Nano- and Microtechnology Development for Advanced Scientific Measurements throughout the Solar System

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Goddard Space Flight Center

NASA Centers (10):

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Nano and Microfabrication Facilities

SWCNT and MWCNT growth

Wet Chemistry Benches

Electron Beam Lithography

Physical Vapor Deposition Systems

Front/Back-Side Mask Aligners

Low Pressure Chemical Vapor Deposition

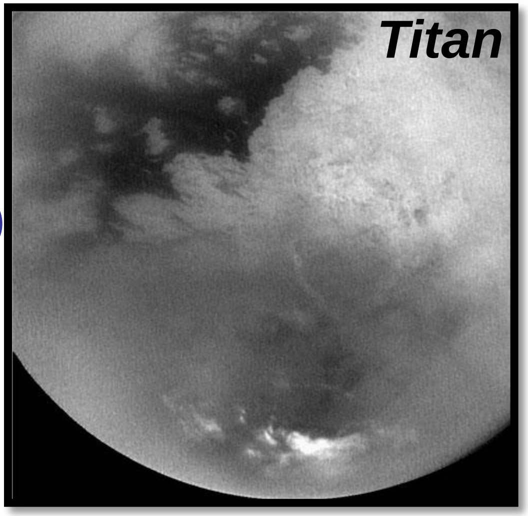
Reactive Ion Etch Systems

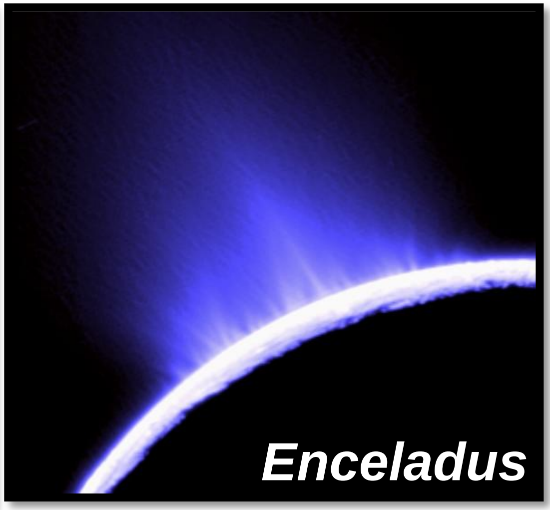
Scanning Electron Microscopy

Planetary Targets for *In Situ* Instruments

Mass Spectrometry

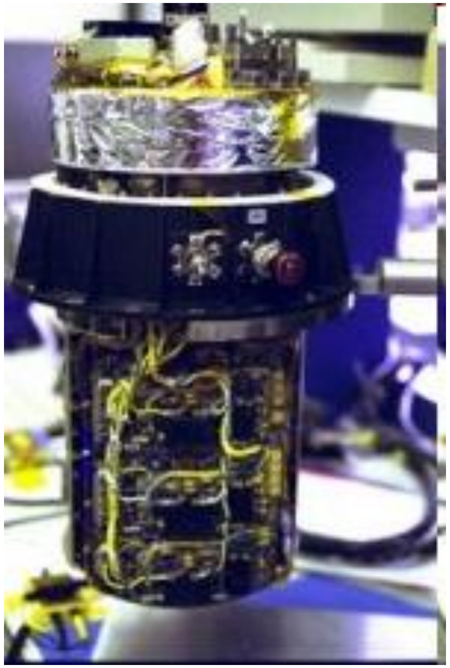
- Well established technique for in situ chemical analysis in planetary science missions
- Provides mass-to-charge ratio for every sample constituent – non-specific
- Can be interfaced to complementary analytical techniques for thorough sample characterization, e.g.
  - Pyrolysis (soil)
    Thermally evolved volatiles
  - Gas chromatography
    (atmosphere, pyrolysis products)
    Small, robust molecules
  - Liquid chromatography
    (soil extract, liquid)
    Complex molecules





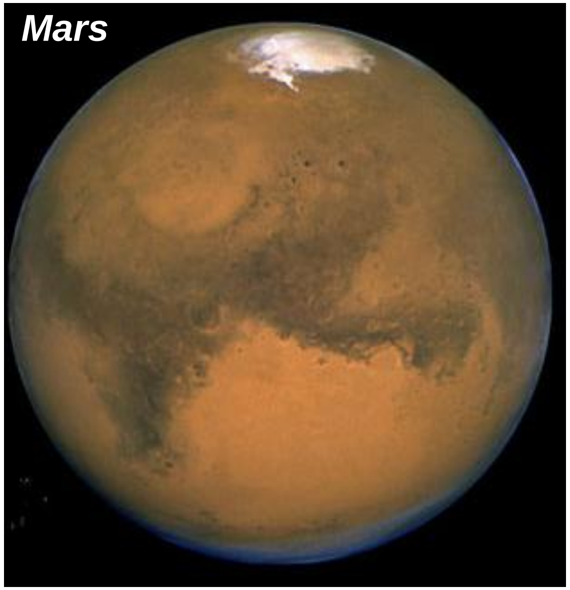
Planetary Mass Spec: State of the Art

Gas Chromatograph Quadrupole Mass Spectrometer
- part of Huygens lander
- Cassini mission to Saturn and moons



- Quadrupole mass filter uses AC fields to select transmission at a single mass
- Mass spectrum is acquired by scanning through operating range
- Thermionic filaments are used for electron impact ionization of gas

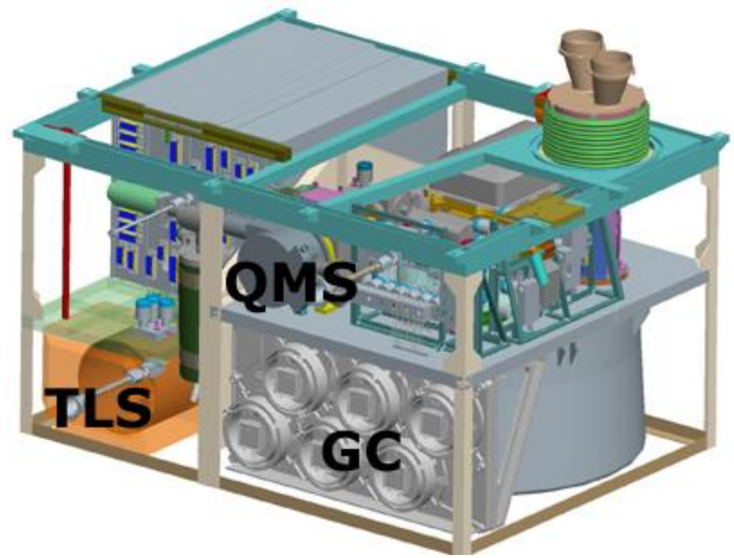
Planetary Mass Spec: State of the Art



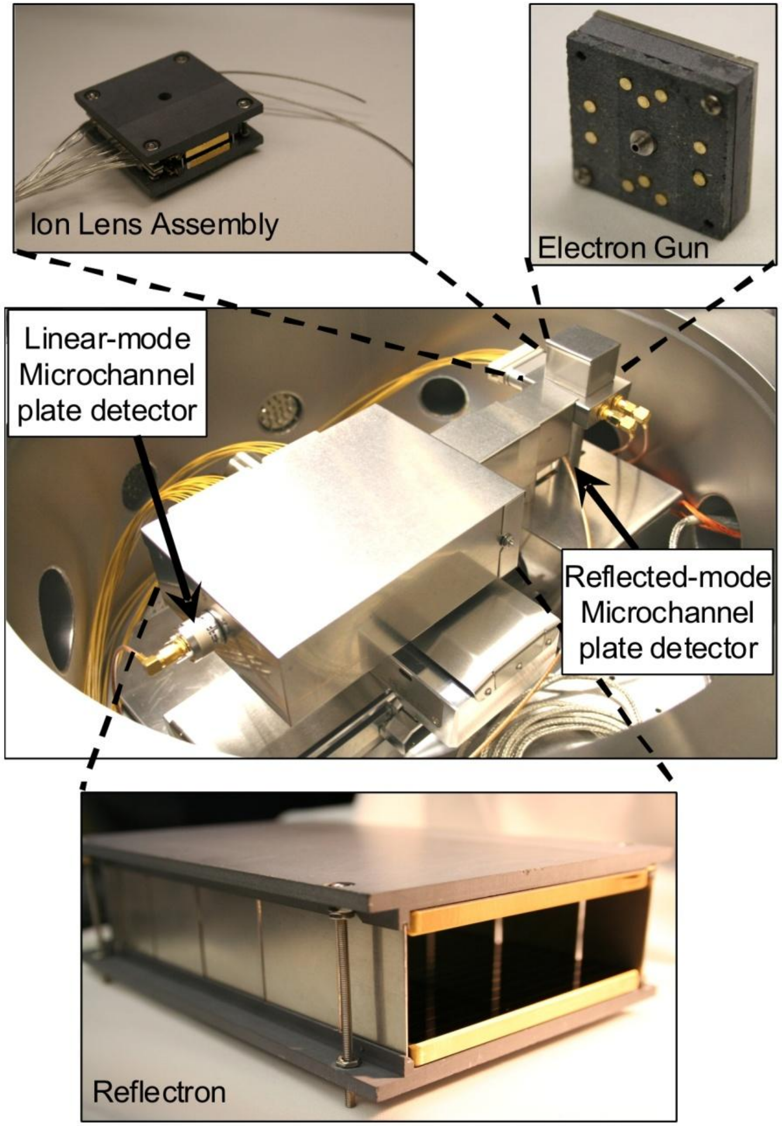
Pyrolysis Gas Chromatograph Quadrupole Mass Spectrometer

- part of Sample Analysis at Mars Instrument Suite
- Mars Science Laboratory rover mission

- QMS similar to Huygens
  - m ~ 1.3 kg, P ~14.5 W
- Thermionic filaments are used to ionize pyrolysis products or atmospheric gases

