Exploring the Radiation Tolerance of Ceramic Nanoparticles via In-situ Ion Irradiation TEM

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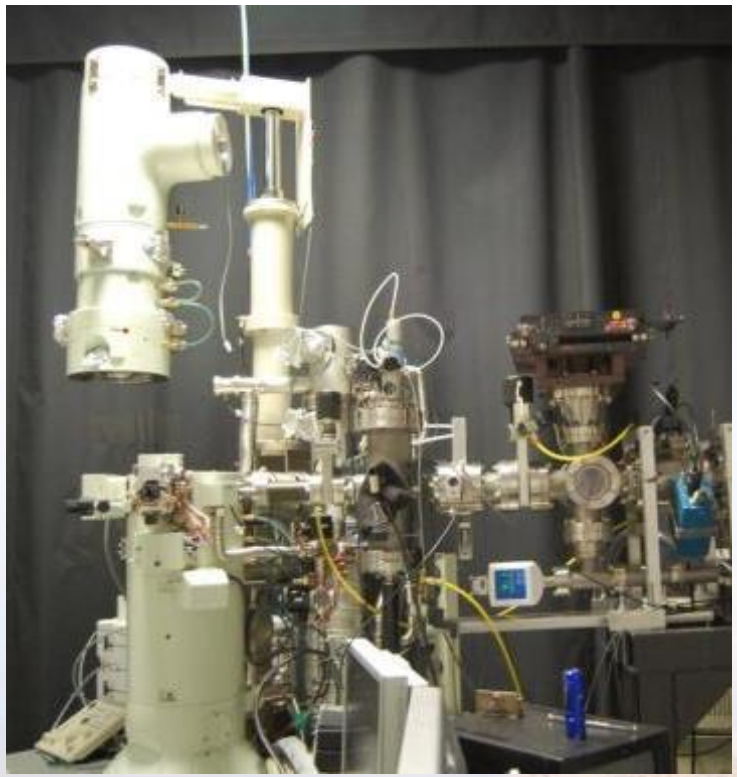
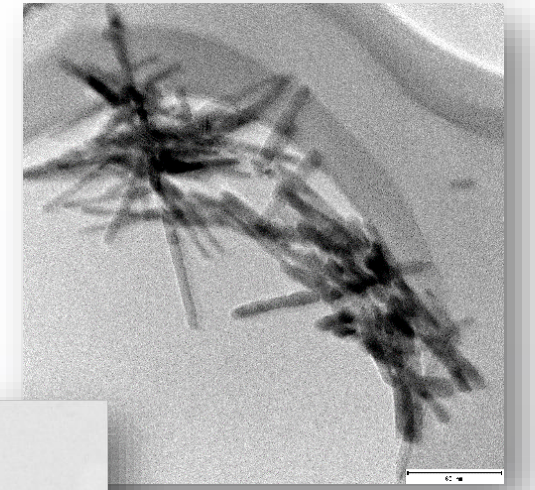
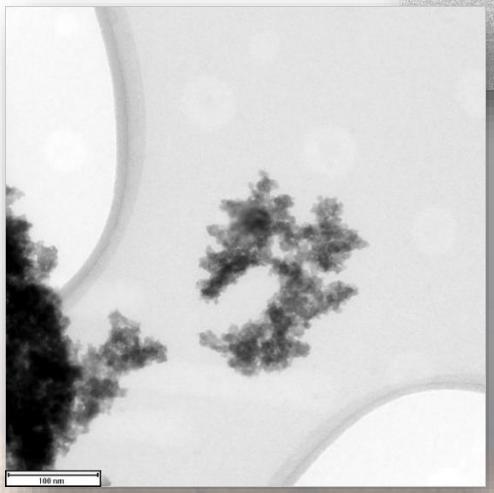
Sandia National Laboratories

May 23rd, 2017



***In situ* TEM microscopy has recently undergone significant growth providing capabilities to investigate the structural evolution that occurs due to various extreme environments and combinations thereof**

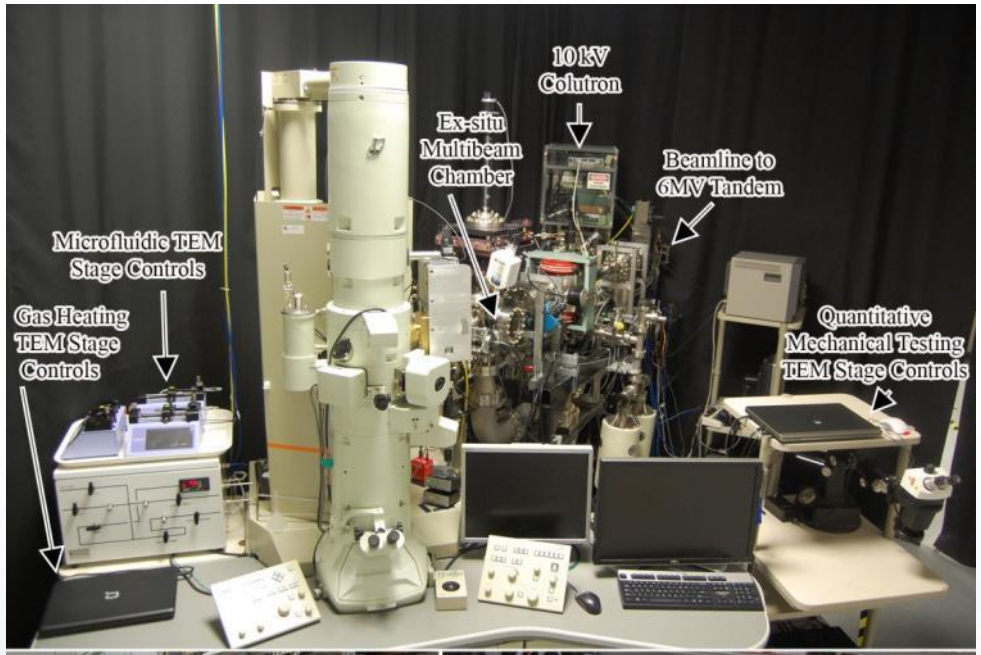




This work was supported by the US Department of Energy, Office of Basic Energy Sciences.

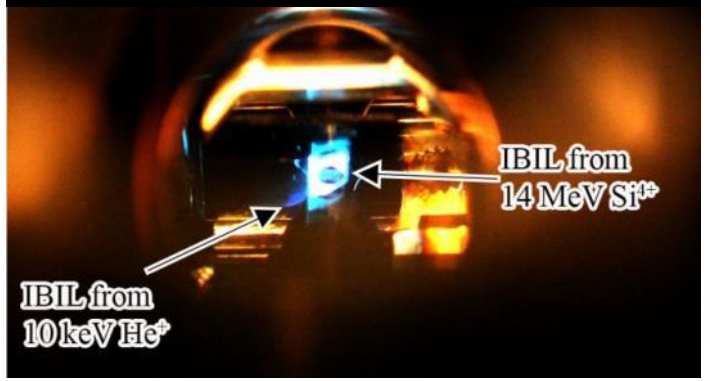
Sandia's Concurrent *In situ* Ion Irradiation TEM Facility

10 kV Colutron - 200 kV TEM - 6 MV Tandem

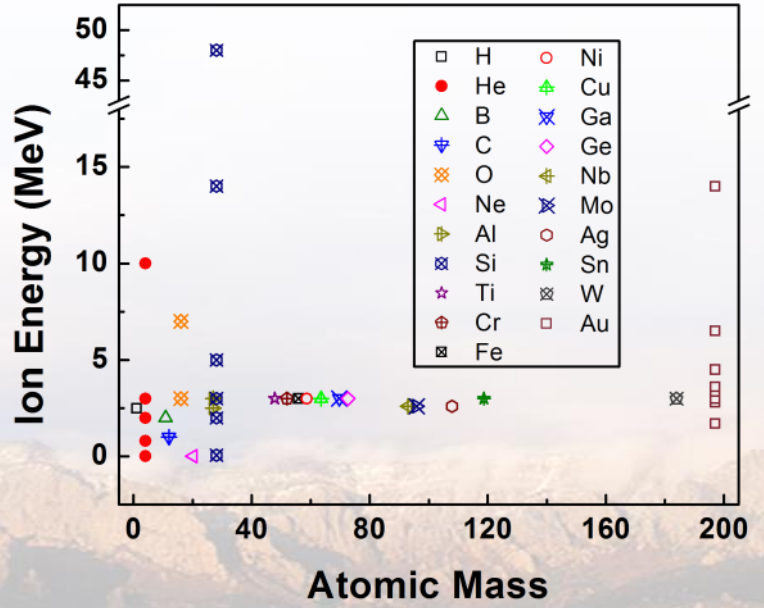


Direct real time observation of ion irradiation, ion implantation, or both with nanometer resolution

