Fabrication and Testing of Micro-Cylindrical Ion Trap Arrays for Miniaturized Mass Spectrometer Development

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Miniaturization and Microfabrication of Cylindrical Ion Trap (CIT) Mass Spectrometers

- Miniaturize ion trap mass spectrometers
  - Low power and portable
- Array approach
  - High sensitivity and parallel analysis
- Microfabrication
  - High precision and batch fabrication

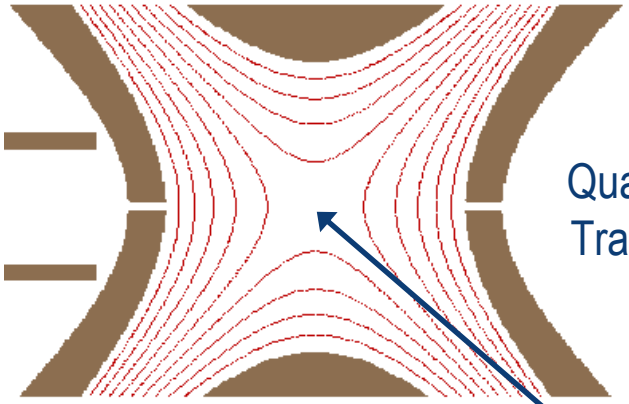
Hyperbolic

Cylindrical

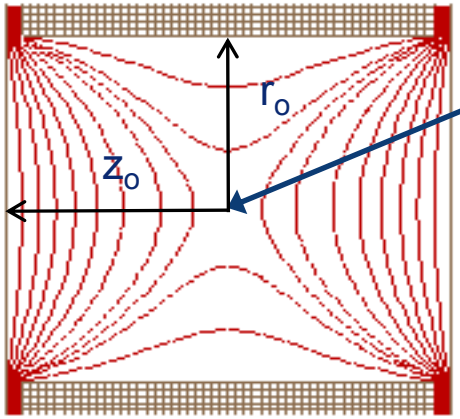
Micro-array

Cylindrical Ion Trap Easier to Miniaturize

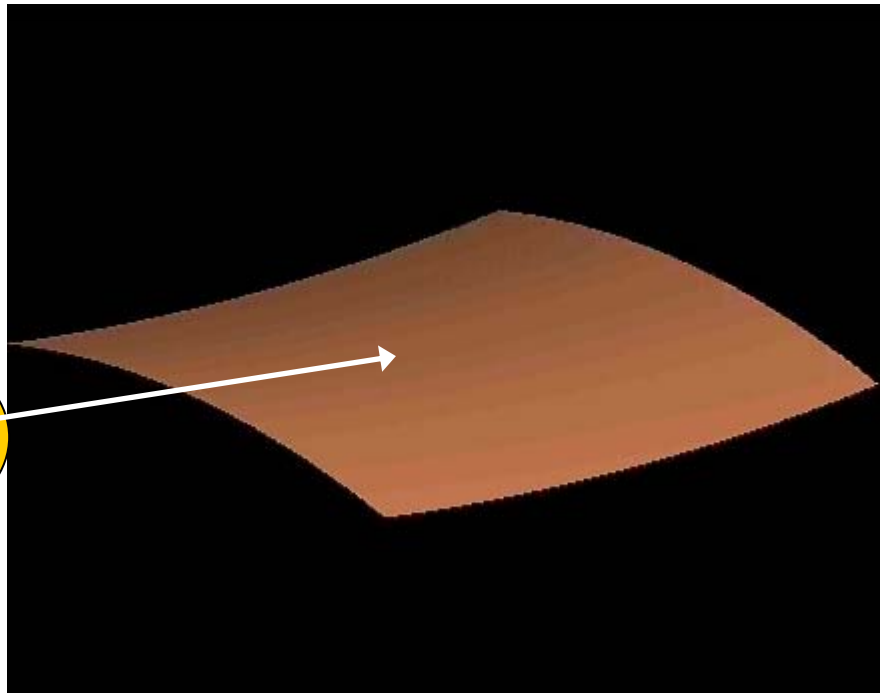
$$V_{rf} = (q_z\, r_0^2\, \Omega^2\, m) / (4A_2 e)$$



Quadrupole Ion Trap Geometry



Minimum of RF potential

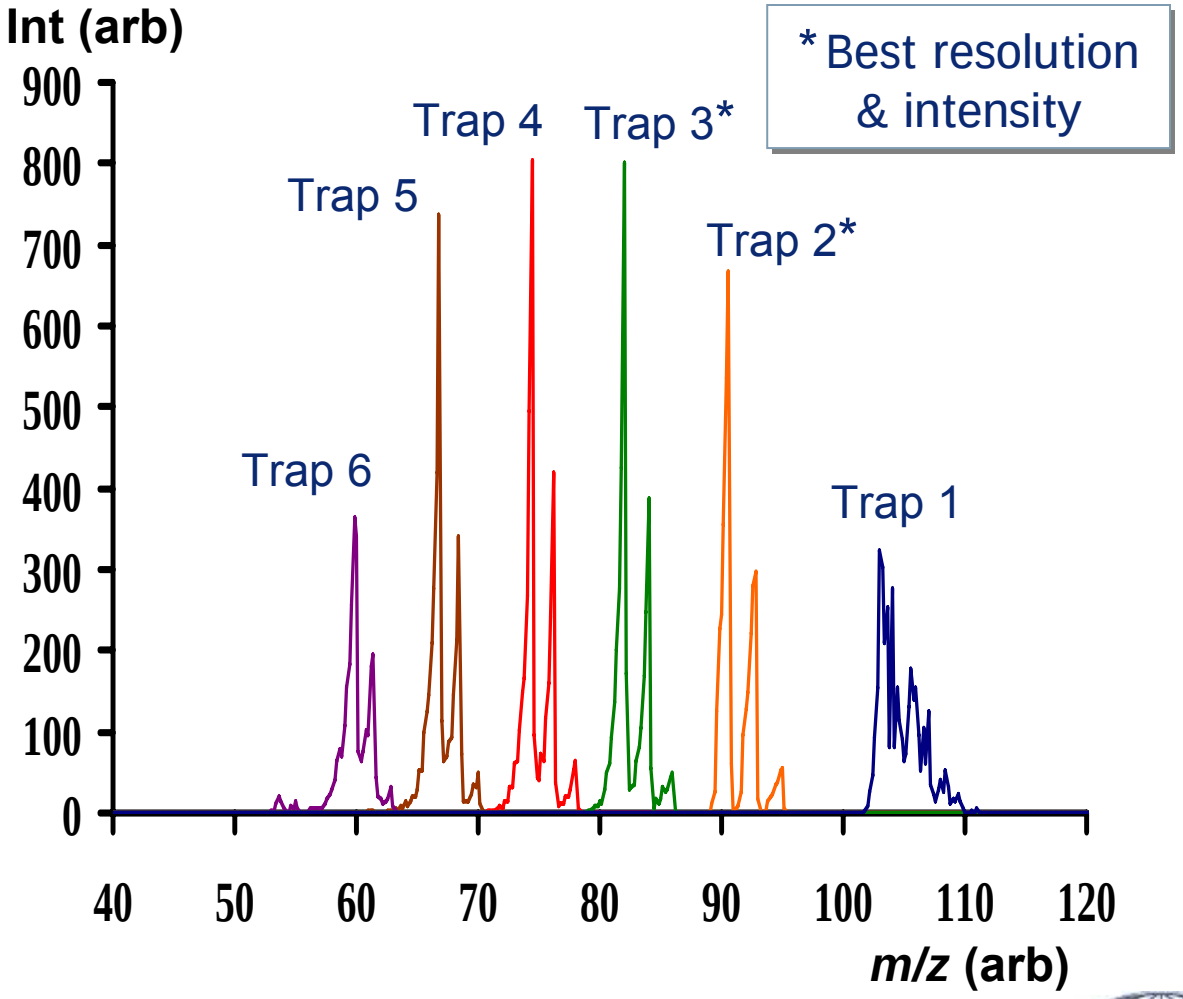


CIT Geometry
(z_o/r_o is critical)