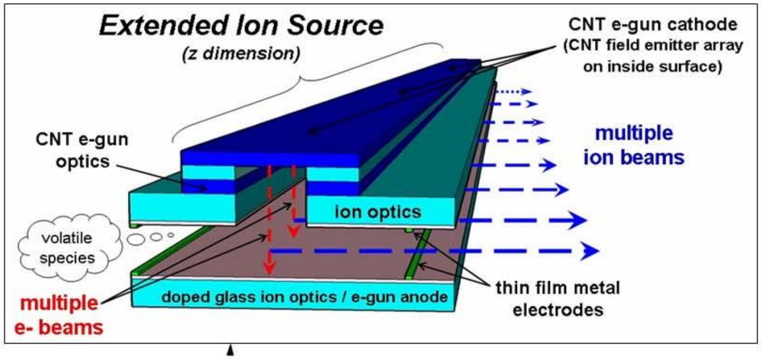


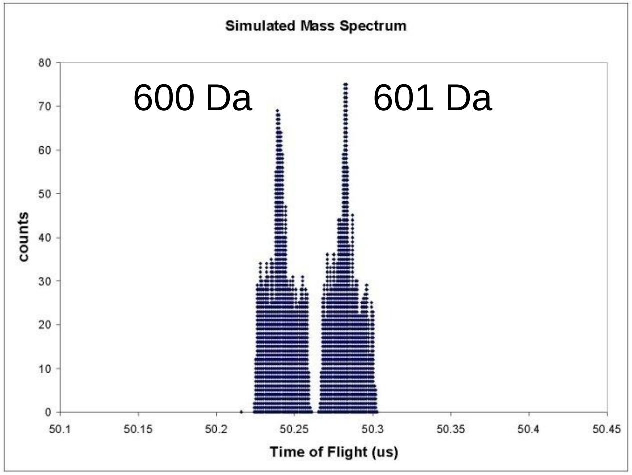
In Development: Miniaturized Time-of-Flight Mass Spectrometer

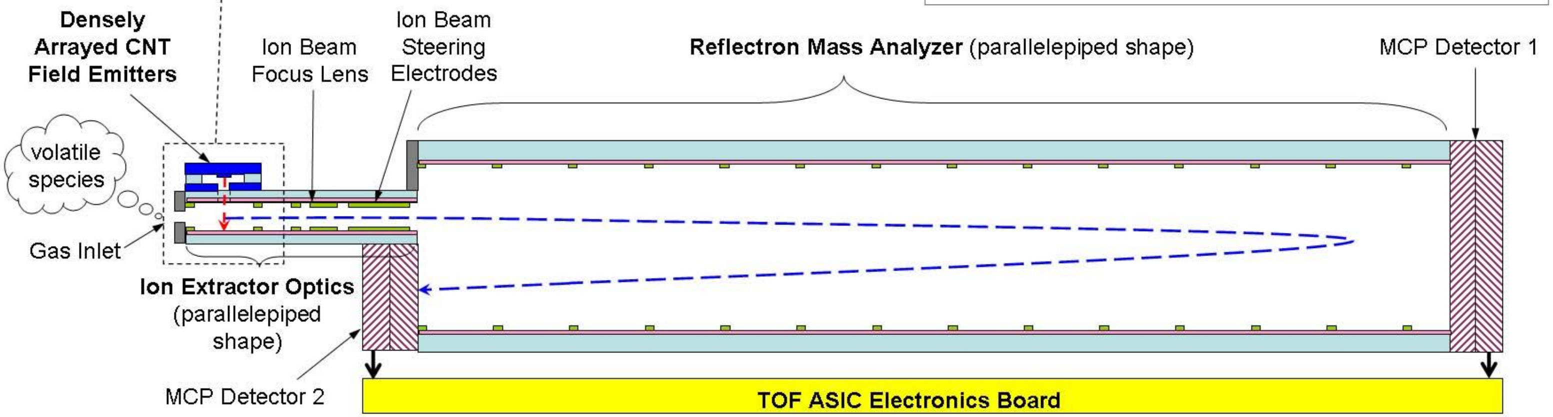


- Time-of-Flight Mass Spectrometer
  - Field emission **Electron Gun** for electron impact ionization
  - Ions are accelerated to a uniform kinetic energy in the **Ion Lens Assembly**
  - Heavier masses travel more slowly than light ones in the **Reflectron** analyzer
  - Arrival of isomass ion packets is registered as a function of time at the **Microchannel Plate Detector**

Ionization for Time-of-Flight Mass Spectrometer





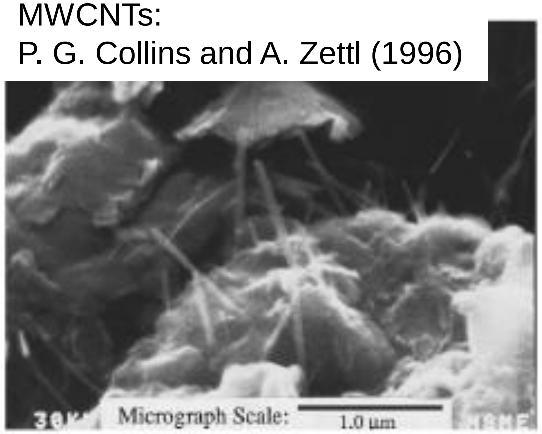


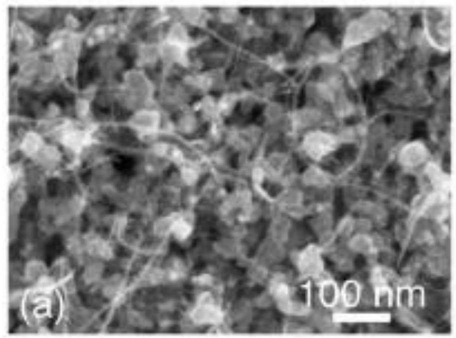
Previous Work: Field Emission in CNTs

- SWCNT, MWCNT, and CNF
  - Various growth techniques

Our approach:
Tower x-section << spacing

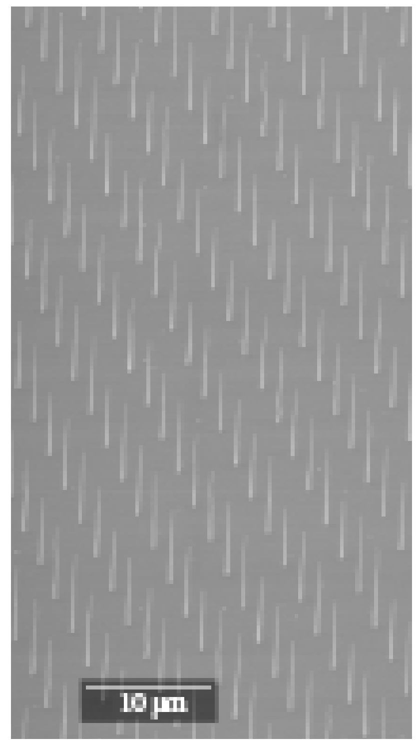
MWCNTs:
P. G. Collins and A. Zettl (1996)



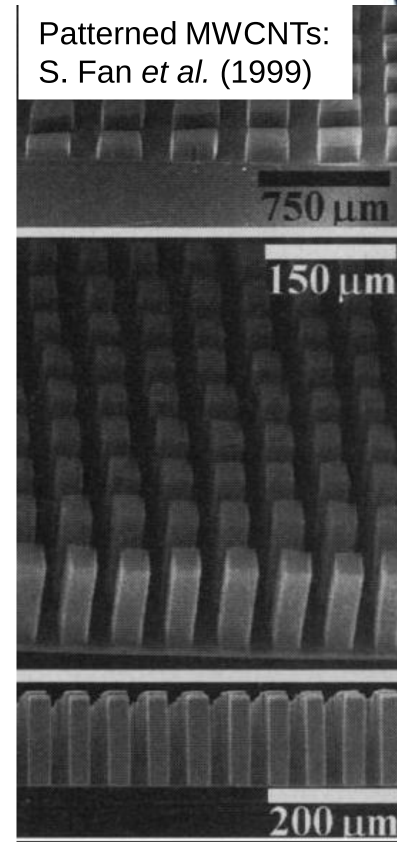


SWCNTs:
J.-M. Bonard, *et al.* (1998)

CNFs:
E. Minoux *et al.* (2005)



Patterned MWCNTs:
S. Fan *et al.* (1999)



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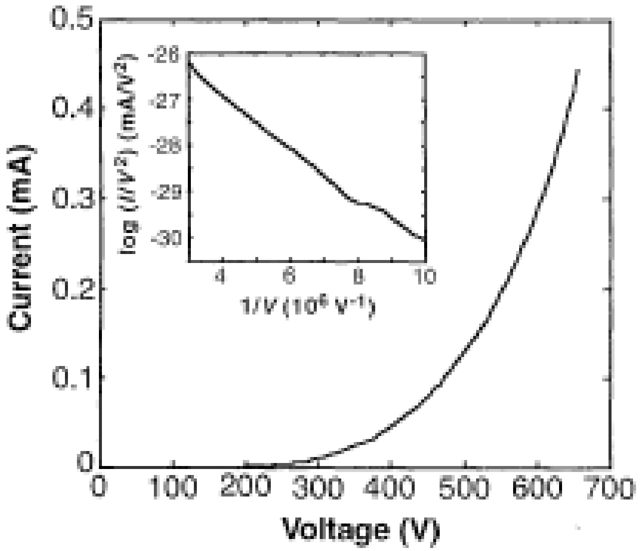
Previous Work: Field Emission in CNTs

- Fowler-Nordheim Tunneling
  - –Field enhancement factor
  - –Reported values 400-1200 (for MWCNTs)

