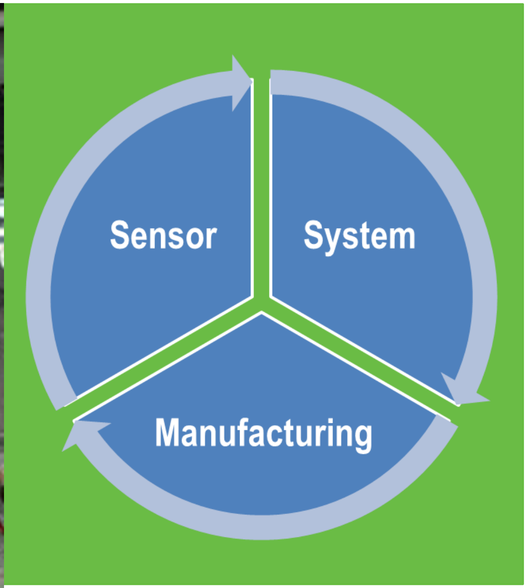
Peel and Stick Sensor for Refrigerant Leak Detection



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Project Summary

Timeline:

Start date: 10/01/2017

Planned end date: 09/30/2019

Key Milestones

1. Evaluate flexible sensor characteristics for flammable refrigerants, 03/31/2019
2. Establish sensor drift characteristics and calibration requirements, 09/30/2019

Budget:

Total Project $ to Date:

- **DOE: $96K**

Total Project $:

- **DOE: $175K**

Key Partners:

Danfoss
Emerson
Mexichem

Project Outcome:

- The project aims to develop a low-cost refrigerant sensor through a combination of direct-write printing and pulse thermal photonic processing to overcome thermal barriers for plastic integration
- Direct-write printing of low-cost refrigerant sensors employing high throughput roll-to-roll manufacturing techniques will define a path towards direct and continuous monitoring of refrigerant leakage

Publication: "A novel strategy to purify conductive polymer particles." RSC Advances 9, no. 9: 4857-4861, 2019.

Disclosure: NUMBER: S-138,840 A Novel Strategy to Purify the Conductive Polymer Particles

Team

Dr. Pooran Joshi: Focus on development of direct-write sensors. Over 20 years of R&D experience on advanced sensors and devices

Dr. Vishaldeep Sharma: Expertise in commercial refrigeration systems. Research interests include energy audits and green roofs

Dr. Brian Fricke: Over nine years of active involvement in the assessment/evaluation of low GWP refrigerants in commercial systems

Dr. Ayyoub Momen: Expertise in energy-efficient technologies for buildings and refrigeration systems. Subprogram manager for the HVAC&R, Water Heating, and Appliances Program

Dr. Teja Kuruganti: Over 14 years of experience in wireless communications and sensor technologies. Manages ORNL's buildings-related sensors, controls, and transactive energy research

Dr. Jaswinder Sharma: Expertise in chemical synthesis of nanomaterials

Dr. Tolga Aytug: Over 20 years of R&D experience in materials processing for integrated devices

Yongchao Yu (PhD Student): Direct-write printing

Christine Fisher (SULI Student): Chemical synthesis

Challenge

Problem Statement

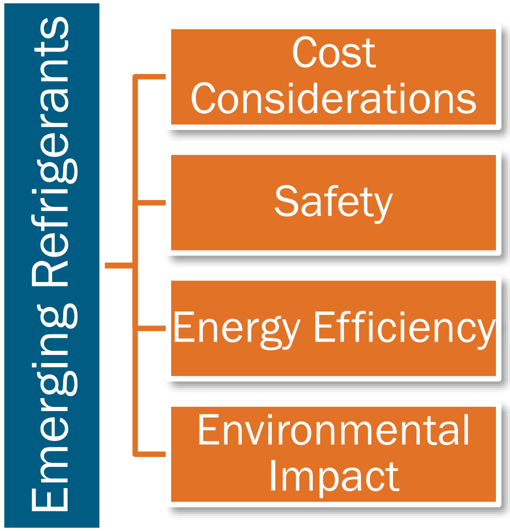
Chlorine-Free Refrigerants

- **Halocarbon-Based Refrigerants**
  - Nontoxic
  - High global warming potential (GWP)
- **Hydrocarbon-Based Refrigerants**
  - Nontoxic but highly flammable (2–10% in air)
  - Low GWP
- **Ammonia as Refrigerant**
  - Environmentally friendly: ODP=0, GWP <1
  - Toxic: Poisonous in high concentration (>100 ppm)
  - Flammable between 16–25% by volume

