Low Power Carbon Nanotube Field Emission Electron Source for Chemical Ionization Mass Spectrometry

HEMS 2015

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- **Introduction**
- **Background and Research Objectives**
- **Experimental Methods**
- **Results and Discussion**
- **Concluding Remarks**

Introduction

TSA Investment in Airport ETD Technology

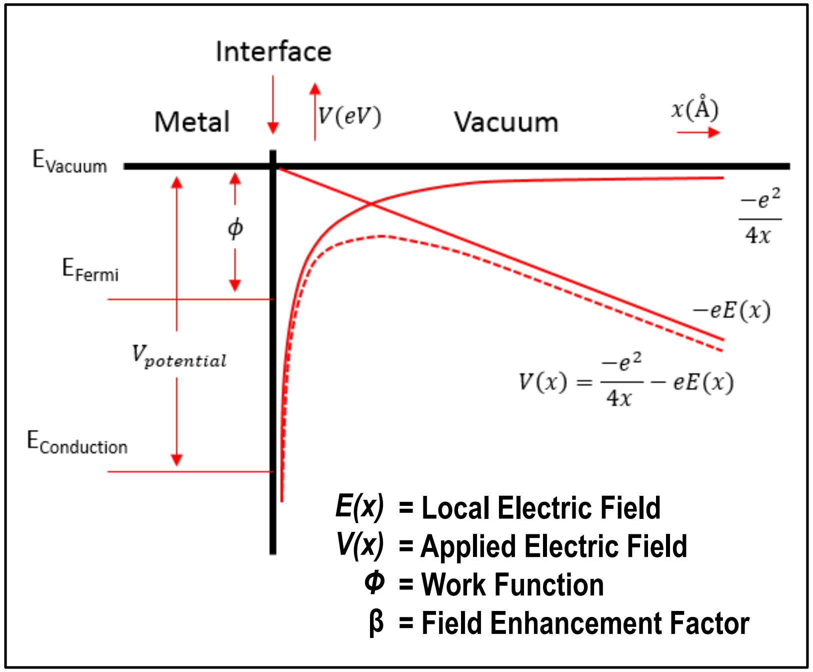
Year	Amount
2012	$15 million
2013	$39 million
2014	$84 million
2015	$140 million

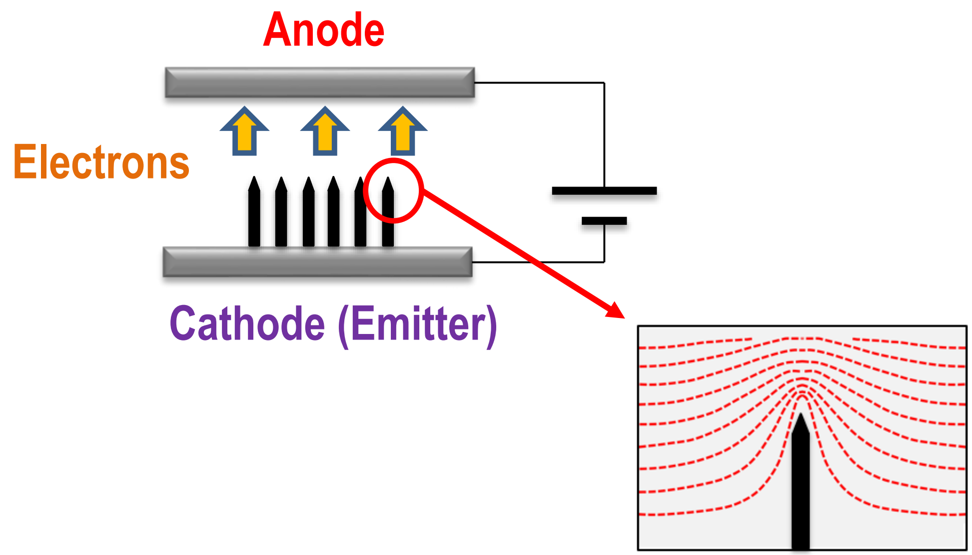
TSA = Transportation Security Administraton
ETD = Explosive Trace Detection

Source: House Report - Department of Homeland Security Appropriations Bills (2012-2015)

- Portable instrumentation can offer rapid and accurate ETD
- Desire to reduce maintenance and consumables.
- Replace filaments with carbon nanotube field emitter.
- Carbon nanotube field emission electron sources demonstrate improvements in the following areas:
  - Power requirements
  - Cycling capability
  - Lifetime

Carbon Nanotube Field Emission





Spatial distribution of E-field potential near a sharp tip

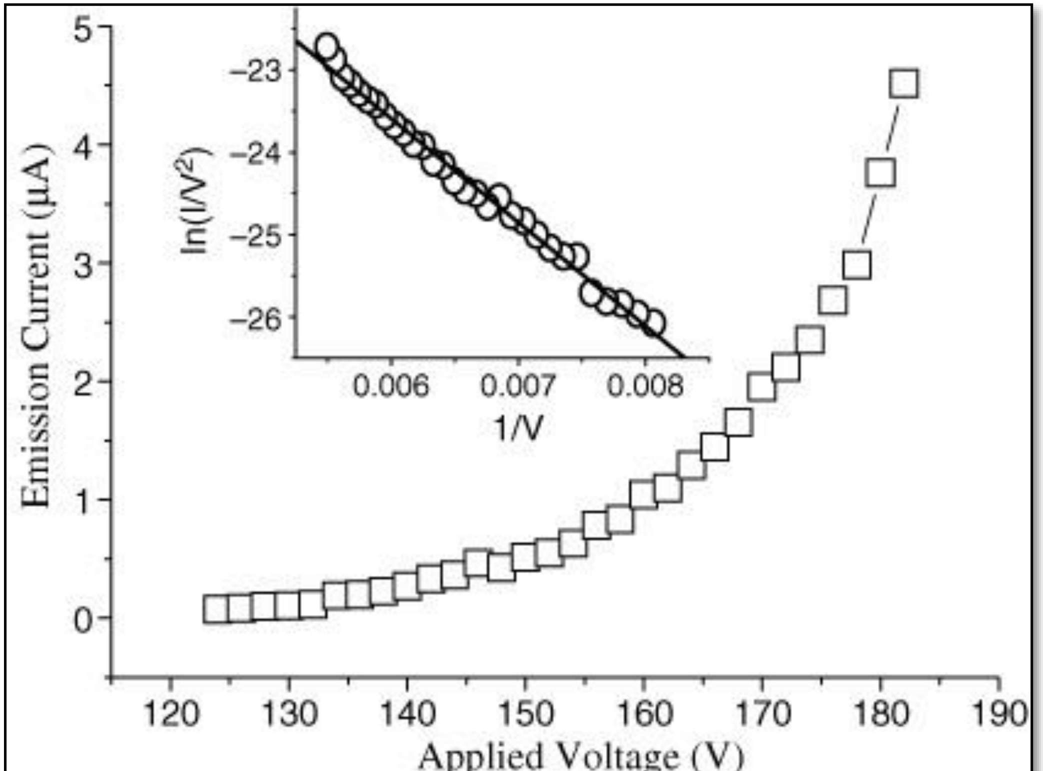
Advantages of CNT Emitters

High electrical conductivity	High chemical & mechanical stability	High current densities	High thermal conductivity
Large aspect ratio (>10,000) = High β values	Large surface migration activation energy	Low Thermal Expansion Coeff.	Very high tensile strength