Axial modulation often used to improve resolution

Computer Simulations to Optimize μ-CIT Geometry (z_o/r_o)

z_0 = 350 μm		
Trap #	z_0/r_0	r_0 μm
1	0.92	381
2	0.97	360
3	1.02	343
4	1.07	327
5	1.13	310
6	1.19	294

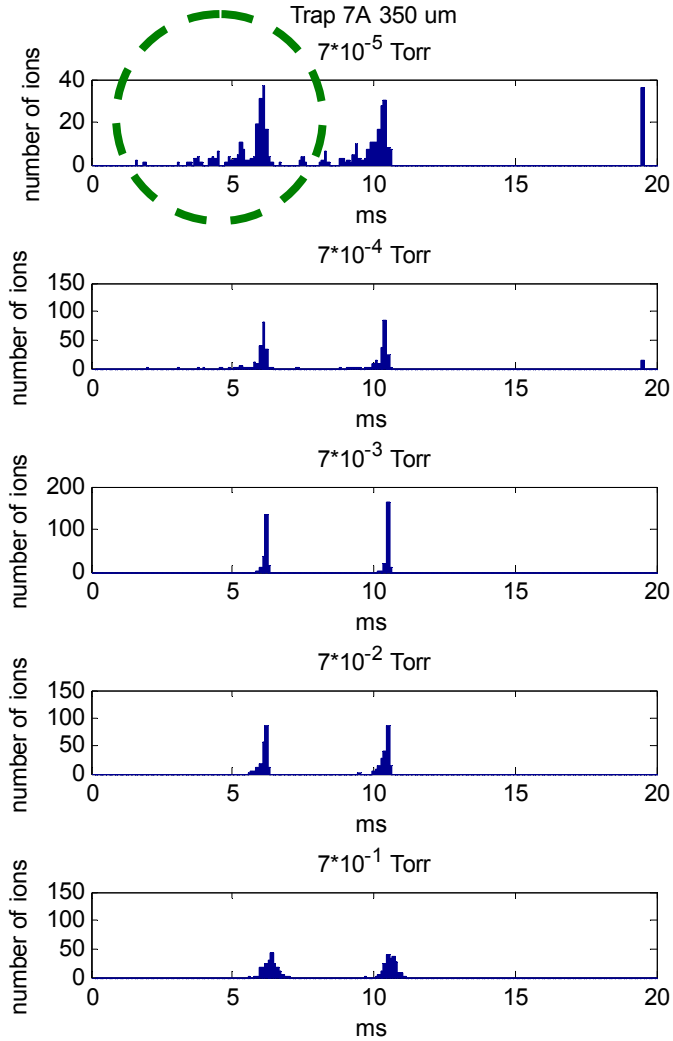


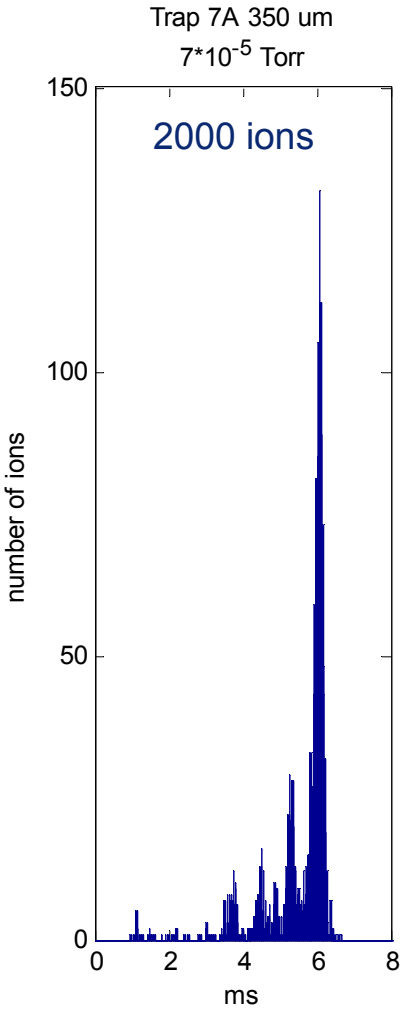
* Best resolution & intensity

Simulated in ITSIM (Courtesy: R. G. Cooks et al.)

Buffer Gas Simulations Indicate μ-CITs can Operate in Low Vacuum

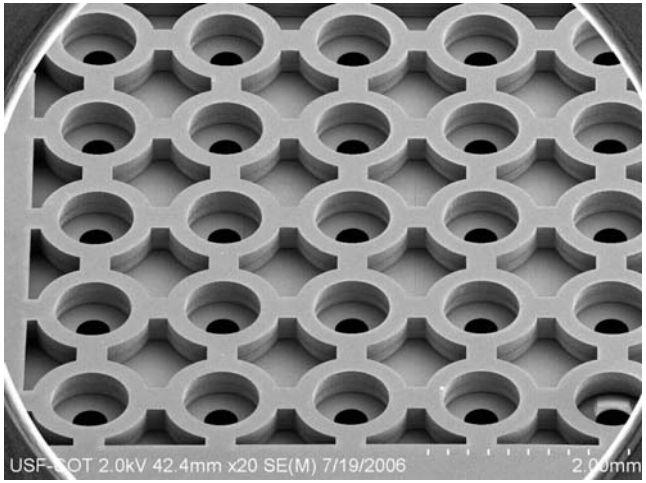
- Simulated spectra obtained from SIMION for a range of pressures inside the CIT with $z_0/r_0 = 0.97$
- ~360 μm radii μ-CITs, *m/z* = 40 & 50 (200 ions/ea)
- Simulations indicate good performance of micro traps at relatively higher pressures
- *No high-vacuum pump?*

