SAND2009-0357 P

Ion Beam Characterization of Advanced Luminescent Materials for Application in Radiation Effects Microscopy

07.21.10

Janelle V. Branson, G. Vizkelethy, K. Hattar, C.J. Powell, B.L. Doyle

Sandia National Labs, Albuquerque, NM

P. Rossi

Dep. of Physics at University of Padua and INFN, Padua, Italy

SEERI Summer 2010

Sandia is a multiprogram laboratory operated by Sandia Corporation, a Lockheed Martin Company, for the United States Department of Energy's National Nuclear Security Administration under contract DE-AC04-94AL85000.

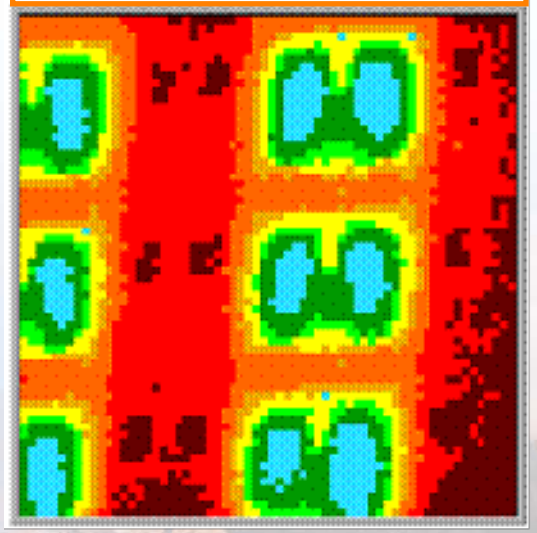
Sandia National Laboratories

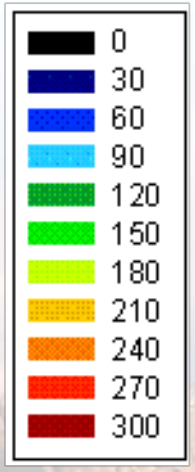
Introduction – Single Event Effects

Integrated circuits/devices facing exposure to radiation environments must be tested for reliability

- Broad beam testing (cyclotron)
  - cm X cm spot size
  - 100s of microns range at sufficient LET
- Micro-beam testing (Van de Graaff accelerators)
  - 1 micron or less spot size
  - 10s of microns range at sufficient LET

Ion beam induced charge (IBIC) imaging





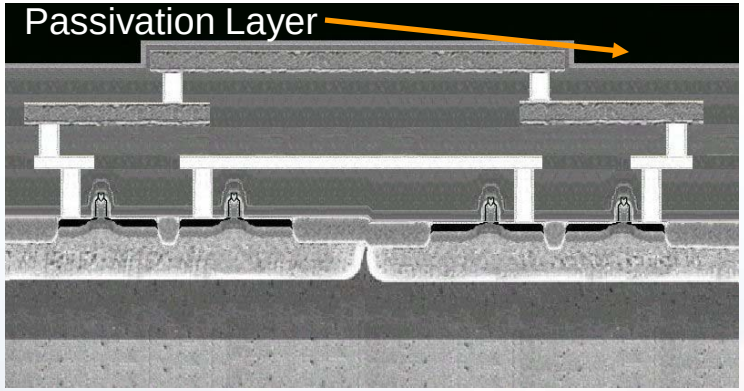
Charge (fC)

Single event upset (SEU) imaging

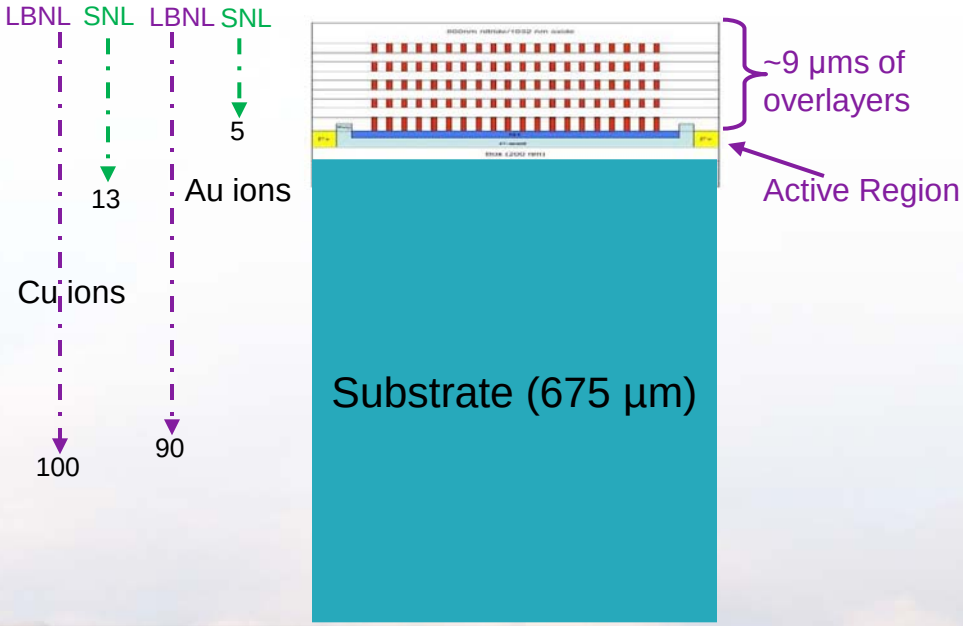
Background and Motivation

Radiation Effects Microscopy (REM) – used in IC design, pinpoints radiation intolerance to single event effects (such as single event upsets)

- Metal, dielectric and passivation layers (as well as flip-chip) → hundreds of microns of material → only high energy ion beams can penetrate
- Current GeV ions from cyclotron: a potential solution, but hard to focus due to high magnetic rigidity and get poor energy resolution



SEM image of CMOS transistor from Analytical Solutions, Inc.

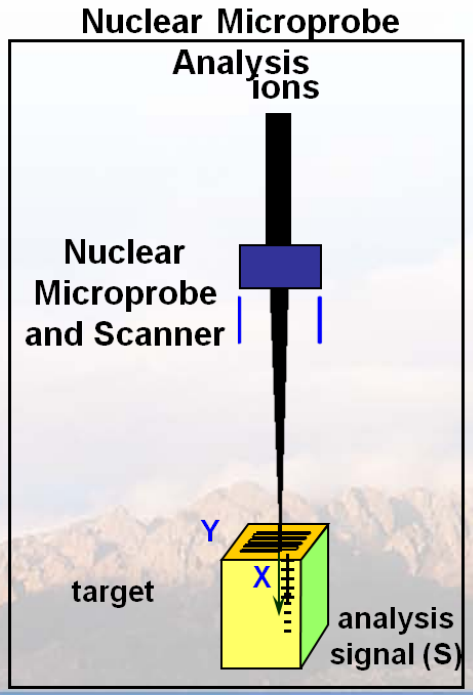


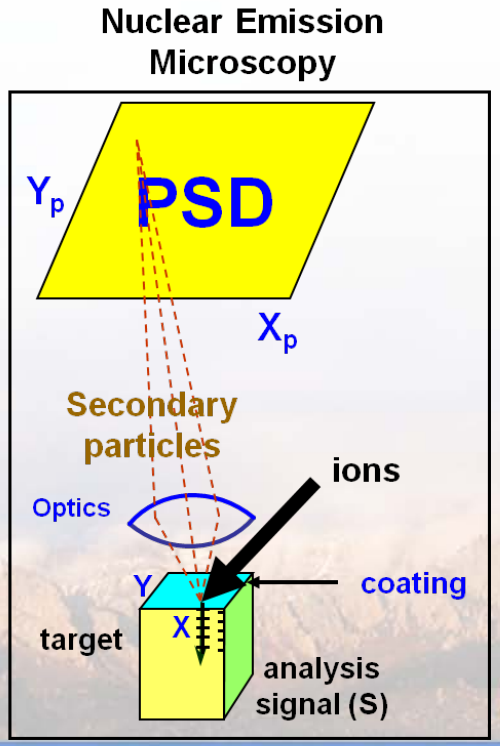
- Our approach (IPEM): use photons emitted from the location of the ion impact to determine the position of the ion, and thus rely on these photons and the ion's effect on IC to image radiation sensitivity.

Approach - Proof-of-Principle

This tabletop version of the IPEM was used to study various luminescent materials

- Ions have two major interactions
  1) Ions hit luminescent film and get ion beam-induced luminescence (IBIL)
  2) Ions hit chip and create ion beam induced charge (IBIC) and possibly upsets
- Photons produced by film detected with single photon position-sensitive
- detector (PSD)
- However, cannot cause single-event effects with a-IPEM

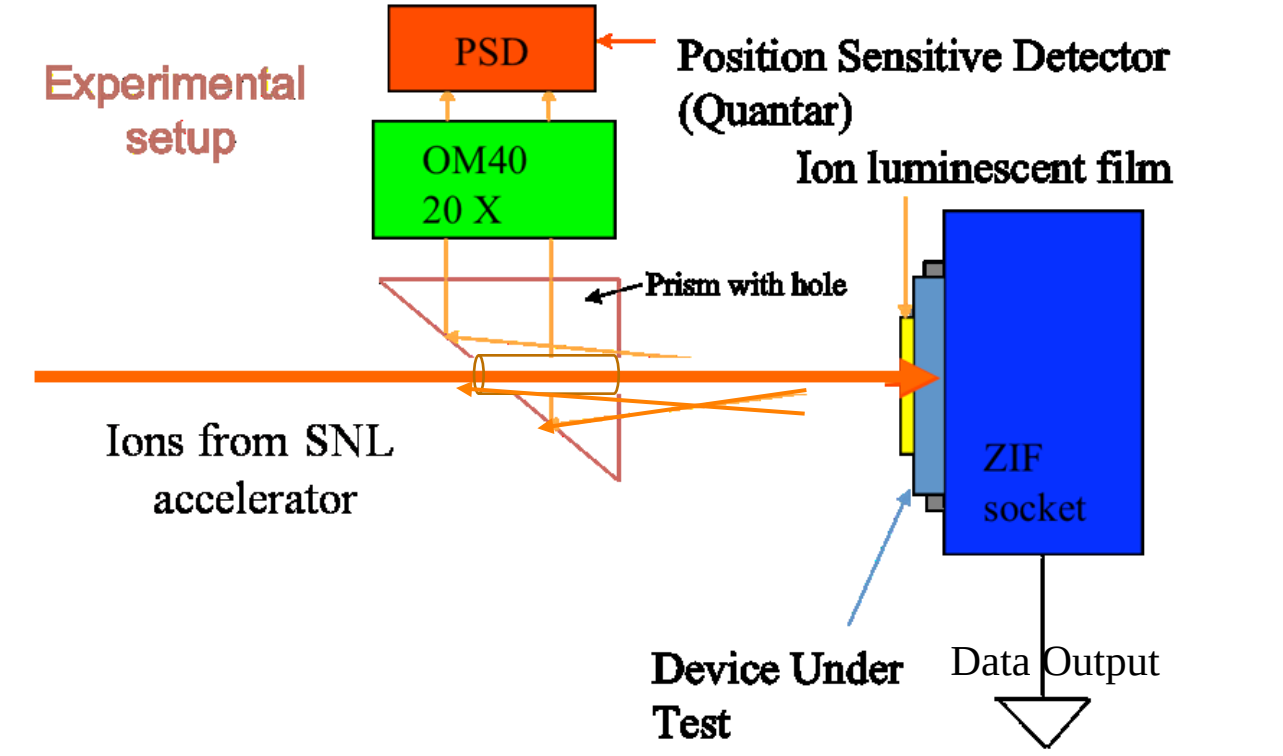




Approach – Accelerator IPEM

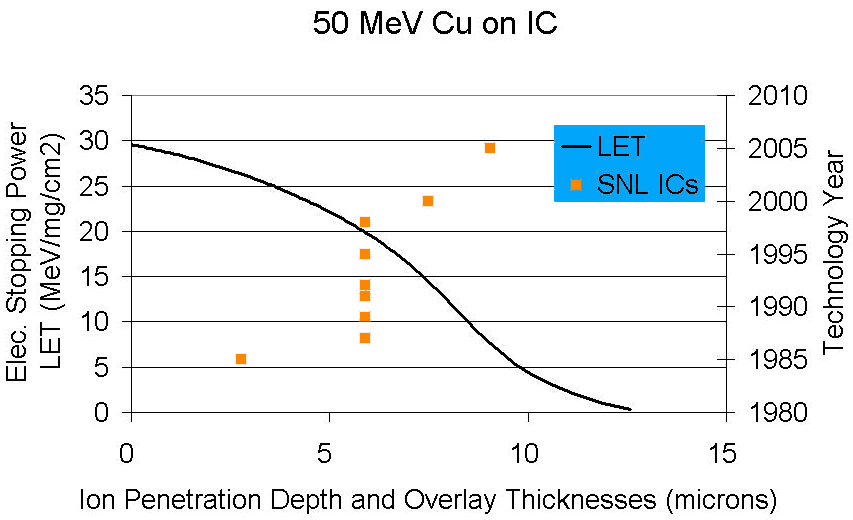
Developed IPEM system on nuclear microprobe line on SNL's tandem accelerator

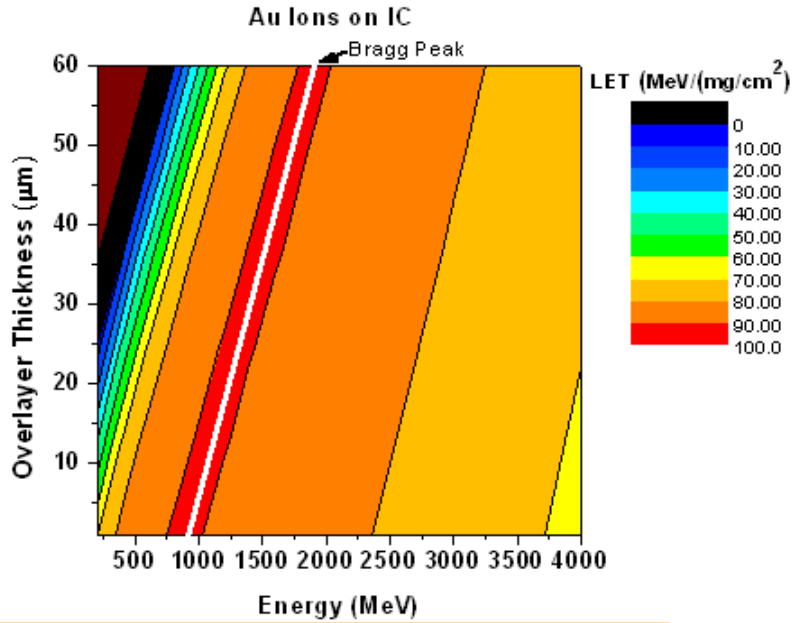
- Availability of various ion species/energies (2 MeV protons to 50 MeV Cu ions)
- Allows us to compare results obtained with traditional nuclear microscopy and IPEM technique



