MADCOR

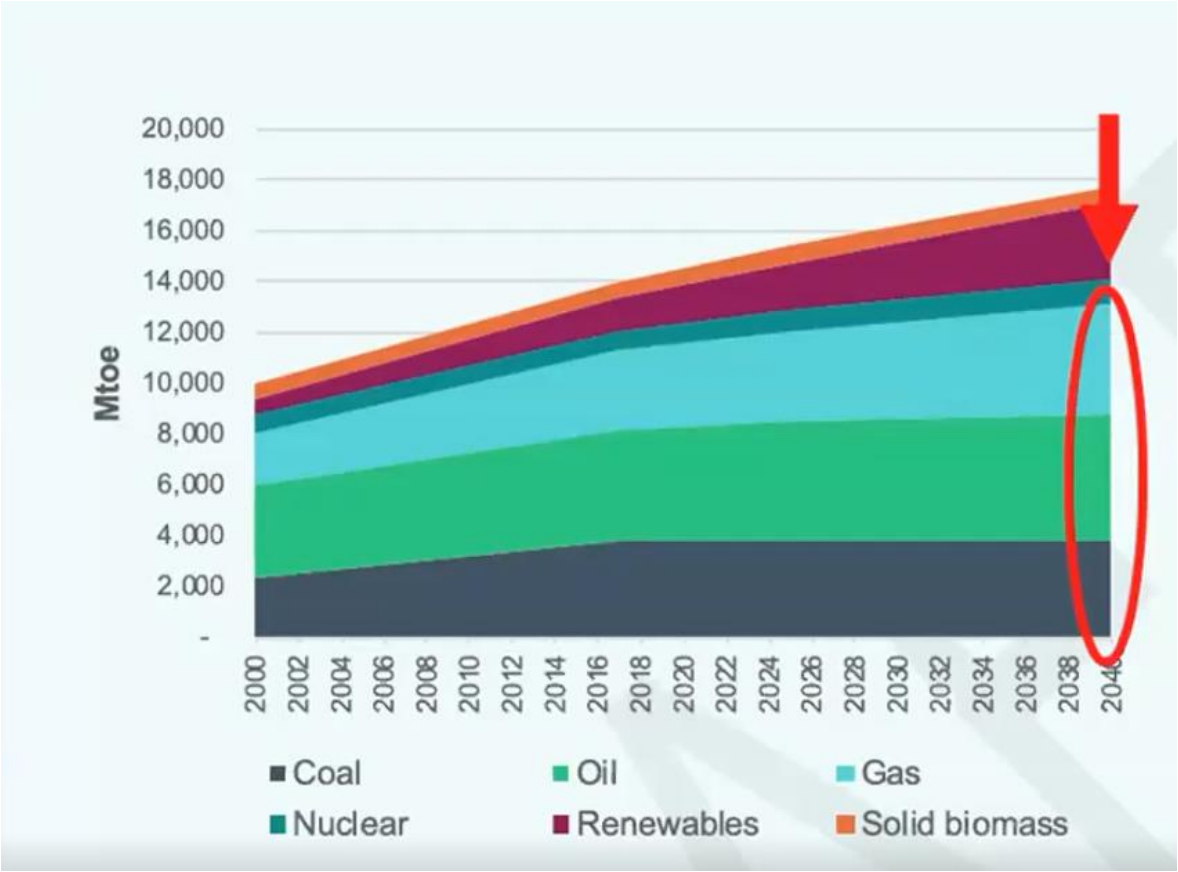
Irradiation with Low Energy Heavy Ion at University of Wisconsin and PIE capability

Adrien Couet – Associate Professor
UW-Madison

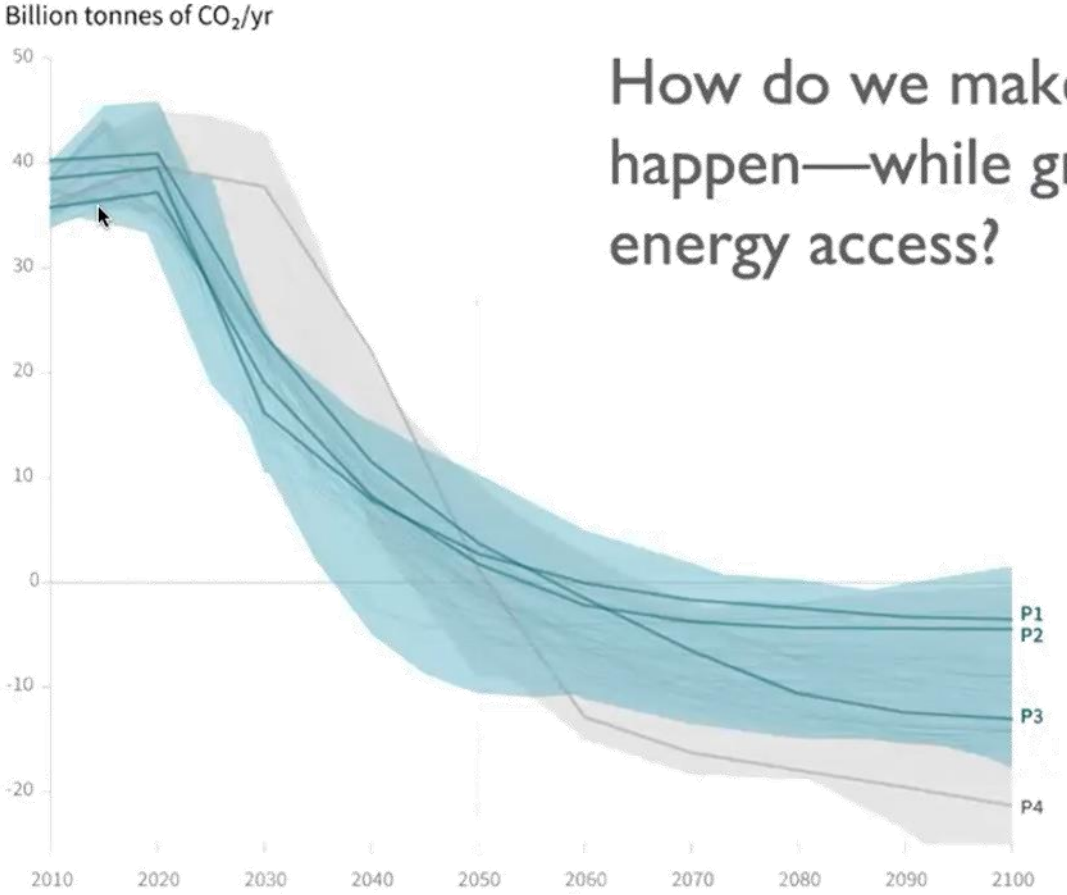
UW-Madison: Prof. Kumar Sridharan, Dr. Hongliang Zhang, Dr. Zefeng Yu, Dr. Benoit Queylat, Kim Kriewaldt, Michael Moorehead, Cody Falconer

World Primary Energy Source

IEA 2018 report
Lucid Catalyst



IPCC Special Report on Global Warming of 1.5C (2018)

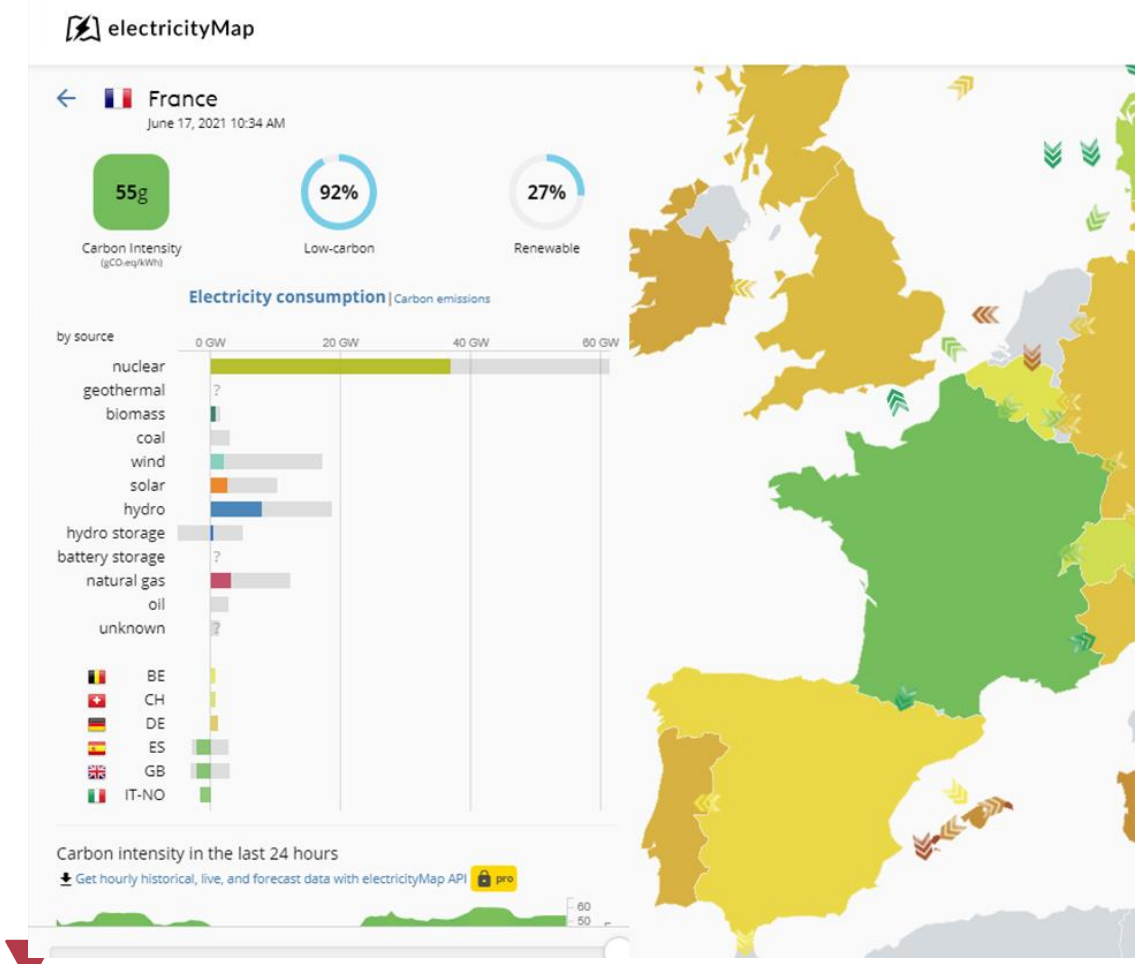


How do we make this happen—while growing energy access?

