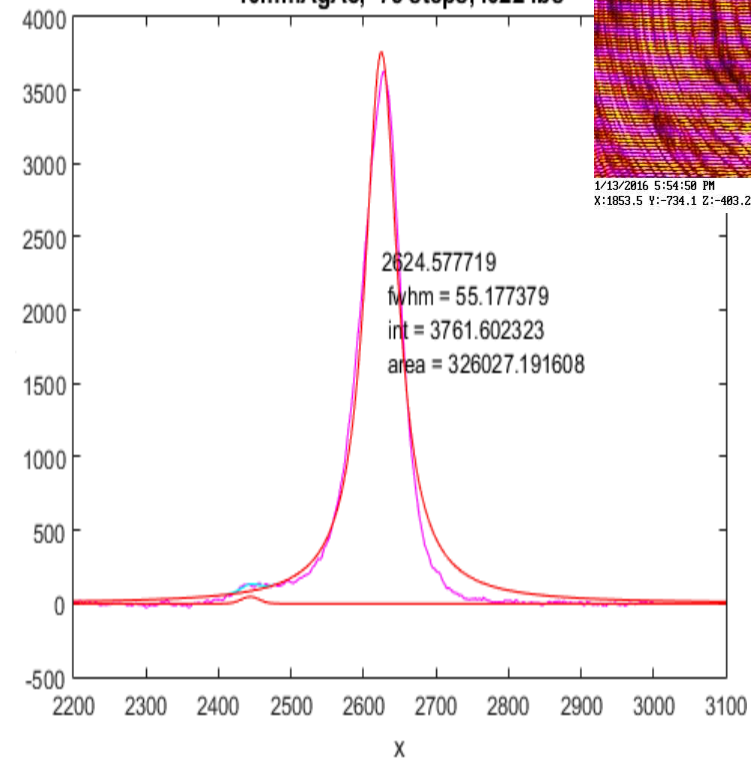


632.8 nm
5.5 mW
5 sec Exposure Length x 5
accumulations

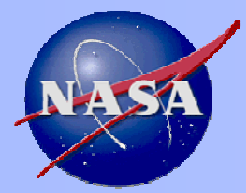


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X:5.3 Y:-14.3 Z:38.9

53332199.spc
10mmAgAc, 75 steps, .022 lbs

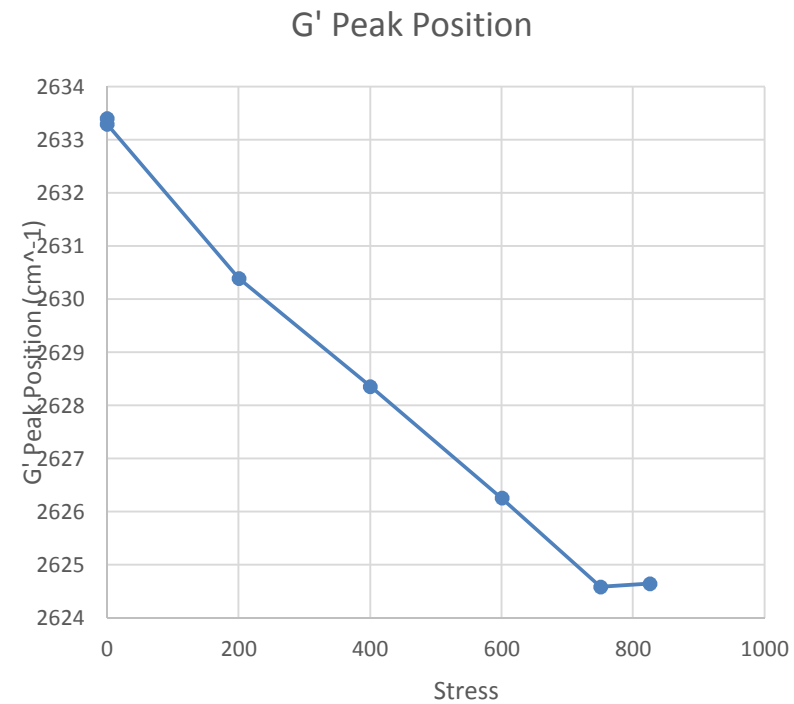
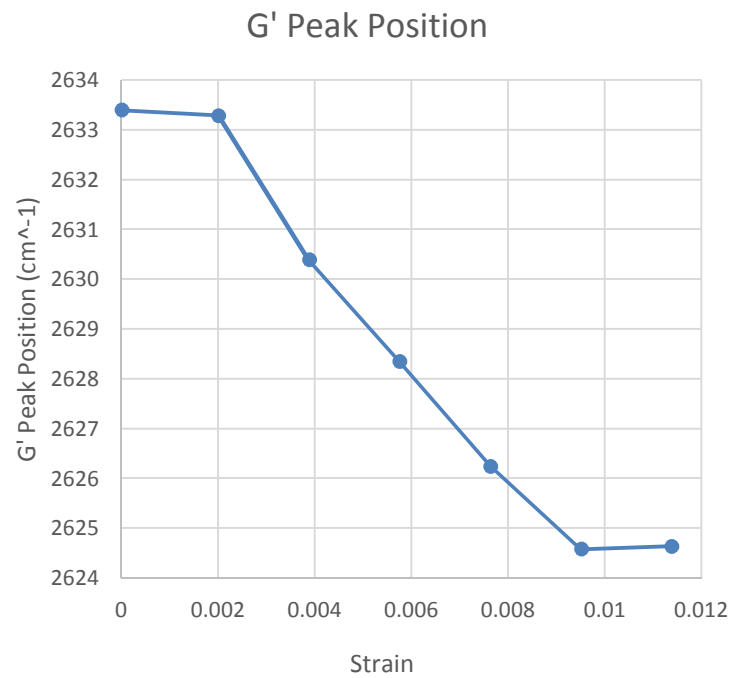


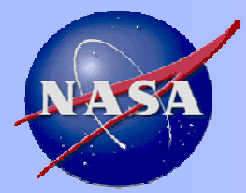
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X:1853.5 Y:-734.1 Z:-403.2



G' Peak Position Versus Stress and Strain

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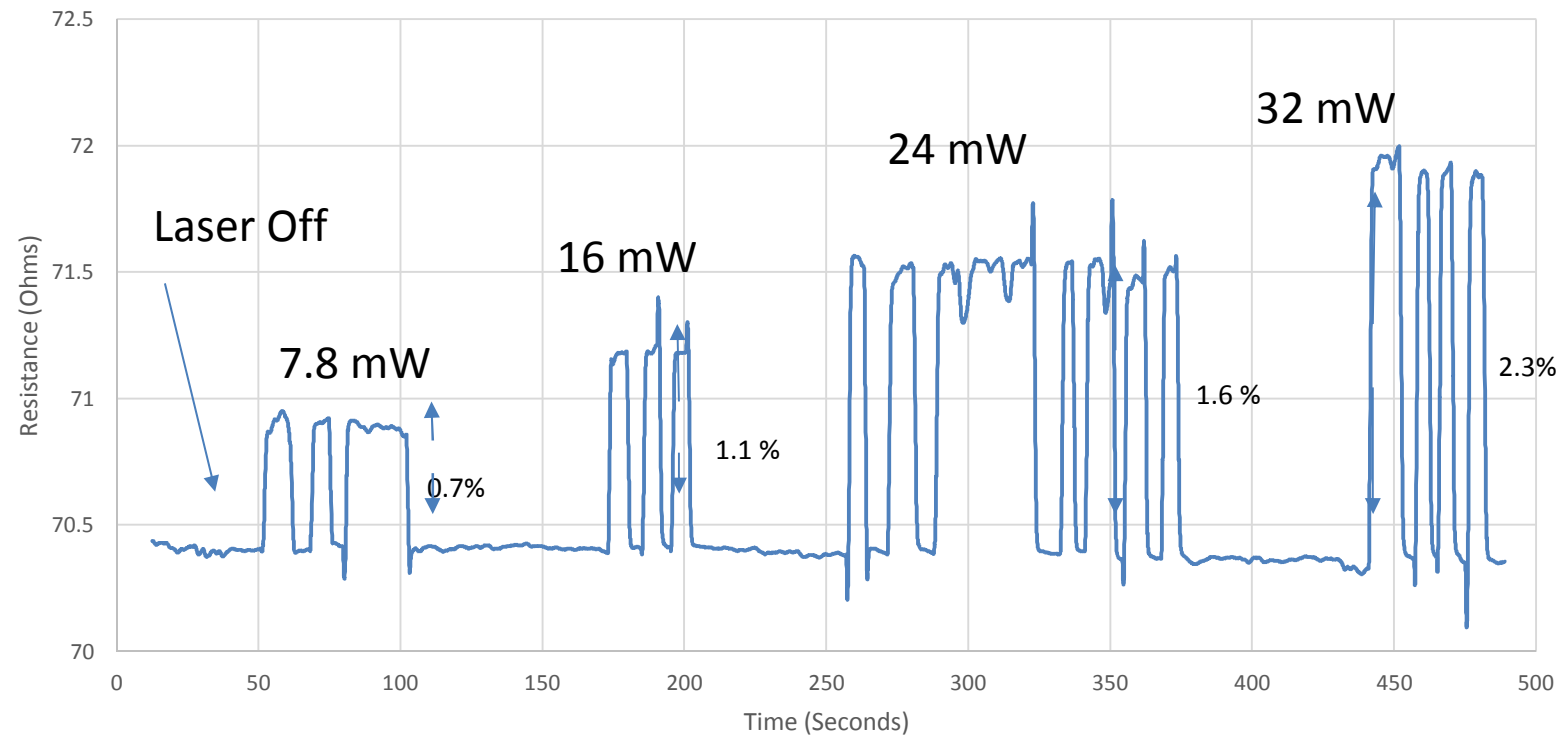




