Superconducting High-Resolution X-Ray Spectrometers for Chemical State Analysis of Dilute Samples

Stephan Friedrich

QuickTime™ and a
BMP decompressor
are needed to see this picture.

BERKELEY LAB

Lawrence Livermore National Lab

Owen Drury, Simon Labov

Lawrence Berkeley National Lab

Tobias Funk, Steve Cramer

Funding:DOE-OBER, NIH-GM, NSF-IMR, NASA SR&T

SRI-2003, San Francisco, 08/28/03

Outline: Why Cryogenic Detectors?

- Why low T? → Low thermal noise / Small excitation energies → High energy resolution

- Which technologies?

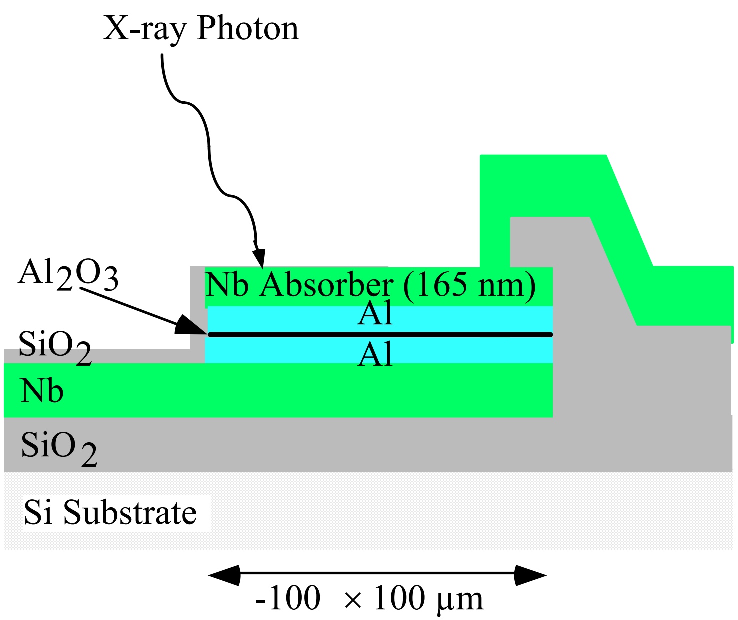
	Tunnel Junctions	Microcalorimeters
Operating Principle	$E \rightarrow \Delta Q$	$E \rightarrow \Delta T$
Resolution (0.1 to 6 keV)	2 - 12 eV FWHM	2 - 5 eV FWHM
Max. count rate	~10,000 cts/s	~500 cts/s

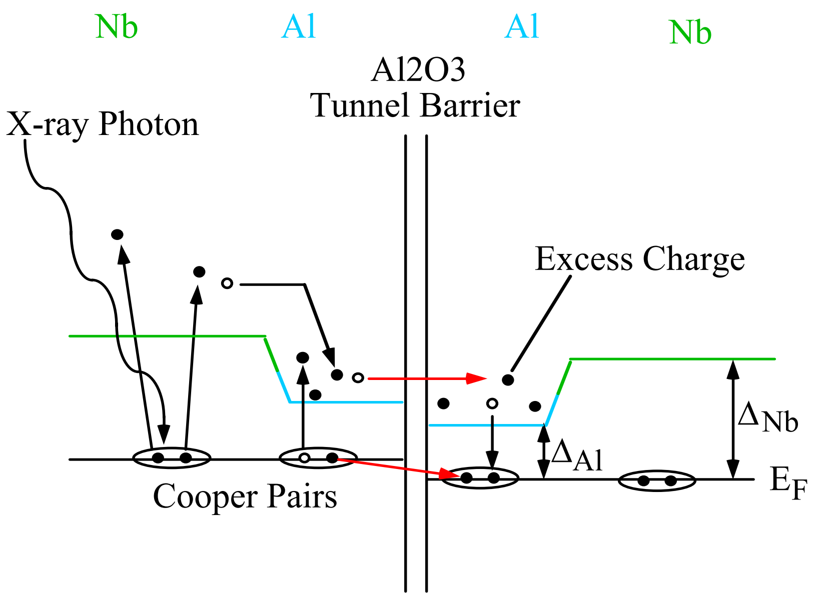
Both detectors have small pixel sizes $(\sim 0.2\ \mathrm{mm})^2$ and are operated around 0.1 K.

- What for?

