MADCOR

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

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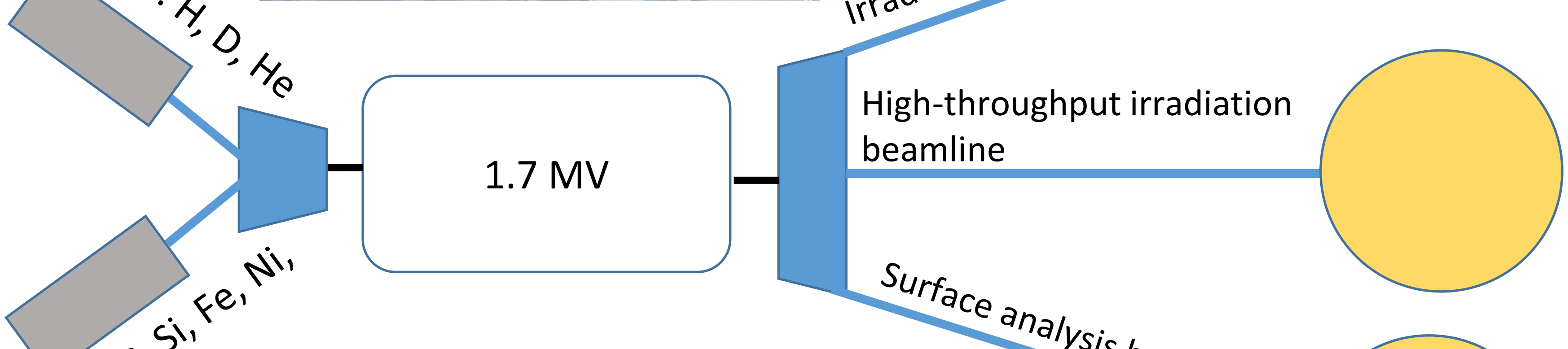
UW – Ion Beam Laboratory (IBL)

- 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)



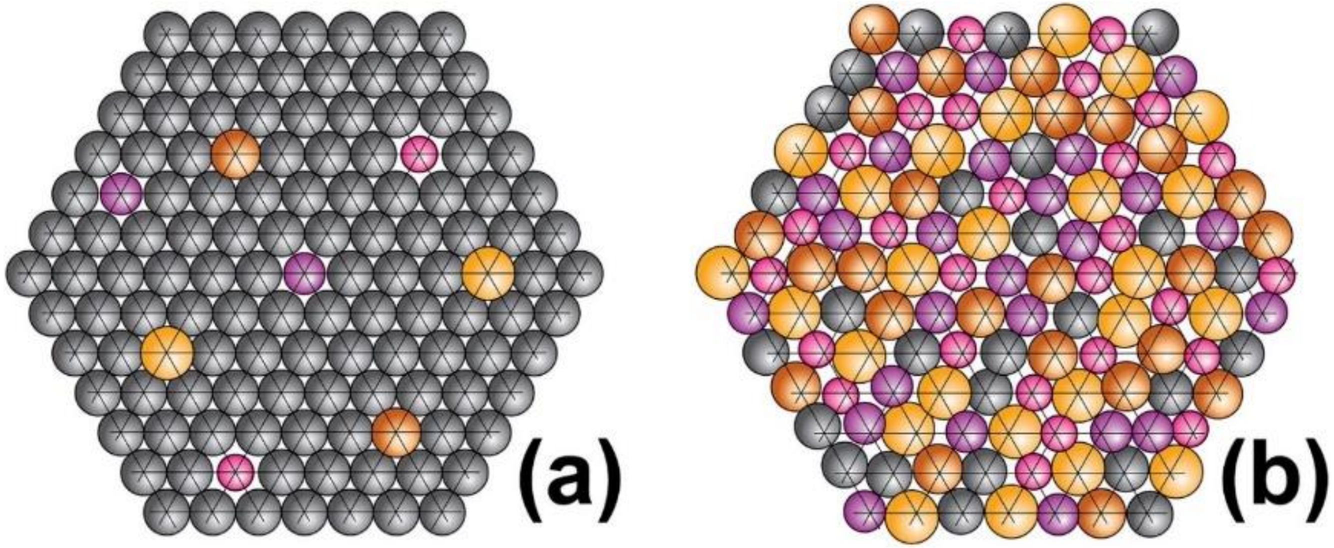
- Water chiller and liquid metal cooling for precise temperature control,
- Heating cartridges (up to 1000C)

- **New installation of high-throughput irradiation beamline (2021/2022)**
- Large chamber with load lock
- 3D moving stage for high-throughput irradiation
- Controlled laser heating from front

- **New installation of ion beam analysis beamline (2022/2023)**
- Rutherford backscattering spectrometry (RBS)
- Particle-induced X-ray emission (PIXE).

High-Entropy Alloy (HEA) Overview

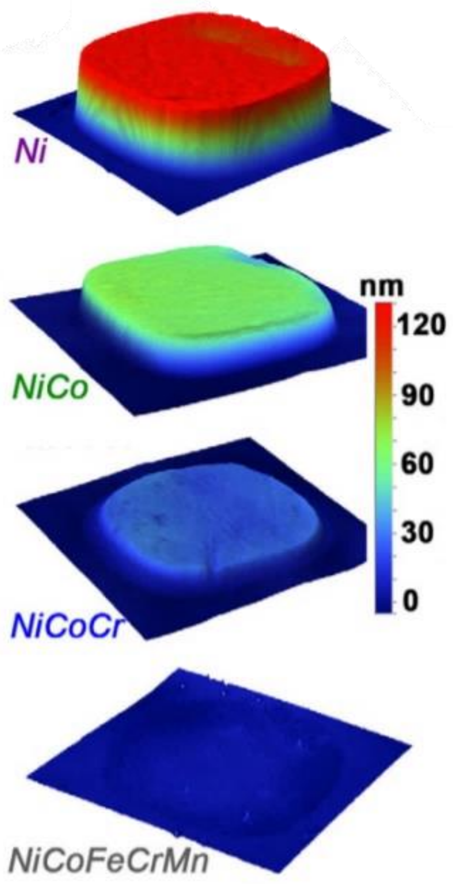
- Develop robust beam-intercepting devices (targets, beam windows, beam dumps, collimators…) to maximize physics benefits of future experiments
- Better assessment of current materials used in accelerators
- Develop novel materials to improve lifetime of components under extreme conditions → new materials specifically addressing the requirements of future target facilities.



(a) Conventional alloy, (b) High-entropy alloy (Miracle, Senkov | 2017)

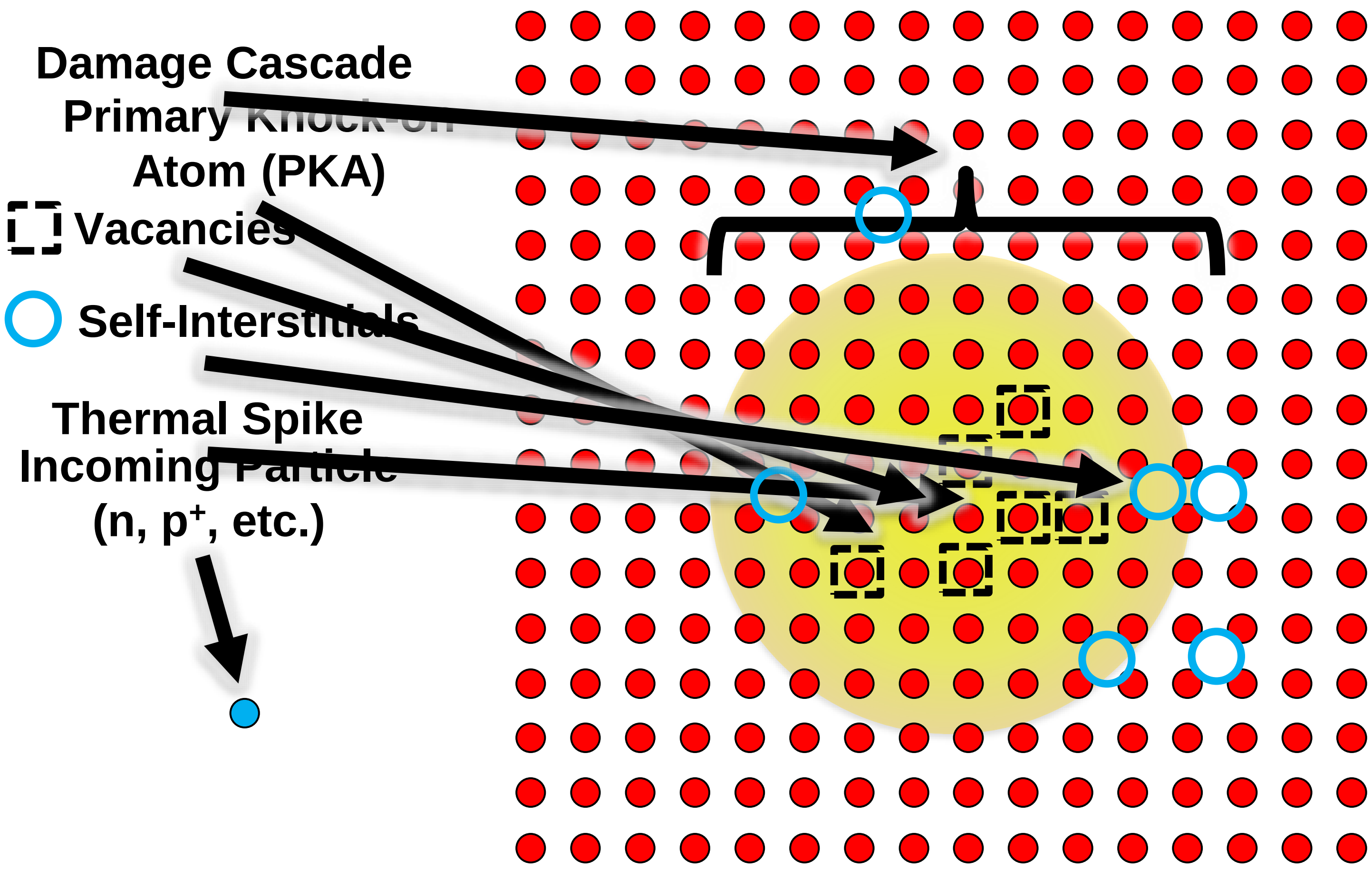
High-Entropy Alloy (HEA) Overview

- High-entropy alloys (HEAs) are alloys with multiple principal elements
- Typically, no element >35 at%
- Usually defined as being primarily a solid-solution matrix
- Promising properties have been observed:
  - High-temperature strength
  - High specific strength
  - Enhanced radiation tolerance



Swelling of increasingly complex alloys under ion irradiation (Jin | 2016)

Radiation Damage in Metals



Primary Collision → Thermal Spike → Quench → t⟶∞

Long-Term Defect Evolution:

Quench:

Thermal Spike:

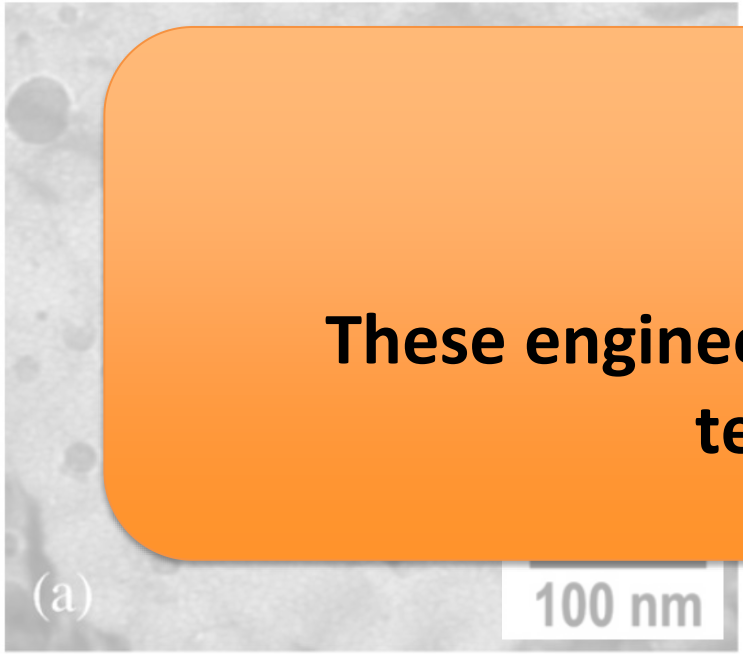
Primary Collision:

As time progresses and more damage cascades are produced, the population of point defects increases. Over timescales (minutes, hours, days), point defects can diffuse towards free surfaces or interfaces (sinks) to be annihilated. Dissimilar point defects can annihilate with one another. Alternatively, similar point defects can cluster together, giving rise to voids and dislocations.