PhotoResponse of CNT Fiber

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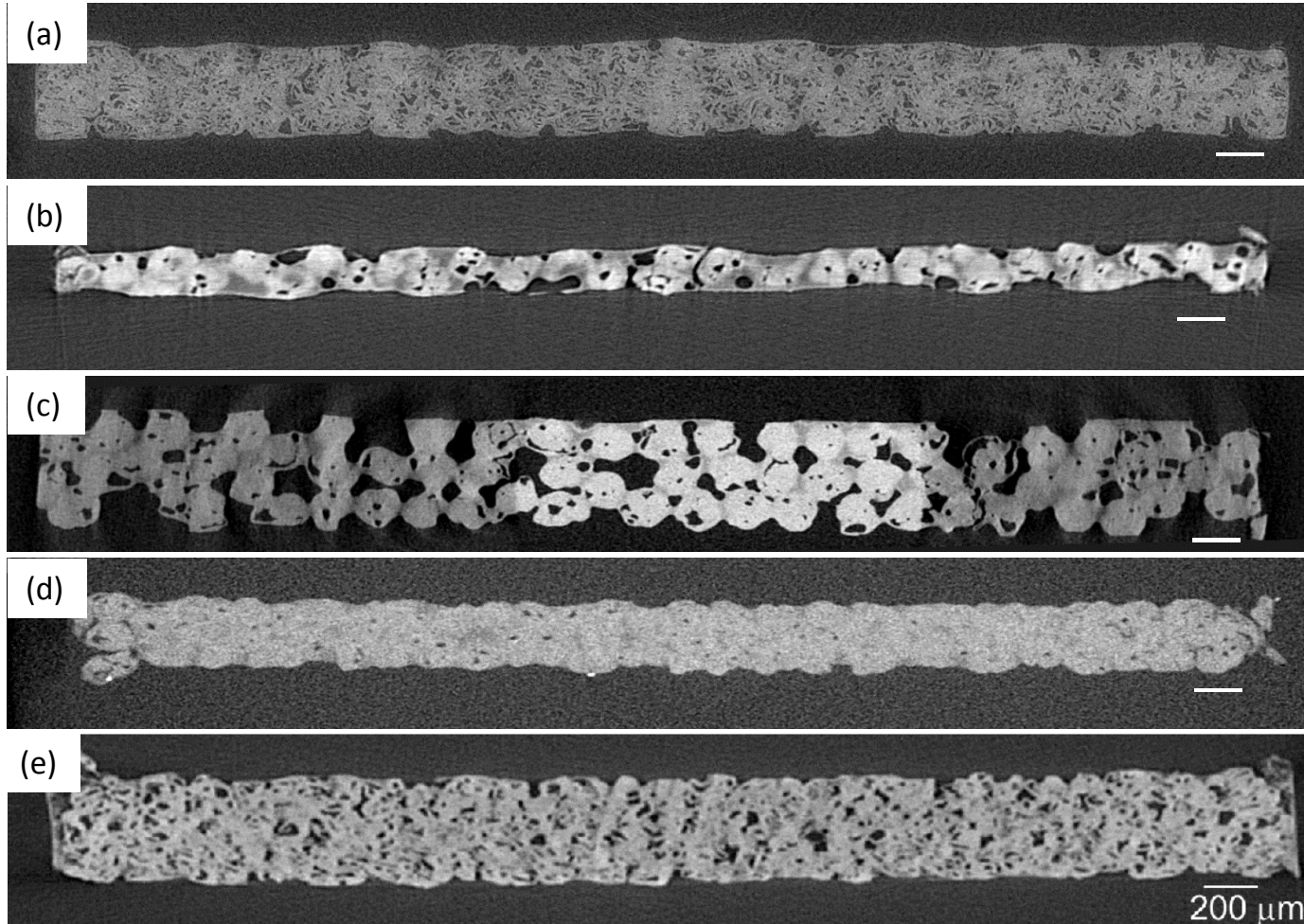
PhotoResponse of Suspended SWCNT Filament to 785nm Laser (~ 100 micron x 20 micron exposure area)



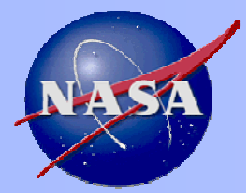


CNT Composites

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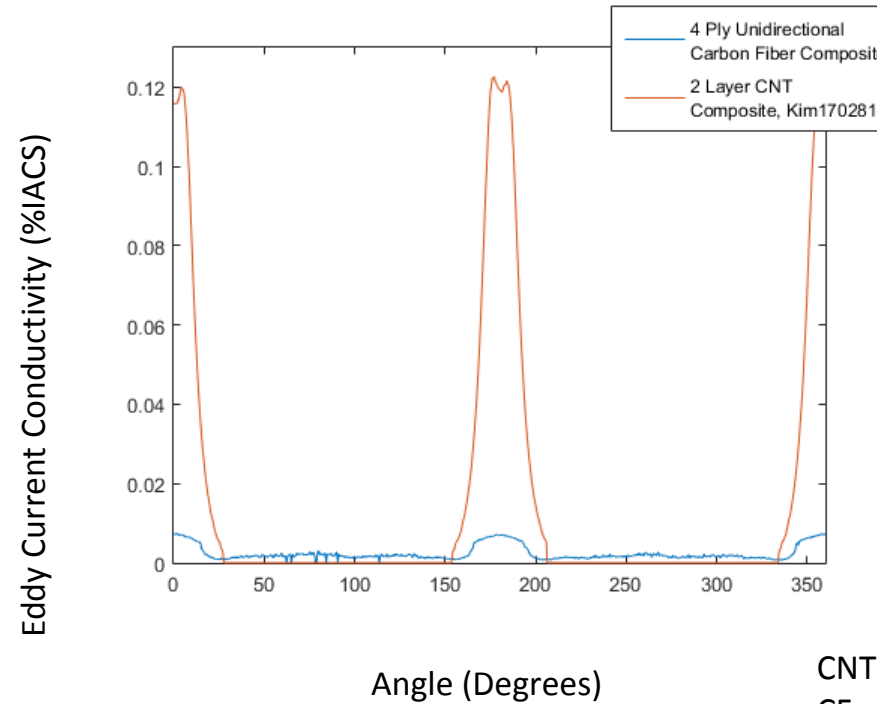


Cross sectional X-ray CT images of unidirectional CNT yarn/API-60 composites prepared at various processing conditions such as (a) 2 layers, 2.2 lbf in winding tension, and 200 psi in cure pressure, (b) by using prepreg, 2 layer lay up, and 200 psi in cure pressure, and (c) 2 layers, 3.0 lbf in winding tension, and autoclave cure. (d) Cross sectional X-ray CT image of unidirectional CNT yarn/cyanate ester composites prepared with 2 layers of CNT yarn at 3.0 lbf of winding tension and 1500 psi of cure pressure. (e) Cross sectional X-ray CT image of unidirectional CNT yarn/cyanate ester composites prepared with 2 layers of 2 ply CNT yarn at 1.0 lbf of winding tension and 300 psi of cure pressure.



Eddy Current Conductivity of CNT and Carbon Fiber Composite

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$$J_x = J_0 e^{-(x/\delta)}$$

$$\delta = 1 / \sqrt{\pi f \mu \sigma}$$

$$\delta(f = 10\text{MHz}, \sigma = 0.12\% \text{IACS}) = .603 \text{ mm}$$

$$\delta(f = 10\text{MHz}, \sigma = 0.007\% \text{IACS}) = 2.5 \text{ mm}$$

CNT part thickness = 0.419 mm

CF part thickness = 0.841 mm

Directional conductivity measured with inductive probe at 10 MHz

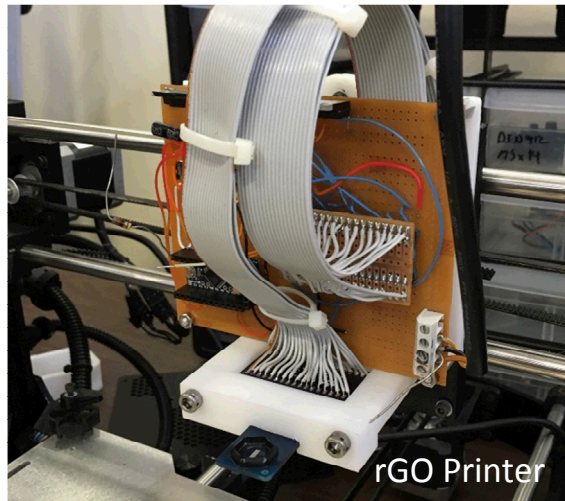
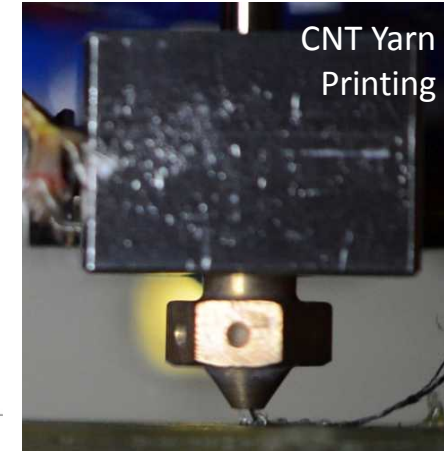
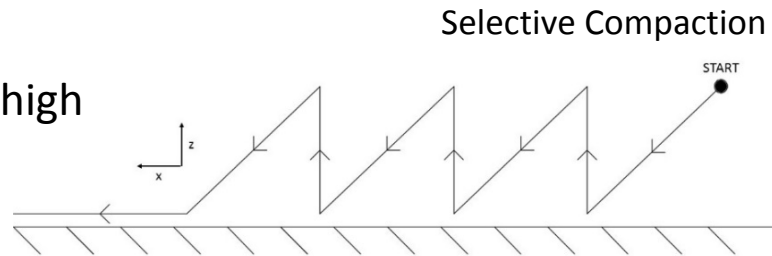


CNT and Reduced GO Printing

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CNT Yarn Printing

- High density CNT yarn coated with polymer
- Selective compaction
- CNT compliance allows for high fidelity printing



Reduced Graphene Oxide (rGO) Printing

- Developed cooperatively between NASA and MIT
- GO/water suspension
- Spray deposition
- Reduced via heat and ascorbic acid

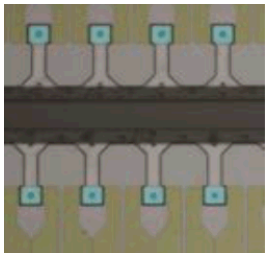


Image Credits: NASA



ecoDemonstrator Flight Test

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Overview

- Print CNT and rGO sensors on flight article
- Collect data during flight
- Test survivability and response of nanomaterial sensors

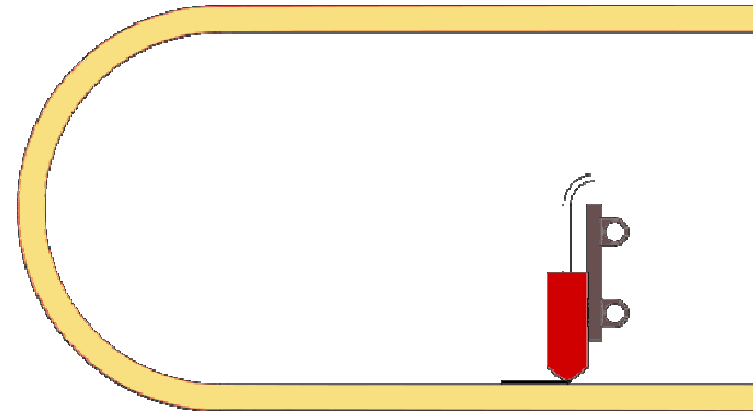


Image Credit: Boeing

Boeing 777 ecoDemonstrator

Challenges

- Unique printing parameters for rGO and CNT
- Geometric constraints: printing in constrained spaces
- In-situ post-processing