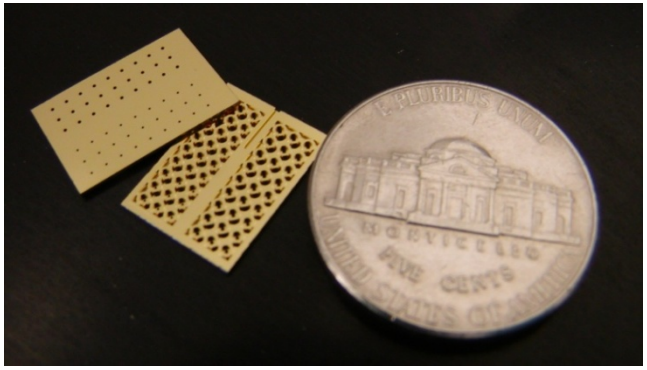




Microfabrication Provides Uniform 3-D Structures

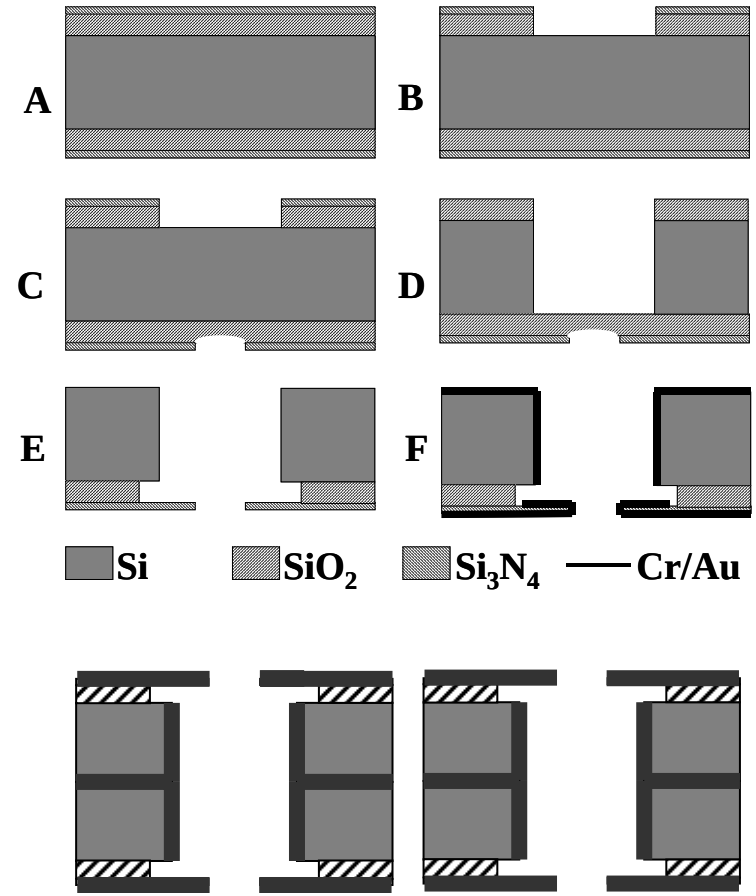
- Create high-aspect-ratio structures in silicon with high precision
- Arrays of half-CITs bonded back-to-back
- Capacitance reduced by etching excess silicon
- Simulations indicate axial modulation required
  - Strong influence of higher-order multipoles





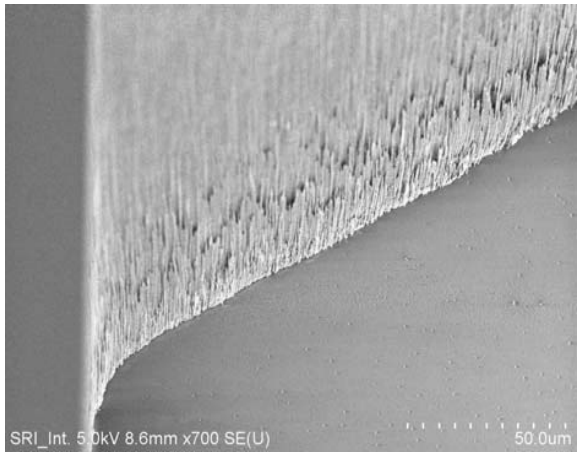
Microfabrication Approach for CIT Array in Silicon

- Wafer-level batch fabrication approach
- CIT structural layer
  - Ring electrode: Au-coated Si (350 μm)
  - Endplate electrode: Au-coated Si_3N_4 (3 μm)
  - Dielectric gap: 5 μm thermally grown SiO_2
  - Cr/Au layer: 150/2500 Å
  - A range of radii (325–375 μm) was incorporated in the optical mask to account for variation in wafer thickness
- Processes used
  - i-line lithography, RIE, DRIE, metal sputtering, dicing, flip chip bonding, Au wire bonding, SEM analysis, Wyko surface analysis

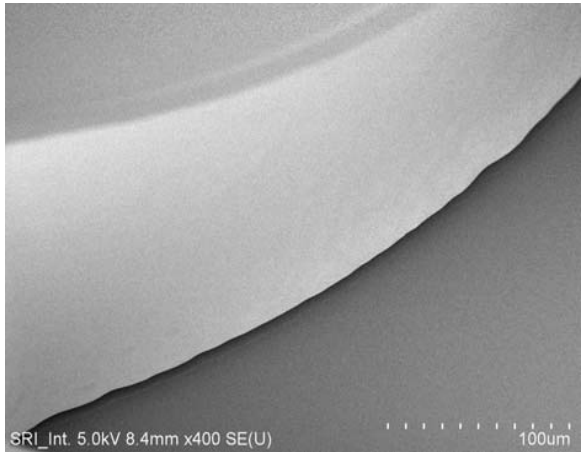


Two arrays of half μ-CITs bonded back-to-back using Au-Au thermo-compression bonding

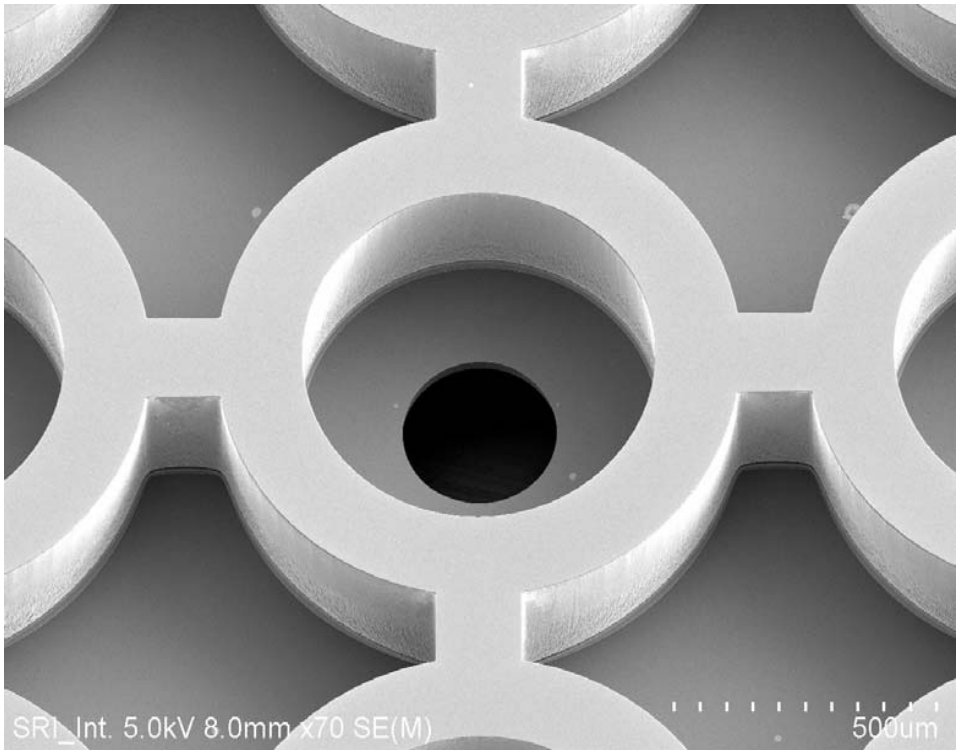
KOH Etch after DRIE to Smooth Electrodes



Wall roughness after deep Si etch step in DRIE

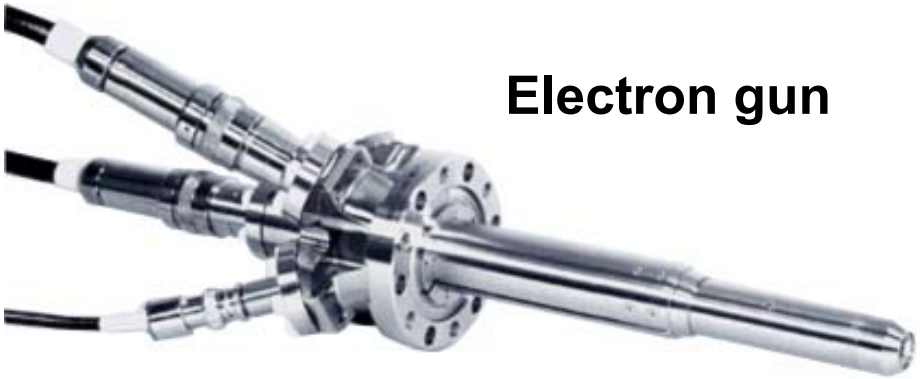


Wall roughness after 1.5 min of KOH etching



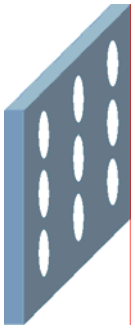
SEM of μ-CIT with aperture diameter 30% of ring electrode diameter (~360 μm)