Approach – SNL Limitations

Advancing IC technology is hindering our ability to microscopically study radiation effects, even with ions from the tandem



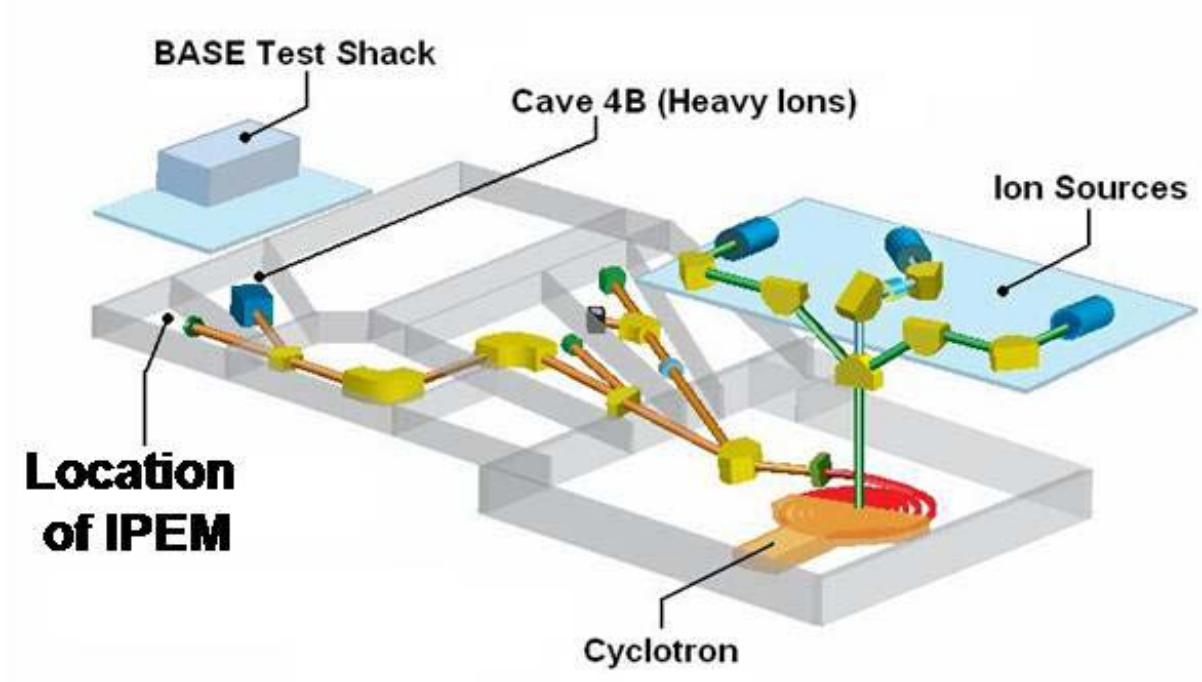


- New technologies continually increase the overlayer thickness on ICs, limiting our ability to probe the active region and study the devices using the tandem.
- Bragg peak for Au ions occurs at 0.9 GeV with an LET of ~90 MeV/(mg/cm^2); With 20 µm of overlayers, 1.2 GeV is required to position the Bragg peak into the sensitive depth.
- If start with lower incident energies, LET will decrease as material is penetrated, and before reaching the silicon active region.

Approach - Newest IPEM Location

In progress: developing an IPEM system on 88" Cyclotron at Lawrence Berkeley National Labs

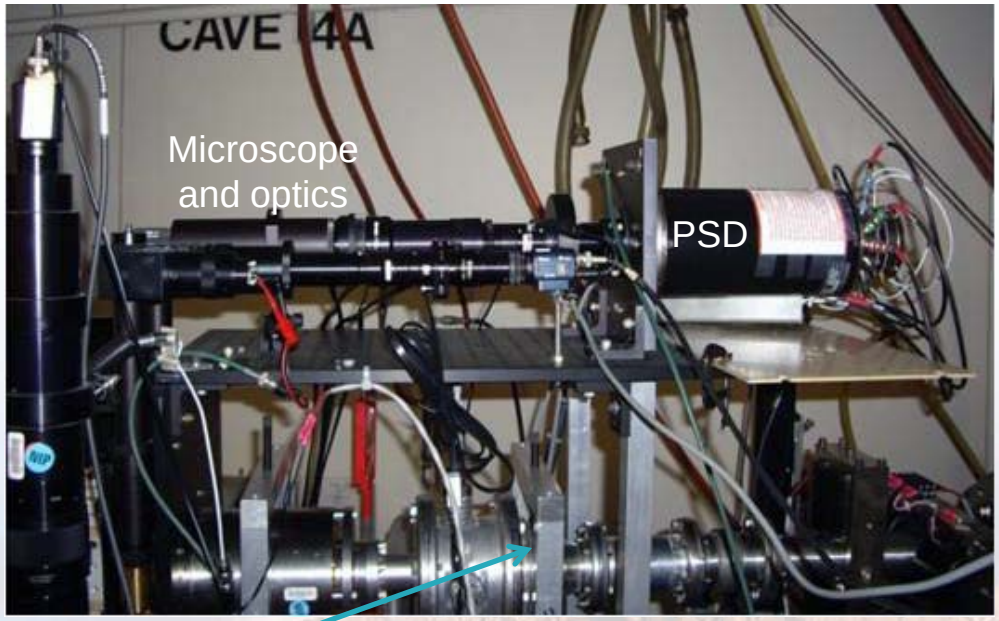
- Availability of various ion cocktails (4.5 MeV/amu to 16 MeV/amu heavy ions)
- Allows us to get ions with sufficient mass/energy to probe modern devices, while still operating externally
- Footprint of IPEM: ~5' X 2' optics and ~2' X 3' electronics
- System ideally will be portable



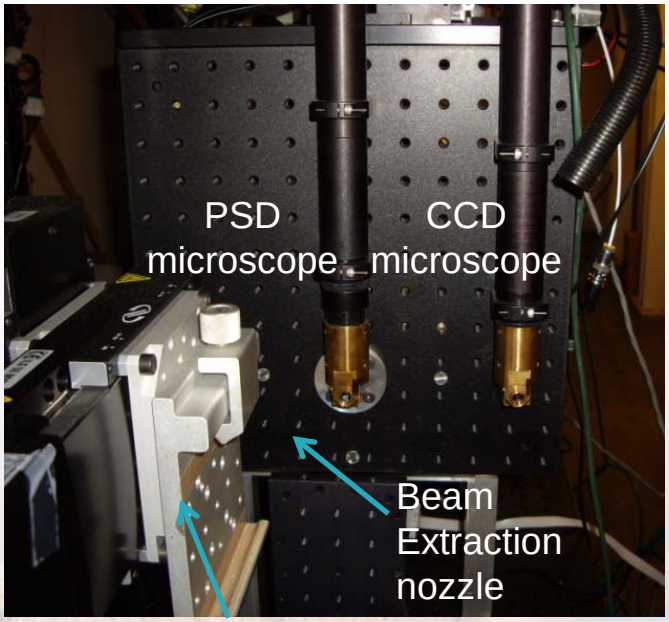
Approach - Design of Cyclotron IPEM

Mark I IPEM on LBNL's 88" Cyclotron

- Tandem IPEM was significantly modified for initial attempt at cyclotron capability.
  - Separate optics lines for CCD and PSD imaging
  - Aperture for self-standing GaN
  - Exit foil for in-air operation
  - Remote control of all operations



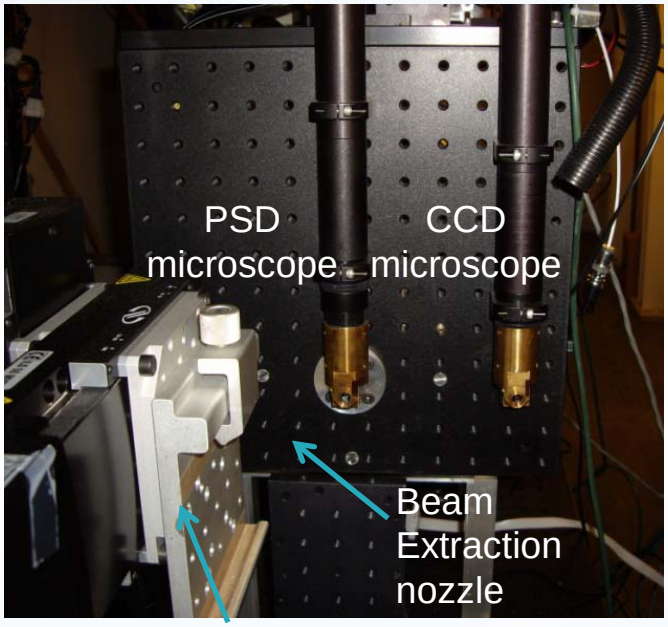
Cyclotron beamline



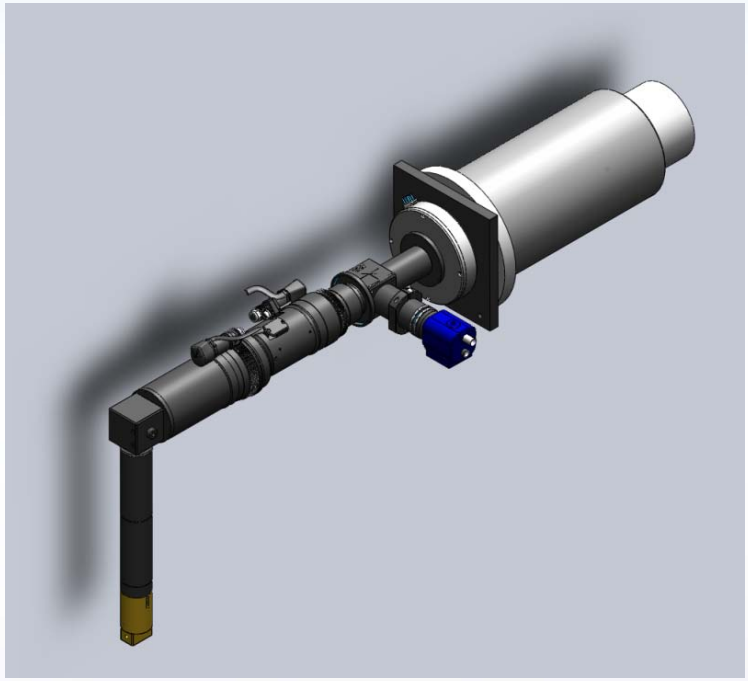
3D stage for device board

Approach - Design of Cyclotron IPEM

Mark II IPEM on LBNL's 88" Cyclotron



3D stage for device board



- Mark I had trouble with focusing – translation vector between two microscopes was not reliable
- Mark II had one single optics line with a 92/8 mirror splitting the light between CCD and PSD; locations of all optics pieces have been optimized
- Mark II currently being installed at LBNL – awaiting first test

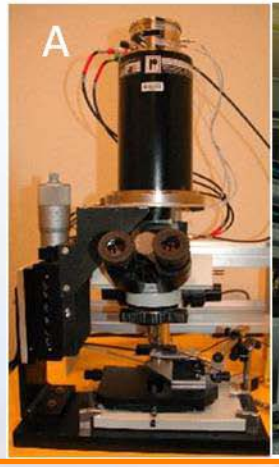
Research - Techniques

SNL's Tandem Accelerator

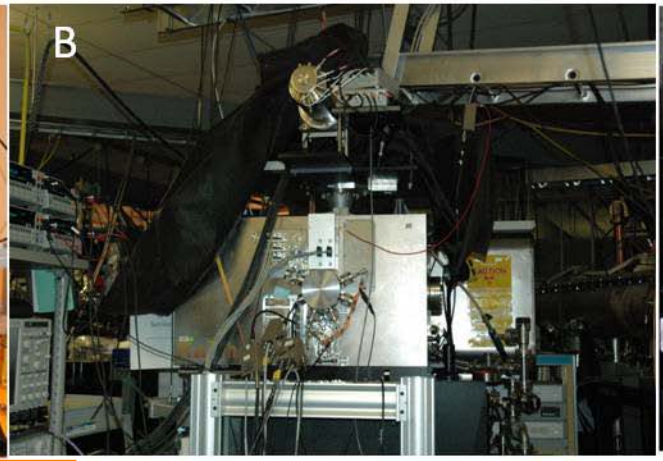
- Excitation source:
  - 7.5 MeV α particles
  - 30 MeV C^{5+} ions
  - 35 MeV Cl^{5+} ions
- Focused scanning microbeam
- 5 μm GaN film
- One OM-40 microscope
- Bialkali single photon position-sensitive detector

LBNL's 88" Cyclotron

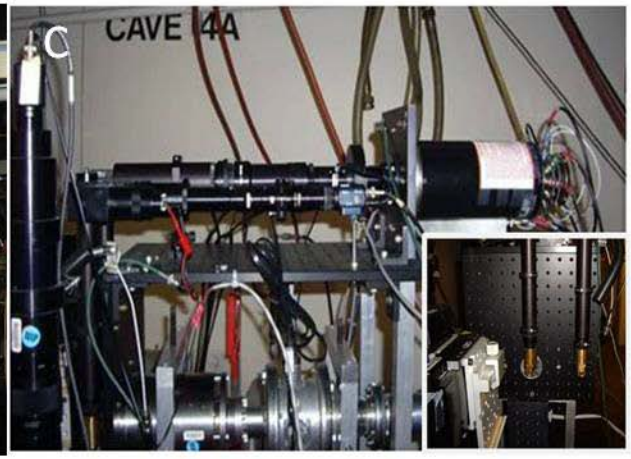
- Excitation source: 10 MeV/amu ion cocktail
  - B^{+}, O^{5+}, Ne^{6+}, Al^{7+}, Ar^{11+}, Cu^{18+}, Co^{16+}, and Xe^{38+3}
- Broad (~ 1mm) beam
- 5 μm Al exit foil for in-air
- Filter to eliminate air luminescence
- S25 single photon position-sensitive detector