Radiation Tolerant Microstructures

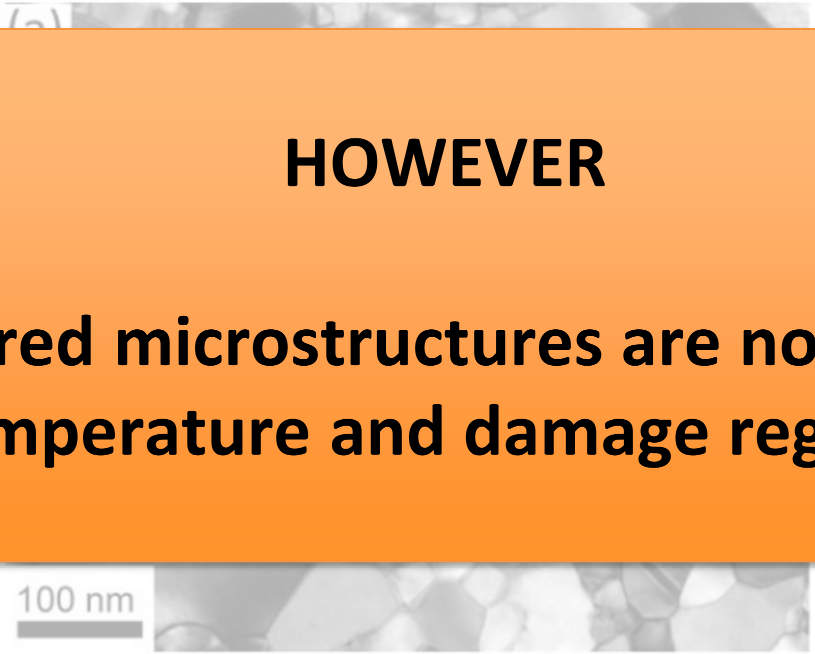
Typical approach to improving radiation tolerance: increase sink density

Oxide-dispersion strengthened (ODS) alloys



(Allen | 2008)

Nanocrystalline alloys



(Barr | 2018)

Cold-worked alloys

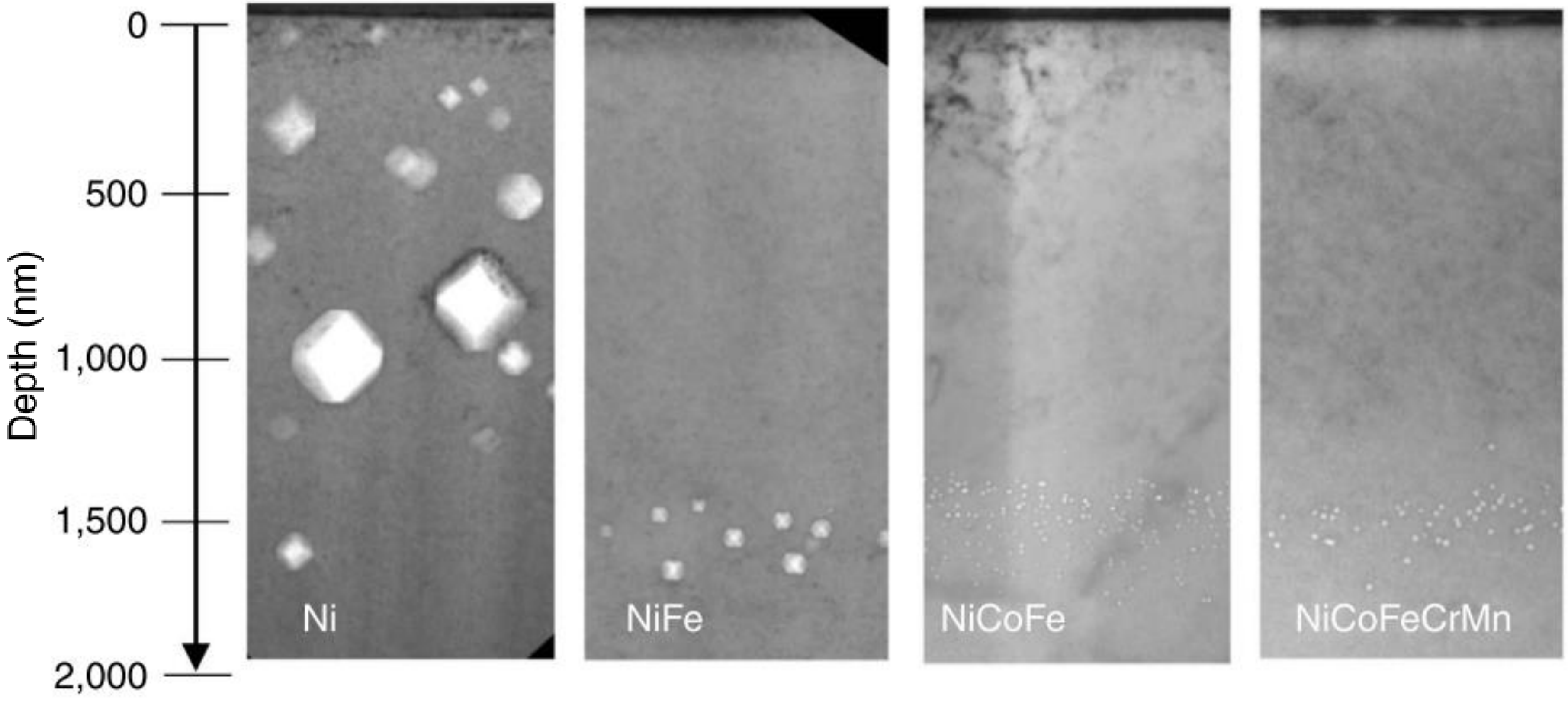
(Alshater | 2018)

HOWEVER

These engineered microstructures are not stable under all temperature and damage regimes

Radiation Tolerance in HEAs

Both modeling and experimentation have shown HEAs can exhibit enhanced radiation tolerance <u>in the matrix</u>



3 MeV Ni^{+} ions
5×10^{16} cm^{-2}

T = 500 °C

(Lu | 2016)

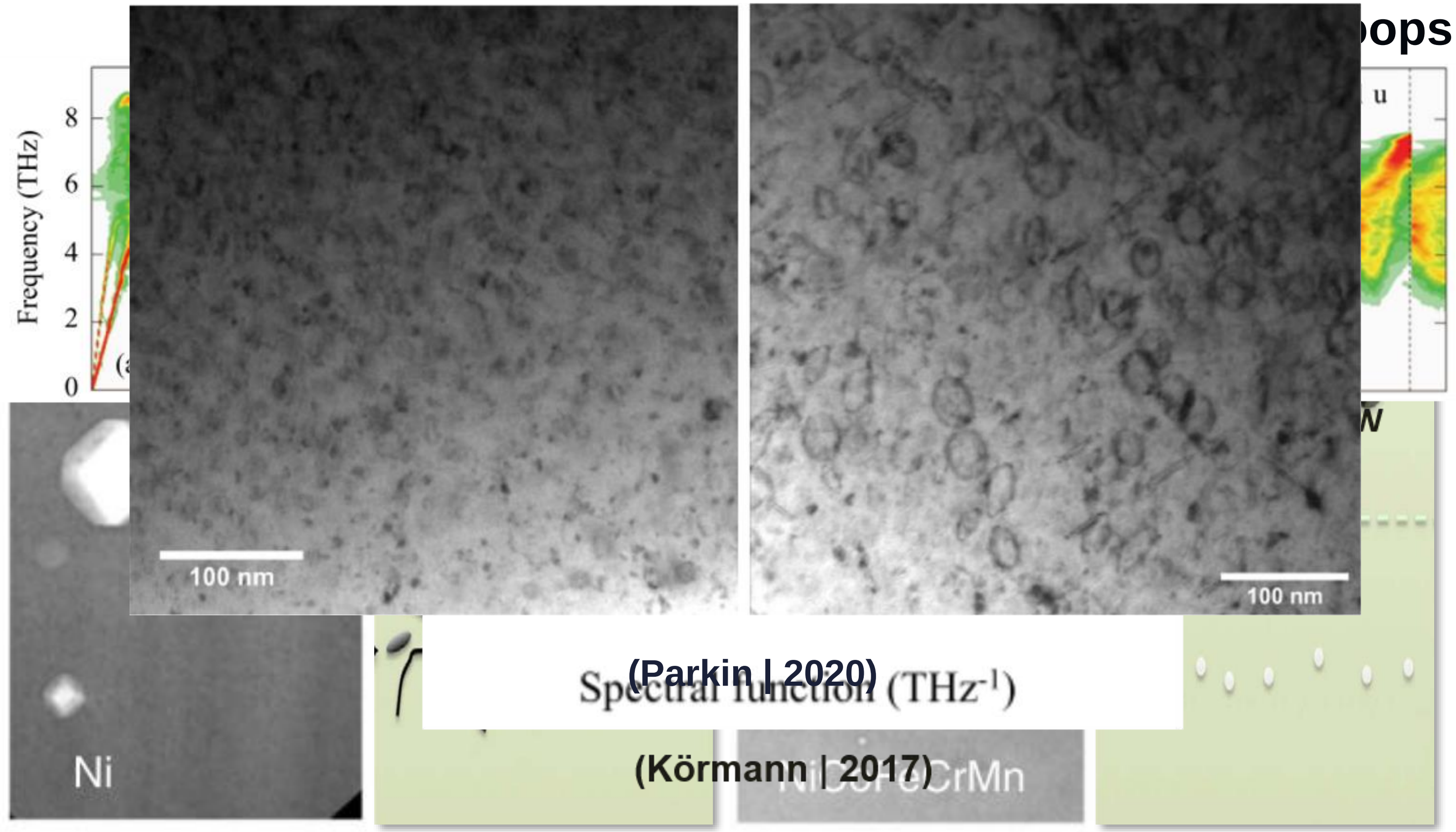
Radiation Tolerance in HEAs

Proposed mechanisms:

- Greater phonon scattering
- Sluggish diffusion of:
  - Atoms and point defects
  - Interstitial loops

⋮

- More trapping sites
- Broad migration energies
- And more!



(Parkin | 2020)

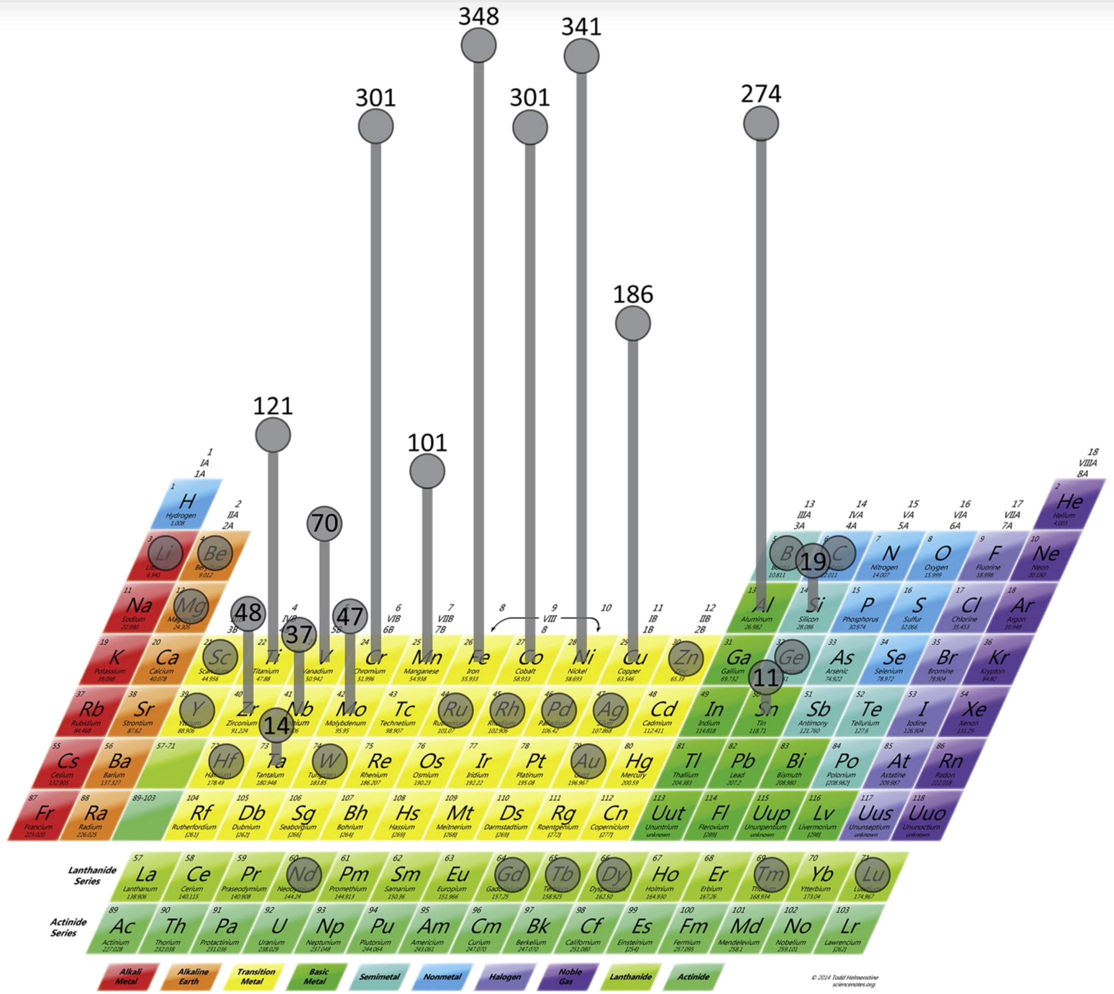
(Körmann | 2017)

(Lu | 2016)

Which HEA Composition to Use For Accelerators?

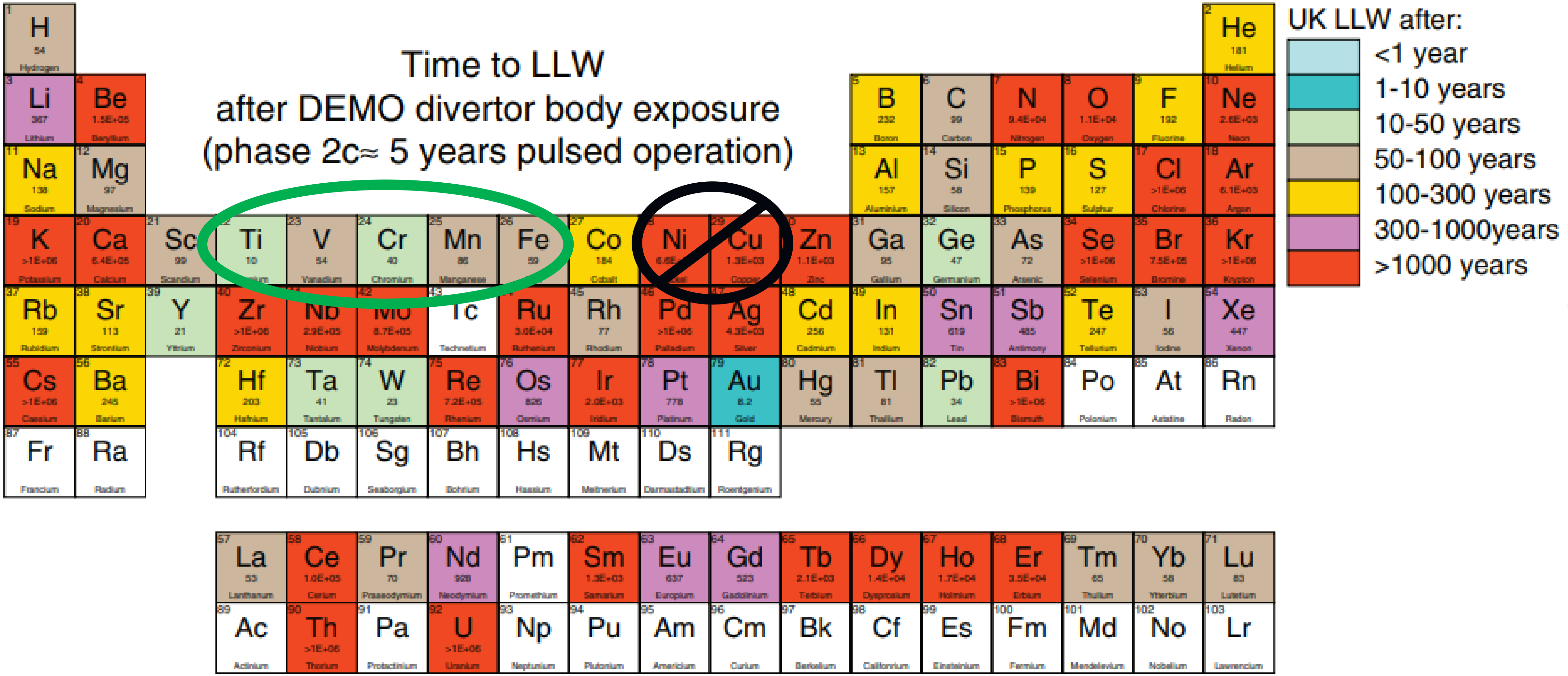
- Most commonly studied HEAs:
  - CoCrFeMnNi (Cantor alloy)
  - $Al_xCoCrFeNi$
  - $Al_xCoCrCuFeNi$

