

Ion Beam Induced Luminescence and *In situ* Ion Irradiation TEM (I³TEM)

S.M. Hoppe^a, K. Hattar^a, B. A. Hernandez-Sanchez^a, T. J. Boyle^a, J. Villone^a, P. Yang^a, P. Feng^b, F. P. Doty^b

^aSandia National Laboratories, Albuquerque, NM 87185

^bSandia National Laboratories, Livermore, CA 84550

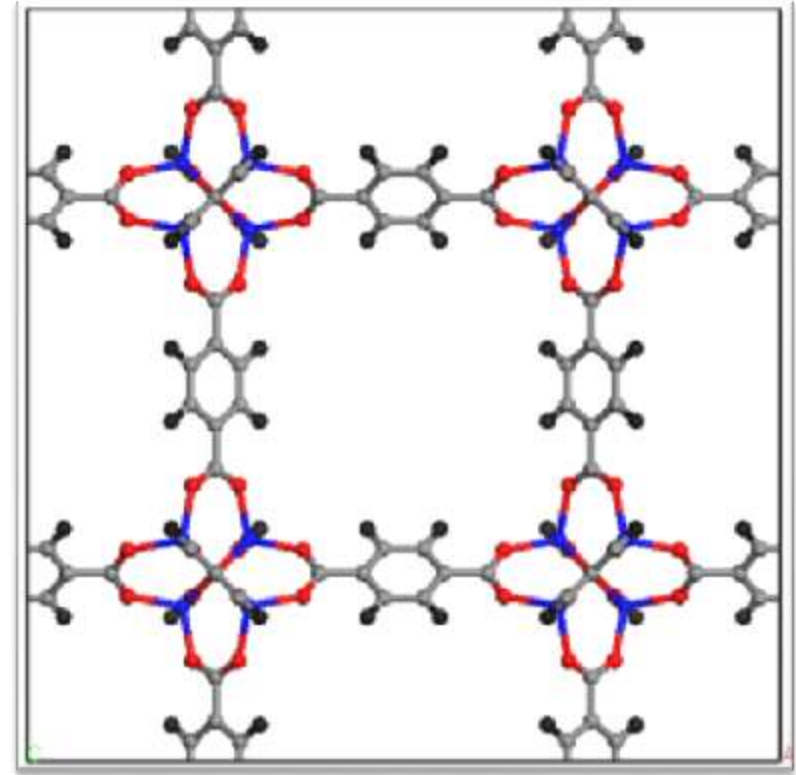
Introduction

Scintillators used for radiation detection require materials that have high density, radiation hardness, high stopping power, short decay time, and high light output. New materials being investigated offer more control and tunability to yield the desired properties.

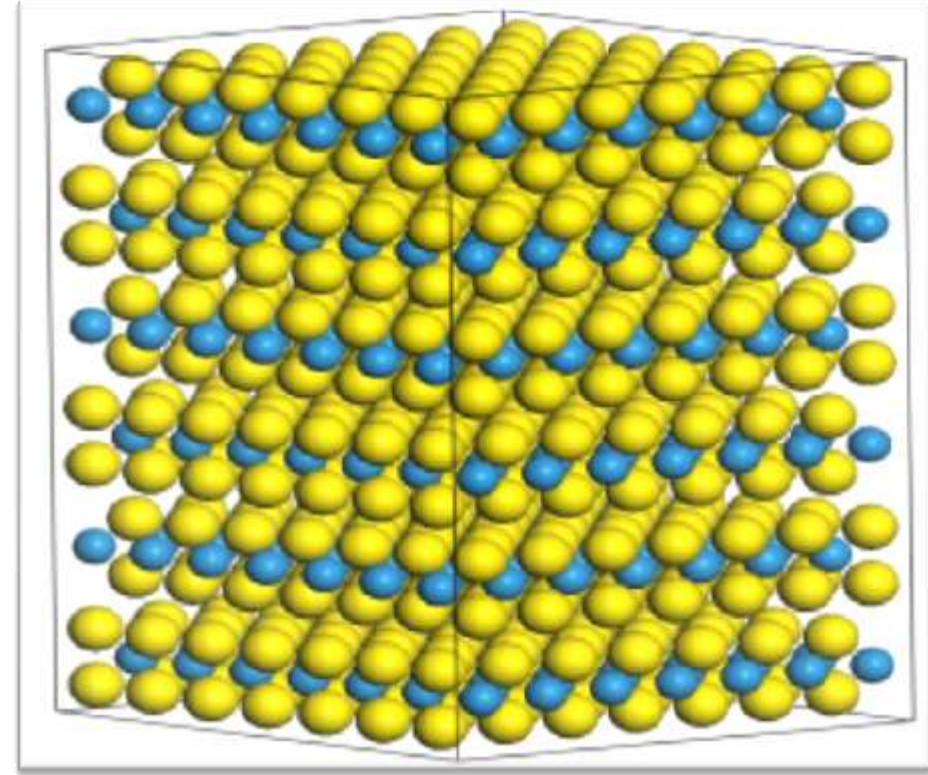
Metal-organic framework (MOF)