Tabletop IPEM



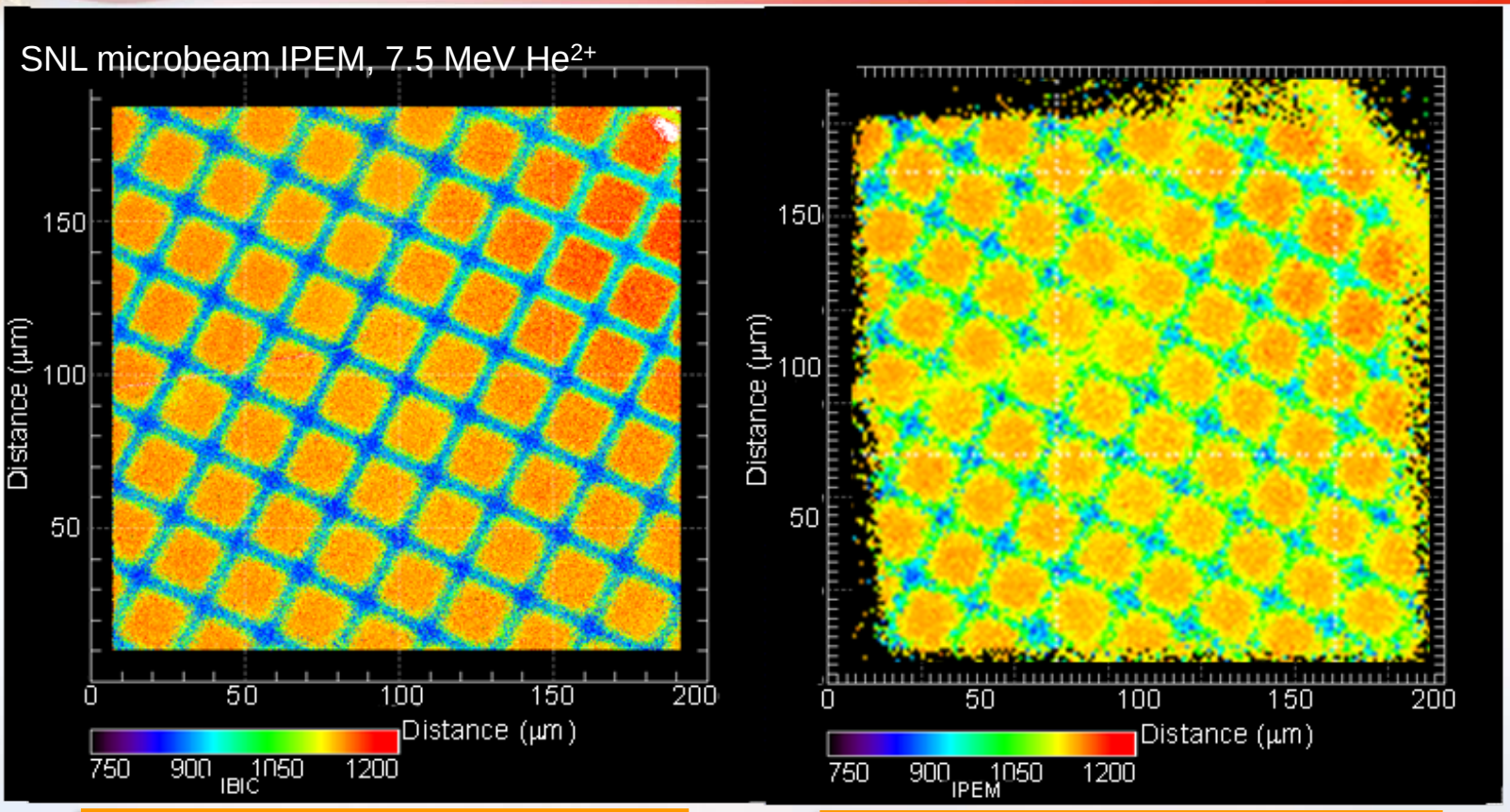
IPEM on SNL's micro-ONE beamline



Cyclotron-IPEM

Research – SNL Results

Focused beam IBIC and IPEM IBIC from SNL



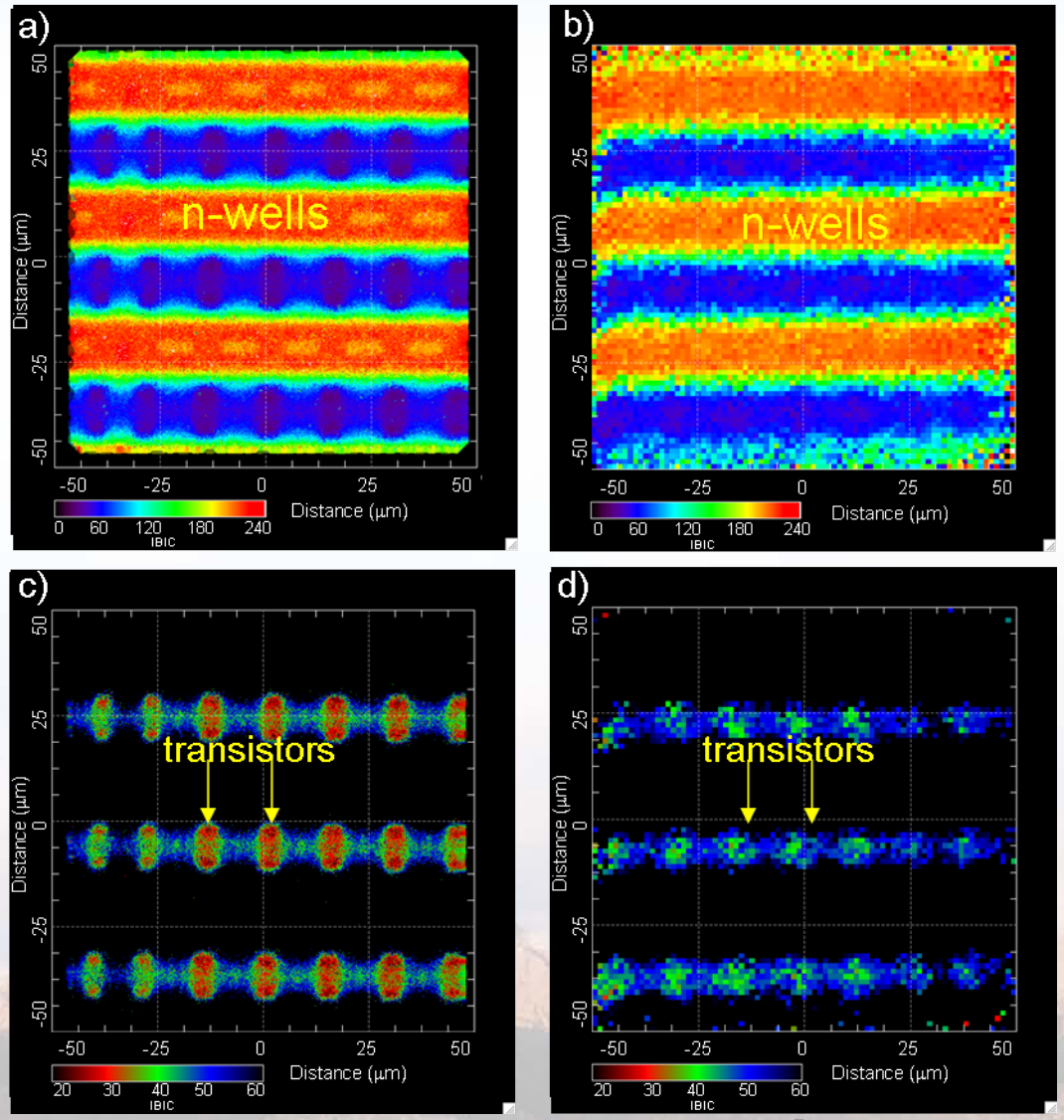
- New IPEM system installed on SNL's tandem accelerator
- IBIC on diode + TEM grid + GaN film

- IPEM IBIC image
- Signal from diode put in coincidence with photons arriving at detector
- See damage, cracks in GaN

Research – SNL Results

Focused beam IBIC and IPEM IBIC from SNL

- IBIC images of TA788 from scanning microbeam of 30 MeV C^{5+} ions
- n-wells easily distinguishable – high charge collection in junctions between n-well and p-substrate
- Intensity range modified in bottom image to resolve details in p-substrate
- IBIC on device + GaN film

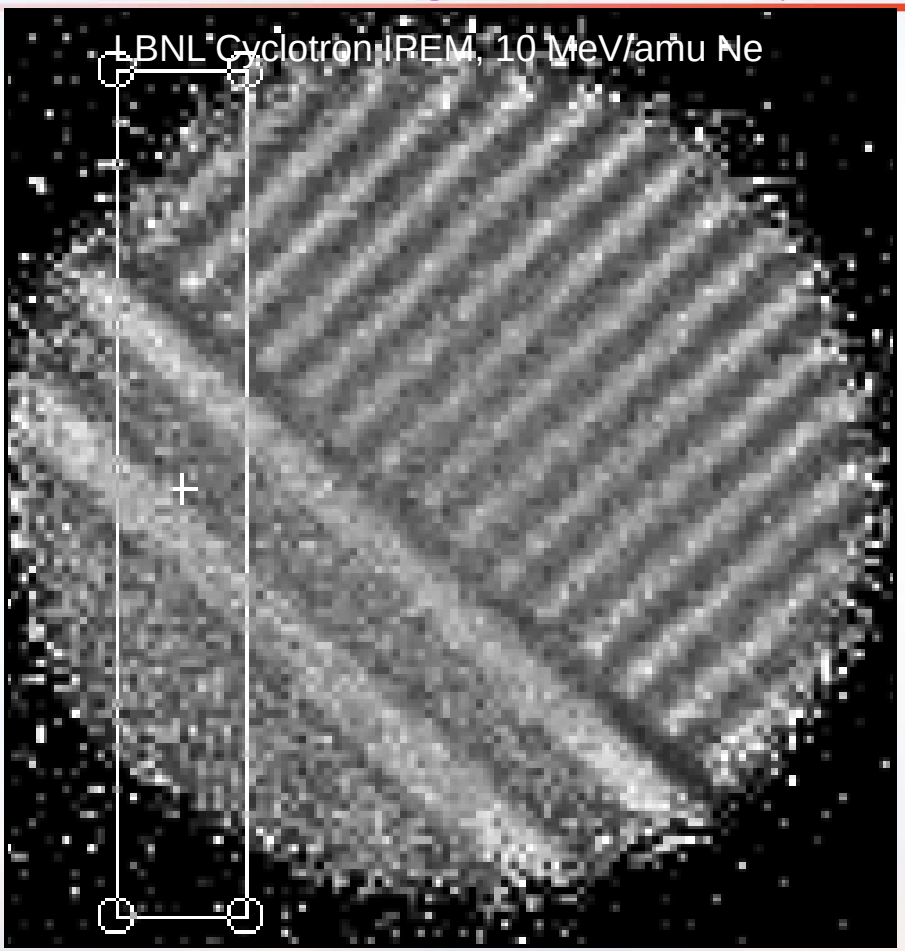


- IPEM/IBIC image of TA788 with 5 µm GaN as luminescent film
- Can still distinguish details, but resolution not comparable to focused scanned-beam IBIC
- Intensity range modified in bottom image to resolve details in p-substrate
- IPEM/IBIC on device + GaN film

SNL microbeam IPEM, 30 MeV C^{5+}

Research – Cyclotron Results

Broad beam IPEM image from LBNL cyclotron



- First IPEM image obtained from LBNL cyclotron
- Device is a Sandia TA788 64k SRAM
- Bright bands at ~10-12 μm
- Current resolution of this system is ~5 μm
- Cyclotron IPEM is not yet optimized

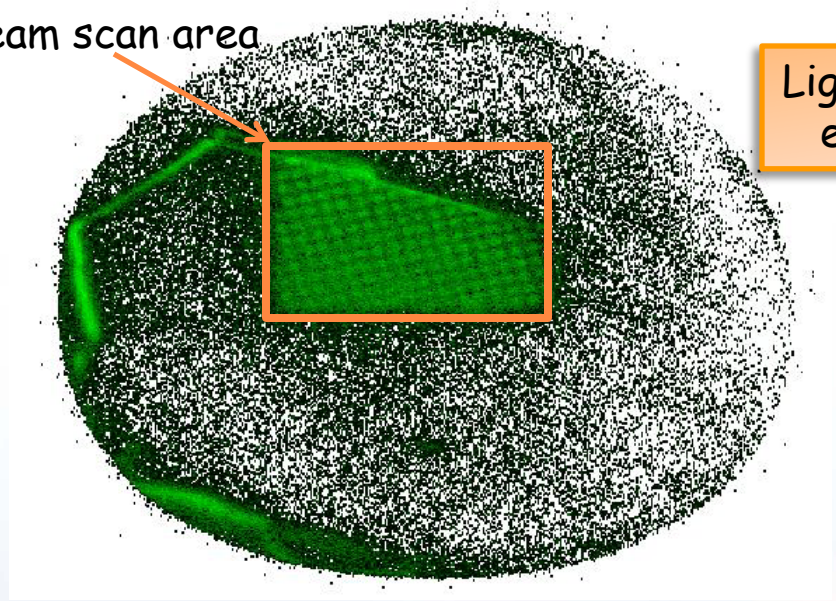
Background – Phosphor Requirements

A variety of parameters need to be met for phosphor to work in IPEM application

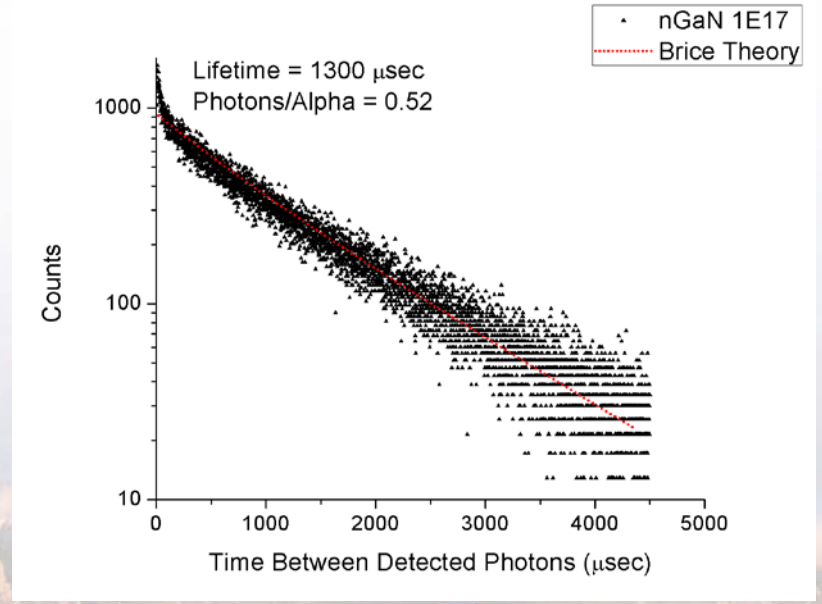
- Emission wavelength to match detector sensitivity
- High efficiency (detect at least 1 photon/ion)
- Short luminescent decay time
- Easy to handle
- Thin single crystal or nanophosphor layer to optimize resolution
- Tolerant to radiation damage, esp. at GeV energies for long periods of time
- Homogeneous luminescent properties