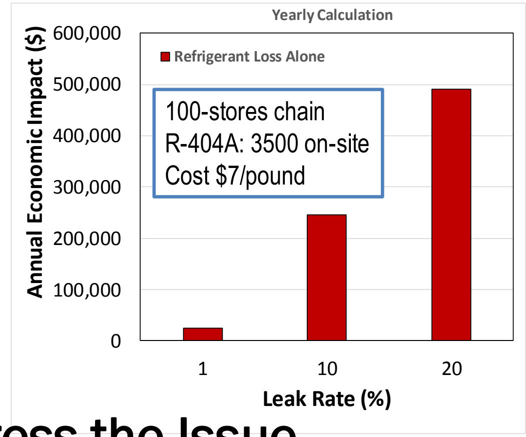
Anthropogenic Activity

- Refrigerant leakage is the most frequent fault in a refrigeration system
- Annual leakage in commercial refrigeration systems can vary from an average of 11% up to 30% in some cases

Refrigerant Gas Detection



Early Detection: Crucial



Advanced Sensors and Controls Critical to Address the Issue

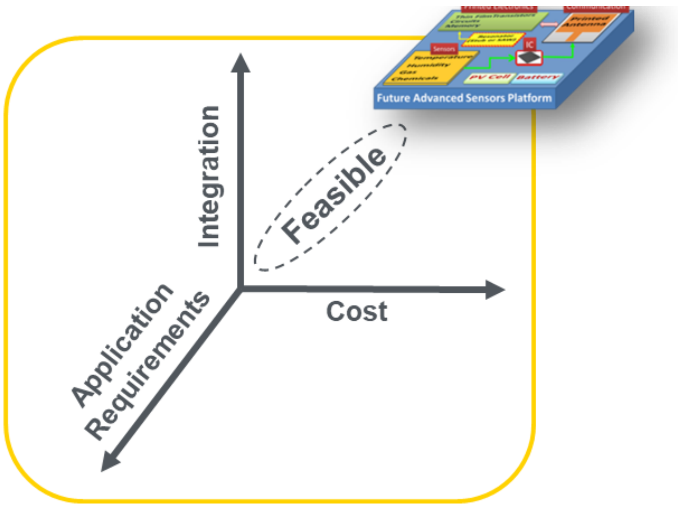
Path to Success

Address Refrigerant Leak Detection Issue: Both Commercial and Residential Applications

- Inertia in use of energy-efficient toxic and/or flammable refrigerants
- Currently, sensors used to prevent a potential combustible event

Refrigerant Sensor Development

- Sensitivity/selectivity
- Response time
- Reliability
- System integration
- Cost



Low-cost sensors required to address health, safety, environmental, and financial issues associated with current and emerging environmentally friendly refrigerants.

Technology Space

Sensor Technologies for Refrigerant Applications

Sensor	Range (ppm)	Response Time (s)	Operating T (°C)	Lifetime (years)	Cost
Infrared (PIR/NDIR)	0 -10,000	5 - 300	-40 - 75	5 - 15	$300-$12,000
Electrochemical Cell	0 – 1,000	<90	-20 - 50	1 - 3	$250 - $1,600
Metal-Oxide-Semciconductor	20 – 10,000	15 - 90	-34 - 170	3 - 5	$500 - $1,300
Catalytic	0 – 1,000	20 - 30	40 - 150	2 - 5	$700 - $1,500
Heated Diode	<0.1 – 6.6oz/yr	0.5 – 1	-20 - 50	2 - 3	$100 - $500
Virtual Sensor	Indirect estimation based on algorithm; no leak measurement				

* AHTRI Report No. 9009

Opportunity

- Conducting polymers show promise for electrochemical detection of ammonia.
- Low-cost, small-footprint metal-oxide-semiconductor (MOS) sensors suitable for refrigerant monitoring in residential environment