Fluorescence-detected absorption spectroscopy of dilute samples

Superconducting Tunnel Junction Detectors



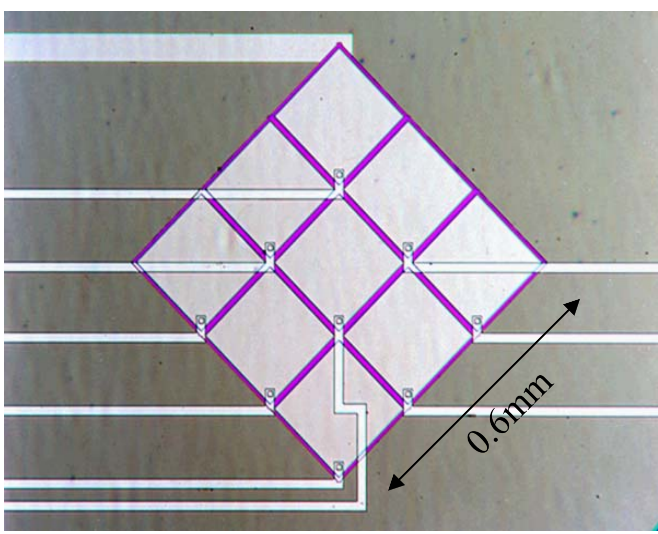


Signal = Current Pulse

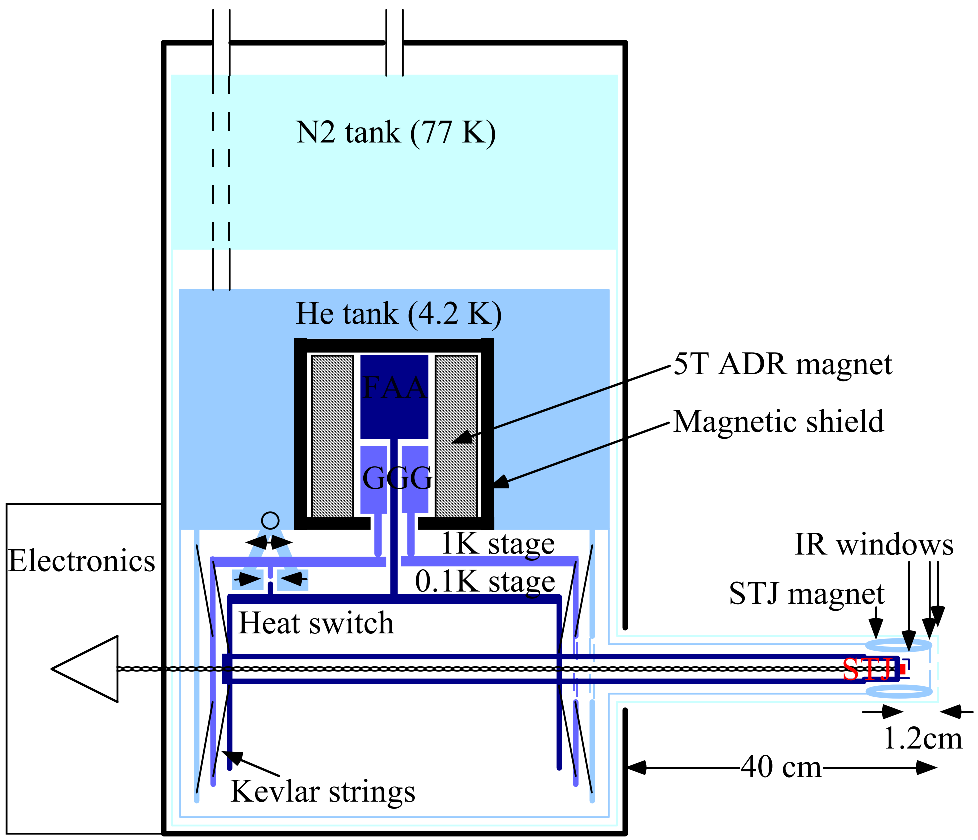
Small energy gap (Δ≈1meV) ⇒High energy resolution (≈10 eV FWHM)
Short excess charge life time (µs) ⇒ (Comparably) high count rate (≈10,000 counts/s)

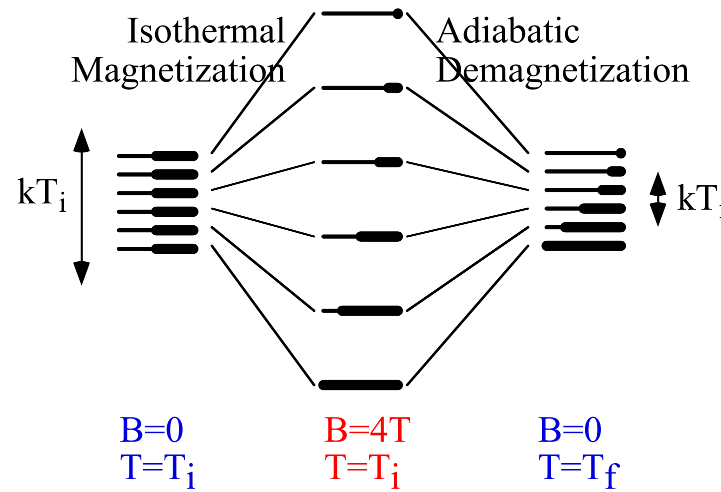
Two-Stage ADR with Cold Finger

- 70 mK base T, 20h below 0.4K
- 3×3 array at ≈15mm $\Rightarrow \Omega/4\pi \approx 10^{-4}$
- ≈15eV FWHM, >100,000 cts/s max



Adiabatic Demagnetization Refrigeration





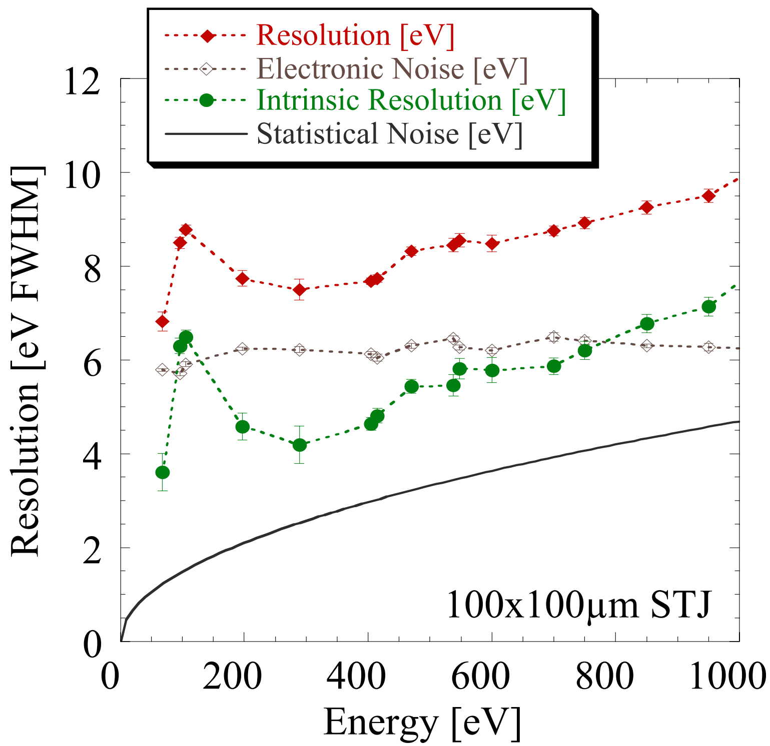
1) Close heat switch

2) Apply B (lower entropy S)