Research – GaN as Luminescent Material

GaN demonstrated problems with light traveling and long lifetime



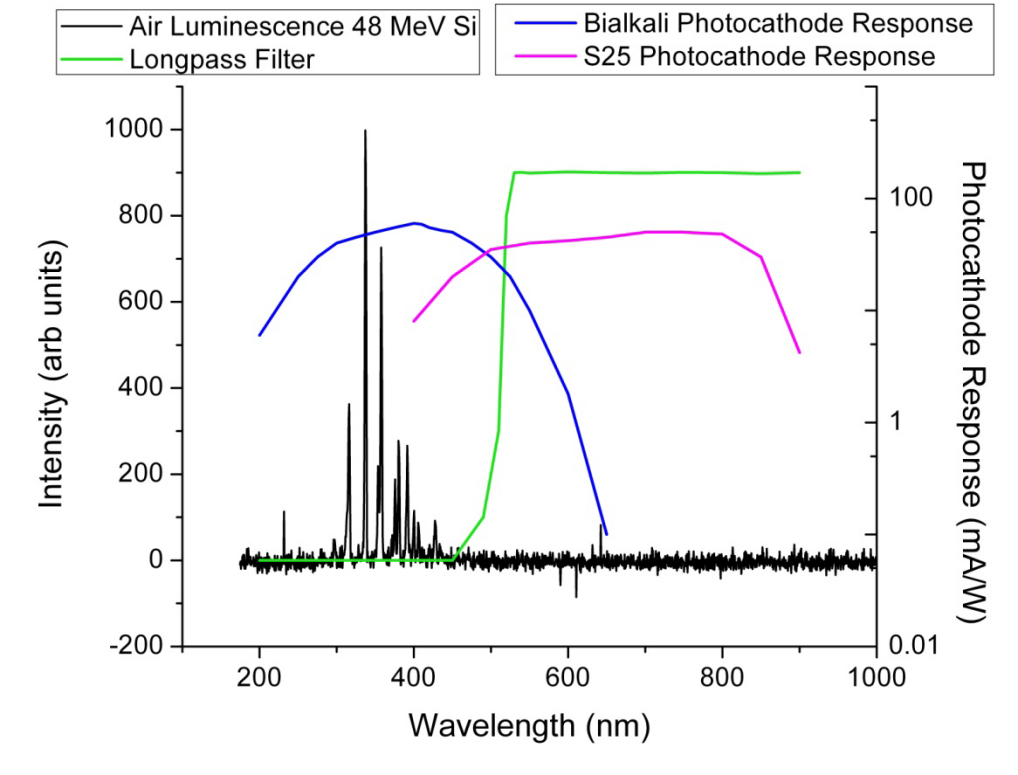
Light from GaN travels through material and exits at cracks/edges, limiting resolution



Long luminescent decay time of nGaN increases chance of accidental coincidences

Approach – Design of Cyclotron IPEM

Air luminescence must be considered for ex-vacuo system



- When testing Mark I, realized problem of air luminescence
- To maintain ease-of-use with ex-vacuo system, had to re-design system
- Mark II uses a longpass filter to cut out air luminescence and PSD with a S25 photocathode – sensitive to the longer wavelengths
- This also changed the materials of interest

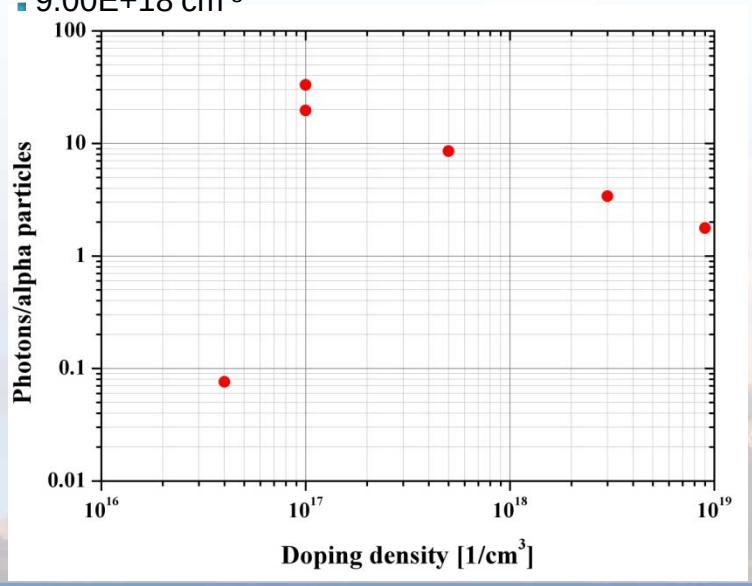
Background – Materials Studied

Variety of materials tested for application in IPEM

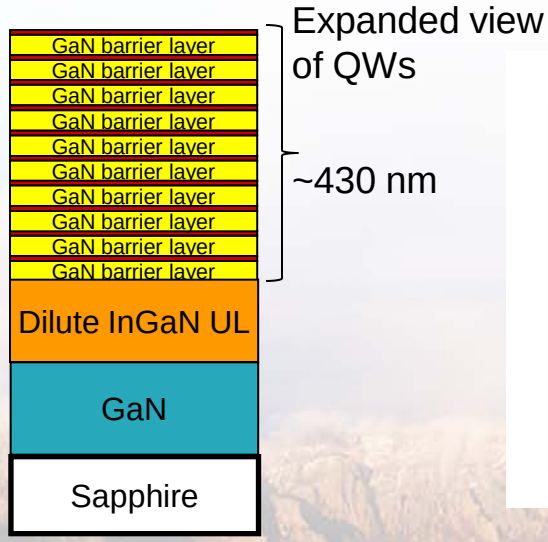
- 3 types of materials are currently the focus of our phosphor research:
  - Plastic scintillators (Bicron)
  - n-type GaN; InGaN/GaN quantum wells
  - Lanthanide-doped ceramic phosphors (YAG:Ce, Y_2O_3:Eu, etc.)

n-GaN Materials Studied

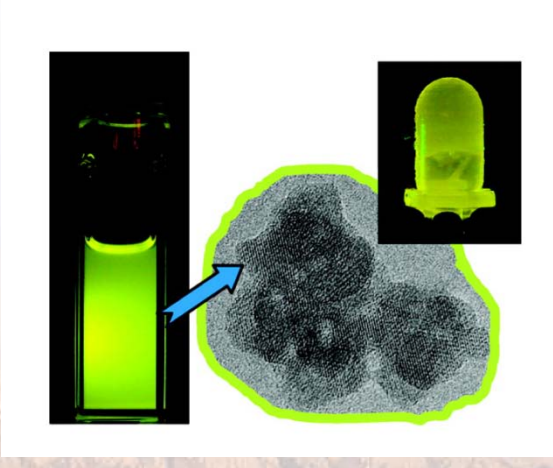
- 4.00E+16 cm^{-3}
- 1.00E+17 cm^{-3} **
- 5.00E+17 cm^{-3}
- 3.00E+18 cm^{-3}
- 9.00E+18 cm^{-3}



Example Quantum Well Structure



Nanophosphors



Experimental – Photoluminescence & Cathodoluminescence

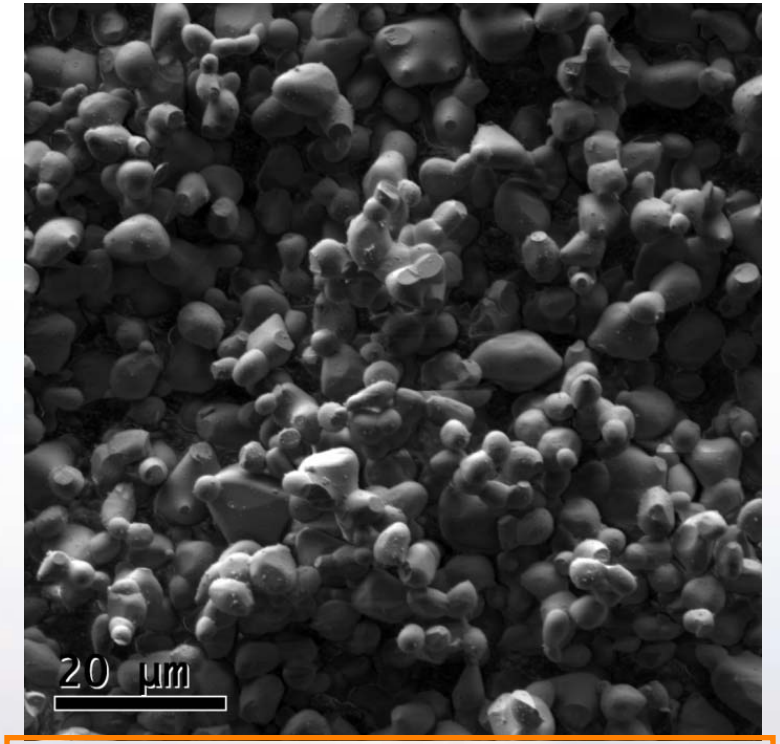
It is critical to study materials with various excitation mechanisms

Photoluminescence

- PL measurements performed with a Horiba Jobin Yvon Fluorolog spectrofluorometer
- Halogen lamp excitation with monochromator to select wavelength
- Samples excited at 337 nm unless observed emission depending on excitation wavelength

Cathodoluminescence

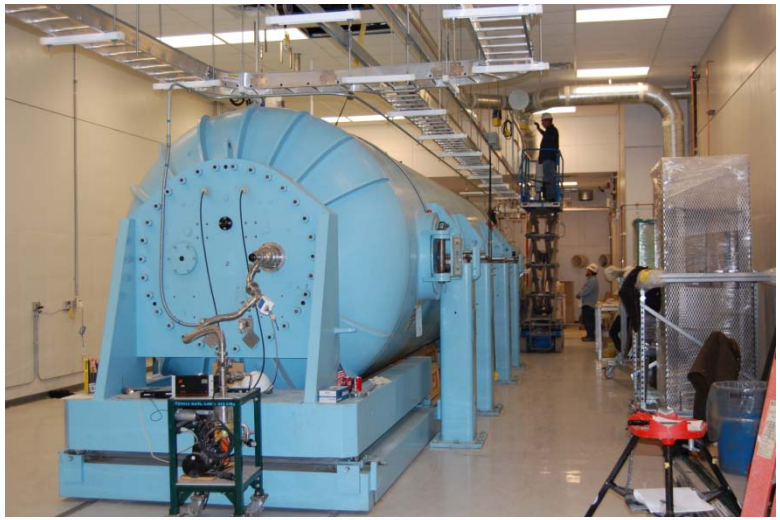
- 3 kV electrons, 2.6 nA current
- PMT operating at -750 V
- Spot size = 6
- Magnification: 1300X or 5000X



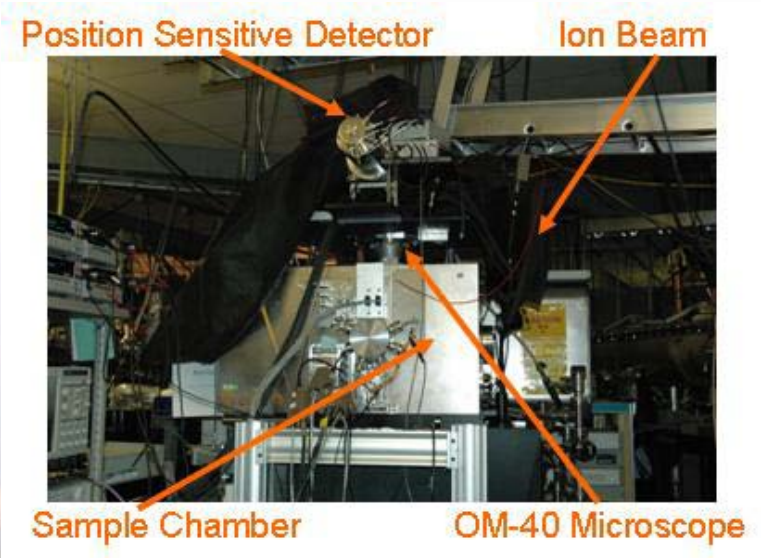
1300X SEM image of Y_2O_3:Eu powder – location of CL spectrum

Experimental – Ion Beam Induced Luminescence

IBIL experiments performed with tandem or Van de Graaff accelerator



- 2.5 or 4.5 MeV H+ beam used as excitation mechanism
- Light collected with OM-40 microscope or fiber optics mounted close to sample
- Avantes AvaSpec 2048 spectrometer