Approach

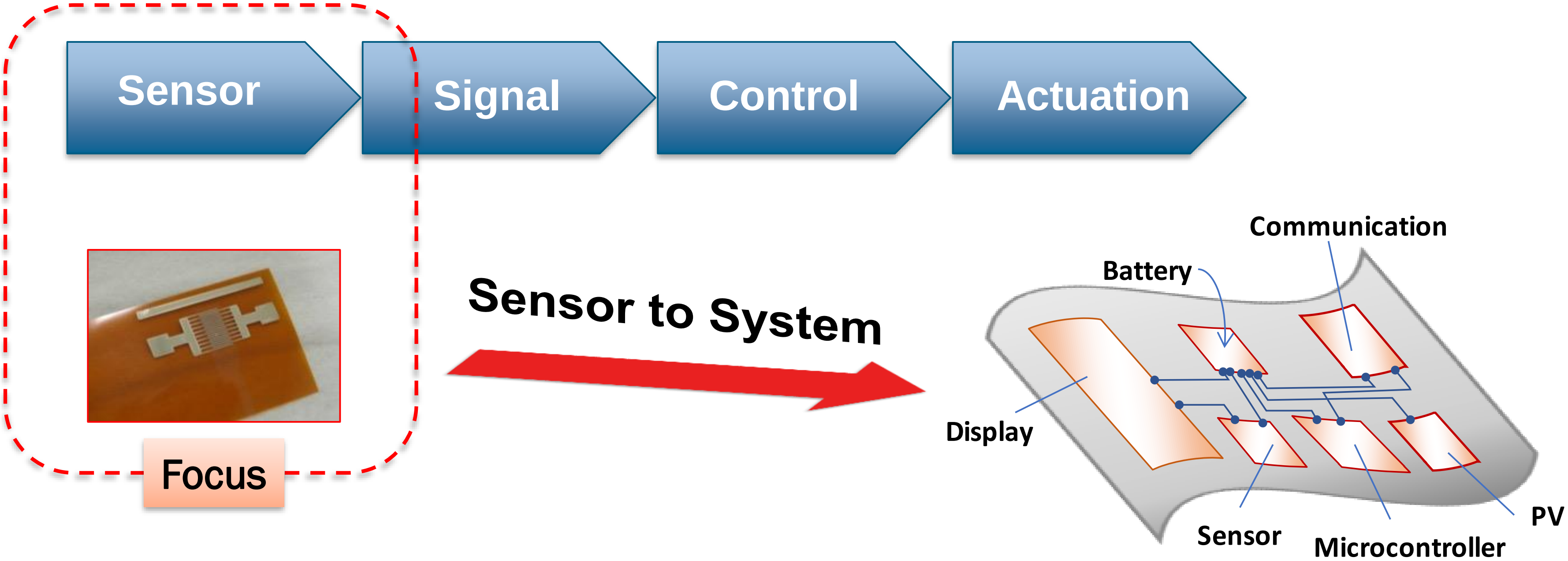
Direct-Write Printed Sensor Development

- Develop process for conductive polyaniline nanoparticle (PANI) printing
- Evaluate coating quality and material characteristics
- Develop process and evaluate the impact of 2D materials (carbon nanotube [CNT] and reduced graphene oxide [RGO]) on sensor performance
  - Main focus: Ammonia sensitivity, selectivity, and reliability characteristics

Metal-Oxide Sensor Development

- Low-temperature pulse thermal processing of thin film on low-cost plastic substrate
- Interdigitated electrode (IDE) development for resistive sensor configuration
- Binary metal-oxide printing: Focus on metal-oxide (ZnO)
  - Main focus: Evaluate material performance for F-sensor applications

Impact



Low-Cost Refrigerant Sensor

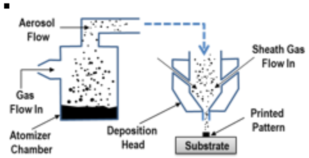
- High penetration of environmentally friendly refrigerants is enabled
- Sensitivity, selectivity, and power consumption will dictate synergistic integration on multifunctional sensor platform
- Low cost is critical for widespread deployment of sensor in areas where refrigerant from a leak will concentrate
- Low-power operation is critical for sensor connectivity with low-cost, low-power IoT platform

Enabling Capabilities

Direct-write Inkjet and Aerosol Jet Printing

Printing Challenges:

- Resolution
- Process tolerance
- Defect density
- Printing yield

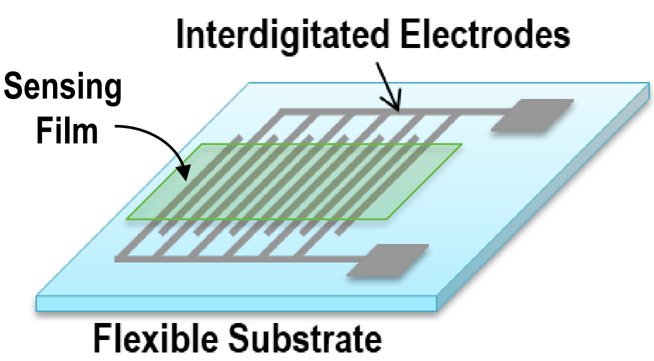


Fujifilm Dimatix

Optomec Aerosol Jet

Line Width/Spacing Control

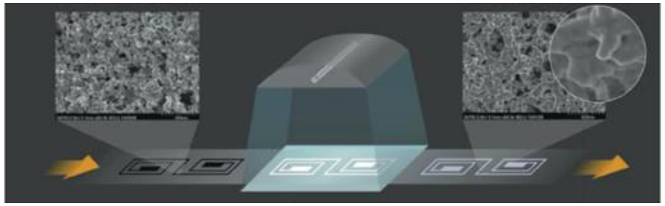
- Down to 10 µm

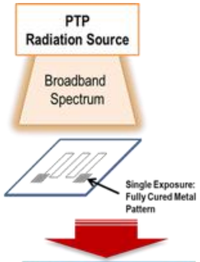


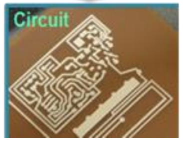
Direct-write Printing

- Sensors
- Active/passive layers
- Antennas
- Electrical contacts

R2R Compatible Pulse Thermal Processing



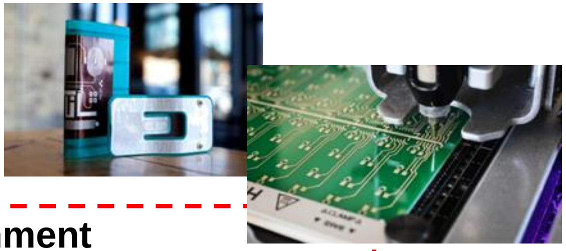




Power Density: >20 KW/cm^2
Process Window: µs-milliseconds-continuous

Direct-Write PCB
Concept → Prototype→ Production

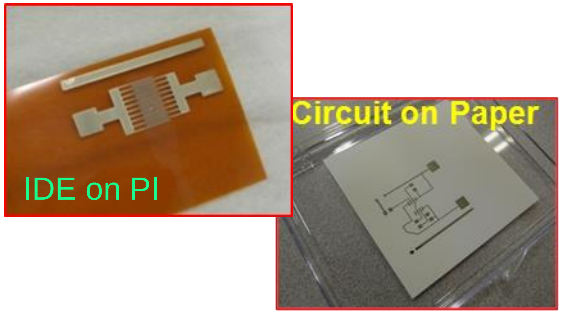
Sensors and Circuits on Rigid and Flexible Substrates

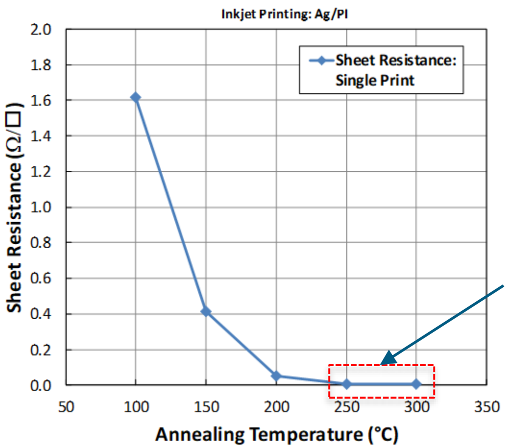


→ **SMART alignment**
→ **Automatic height compensation**
→ **Feature control**: 200 µm
→ **Pin-to-pin pitch**: 650 µm
→ **Substrate thickness**: up to 3 mm