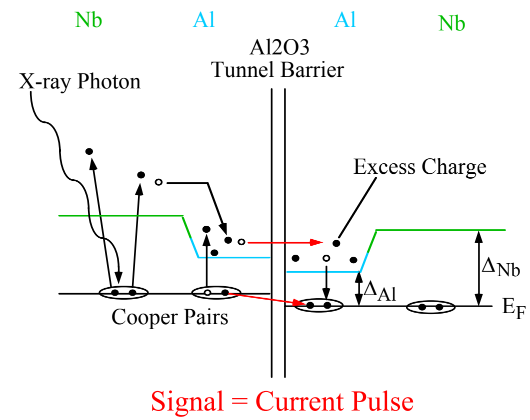
3) Open heat switch (decouple T)

4) Reduce B slowly (keeping entropy constant ⇒ reduce T)

STJ Performance: Energy resolution



≤10eV resolution below 1keV



Statistical limit:

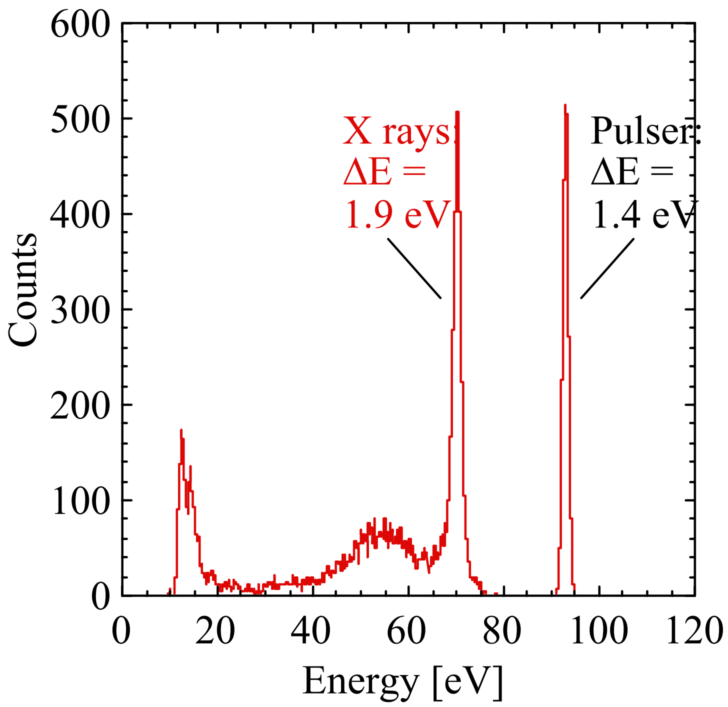
$$\Delta E_{FWHM} = 2.355\sqrt{\varepsilon E_x\left(F + 1 + 1/\langle n \rangle\right)}$$

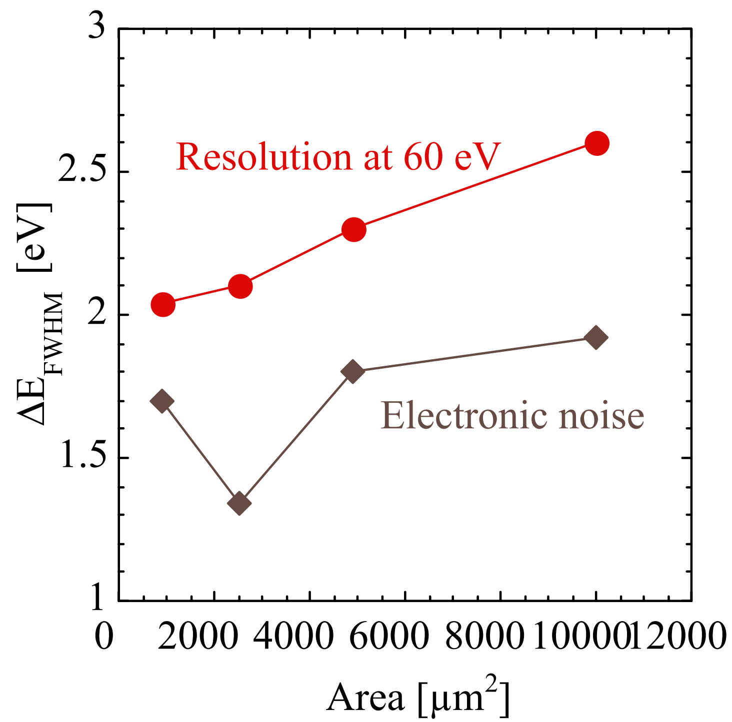
$\varepsilon \approx 1.7\Delta \approx \text{meV}$ = charge creation energy

F = Fano factor (charge generation noise)