Experimental Characterization of μ-CIT MS Arrays

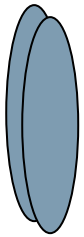


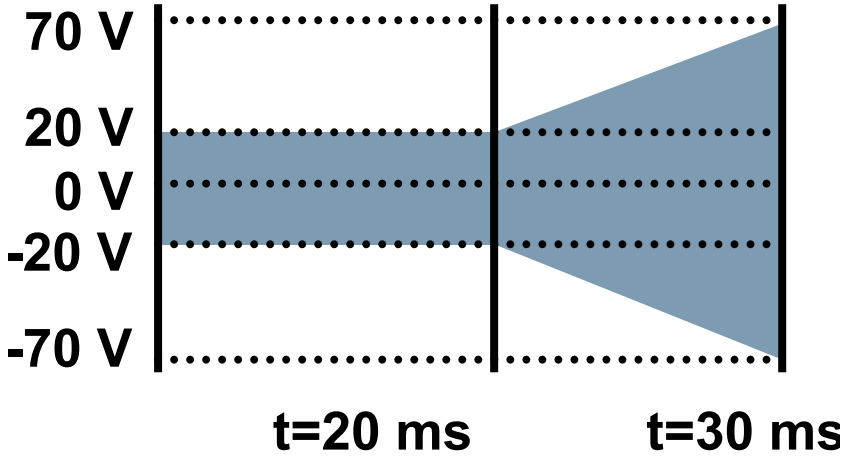
Electron gun

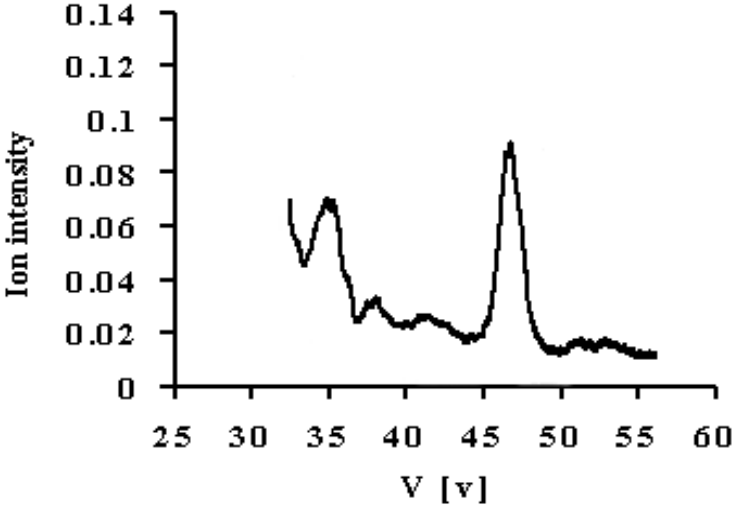
μ-CIT array



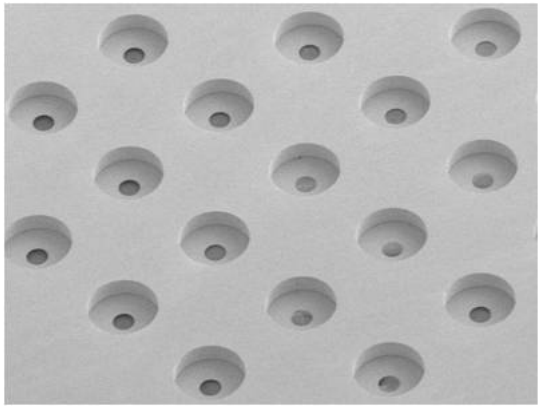
Microchannel plate detector



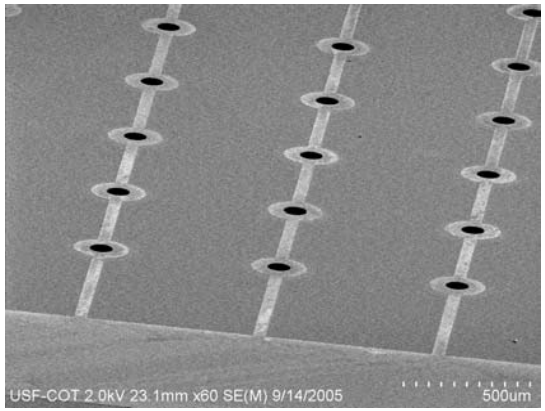




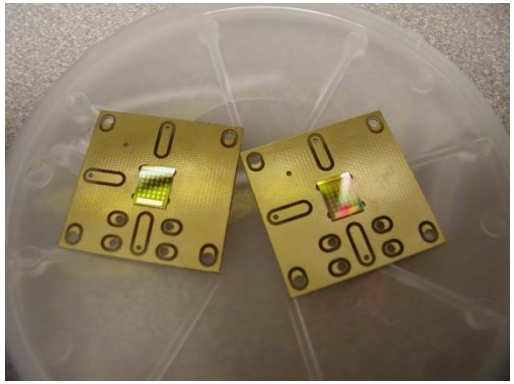
μ-CIT Array Fabrication Iterations

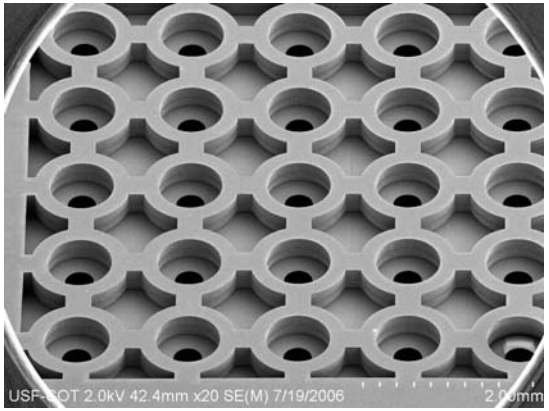


Generation I
- Developed a process to fabricate CITs in Si
- Thermal compression bonding method established
- Feasible SiO_2 & Si_3N_4 layer thicknesses ascertained
- High capacitance 640 pF

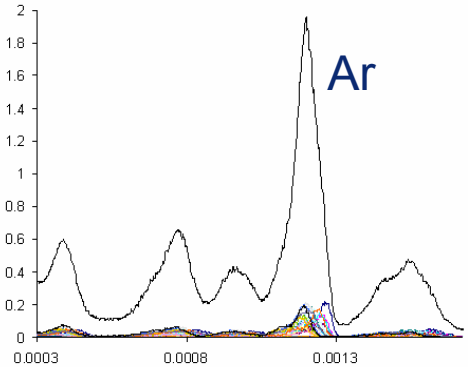


Generation II
- Au layer patterned
- Low capacitance 90 pF
- RF testing indicated arcing/plasma generation
- Need for integration with matched ionization source

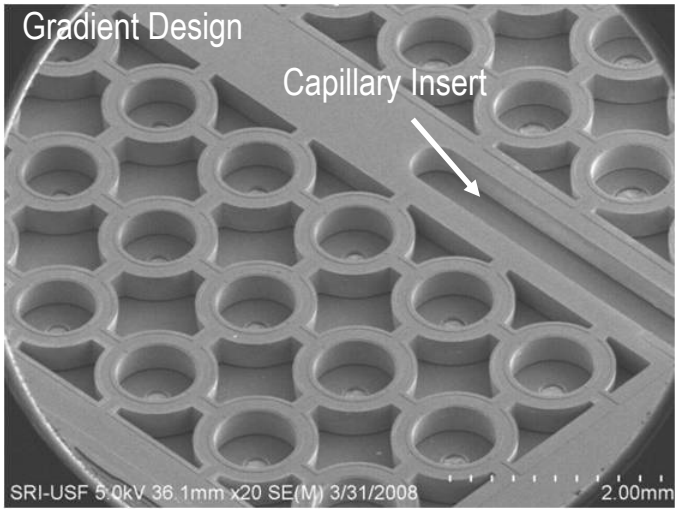




Generation III
- Si etched between μ-CITs
- Device capacitance 265 pF
- Cylinder wall verticality ~ 89°
- Working prototype (low resolution)
- Summation of spectra