Progress

Printed Ag Metal Line



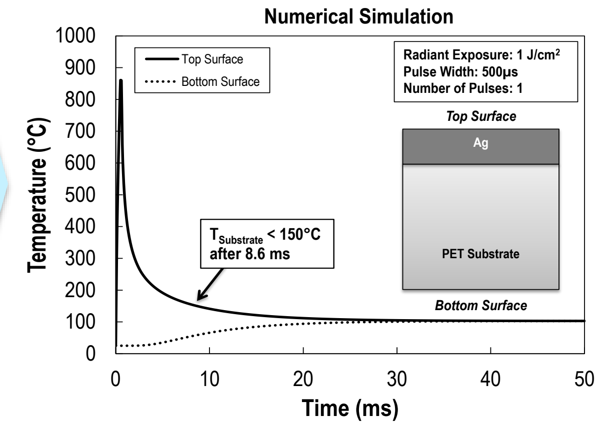


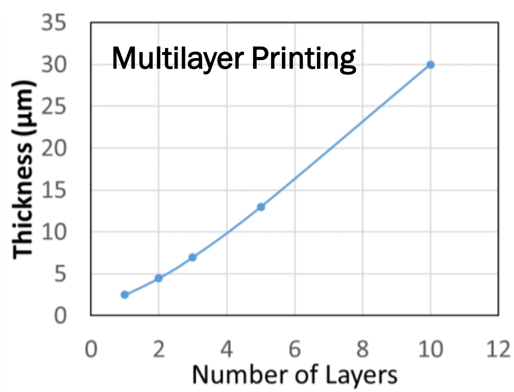
Conductivity approaching bulk value

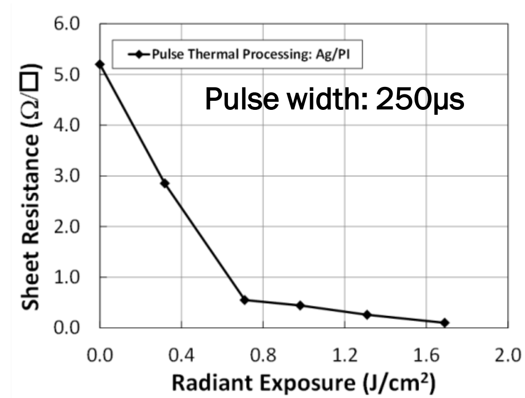
Nanoparticle ink conductivity approaches the bulk value