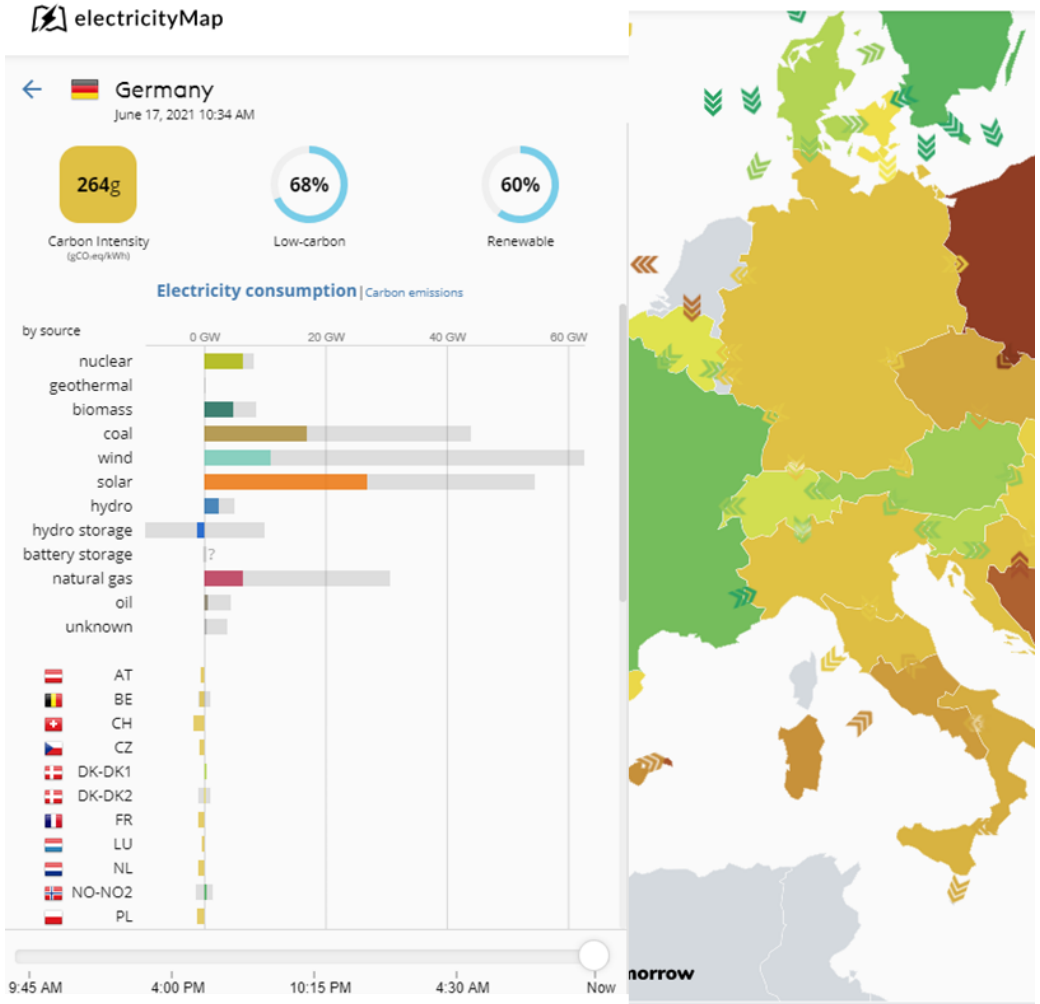
ENERGY FOR HUMANITY

Comparison France-Germany for Electricity Consumption

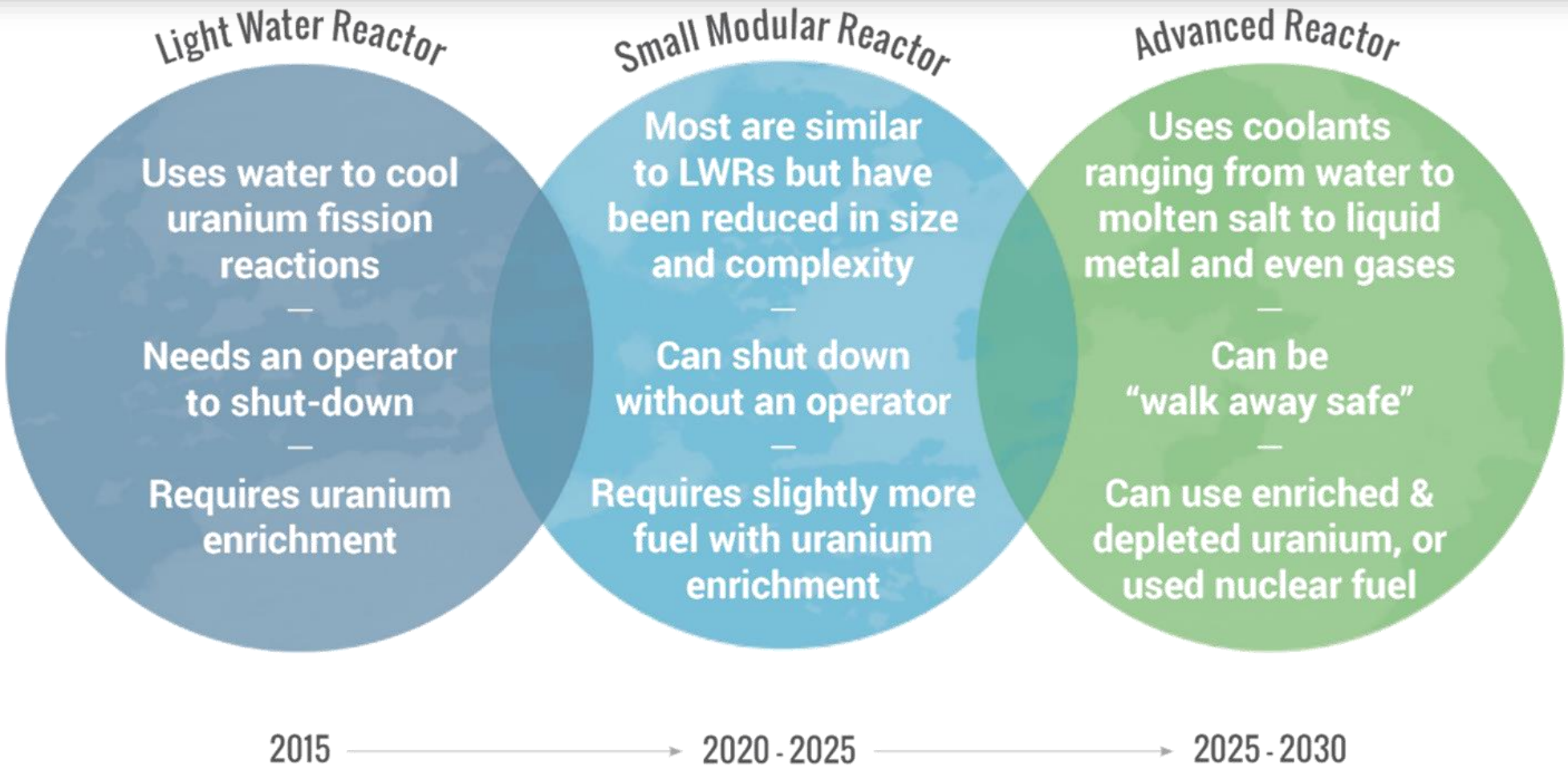
Electricity Consumption in France
This afternoon (5:44PM, 6/17/2021)



Electricity Consumption in Germany
This afternoon (5:44PM, 6/17/2021)

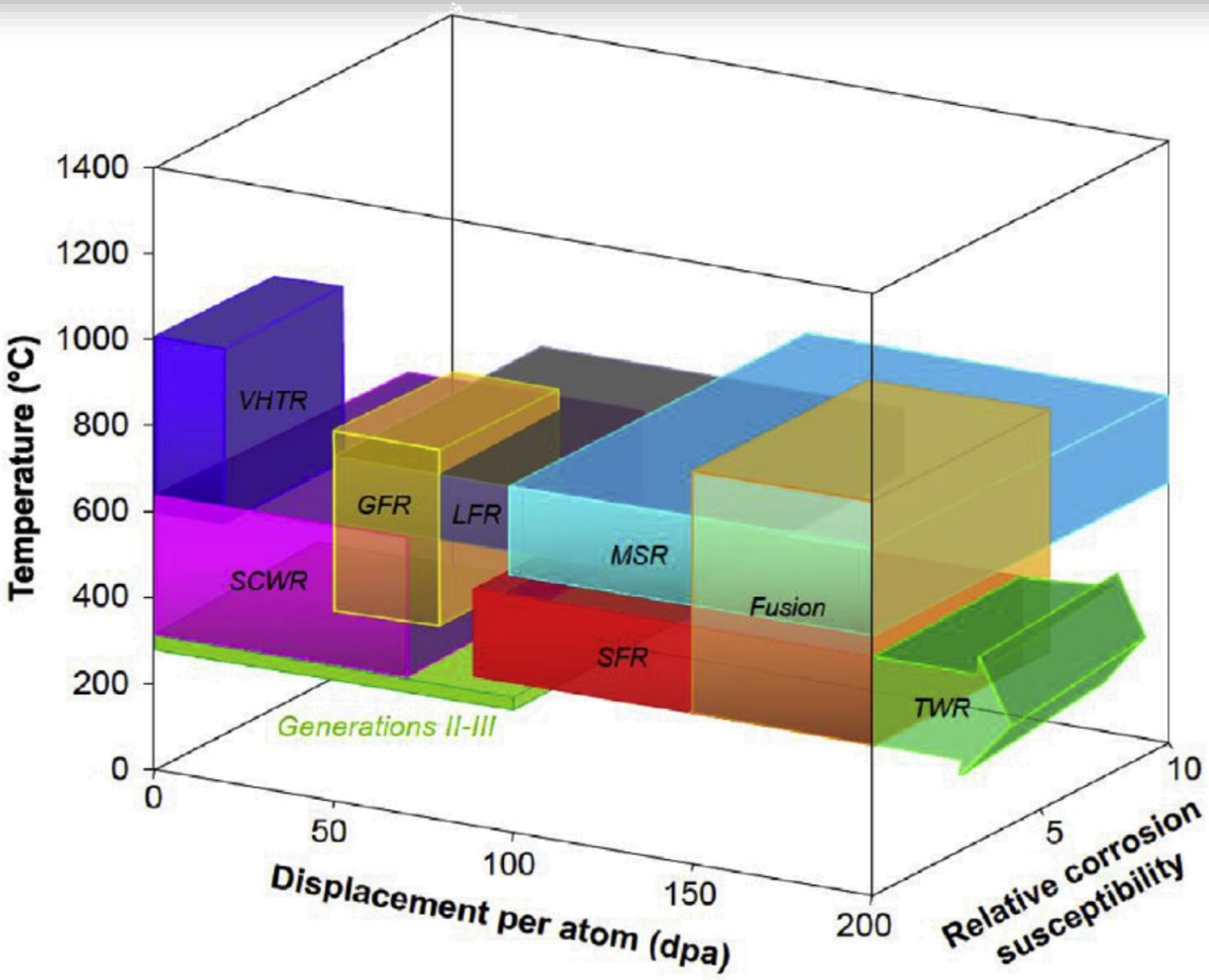


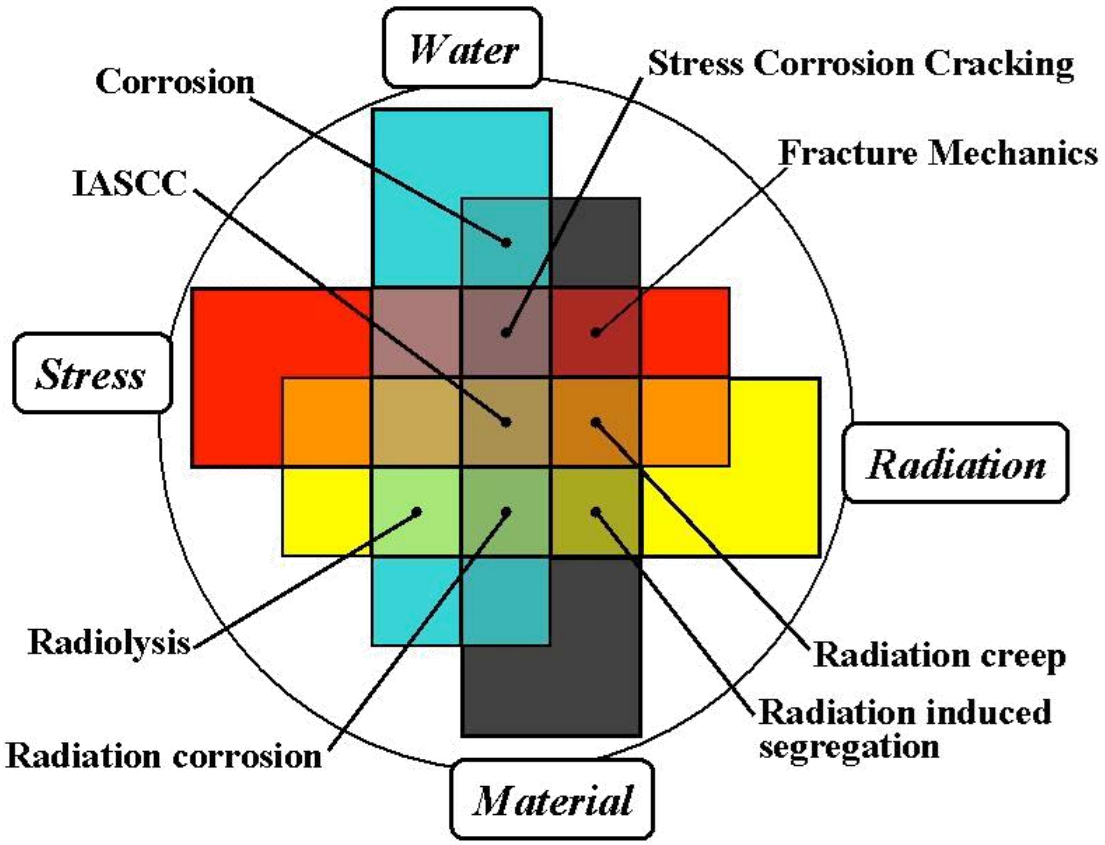
The Future of Nuclear Industry in the US



third way
fresh thinking

MAIN ACTIVITY: IRRADIATION DAMAGE IN MATERIALS



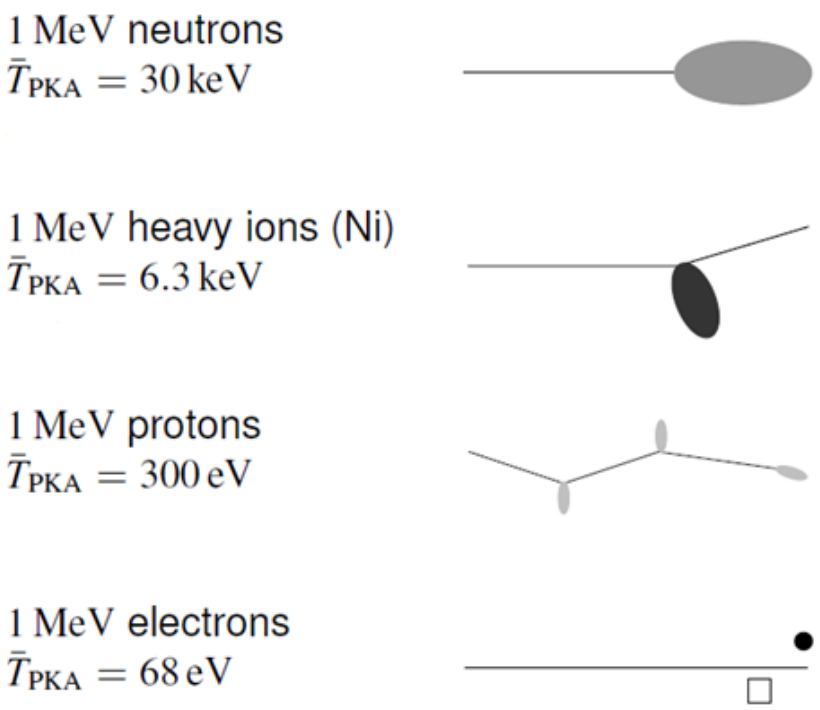


*G.S. Was, D. Petti, S. Ukai, S. Zinkle, Materials for future nuclear energy systems, Journal of Nuclear Materials 527 (**2019**) 151837.*