- Elemental selection considerations:
  - Primarily a single-phase alloy
    - Better ductility and manufacturability
  - Light-weight elements / low density
    - Reduced interaction with protons
  - Minimal activation under proton beam
    - Safer/easier to handle after use



Most commonly used elements in HEAs from literature (Miracle | 2017)

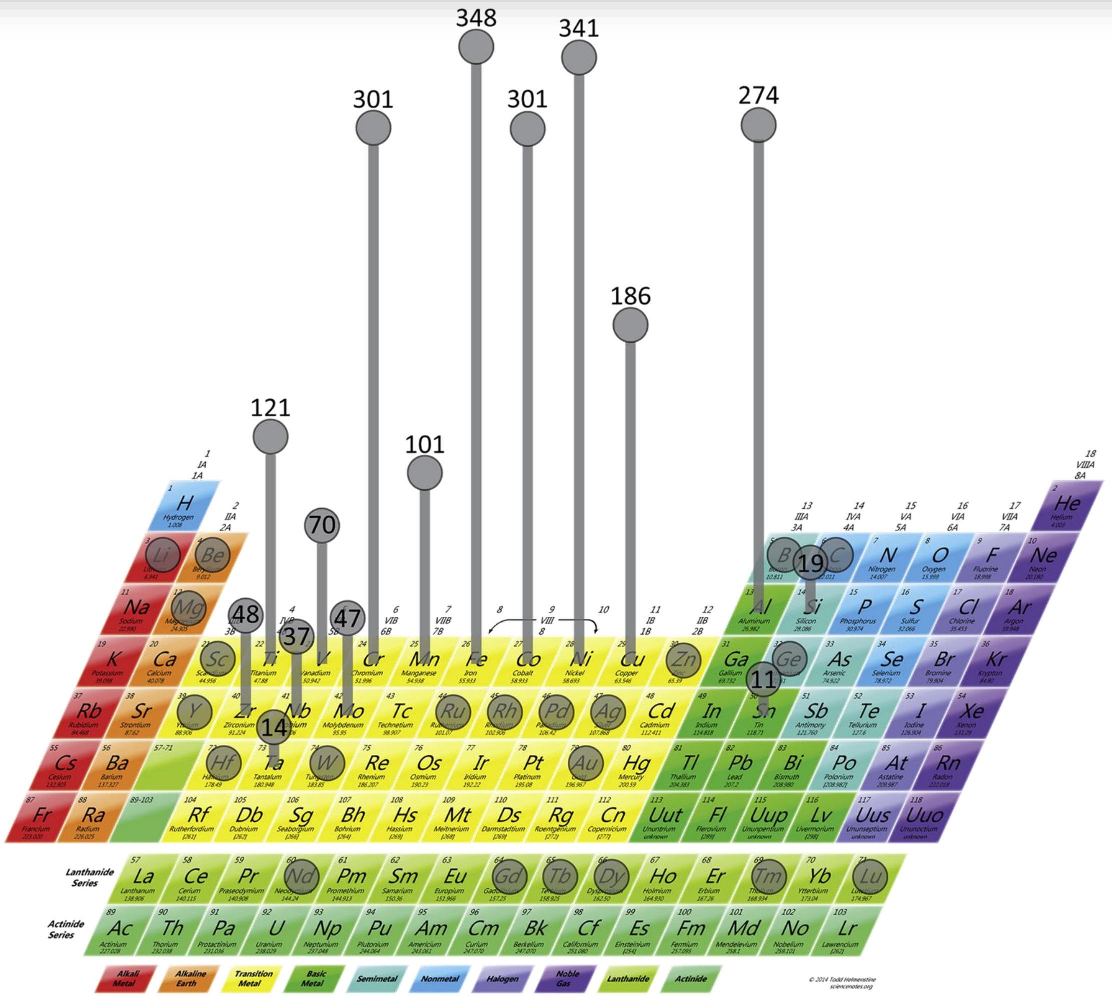
Which HEA Composition to Use For Accelerators?



Predicted cooldown time for elements used in DEMO reactor divertor environment (Gilbert | 2019)

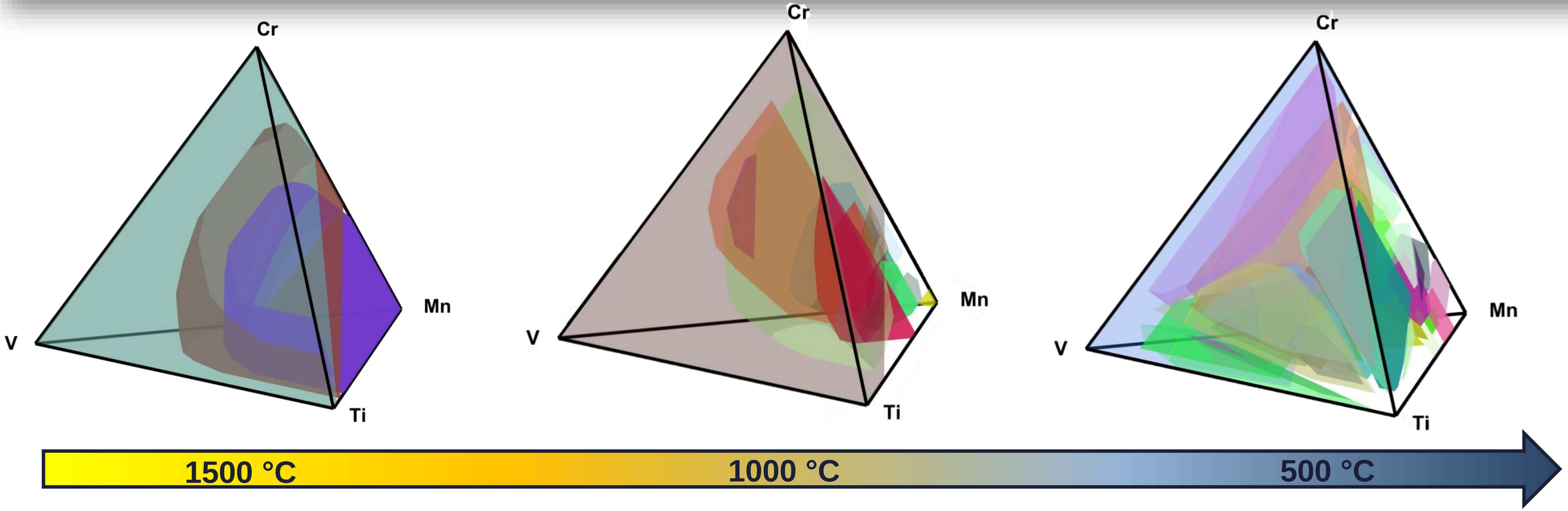
Which HEA Composition to Use For Accelerators?

- Most commonly studied HEAs:
  - ~~CoCrFeMnNi (Cantor alloy)~~
  - ~~$Al_xCoCrFeNi$~~
  - ~~$Al_xCoCrCuFeNi$~~
- New HEA using Cr, ~~Fe~~, Mn, Ti, V
  - Fe forms intermetallic compounds near the equimolar ratio with Cr, Ti, and V



Most commonly used elements in HEAs from literature (Miracle | 2017)

Which HEA Composition to Use: CALPHAD



Takeaways:

Mn reduces the melting point substantially

V greatly promotes the formation of a single BCC phase

At lower temperatures, high **Ti** and **Mn** lead to the formation of many secondary phases

Starting from Literature

Contents lists available at ScienceDirect

Scripta Materialia

journal homepage: www.elsevier.com/locate/scriptamat

ELSEVIER

Towards V-based high-entropy alloys for nuclear fusion applications

P.J. Barron[a,*], A.W. Carruthers[a], J.W. Fellowes[a], N.G. Jones[b], H. Dawson[c], E.J. Pickering[a]

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[c] Culham Centre for Fusion Energy, Abingdon OX14 3DB, UK

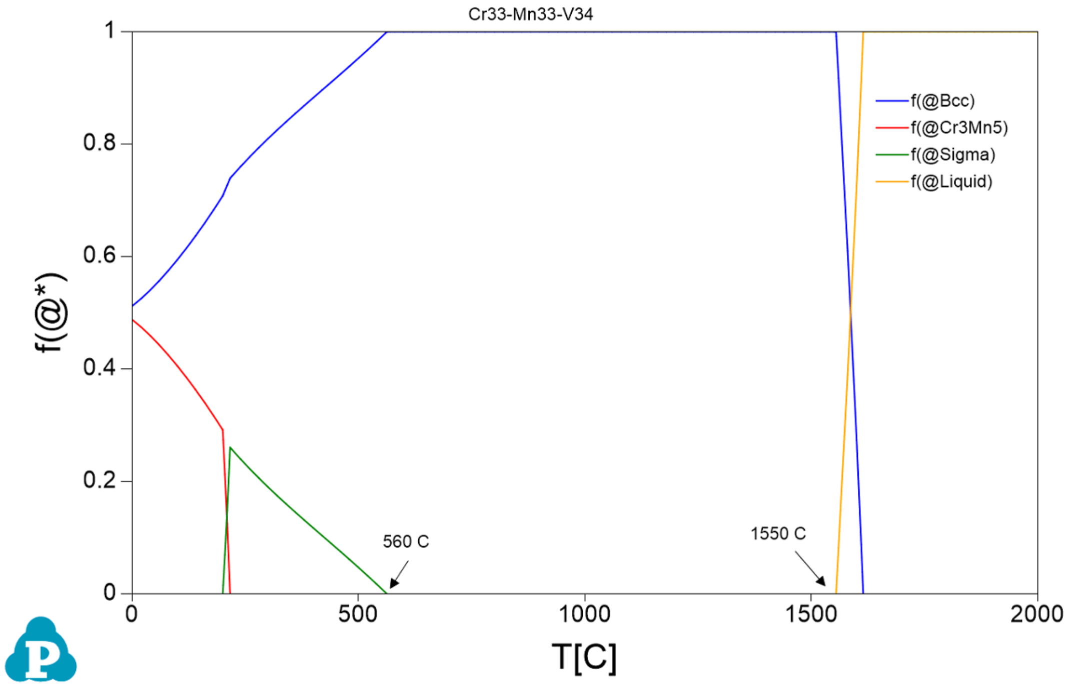
Check for updates

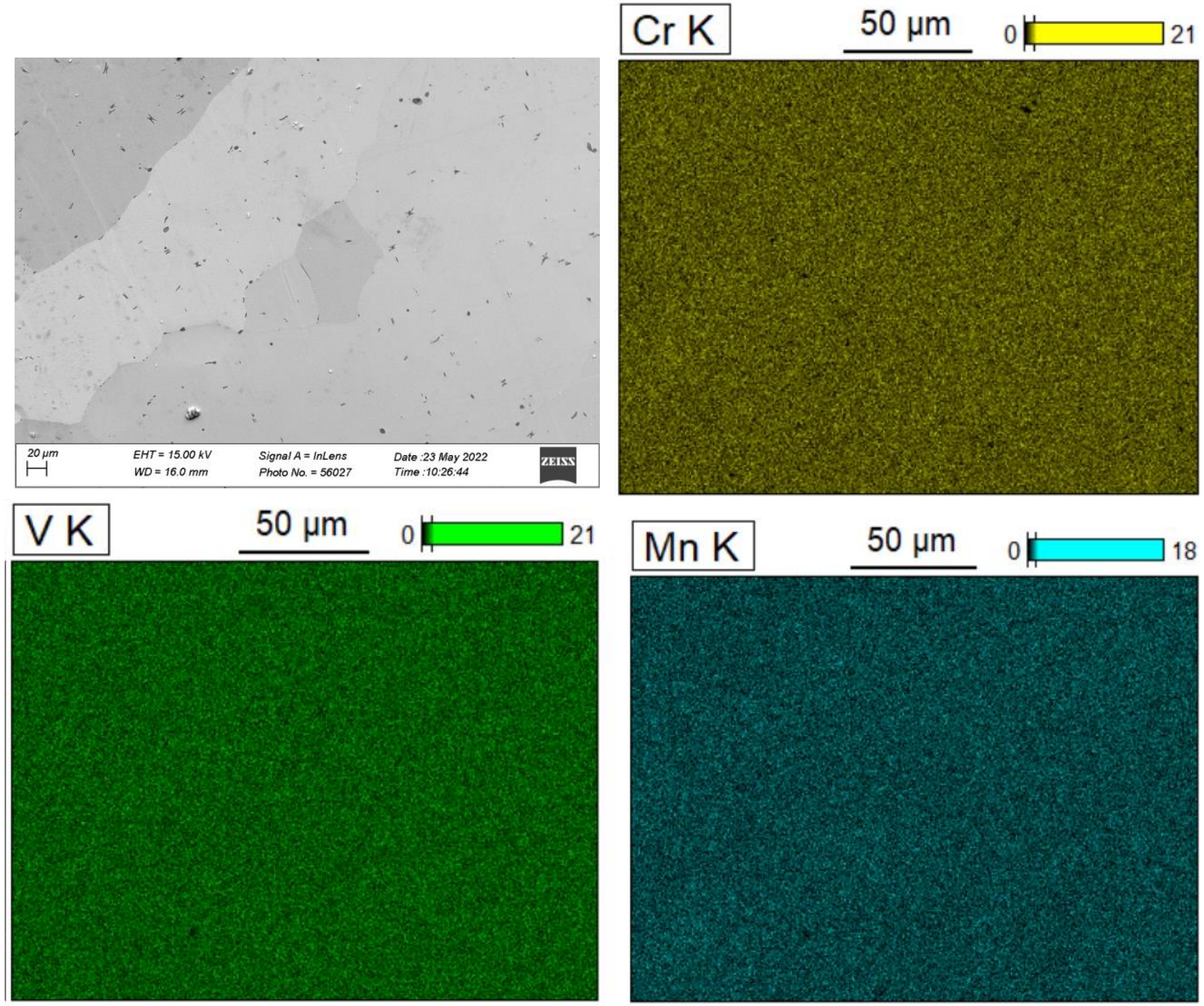
- Equimolar Cr-Mn-V shown to be single-phase BCC
- **Ti additions reduce concentration of interstitial impurities** by forming Ti-(C,O,N)
- Concerns **Ti concentrations above ~8% will promote Laves phase formation**
- 1200-°C, 100-hour heat treatment followed by water cooling to homogenize