W. A. de Heer, A. Chatelain, D. Ugarte (1995)



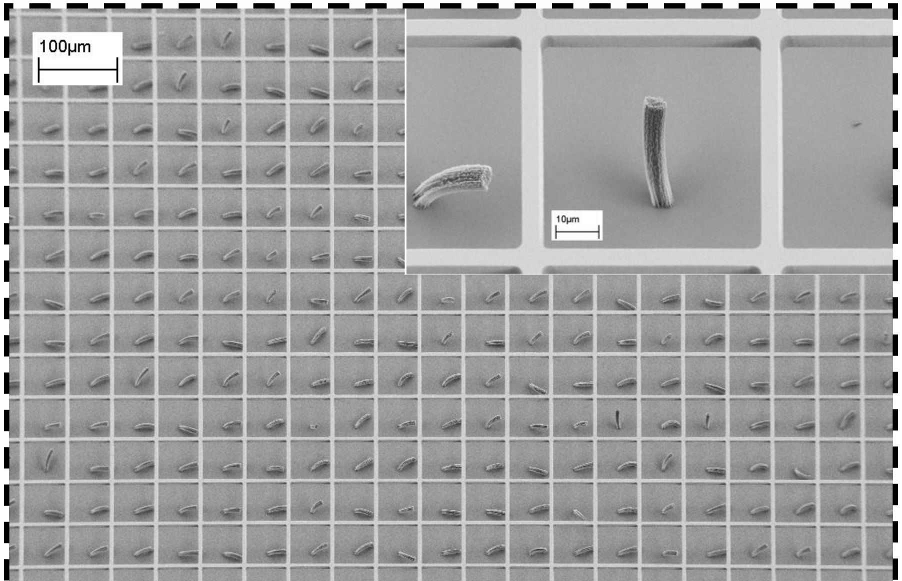
$$J = K_1 E^2 \exp\left(-\frac{K_2}{E}\right)$$

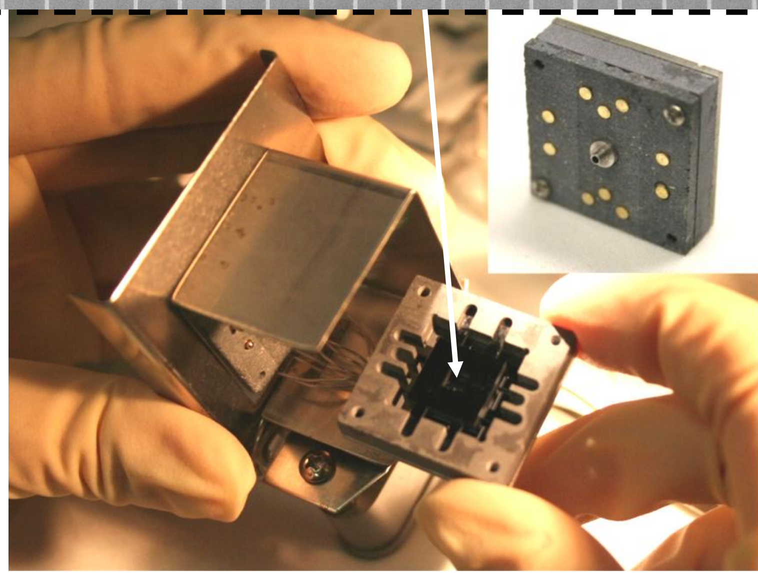
$$K_2 = \frac{B\Phi^{3/2}}{\beta}$$

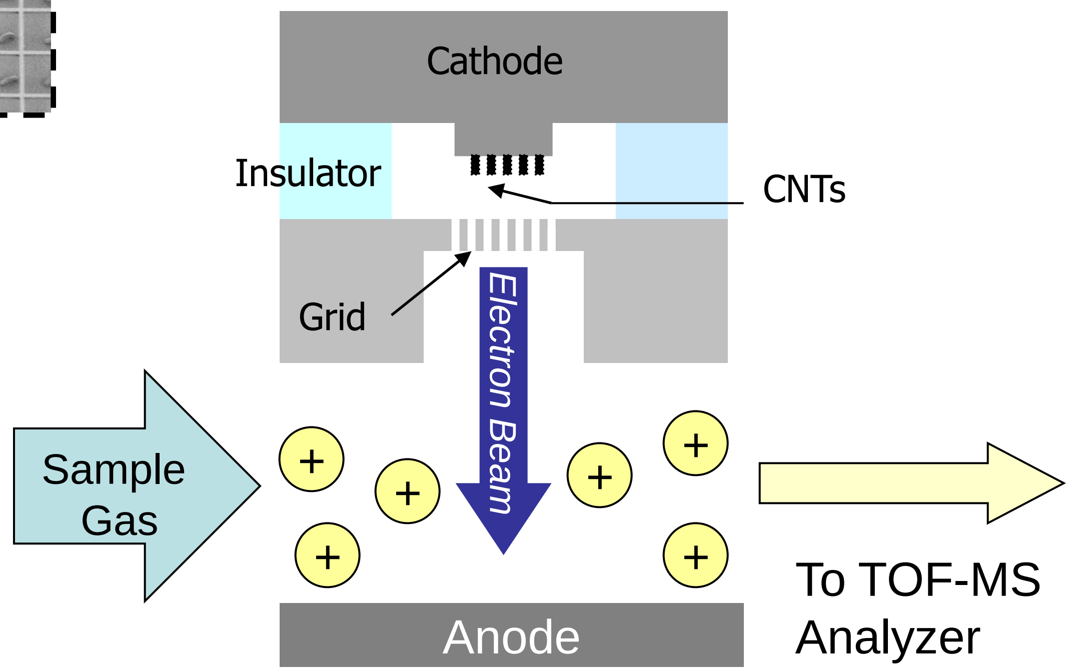
Carbon Nanotube Electron Gun

Carbon Nanotube field emitters

- Low power (100x < thermionic)
- Scalable for high sensitivity, redundancy
- Efficient ionization of the parent species
- Challenges: Lifetime and stability