$1 + 1/\langle n \rangle$ = charge tunneling noise

STJ Performance: Area Dependence

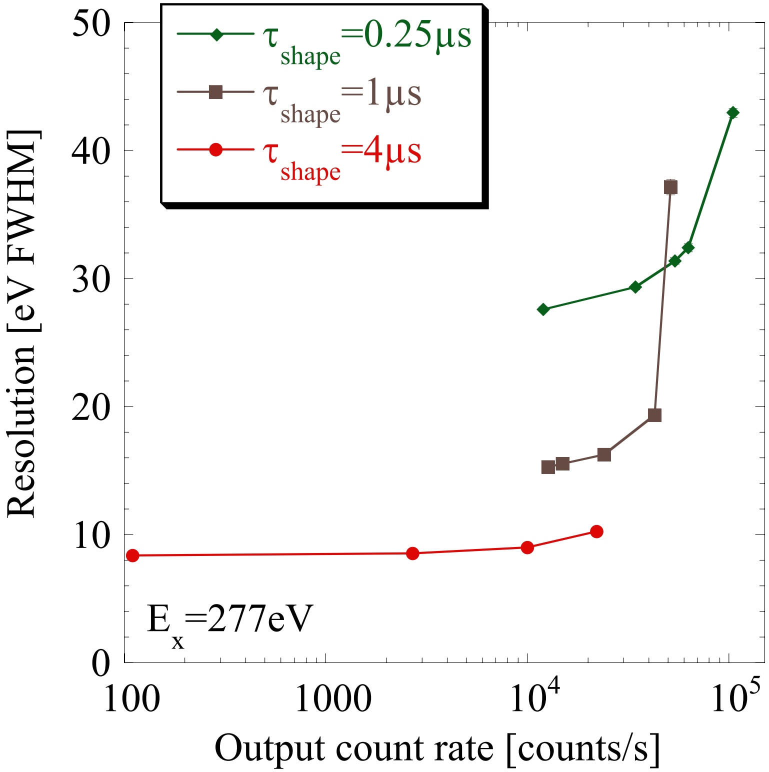




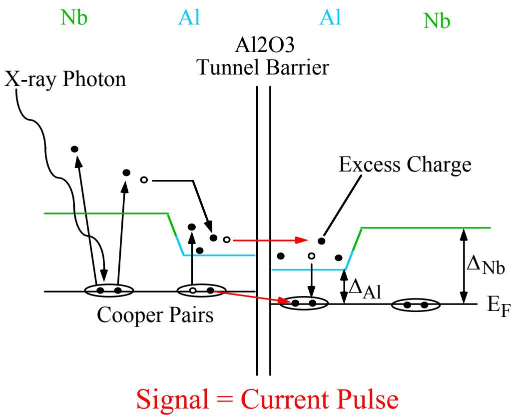
Low energy operation possible

Trade-off between area and resolution

STJ Detector Performance: Count Rate



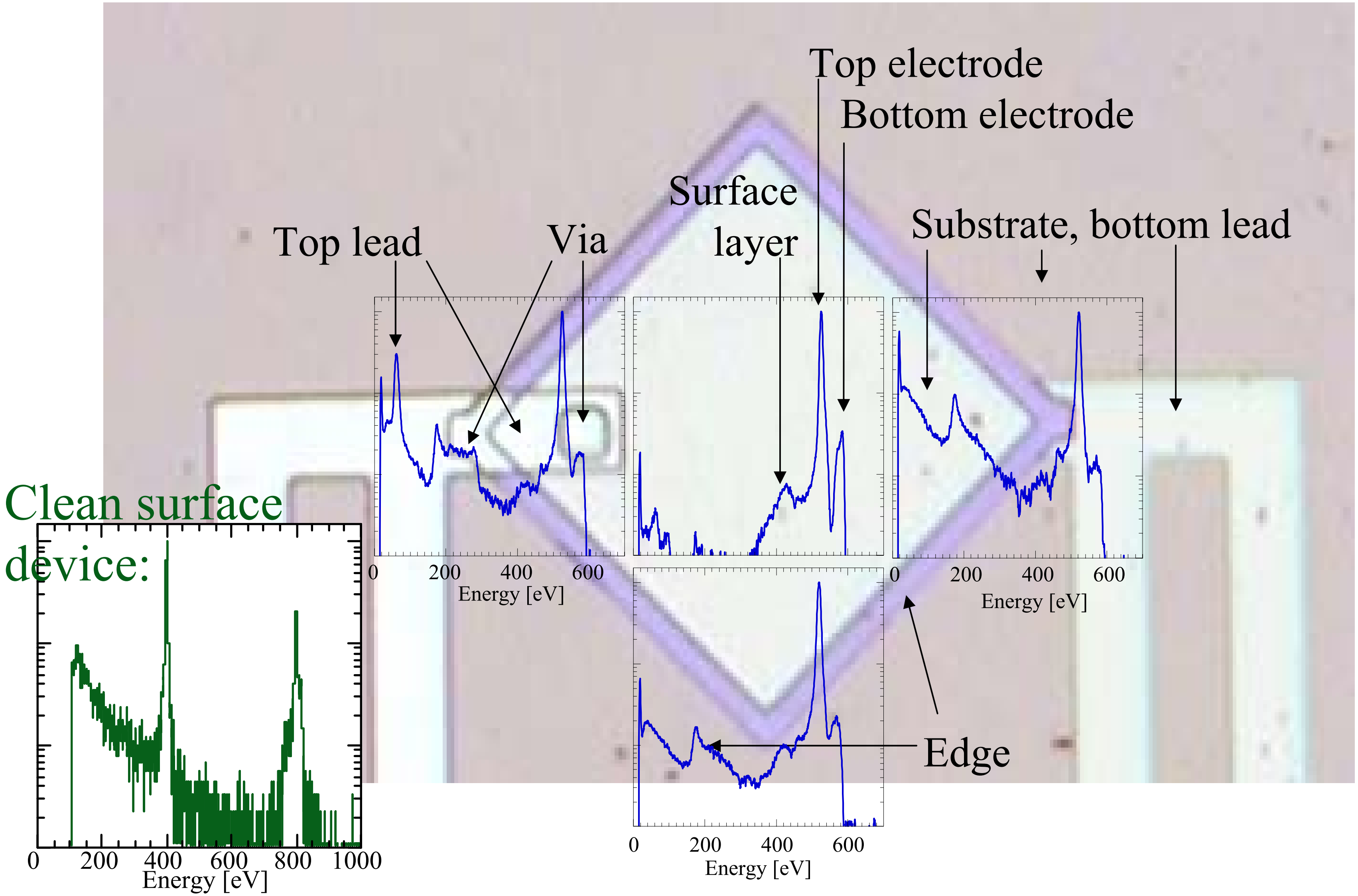
≥10,000 counts/s throughput



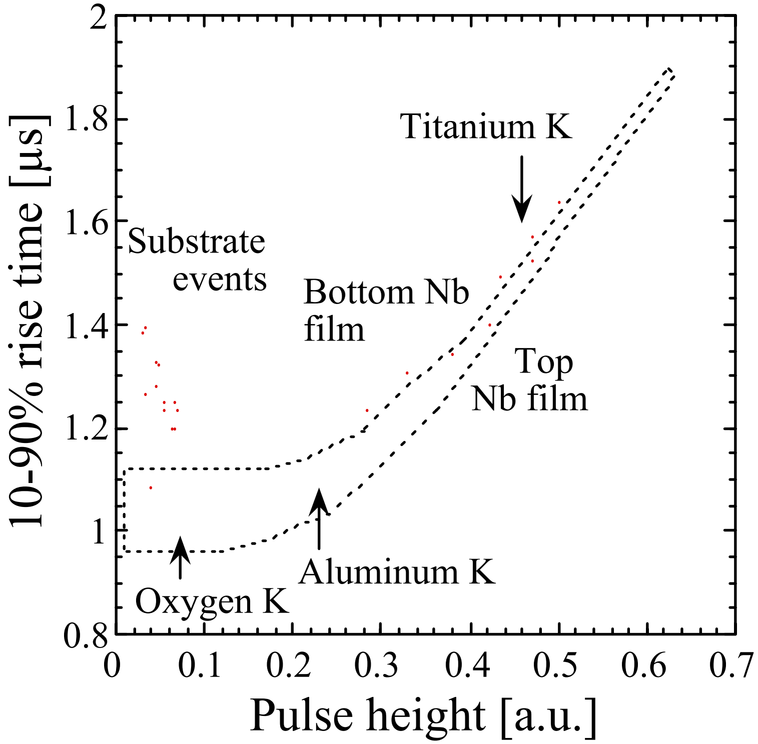
Current sensitive preamplifier :

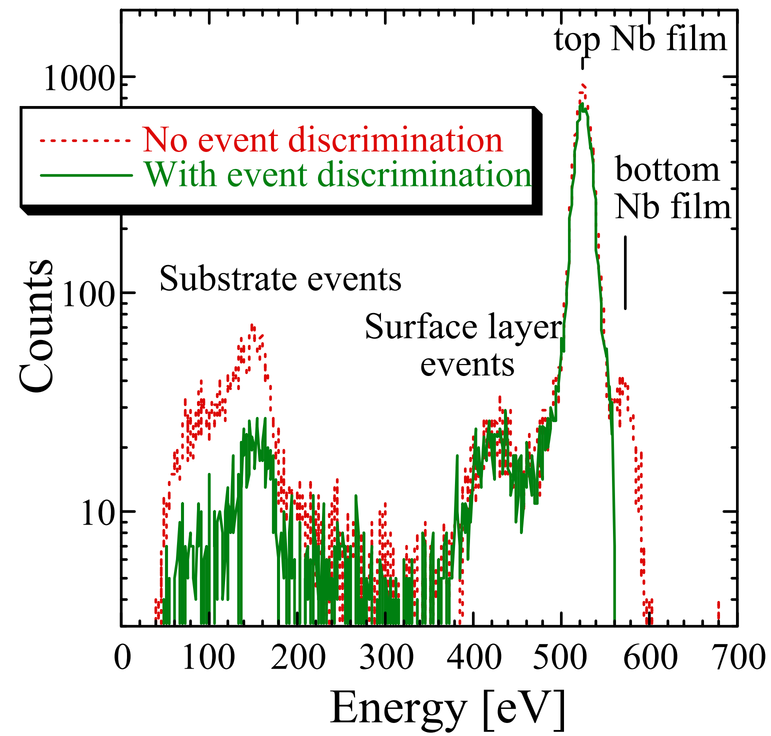
$\tau_{decay} = \tau_{life\ time} = \ \sim$ few μs

STJ Detector Performance: Lineshapes



Digital Signal Processing





Rise time differences between top and bottom electrode and substrate

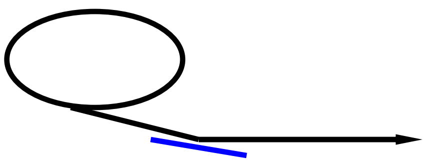
On-line event discrimination at 5000 cts/s to reduce artifacts

Digital signal processors courtesy of X-Ray Instrumentation Associates, www.xia.com

X-Ray Absorption Spectroscopy

Synchrotron beam

Intense, monochromatic, tunable
$I_0 \approx 10^{12}$ photons/s
$\Delta E = 0.1$eV



Monochromator

Sample x

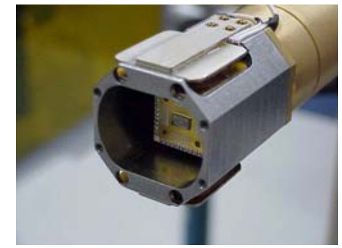
Absorption $\mu_x(E)$

Photons
(Fluorescence)

Electrons
(Auger, photoelectrons)

Detection

3) Fluorescence $\propto I_0 \cdot \mu_x$ (E)