Print substrate geometry

Image Credits: NASA



CNT Printing

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Compliant Toolhead

- Tool suspended by springs
- Mitigates issues caused by uneven surfaces or machine accuracy

Sensor Parameters

- CNT Yarn
- Geometry: 32 line raster pattern
- Resistance: $\sim 50\ \Omega$

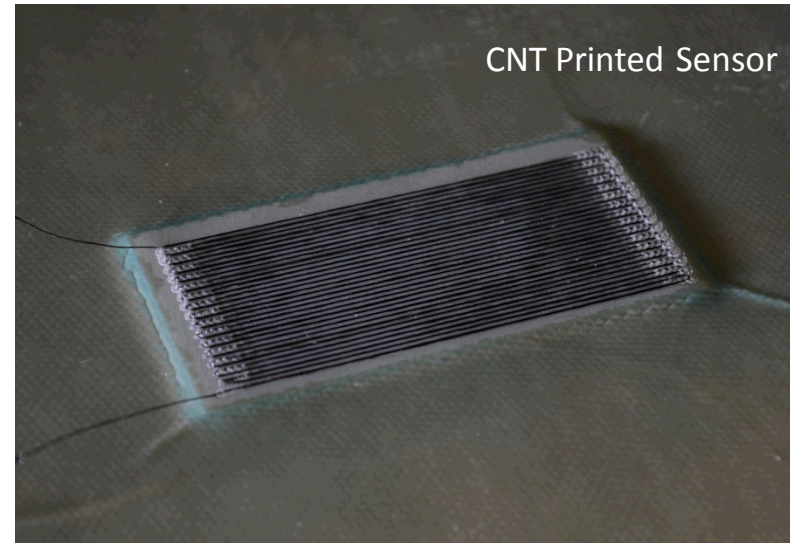
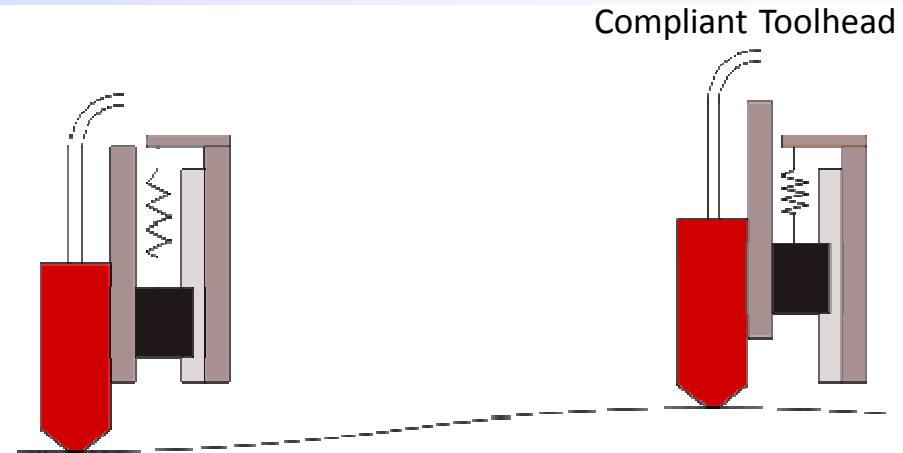


Image Credits: NASA



rGO Printing

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Sensor Parameters

- Geometry: 10 mm x 3 mm print area
- Flexible heating plate for rGO reduction
- Resistance: $\sim 5 \text{ k}\Omega$

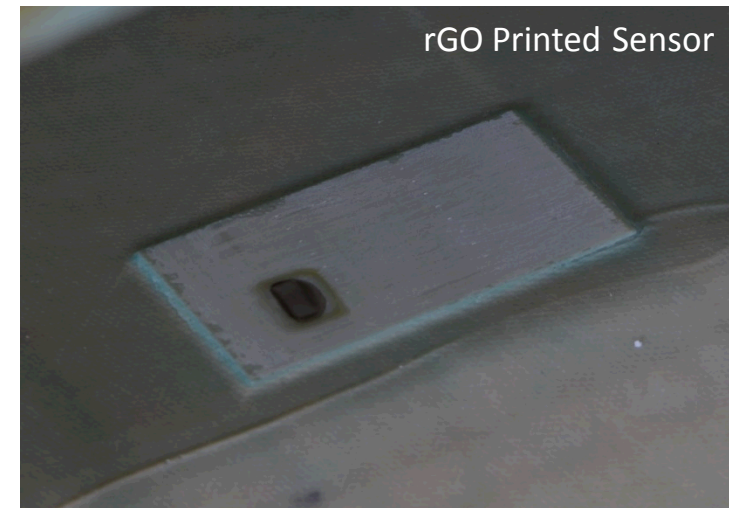
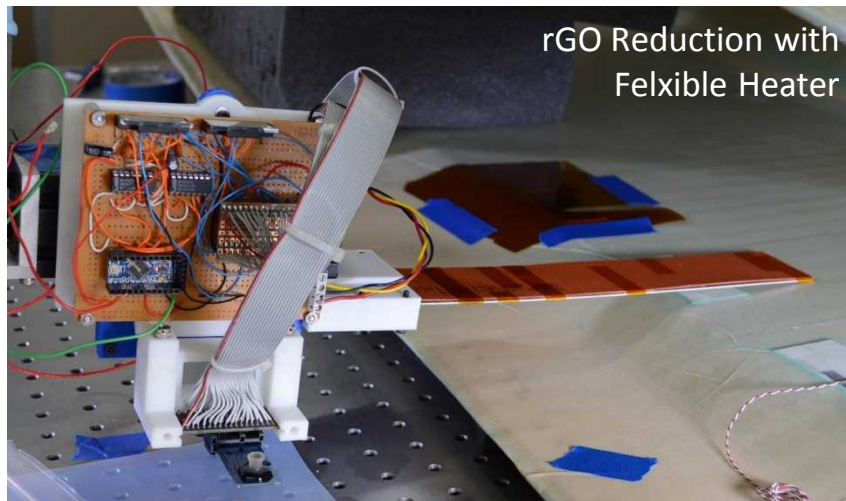
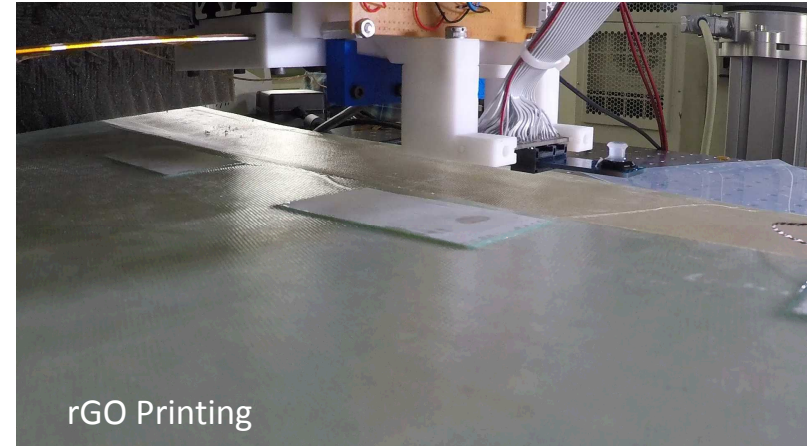
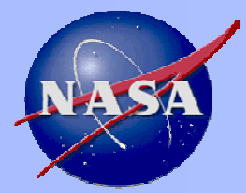
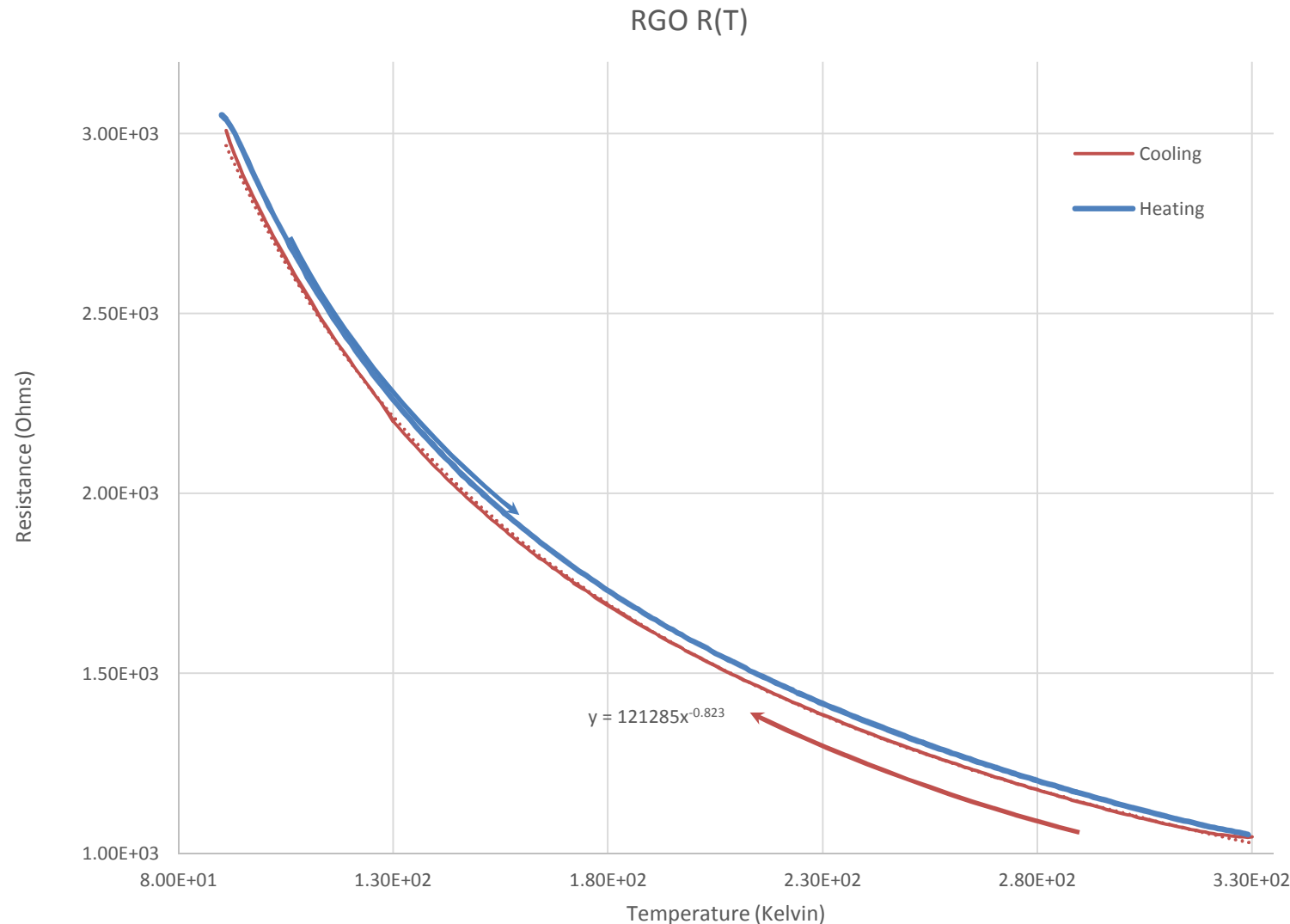