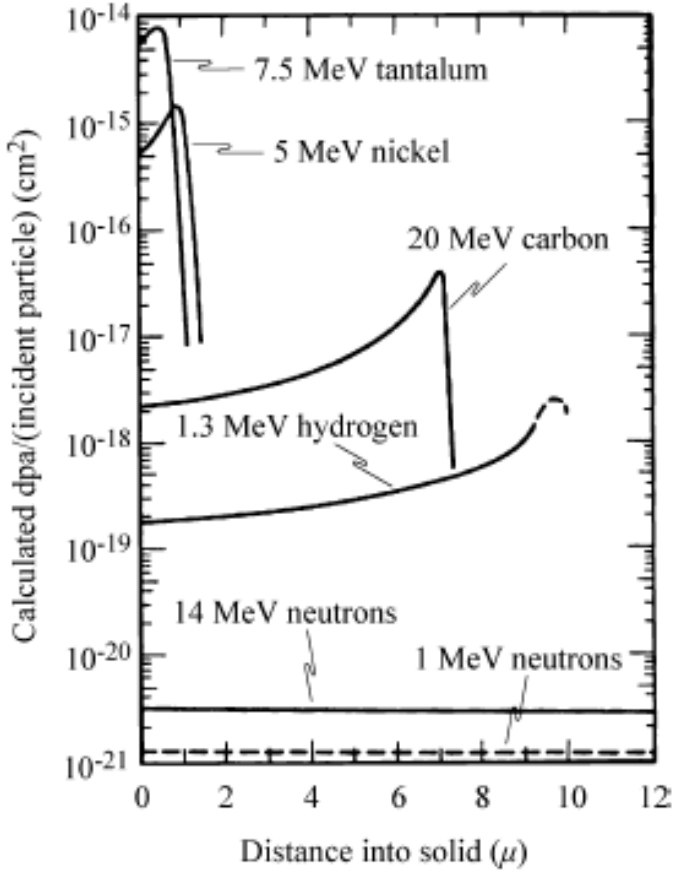
Main Activity: Irradiation Damage in Materials

- Stable defect migration
- Nucleation and growth of extended defects (dislocation loops, voids, bubbles)
- Chemical segregation or mixing,
- Etc… materials properties change!

Why Using Ions to Reproduce Neutron Irradiation?



Difference in damage morphology of pure nickel after irradiation with various types of particles at the same energy ($\bar{T}_{PKA}$ is the average PKA energy)



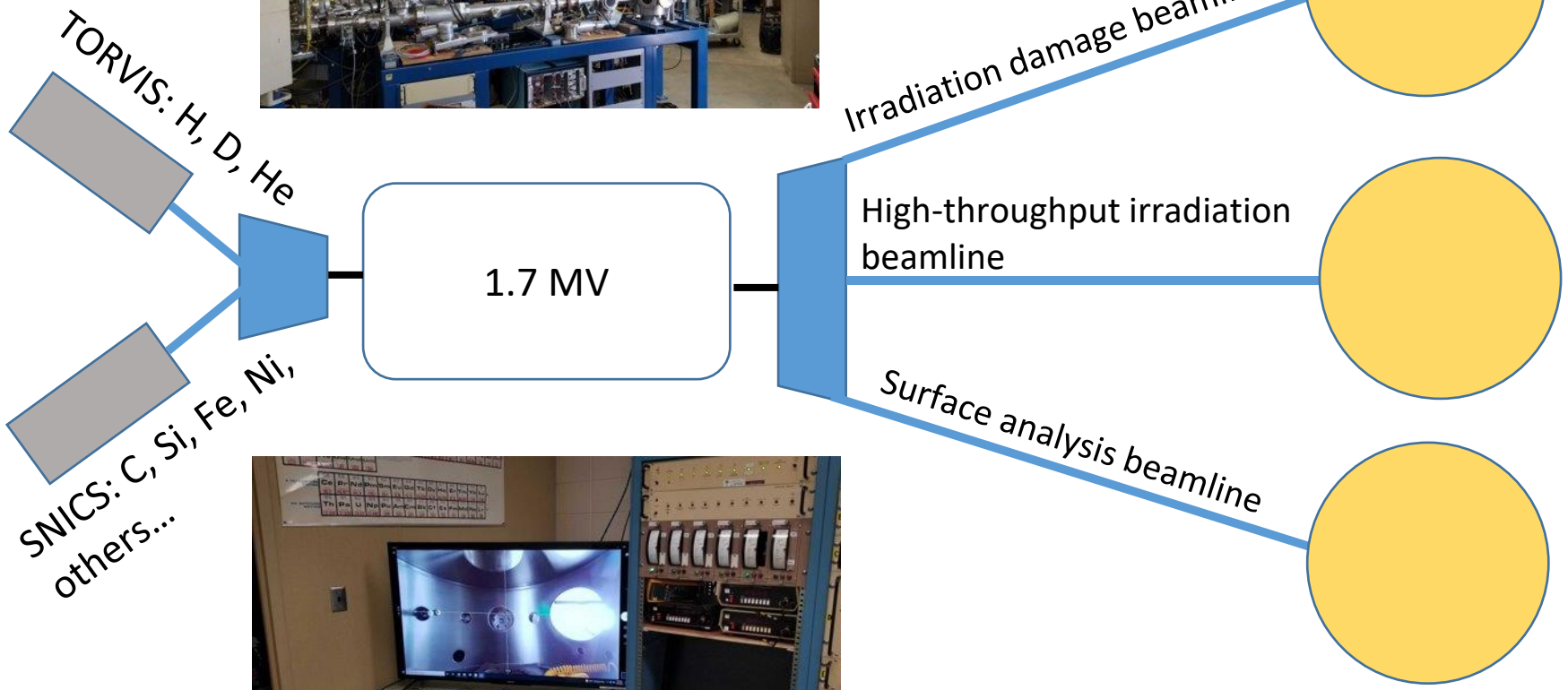
Displacementdamage effectiveness for various energetic particles in nickel. Source: Kulcinski GL, et al. « Production of voids in pure metals by high-energy heavy-ion bombardment », Proceedings of Radiation-Induced Voids in Metals, 1972, p 453

- Higher dpa rate: (can reach "high" fluence "rapidly")
- No or limited material activation
- In-situ monitoring
- Lab scale
- Cheaper

UW – Ion Beam Laboratory (IBL)

- 1.7 MV tandem accelerator from National Electrostatics Corporation (NEC)
- Temperature monitored with thermocouples and IR camera
- Various samples geometries
- Rastered or defocused beam
- Toroidal Volume Ion Source (TORVIS) and Source of Negative Ions via Cesium Sputtering (SNICS) ion sources

UW – Ion Beam Laboratory (IBL)



- Water and liquid metal cooling for precise temperature control,
- heating cartridges (up to 1000C)
- In-situ electronic measurement

- Large chamber with load lock
- 3D moving stage for high-throughput irradiation
- Laser heating from front

- Ion irradiation and ion beam analysis
- Rutherford backscattering spectrometry
- Particle-induced X-ray emission.

UW – Ion Beam Laboratory (IBL)

- TORVIS: High current H, D, and He ions, from < 1 μA to 100 μA. Large flat damage/depth profile
- SNICS: Wide range of heavy ions, Fe, Si, C, V, Nd, and more. Fast to achieve high damage, e.g., 250 dpa peak damage of stainless steels in less than 8 hours. No radiation-induced radioactivity.
- Rastered or defocused beam can be provided

Beamline	Irradiation damage	Surface analysis	High-throughput irradiation
Temp Range:	50°C-1000°C ±30°C	-150°C-1100°C ±5°C	RT to 800C (laser heating)
Temp. control	2 thermocouples + IR camera	3 thermocouples + IR camera	thermocouples + IR camera
Flux Range (cm^2)	$5x10^{12}$ - $2x10^{14}$	$1x10^{10}$ – $2x10^{15}$	$1x10^{10}$ – $2x10^{15}$
Irradiation Area	1.5 - 2.3 cm^2	0.1 - 6cm^2	0.1 - 225cm^2
Vacuum	1e-7 Torr	1e-8 Torr	5e-7 Torr
In-situ analysis	Resistivity measurement	RBS,NRA,PIXE	3D movement and rotation, and variable heating
Sample size	1cm x 2cm	1cm x 1.5 cm	15cm x 15 cm

UW – Ion Beam Laboratory (IBL) History and Upgrade

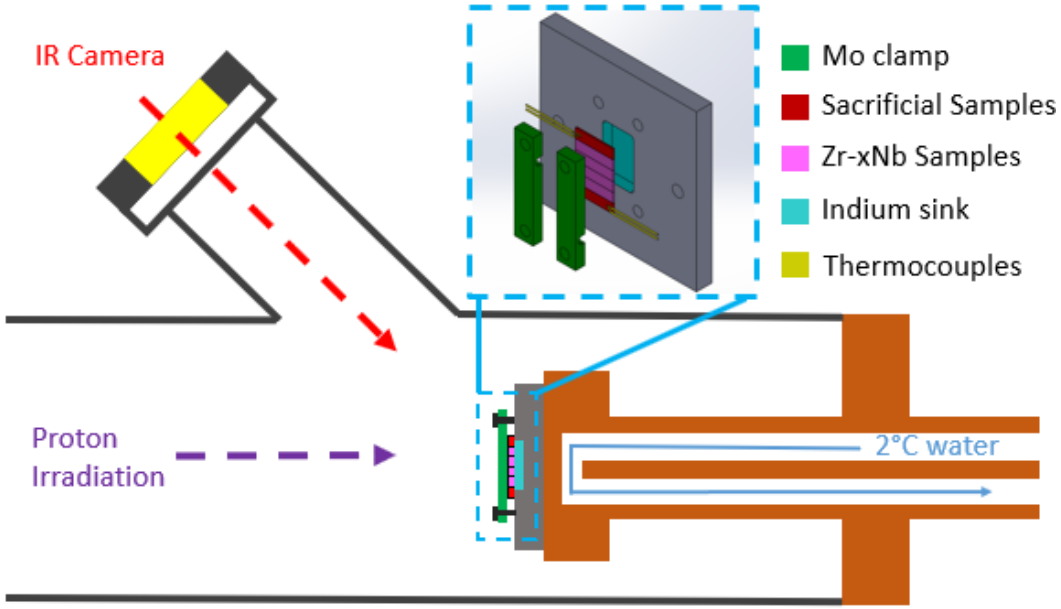
- 2009: Nuclear Science User Facility (NSUF) partner.
- 2011: TORVIS ion source (high beam current for H and He).
- 2014: CLIM laboratory (consolidation of sample preparation tools, TEM and SEM).
- 2015: New chamber upgrade (control of irradiation area, pre-loading chamber).
- 2016: Sample activity screening (count integration over time, better accuracy).
- 2017: Sample stage upgrade (liquid metal contact to ensure proper cooling).
- 2018: Automated 3D sample stage design and manufacturing
- 2019: In-situ resistivity measurement, load lock
- 2020: 2 kW IR laser for surface heating, coupled to a pyrometer and IR camera for temperature measurements
- 2021: Integrated Labview control of sample stage movement, irradiation parameters and monitoring probes