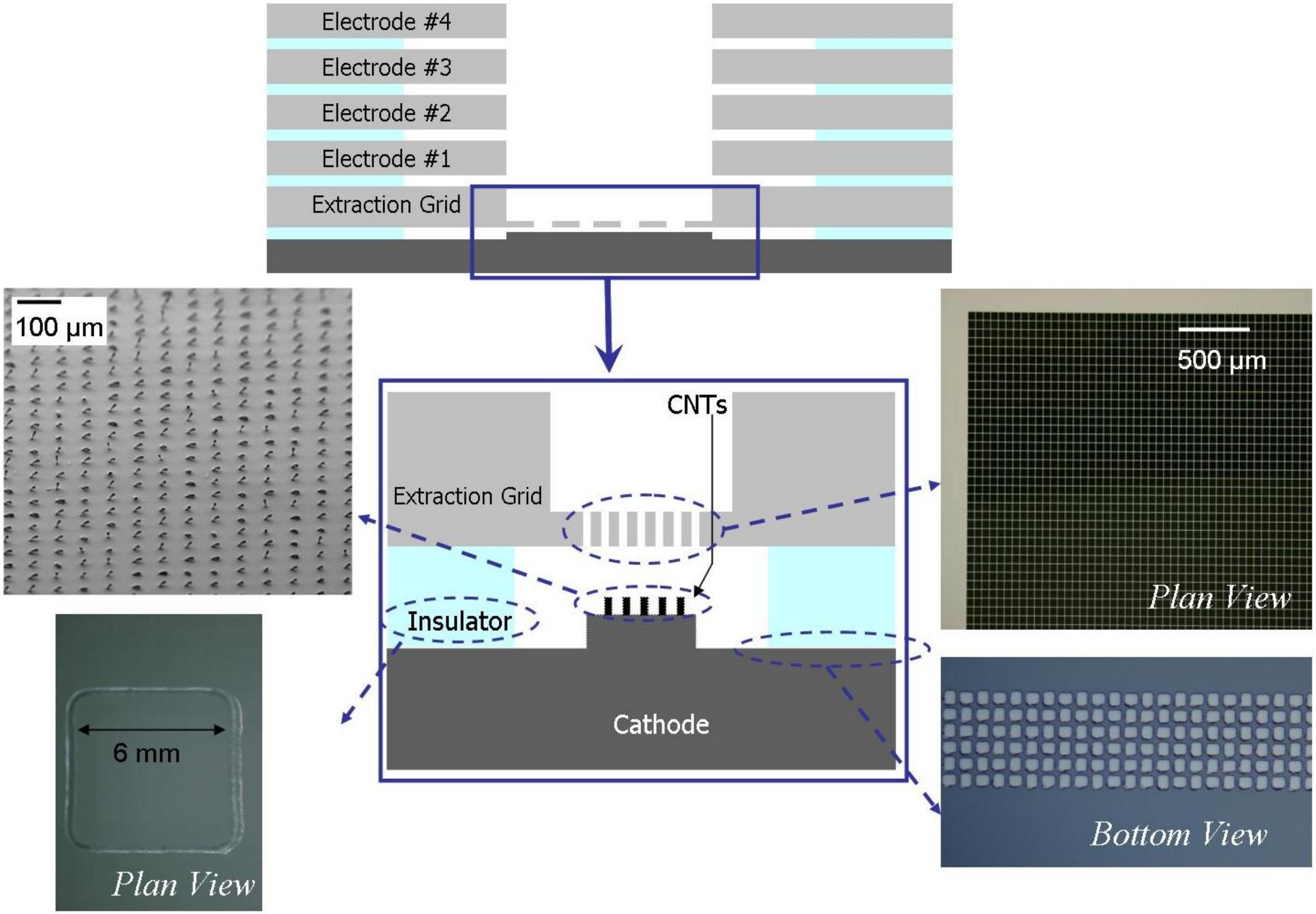






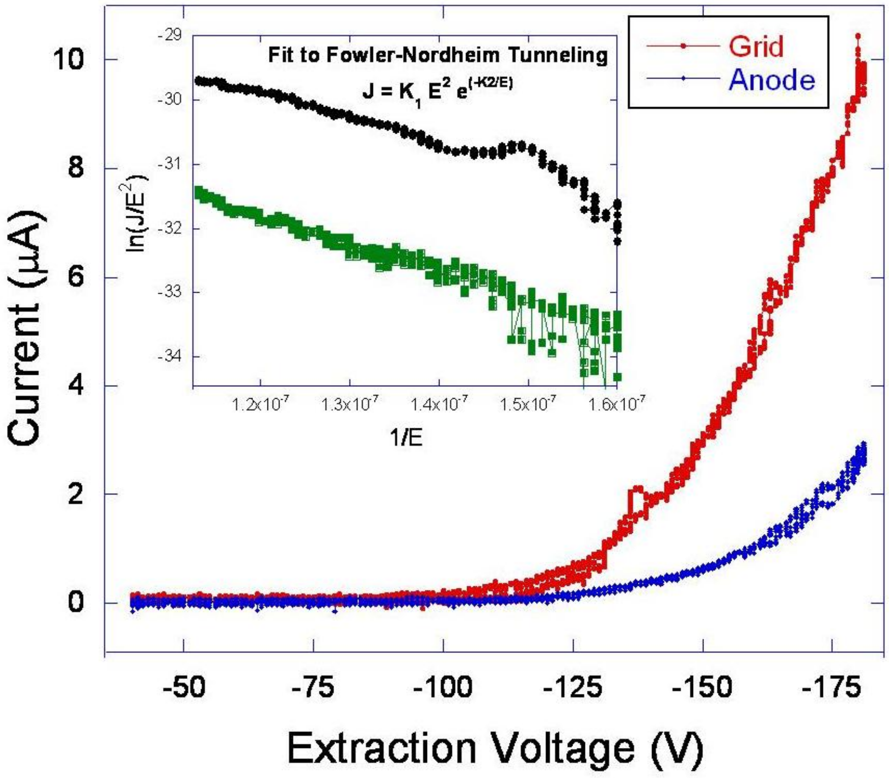
Recent Work: Assembly and Integration

Cathode-grid integration



CNT E-gun for Mini Mass Spec

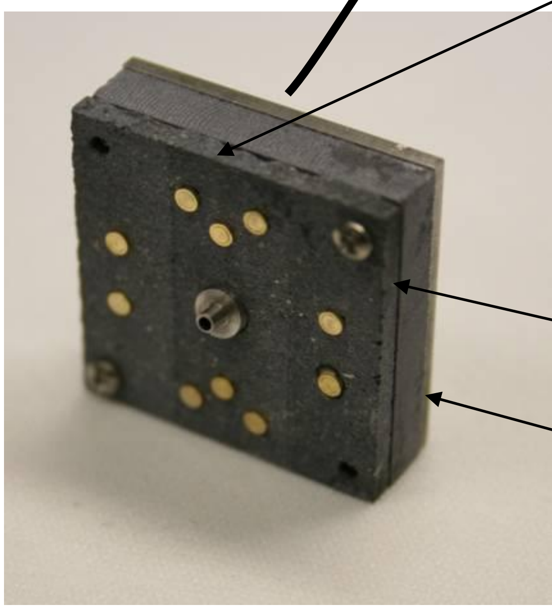
- Low operating voltages achieved for efficient gas ionization
- Fowler-Nordheim behavior confirmed
  - Field enhancement factor ~ 900
- Microamps of transmitted current in triode mode
- Persists for several hundred hours in high vacuum

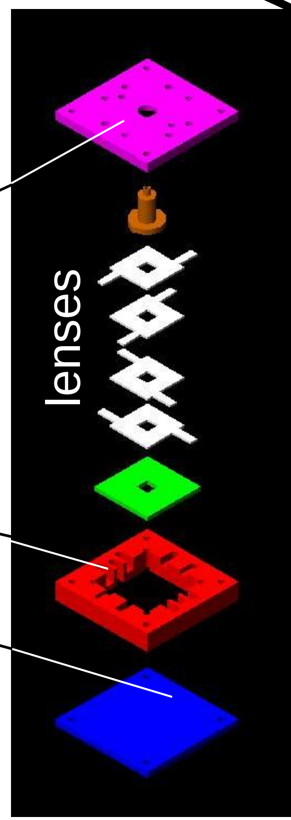


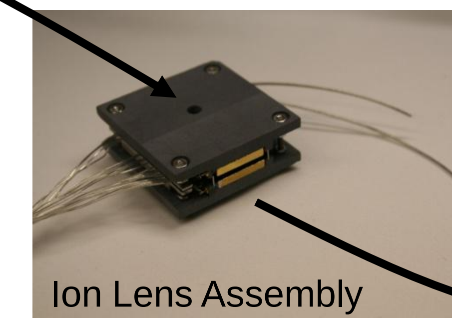
CNT E-gun for Mini Mass Spec

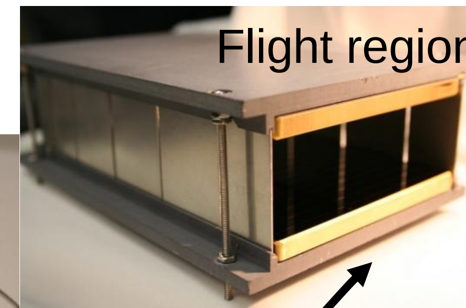
MEMS Integration of CNT e-gun for clean packaging

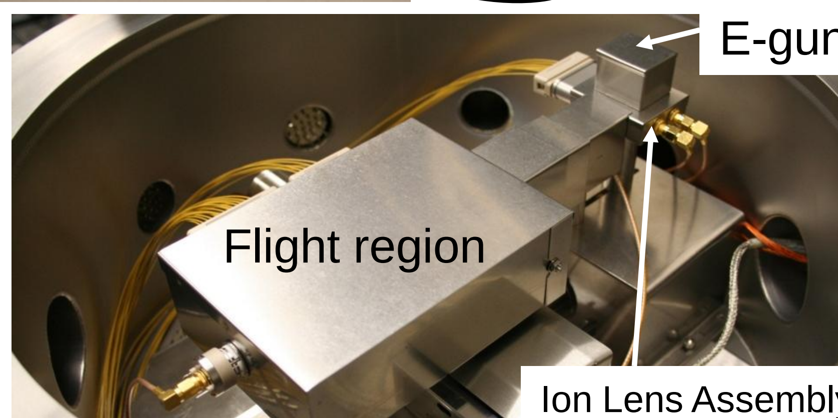
- Long lifetime
- Reduced current noise
- Reliable fabrication