$Cr_{33}Mn_{33}V_{33}$

$Cr_{33}Mn_{33}V_{33}$

- Broad BCC phase field spanning ~1000 °C
- At 500 °C, the alloy is still >90% BCC

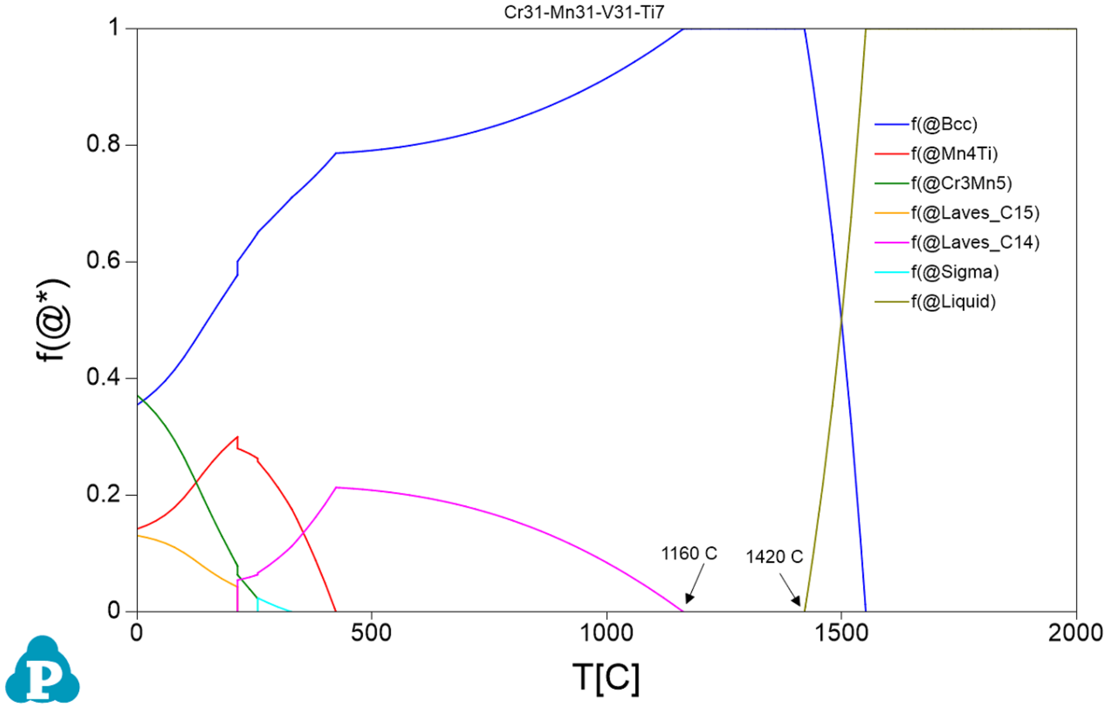


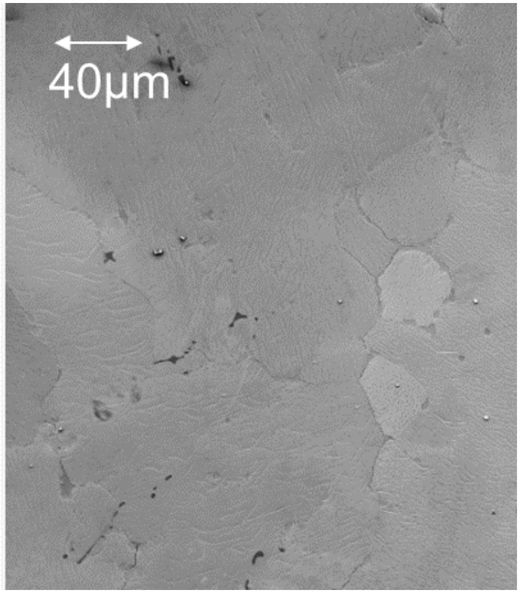


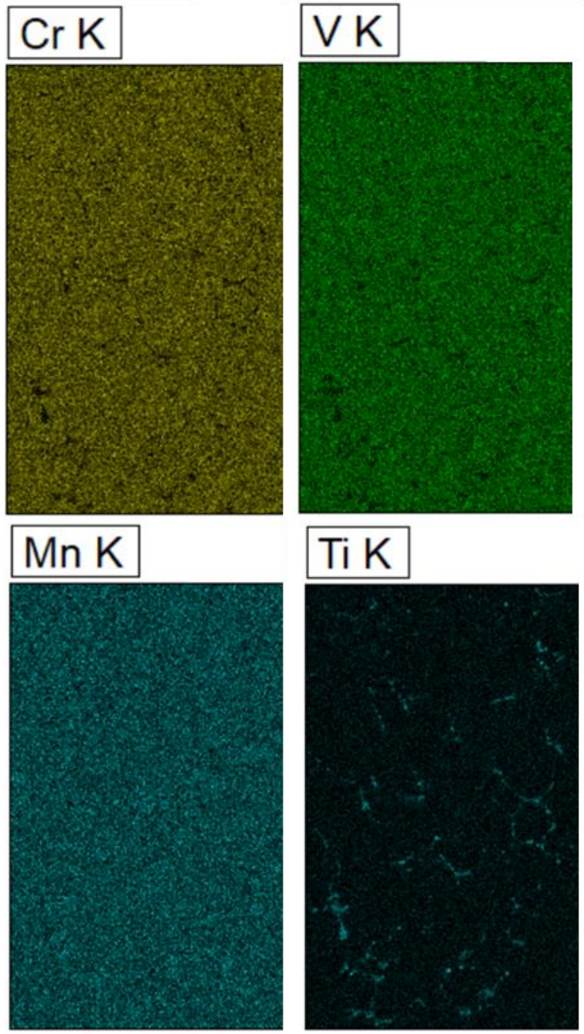
$Cr_{31}Mn_{31}V_{31}Ti_7$

$Cr_{31}Mn_{31}V_{31}Ti_7$

- Narrow BCC phase field spanning ~260 °C
- Laves phase (undesirable) predicted to form below 1160 °C



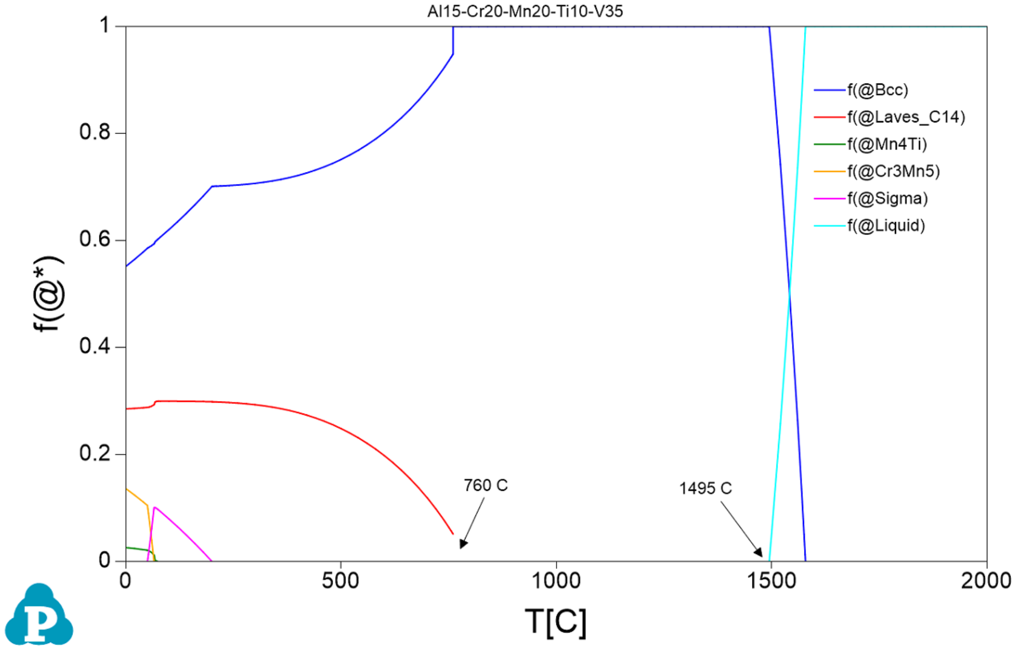


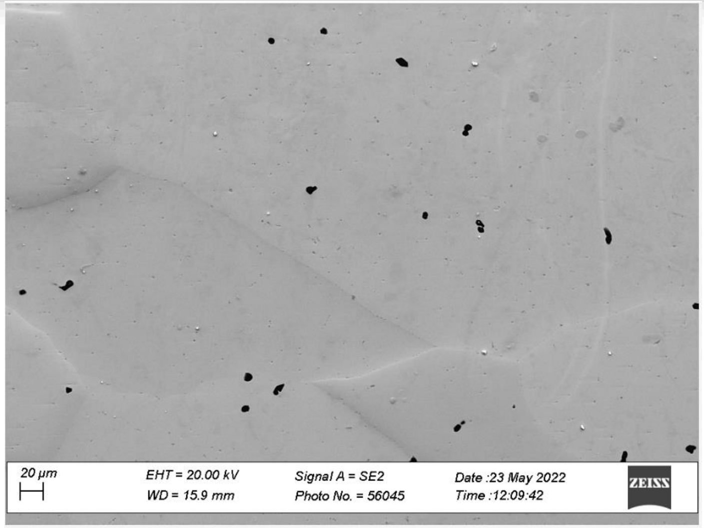


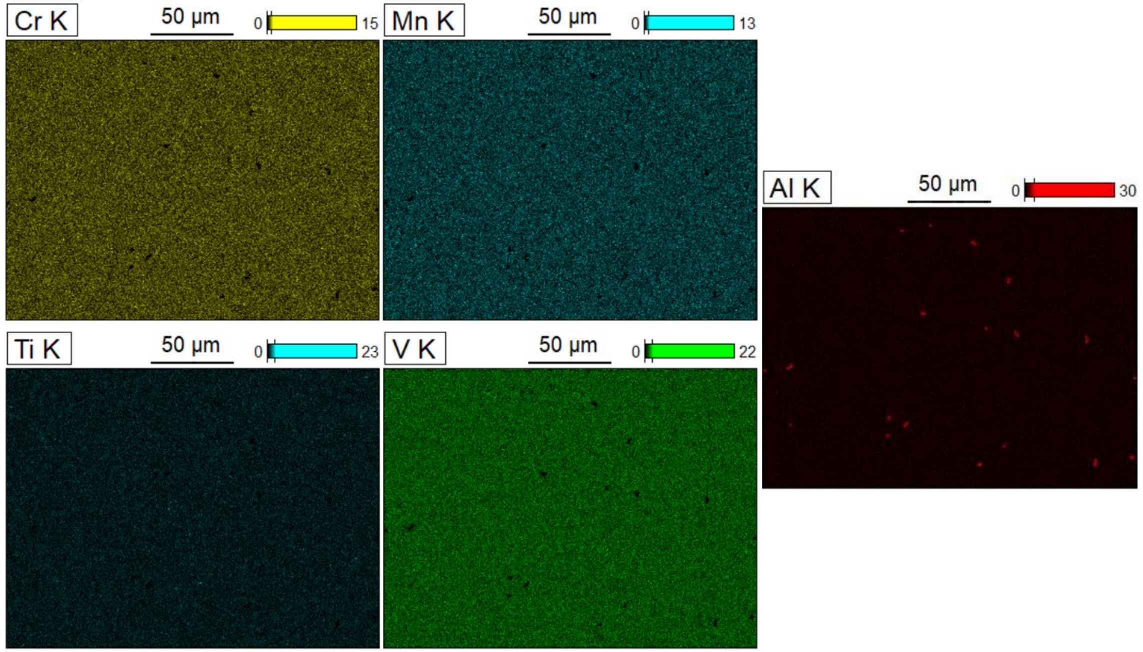
Al-Cr-Mn-Ti-V HEA

$Al_{15}Cr_{20}Mn_{20}Ti_{10}V_{35}$

- Al shown to be a BCC stabilizer in multicomponent alloys + light weight
- Expands BCC stability range (ΔT) to 735 °C
- Increases melting point 75 °C