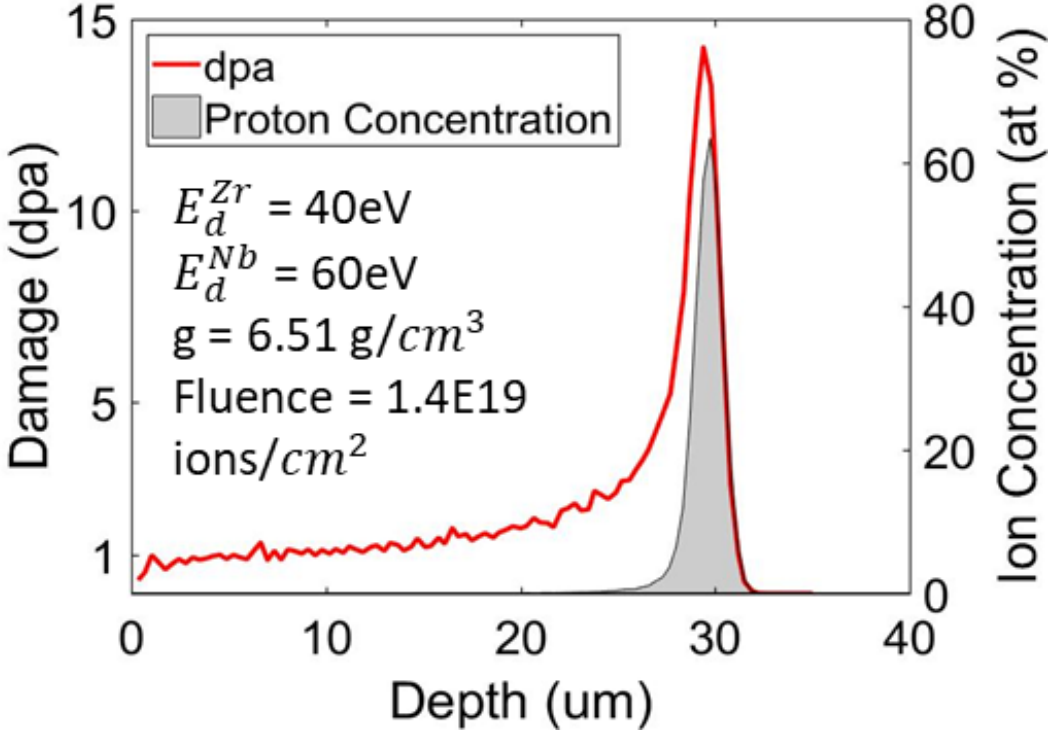
UW – IBL Usage

- 2017-2018:
  - 22% UW-Madison committed non-NSUF projects
  - 50% DOE funded projects
  - 28% others (industry)
  - Total 350 hours
- 2018-2019:
  - 41% UW-Madison committed projects
  - 50% DOE funded projects
  - 9% others (industry)
  - Total 400 hours
- 2019-2020
  - 70% UW-Madison committed projects
  - 30% DOE funded projects
  - Total 260 hours (limited access and operations because of Covid)

UW – IBL: Example 1 – Proton Irradiation of ZrNb alloys

- 2MeV Proton Irradiation
  - 8-10 µA , 1.4E19 ions/cm^2, 1.0 dpa, 123 hrs, 350°C
  - Four Zr-xNb alloys simultaneously (4mm x 15mm x 1mm) and two sacrificial Zr alloys
  - Indium cooling stage ; Temperature control with IR camera (Calibrated)





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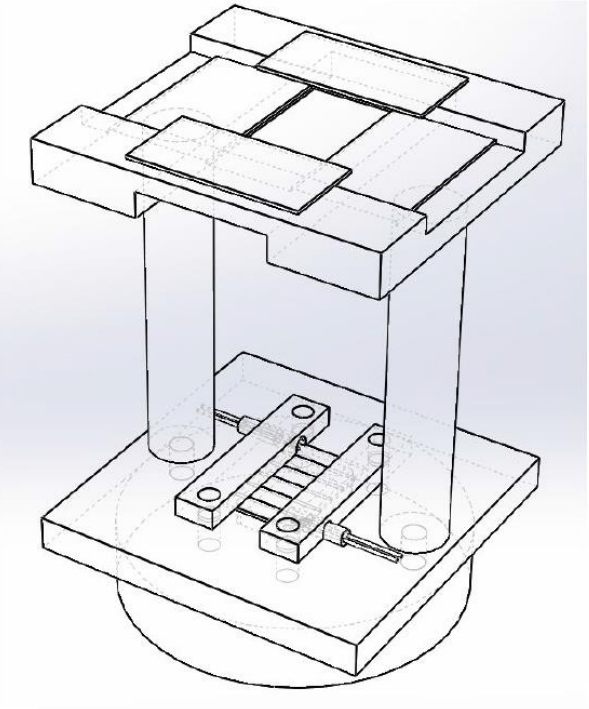

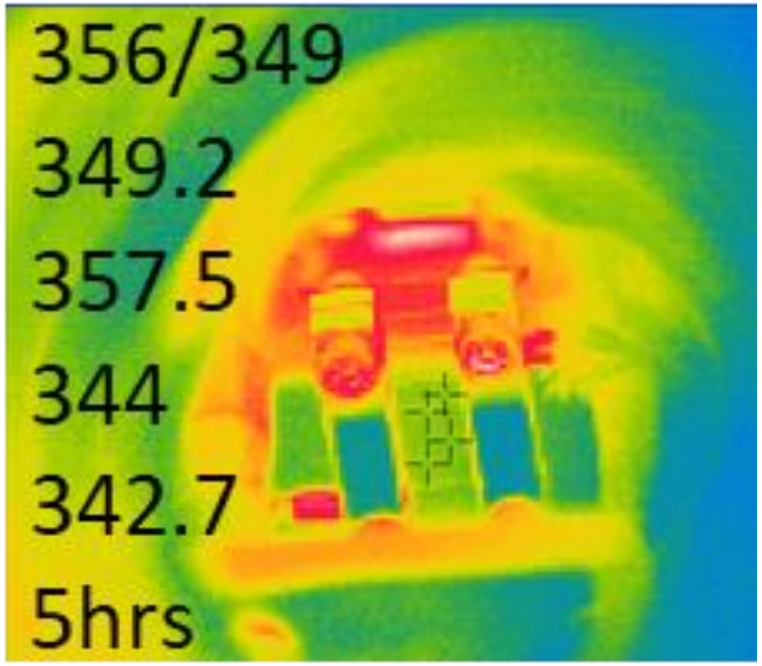

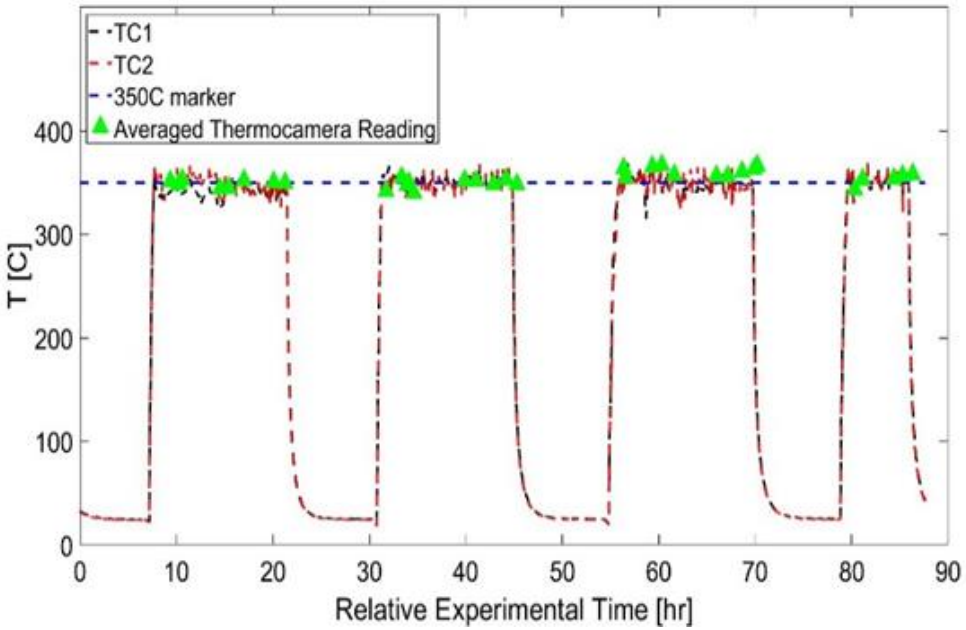

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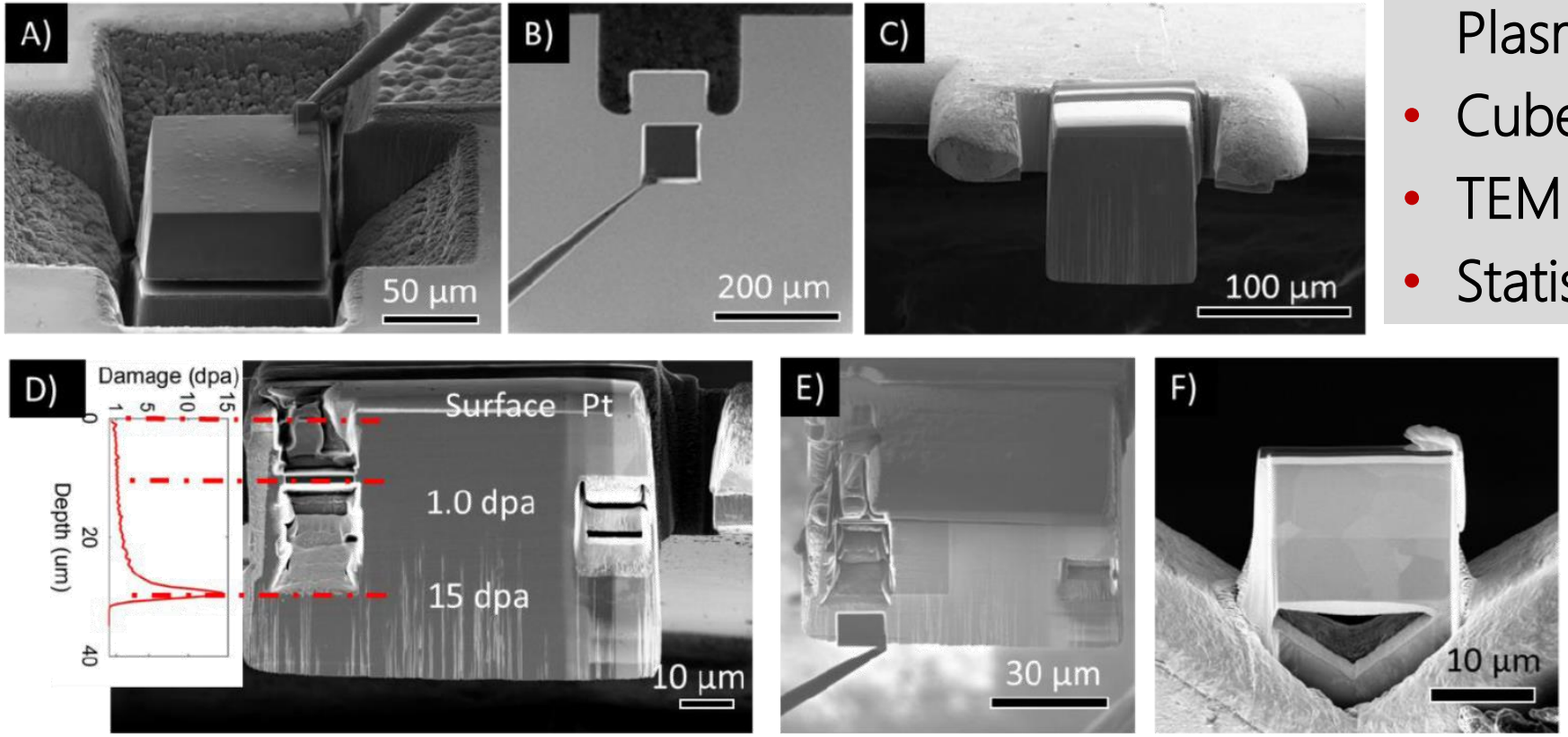