IBIL from a quartz stage inside the TEM



Ion species & energy introduced into the TEM



Hummingbird tomography stage

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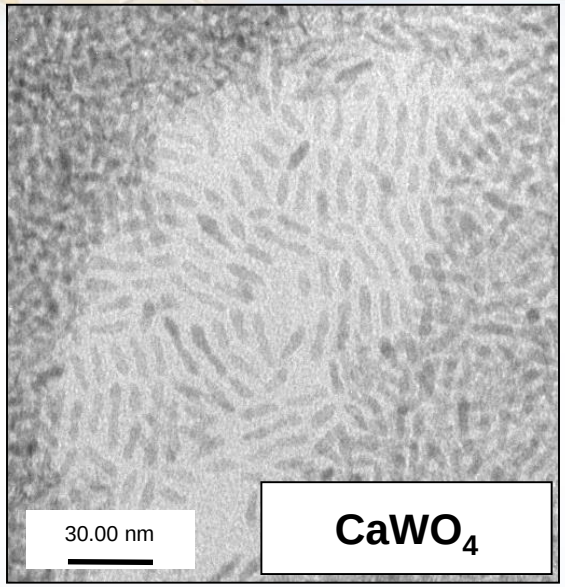
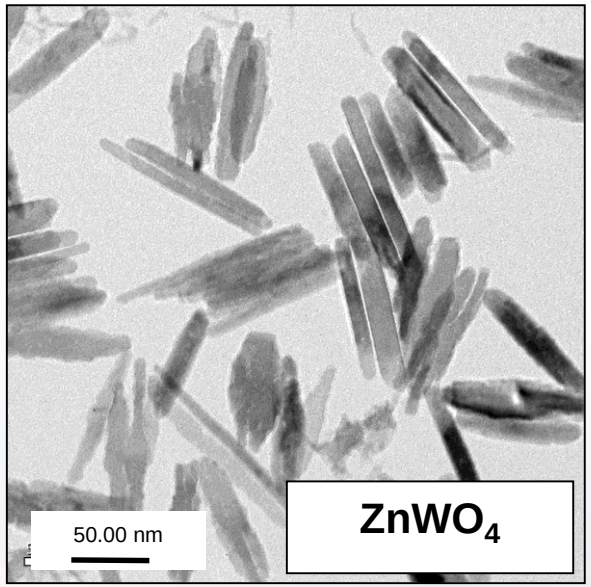
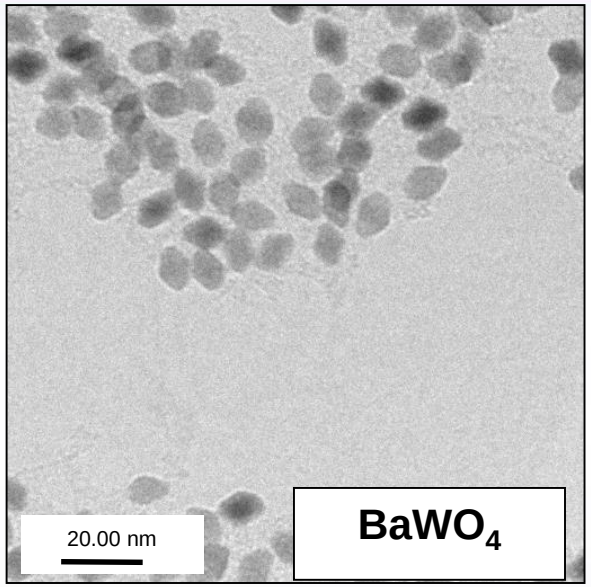
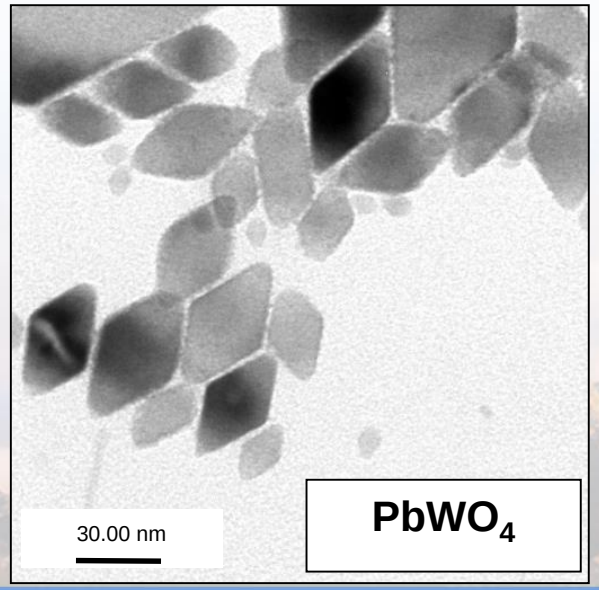
Synthesis Controlled Nanostructured Tungstates

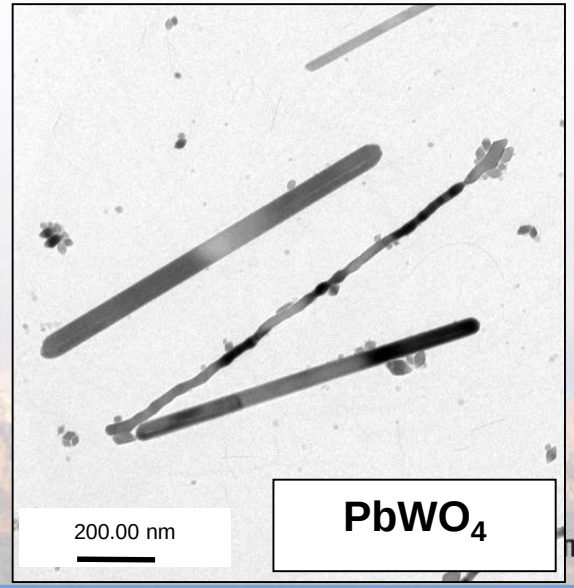
- Entire family of MWO_4 materials synthesized:
  M = Ca, Cd, Pb, Sr, Ba, Zn, Na
- Varying composition and synthesis methods result in a range of interesting morphologies