Experimental – Luminescent Decay Times & Radiation Hardness

Decay and radiation hardness are critical parameters for application

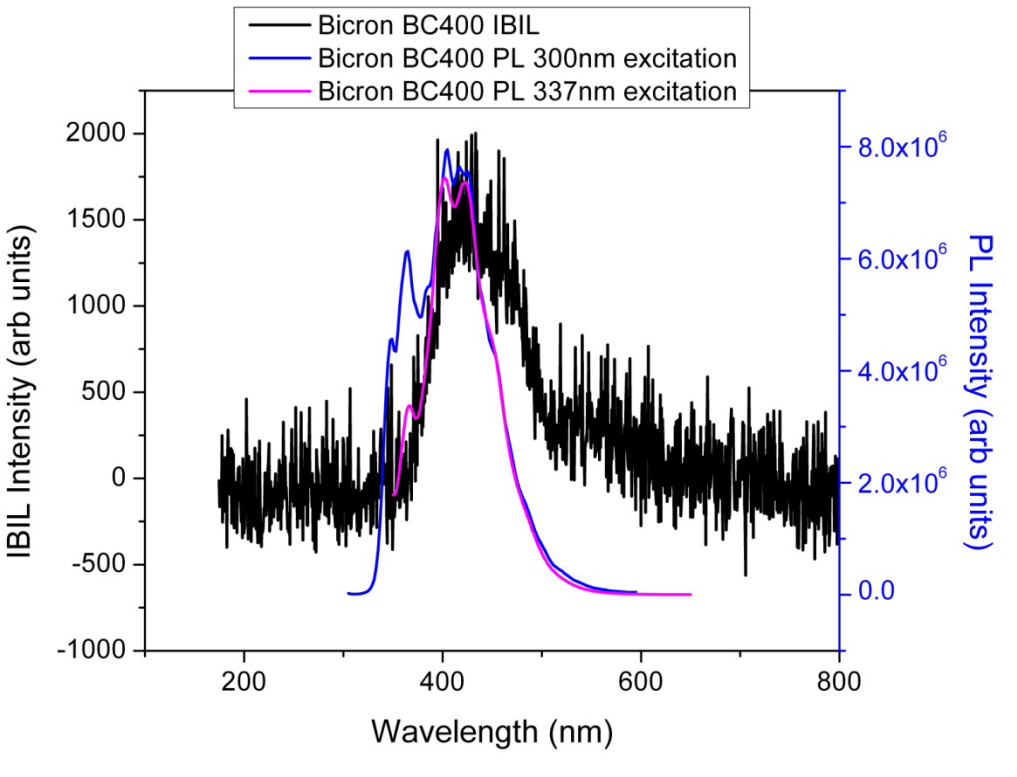
Decay Times

- Photon Technology International (PTI) GL-3300 nitrogen pulsed laser used as excitation method
- Light collected with a ThorLabs 210 or PDA-55 silicon photodiode
- Signal put directly into Tektronix TDS 5104 digital phosphor oscilloscope
- Light intensity measured as a function of time after laser pulse

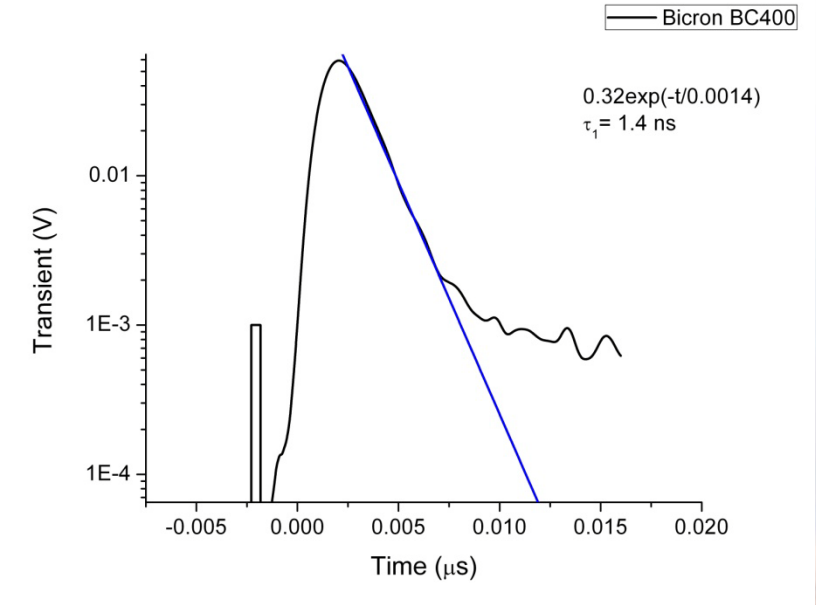
Radiation Hardness

- Radiation hardness experiments performed with 4.5 MeV H^+ beam from tandem accelerator
- Thin films of samples were mounted on PIN diodes
- Hamamatsu PMT was used in single photon counting mode
- Counters measured the number of IBIC signals from PIN diode (# of ions) and number of photons hitting PMT
- Experiment repeated at same position on sample for a period of time

Results - Bicron

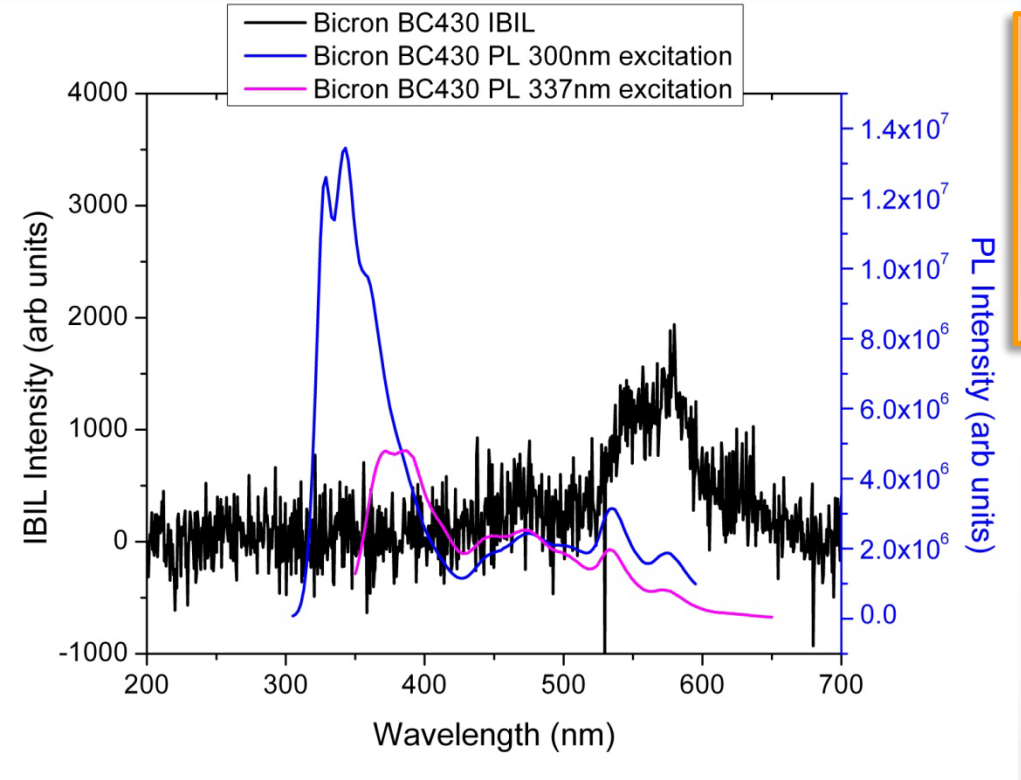


- BC400 is a blue-emitting organic scintillator
- Excitation wavelength can affect emission spectrum
- These emission wavelengths will overlap air luminescence

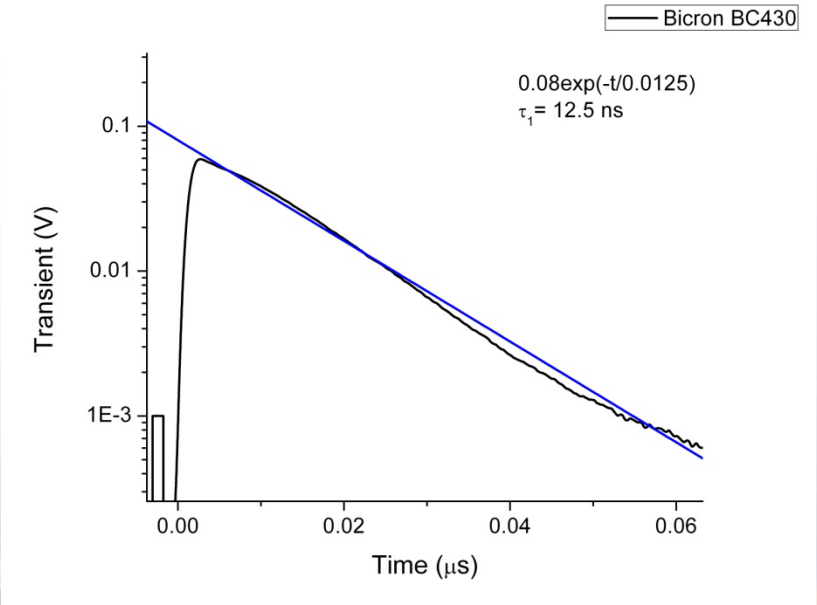


- BC400 has a very fast decay time
- Reported : 2.4 ns

Results - Bicron



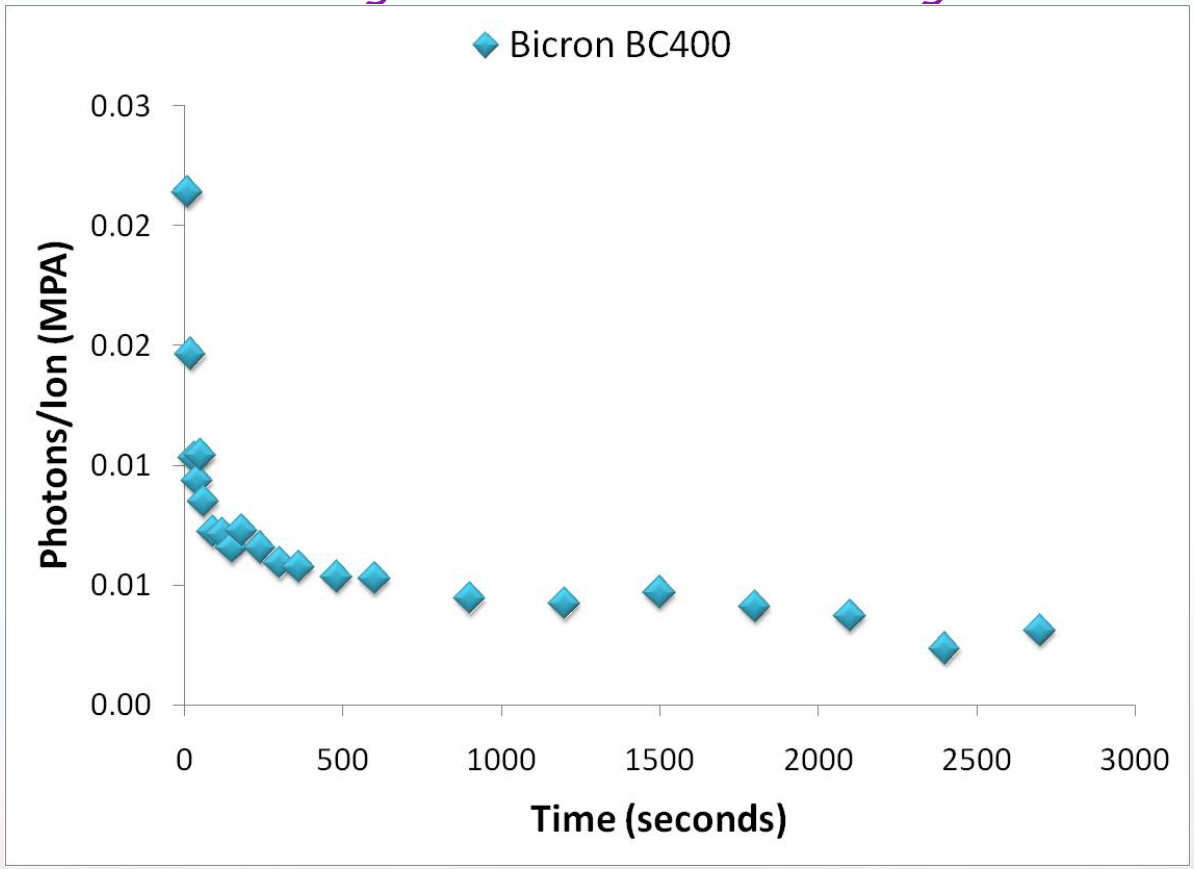
- BC430 is marketed as a red-emitting organic scintillator
- With PL, see very intense UV emission
- Red emission could be useful – long enough to avoid air luminescence



- BC430 also has a very fast decay
- Reported : 16.8 ns
- Emission spectrum and decay time show potential

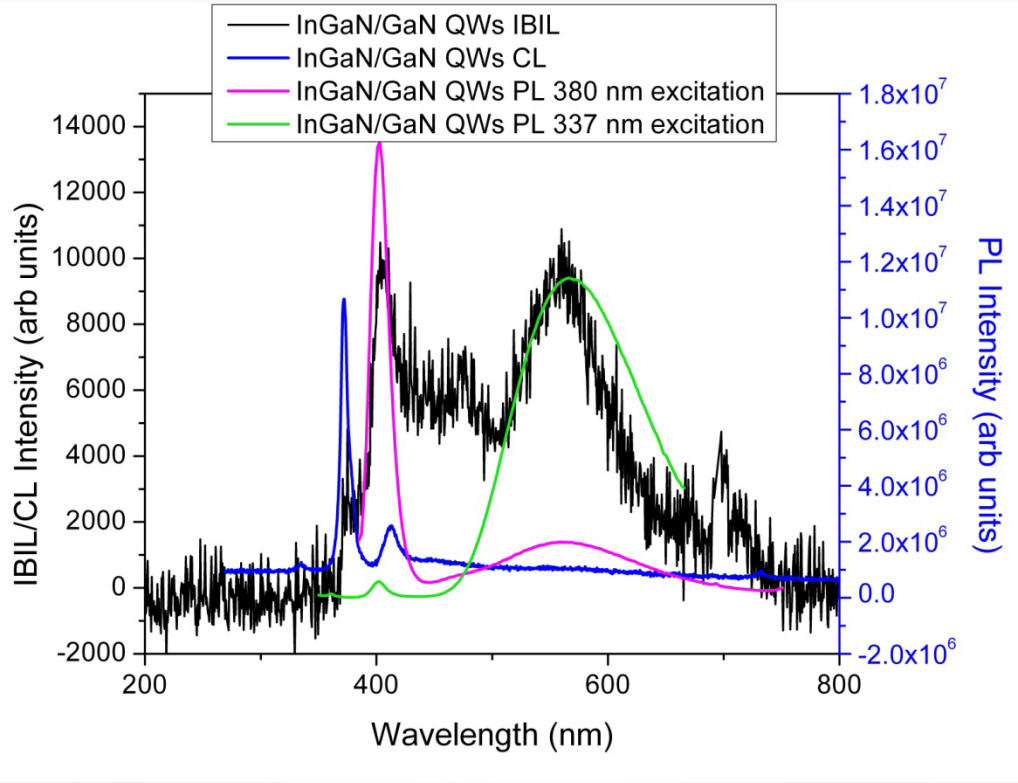
Results - Bicron

Bicron demonstrated very low detected photons/ion and exhibits significant radiation damage