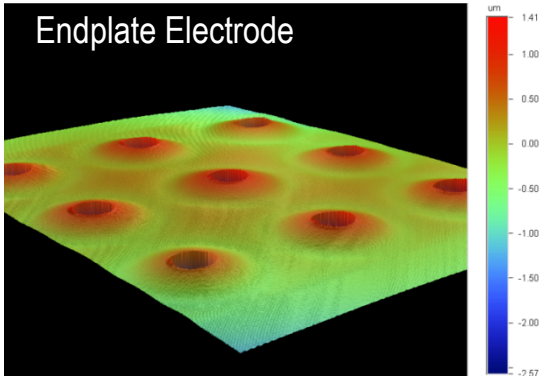


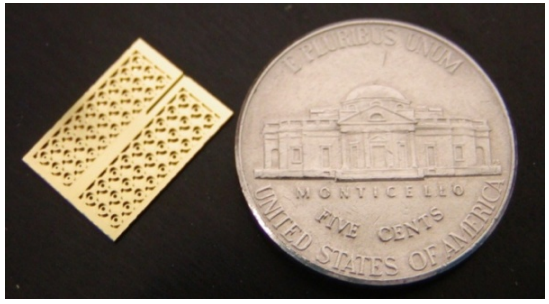
New Generation µ-CIT Array Fabrication

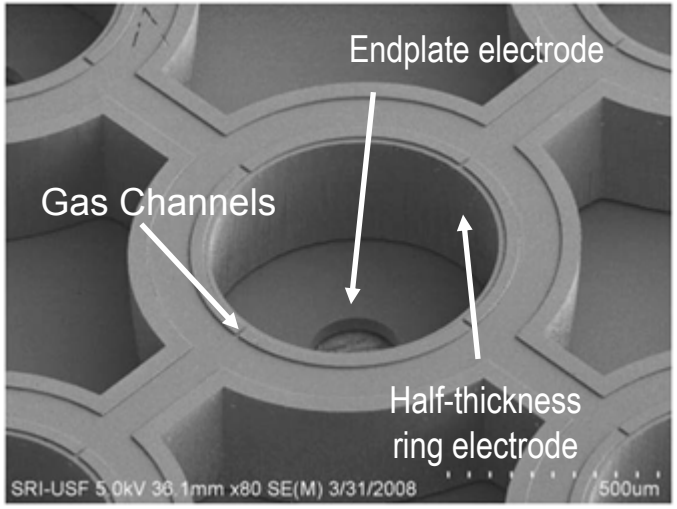


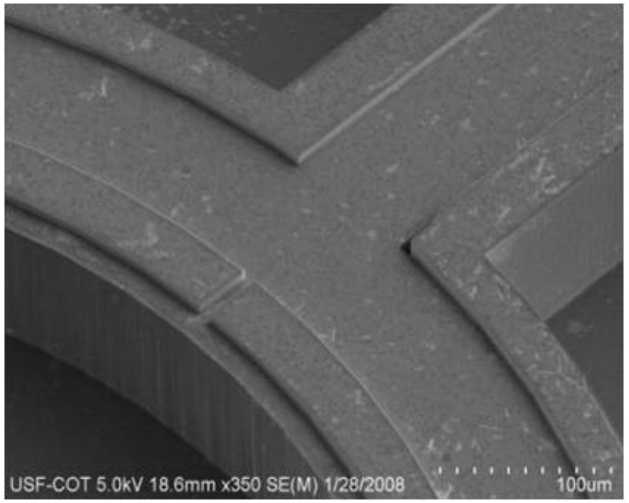
Generation IV

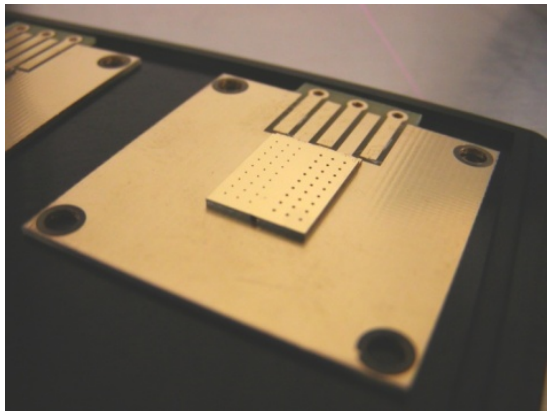
- Fabricated in SOI (Silicon-On-Insulator) wafers
- SOI (40 µm device, 5 µm BOX, 350 µm handle)
- Gas channels for direct gas introduction
- Gradient of z_0/r_0 ratio for fast determination of optimum geometry





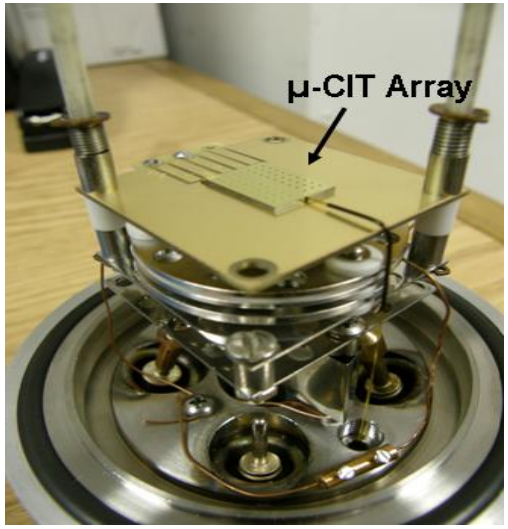




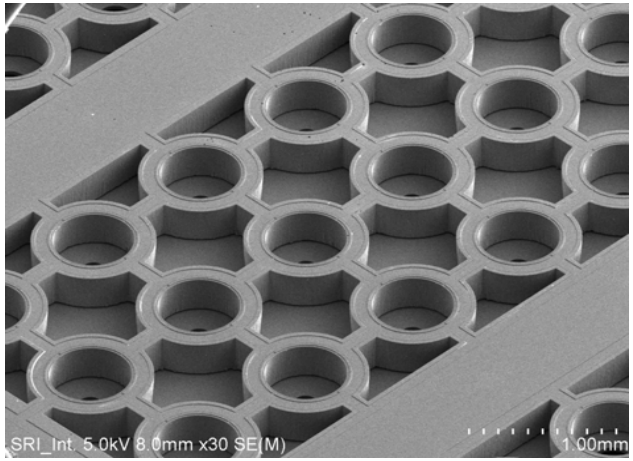


µ-CIT Array mounted on a Au-coated PCB

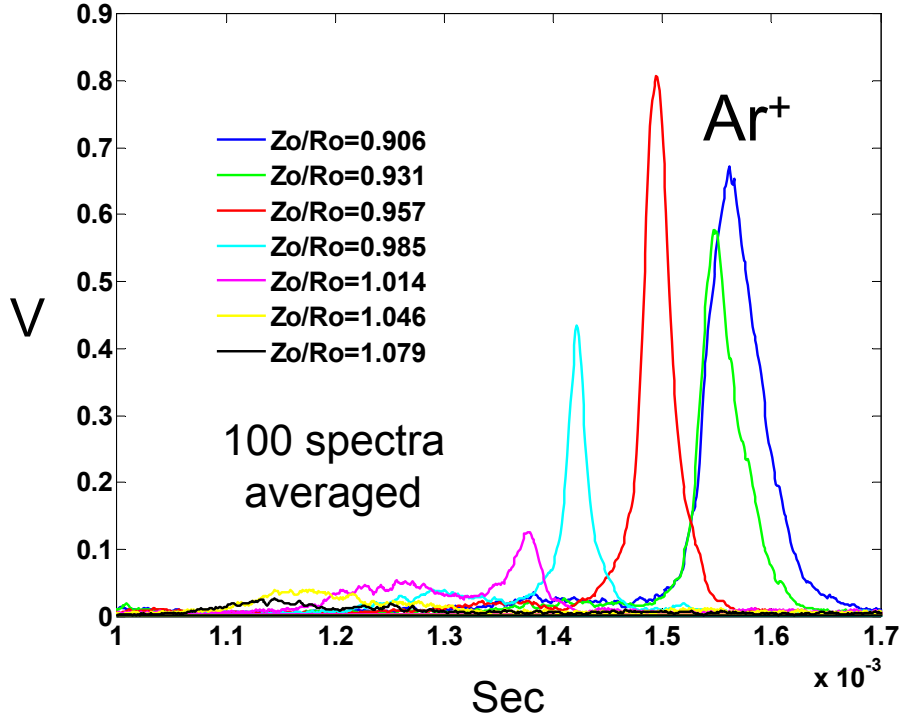
Arrays of μ-CITs with Gradient of z_o/r_o to Determine Optimum Geometry