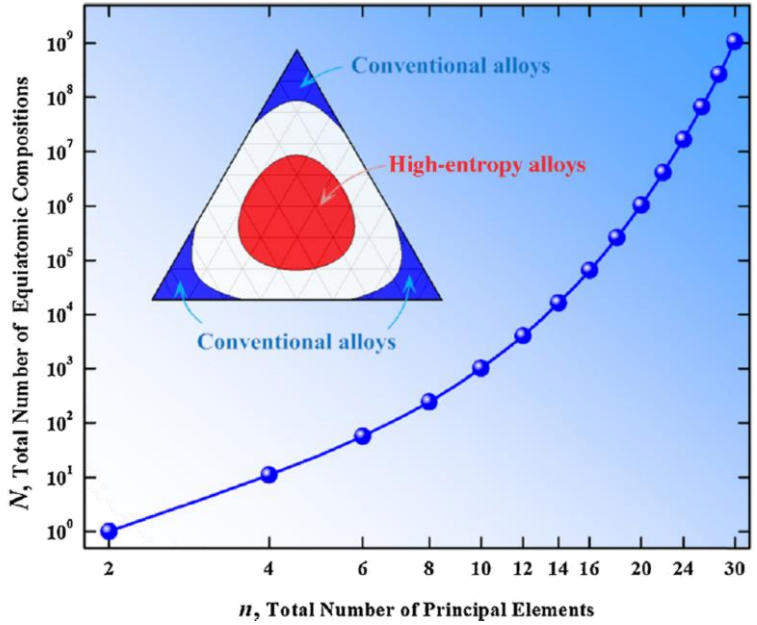
- Plasma FIB: FEI Helios G4 UX Plasma FIB/FE SEM
- Cube liftout
- TEM sample at constant dpa
- Statistical analysis

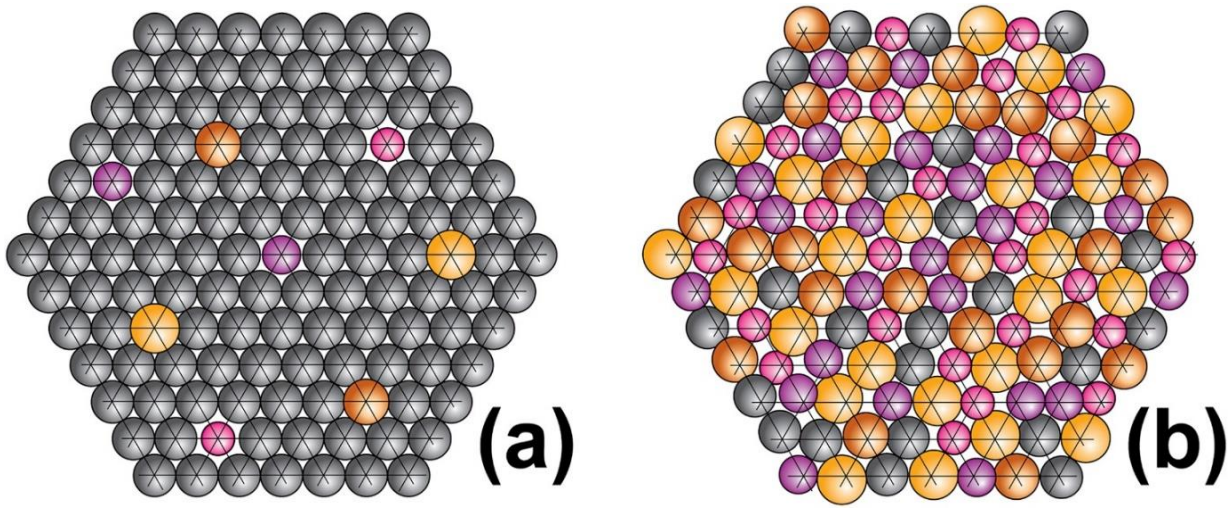
UW – IBL: Example 2 – High Throughput Irradiation of HEAs

What are High-Entropy Alloys?

Multiple primary elements with <35 at% in each

- **Limited modeling studies** and **experimental work** suggest radiation and corrosion resistance of CCA matrix
- HEAs are of interest as a **replacement base matrix** for high-temperature alloy design

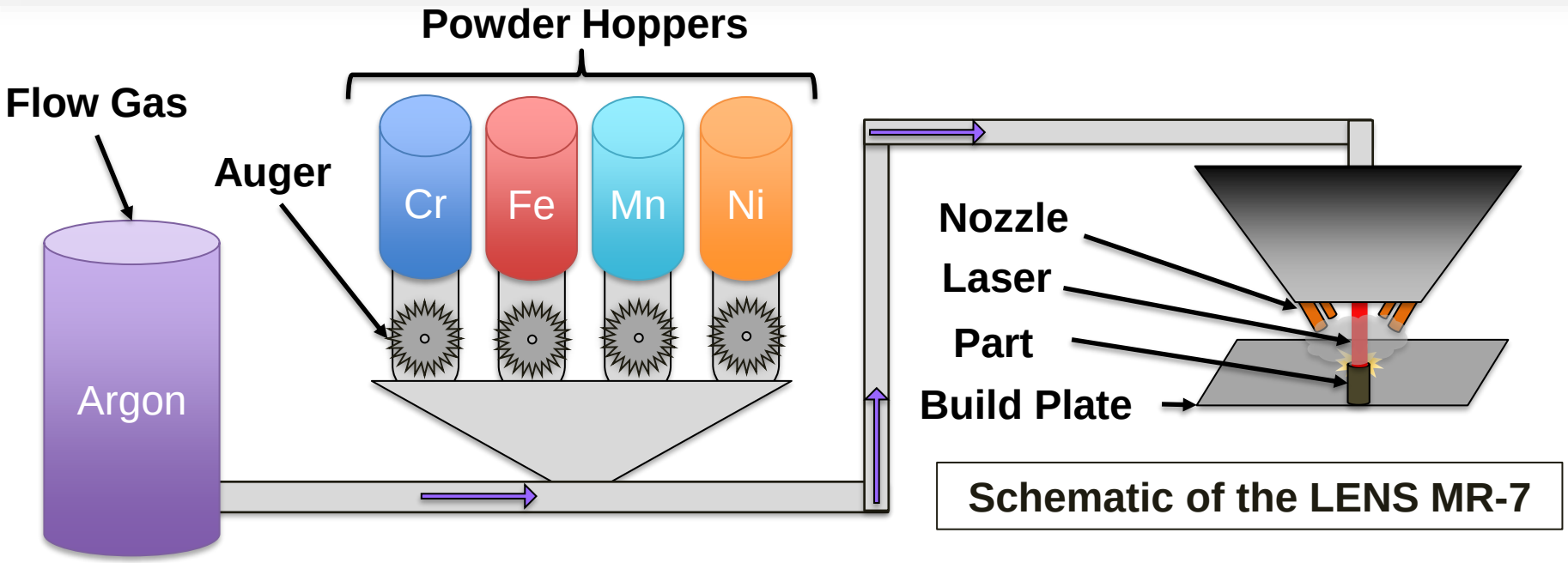




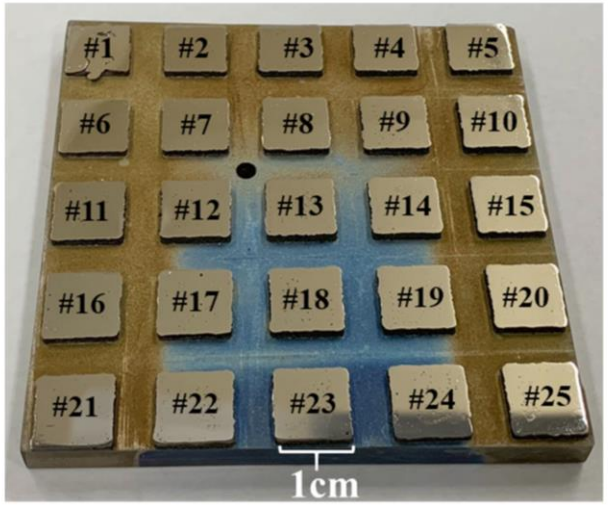
(a) Conventional Alloy, (b) Compositionally Complex Alloy[1]

Testing of one HEA at a time is not conducive with HEA research

UW – IBL: Example 2 – High Throughput Irradiation of HEAs



Schematic of the LENS MR-7



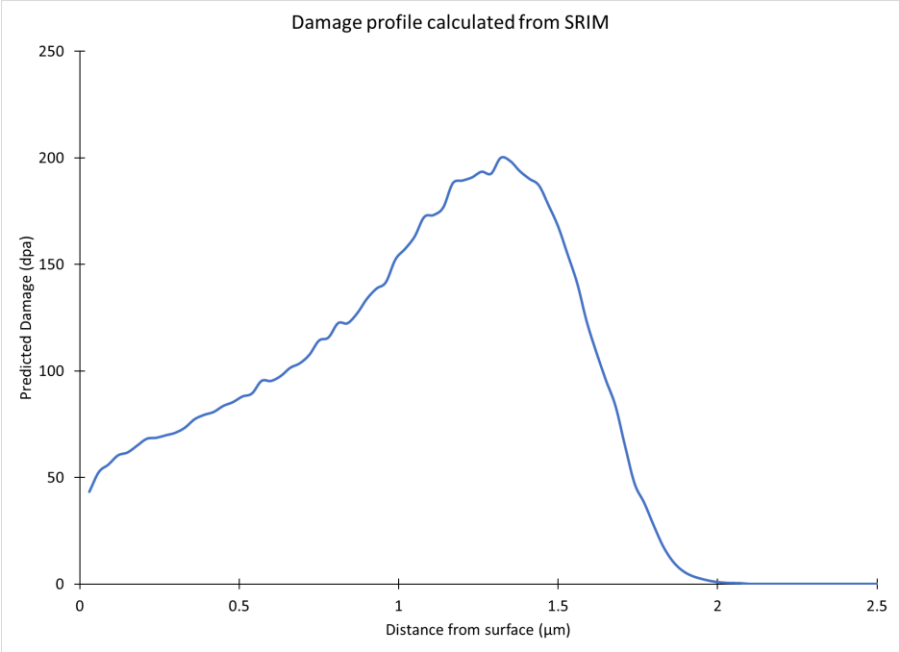
In collaboration with Prof. Dan Thoma at UW-Madison

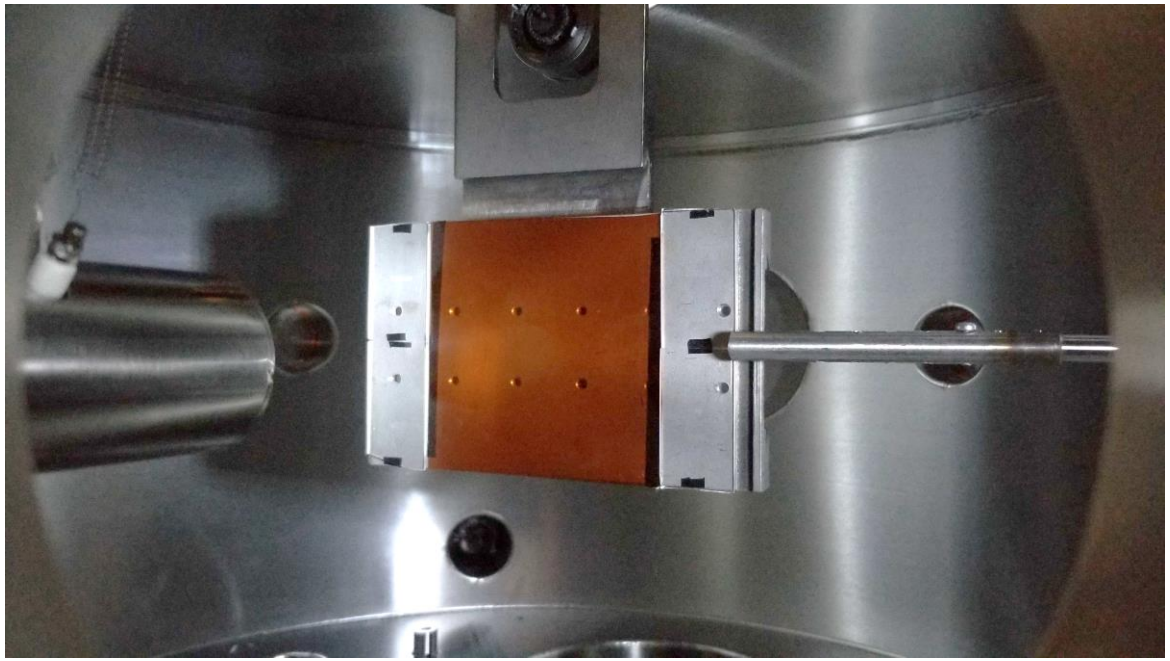
- Elemental powders are controlled independently
- Powders are delivered to print head by argon flow gas
- Laser down optic axis melts powders
- Focus on steels component FeCrMnNi for method validation

Moorehead, M., Bertsch, K., Niezgoda, M., Parkin, C., Elbakhshwan, M., Sridharan, K., Zhang, C,. Thoma, D., Couet, A. High-throughput synthesis of Mo-Nb-Ta-W high-entropy alloys via additive manufacturing. Mater. Des. 187, 108358 (2020).