Image Credits: NASA



Temperature Dependent Resistance of RGO Sensors: Hopping Conduction

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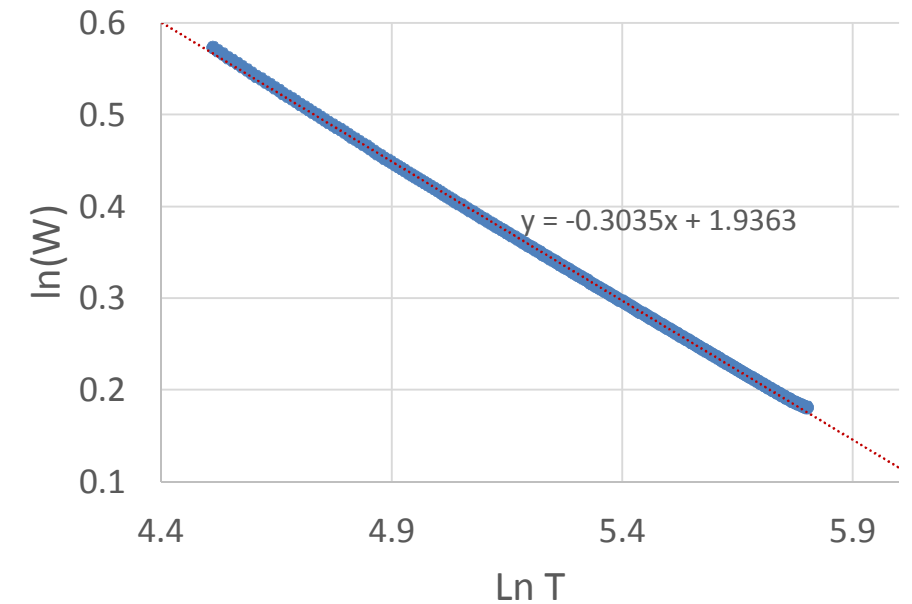


$$R = R_0 \exp \left[\left(\frac{T_0}{T} \right)^p \right], p = \frac{1}{1+d}$$

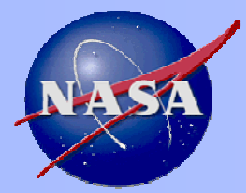
$$W = \frac{d(\ln R(T))}{d(\ln T)}$$

$$\ln W = A - p \ln(T)$$

ln(W) vs. ln(T)

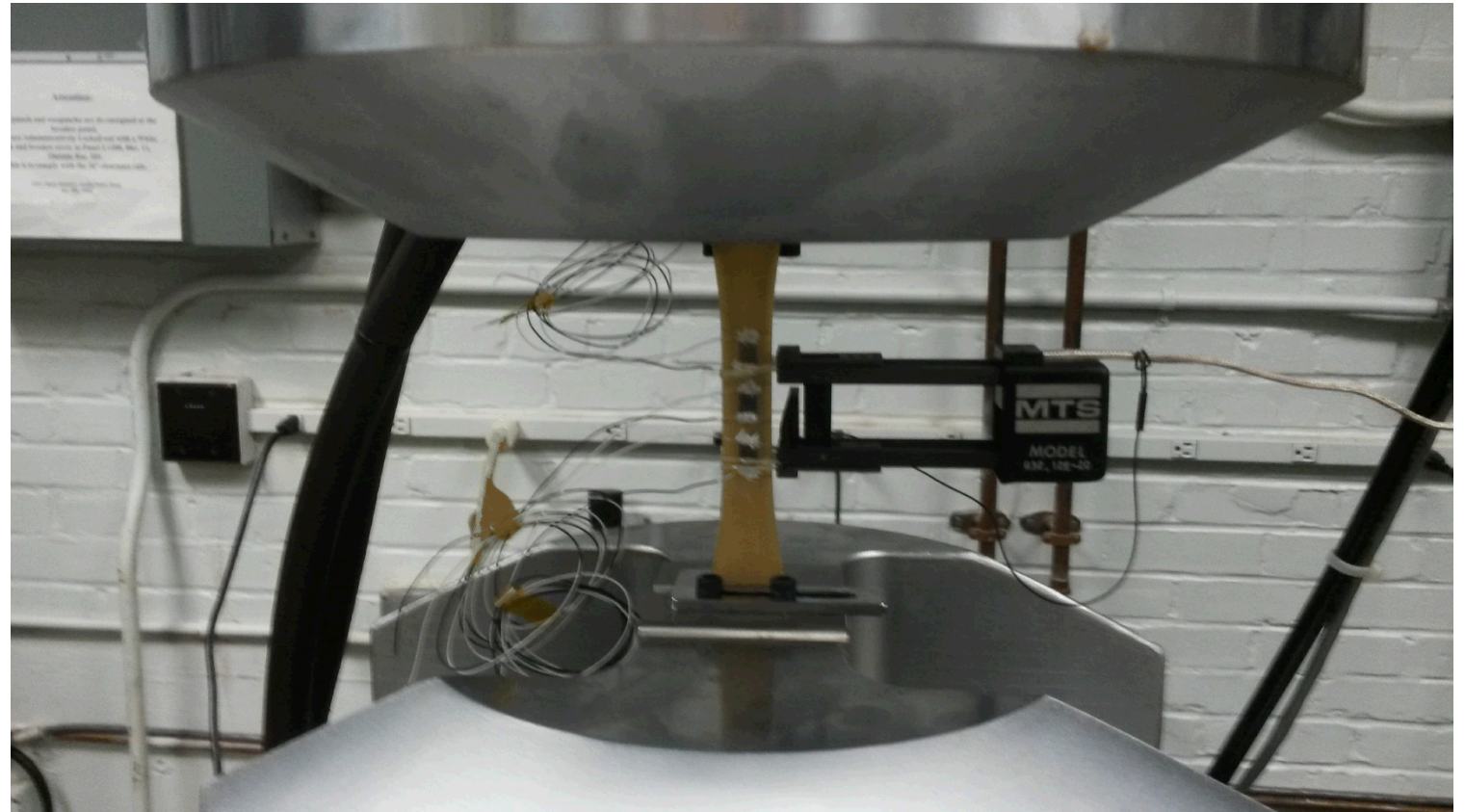
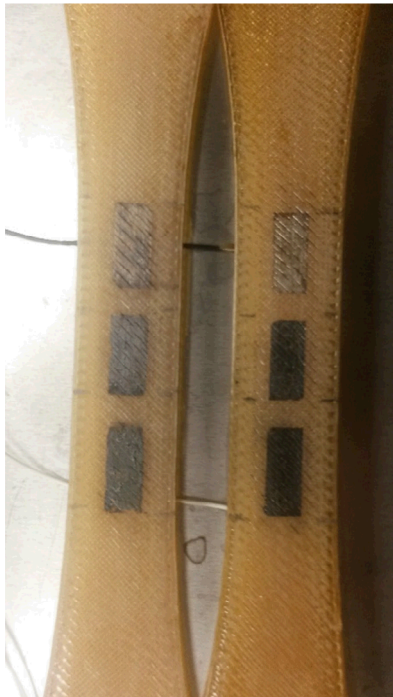
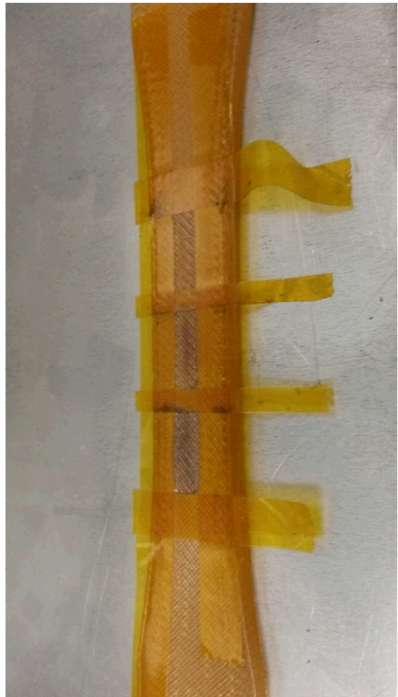


$$d = \frac{1}{p} - 1 = 2.29$$



Piezoresistance Measurements

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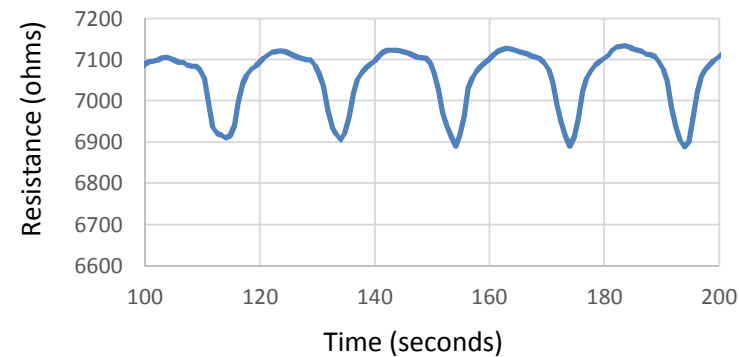


Strain Tests for RGO Sensors Printed on Ultem

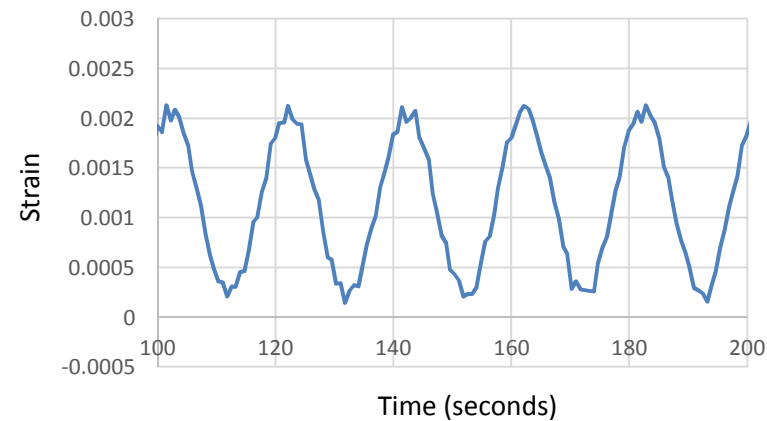
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Graphene2 Sensor1