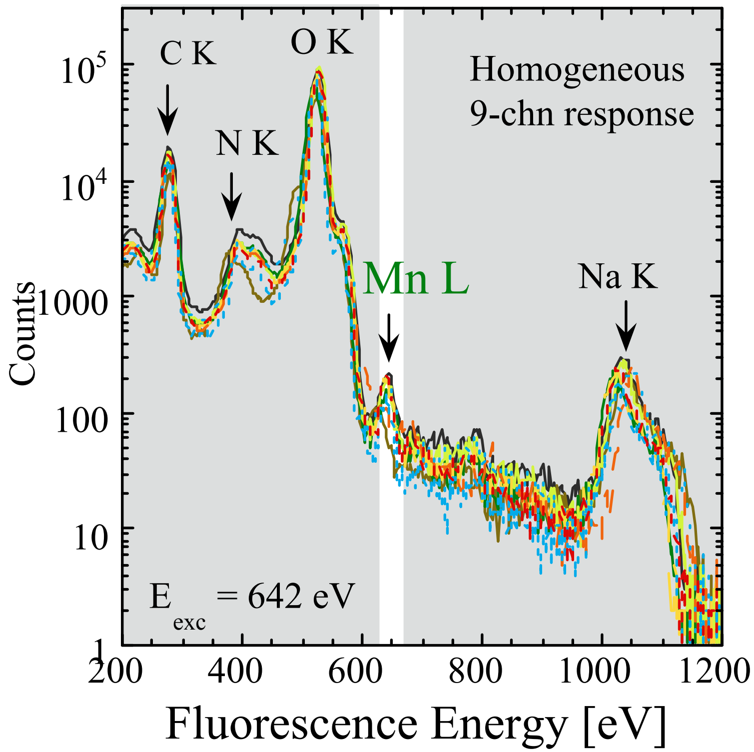


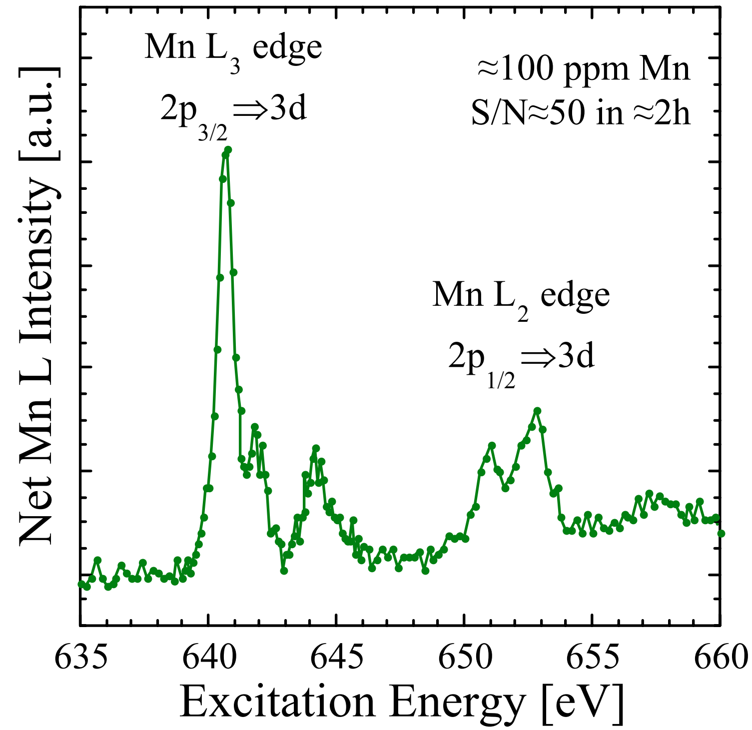
1) Transmission $\propto I_0\,(1-\mu_x-\mu_{bgnd})$
Thin samples
High background

2) Electron signal $\propto I_0 \cdot \mu_x$ (E)
Surface sensitive
Moderate background

ALS, BL 4.0.2., A.T. Young et al.,
Nucl. Inst. Meth A467, 549 (2001)

X-Ray Absorption Spectroscopy on Proteins



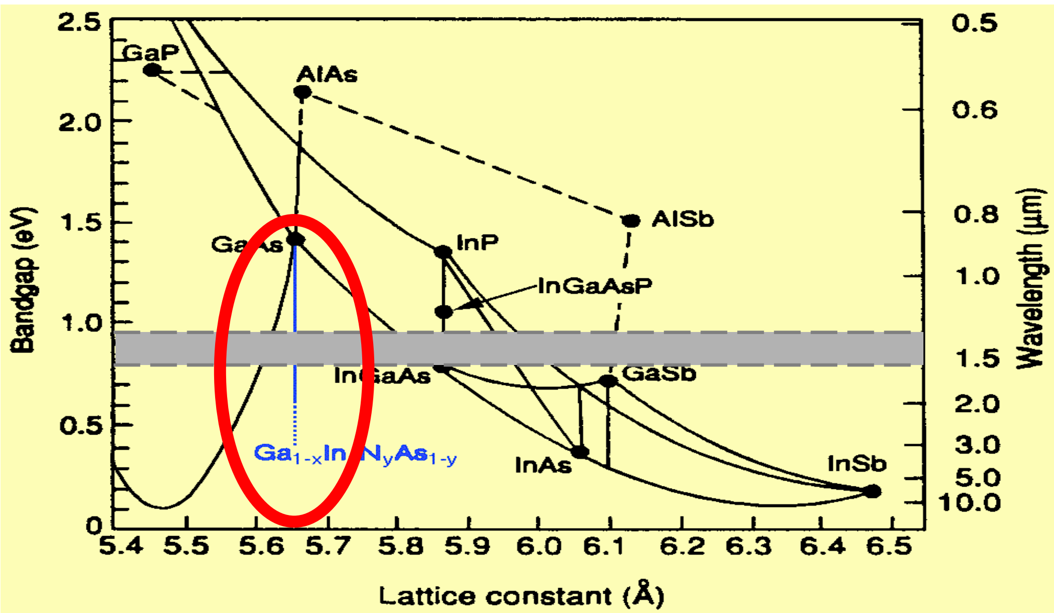


Spectrometer sufficiently sensitive for few ~100 ppm sample analysis.

Spectroscopy on active metal sites in proteins (photosystem II)

GaInNAs: A material for 1.3-1.55µm lasers

Collaboration with V. Lordi from Prof. Harris' group at Stanford



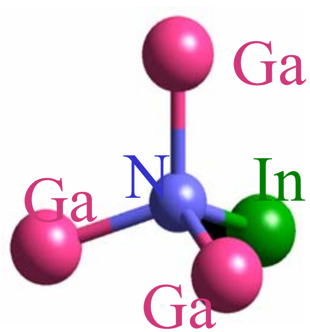
Anneal GaInNAs to increase luminescence.