UW – IBL: Example 2 – High Throughput Irradiation of HEAs

- 4 MeV Ni2+ ions
- High temperature: 500 ºC
- 200 dpa at peak damage



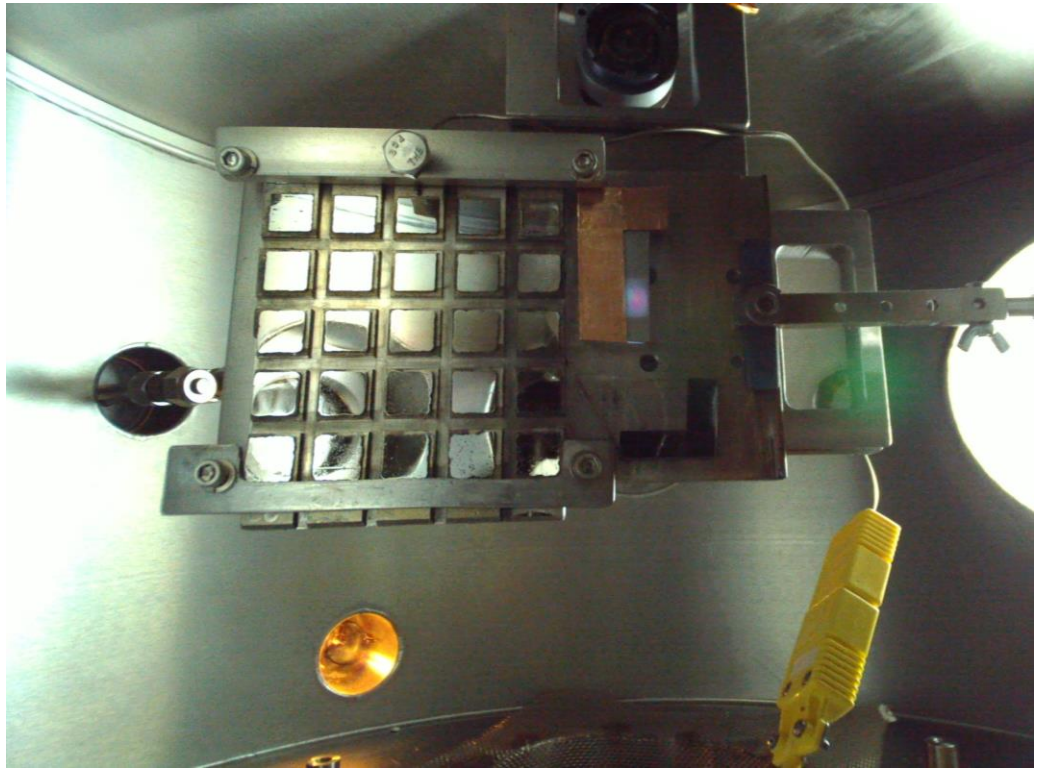


Challenge: Irradiate 25 samples in a limited period of time

Development of the high-throughput irradiation stage and chamber

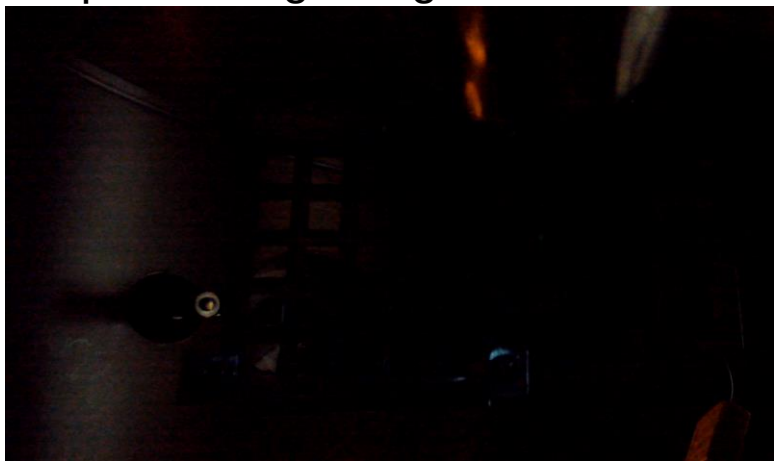
UW – IBL: Example 2 – High Throughput Irradiation of HEAs

- Necessity to shape the ion beam to irradiated only half of each sample → Comparison between irradiated and un-irradiated areas



HEA additively printed plate in chamber

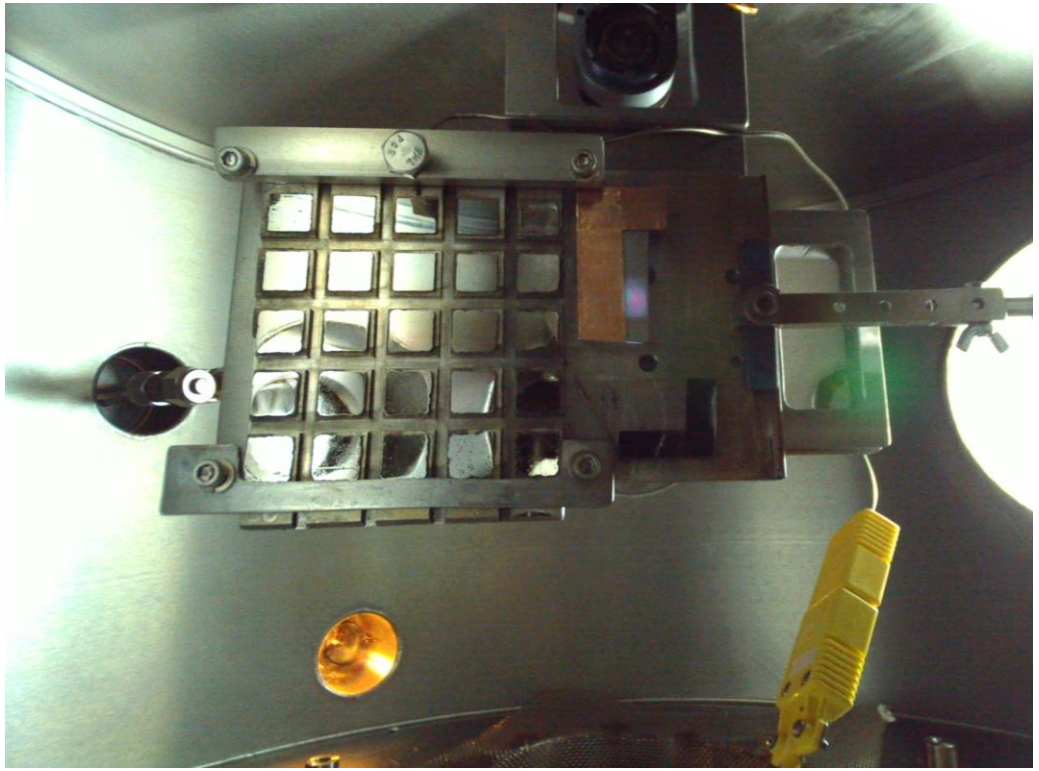
Ion beam positioning using electrostatic deflectors



Ion beam shaping (Ta apertures) and Automated Faraday cup insertion for current reading

UW – IBL: Example 2 – High Throughput Irradiation of HEAs

- Need to perfectly align three beams (ion beam, IR laser, pyrometer) on the same spot → Camera inside the irradiation chamber



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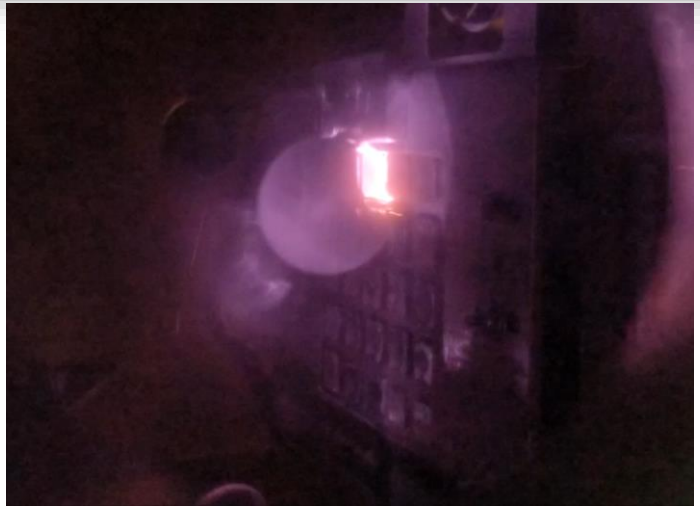
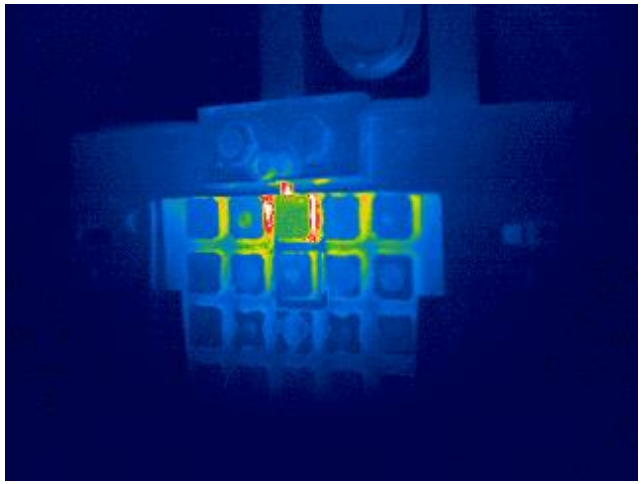
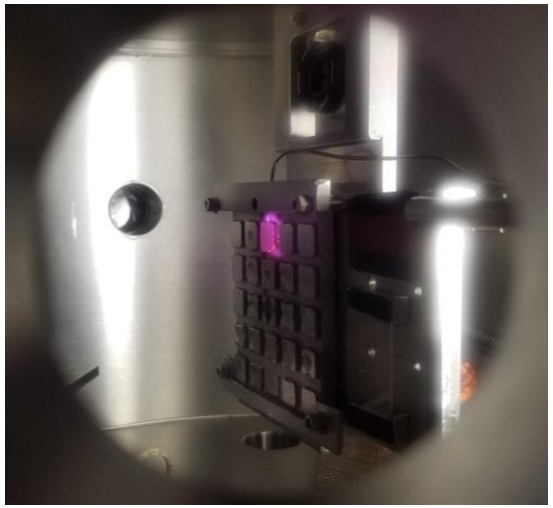


2kW laser turning off and still glowing red hot while the surrounding samples are cool

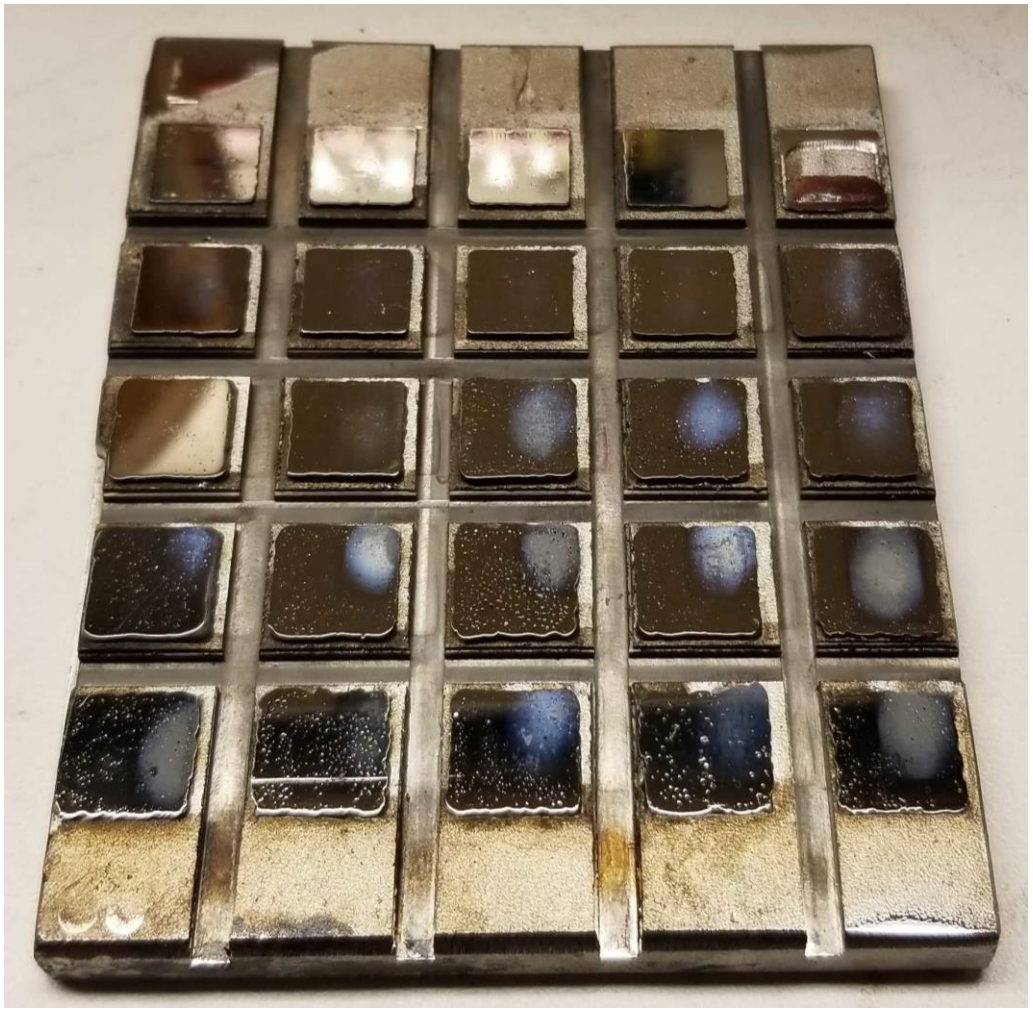




In-situ IR camera reading for temperature control

UW – IBL: Example 2 – High Throughput Irradiation of HEAs

Number of samples	25
Ions	4 MeV Ni^{2+}
Irradiation temperature	500 ± 20 ºC
Vacuum	10^{-7}-10^{-6} torrs
Peak damage	200 dpa
Damage rate	0.04 dpa.s^{-1}
Total irradiation time	32 hours