Resistance



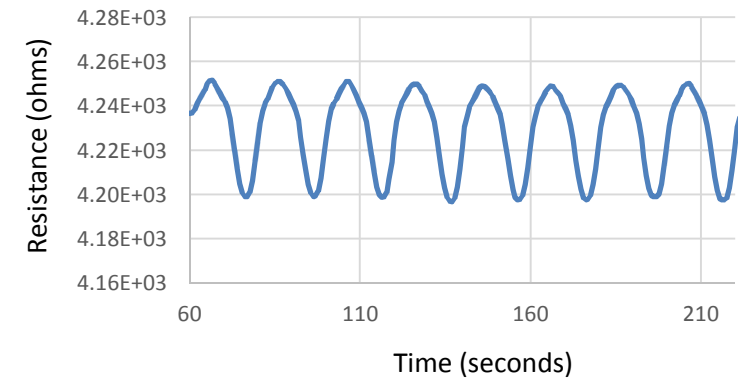
Strain



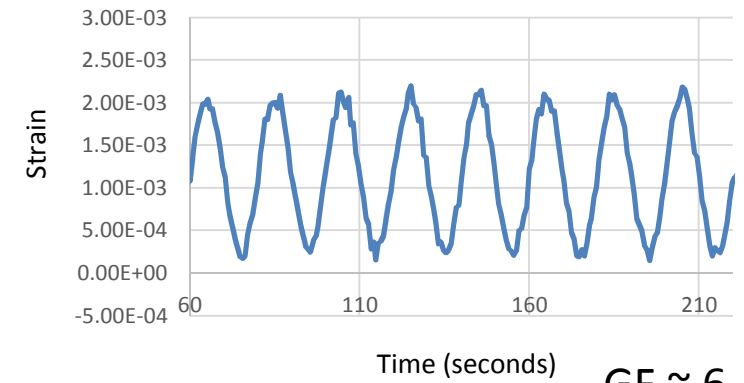
GF ~ 14

Graphene2 Sensor2

Resistance

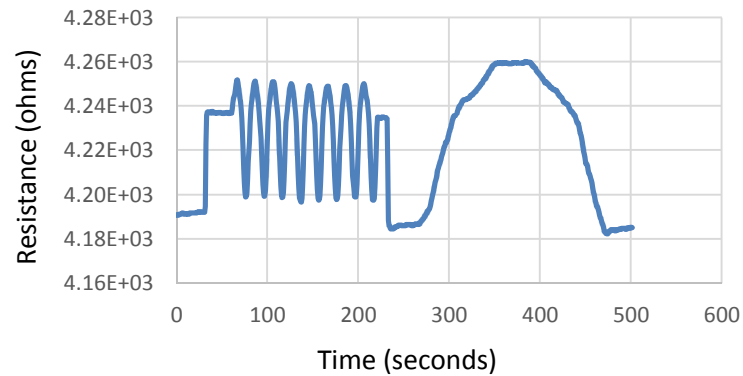


Strain

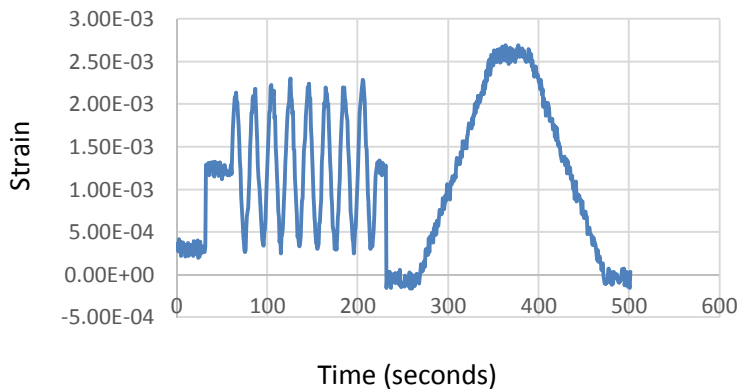


GF ~ 6

Resistance



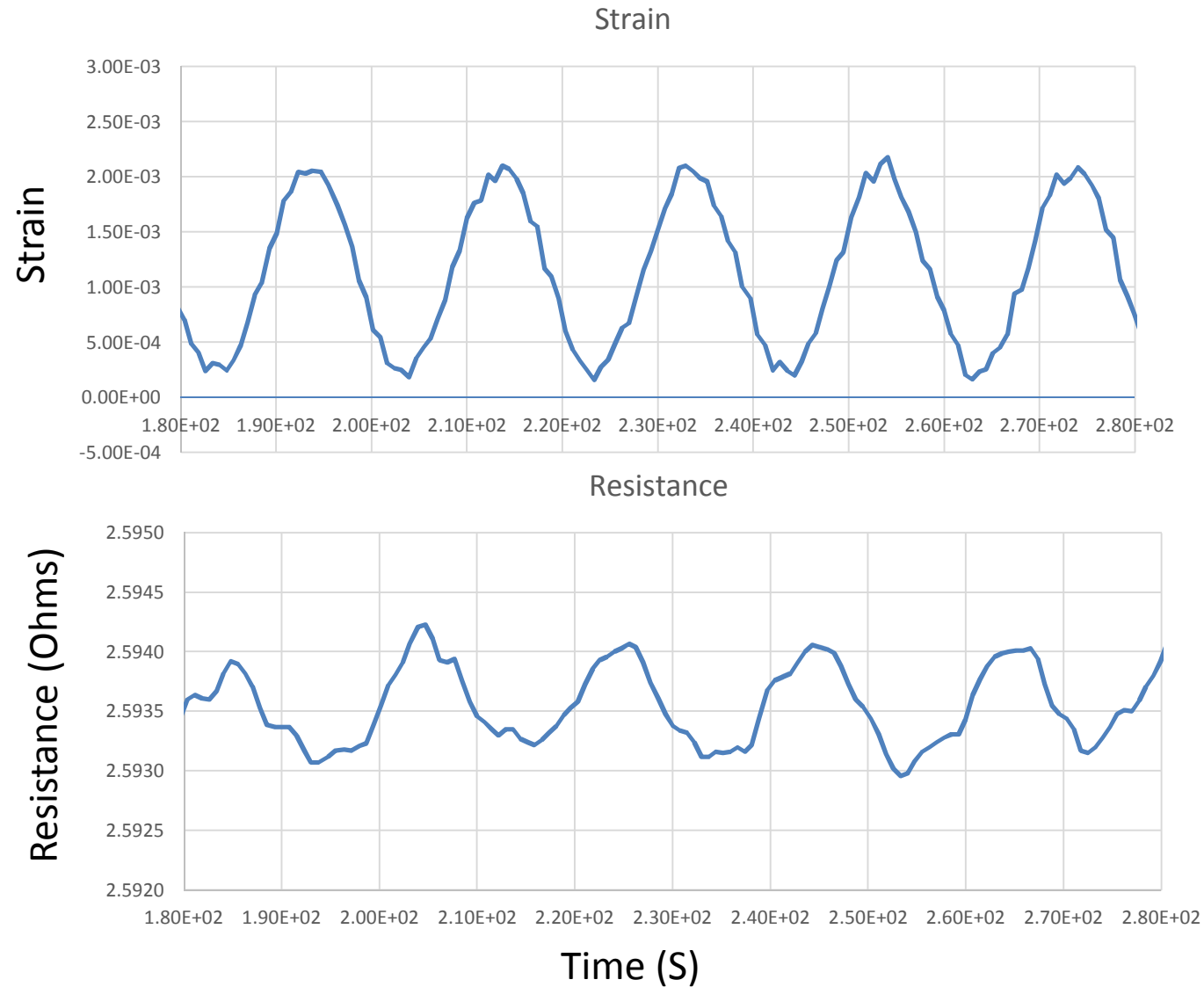
Strain2





Piezo-Resistance of CNT Fiber

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Wireless Strain Sensors for Flight Testing

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Phase III SBIR with Invocon Technologies for procurement of wireless DAQ devices for flight testing.

11 bit DAQ sensors have completion bridge wiring for RGO, CNT and commercial foil strain gauges. Gains set to accommodate anticipated temperature response of sensors.

Data acquisition rate fixed at 25 Hz for all sensors.

RGO and CNT sensors printed directly onto flight test article.

Sensors located in inaccessible area, wireless communication and self-powering required.

DAQ devices ~ 1.75" x 1.5" x 0.2" with 10 gm mass.

SAFT LSH20, 3.6V lithium-thionyl chloride (Li-SOCl₂) Battery.
34mm diameter x 61.5mm height (D-size) with 100 gm mass.
Operating temperature range -60 °C to +85 ° C.





Wing Panel

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- Wing leading edge panel
- Sensor orientation
 - Both CNT sensors lateral
- Commercial strain gauges added as control
- Data acquisition system
 - Wireless interrogation

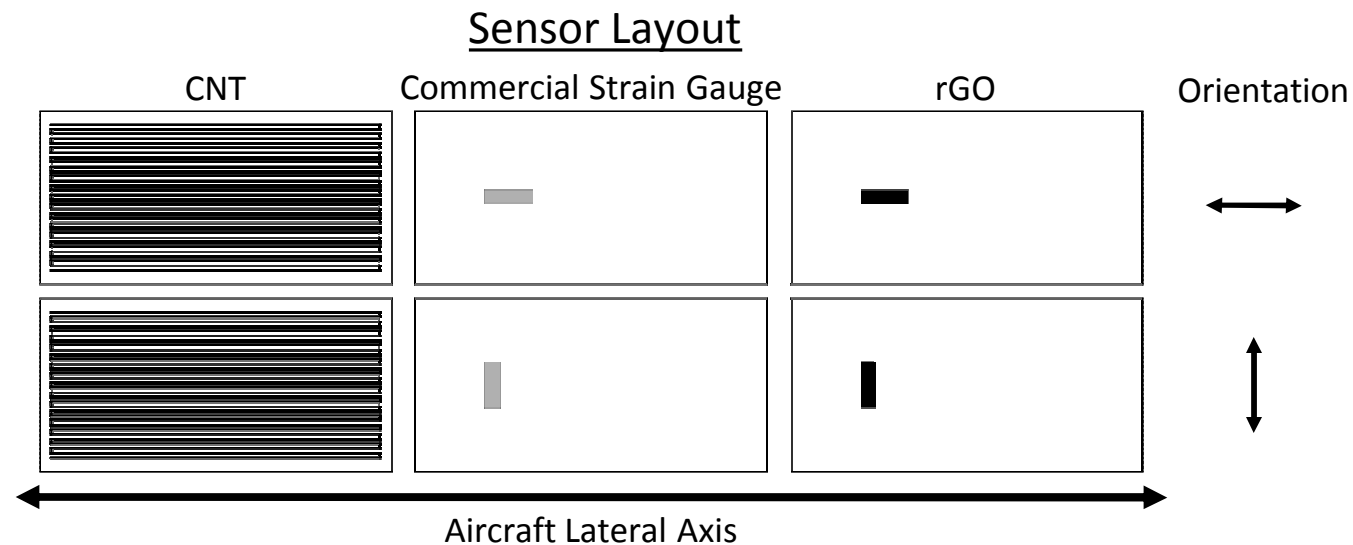
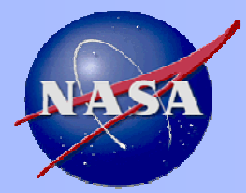