High-Z Nano-tungstates (MWO₄)

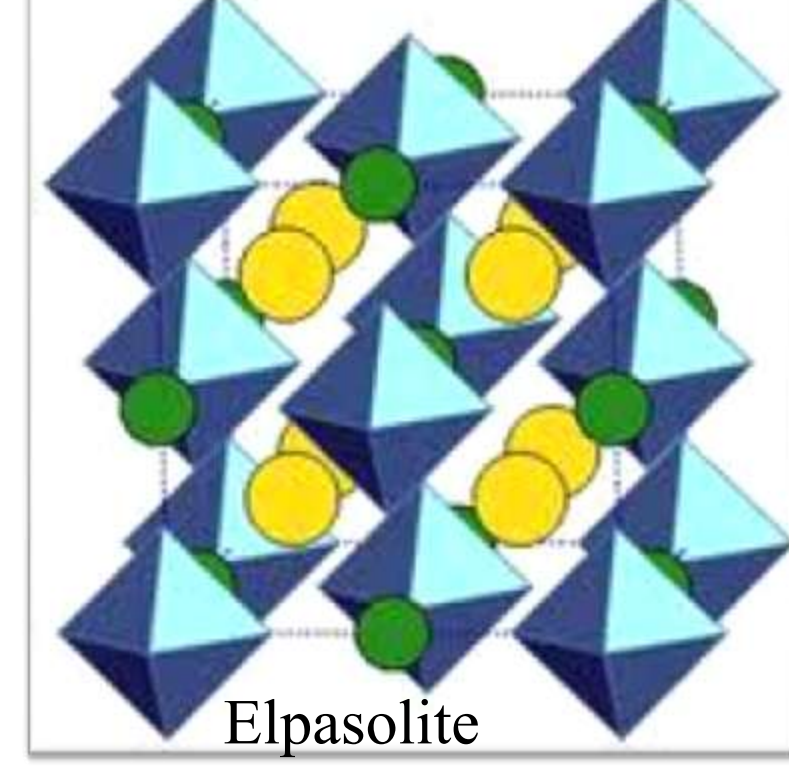
Halides



Synthetic versatility of metal clusters linked by fluorescent organic compounds offers control over electronic and crystal structures