Fowler-Nordheim (F-N) Model and CNT Field Emitters

$$J = \frac{1.54\times10^{-6}}{\phi}\left(\frac{V}{d}\right)^2\beta^2 \exp\left(\frac{10.4}{\sqrt{\phi}}\right)\exp\left(\frac{-6.83\times10^{9}\phi^2 d}{\beta V}\right)$$

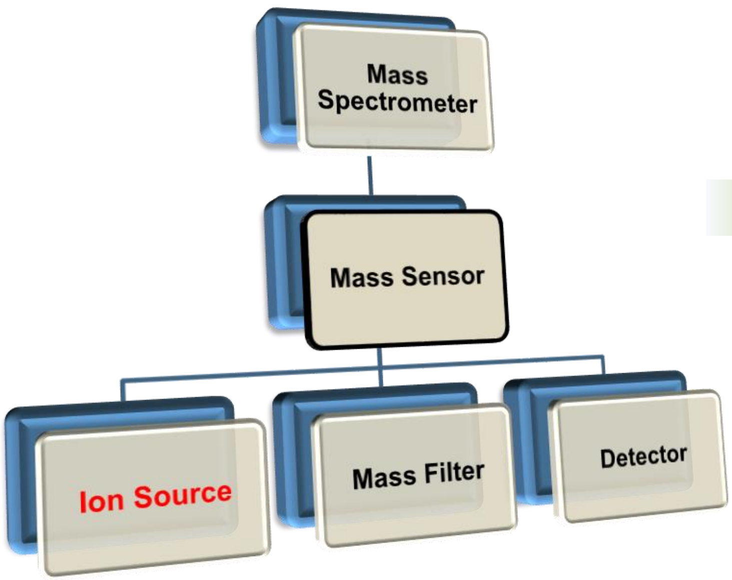
$$\ln\left(\frac{J}{E^2}\right) = \ln\left(a\frac{\beta^2}{\phi}\right) - b\,\frac{\phi^{3/2}}{\beta E}$$

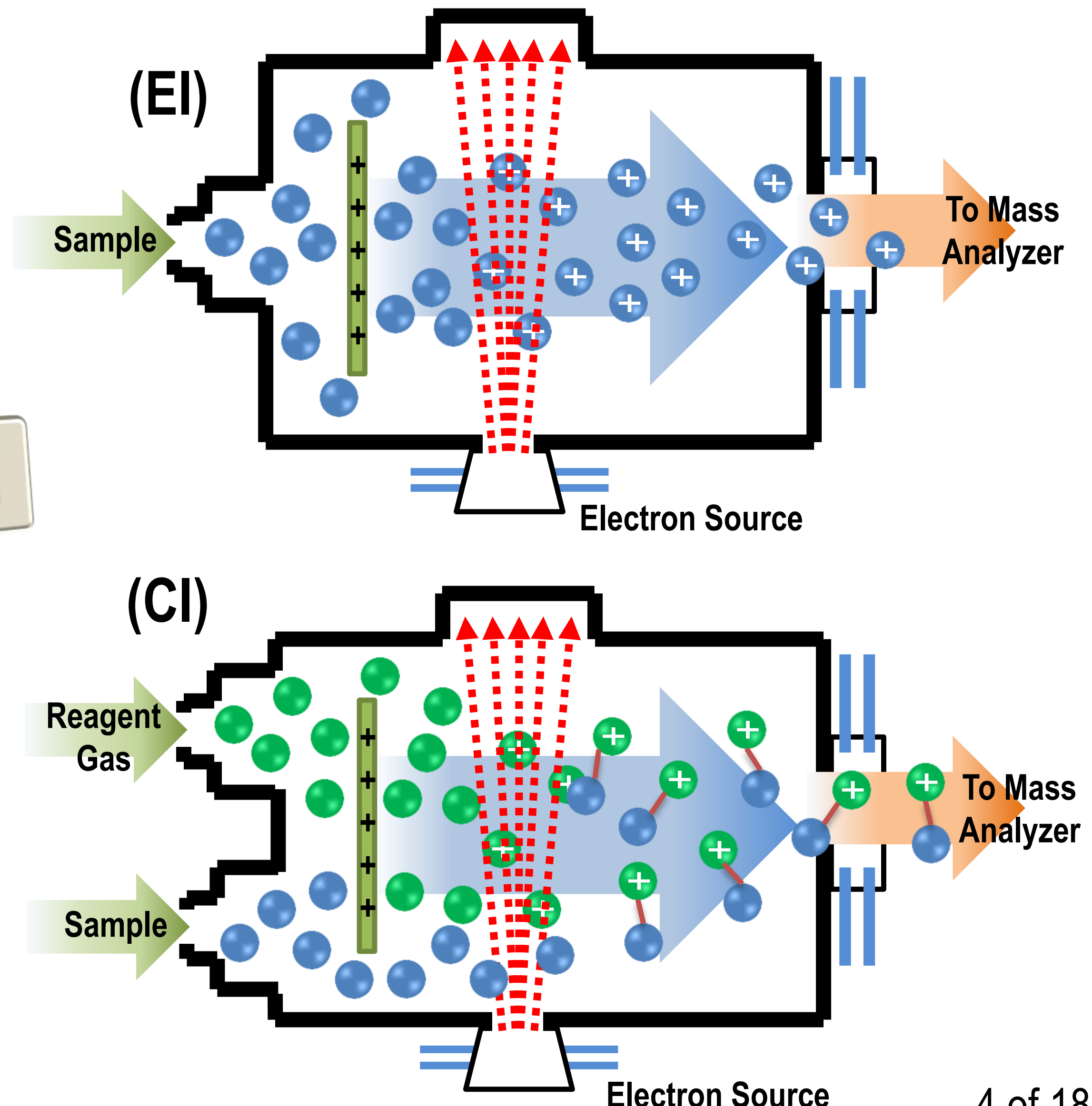


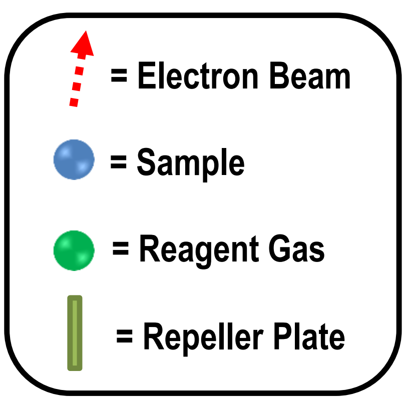
- A relationship between J, β, φ, and E can be developed using Schrodinger equation
- F-N model is used to characterize CNT field emitters