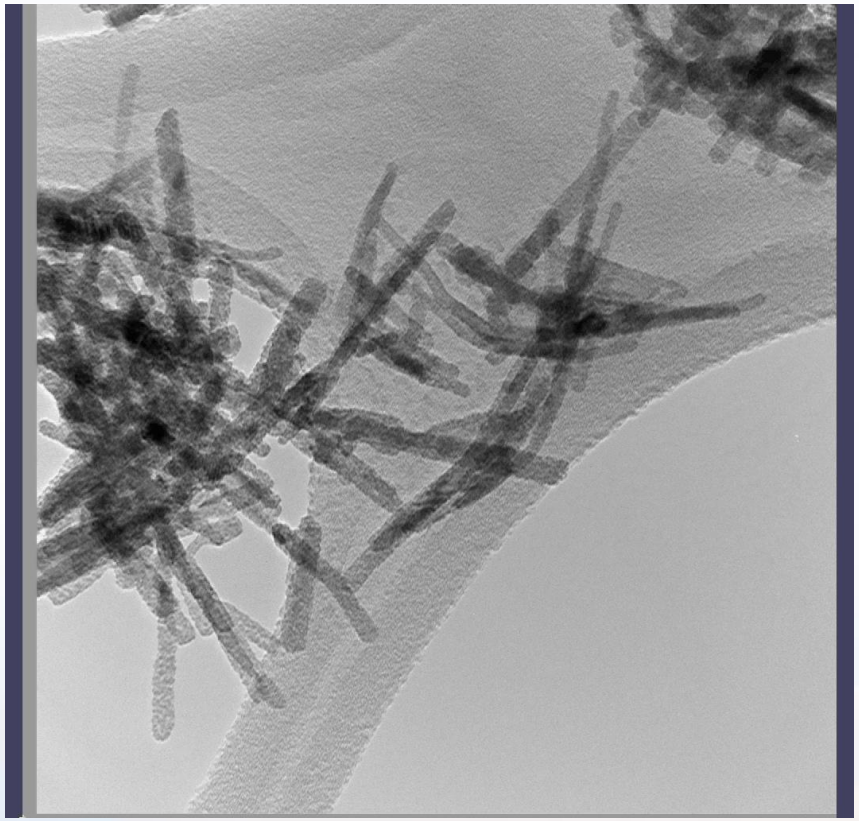






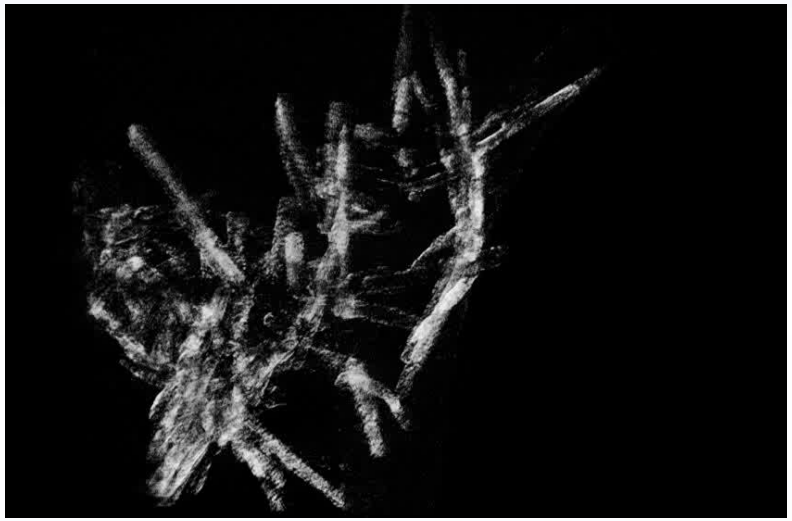


Tomography of $CdWO_4$ irradiated with 30 nA of 3 MeV Cu^{3+}

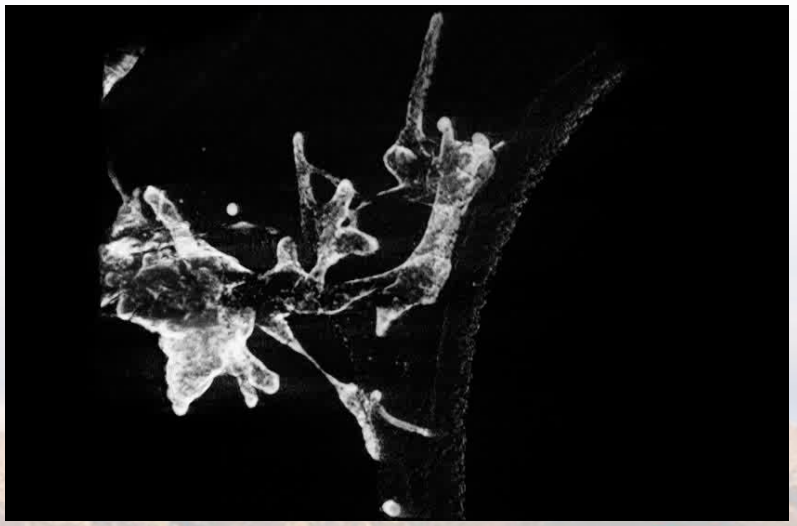


Aligned Tilt Series

Unirradiated Reconstruction

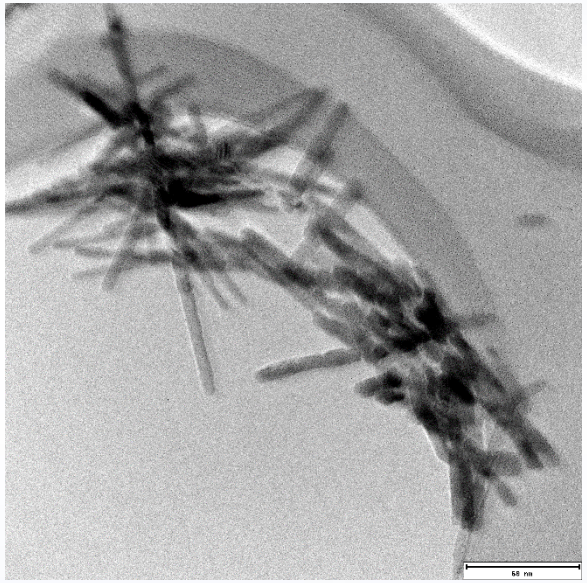


Irradiated 30 min Reconstruction

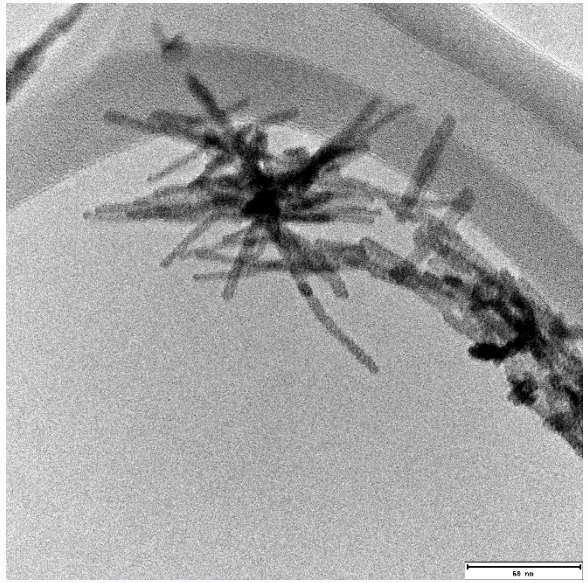


Current work: *In situ* Proton Irradiation as First Order Simulation of Neutrons

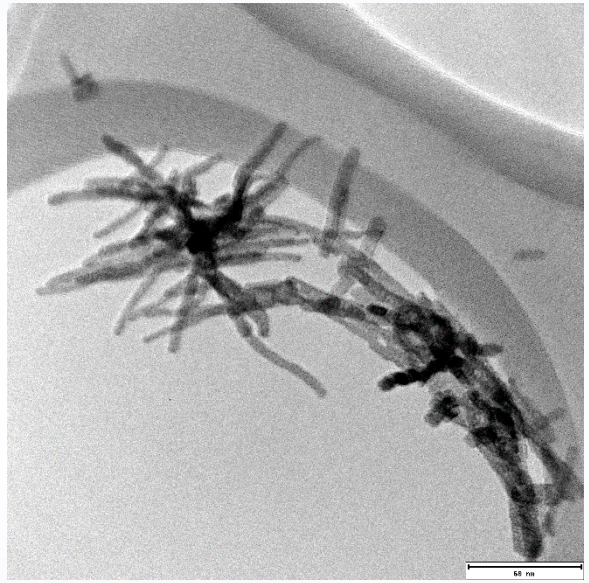
Unirradiated

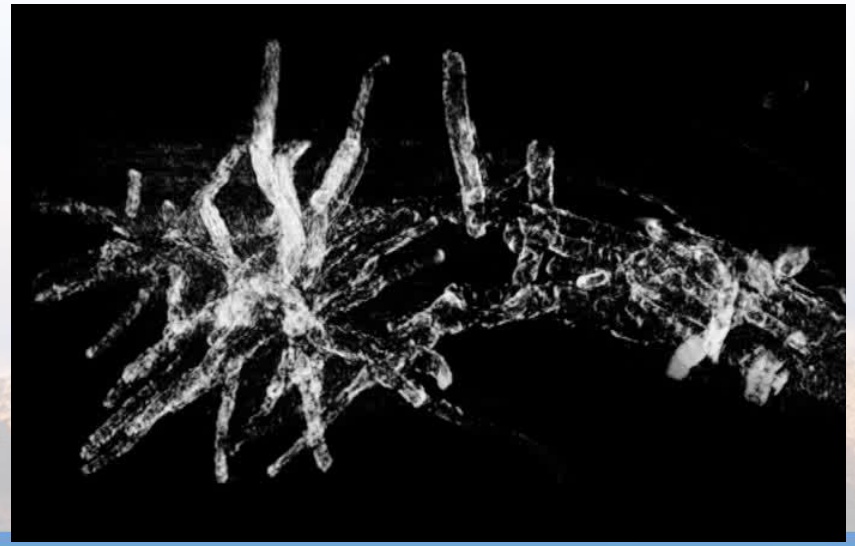


15 minutes



60 minutes

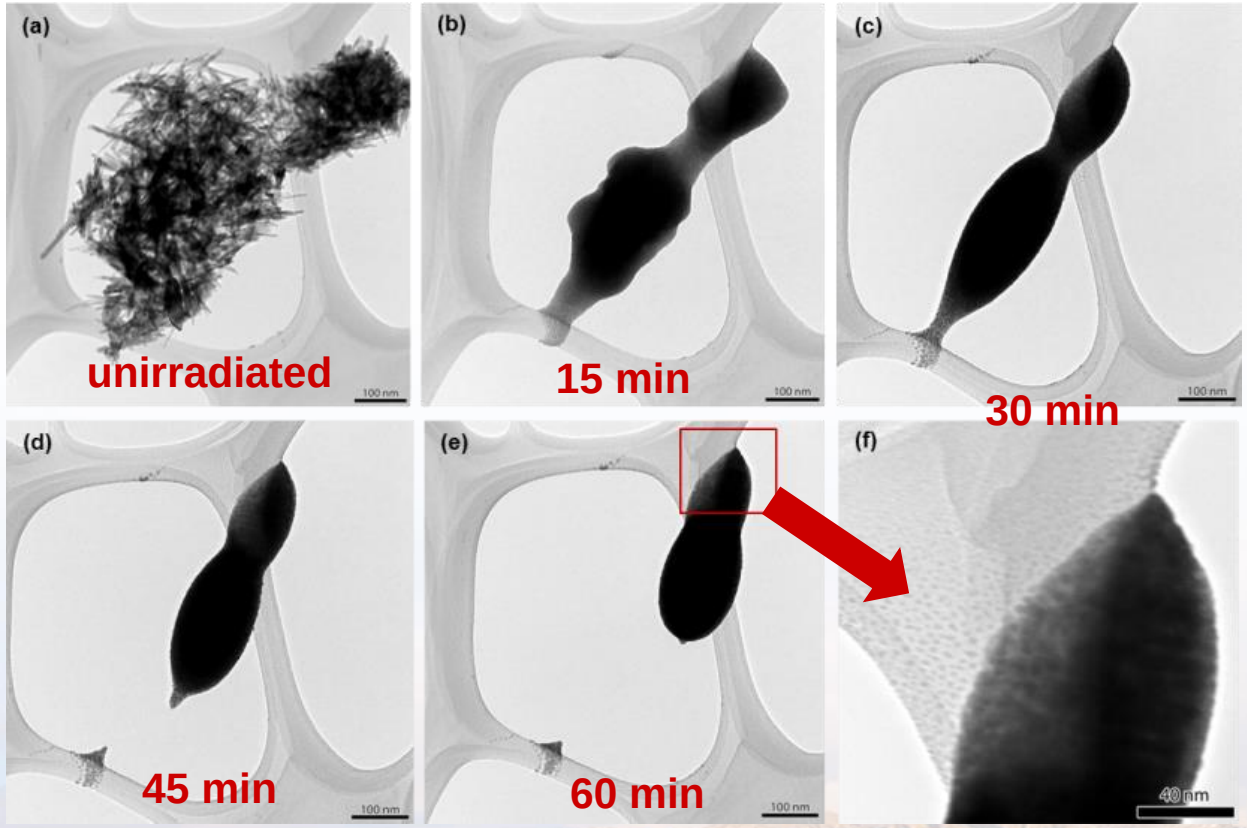




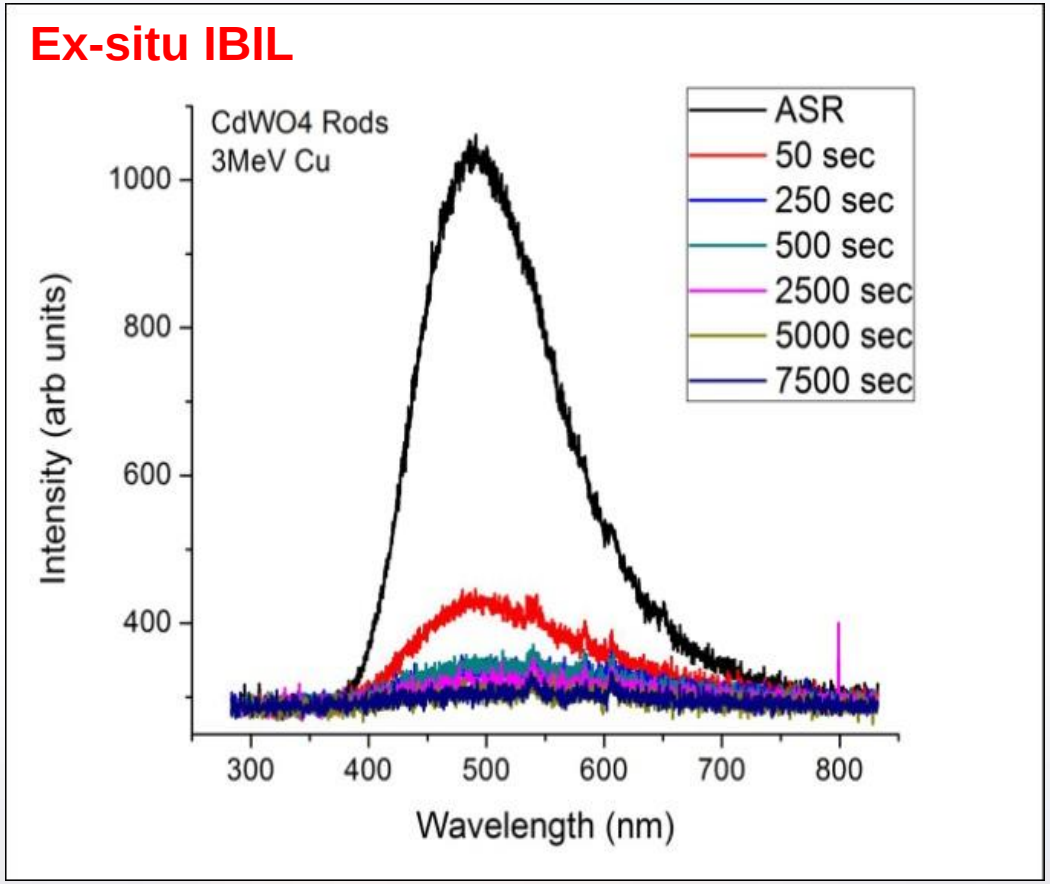
160 nA of 2.5 MeV H^+ used to simulate neutron radiation shows less change. Results suggest good radiation hardness for tungstate nanorod-composite scintillators.