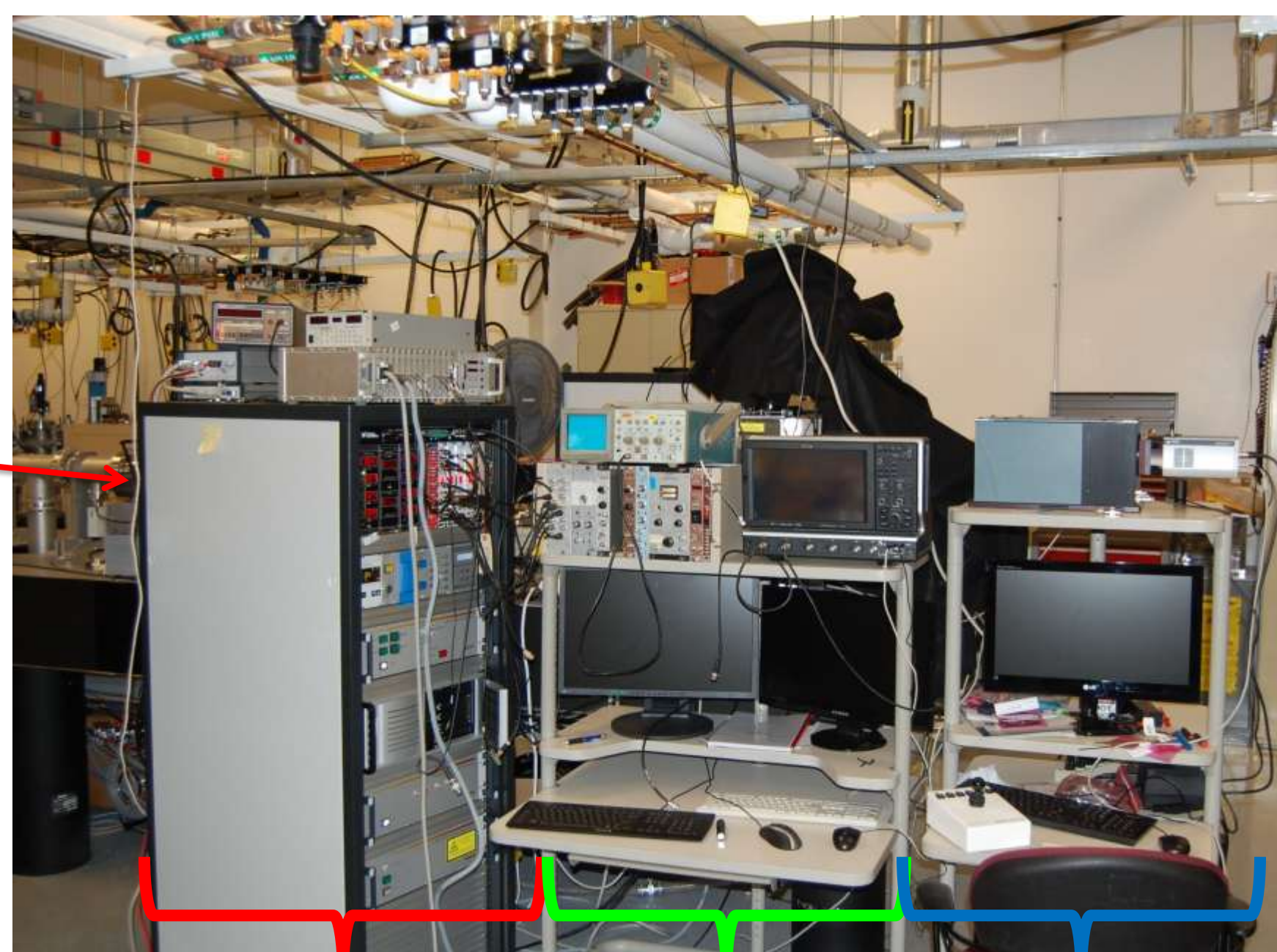


High-Z good for gamma ray detection; may benefit from increased strength and novel morphologies exhibited by nanoscale materials



Investigating Elpasolite and Sorohalides, a novel class of halide-containing compounds, while varying compositions and crystal structures

Ion Beam Induced Luminescence (IBIL)



The Ion Beam Lab's microbeam end station on the 3 MeV Pelletron accelerator permits precise location of the irradiated regions, as well as IBIL spectra and lifetime measurements