Low-Temperature Integration

Millisecond processing on flexible substrates





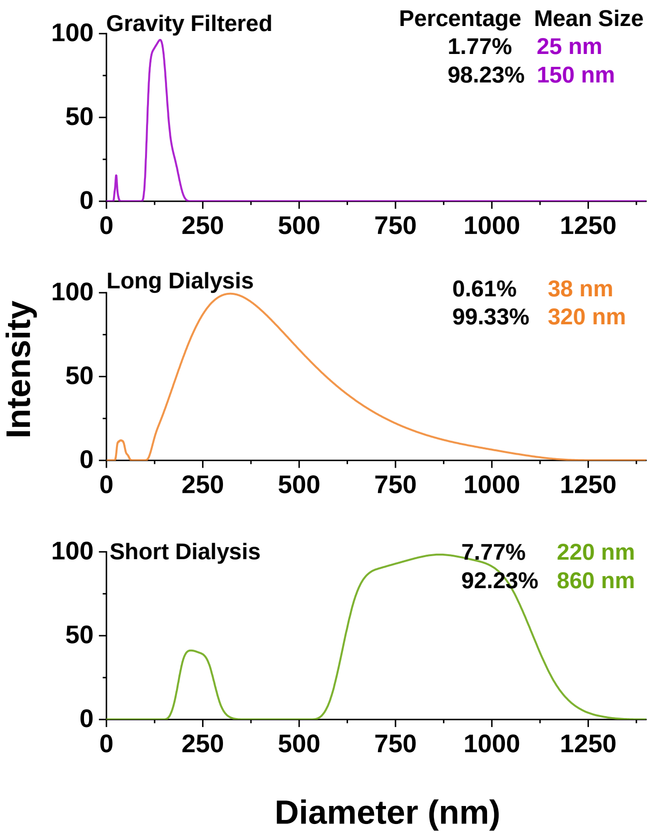


- Low thermal budget processing

Progress

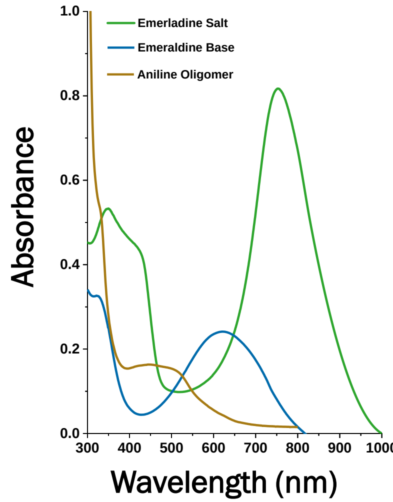
Nano PANI Synthesis

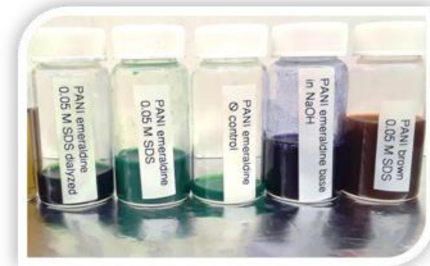
Particle Size Distribution



Combination of dialysis and filtering for nano-PANI synthesis

Conductive vs Nonconductive PANI





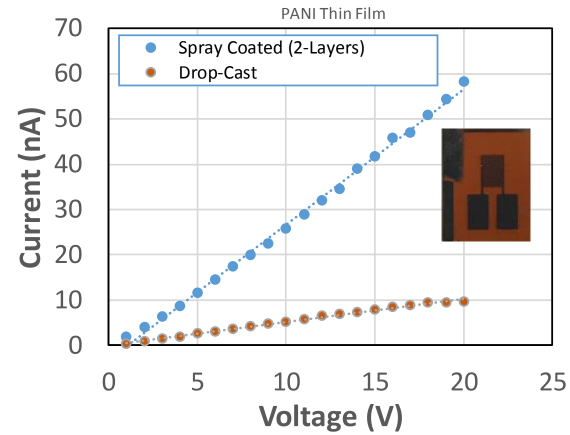
Green emeraldine salt (ES)

– Intrinsically conducting

Blue emeraldine base (EB)

– No conductivity

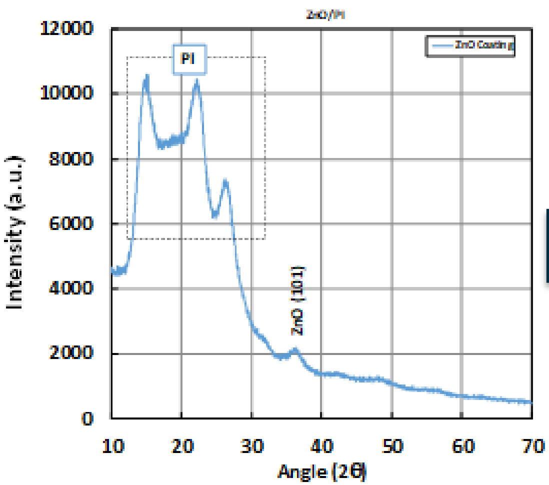
Electrical Conductivity



- Linear I-V response measured on PANI thin films deposited on interdigitated electrodes