In-situ Ion-irradiation and IBIL measurements of $CdWO_4$ nanorods

Region irradiated with 50 nA of 3 MeV Cu^{3+}:

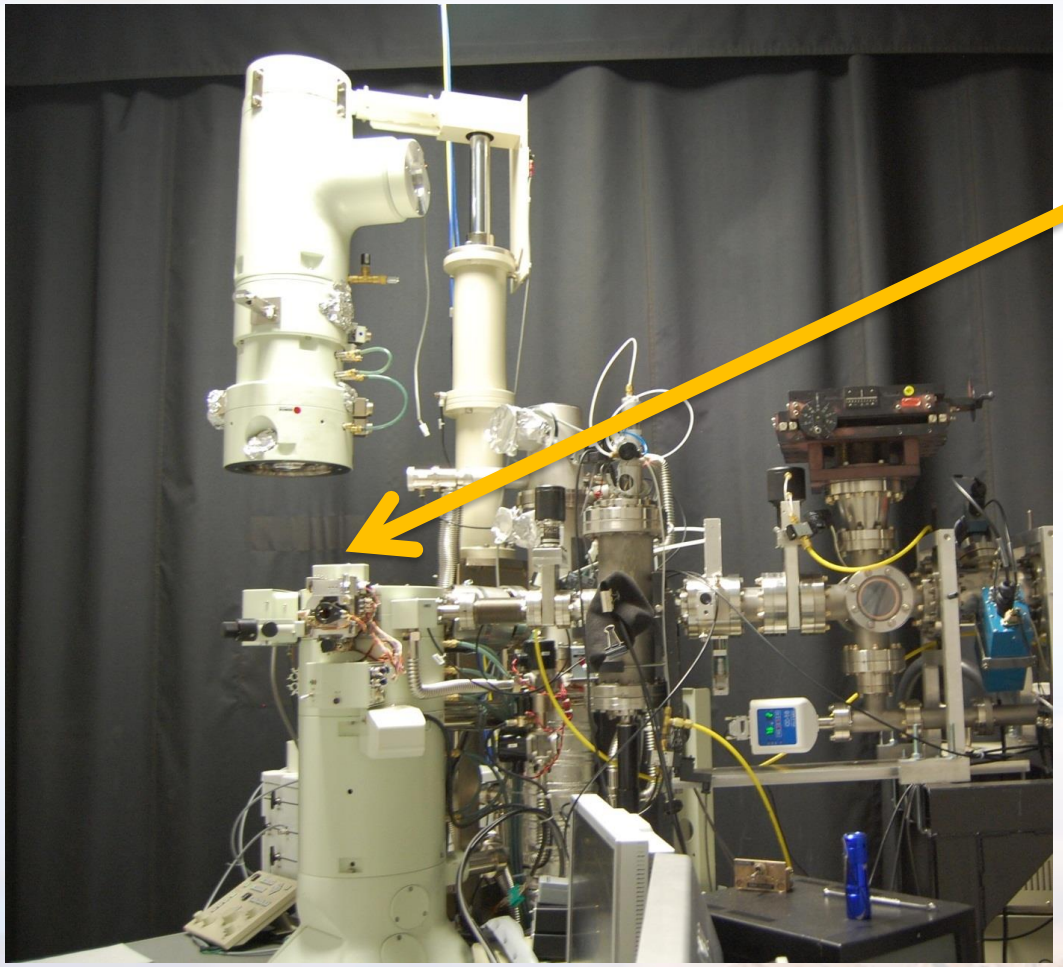


Ex-situ IBIL



In-situ TEM combined with Ex-situ luminescence measurements: Decrease in IBIL peak intensity with loss of nanorod structure

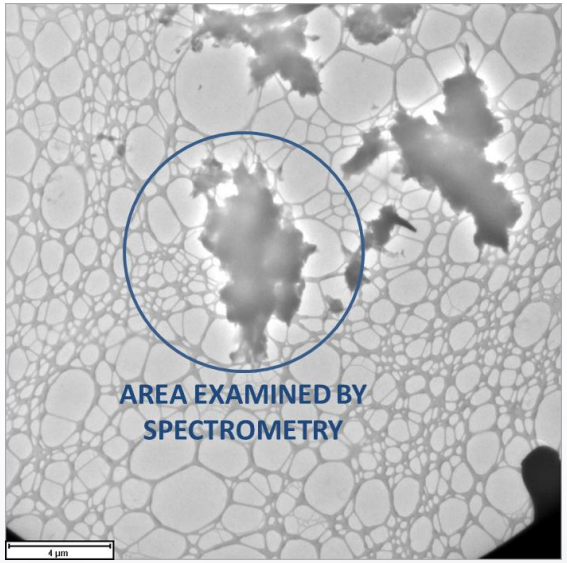
Current work: *In situ* TEM Luminescence

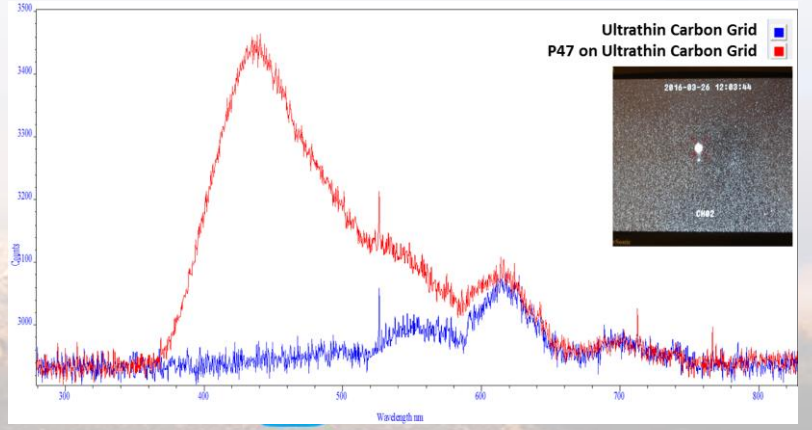


Optical Pathway in an I^{3}TEM

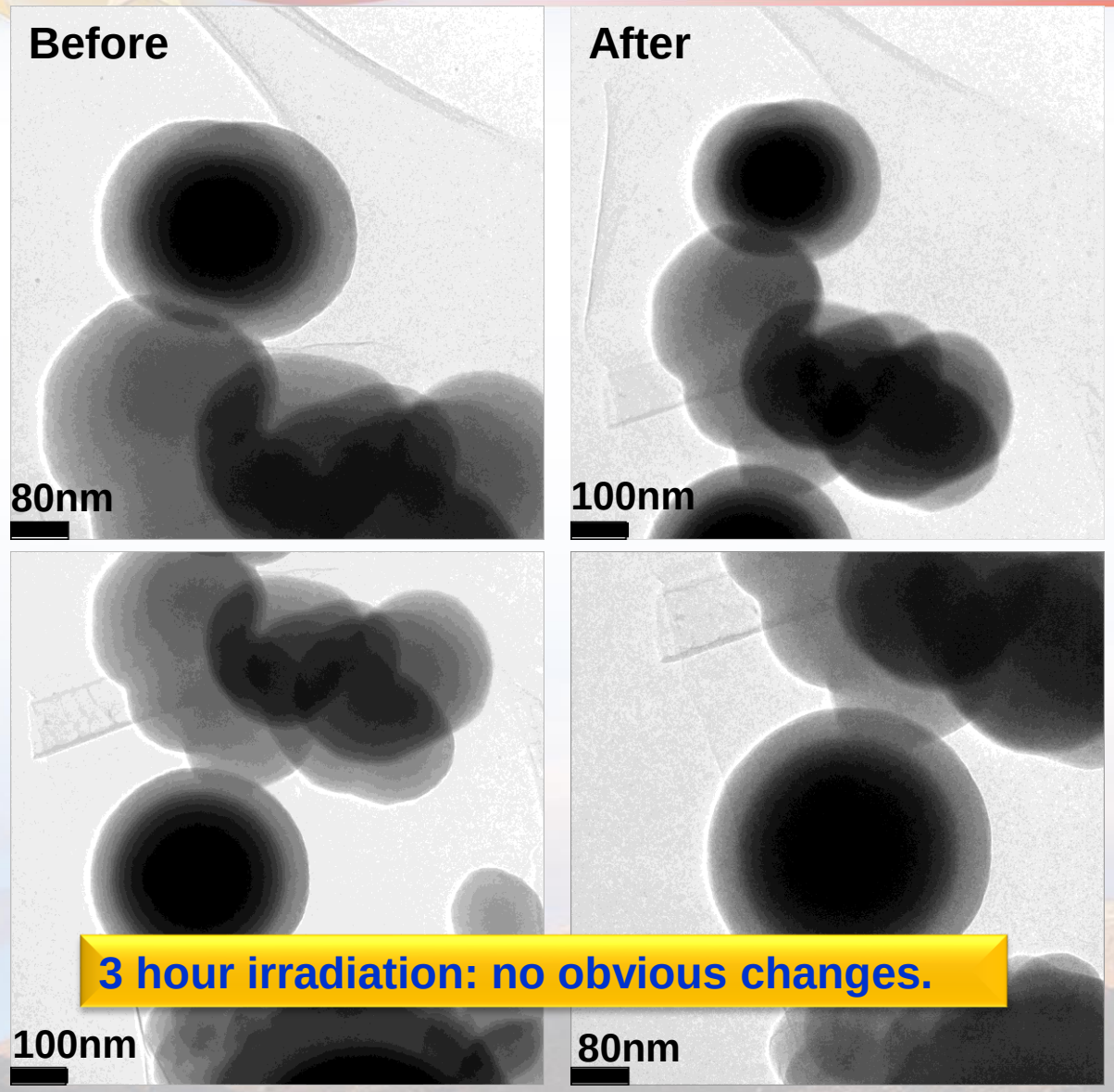
- Angled mirror with bore hole for the electron path was installed.
- Mirror is located on top of the objective polepiece “heart of the TEM”
- Port was constructed with thick leaded glass to permit light through, but not x-rays created by ion or electron beams.