Optical path to spectrometer

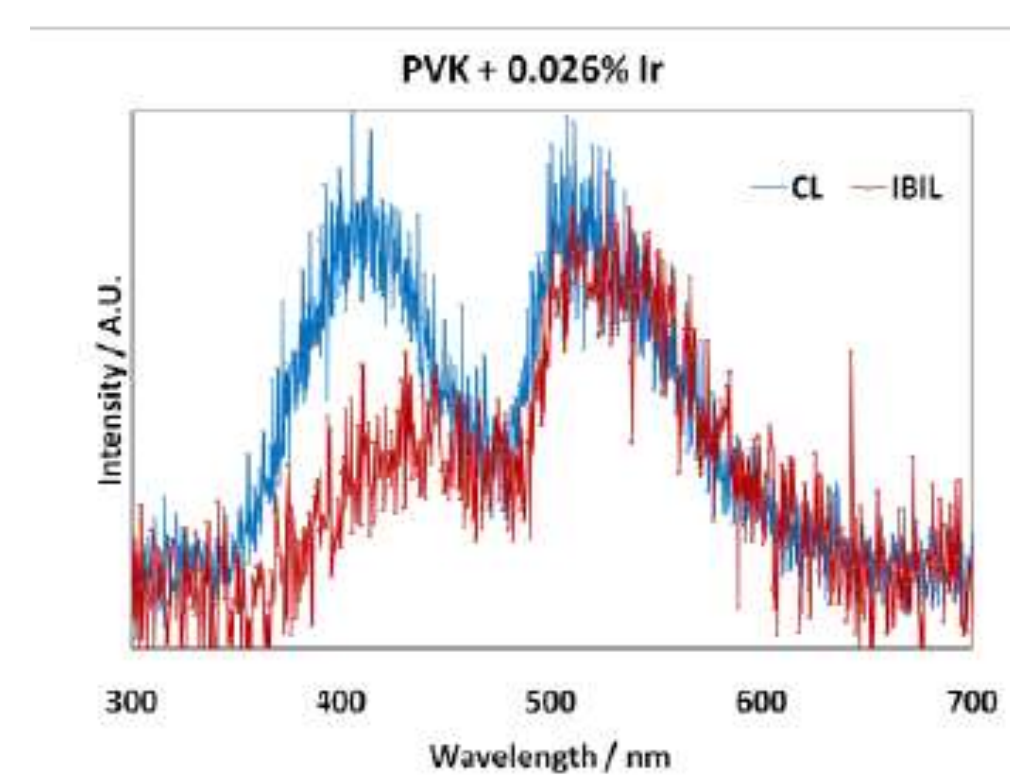
Ion beam

Photodiodes & PMT for lifetime measurements

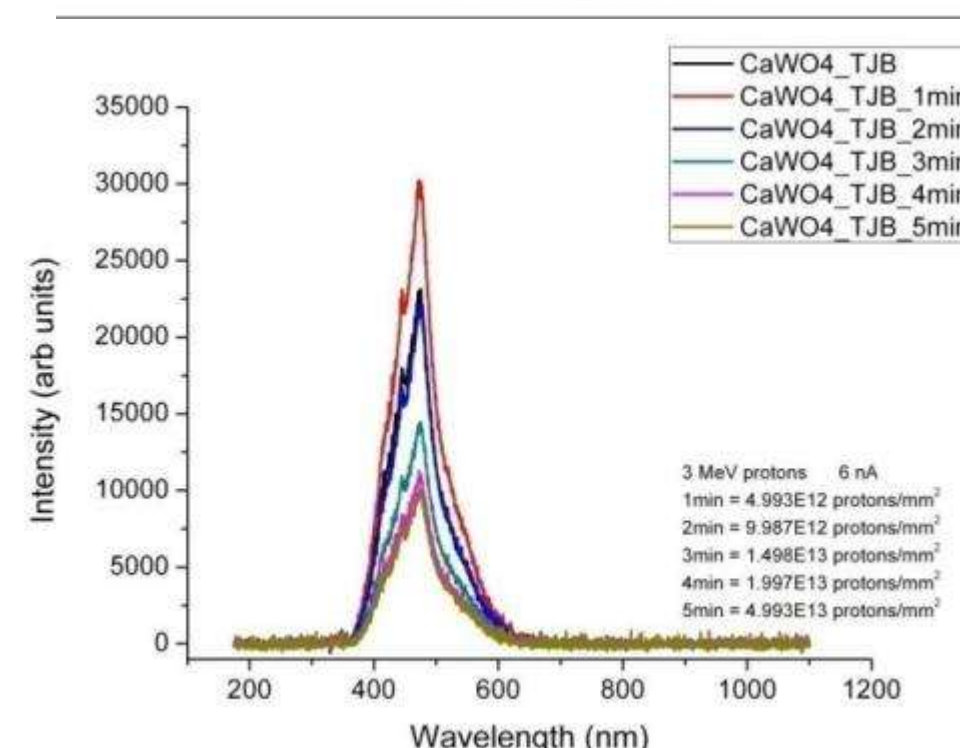
- A modified JEOL OM40 provides direct observation of the region irradiated for spectra collection
- An Andors spectrometer was optimized by PTI for greatest light collection
- Thin samples (films or particles) are deposited on PIN diodes.
- Diode and PMT signals are used to correlate the ion strike to the photon emitted to determine photon lifetime

IBIL results

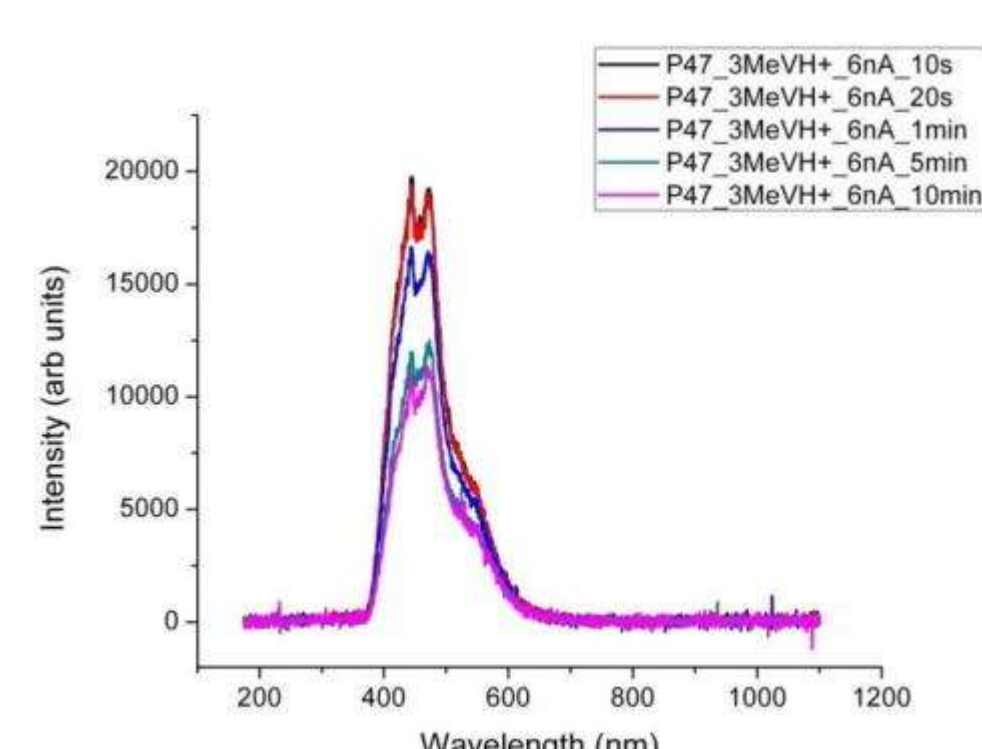
Metal-organic framework (MOF)