Image Credits: NASA



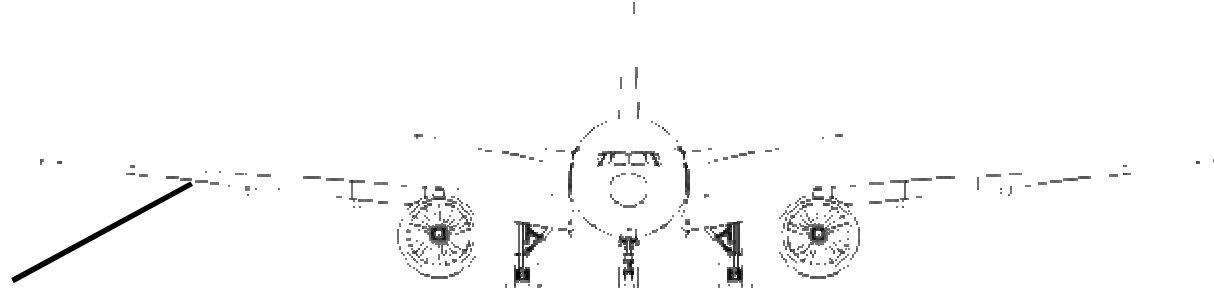
Flight Test Overview

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- Sensors printed on wing panel
- Data analyzed for three flights over one week
 - March 1 data shown
- Altitude up to 43,000 ft (~13 km)
- Correlate sensor data with strain and temperature

Panel Location



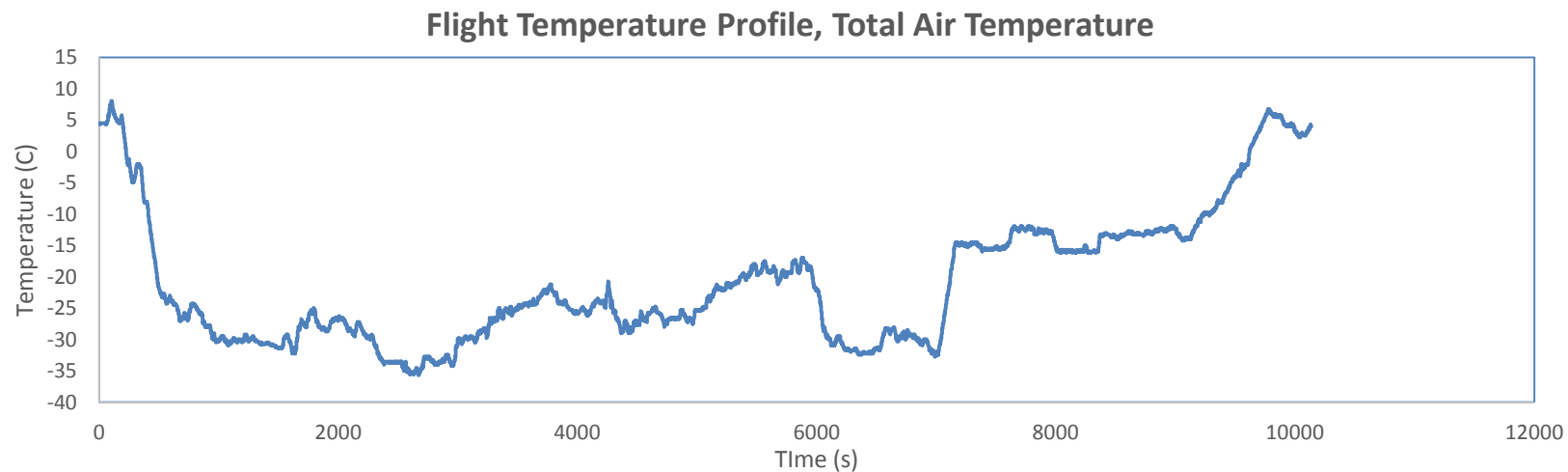
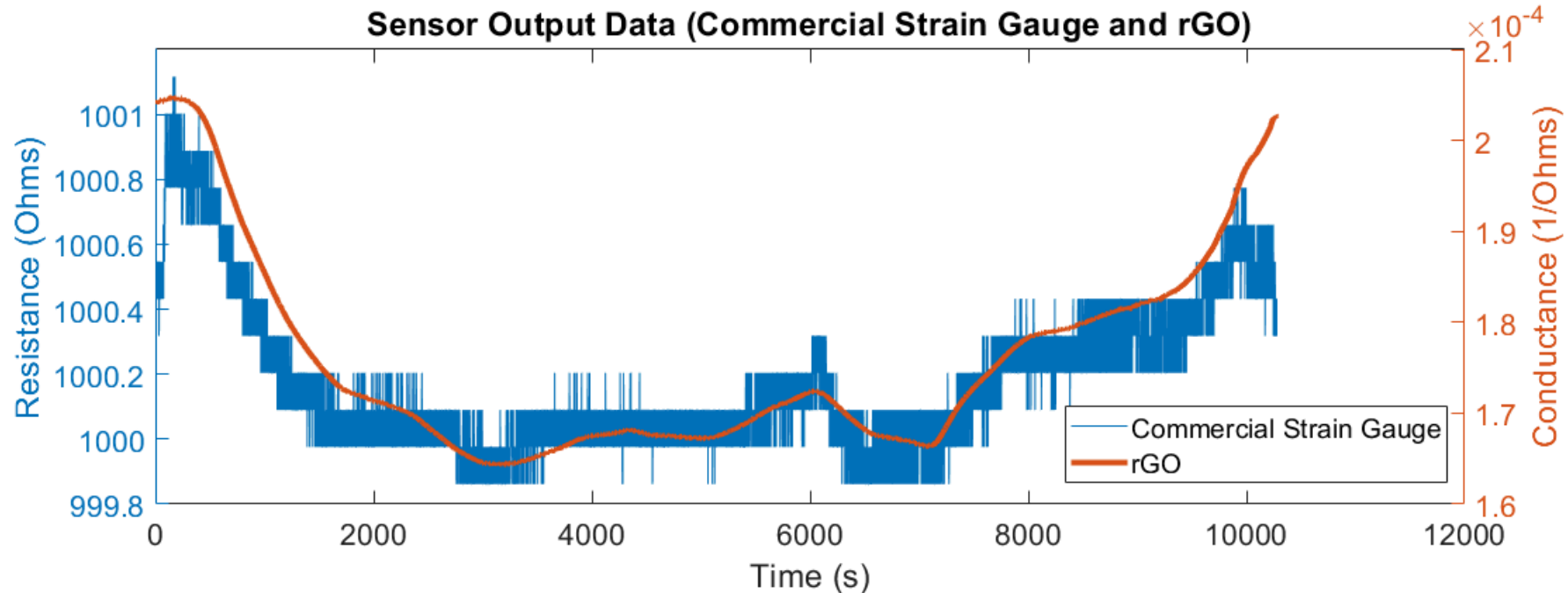
"B777FAMILYv1.1.png" by Julien.scavini CC BY-SA 3.0



Collected Data – rGO

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3/1/2018 Data

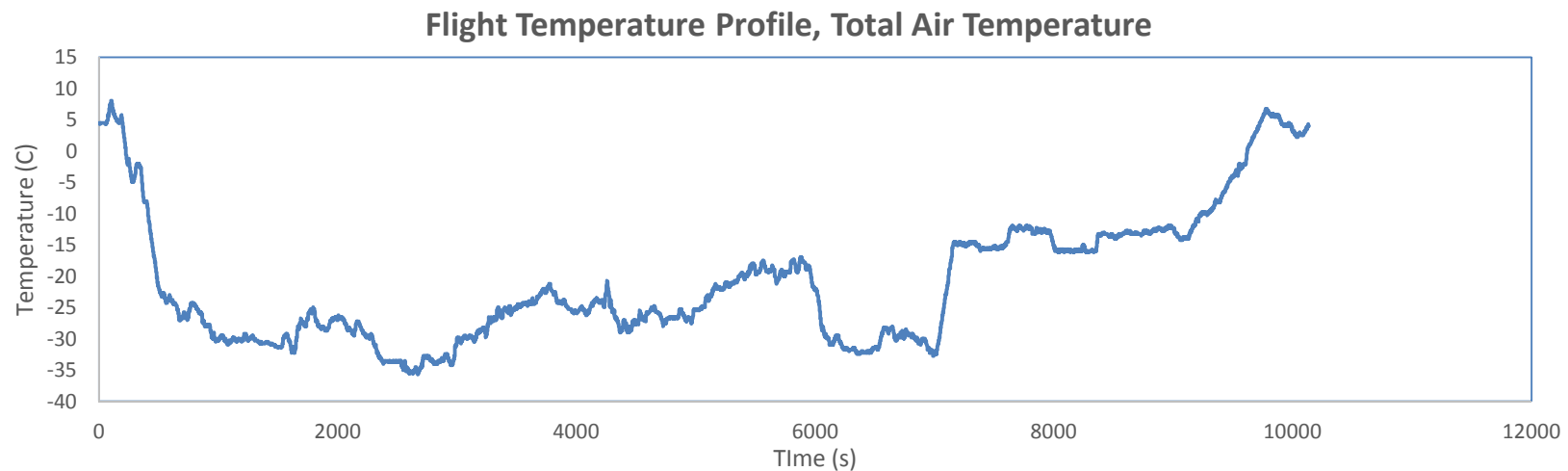
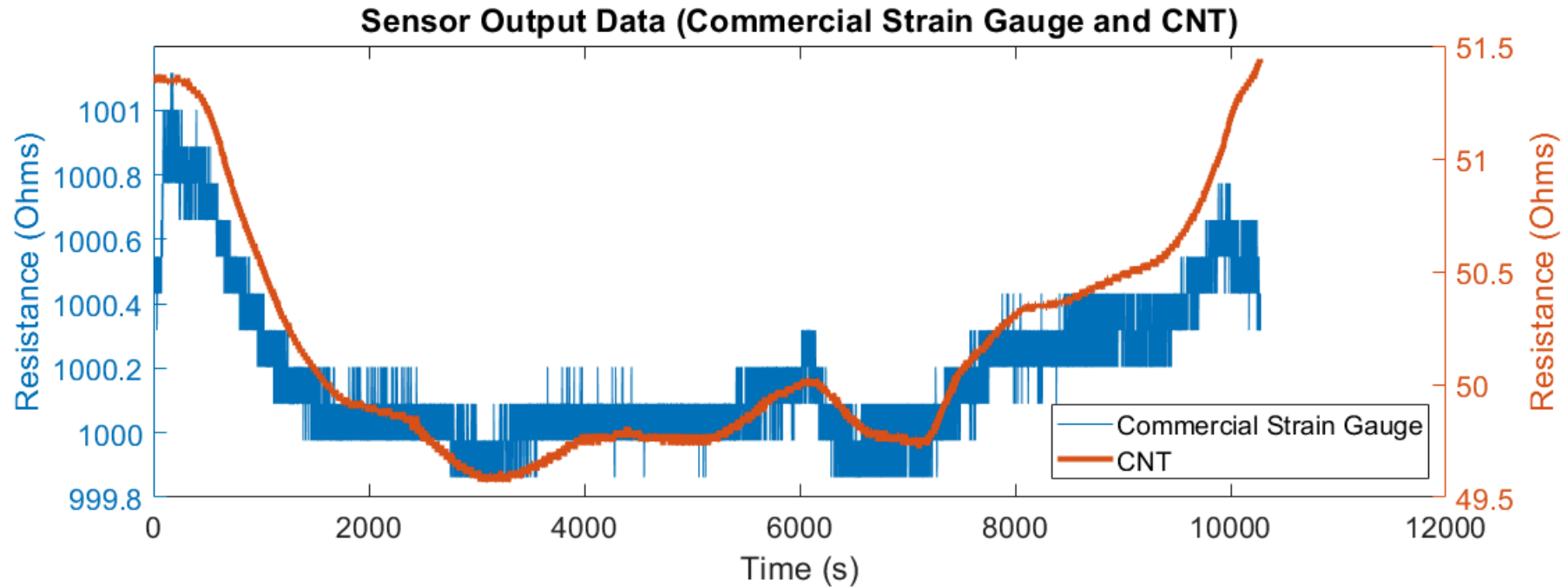