Sample in-situ CL on quartz

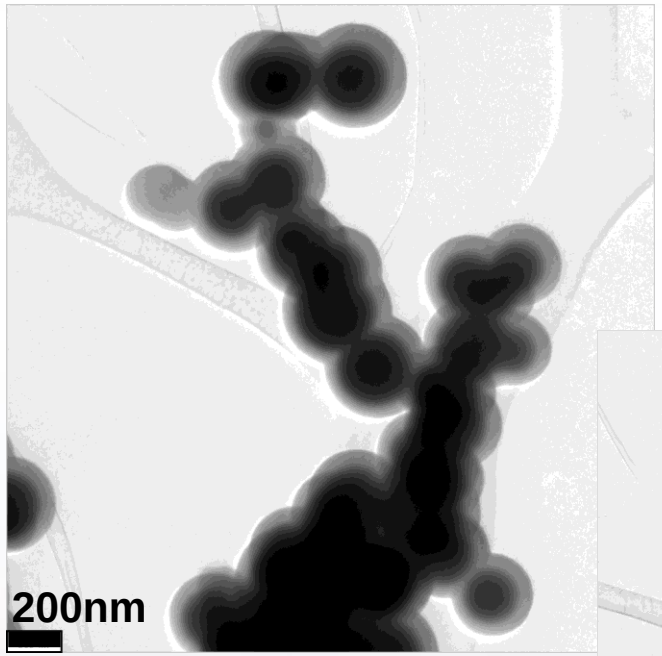




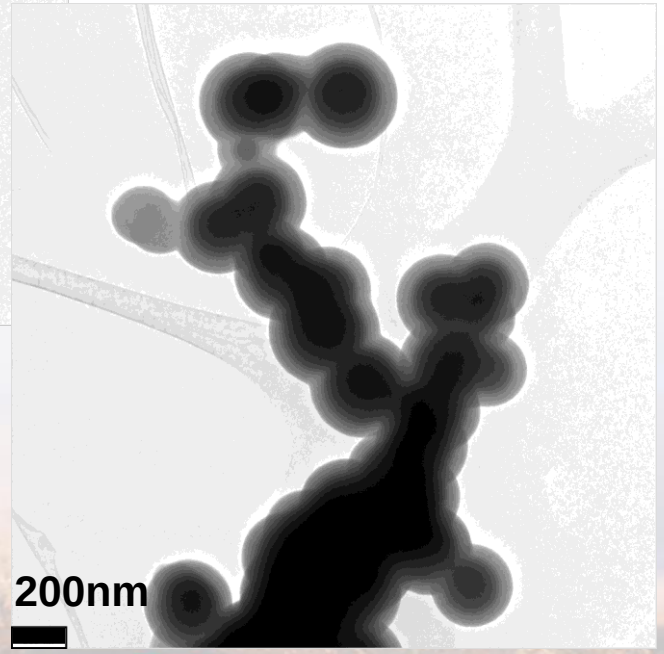
Irradiation of Amorphous Hf Oxide Nanoparticles with 10 nA of 3 MeV Cu Resulted in No Obvious Changes



3 hour irradiation: no obvious changes.

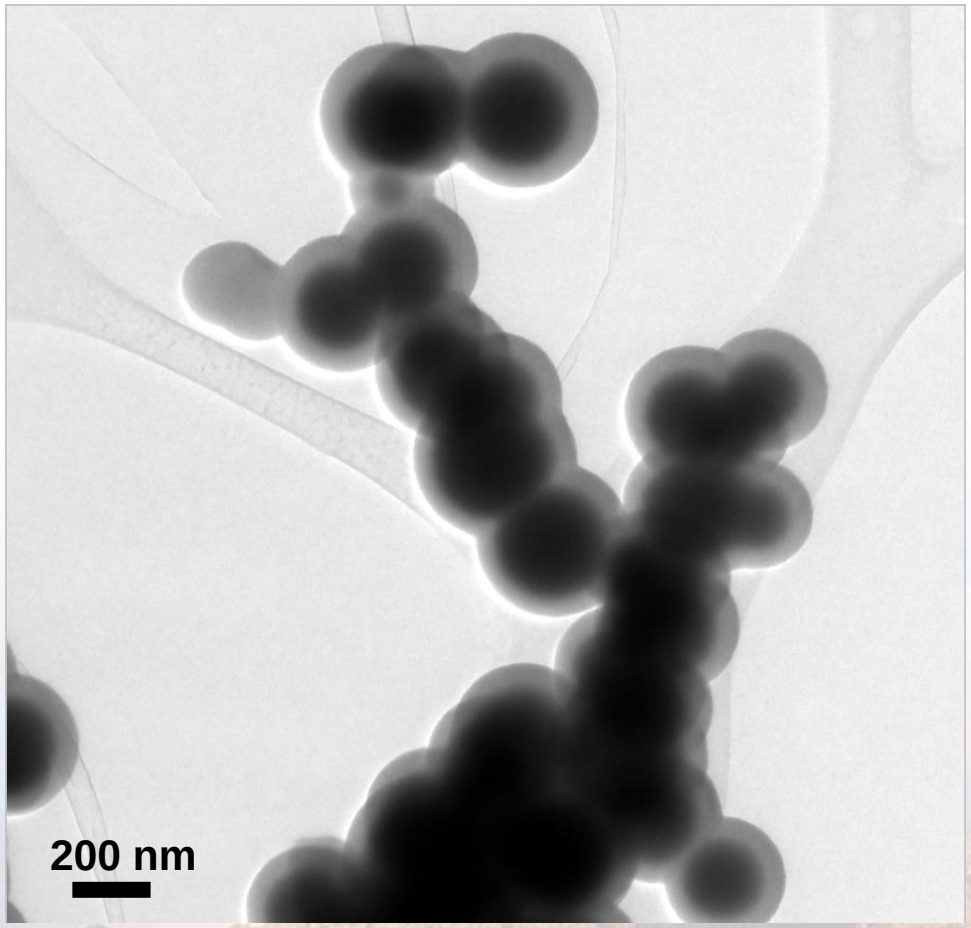


Sample was placed in tomography holder, tilted to 80°, and irradiated for an additional 2.5 h. No obvious changes.

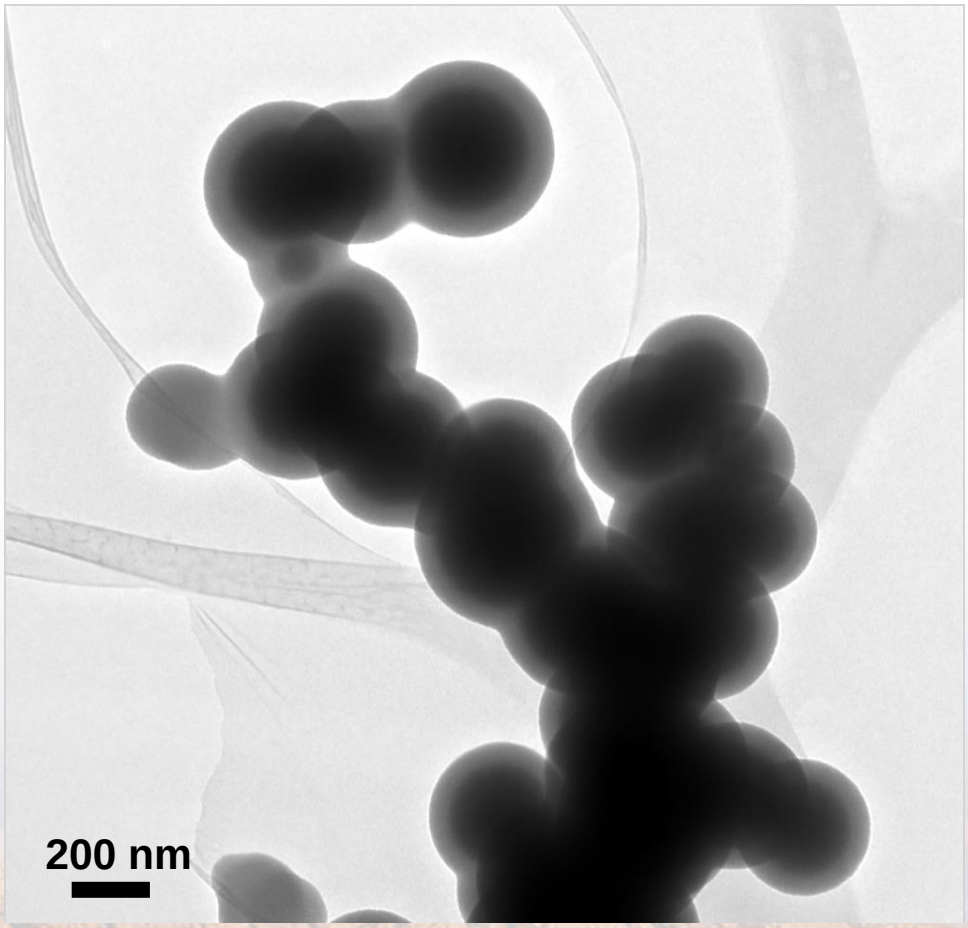


Additional Concurrent 3 MeV Cu/ 10 keV He Irradiation for 4.5 h Caused Significant Swelling of the Hf Oxide Nanoparticles