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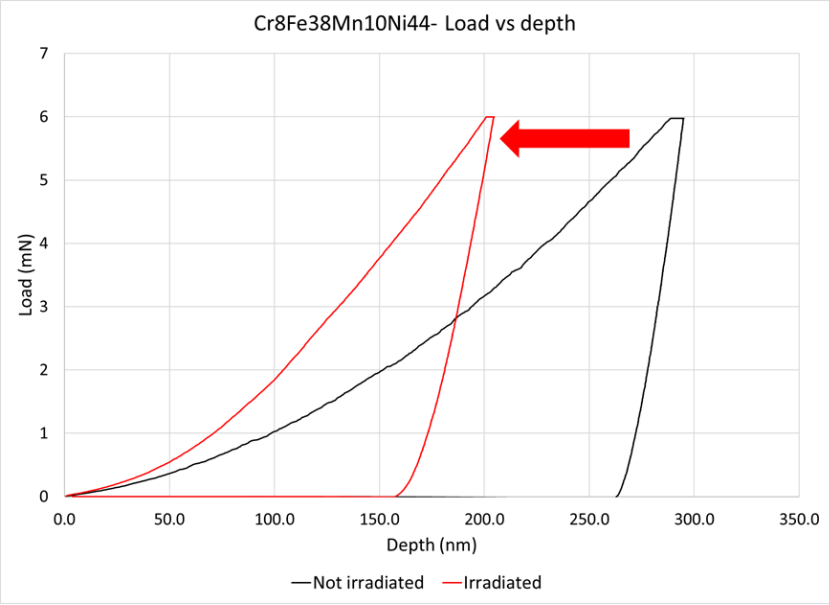
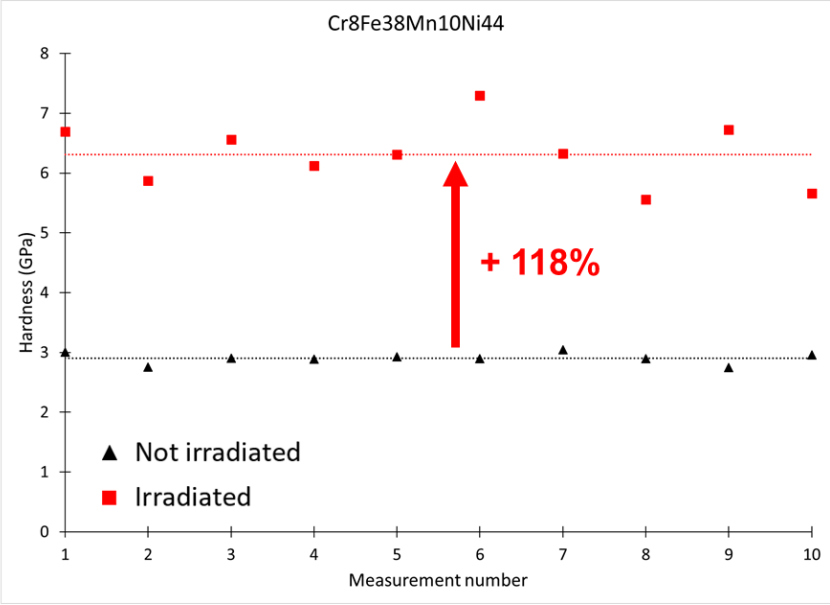
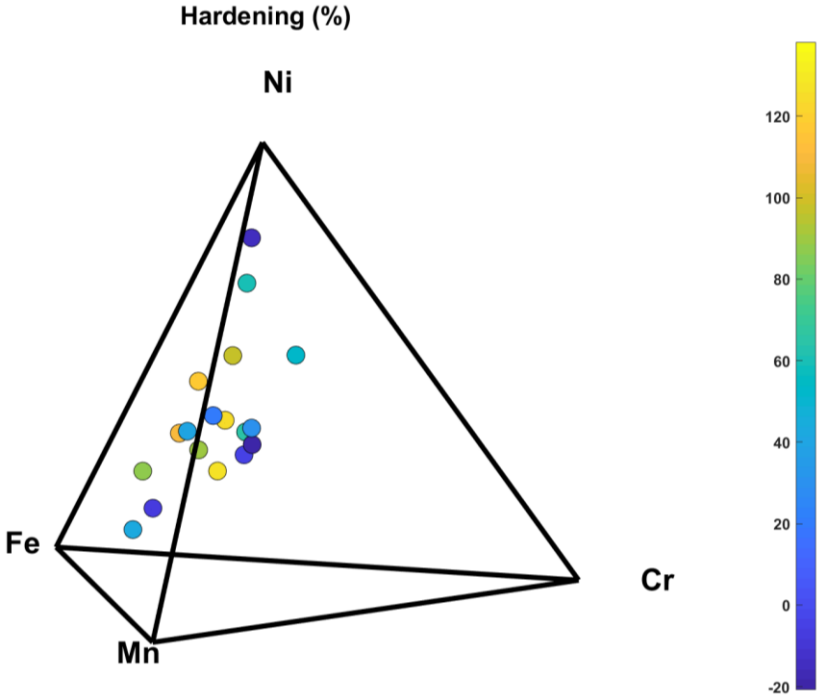