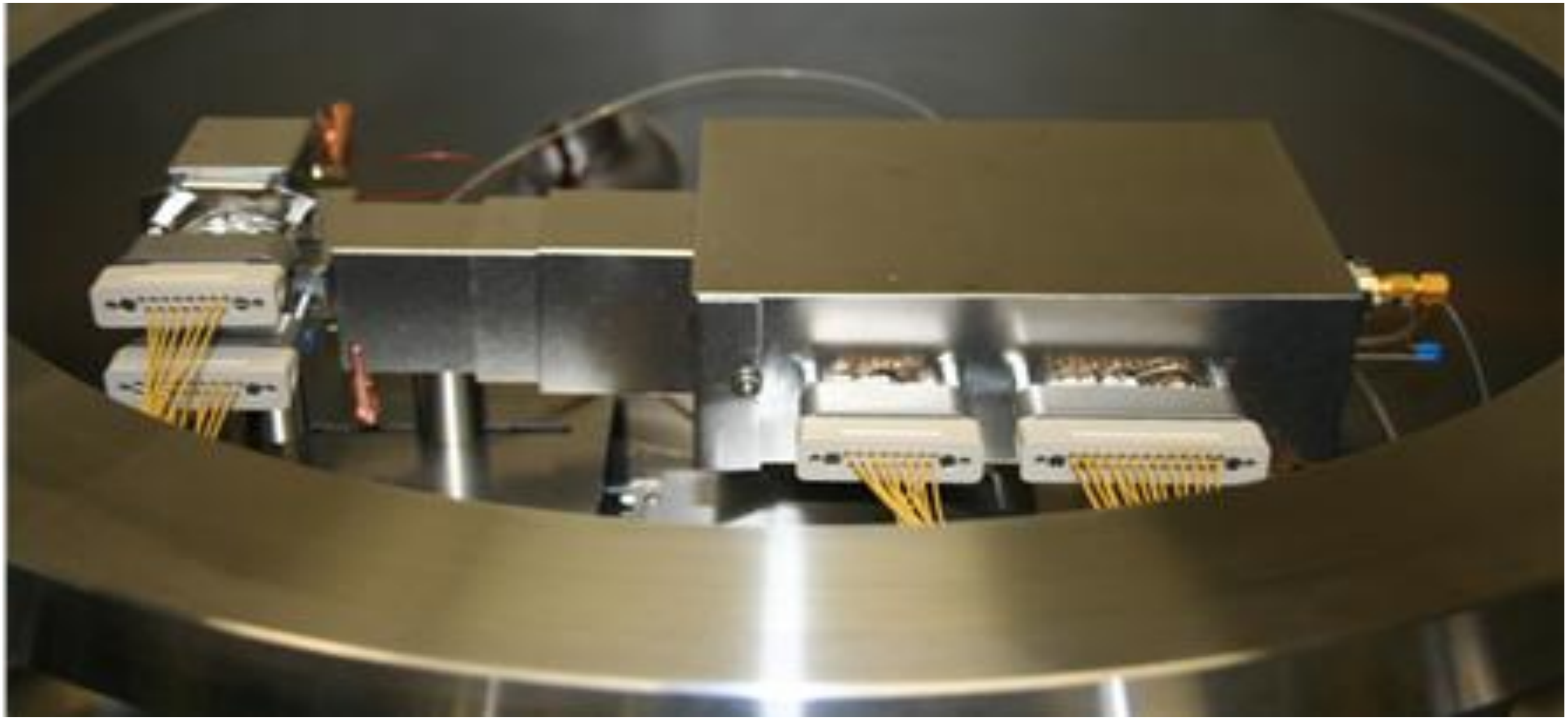






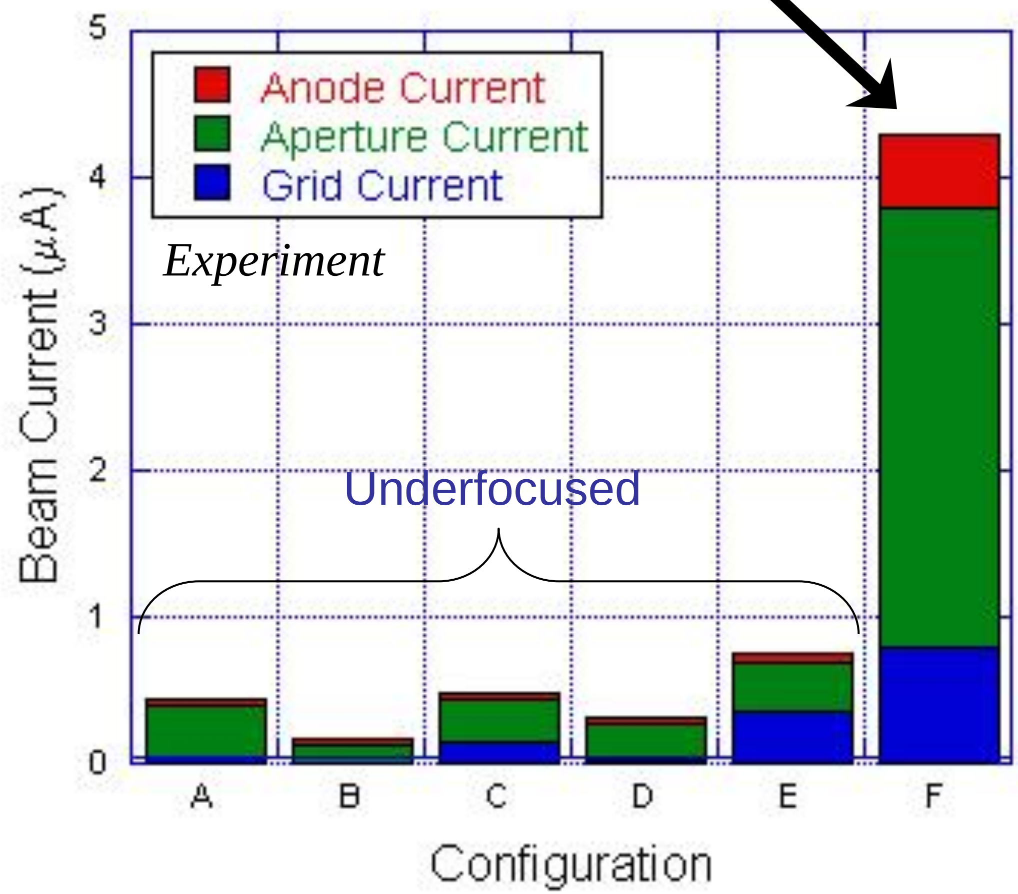


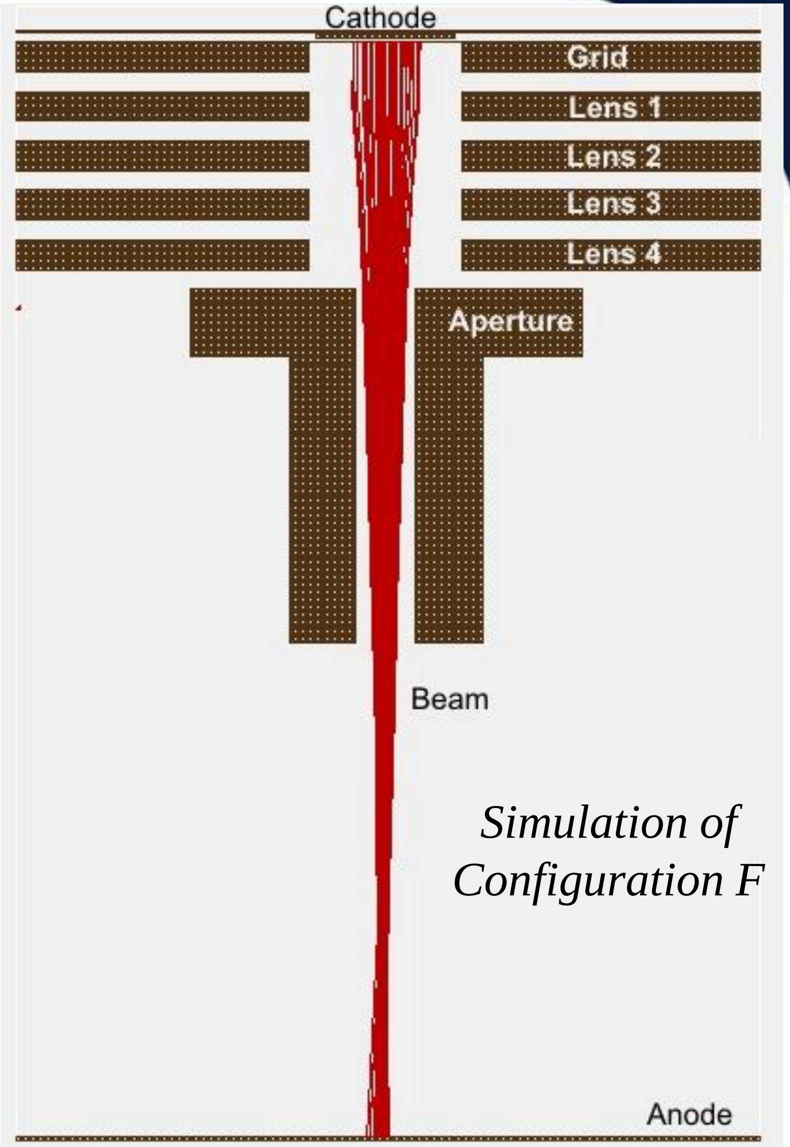
TOF-MS Integrated Testing



Lens Optimization

Focal plane coincident with Ionization region

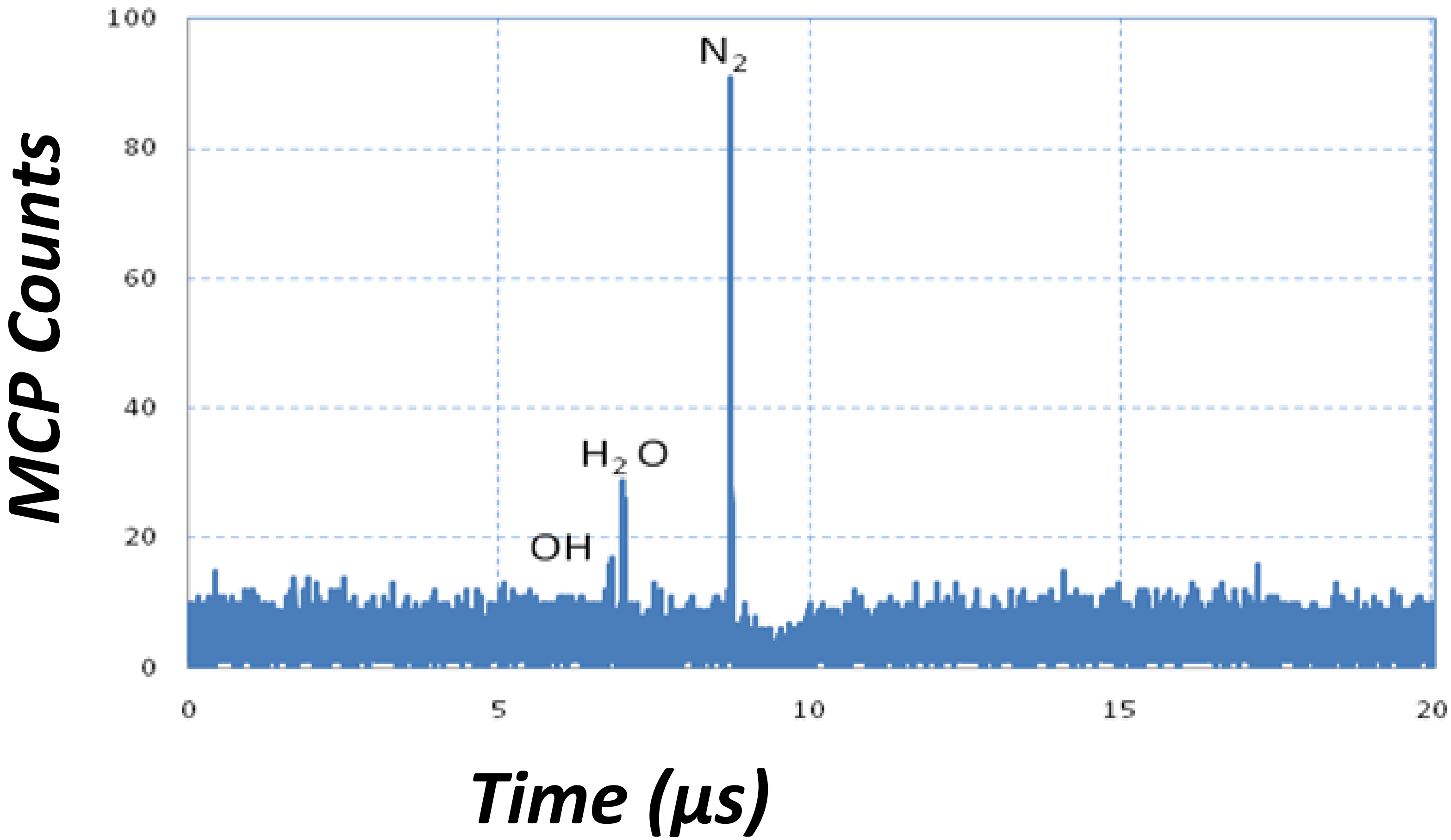




Simulation of Configuration F

Integrated TOF-MS Performance

- Mass resolution > 270



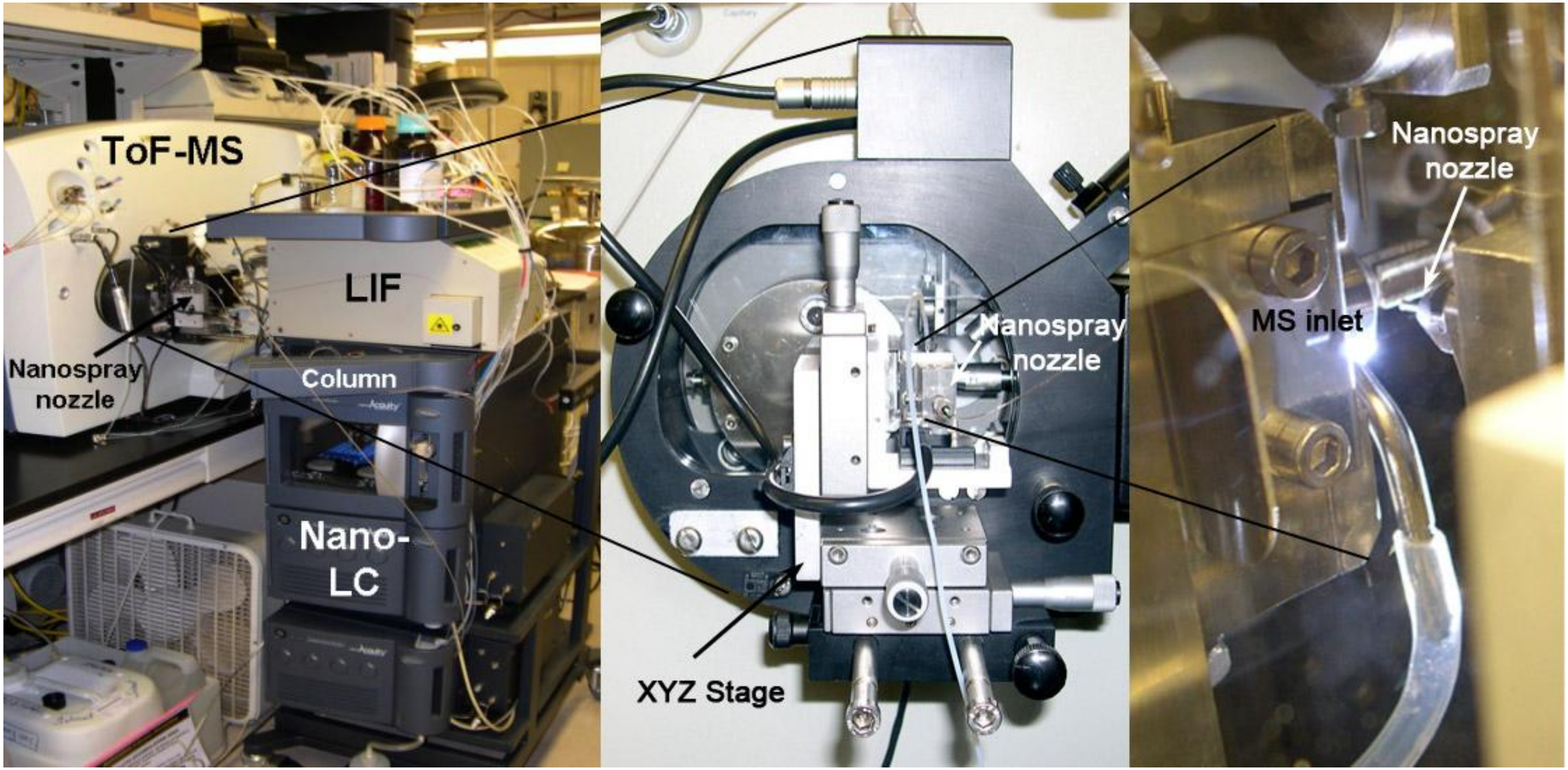
Future Steps

- Opportunities for TOF performance improvement:
  - **Increased sensitivity**

    Electron current –
    - Better transmission through revised geometry
    - Optimized emitter geometry
    - Pulsed e-gun

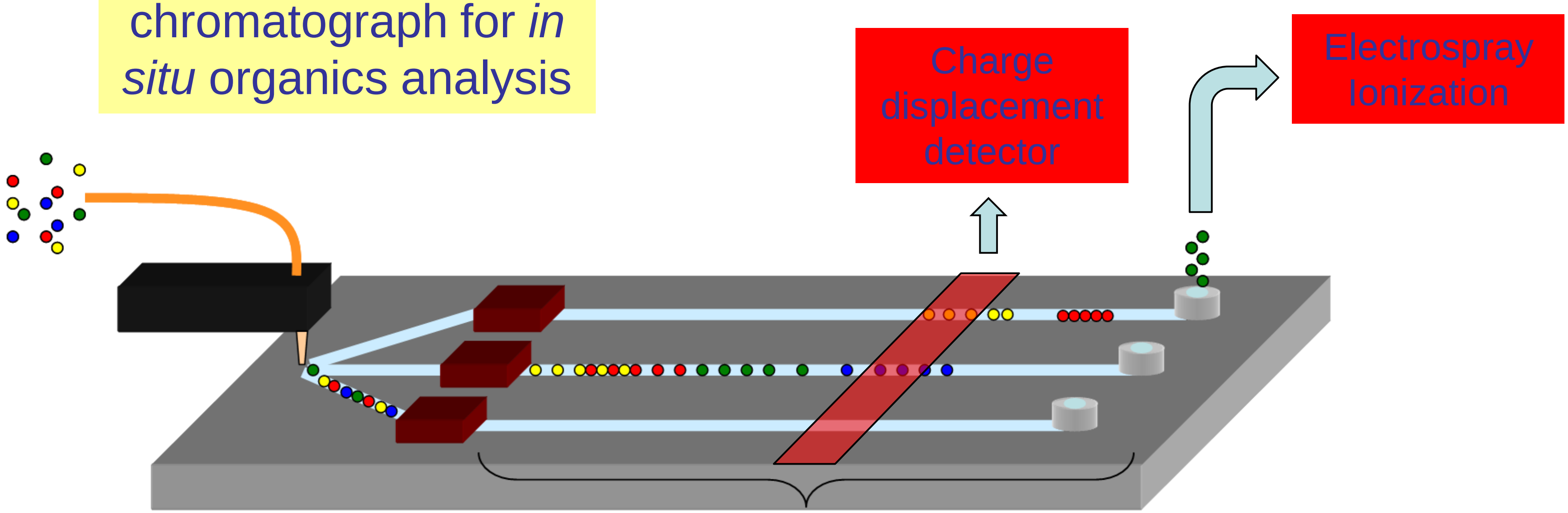