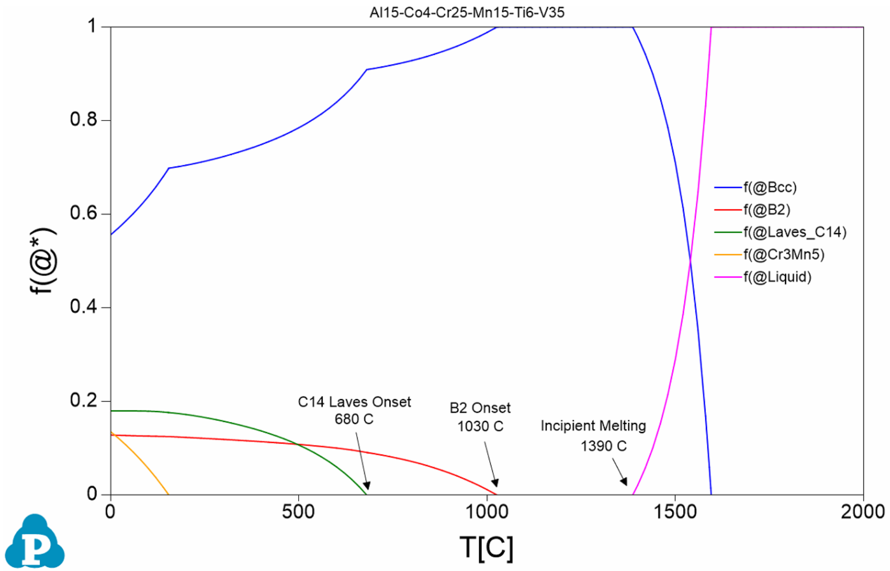


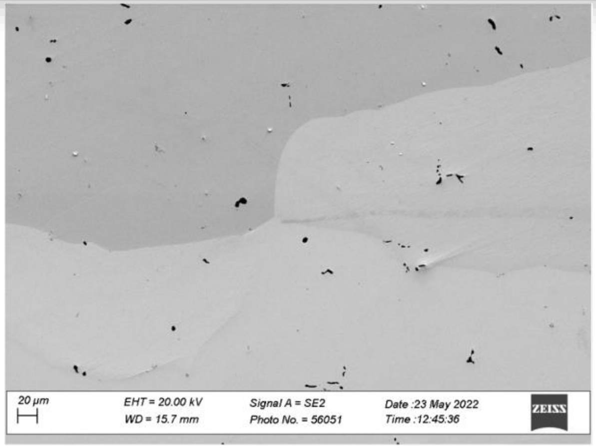
AL-CO-CR-MN-TI-V HEA

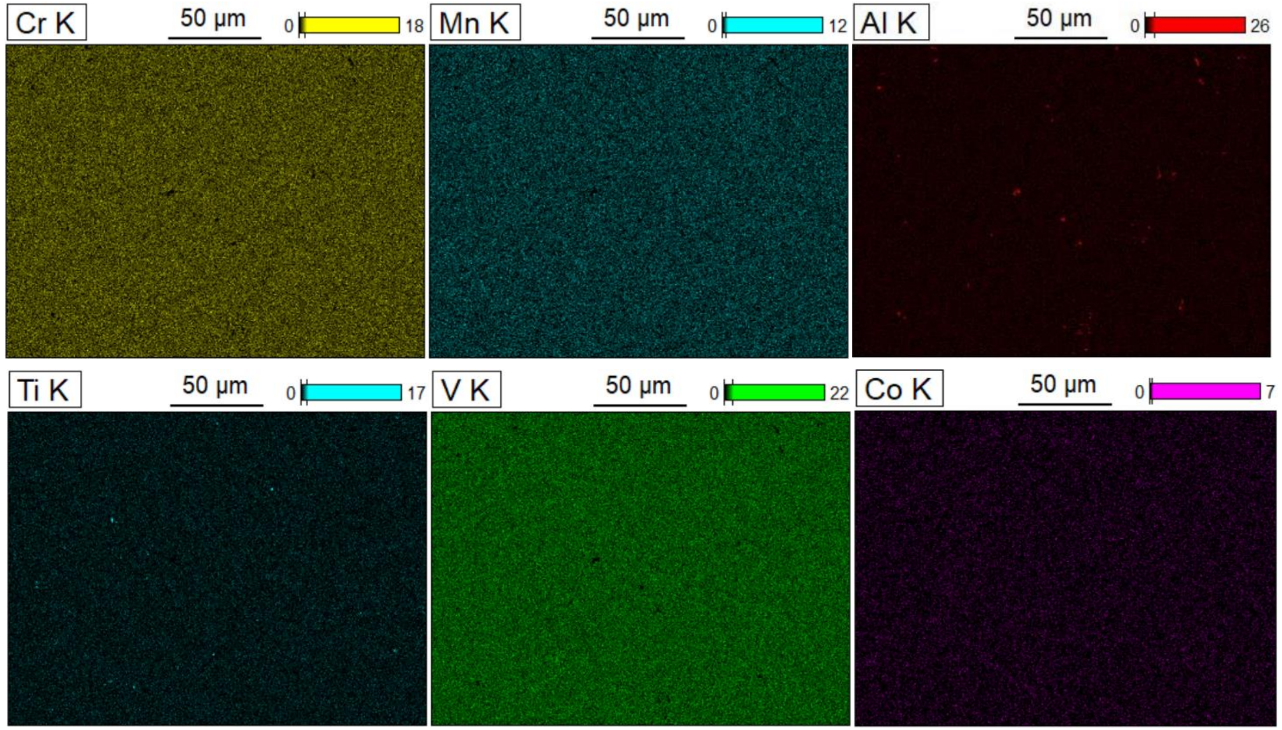
$Al_{15}Co_4Cr_{15}Mn_{15}Ti_6V_{35}$

- 4% added Co at the cost of Ti promotes moderate B2 phase formation while maintaining sufficient Ti for impurity gettering

- Further substitution of Cr for Mn helps to mitigate solidus temperature depression from Co additions

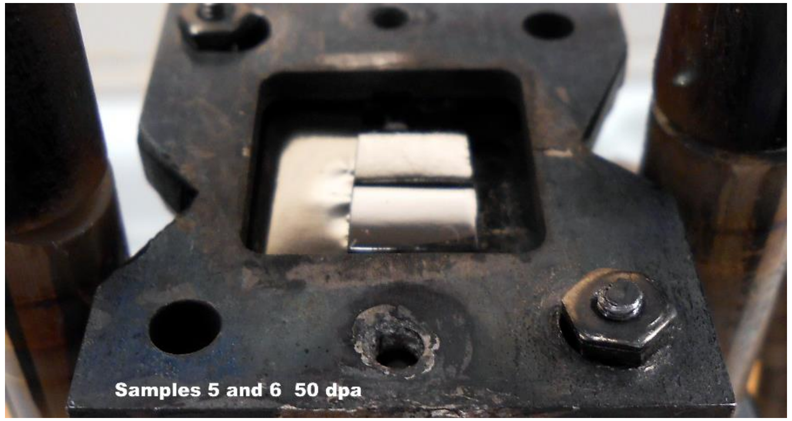


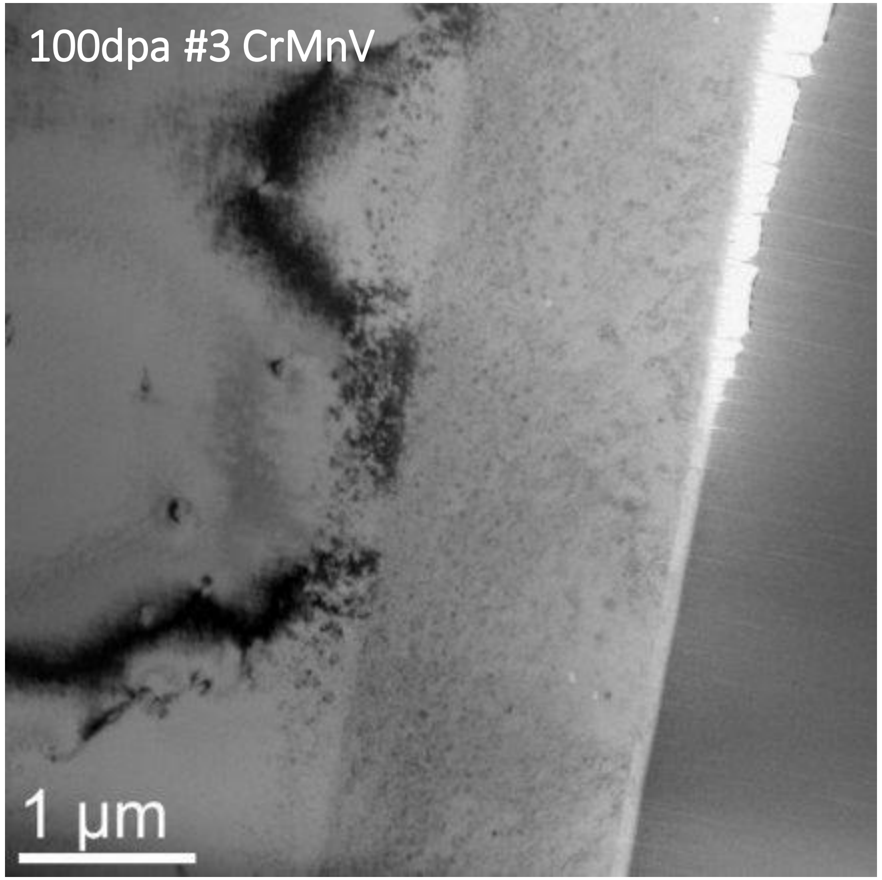




V^{2+} Ion irradiation 50/100 dpa at 500C

- PIE is ongoing

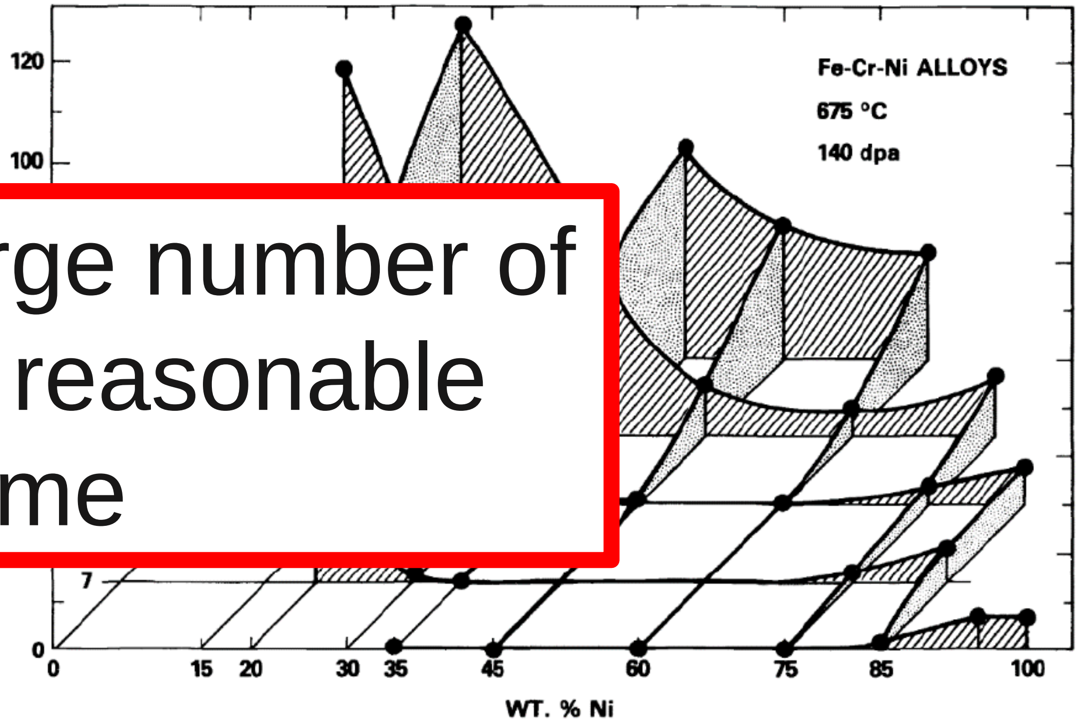




UW – IBL: Example 2 – High Throughput Irradiation of HEAs

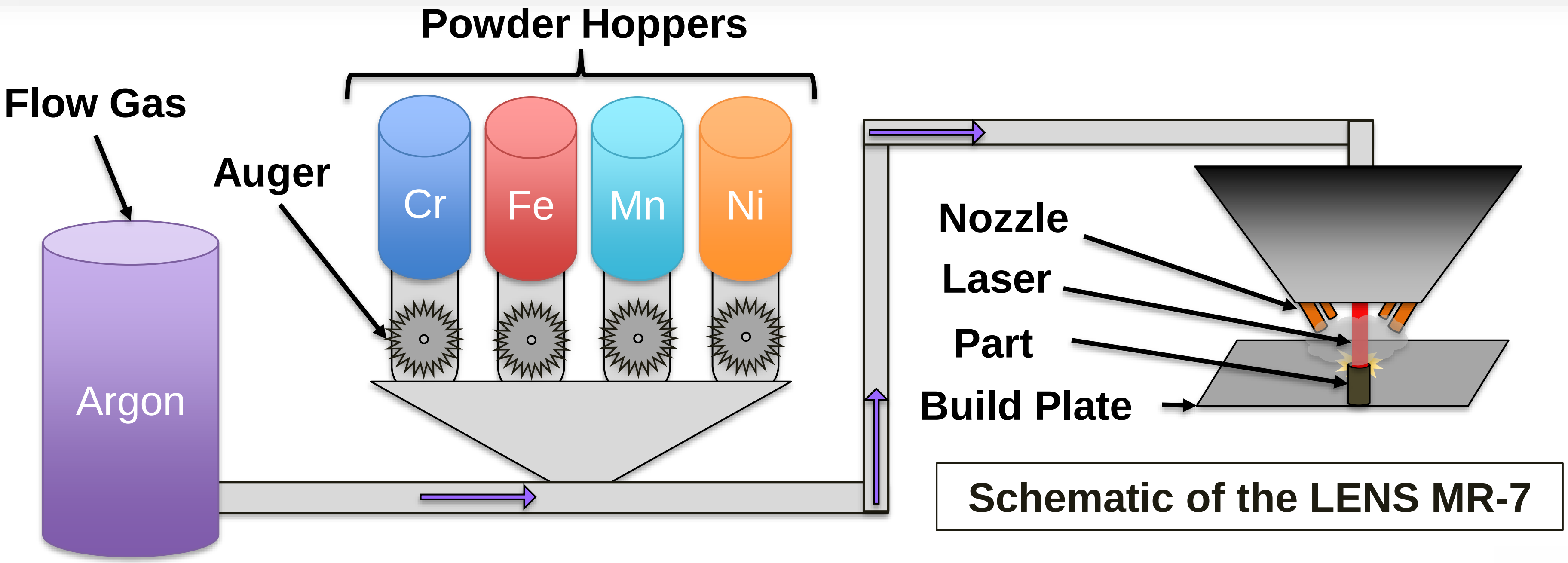
- Metallurgic study of large number of alloys is both expensive and time consuming
- Study of n… is expensi… require sp…
- Standard ion-irradiated materials study time increases linearly with number of alloys