Collected Data – CNT

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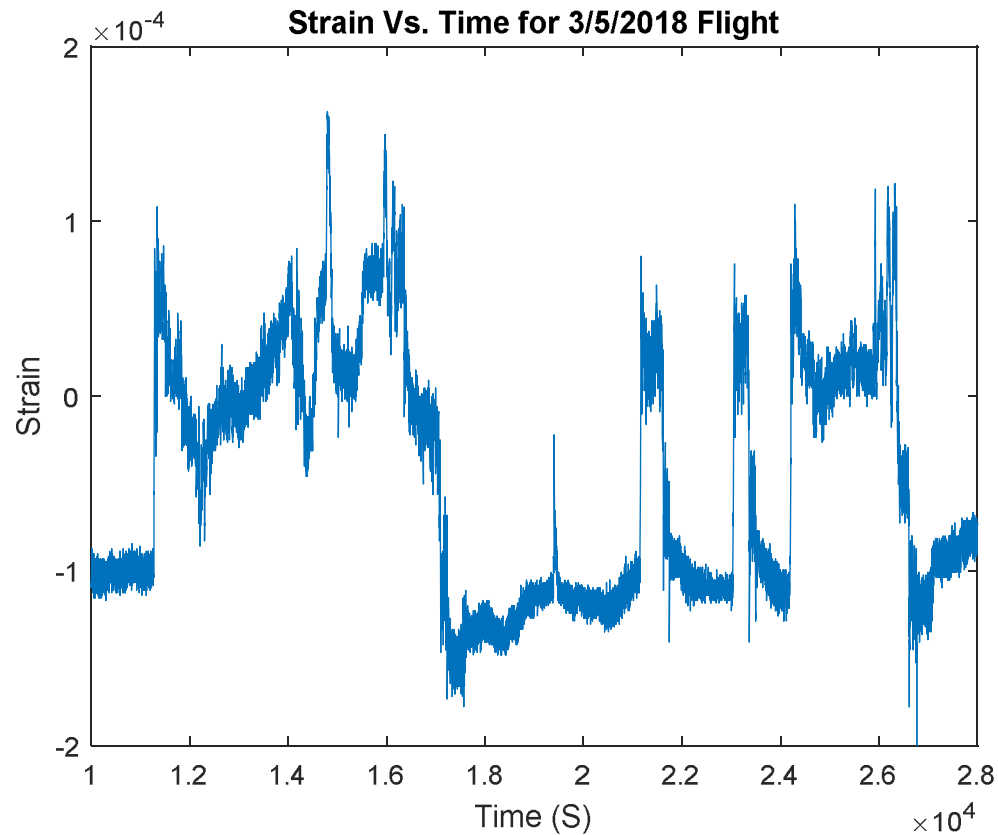
3/1/2018 Data



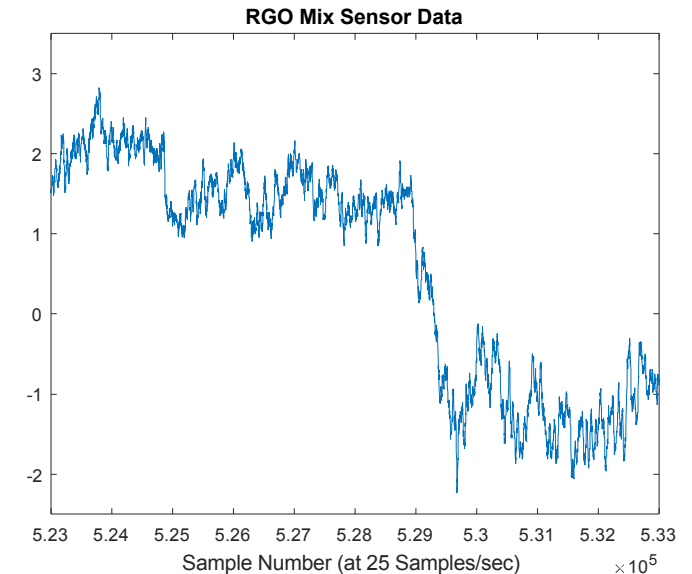
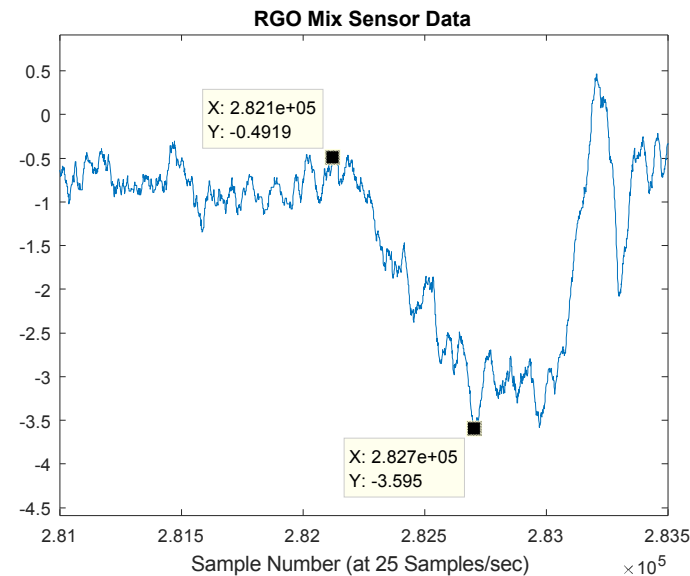
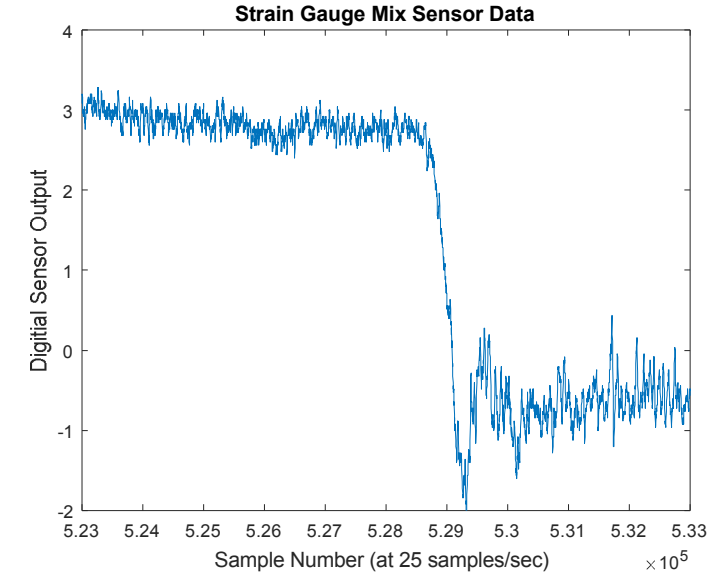
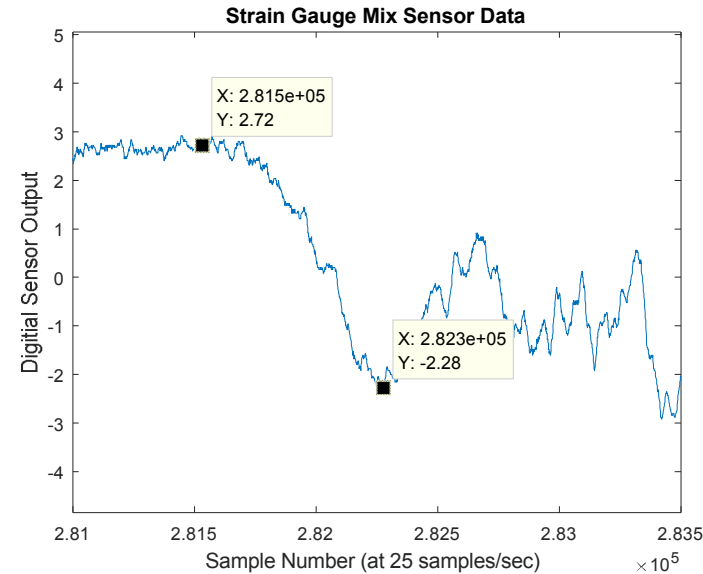


Processed Foil and RGO Strain Gauge Data 3/5/2018 Flights

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$$\epsilon = \frac{\left(\frac{\Delta R_1}{R} - \frac{\Delta R_2}{R}\right)}{G(1 + \nu)}$$