High-Z Nano-tungstates (MWO₄)

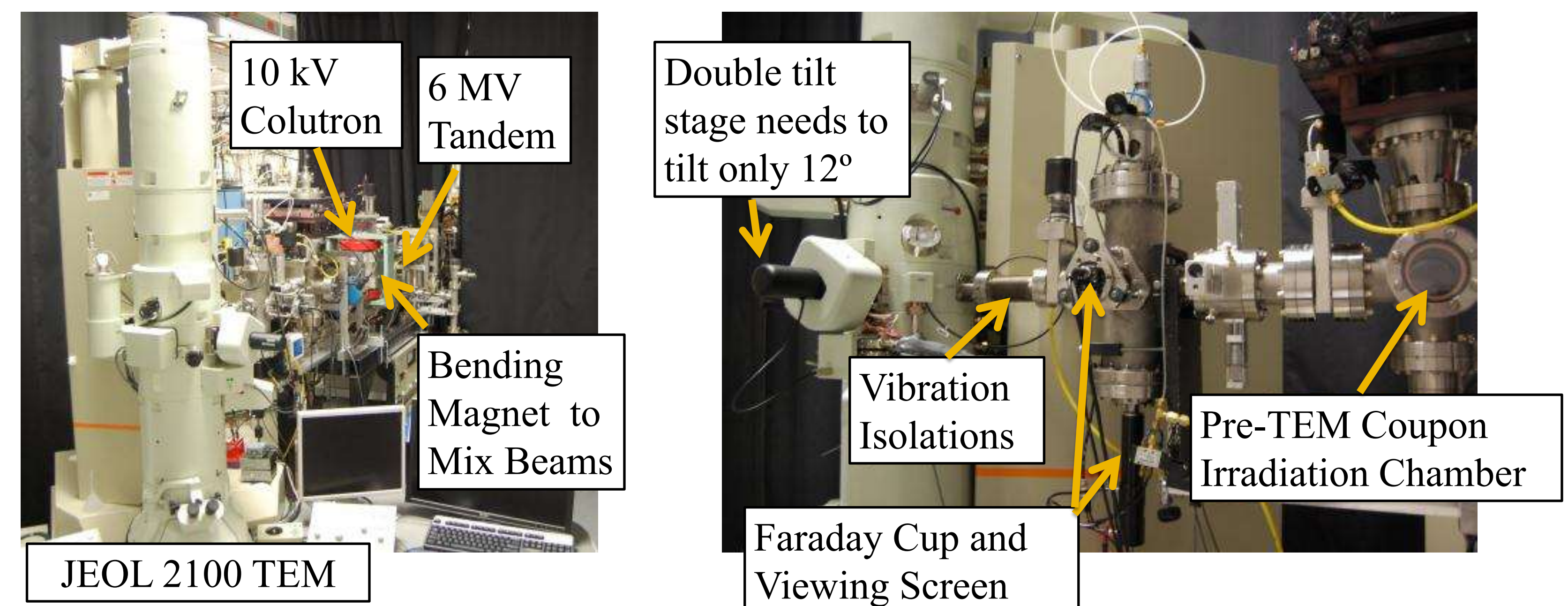


Sorohalides



IBIL provides insight into properties, but lacks the associated microstructure understanding needed to understand underlying physics

In situ Ion Irradiation TEM (I³TEM)