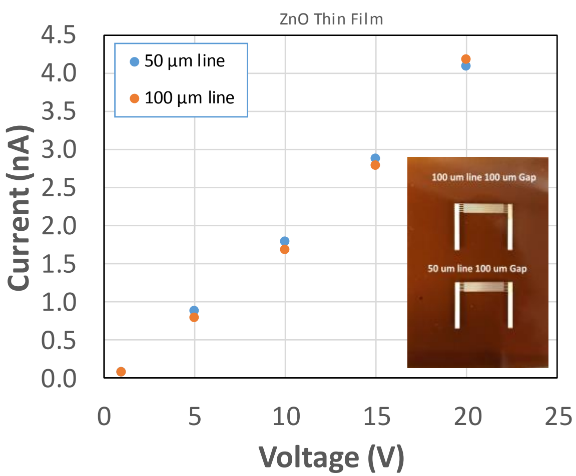
Progress

ZnO Thin Film



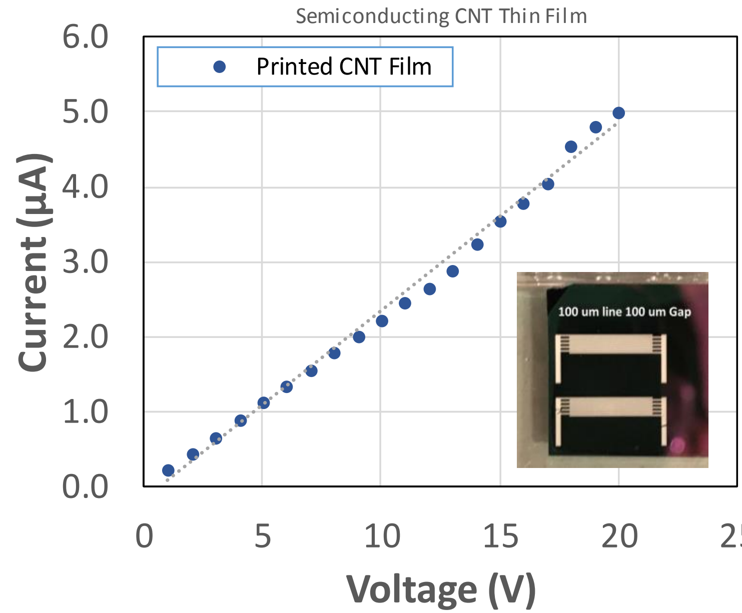
Printed thin films are crystalline

Electrical Conductivity



CNT Thin Film

Direct-write printing using 99.9% semiconducting CNT



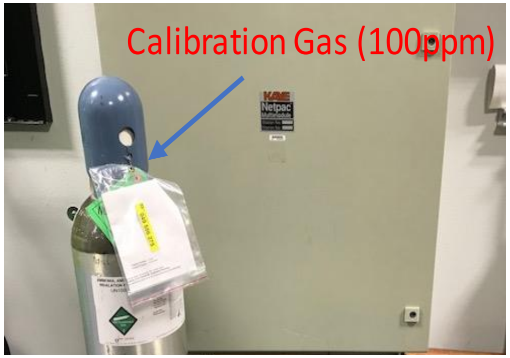
- Process developed for both p-type and n-type CNTs
- Linear current-voltage response measured on Ag interdigitated electrodes

Progress

Measurement Setup

- Setup is currently being pressure and leak tested

Setup Qualification



Steps

- Two calibration gases:
  - N_2/NH_3 (100 ppm)
  - N_2/C_3H_8 (100 ppm)
- Baseline data: Commercial sensors
- Test printed prototypes

Stakeholder Engagement

- **Danfoss has shown interest in working with ORNL on refrigerant sensors**
- **The Air-Conditioning, Heating and Refrigeration Technology Institute, Inc. (AHRTI) is interested in the project**
  - AHRTI: Identified refrigerant sensors as one of their top research priorities
  - Emerson: Leader in refrigeration technologies
  - Mexichem: Global leader in the development, manufacture, and supply of fluoroproducts

Remaining Project Work

- Develop direct-write printing process for sensor and electrodes integration
- Evaluate performance in a relevant environment

- Optimize fabrication process through process-property correlation
- Evaluate sensor drift characteristics as a function of environmental parameters

Oct	Nov	Dec	Jan	Feb	Mar	Apr	May	Jun	Jul	Aug	Sep

- Evaluate flexible sensor characteristics for flammable refrigerants
- Establish sensor drift characteristics and calibration requirements

Go/No-Go Decision: Sensor performance at 100 ppm level

Thank You

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REFERENCE SLIDES

Project Budget

Project Budget: **$75K (2018), $100K (2019)**
Variances: None
Cost to Date: $**21K**
Additional Funding: None

Budget History					
FY 2018 (past)		FY 2019 (current)		FY 2020 (planned)	
DOE	Cost-share	DOE	Cost-share	DOE	Cost-share
$75K		$21K			

Project Plan and Schedule

Project Schedule												
Project Start: 10/01/2017		Completed Work										
Projected End: 09/30/2018		Active Task (in progress work)										
		Milestone/Deliverable (Originally Planned) use for missed										
		Milestone/Deliverable (Actual) use when met on time										
	FY2018				FY2019				FY2020			
Task	Q1 (Oct-Dec)	Q2 (Jan-Mar)	Q3 (Apr-Jun)	Q4 (Jul-Sep)	Q1 (Oct-Dec)	Q2 (Jan-Mar)	Q3 (Apr-Jun)	Q4 (Jul-Sep)	Q1 (Oct-Dec)	Q2 (Jan-Mar)	Q3 (Apr-Jun)	Q4 (Jul-Sep)
Past Work												
Q2 Milestone: Develop metal oxide and nanomaterial coatings on flexible substrates												
Q4 Milestone: Evaluate sensor characteristics for flammable refrigerants												
Current/Future Work												
Q2 Milestone: Evaluate flexible sensor characteristics for flammable refrigerants												
Q4 Milestone: Establish sensor drift characteristics and calibration requirements												