Nanoparticles after 5.5 h of 3 MeV Cu Irradiation

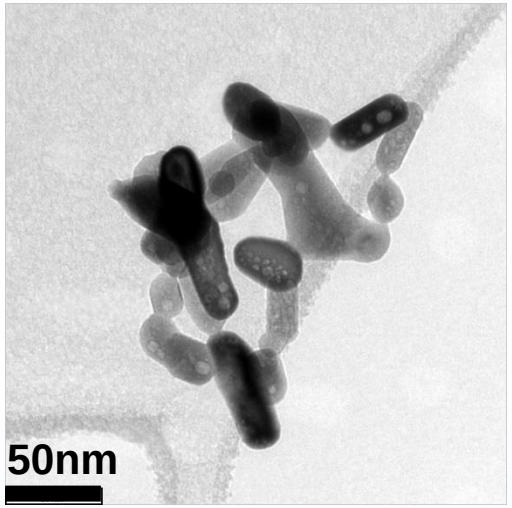


Nanoparticles after He + Cu Dual Beam Irradiation

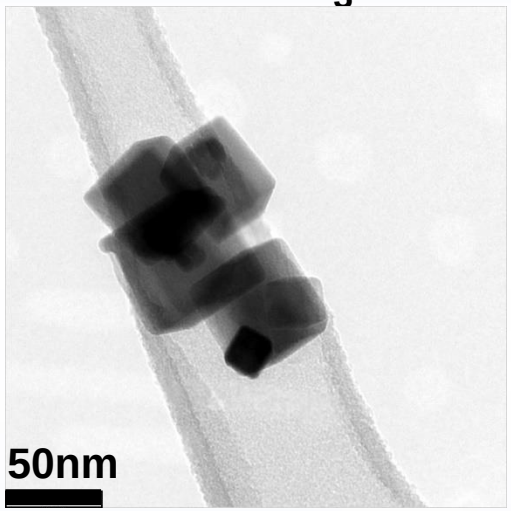


Similar to Tungstates, CeOx Nanoparticles were Synthesized into Various Shapes

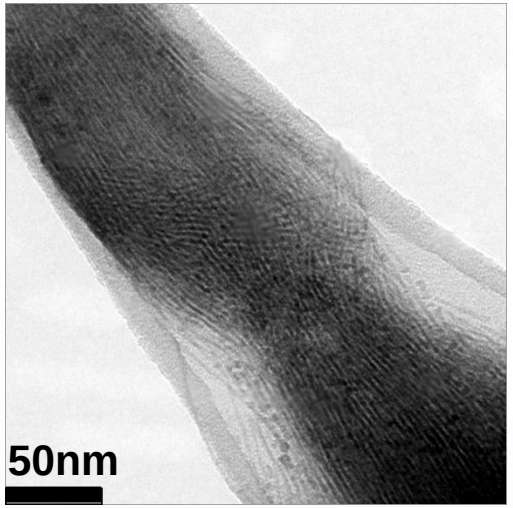
CeCl



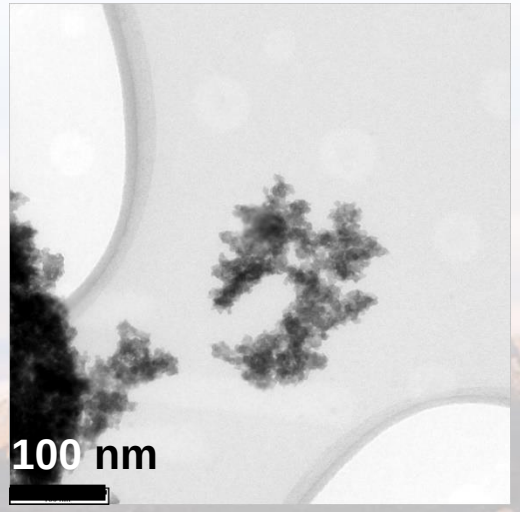
$CeNO_3$



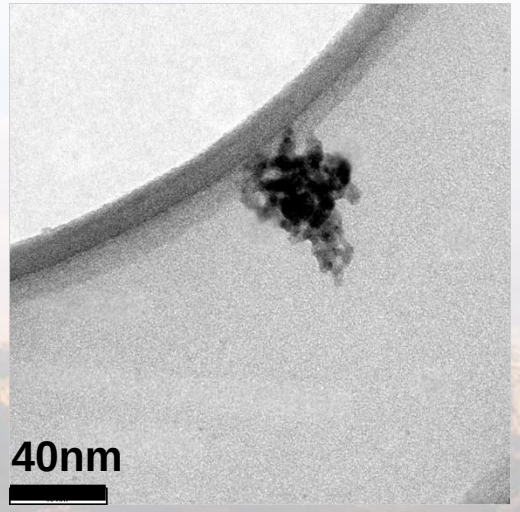
CeNR



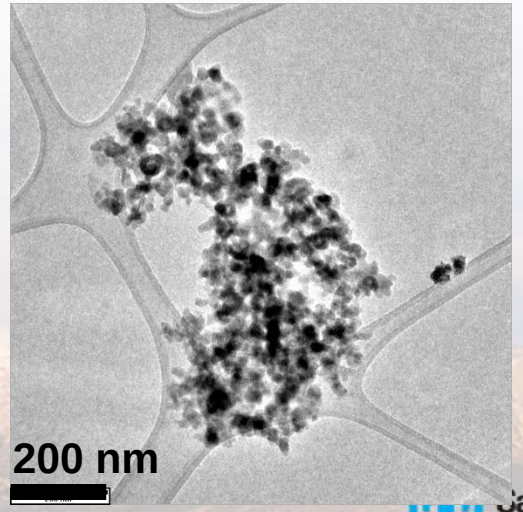
CeDBP



CeDIP