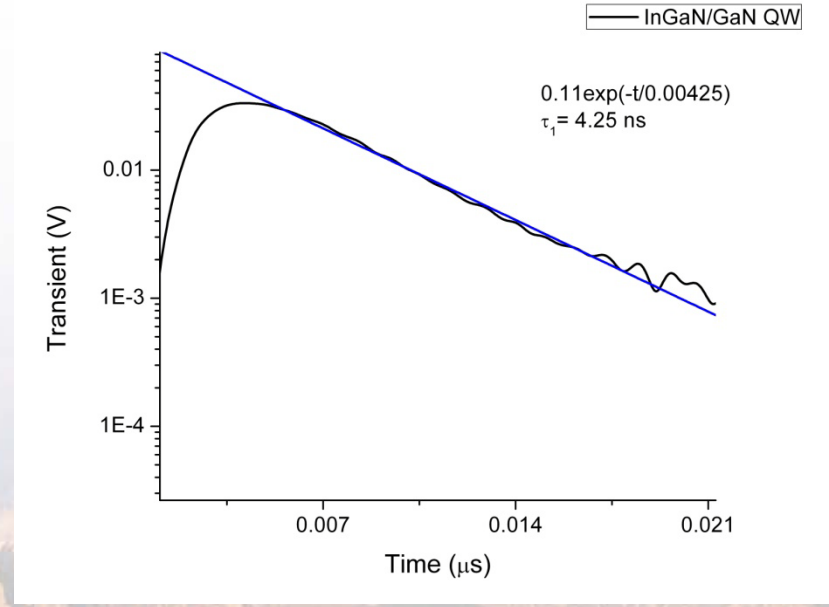


- BC400 and BC430 both demonstrate high sensitivity to radiation damage
- Despite promising emission and decay time from BC430, it will not be useful for IPEM

Results – GaN and InGaN/GaN Quantum Wells



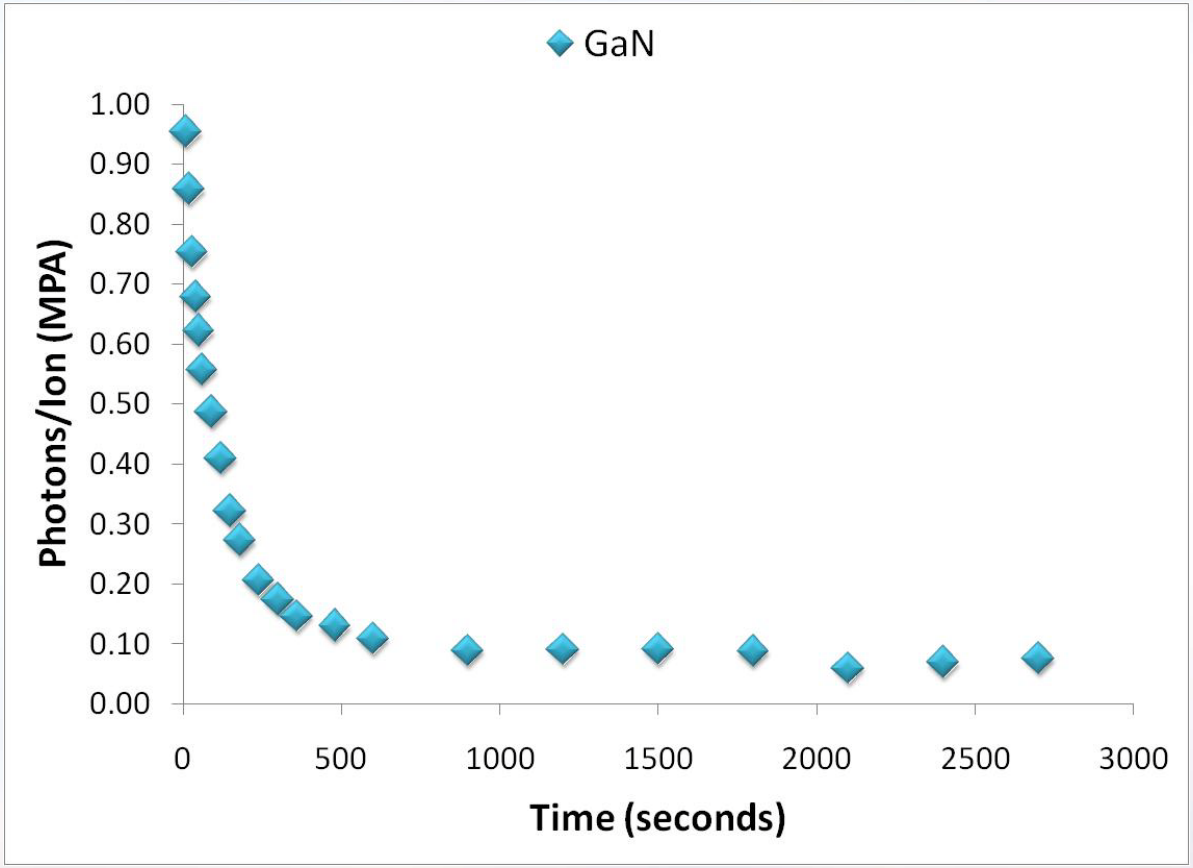
- InGaN/GaN quantum wells were designed and grown at SNL to optimize blue, fast bandedge emission
- Relative intensities of emission bands greatly dependent on excitation mechanism and energy



- Blue bandedge emission has a very fast decay, but overlaps with air
- Yellow band has good emission wavelengths, but has demonstrated very long decay times

Results – GaN and InGaN/GaN Quantum Wells

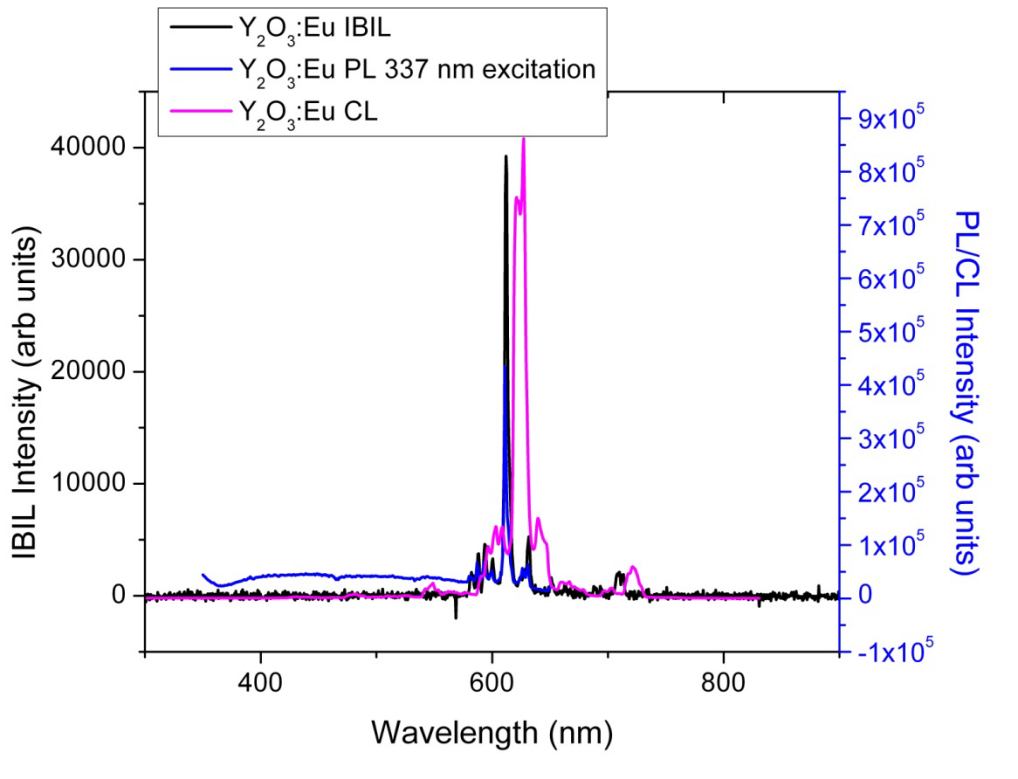
GaN shows sharp decrease in detected photons/ion but remains steady



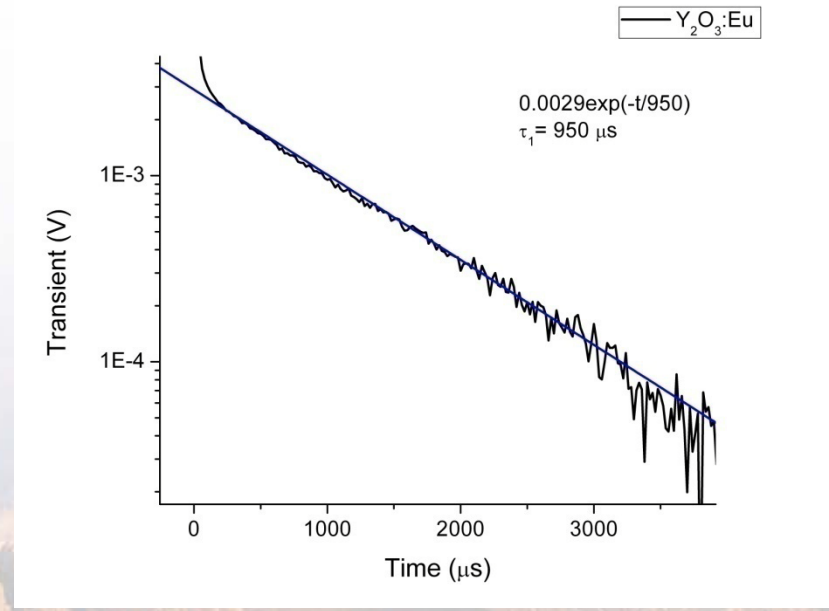
- Despite initial sharp drop-off GaN's efficiency remains higher than that of other materials studied
- Radiation hardness and yellow emission are promising, but long decay time of yellow light will cause accidental coincidences

Results – Lanthanide-Doped Ceramics

Europium-doped ceramics have easily engineered emission properties



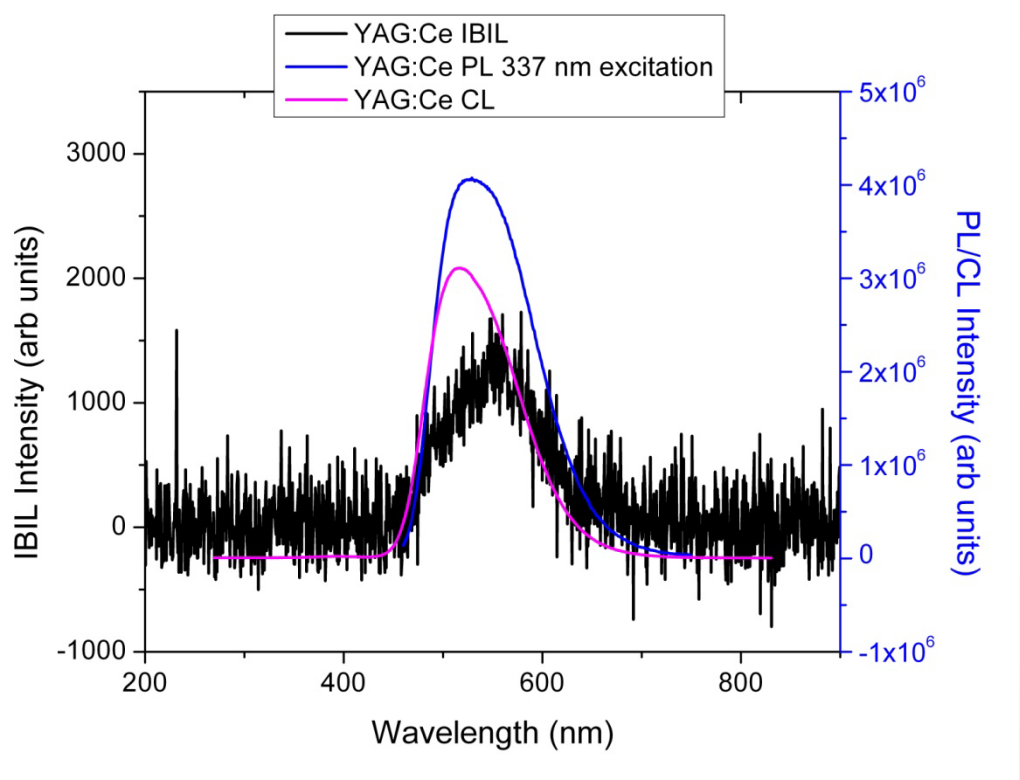
- Y_2O_3:Eu has a great emission spectrum – intense lines at wavelengths much above that of air
- IBIL and PL characteristics very similar