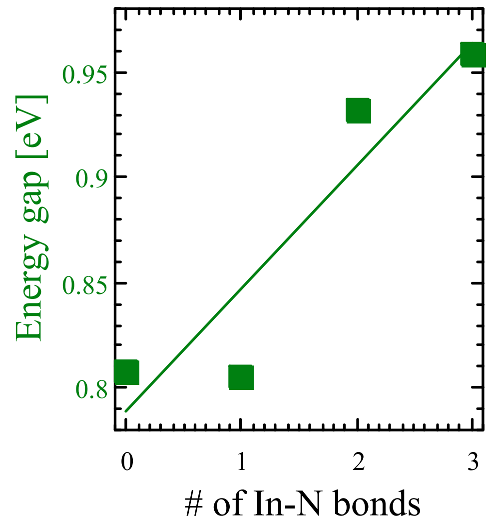
Telecommunications Application

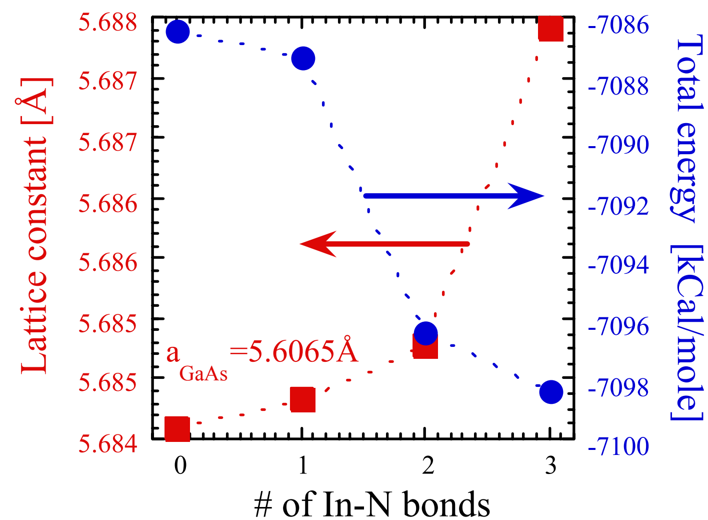
- $Ga_xIn_{1-x}N_yAs_{1-y}$ ($x \approx 0.70$, $y \approx 0.03$) has a bandgap of ~1.3 - 1.55 µm
- It is nearly lattice-matched to GaAs
- Fabrication of inexpensive surface-emitting lasers for optoelectronics
- Optical fibers best at ~1.3 - 1.55µm

Problem: Band gap increases upon annealing

Nitrogen nearest neighbors affect optical properties







- MBE, ~2% nitrogen
- III-V random alloy
- FCC lattice

- Anneal at 780 ˚C, 1min

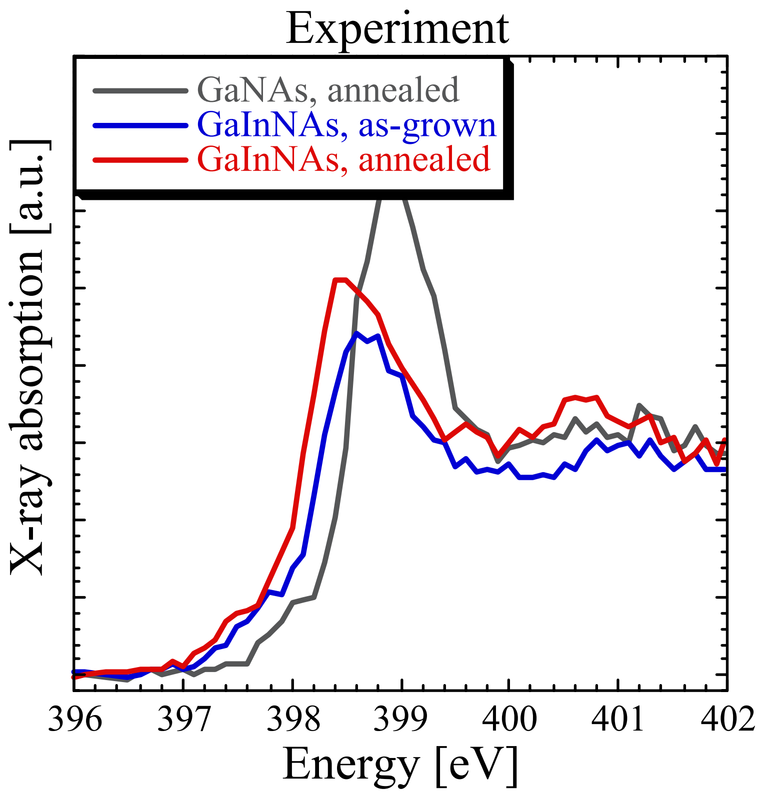
⇒ Luminescence increases
⇒ Bandgap increases

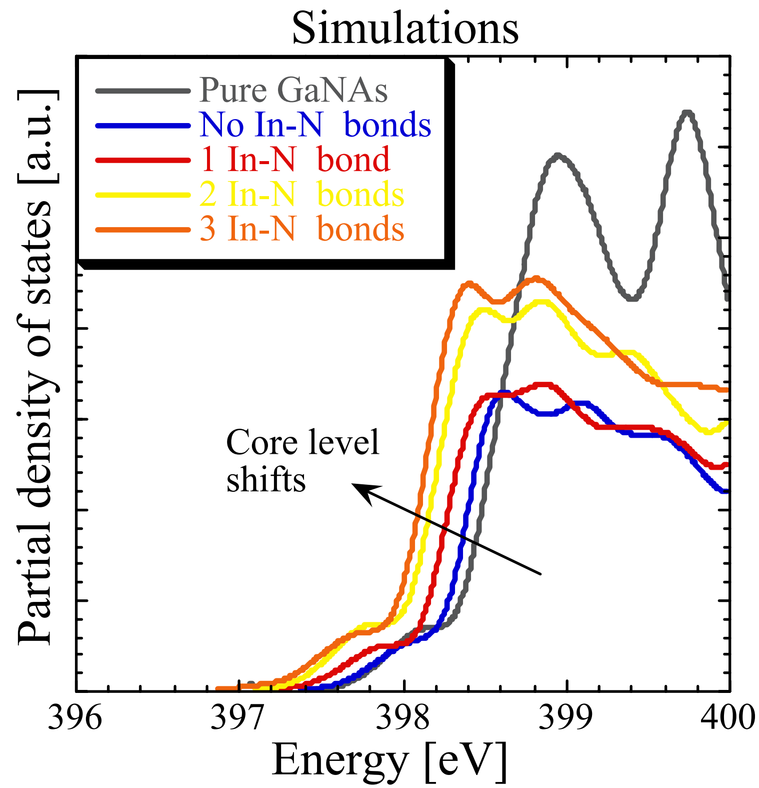
- Energy gap increases with # of In-N bonds