Xu et al, J. Phys.: Condens. Matter v17 pp L507-L512 (2005)

Mass Spectrometry: Electron Ionization (EI) & Chemical Ionization (CI)

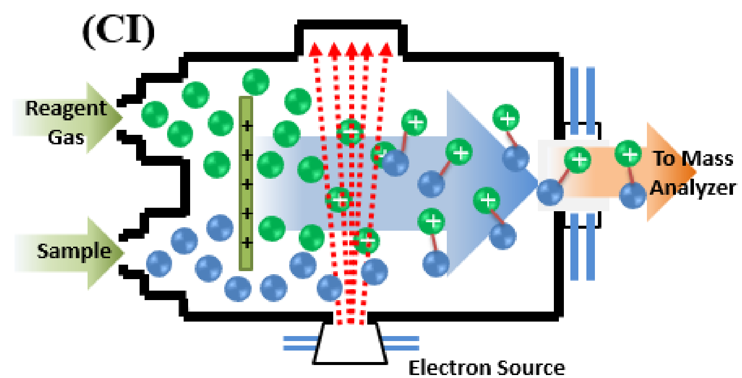






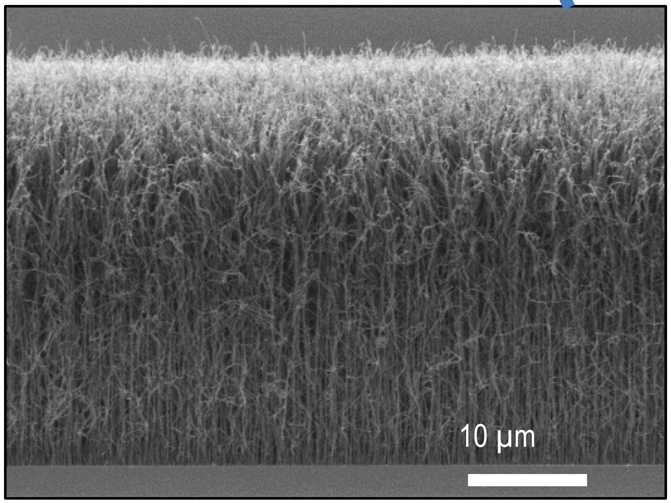
Integration and Evaluation Goals

1. Design robust & replaceable CNT electron source
2. Characterize and optimize performance

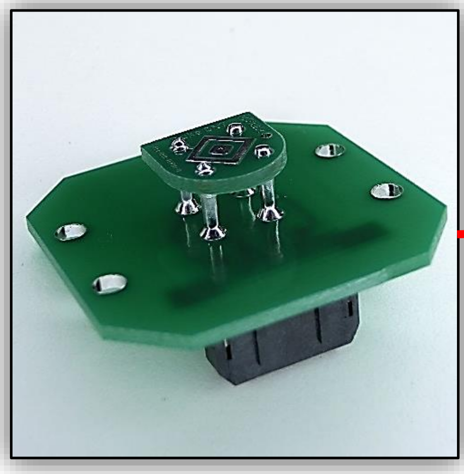


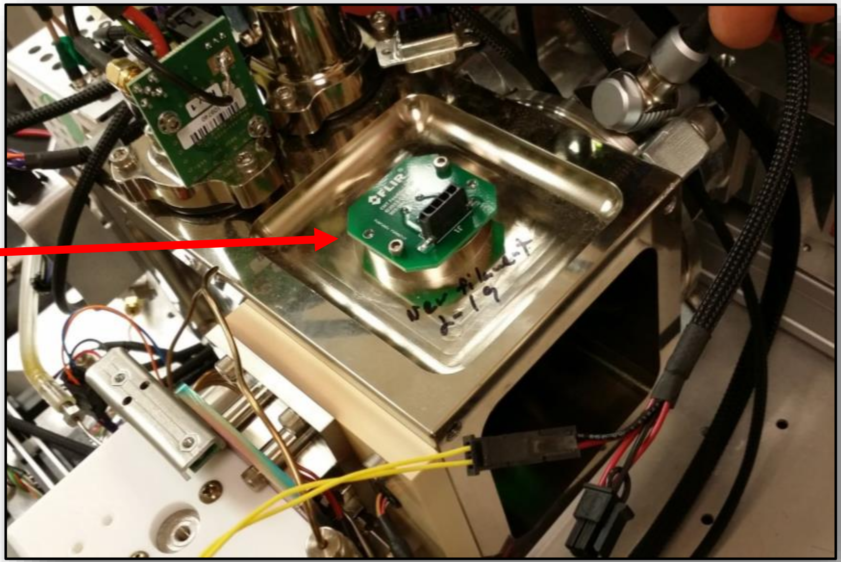
1. Compare CNT field emitters to a filament
2. Evaluate CNT source in a CI Mass Spectrometer

Carbon Nanotube Forest



Electron Source Platform

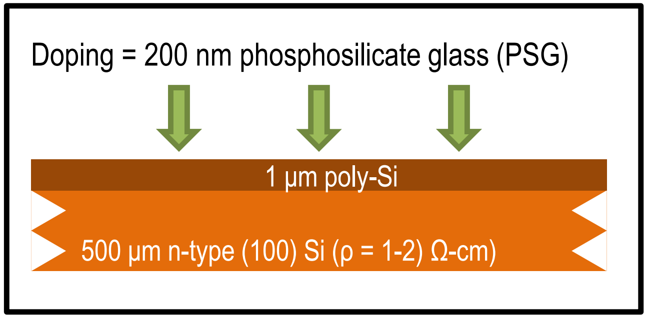


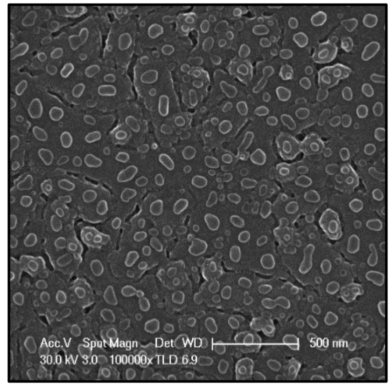


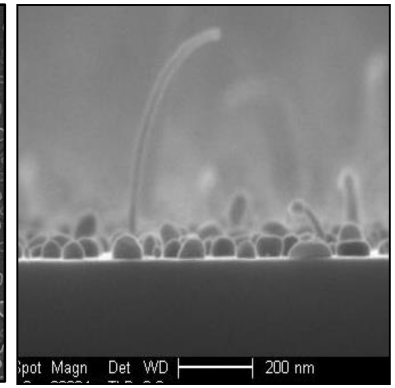
Platform design considerations:

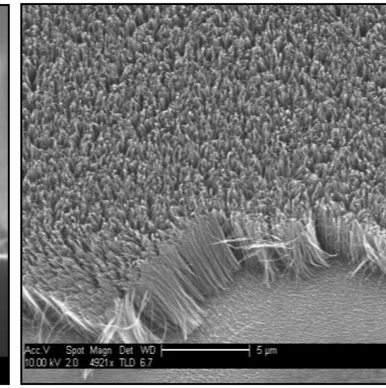
- Can be integrated into existing system
- Durable, can withstand voltages and pressures needed
- Straightforward assembly and replacement
- Minimal effects on field emission

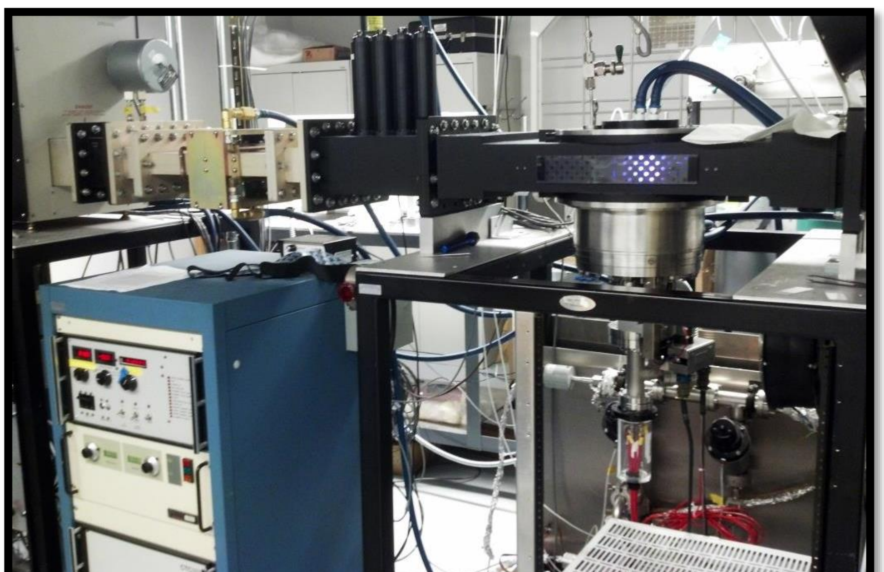
Substrate Design and CNT Growth







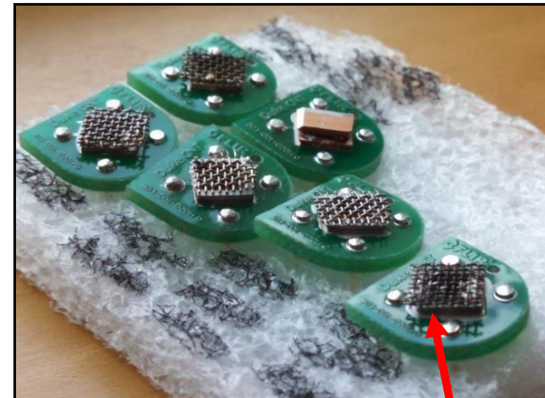


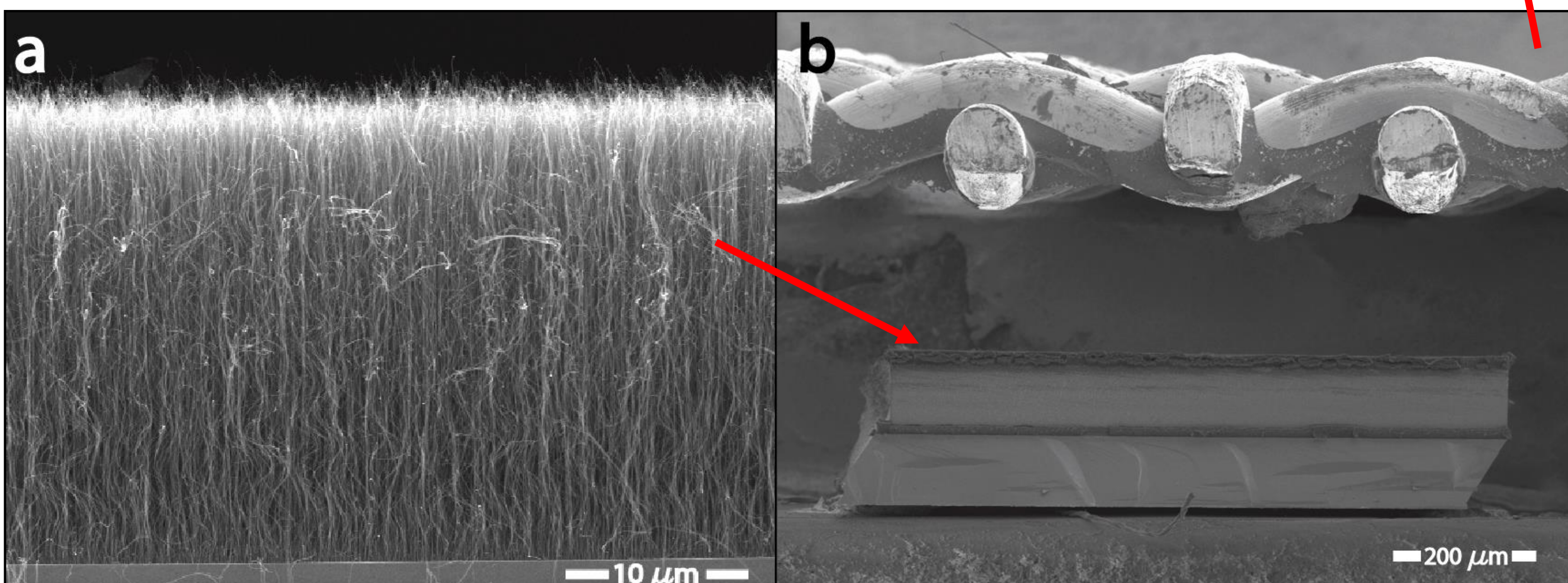


- Thin conductive poly-Si film used to reduce transport barrier
- Growth parameters tuned for improved catalyst control
  - Lower temperature
  - Shorter dwell times

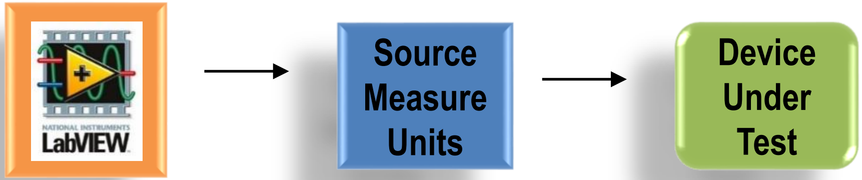
Adhesion and Field Emission Factors

- Investigated adhesion techniques for FE optimization
- Investigated various mesh materials
- Bulk testing provided means of FA and correction