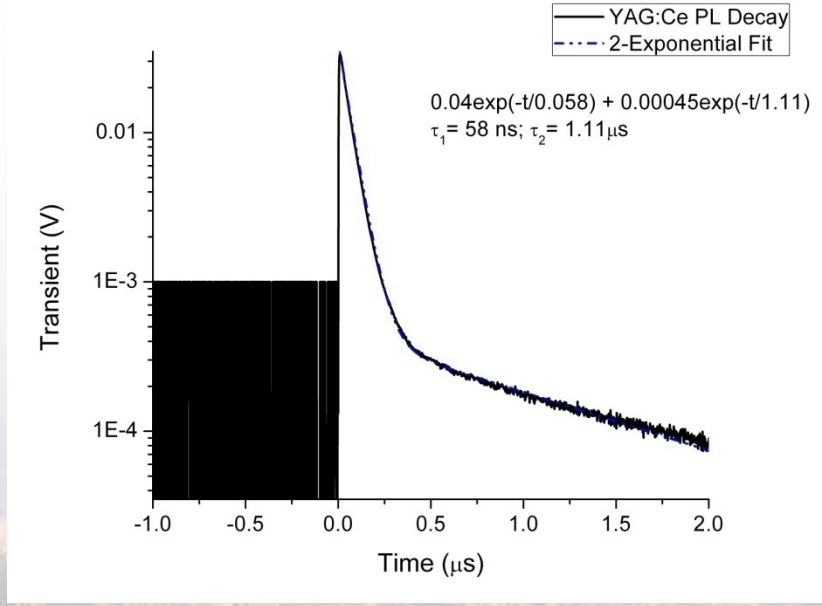
- Decay time of Y_2O_3:Eu is incredibly long
- Would lead to accidental coincidences for IPEM application

Results – Lanthanide-Doped Ceramics

YAG:Ce is most promising IPEM material studied



- YAG:Ce has broad emission band centered around 520 nm
- Not as intense as Y_2O_3:Eu



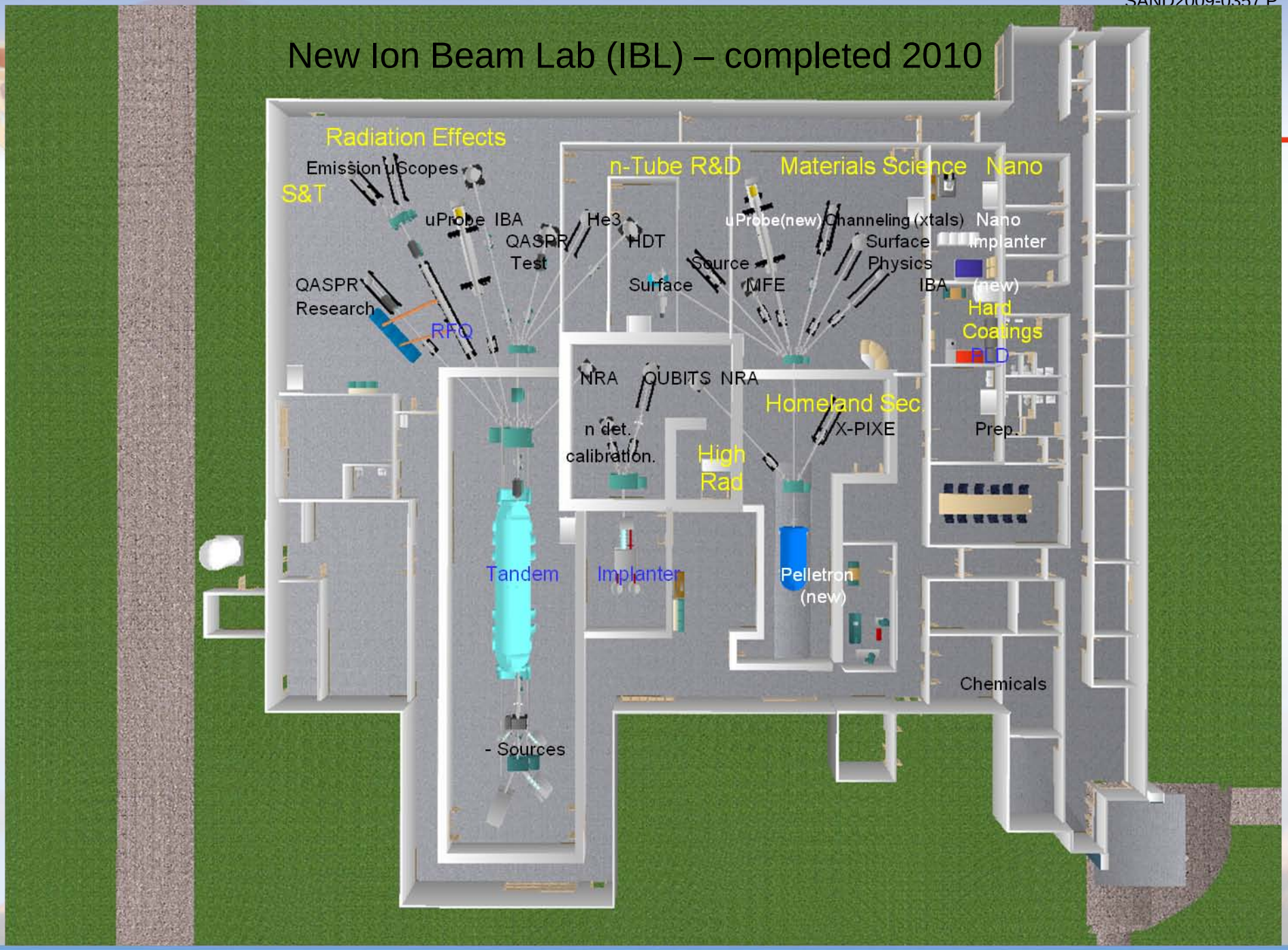
- YAG:Ce decays with a bi-exponential
- Both decays are sufficiently short to be useful

Results – Materials Overview

	Material	IBIL Emission Wavelength Range	IBIL Peak Emission Wavelength	Radiation Hardness	Reported Luminescence Decay Time	Experimental Photoluminescence Decay Time
Plastic	Bicron BC400	410-480 nm	423 nm	Low	2.4 ns	1.4 ns
	Bicron BC430	540-610 nm	580 nm	Low	16.8 ns	12.5 ns
Semiconductor	n-GaN	480-700 nm	555 nm	Medium		
	InGaN/GaN QWs	410-700 nm	430 nm, 555 nm	Medium		4.25 ns, 1300 µs
Ceramic	Y_2SiO_5:Ce	370-480 nm	400 nm	High	80 ns	47 ns
	YAG:Ce	490-620 nm	530 nm	High	60 ns	58 ns, 1.11 µs
	$KGdTa_2O_7$:Eu	560-640 nm	610 nm	High		1.6 ms
	Y_2O_3:Eu	500-650 nm	550 nm	High	1.3 ms	950 µs
	$Y_4Al_2O_9$:Eu	580-710 nm	615 nm	High		2.4 ms
	$ZnSiO_4$:Mn	480-580 nm	525 nm	High		

- Based on emission wavelength, decay time, and radiation hardness, YAG:Ce is so far the best option for application in IPEM

New Ion Beam Lab (IBL) – completed 2010

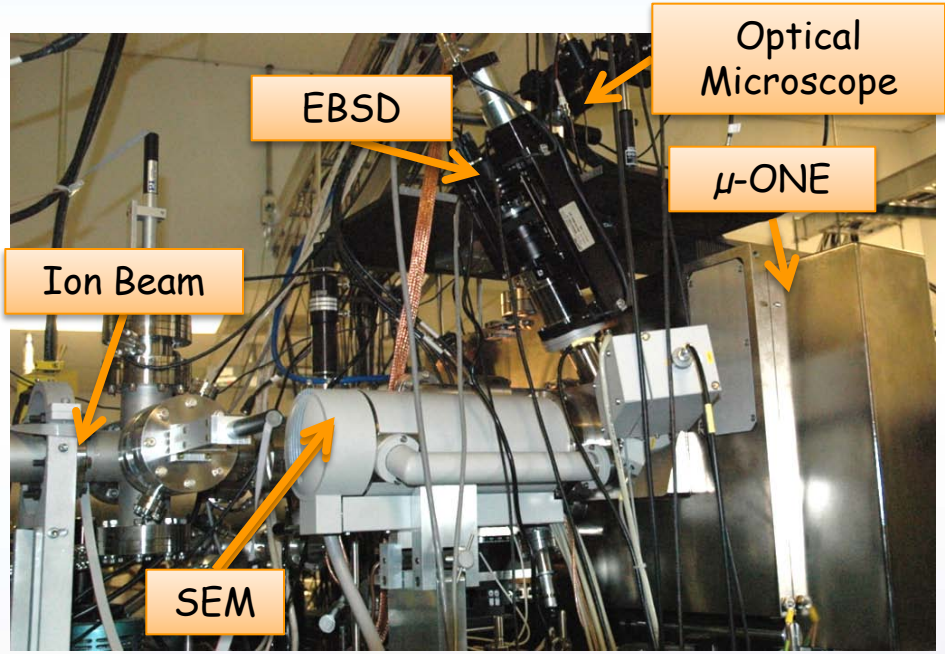


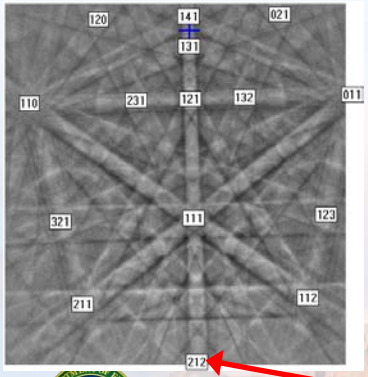
Micro-ONE Capabilities

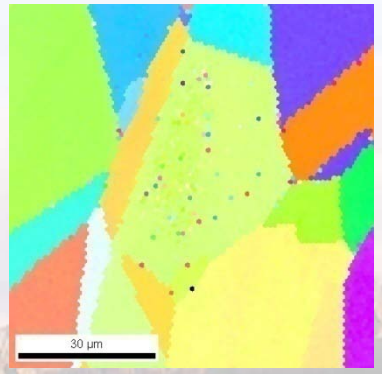
Micro-ONE = Micrometer resolution Optical, Nuclear, and Electron Microscope

Micro-ONE capabilites

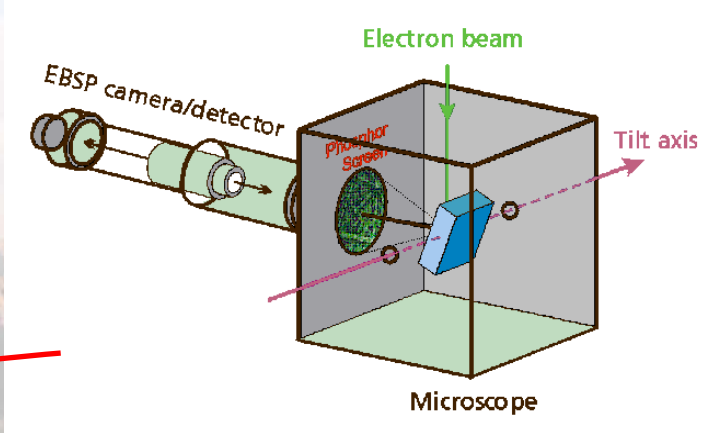
- Beam size: < 1μm (~ 0.5 μm)
- Current: single ions to 10,000 ions/s
- Ions: H, He, heavy ions
- Energy: (q+1)*6 MeV for heavy ions
- Scan size: ~ 100x100 μm^2
- Stage position with 50 nm resolution
- Fast blanking capabilities
- Navigation based on GDS II files
- IBIC and TRIBIC capabilities
- EBSD mapping







First EBSD Pattern and Map obtained with this system



Will allow *in situ* imaging of changes in microstructure: grain size, phase transformations.

In situ Ion Irradiation TEM

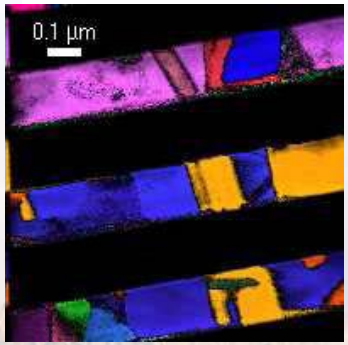
Proposed Capabilities

- 200 kV LaB_6 TEM
- Ion beams considered:
  - 32 MeV Fe^{4+}
  - 250 keV D^{2+}
  - 250 keV He^{+}
  - 300 keV e^{-}
- All beams will hit same location
- 30 to 60 degree considered
- nanosecond time resolution (DTEM)
- procession scanning (EBSD in TEM)
- *In situ* PL, CL, and IL
- *In situ* heating and cooling stage
- *In situ* electrical measurement stage
- *In situ* straining stage
- *In situ* vapor phase stage
- *In situ* liquid mixing stage

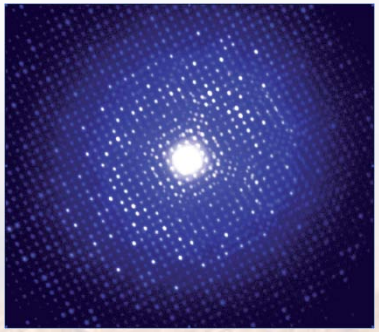
Delivery December 2010

JEOL 2010 at
Shimane Univ.
Japan

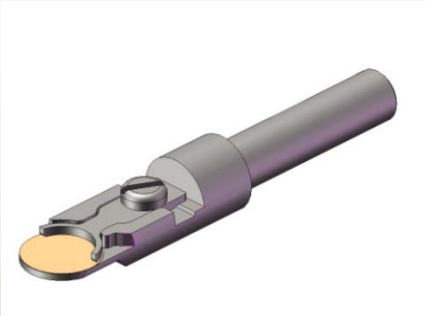
1 in the US (decommission planned)
11 world-wide