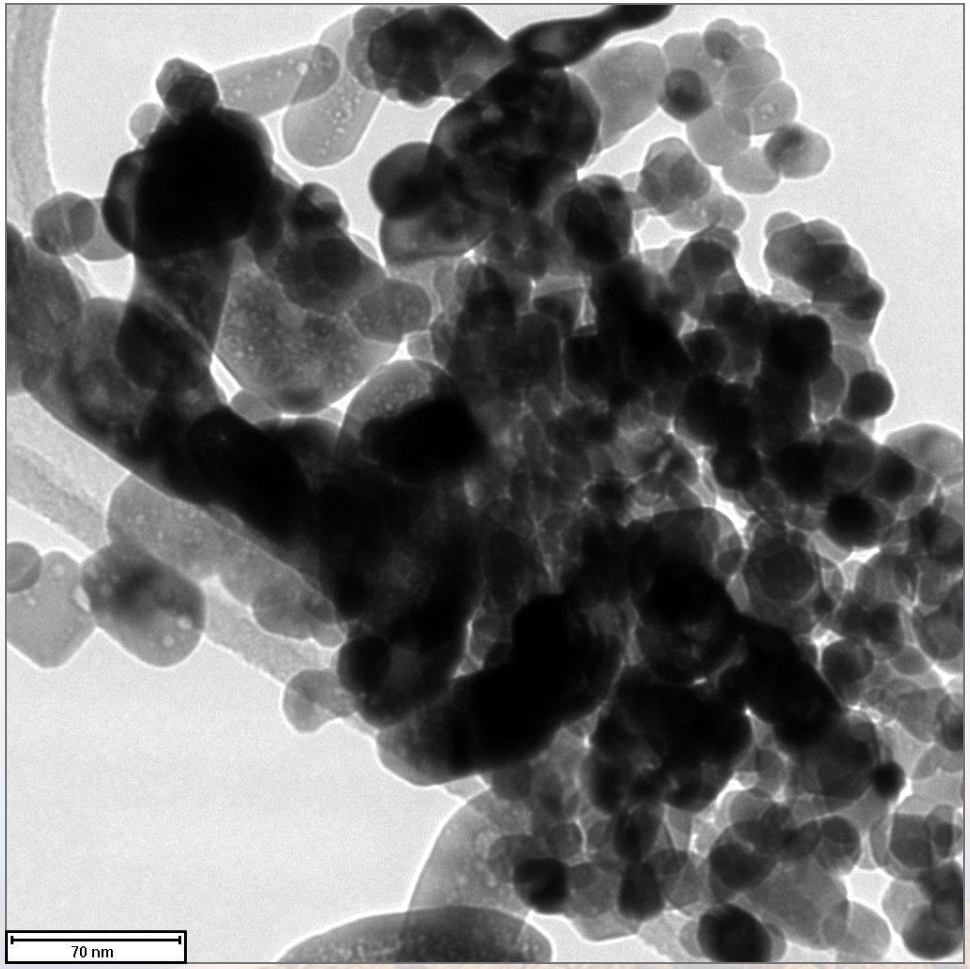


CeONeP

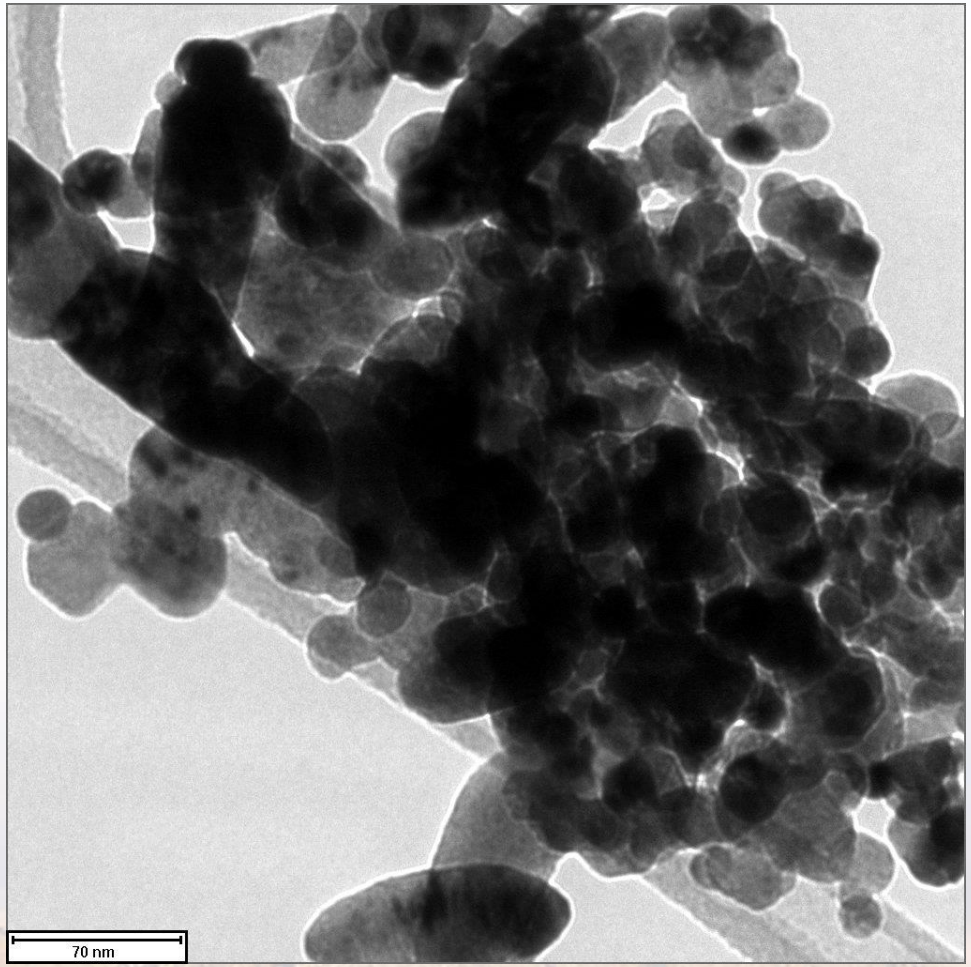


CeOx Nanoparticles Showed Varying Irradiation Resistance Depending on Initial Structure when Irradiated to 10 dpa with 1.7 MeV Au

Before



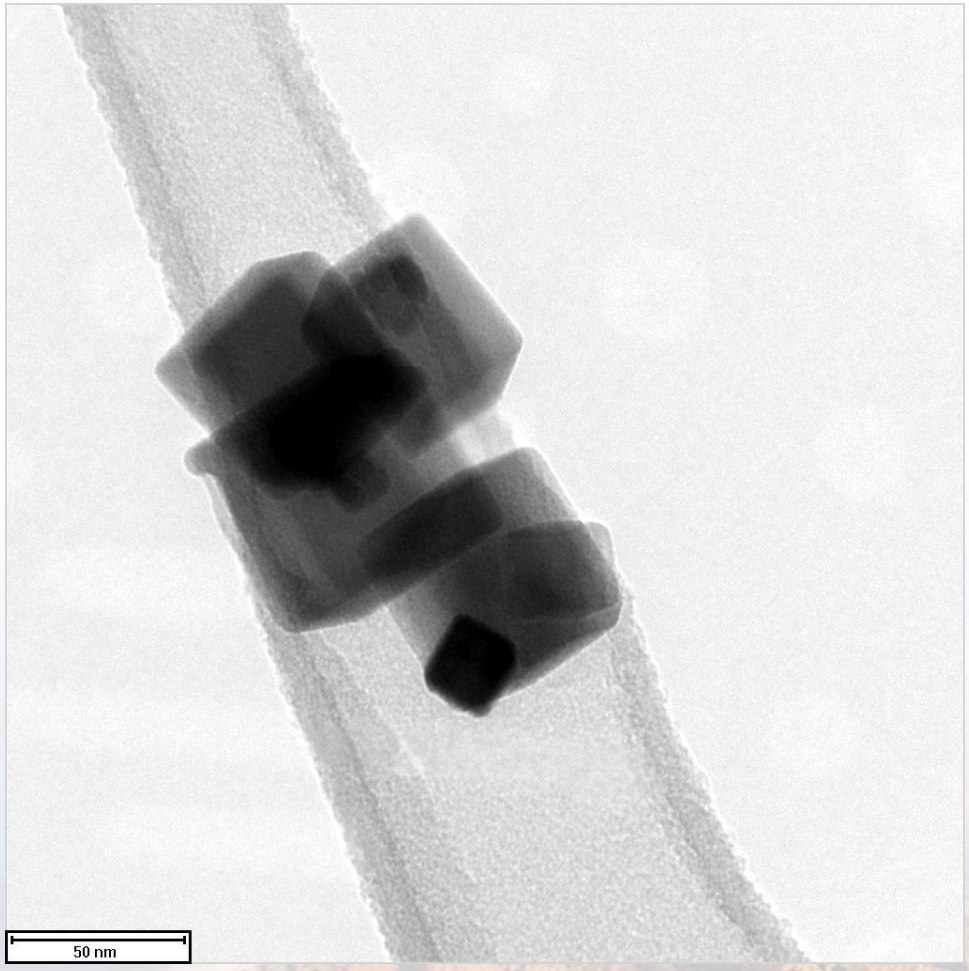
After



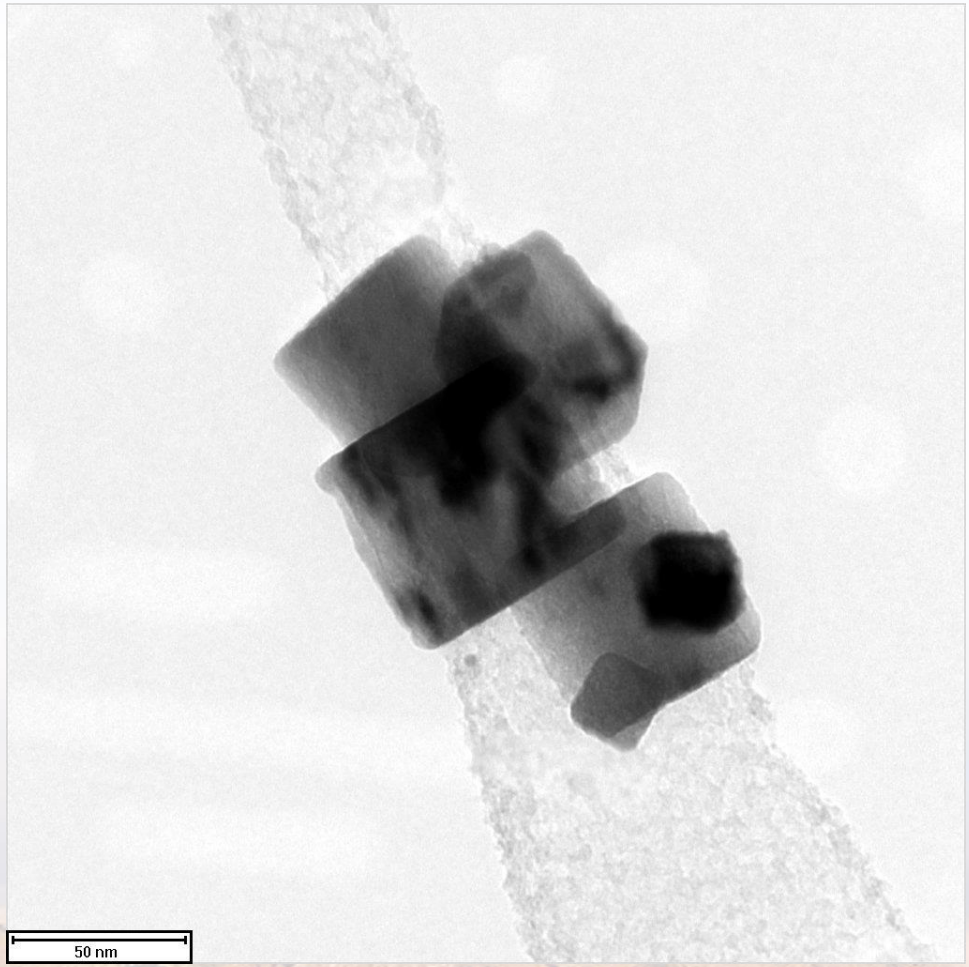
CeOx nanorods: no obvious changes after 10 dpa

CeOx Nanoparticles Showed Varying Irradiation Resistance Depending on Initial Structure when Irradiated to 10 dpa with 1.7 MeV Au