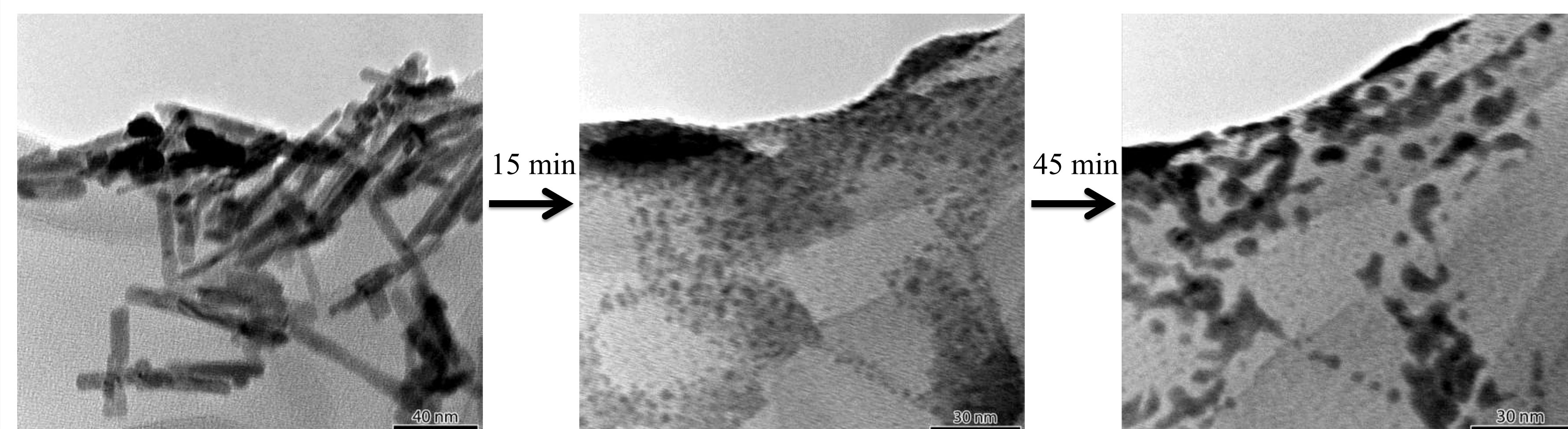


I³TEM Current Status

1st Generation: *in situ* heavy ion irradiation completed and operated regularly with 2.5 MeV H; 3 MeV He, Si, Ni, Cu, and Au; and 14 MeV Si
2nd Generation: concurrent high energy ion beam from the Tandem accelerator and low energy ion beam from the Colutron was assembled and placed under vacuum, but is not operational.

Nano-tungstates Undergo Structural Changes

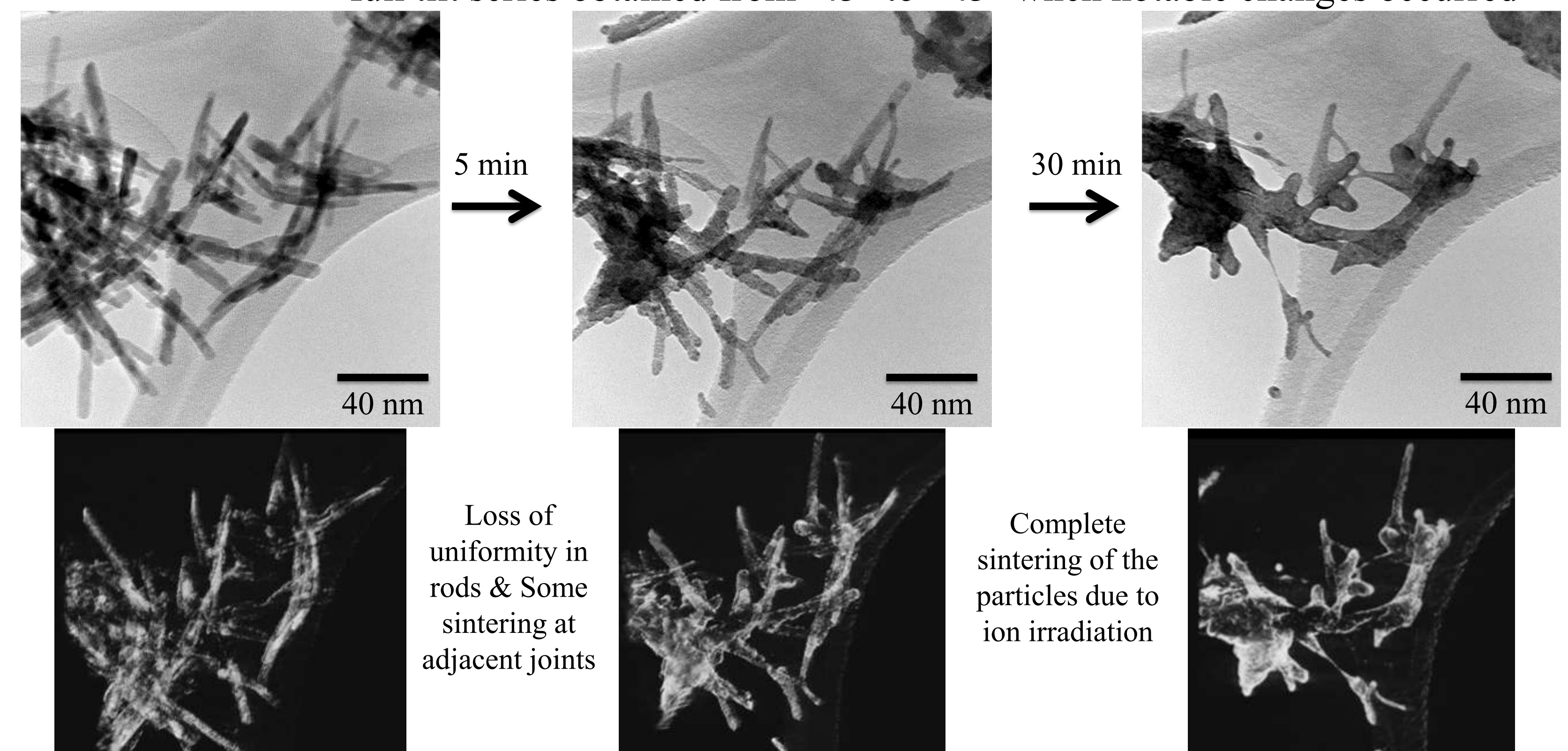
CdWO₄ nanorods irradiated with 40 nA of 3 MeV Cu⁺
- imaged every 15 minutes for 1 hour