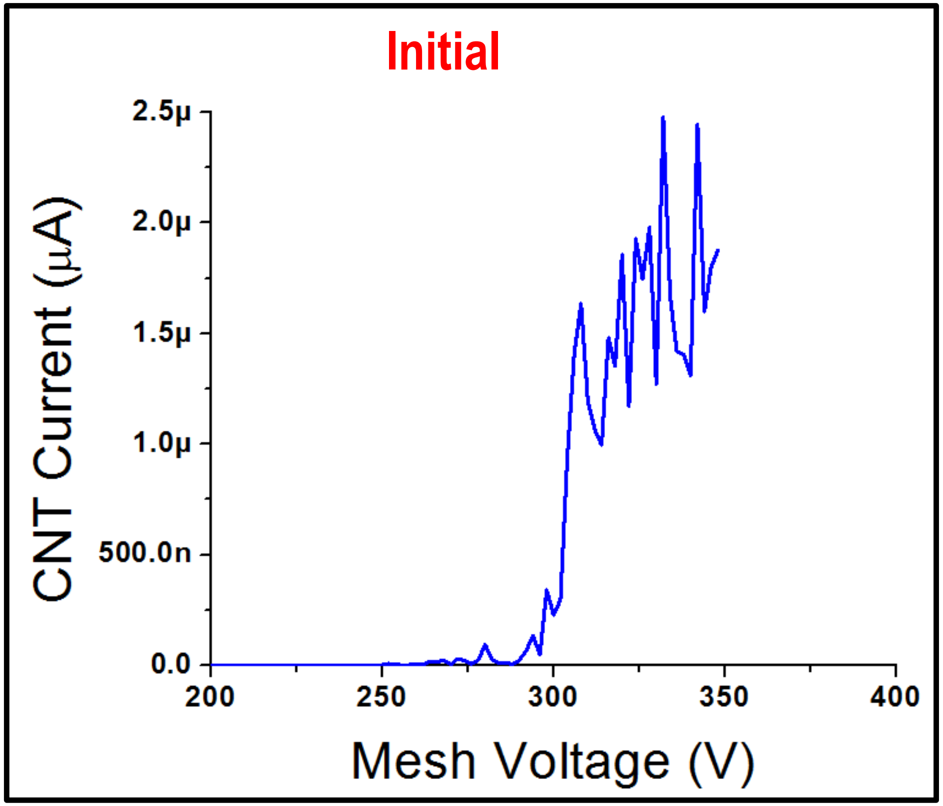
Ex-Situ Characterization

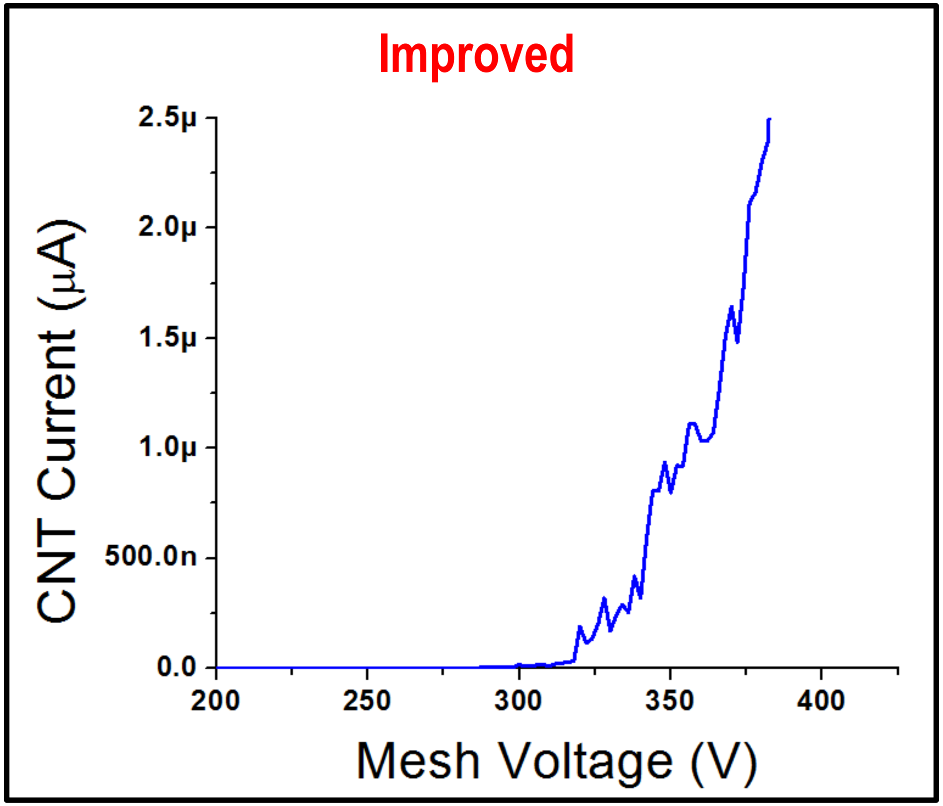


Test Bed Provides

- Controlled Environment
  - Pressure
  - Gas Species
- *I-V* Curve Analysis
  - V_{to}
  - V_{th}
- Lifetime Analysis
  - V controlled I

UHV *I-V* Characterization





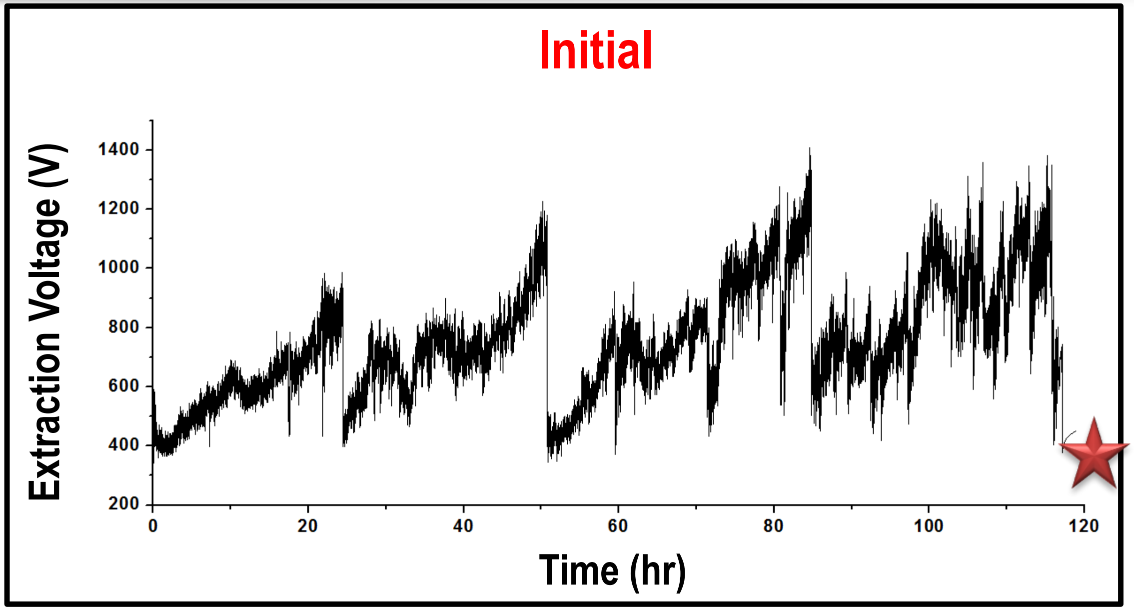
Enhanced substrates and improved CNT growth led to the following:

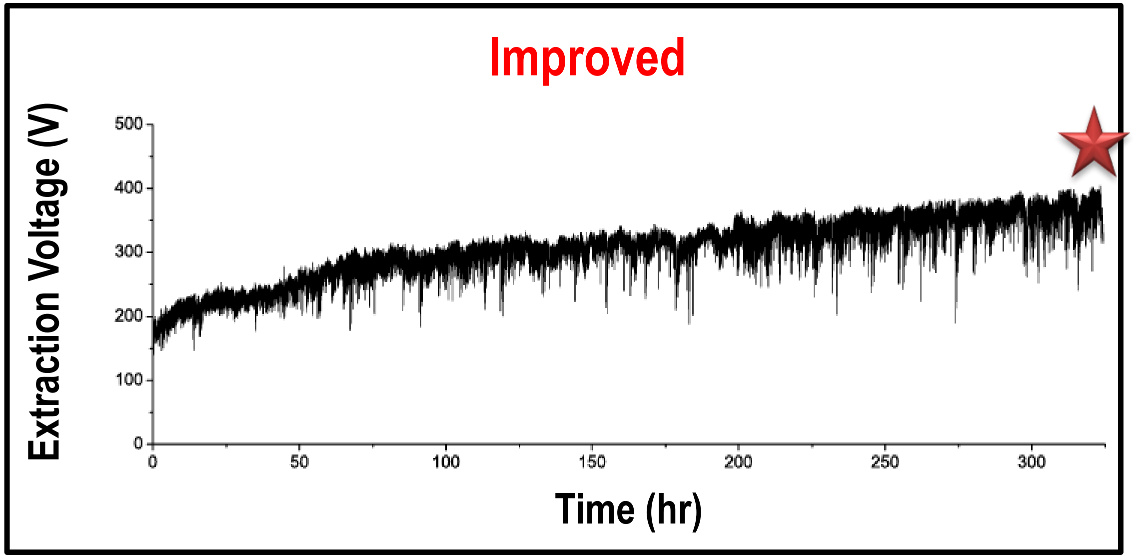
- Superior device *I-V* characteristics
- Better control of desired emission current

Lifetime Characterization

Fixed current tests of improved CNT emitters. Operated at 10^{-4} Torr of lab air and a fixed 5 μA CNT emission current

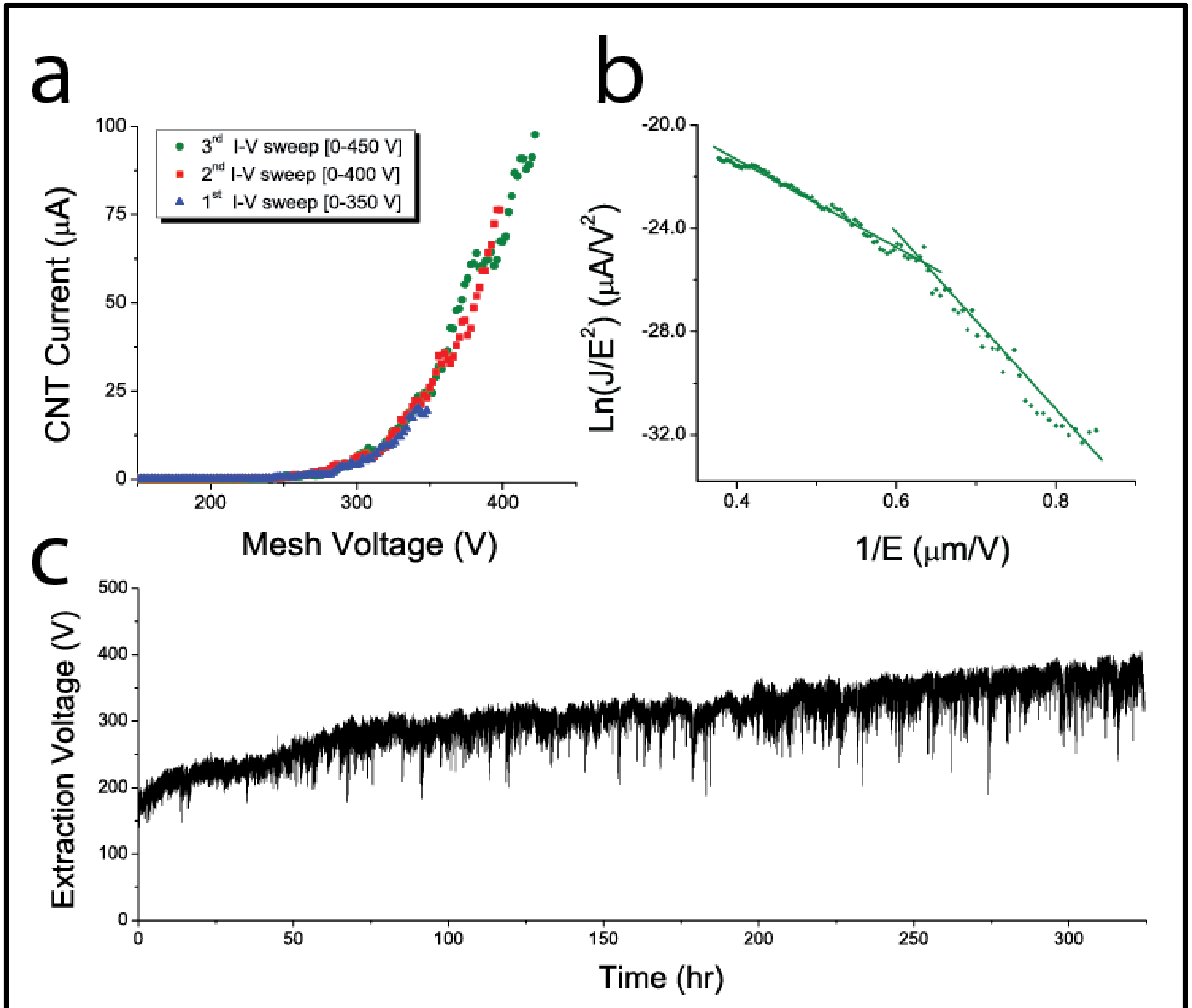
- ~3x lifetime increase over initial CNT emitters
- Decreased variation in emission current and extraction fields