Before



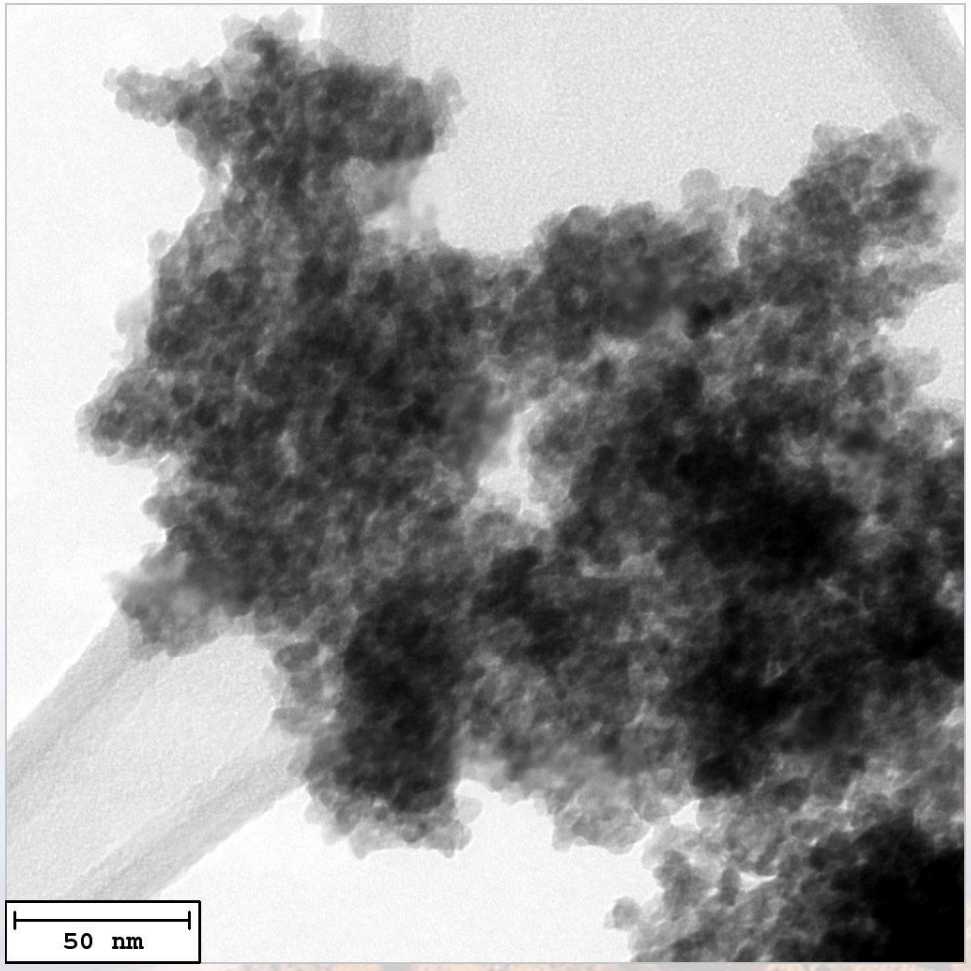
After



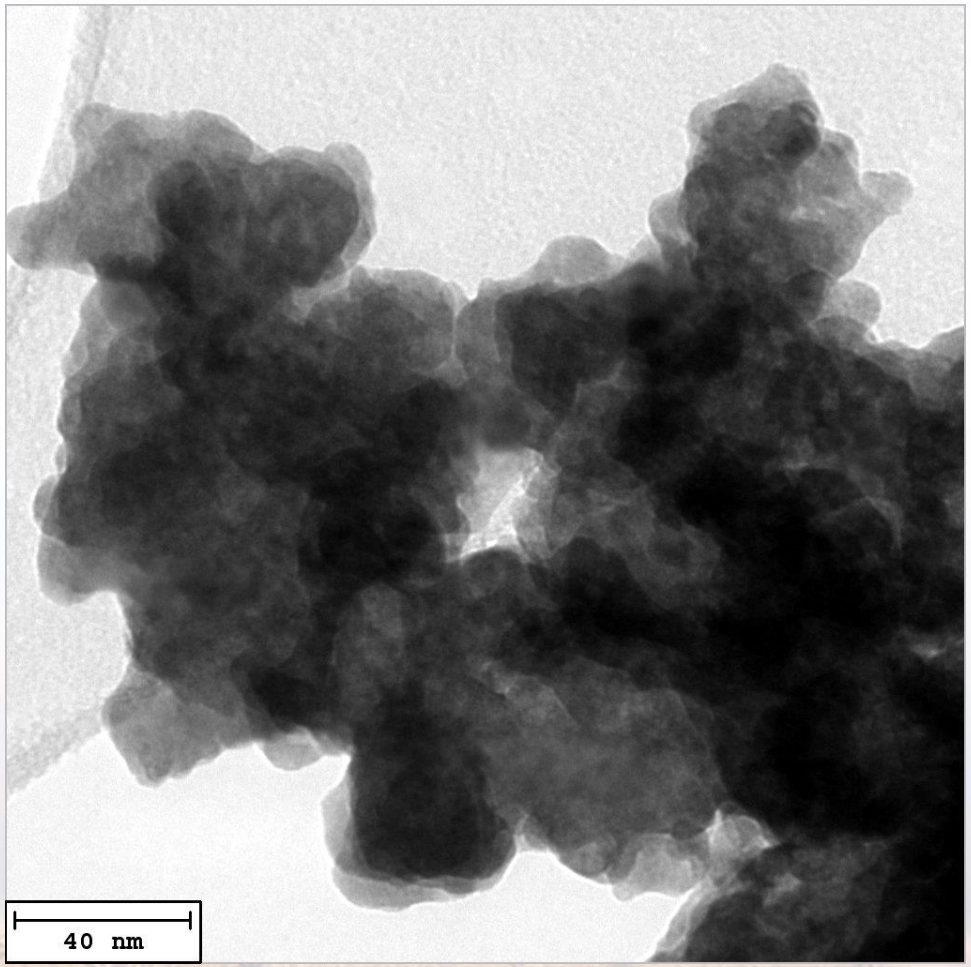
CeOx nanocubes: evidence of sputtering and possible swelling

CeOx Nanoparticles Showed Varying Irradiation Resistance Depending on Initial Structure when Irradiated to 10 dpa with 1.7 MeV Au

Before



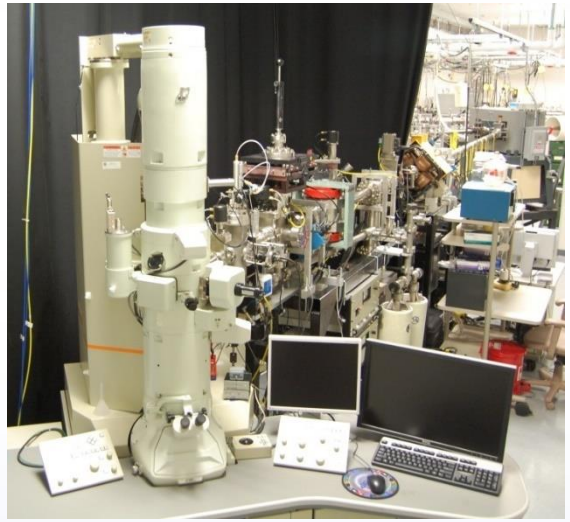
After



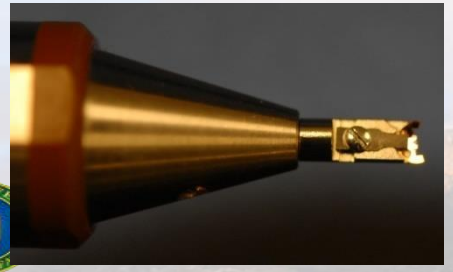
CeOx particles with no initial shape sintered to form larger particles

In-situ TEM was Utilized to Examine Irradiation Effects on Ceramic Nanoparticles

***In situ* Ion Irradiation TEM (I^3TEM)**

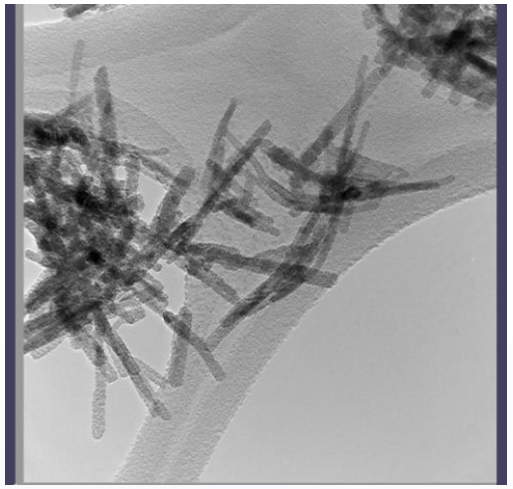


Hummingbird tomography stage

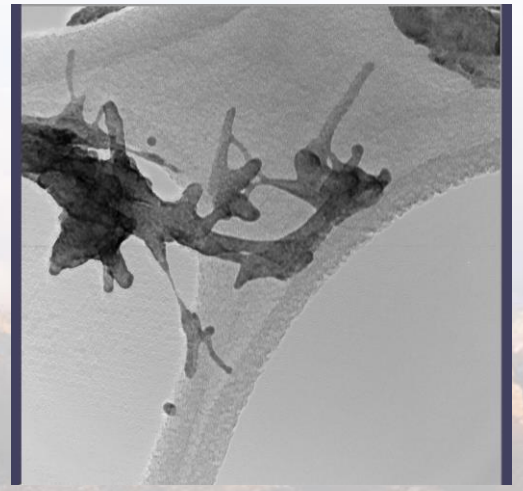


Aligned $CdWO_4$ tilt series

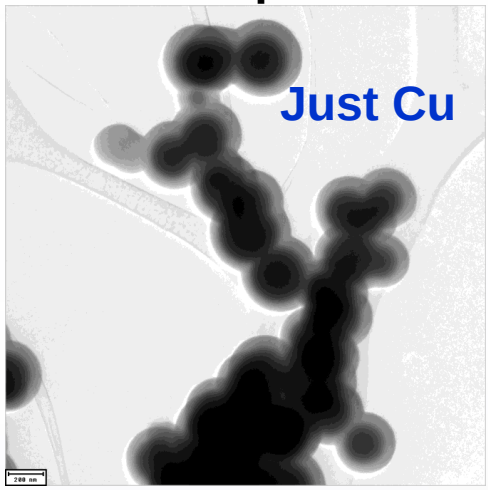
Unirradiated

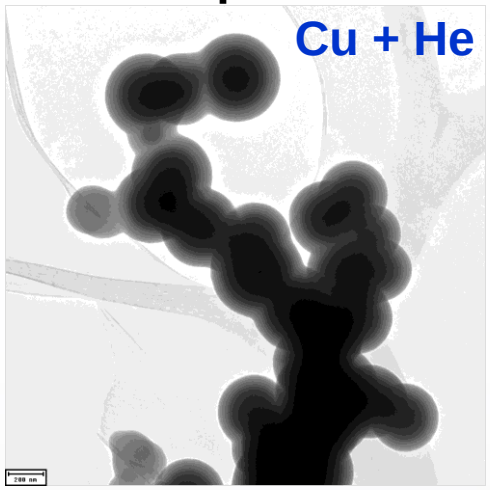


Irradiated



Swelling After Concurrent He+Cu Irradiation in Amorhpous Hf Oxide Nanoparticles





Sputtering in CeOx