- After initial irradiation, rods appear to break into spherical pieces
- With continued irradiation, the small pieces appear to agglomerate into larger masses

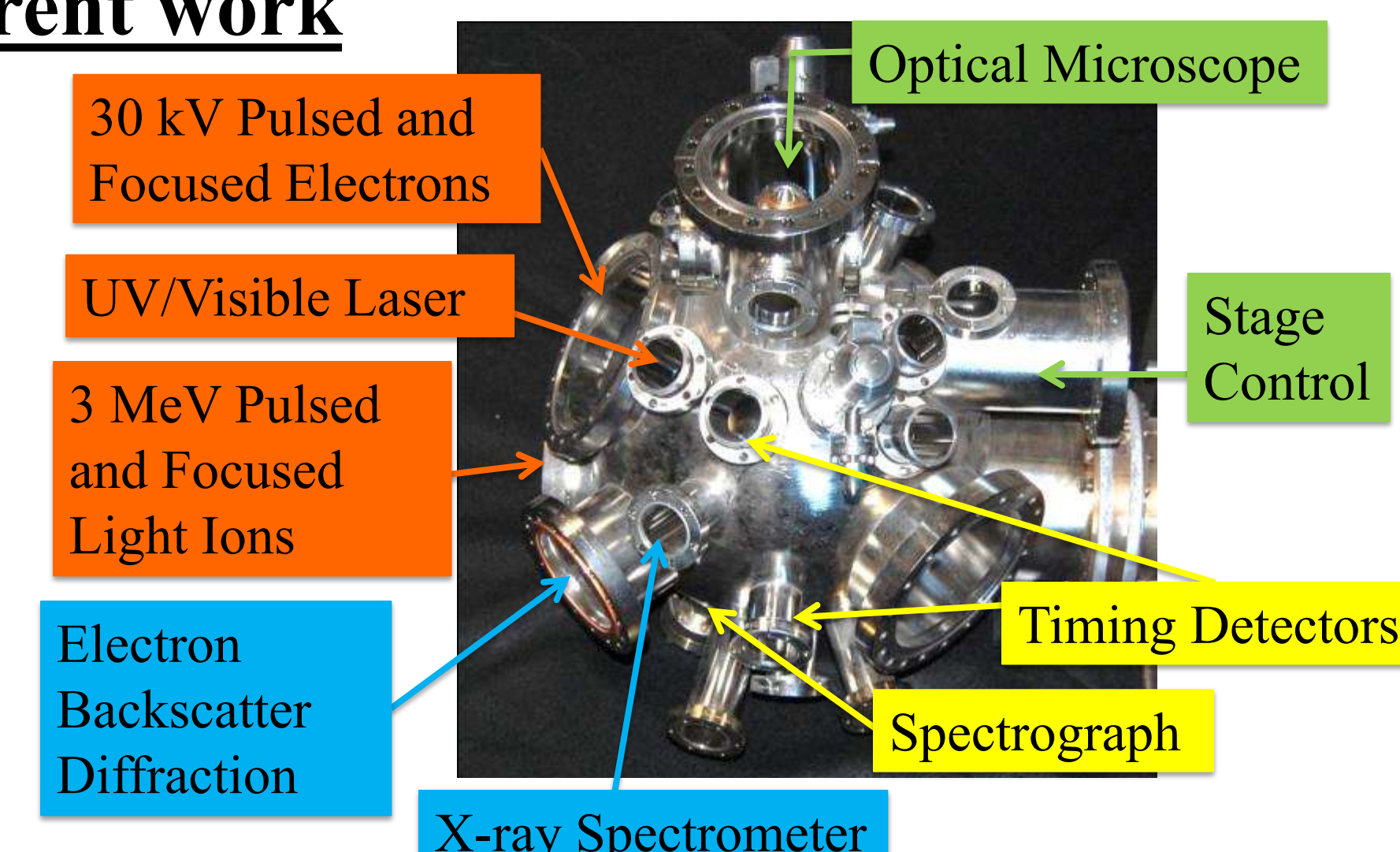
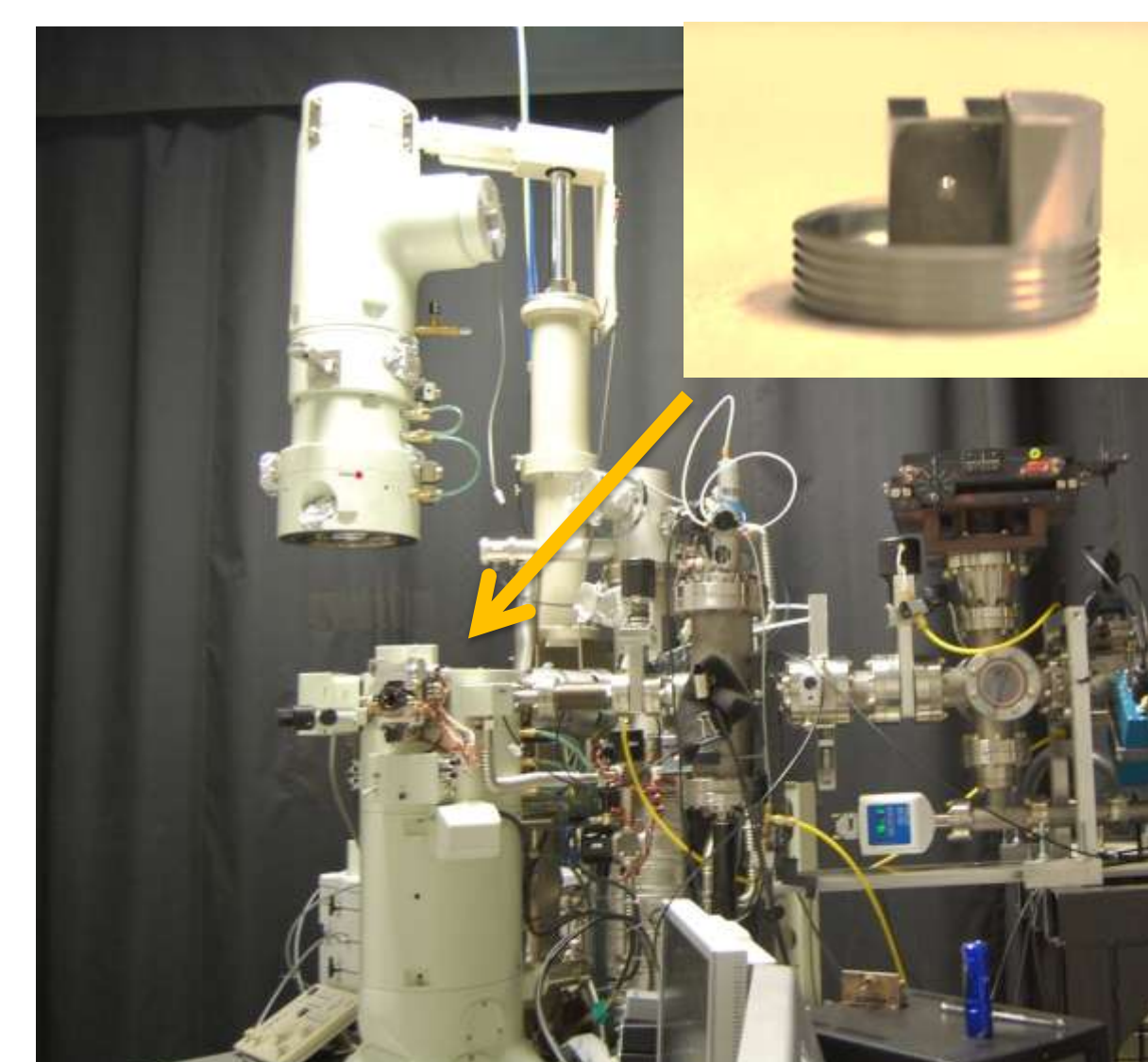
I³TEM Combined with 4D Tomography for Better Understanding

CdWO₄ nanorods irradiated with 20 nA of 3 MeV Cu⁺
- imaged every 5 minutes for 30 minutes
- full tilt series obtained from -43° to +43° when notable changes occurred



- Processing of tomography tilt series yields 3D rotating models
- Constructing models over time leads to better understanding and visualization of damage

Current work



An optical port is currently being added to the I³TEM, which, if successful, will permit *in situ* TEM CL and IBIL

A large chamber is proposed that will permit both lifetime and spectra PL, CL, and IBIL measurements of advanced scintillators.