Objective: Study a large number of different alloys in a reasonable period of time

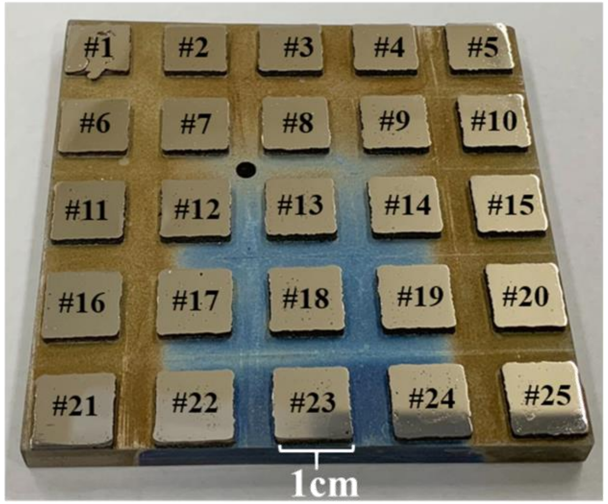


Johnston et al., 1982

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

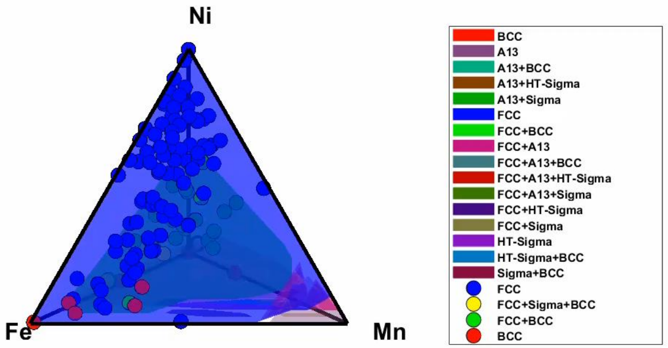


Schematic of the LENS MR-7



In collaboration with Prof. Dan Thoma at UW-Madison

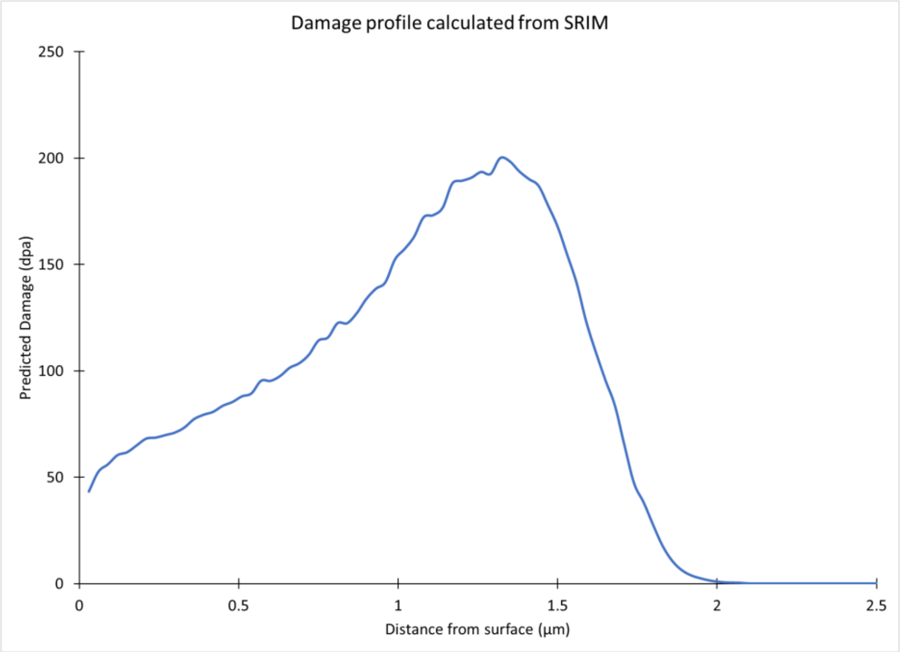
- Elemental powders are controlled independently
- Powders are delivered to print head by argon flow gas
- Laser down optic axis melts powders
- Focus on steels component FeCrMnNi for method validation



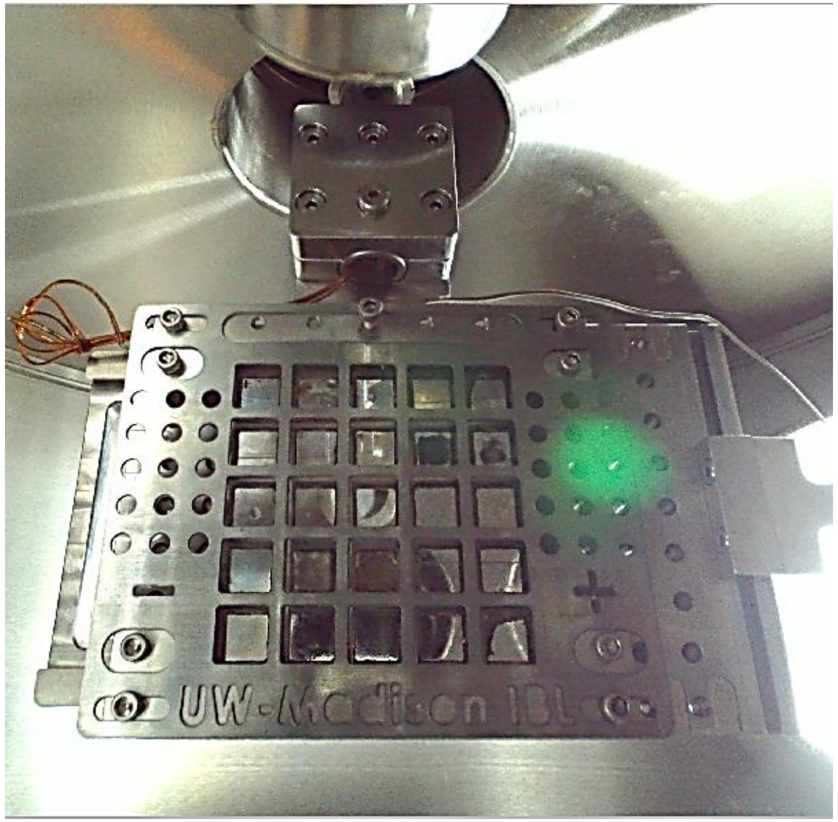
Moorehead, M., Bertsch, K., Niezgoda, M., Parkin, C., Elbakhshwan, M., Sridharan, K., Zhang, C,. Thoma, D., Couet, A. High-throughput synthesis of Mo-Nb-Ta-W high-entropy alloys via additive manufacturing. Mater. Des. 187, 108358 (2020).

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

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



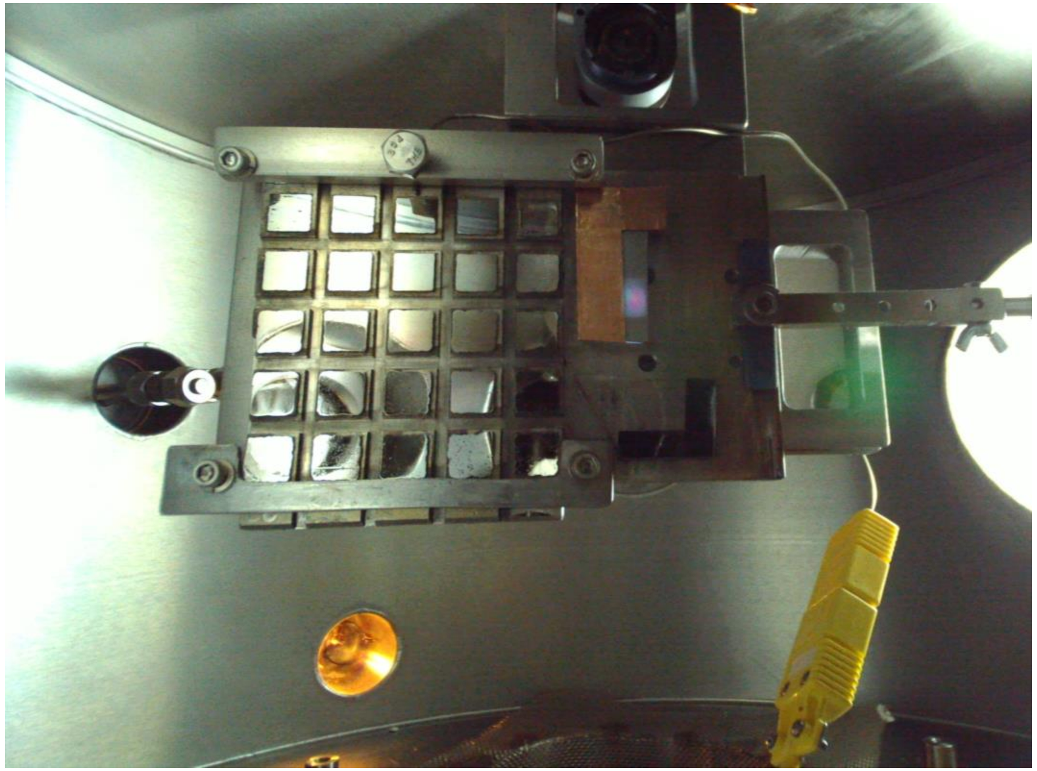
Challenge: Irradiate 25 samples in a limited period of time



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



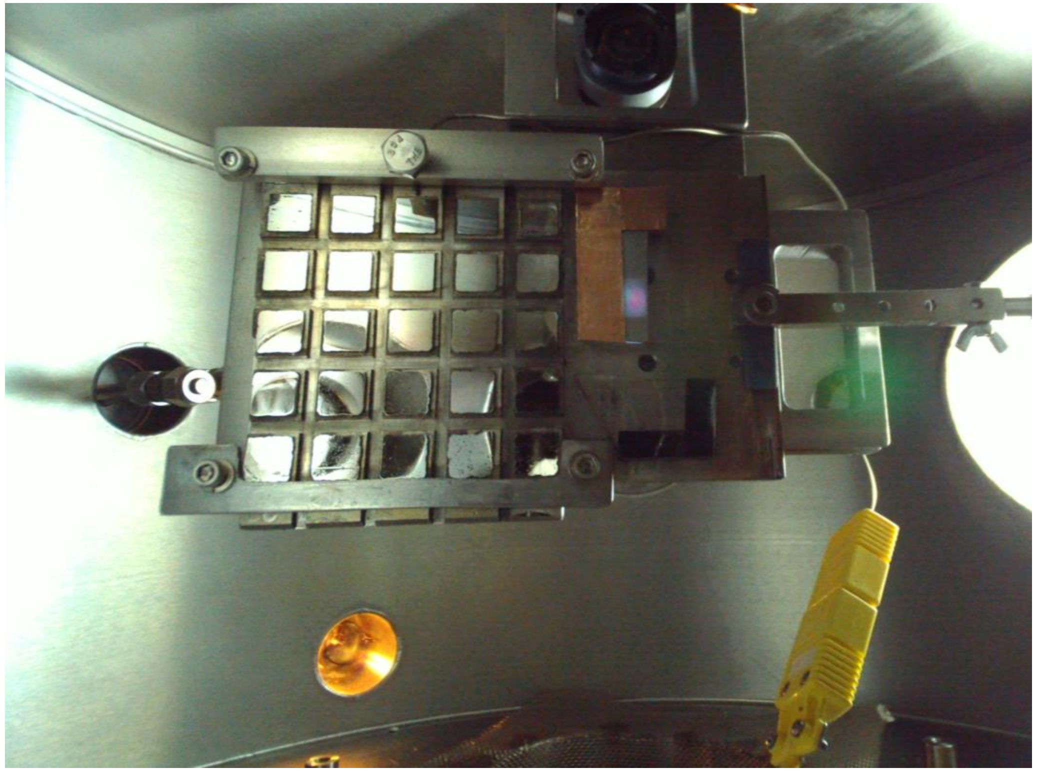
HEA additively printed plate in chamber

Ion beam positioning using electrostatic deflectors

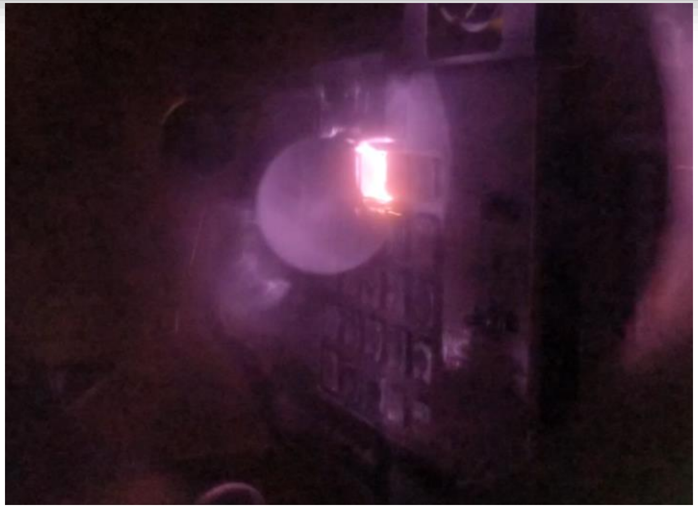
Ion beam shaping (Ta apertures) and Automated Faraday cup insertion for current reading

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

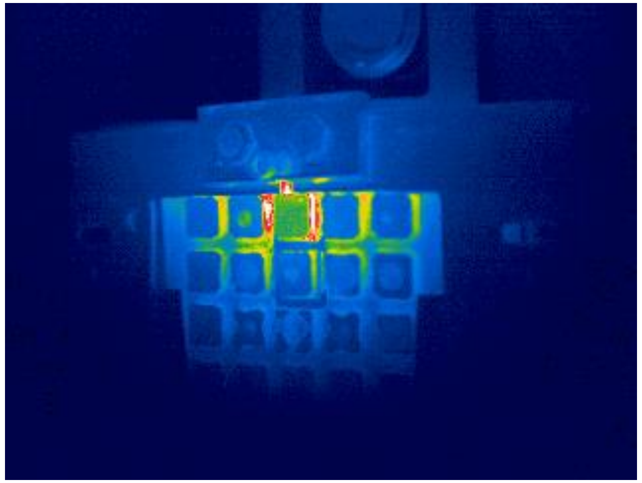
- Need to perfectly align three beams (ion beam, IR laser, pyrometer) on the same spot → Camera inside the irradiation chamber