- Strain favors decreasing # of In-N bonds

- Thermodynamics favors increasing # of In-N bonds

Nitrogen X-ray absorption fine structure

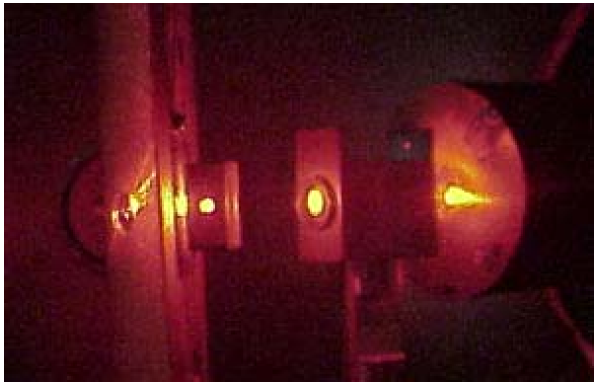


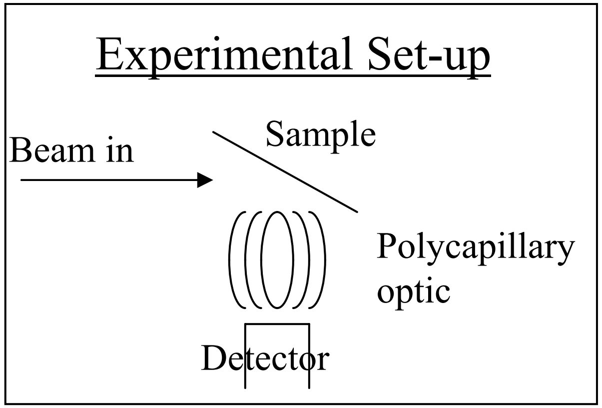


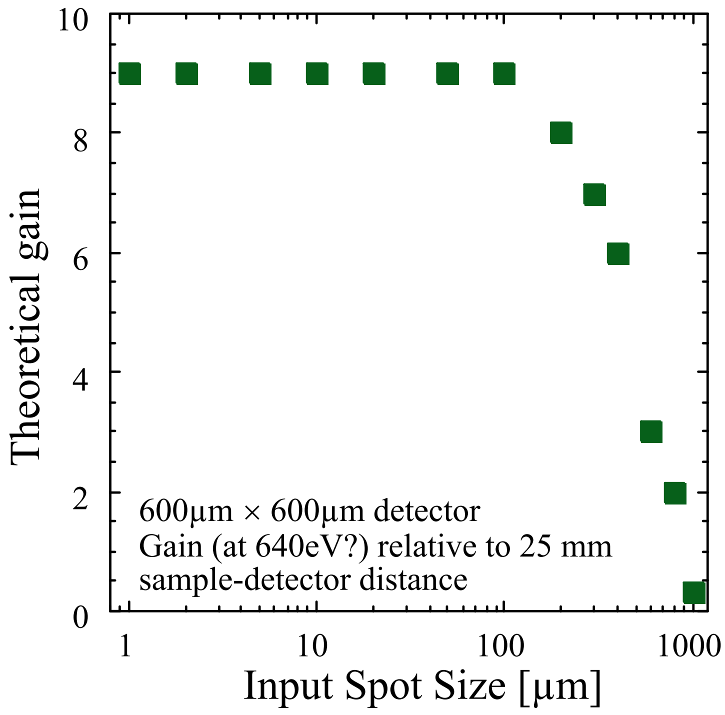
Absorption edge shifts show increasing number of N-In bonds
Nitrogen migrates towards Indium upon annealing

V. Lordi et al., Phys. Rev. Lett. **90**, 145505 (2003)

Current/ Future Work: Enhance Sensitivity







Spectrometer Development:

- Polycapillary Optic (courtesy XOS)
- Larger Arrays (6×6)
- Ta absorber for efficiency at higher E

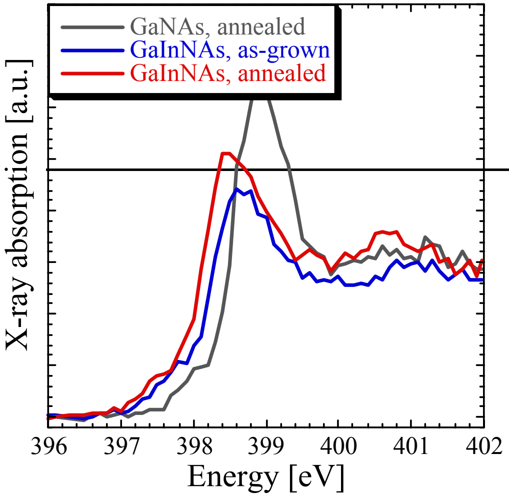
Summary

9-Channel STJ Detector Array

- <10-20 eV FWHM below 1keV
- >100,000 counts/s total count rate

ADR Cryostat with Cold Finger

- 70 mK Base T,
- 20h hold time below 0.4K



Fluorescence-Detected XAS on Dilute Samples

- Few 100 ppm samples with S/N≈50 in ≈1hour
- Protein biochemistry and material science

Current/ Future Work: Increase Sensitivity

- Larger arrays, Closer, Ta absorbers, Optics…