★ = failure

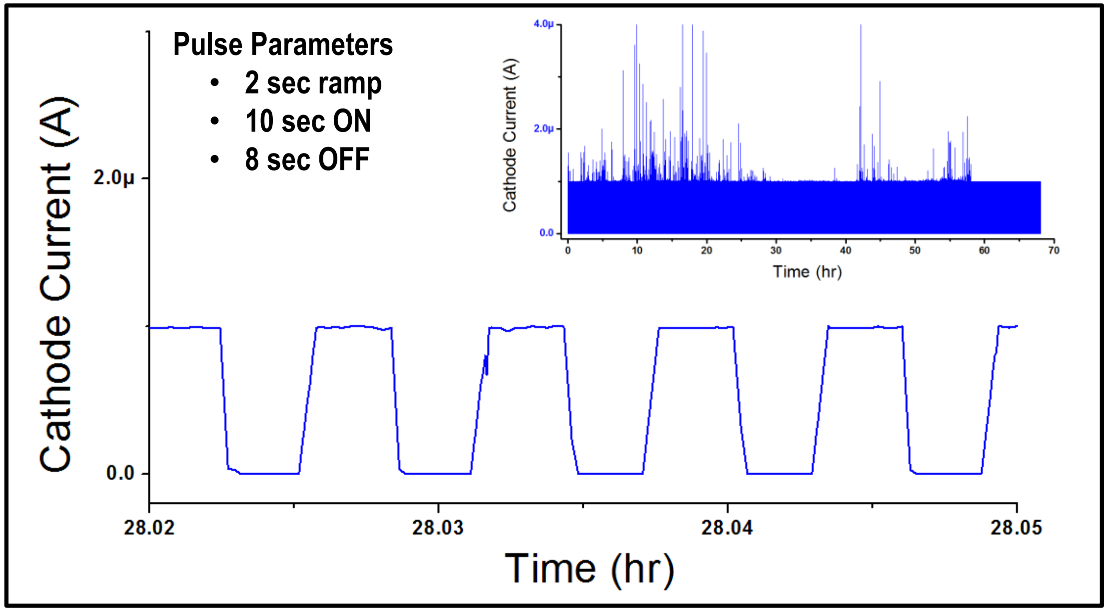
Performance Discussion

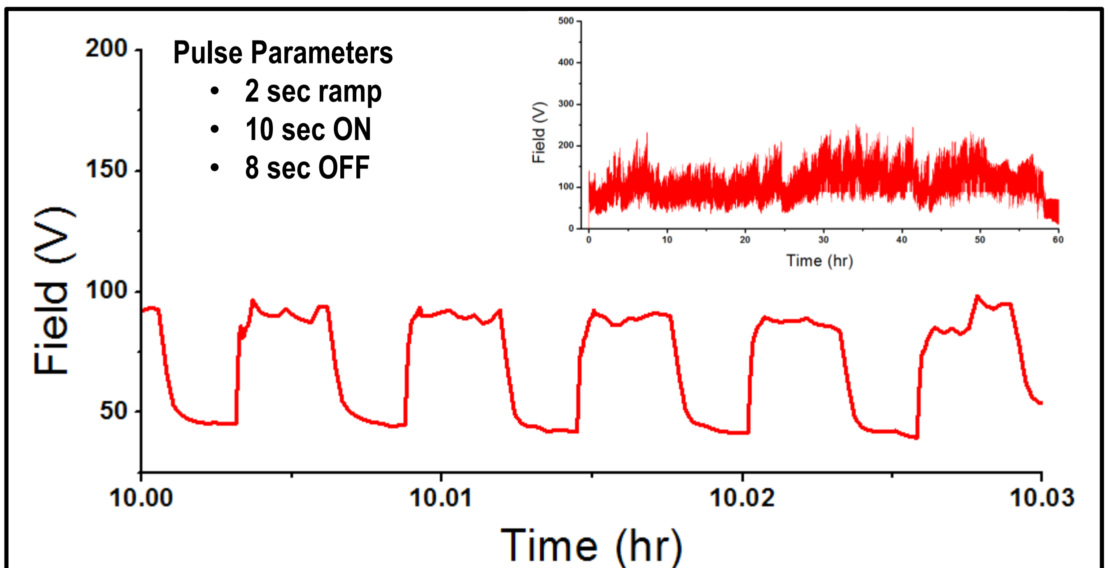


V_{to} = 1.02 +/- 0.026 V/μm
V_{th} = 1.16 +/- 0.029 V/μm

- Experimental optimization resulted in improved lifetime and superb performance
- I-V curves demonstrate excellent *I-V* performance
- Fowler-Nordheim plots show nonlinear characteristics, as expected