Ar pressure	RF voltage frequency	Axial modulation frequency	Axial modulation amplitude
6.7 10^{-6} Torr	7.650 MHz	5.675 MHz	77 mV_{0-p}

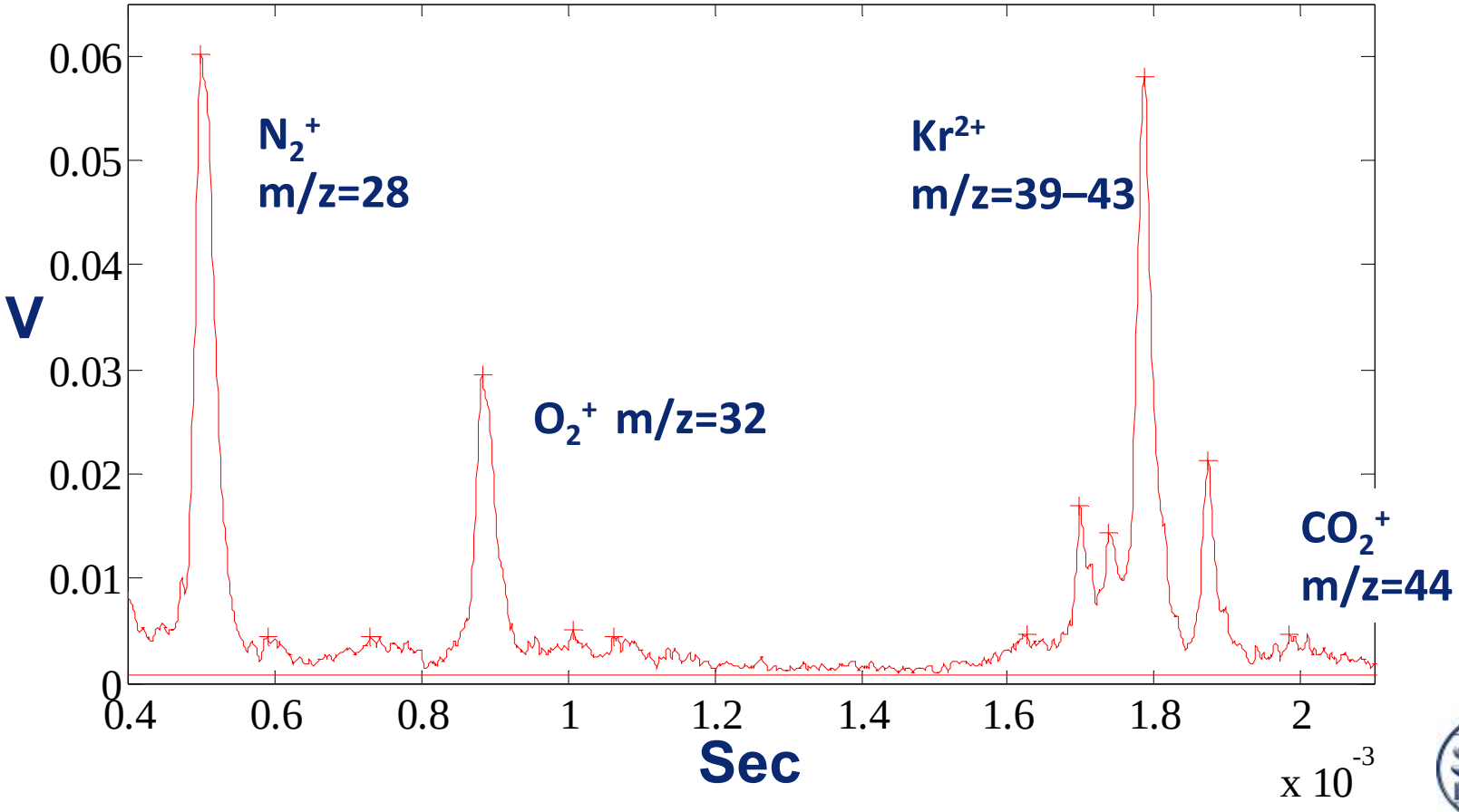


$z_o \approx 360$ μm

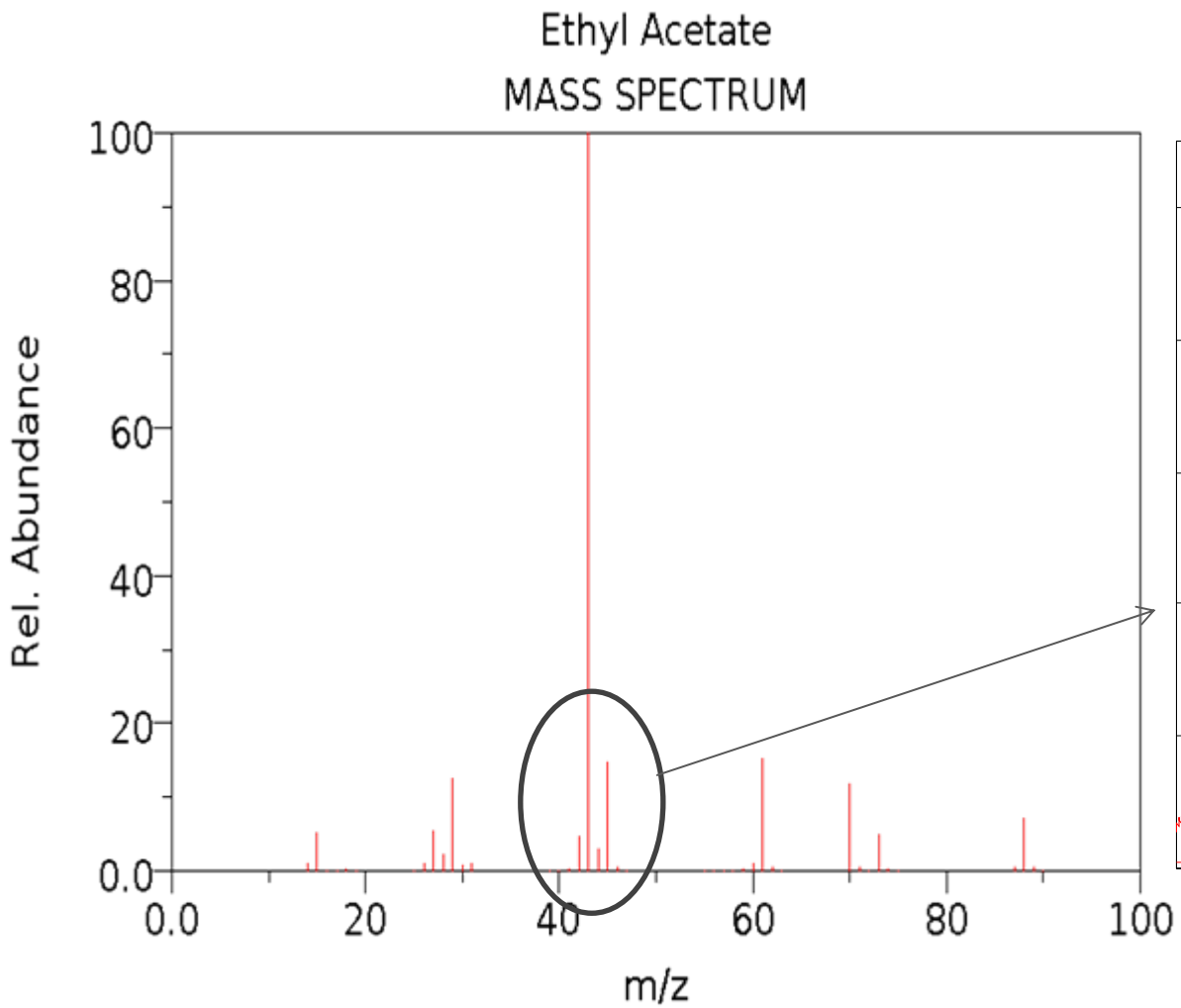


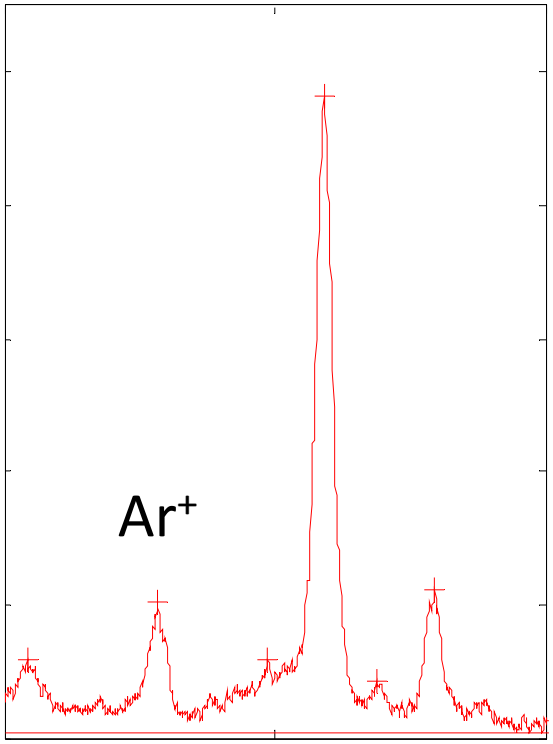
Unit Mass Resolution