Nanomegas



TVIPS

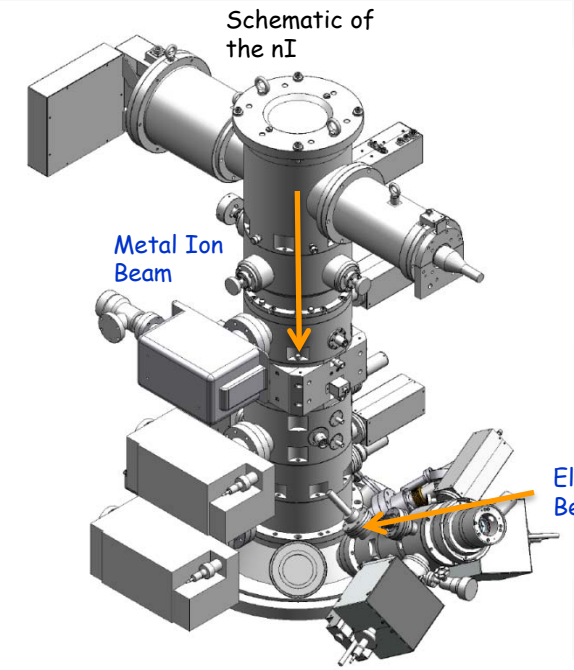


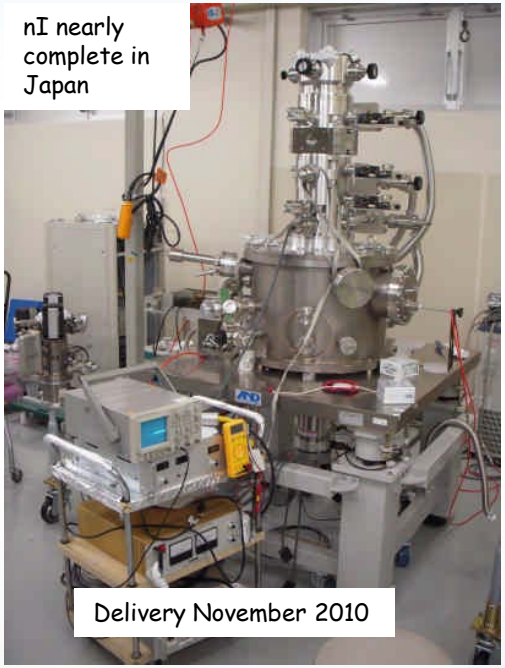
Hummingbird

We are at the beginning stages of planning. Many potential additions for an *in situ* triple beam facility are being considered

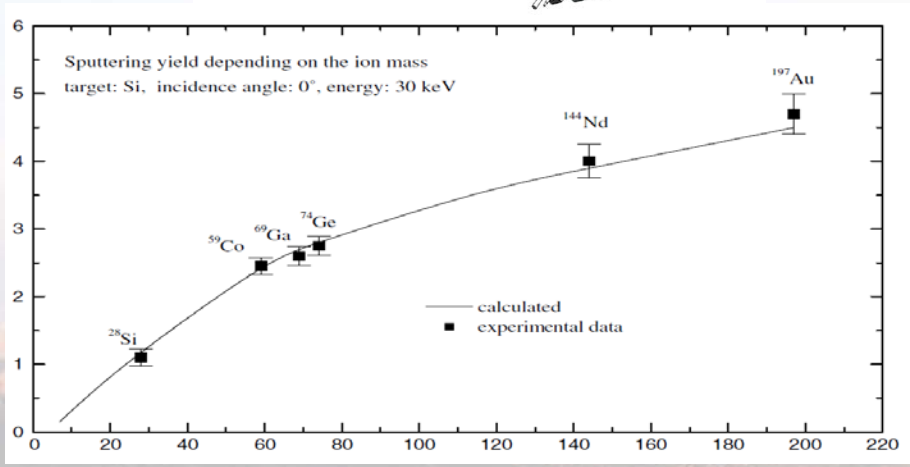
Nanoimplantor

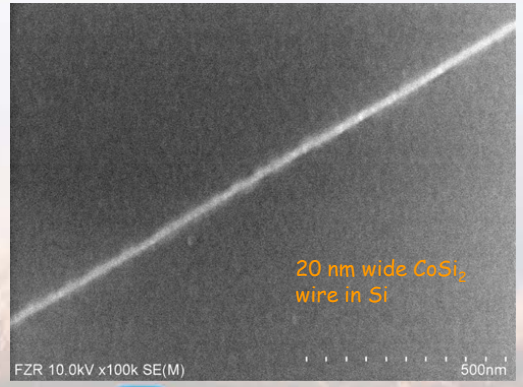
- Multiple liquid metal ion source (LMIS)
- *E x B* filter
- Ion column with a maximum accelerating voltage of 100 kV
- 10 nm spot size
- High-resolution SEM
- 4 manipulators with 10 nm resolution and low current measurement capabilities





These capabilities greatly exceed the capabilities of commercially available Ga^+-based dual-beam FIB systems in terms of irradiation doses, ion species, and nanofabrication.





Sandia National Laboratories

Conclusions & Future Work

- Future of heavy ion radiation effects microscopy depends on developing scanned microbeams and/or emission microscopes that can be easily used on cyclotrons
- IPEM is reasonable solution
- Various materials considered and studied
- Crucial to study optical properties with various excitation methods
- YAG:Ce most promising
- IPEM will allow us to continue using REM as feature sizes shrink, and thicknesses of IC overlayers increase

- Verify IBIL decay times
- Create nanoparticles of YAG:Ce and deposit
- Use new material and cyclotron IPEM design to perform SEE imaging
- Build system for simultaneous IBIL, PL, and CL

Acknowledgements

- Mike Johnson and Larry Phair from LBNL for their patience and willingness to work through all the obstacles
- Dan Buller from SNL for all his time and expertise
- Jim Martin and Lauren Rohwer for the use of their equipment and many enlightening discussions
- Mike Smith for CL measurements
- Bernie Hernandez-Sanchez for her willingness to synthesize various materials

- Sandia is a multi-program laboratory operated by Sandia Corporation, a Lockheed Martin Company, for the United States Department of Energy under Contract DE-AC04-94AL85000.
- Work supported in part by the Italian "Istituto Nazionale di Fisica Nucleare" (INFN).
- Work at LBNL was supported by the Director, Office of Basic Energy Sciences, of the US Department of Energy under Contract No. DE-AC03-76SF00098.

Obstacles/Work in Progress

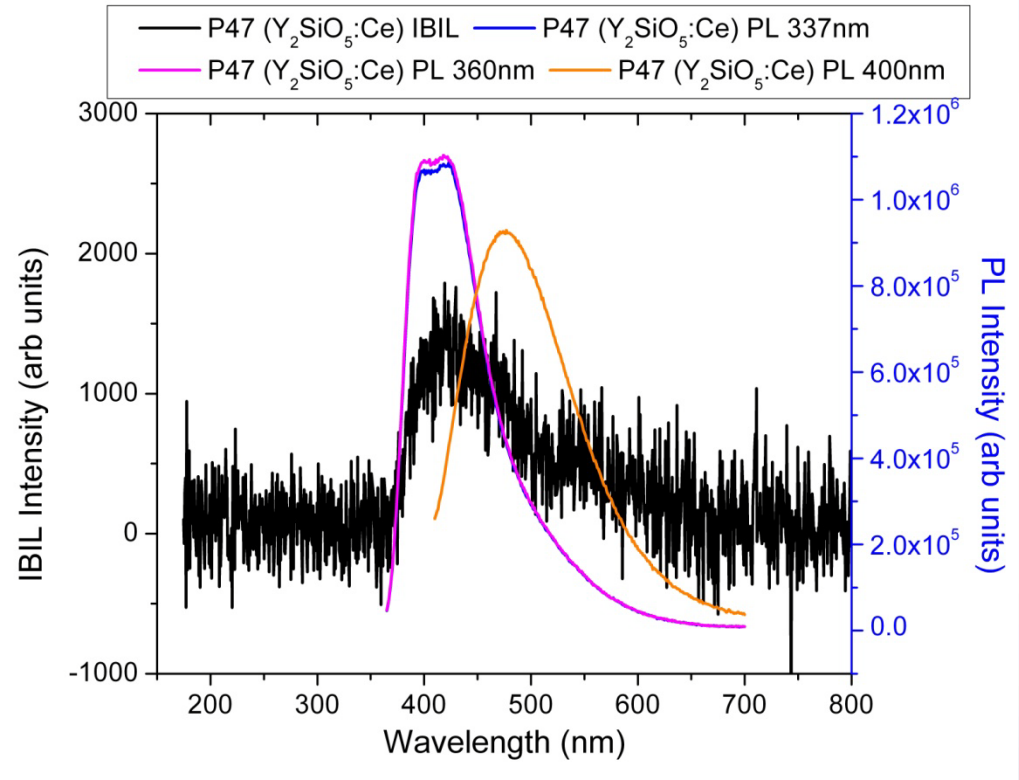
Many obstacles/difficulties have arisen with development and installation of IPEM:

- Timing: luminescent lifetime and incident ion rate → accidental coincidences
- Radiation damage and luminescent properties of materials
- Alignment of separate portion of beamline – 6-inch beam entering a 1 mm nozzle
- Need to redesign optics/microscope system

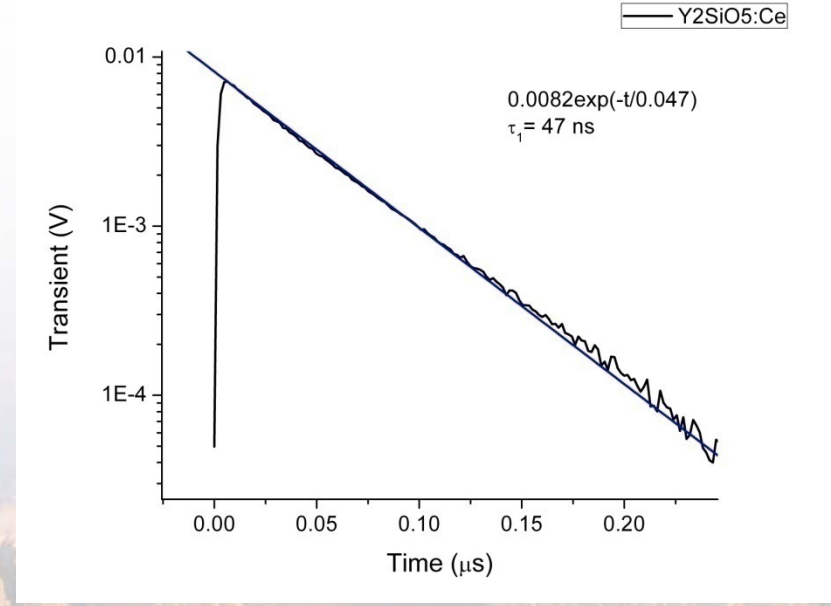
Future Work

- Need to optimize various components
  - Find the best phosphor material/composition/thickness/structure
  - Re-design the optics for ease-of-use and reliability
  - Potentially install IPEM in separate cave at LBNL to avoid continual alignment issues
- Need to understand effect of heavier/more energetic ions on materials and how it will affect system performance
- SEU testing of SNL devices with IPEM to prove its potential in radiation-effects testing

Results – P47



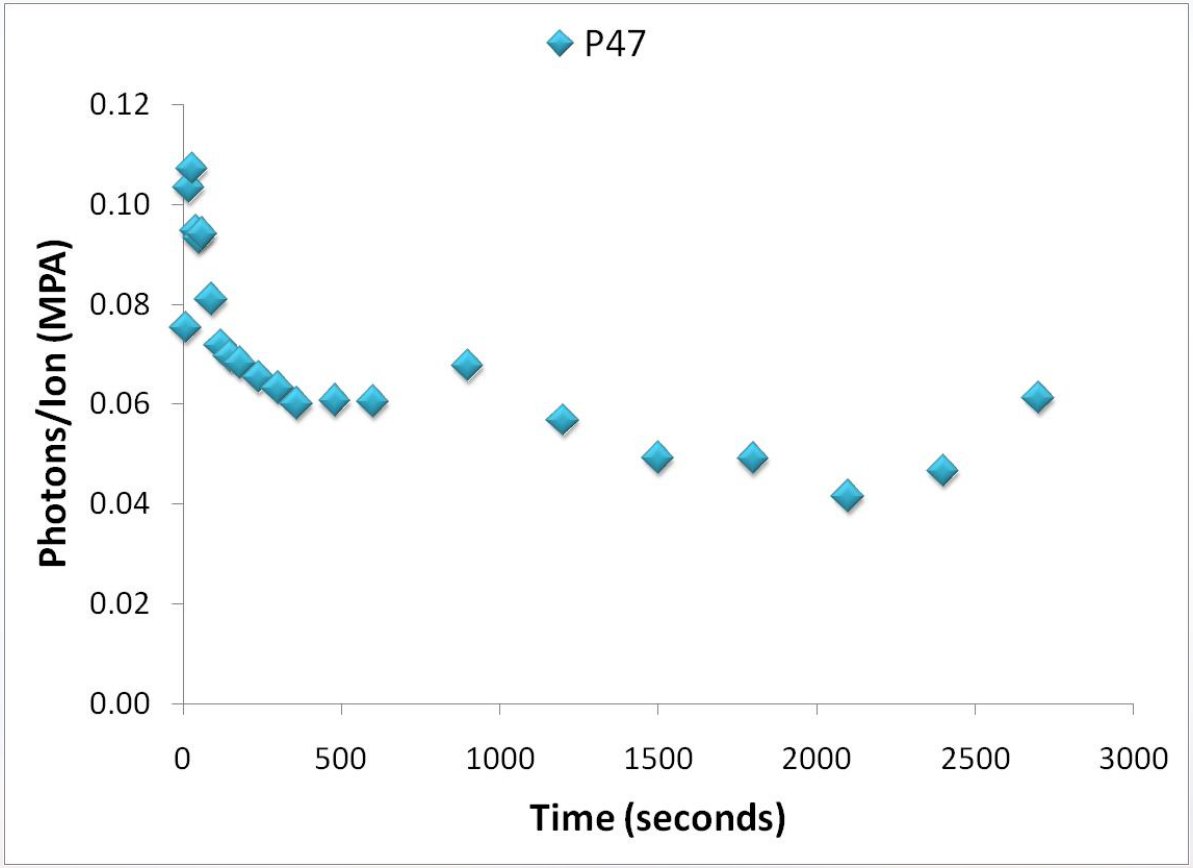
- P47 is a lanthanide-doped ceramic phosphor
- Broad, intense emission centered around 420 nm
- Emission will overlap with air luminescence



- Decay time is very fast
- Reported : 50-60 ns

Results – P47

P47 demonstrates acceptable radiation hardness



- P47 has good radiation hardness; small initial drop in efficiency that levels off
- Despite short decay time and acceptable rad-hardness, the emission of P47 is too close to that of air, and cannot be used for *ex-vacuo* IPEM