    Ion transmission –
    - Improve field uniformity to increase ion yield
  - **Increased mass resolution**
    - Reduce ionization volume
    - Minimize rise time on pulse electronics
  - **Improved emitter lifetime**

Liquid Chromatography-Mass Spectrometry



Miniaturized liquid chromatograph

Miniaturize liquid chromatograph for *in situ* organics analysis

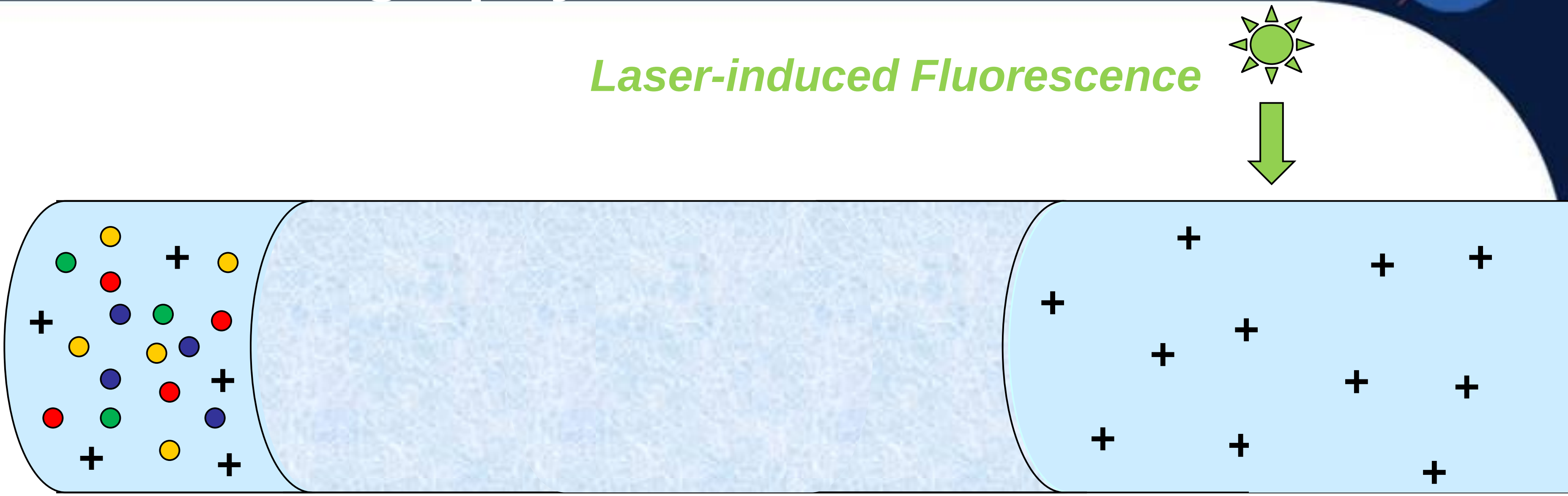


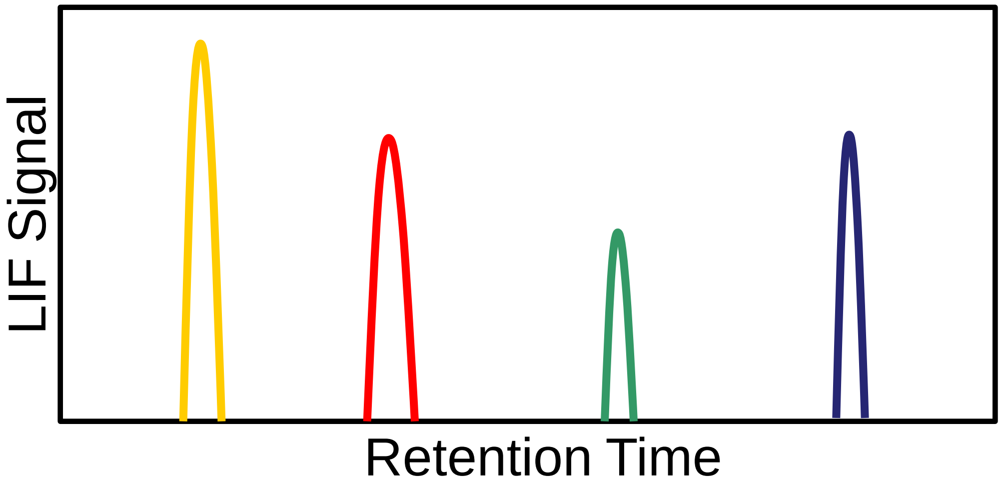
Successful lab techniques → planetary surface

- Nanoelectronic charge displacement detector at output
- Chemicals elute at different rates based on interactions with LC column
- Data = retention time spectrum