Total pressure	RF voltage frequency	Axial modulation frequency	Axial modulation amplitude
6.5 10^{-6} Torr	7.650 MHz	1.983 MHz	25 mV_{0-p}



Organic Vapors Analyzed

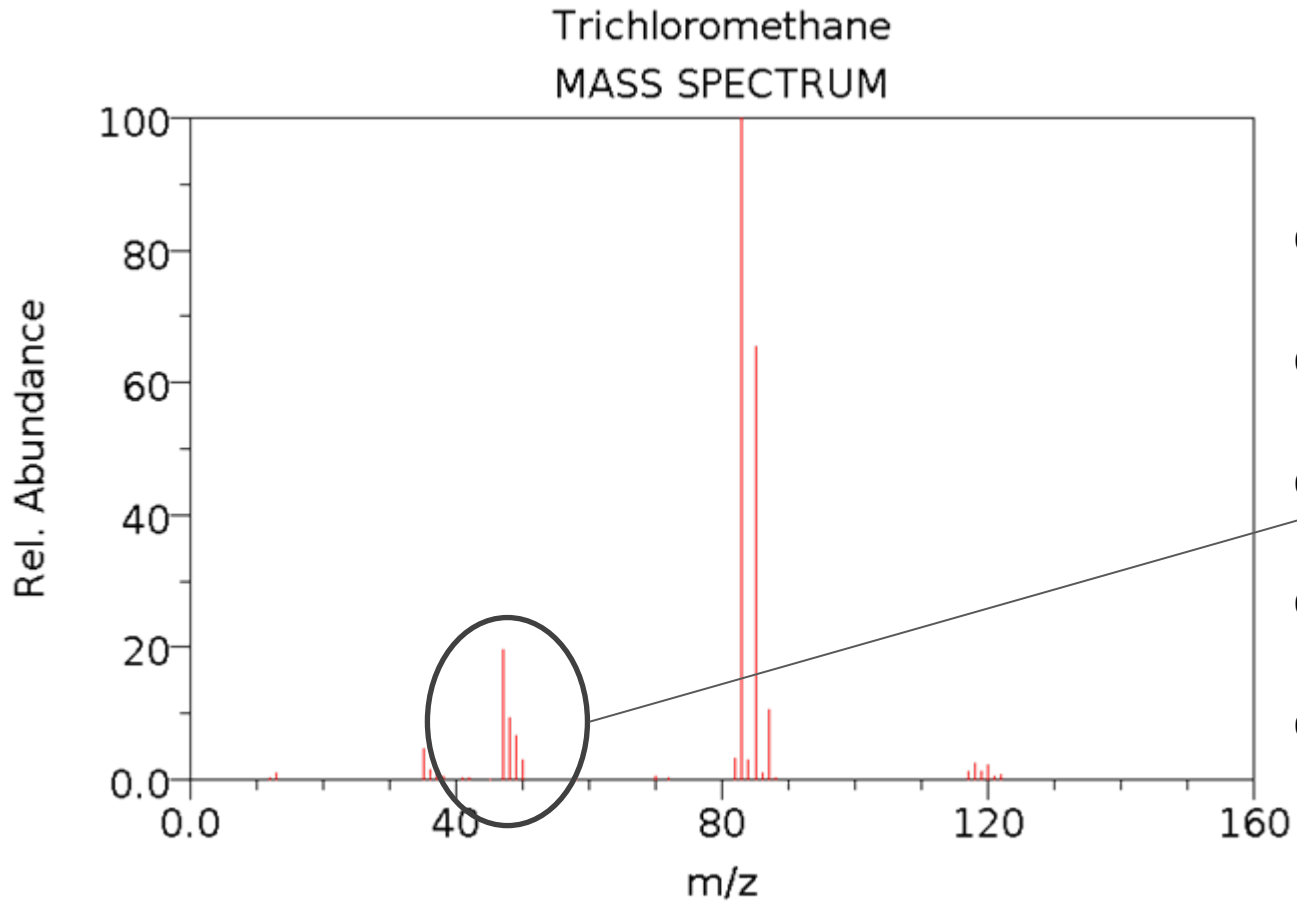




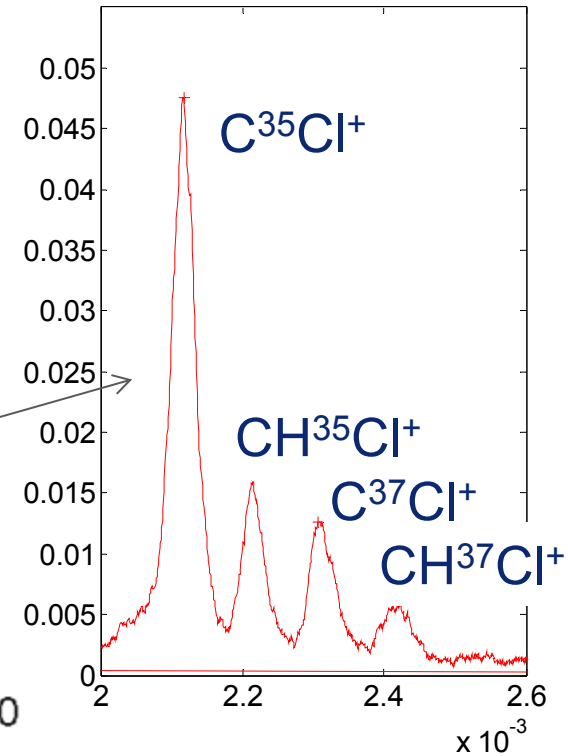
μ-CIT ms
(SRI)

NIST Chemistry WebBook (http://webbook.nist.gov/chemistry)