Pulsed Operation Discussion



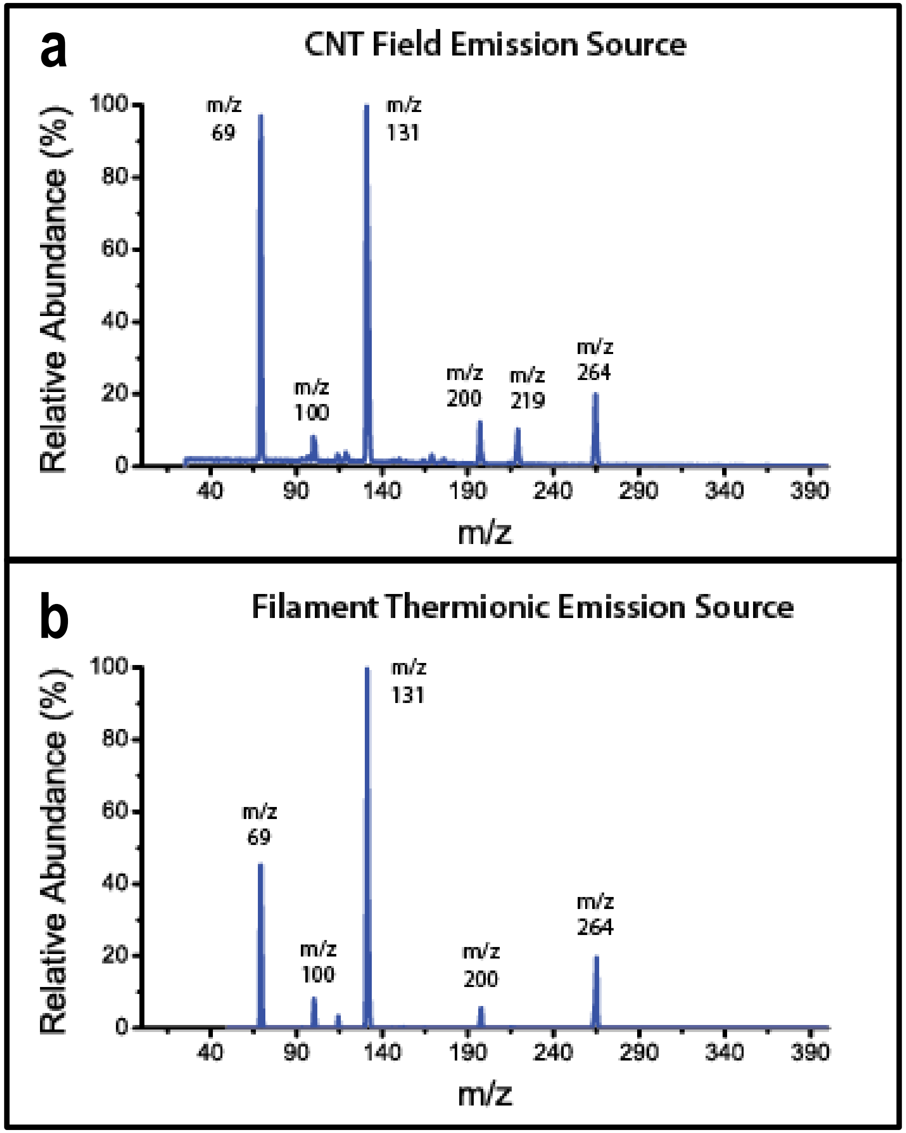


- Data indicates significant pulsing benefits over tungsten/rhenium filaments
- Cycling ON/OFF shows significantly less impact on lifetime compared to filaments
- Assuming one pulse equals sampling of one ticket, CNTs indicate lifetime of 450,000 tickets

<u>Pulsed CNTs have 100x filament lifetime</u>

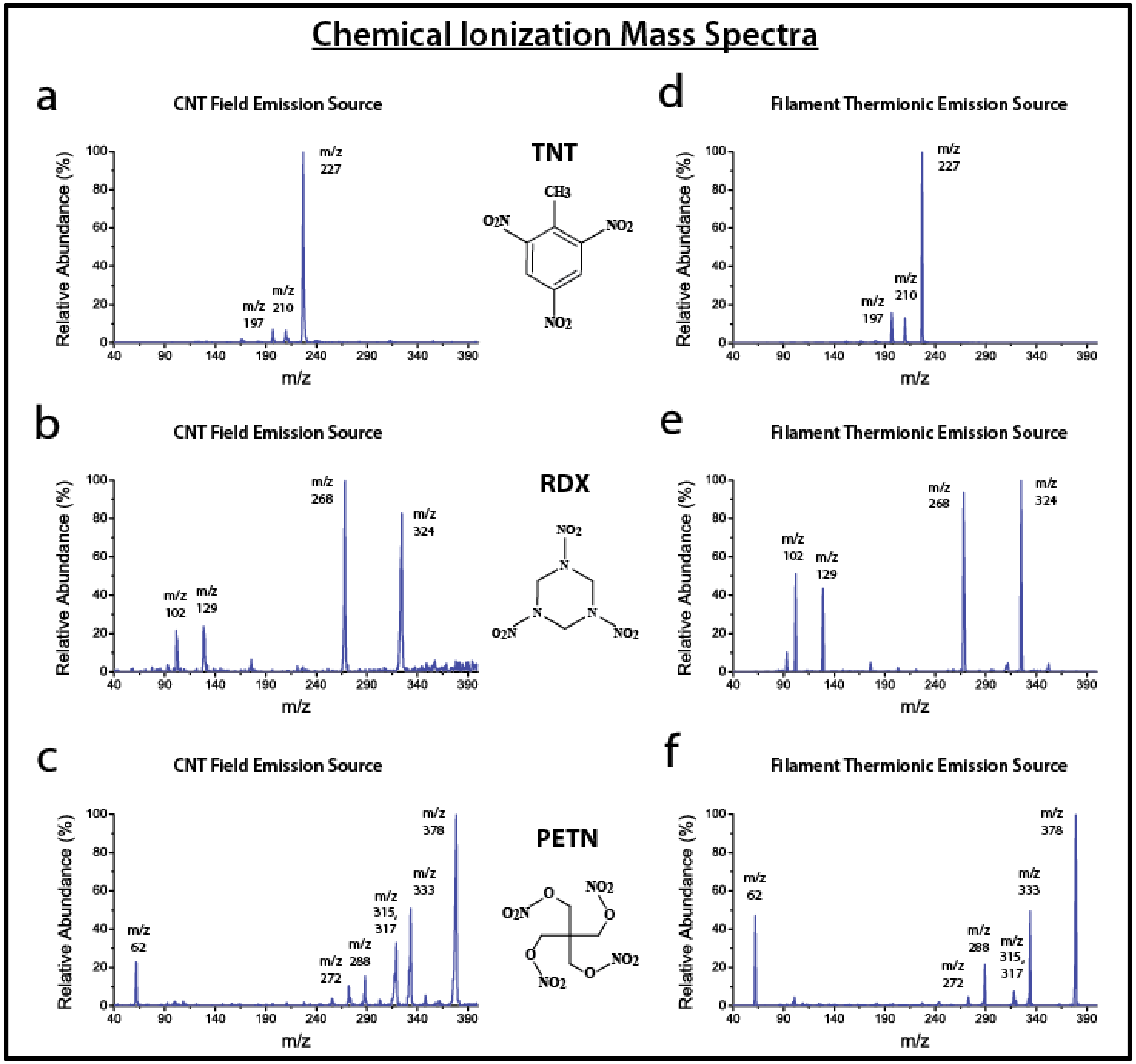
CNT and Filament Source Comparison - PFTBA

- Used standard calibration gas (Perfluorotributylamine [PFTBA])
- Initial electron ionization (EI) performed with both sources to calibrate system
- Good correlation between CNT and filament spectra in EI, positive chemical ionization (PCI), and negative chemical ionization (NCI) modes



* Spectra collected at FLIR Systems, Inc.

CNT and Filament Source Comparison – Trace Explosives



- Trace explosive spectra collected with CNT field emission source is nearly identical to spectra taken with the filament thermionic source
- Three common explosives were analyzed, calibration curves were generally linear as expected

Summary of Results and Discussion

- Designed & integrated a CNT field emission source in a chemical ionization mass spectrometry system

- Optimized CNT emitters to achieve low threshold voltages, long lifetime

- Showed benefits over standard thermionic filaments
    - Number of Tickets (Cycles)
    - Power
    - Lifetime

- Good agreement between spectra generated with filaments and CNT field emission source for electron ionization and chemical ionization spectra.

Closing Information

Future Studies

- Further improving electrical and mechanical characteristics with integration of thin film diffusion/adhesion layers

Publication

- Radauscher, E., A. Keil, M. Wells, J. Amsden, J. Piascik, C. Parker, B. Stoner, and J. Glass, Chemical Ionization Mass Spectrometry Using Carbon Nanotube Field Emission Electron Sources. Journal of The American Society for Mass Spectrometry, 2015
- dx.doi.org/10.1007/s13361-015-1212-0

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