Chemical Separation by Liquid Chromatography

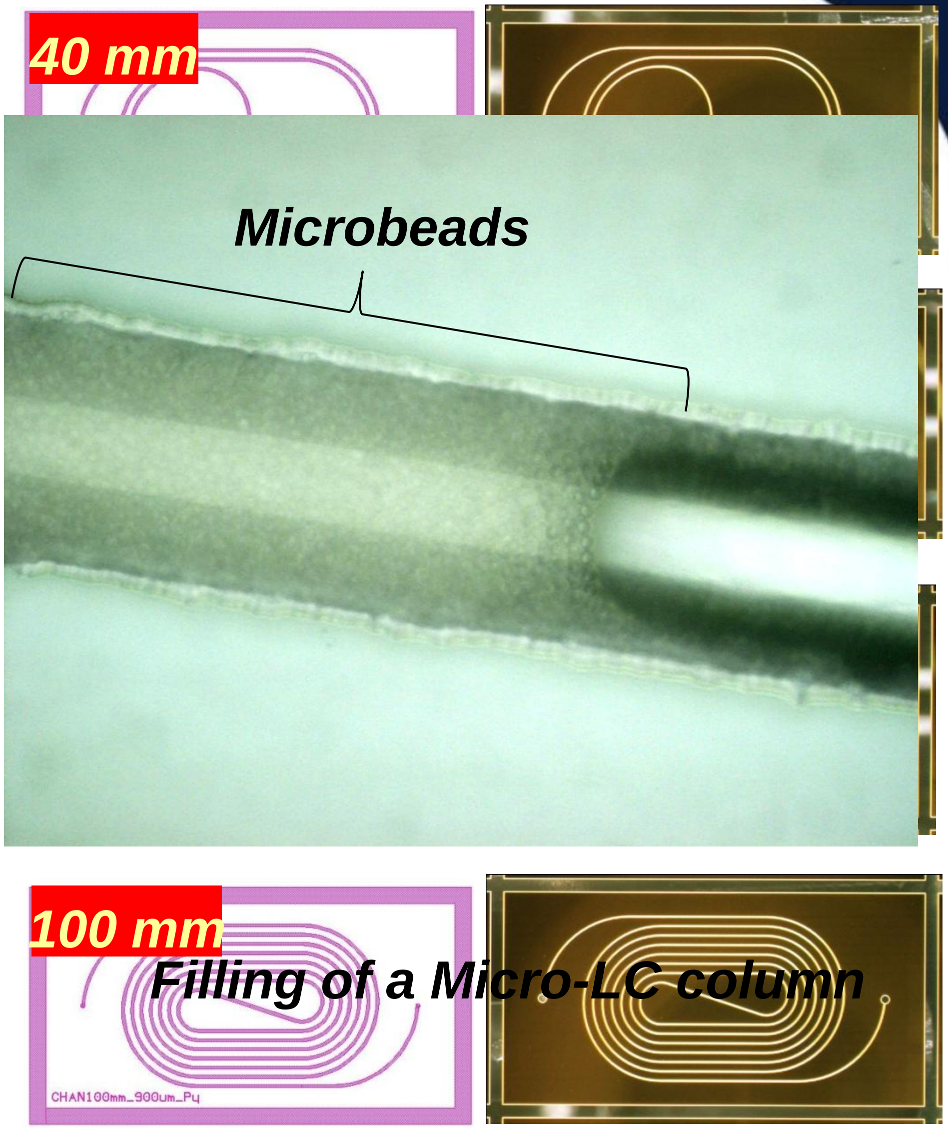
Laser-induced Fluorescence





Design of Micro-LC column

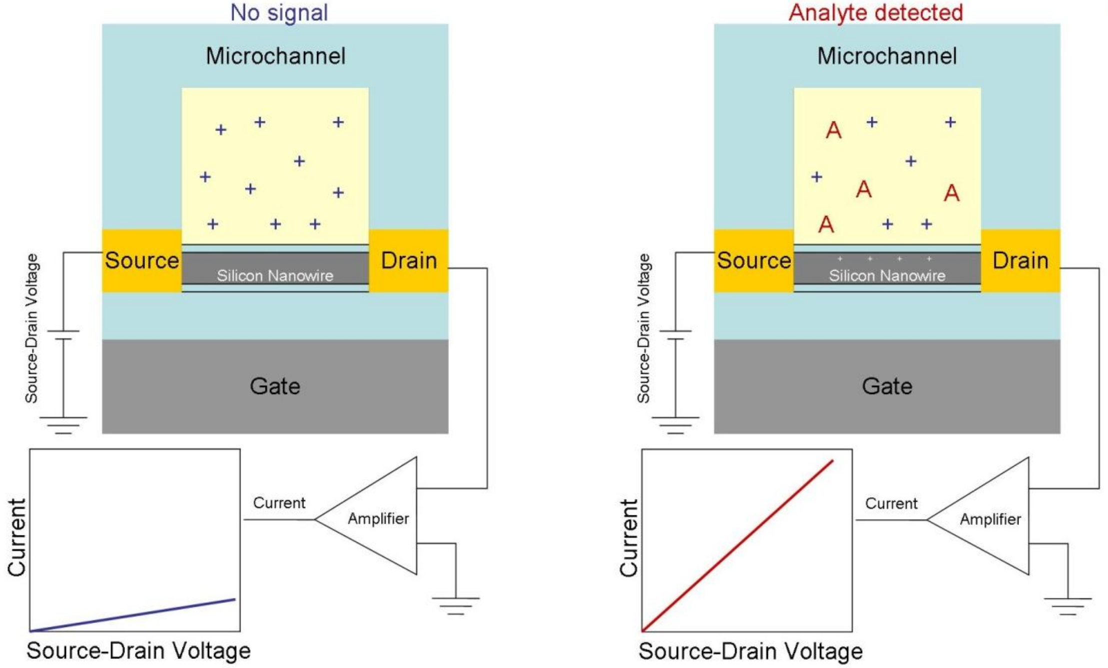
- Packing of microbeads to form stationary phase
- Longer channels → can better distinguish retention times, but higher pressure requirements
- Designed micro channels with varying lengths, 40 mm-100 mm using wafer scale processing.



ChemFET as charge displacement detector

Advantages:

- Fully electronic
  - Microwatts
- Non-specific
  - ChemFET can detect any organic species…
  - …but requires careful calibration

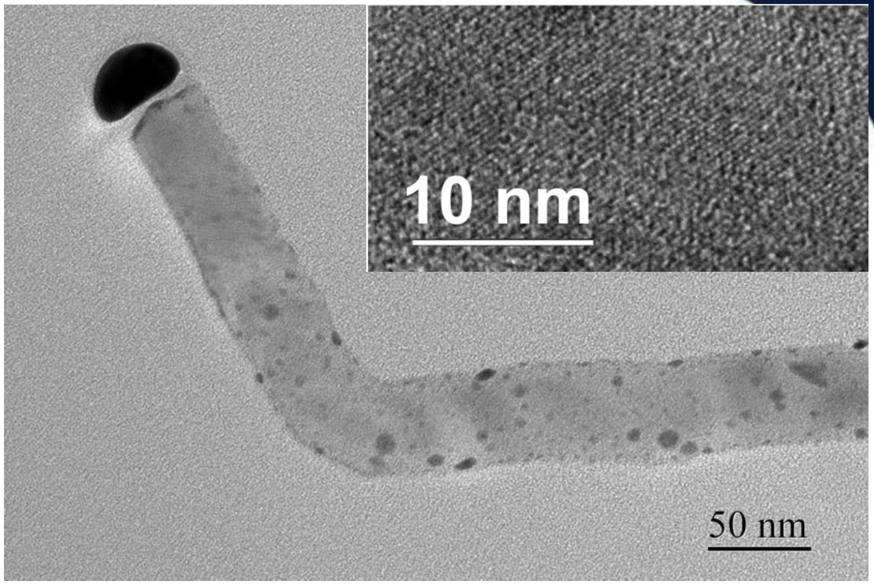


- Nanoscale
  - Redundancy can reduce risk
- Integratable with Si-based technologies
  - Can enable new on-chip functionality (measure pH, flow rate, buffer concentration)

Electronic Means of Chem/Bio Detection

<u>Requirements</u>

- ➢Sensitive/Selective
- ➢Low Power and Compact
- ➢Electronically Addressable
- ➢Autonomously Operable
- ➢Robust and Reliable

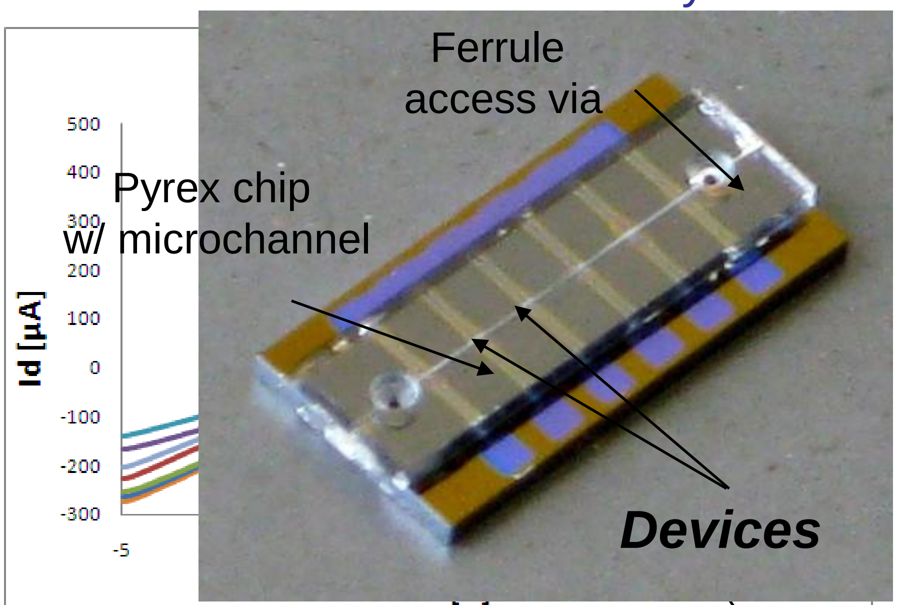


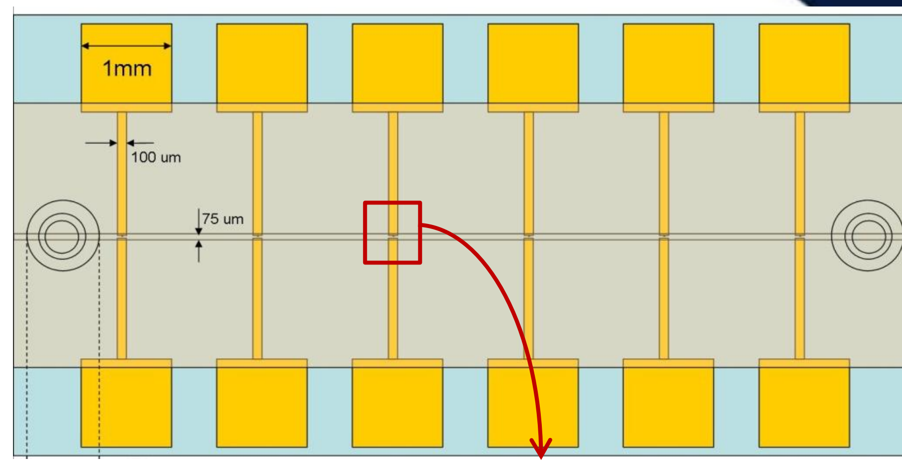
Parameter	SiNW
Metallic or Semiconducting	Semiconducting
Sensor-DNA probe	Direct surface attachment
Channel orientation	Aligned on array
Growth temperature	450°C