Isotopes of Chlorine Resolved



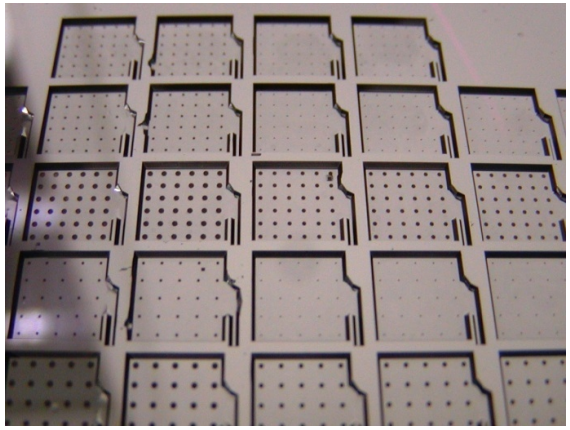
NIST Chemistry WebBook (http://webbook.nist.gov/chemistry)



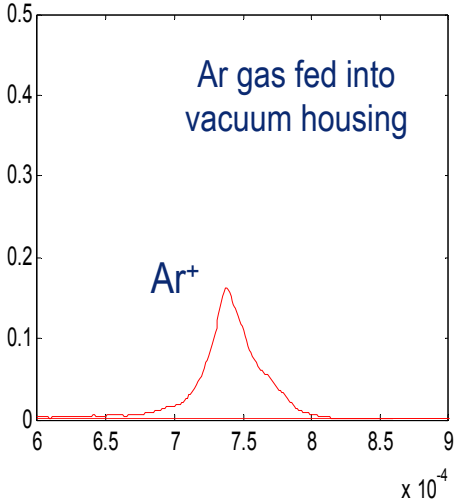
μ-CIT ms
(SRI)

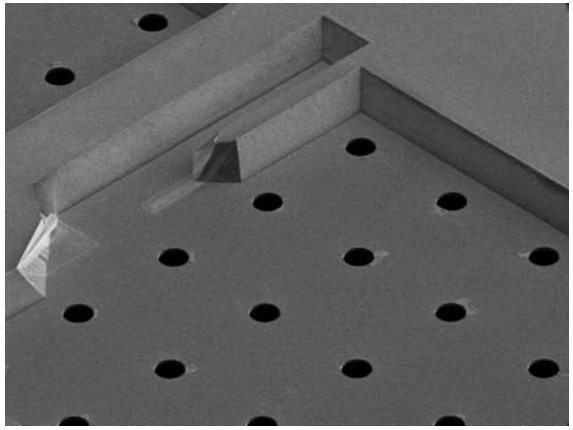
Microfabricated Differential Vacuum Chamber

- Differential chamber (DC) allows more efficient sample introduction
- Increased sensitivity (3x) with apertures 1.2x diameter of CIT apertures
- Pressure gradient allows for testing higher pressures in trap without damaging ion detector
- Investigation into combined inlet system and ionization source

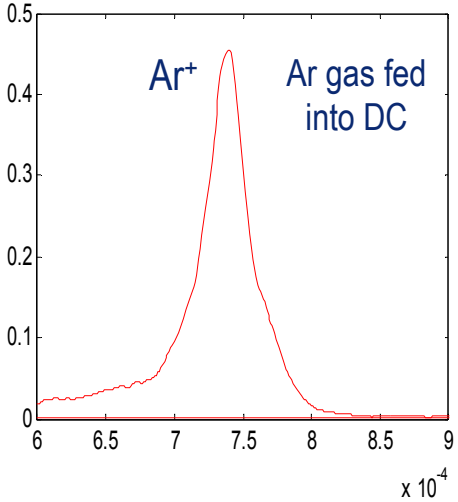


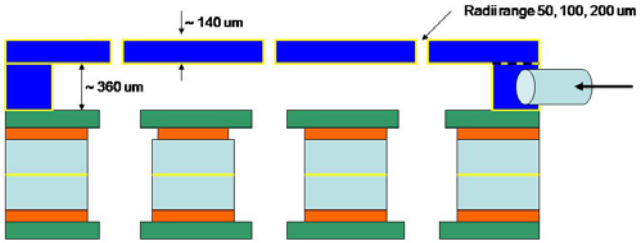
Si wafers with multiple DC chips with different aperture sizes, after etching the Si_3N_4





SEM image of a DC chip showing capillary insert and the apertures for electron beam

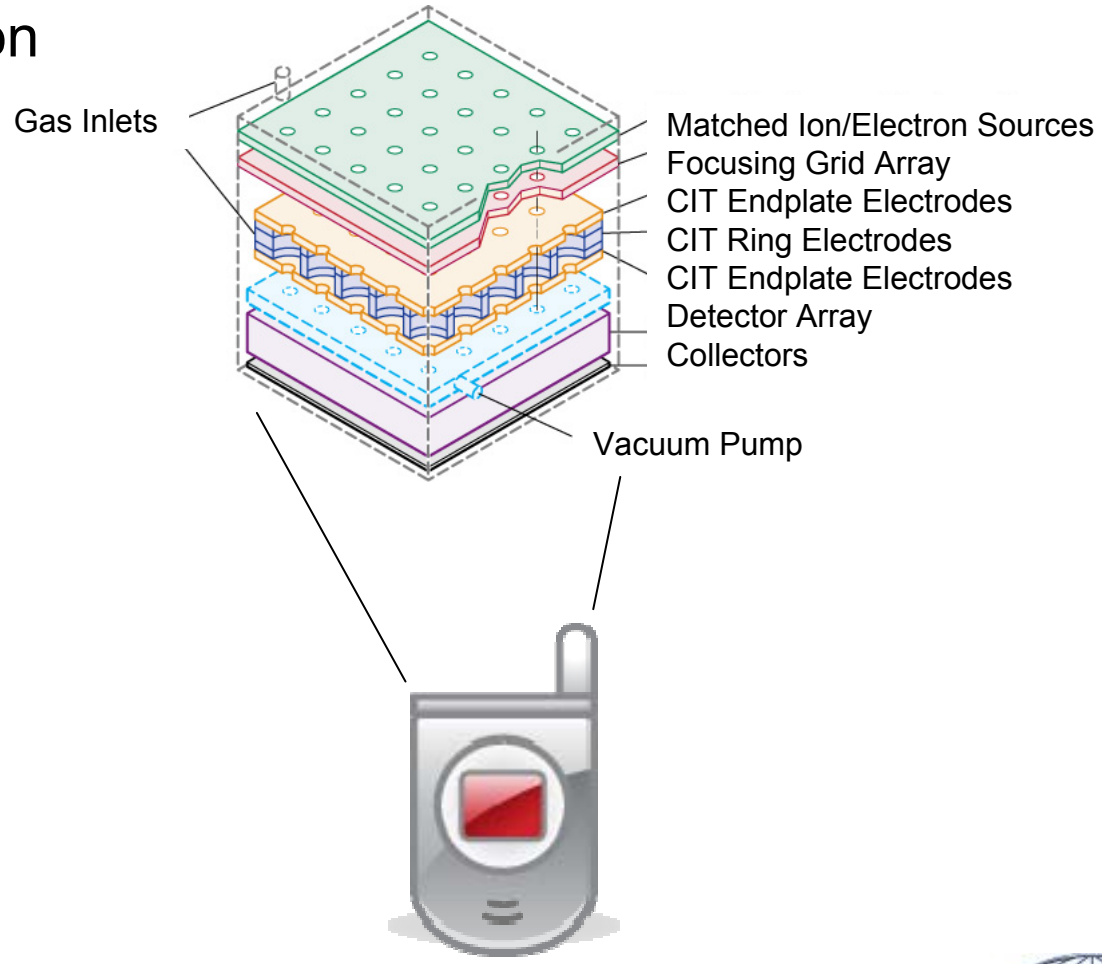




Schematic of DC

Realization of Low-Cost Micro-Mass Spectrometers

- Requires microfabrication and integration of all components
- High-density CIT arrays for increased sensitivity
- Matched ionization sources
- Fast high-gain detector for poor vacuum
- Micro vacuum pumps
- Integration into small package



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