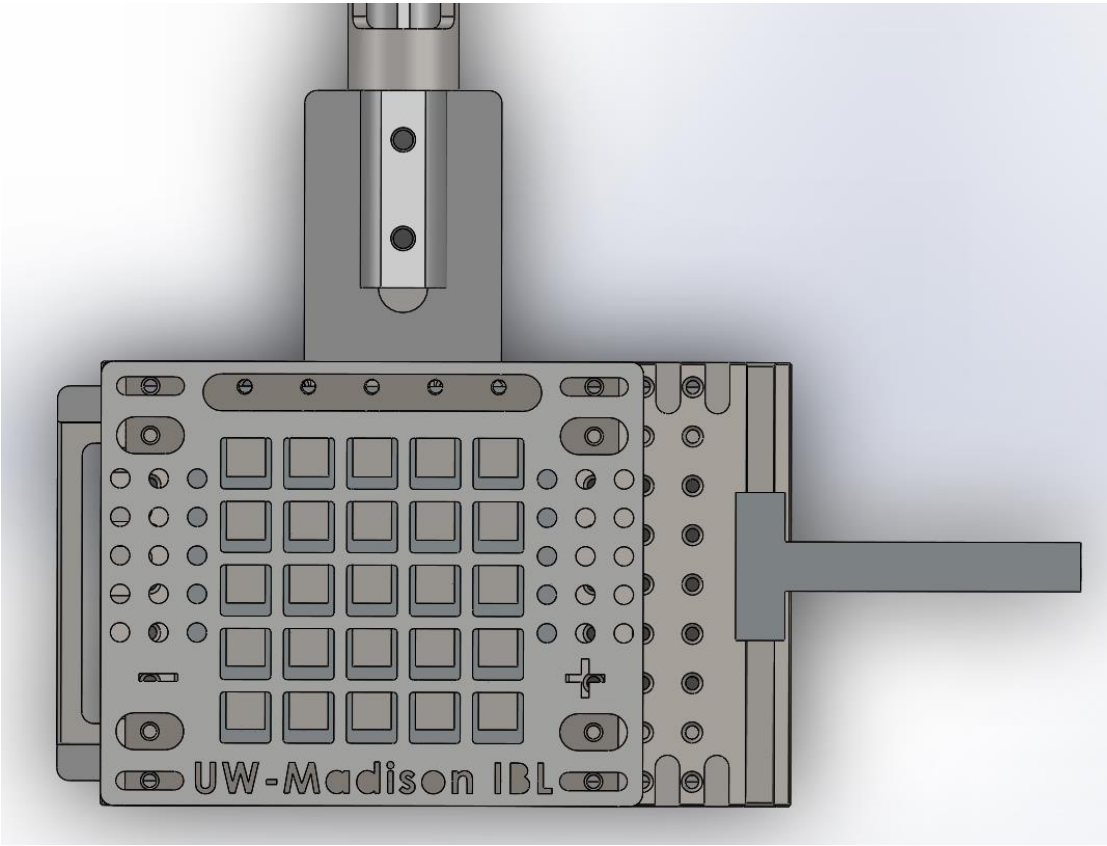


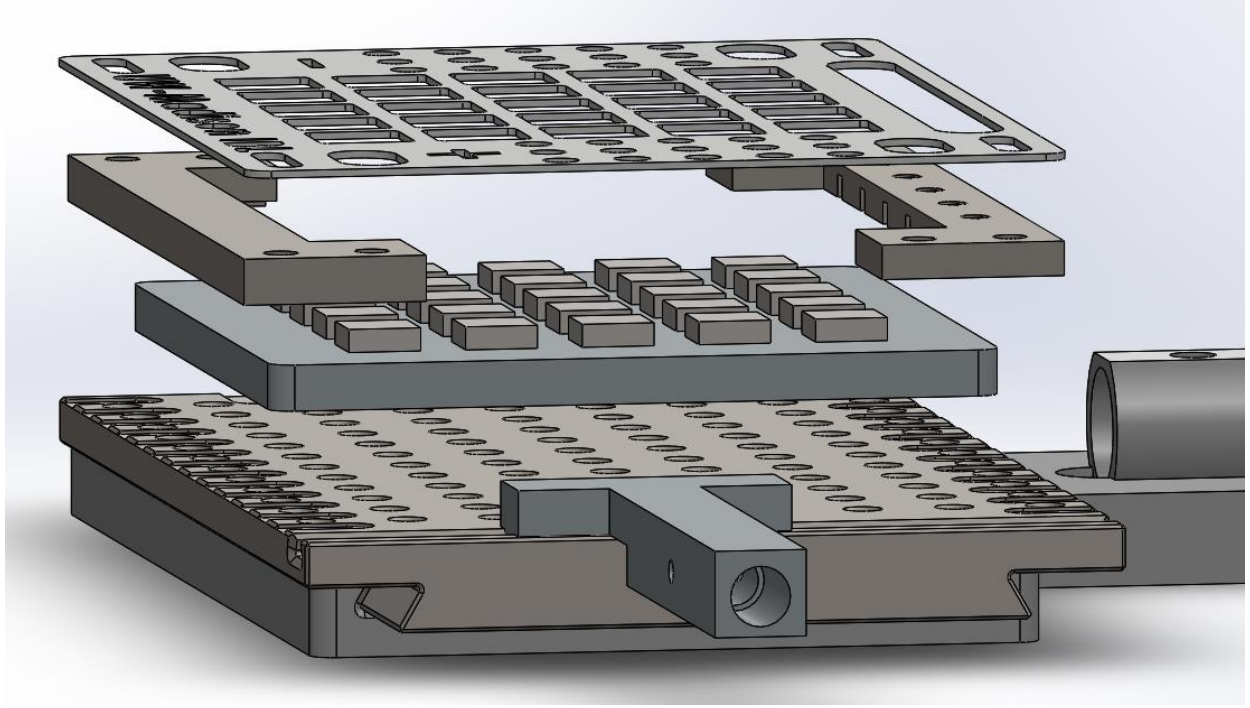


Irradiation induced hardening measured in most samples

UW – IBL: Example 2 – High Throughput Irradiation of HEAs

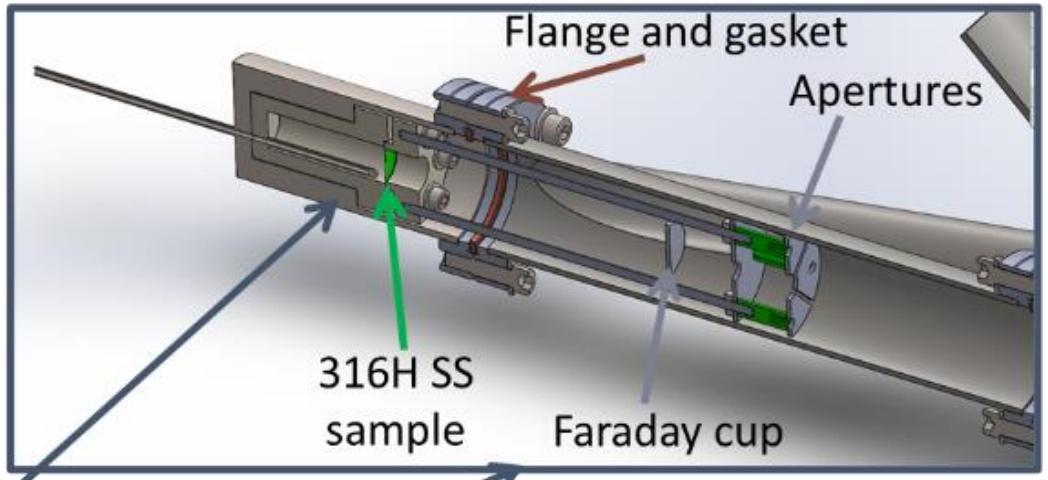
- Better control of unirradiated vs irradiated areas → new sample stage being manufactured
- Image recognition via in-situ camera for automated/automomous Labview control

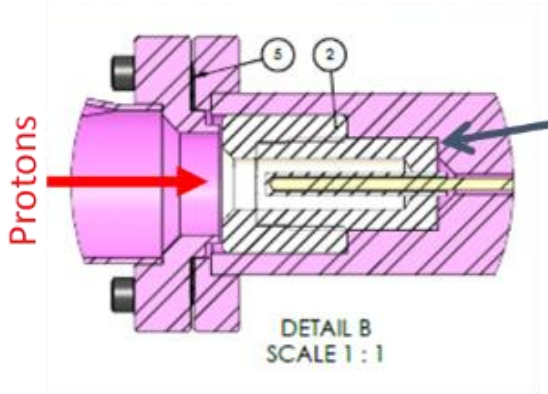


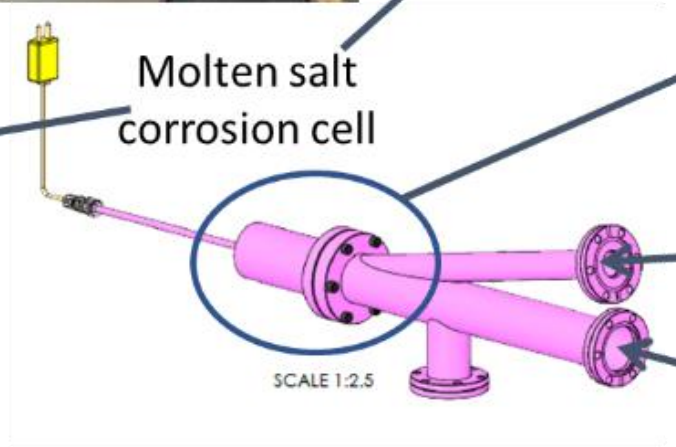


CAD drawing of new sample stage

UW – IBL: Example 3 – In-Situ Irradiation in Molten Salts (2022)







UW – IBL: Post Irradiation Examination (PIE) Capabilities

UW – IBL: Post Irradiation Examination (PIE) Capabilities

- JEOL 6610 SEM with Energy Dispersive Spectroscopy (EDS) and Electron Backscatter Diffraction (EBSD) capabilities

- Rad sample certified (outside of laboratory): X-ray Diffraction.

UW – IBL: Post Irradiation Examination (PIE) Capabilities

- Parallel polisher
- Low speed saw
- Electro-polisher
- Ion mill
- High accuracy balance

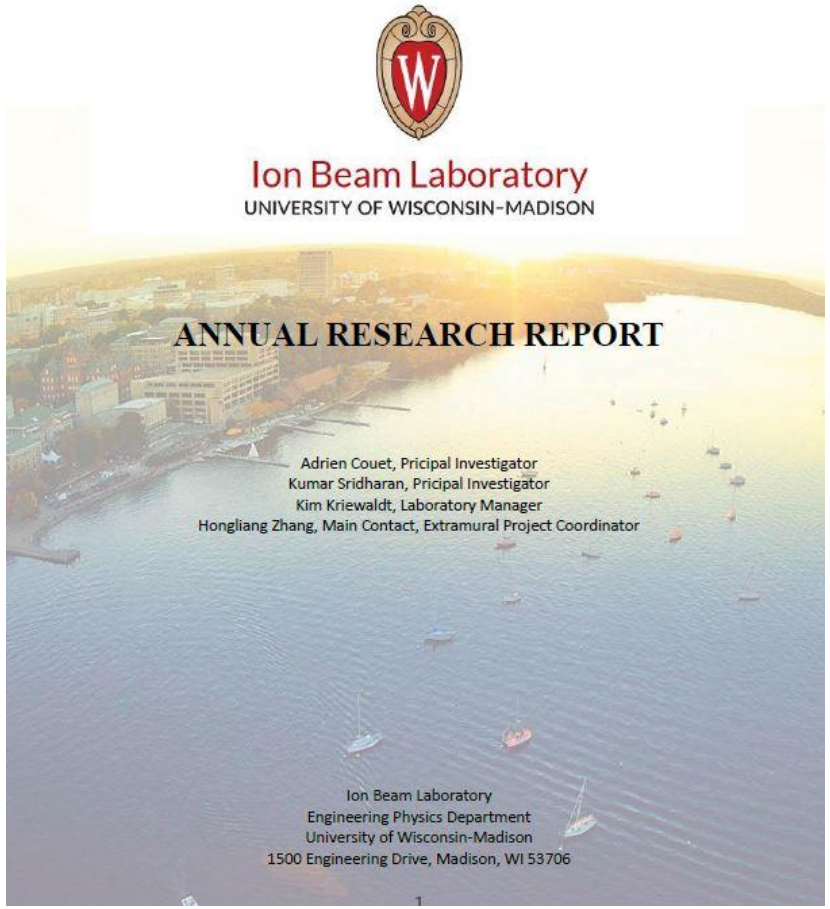
UW – IBL: Post Irradiation Examination (PIE) Capabilities

FEI Helios G4
UX Plasma
FIB/FE SEM

FEI Titan Cs-corrected scanning transmission electron microscope

Hysitron TI 950
TriboIndenter

UW – IBL: 2018-2019 Annual Research Report



- 2020-2021 report to be released
- Possible access via Rapid Turnaround Experiments – 3 calls/year (https://nsuf.inl.gov/Page/rte)
- Details, rates, and application forms:
  - https://ibl.ep.wisc.edu/
- We are happy to upgrade and customize equipment according to users' needs
- Contacts:

Adrien Couet (couet@wisc.edu)

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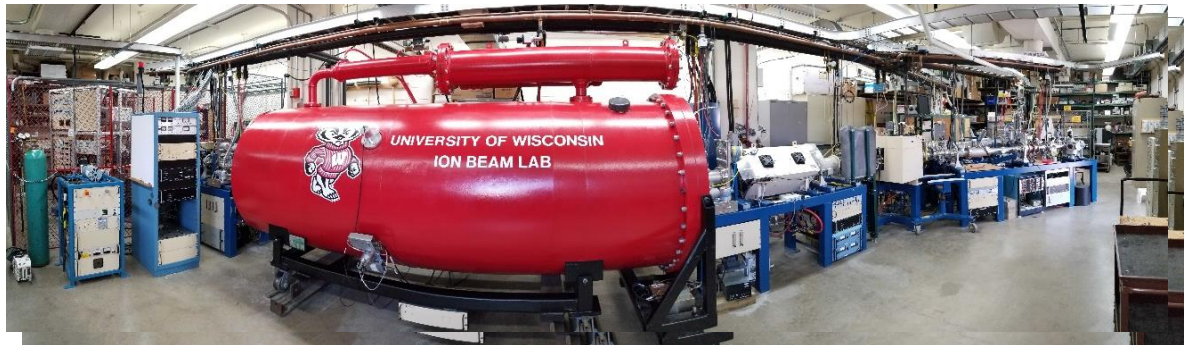

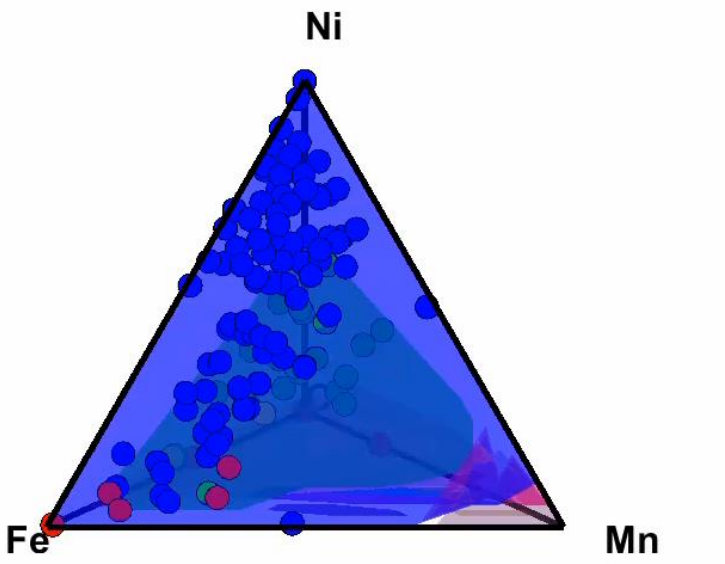

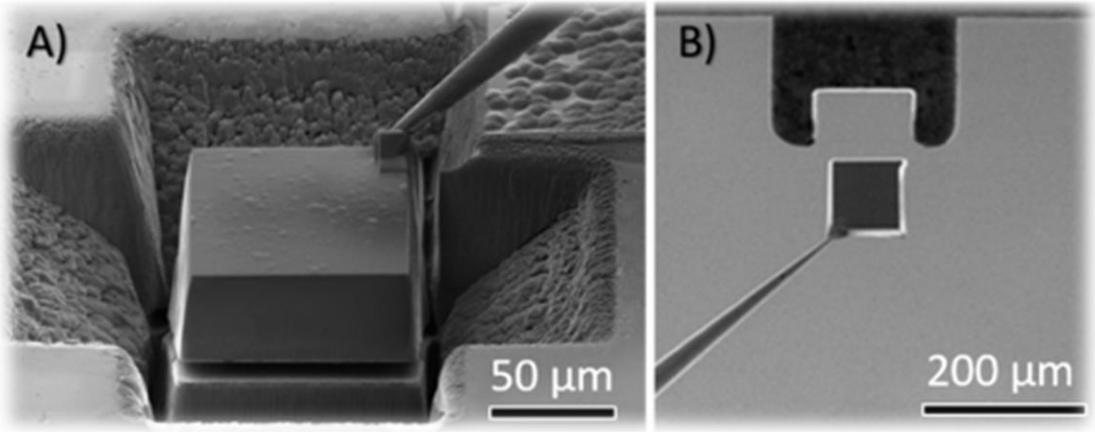

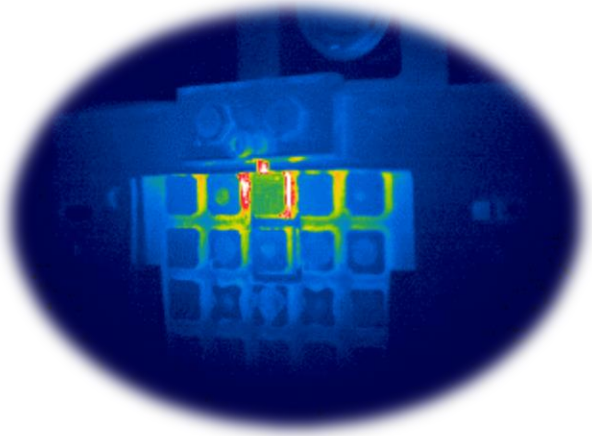

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