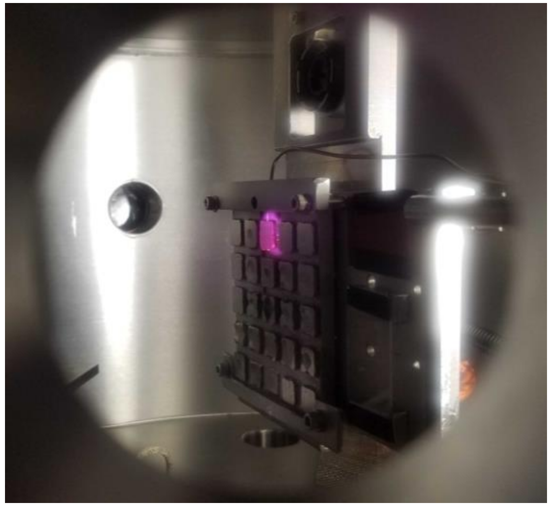


HEA additively printed plate in chamber



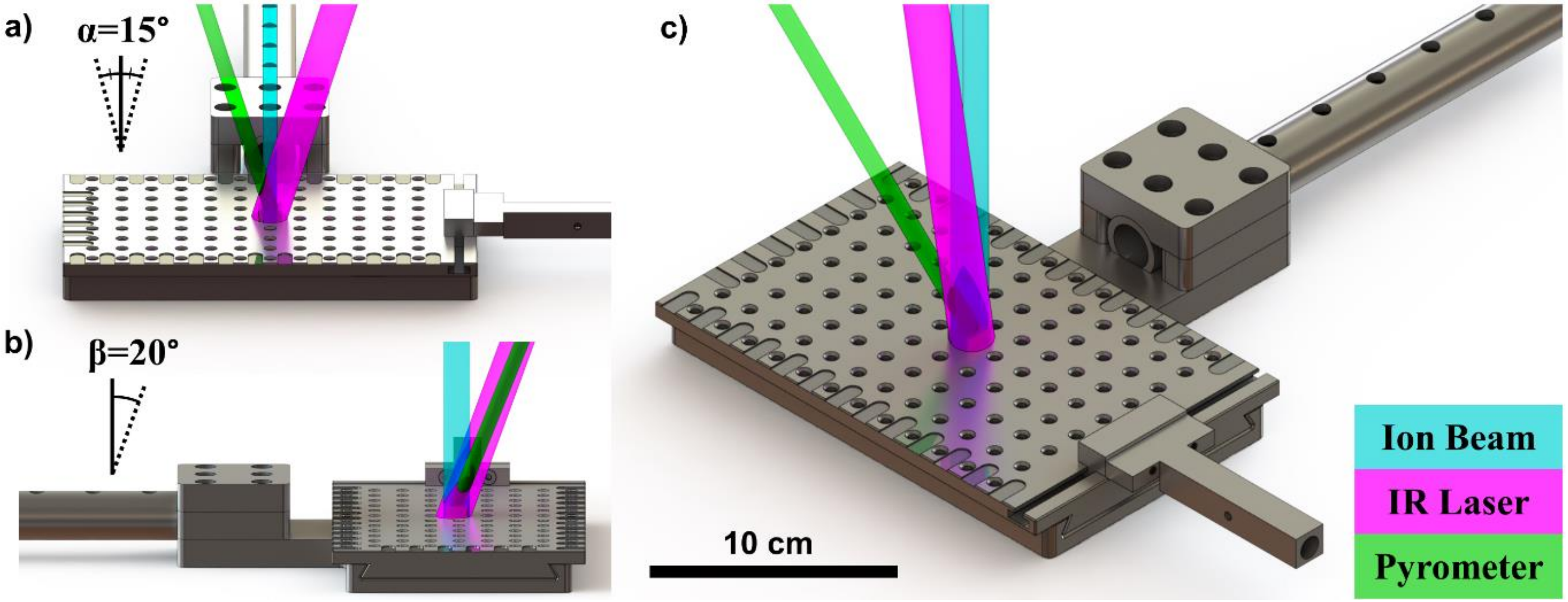
2kW laser turning off and still glowing red hot while the surrounding samples are cool

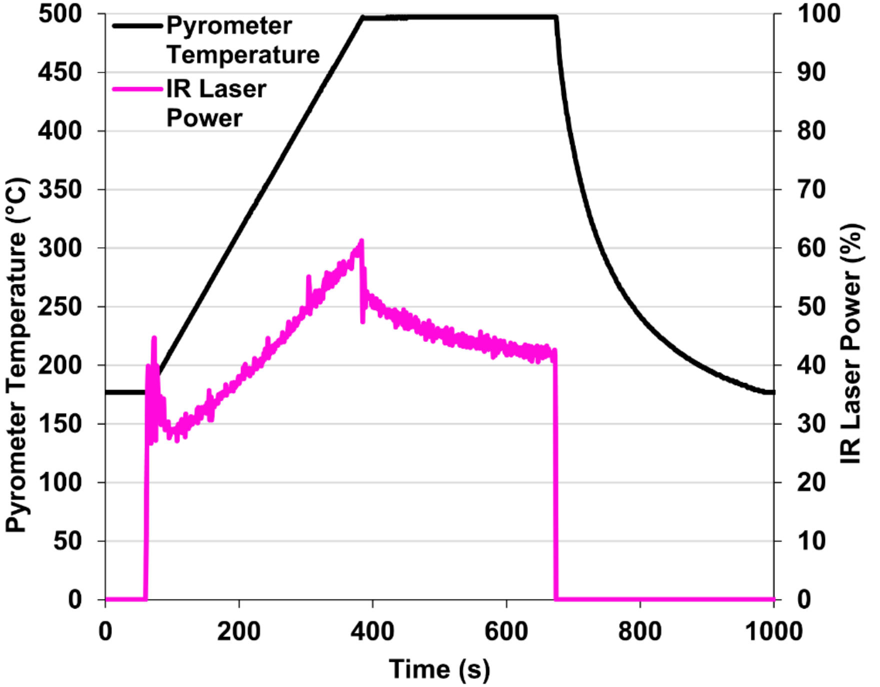




In-situ IR camera and pyrometer reading for temperature control

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





- IR laser (2kW) to heat the sample surface during irradiation (irradiation at temperature)
- Pyrometer to detect temperature in-situ and feedback loop to laser power
- Can reach 500C in 316SS in minutes with excellent temperature control

M. Moorehead, B. Queylat, H. Zhang, K. Kriewaldt, A. Couet, Development of a versatile, high-temperature, high-throughput ion irradiation system, Nucl. Instruments Methods Phys. Res. Sect. A 1020 (2021) 165892.

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

Number of samples	75
Ions	4 MeV Ni^{2+}
Irradiation temperature	500 ºC
Vacuum	10^{-6} torrs
Peak damage	200 dpa
Damage rate	0.04 dpa.s^{-1}
Total irradiation time	100 hours

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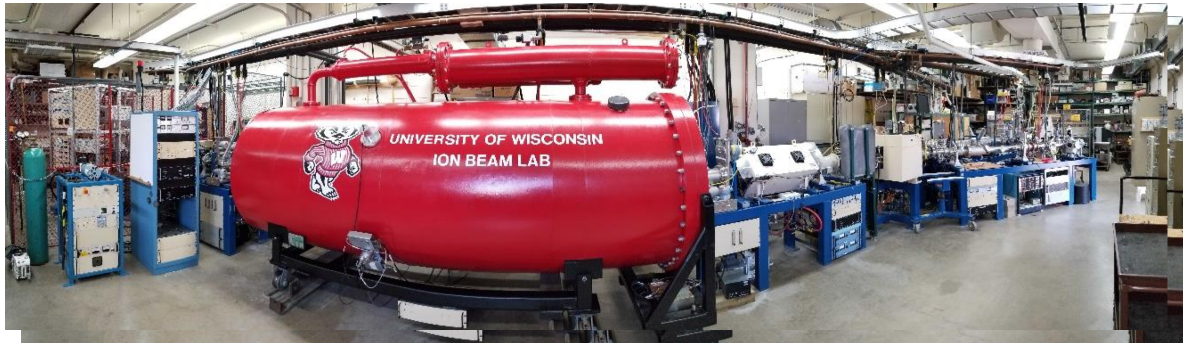

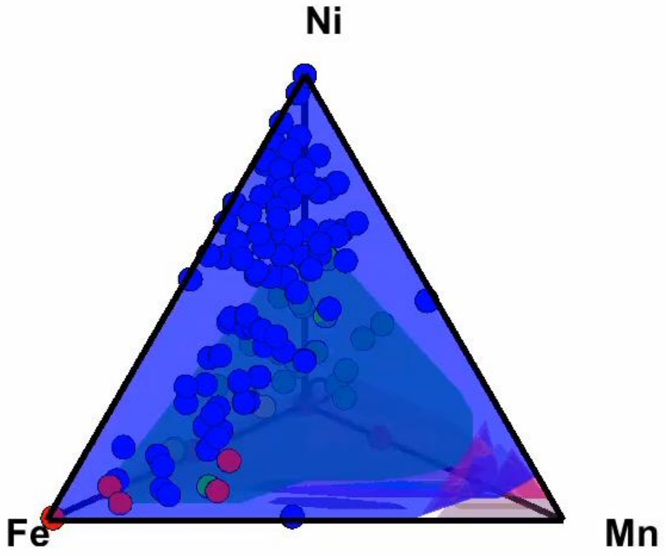

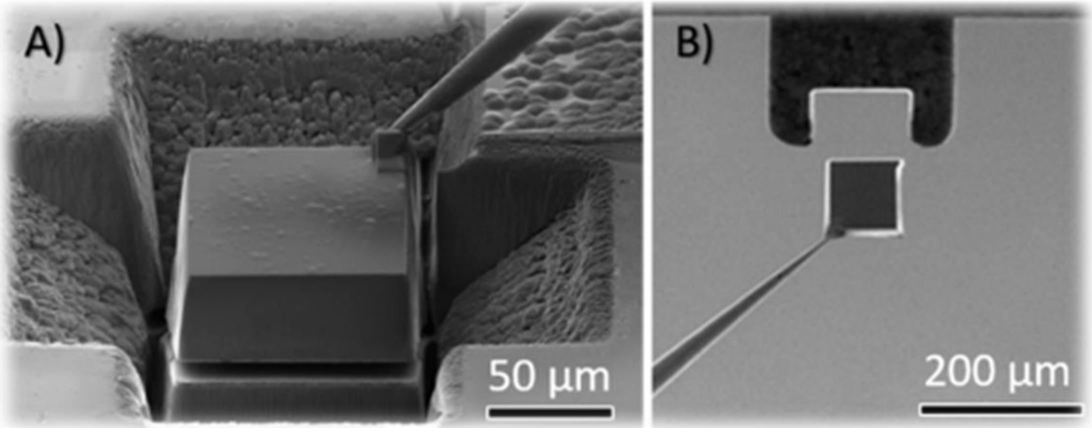

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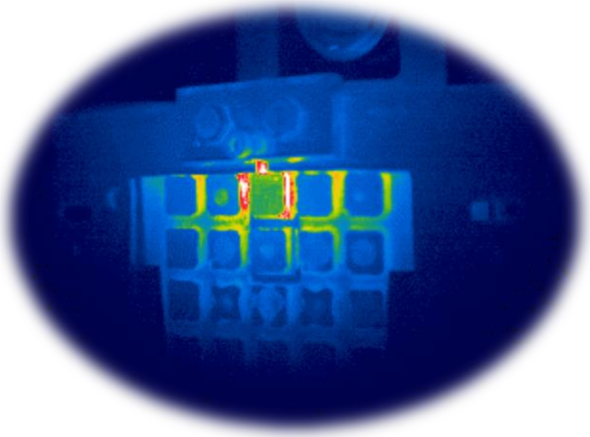

NSUF Nuclear Science User Facilities

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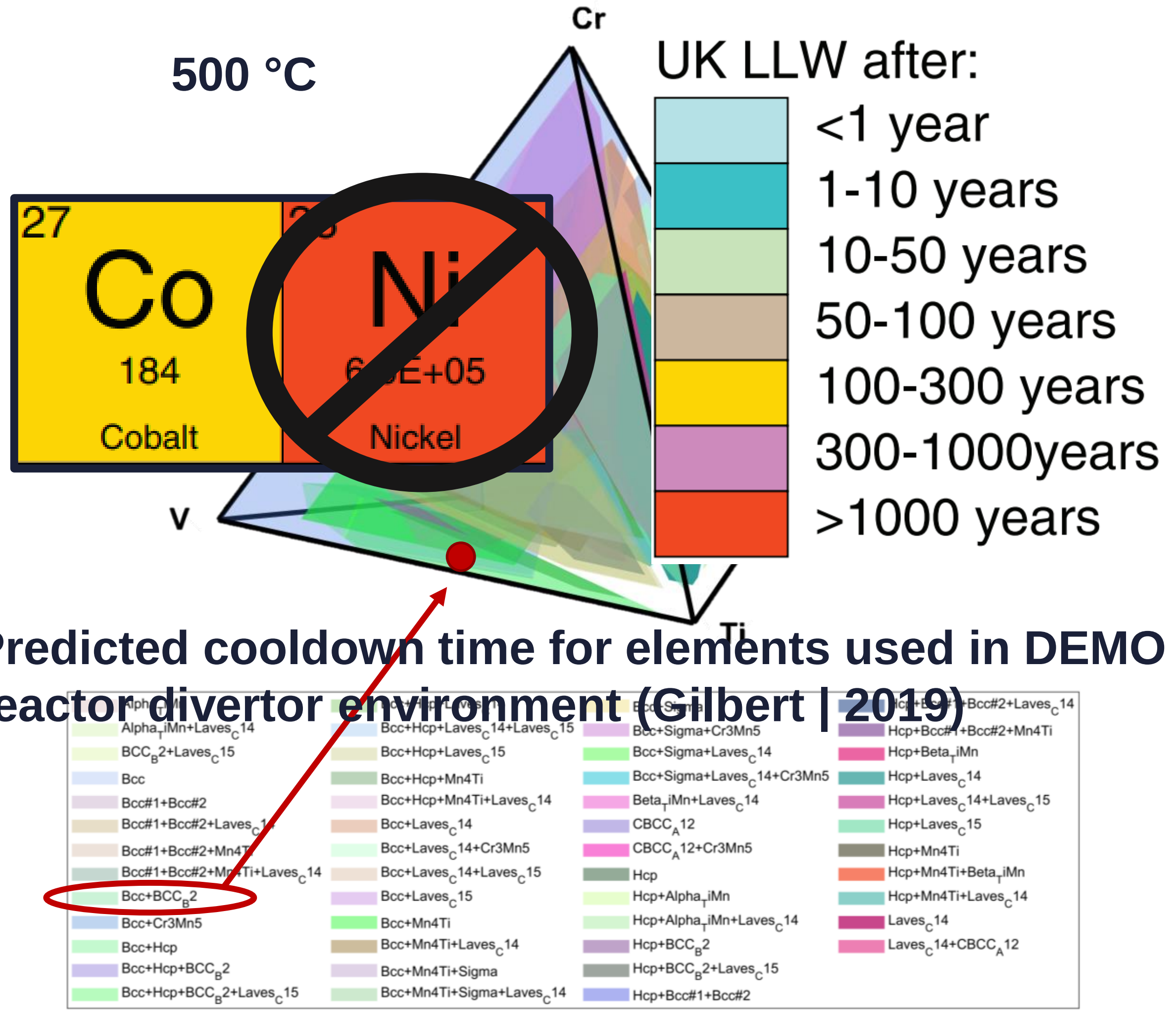
UW – Ion Beam Laboratory (IBL)