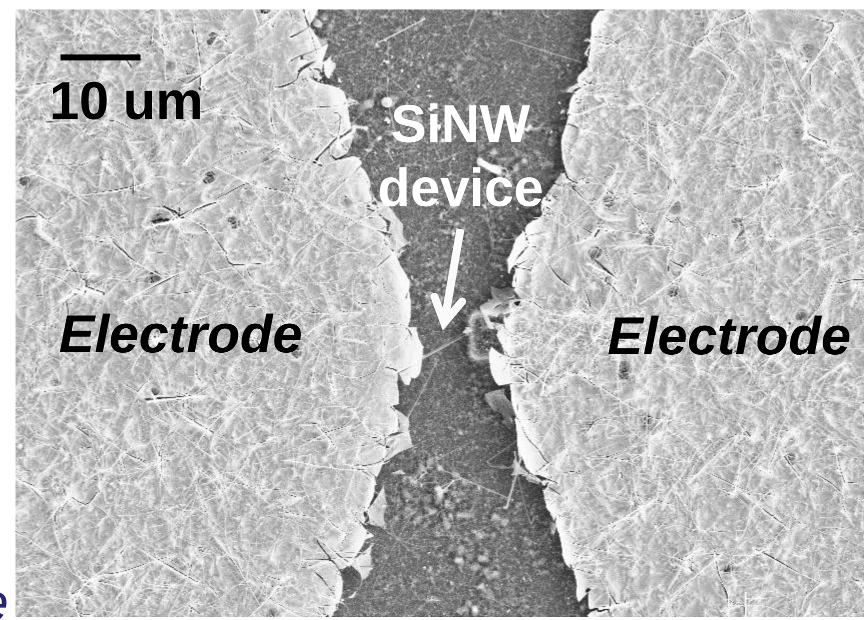
Silicon Nanowires
- Nanoscale diameter
- Single-NW limit attainable
- Scalable for manufacturability and redundancy

SiNW ChemFET

- SiNW fabrication – wafer-scale manufacturability demonstrated
  - Wafer-scale pillar growth for control of nanowire placement
  - Near 100% NW device yield
  - Excellent device uniformity

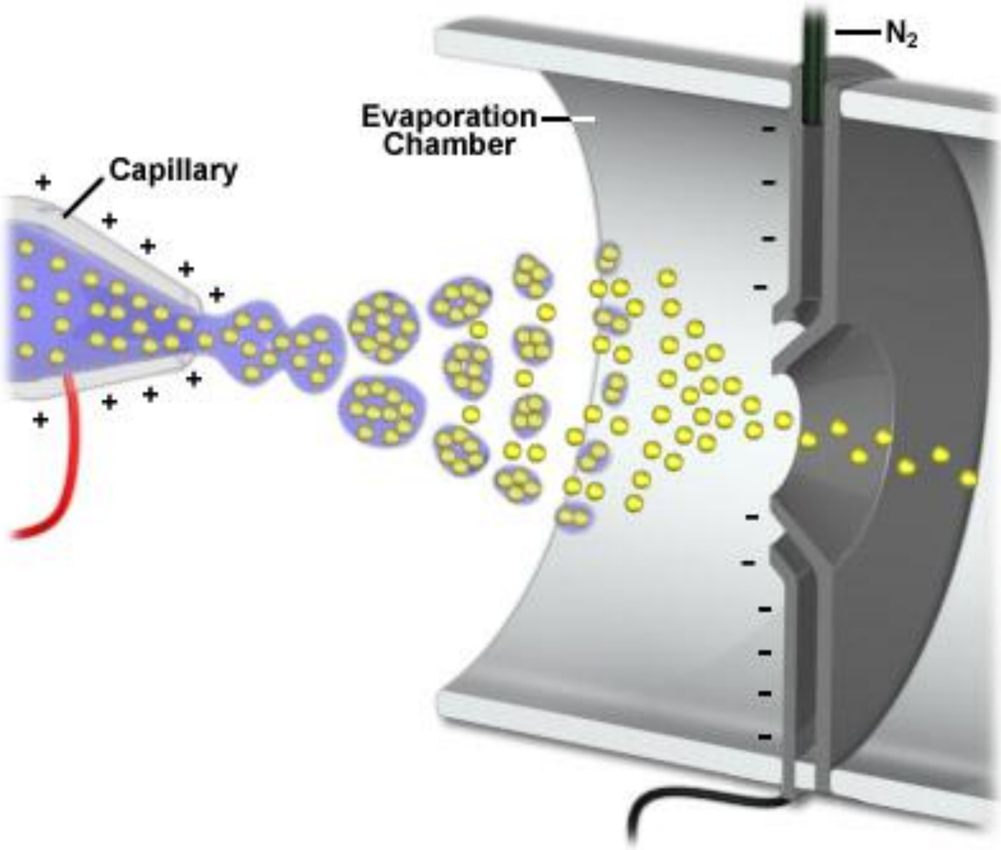






Transistor = current modulation with gate voltage

Electrospray ionization



http://www.magnet.fsu.edu/education/tutorials/tools/ionization_esi.html

Addition of a lens to the ESI nozzle

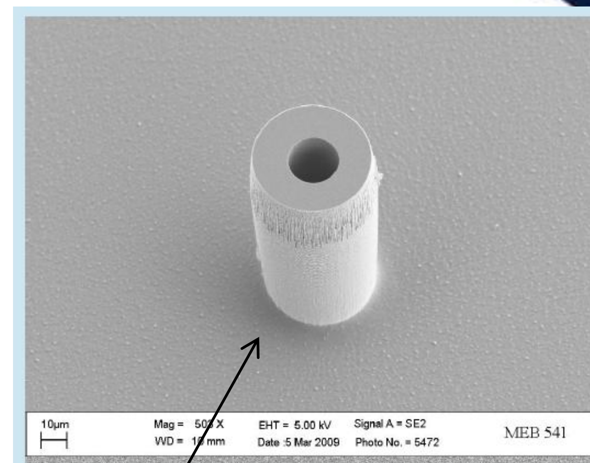
Design features:

• Can be tested with a commercial mass spectrometer and is compatible with microLC and TOF MS.

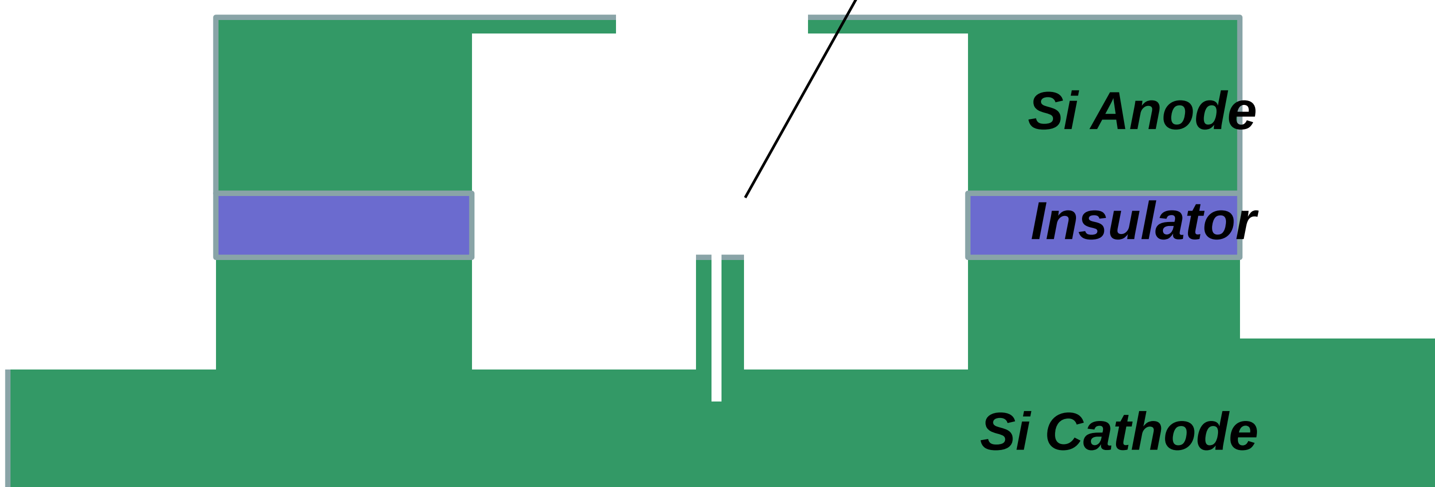
• Utilizes micro-nozzle to enhance atomization of jet.

• Electrodes facilitate formation of an ion beam

• Achieves high electric field for efficient charging of analyte without using high voltages



New design of ESI nozzle



Future Work

- Explore SiNW surface functionalization
  - Specificity
  - Multiplexing
- Liquid sample extraction
  - Extraction from solid samples
  - Concentration of analyte
- Miniaturized liquid sample handling components e.g. valves, pumps
- Higher aspect ratio nozzles

Conclusions

Highly interdisciplinary work

End-to-end systems development

- But this is just one facet of technology development at NASA…
  - Earth-observing instruments
  - Astronomical instruments
  - Spacecraft structures
  - Propulsion
  - Human spaceflight

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Please contact Stephanie Getty for more information or opportunities to collaborate.

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Thank you for your attention.