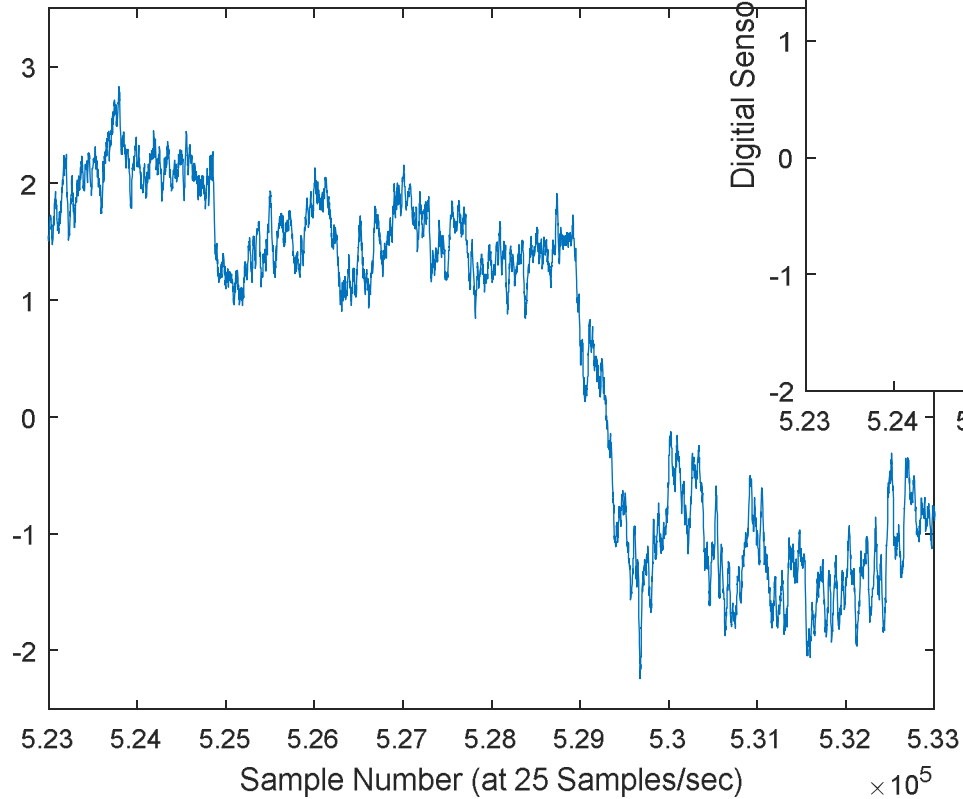




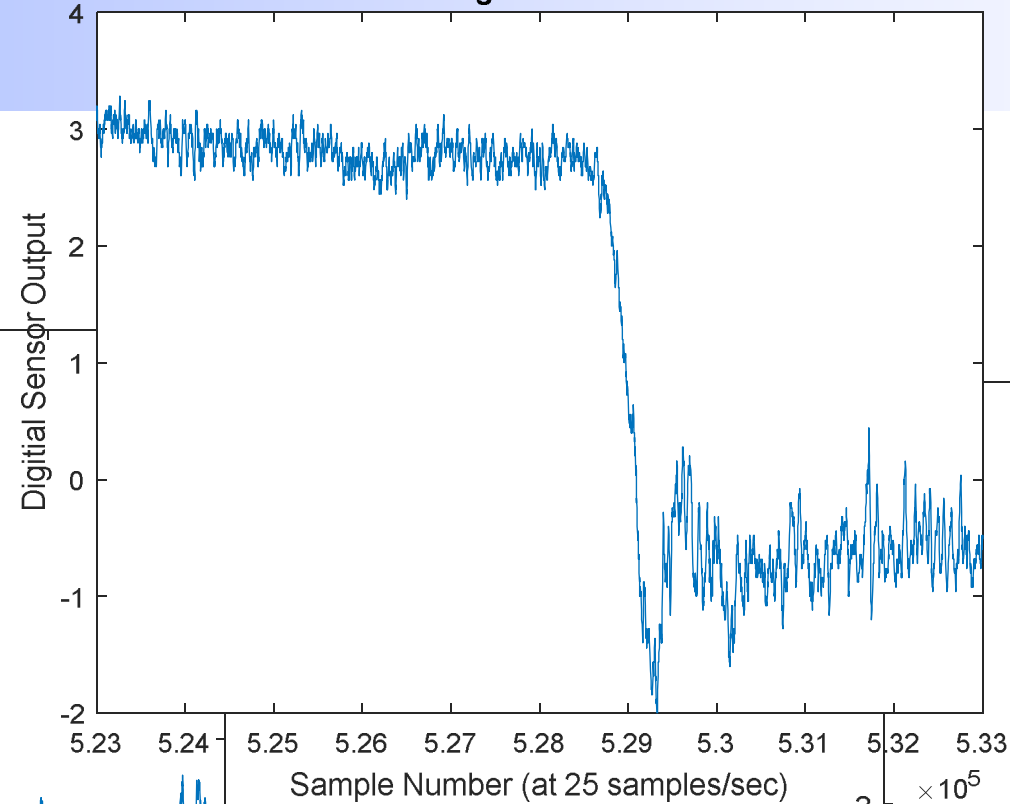
Foil Strain Gauge Vs. RGO Sensor and CNT Sensor: Second lift-off, 3/5/2018

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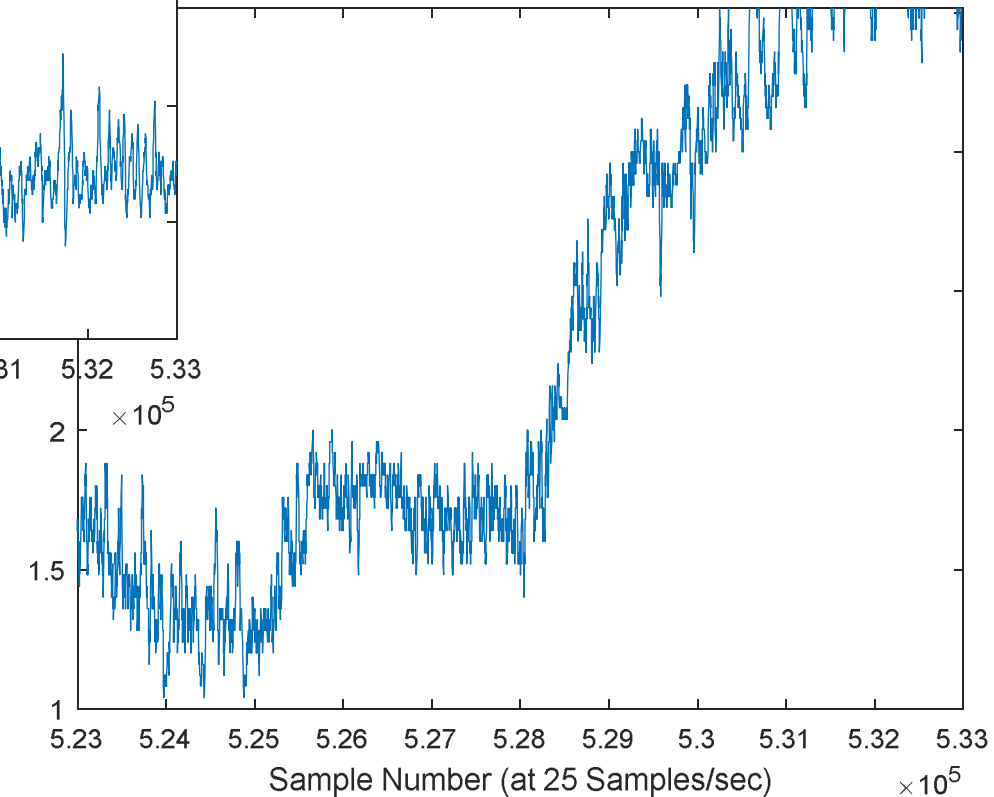
RGO Mix Sensor Data



Strain Gauge Mix Sensor Data



CNT Mix Sensor Data



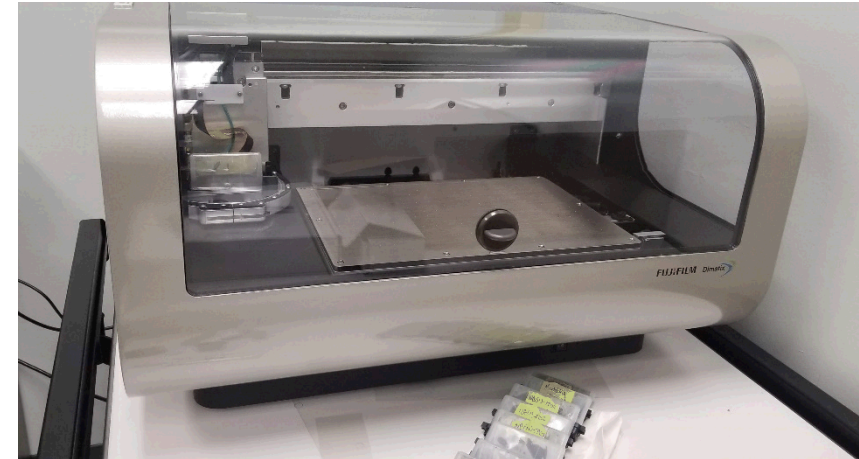
CNT sensor shows opposite response vs. strain. Data is also shifted by ~600 points (40 seconds) due to later start of DAQ



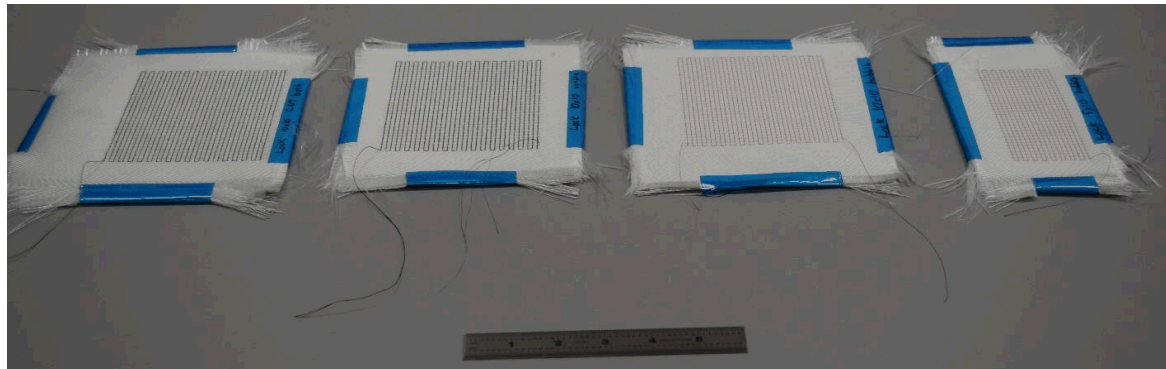
Future Directions

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- Fujifilm Dimatix Material Printer procured and under testing for conductive ink and graphene printing.



- Phase III and Phase IIx SBIR under way to investigate stitched CNT sensor performance and characteristics.





Summary

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- Multifunctional CNT and rGO sensors have been developed, printed on unique substrate geometry and flight tested.
- Nanomaterial sensors survived the flight environment and recorded data with a profile similar to the commercial strain gauge control.
- Temperature changes have a major effect on the sensor outputs. Temperature correction algorithms and techniques incorporating hopping conduction mechanism are being developed and applied for nanomaterial sensors.
- Continued research required for optimization of RGO and CNT sensors and multifunctional systems.
- University funded (NSTRF) research on *2D Materials for Energy Harvesting and Sensing* seeks to add capabilities for structural health monitoring or aerospace structures.