- 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

Stretch Goal: Add Semi-Coherent Precipitates

- Ordered BCC phase (B2) expected to be semi-coherent with BCC lattice
- No Mn-containing (or concentrated) CrMnTiV alloys exhibit a B2 phase at low temperatures
- Dilute Ni or Co additions to $Al_{15}Cr_{20}Mn_{20}Ti_{10}V_{35}$ promote B2 formation

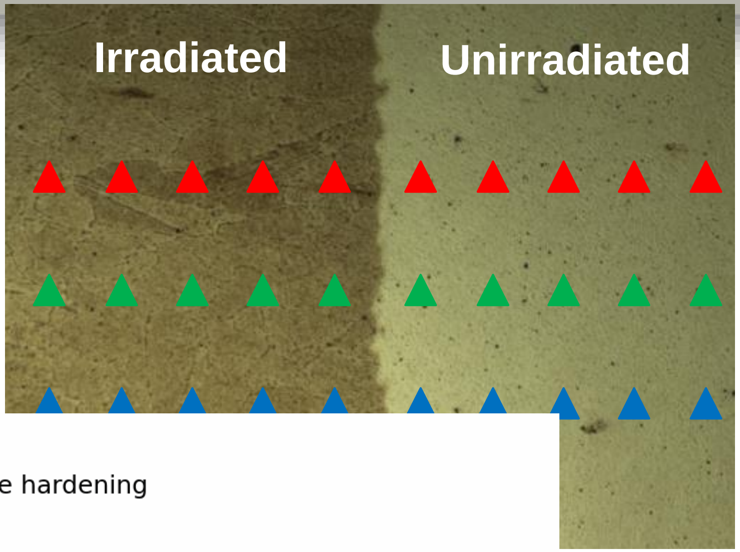


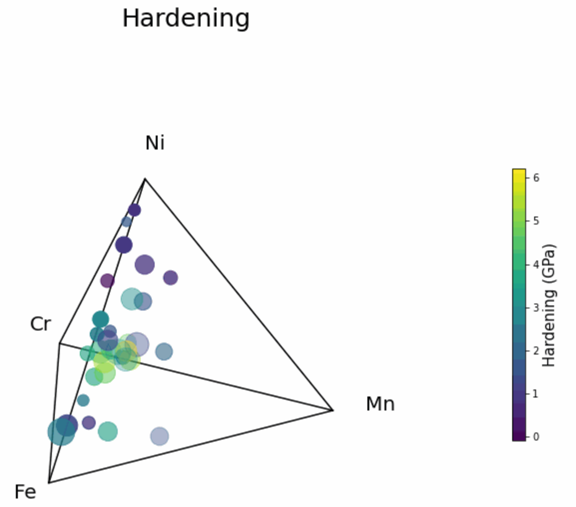
Predicted cooldown time for elements used in DEMO reactor divertor environment (Gilbert | 2019)

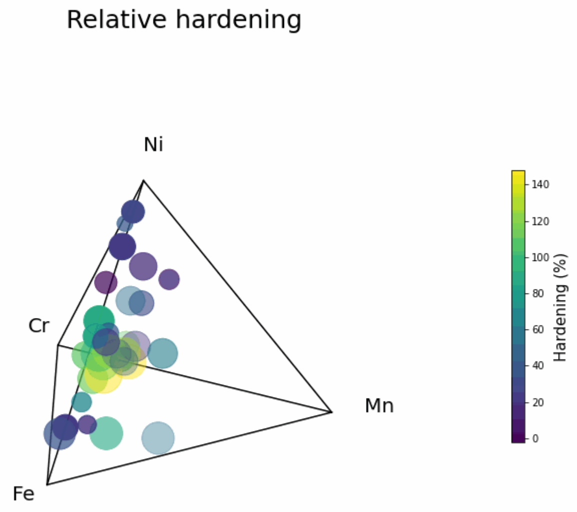
UW – IBL: Example 2 – High Throughput Irradiation of HEAs

Irradiation induced hardening

- For each samples: 3 line-scans (10 indents each) have been done across the interface
- Total time: 44 hours per plate (3mn/indent)







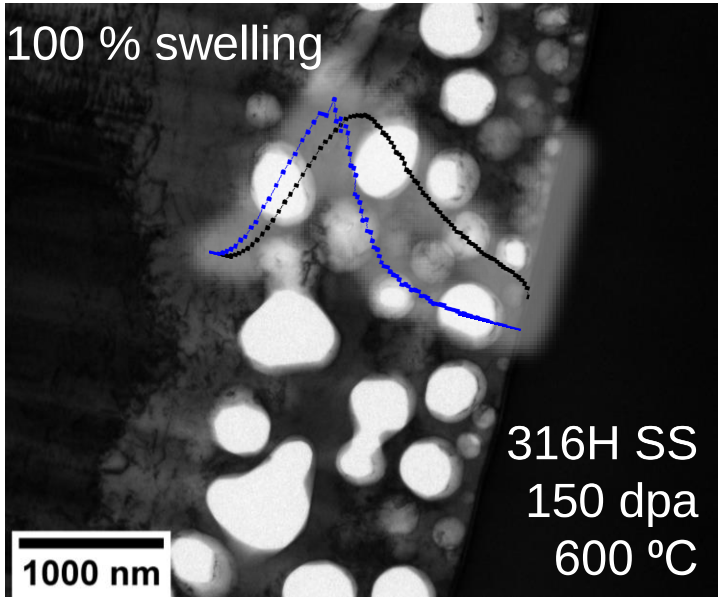
Ni34
1st line-scan
2nd line-scan
3rd line-scan

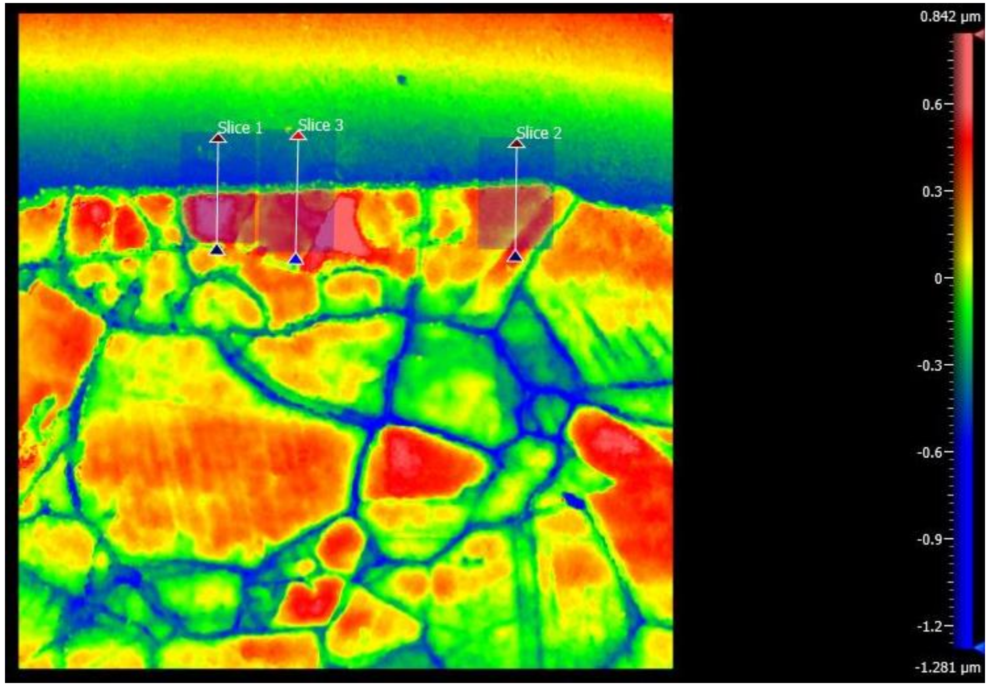
—Not irradiated —Irradiated

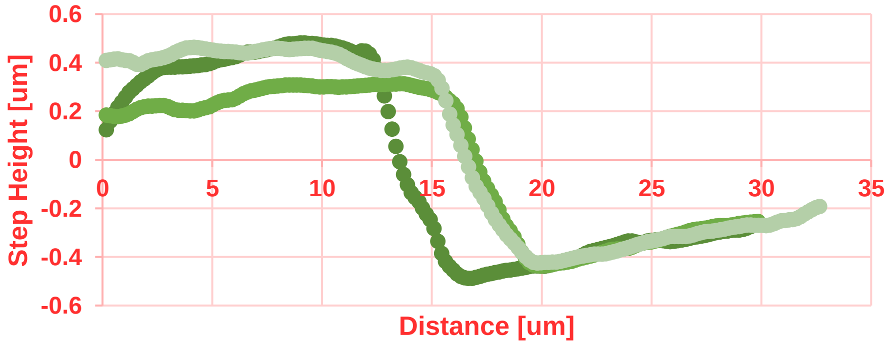
UW – IBL: Example 2 – High Throughput Irradiation of HEAs

Irradiation induced void swelling measured by profilometry:

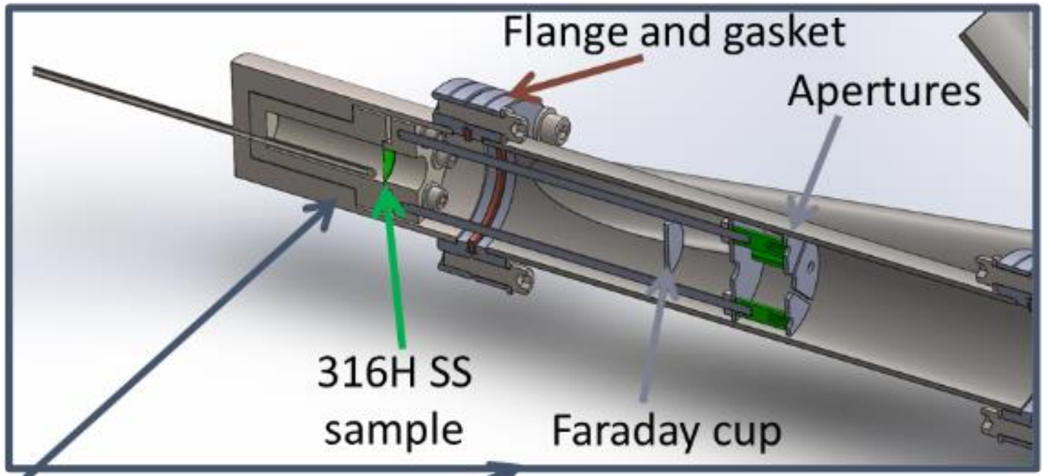
- Profilometry is a relevant semi-high throughput technique to measure irradiation induced void swelling.
- Under development for high-throughput capabilities

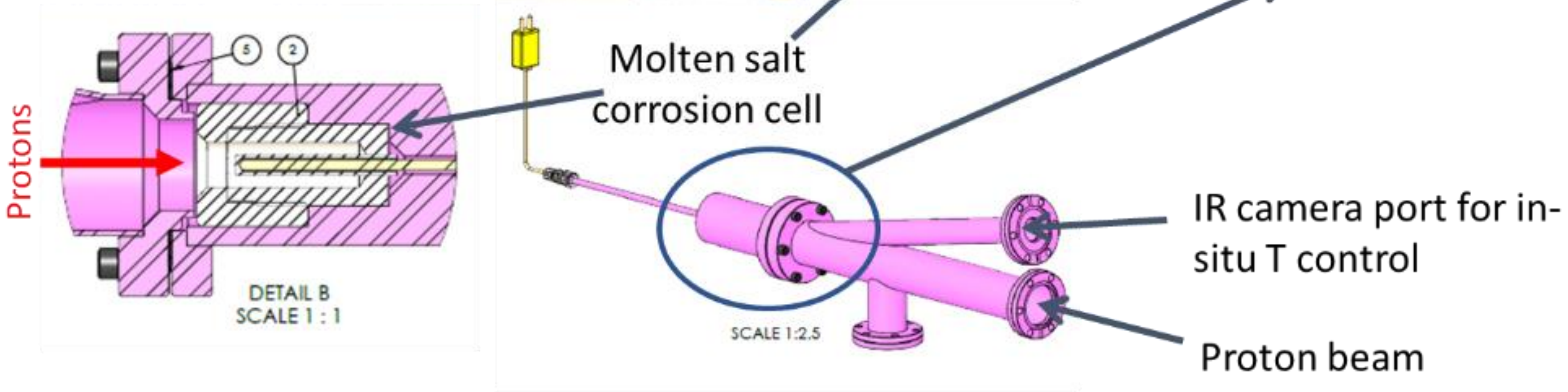






UW – IBL: Example 3 – In-Situ Irradiation in Molten Salts (2022)





UW – IBL: Post Irradiation Examination (PIE) Capabilities

UW – IBL: Post Irradiation Examination (PIE) Capabilities

- For sample preparation of low-active materials
  - Parallel polisher
  - Low speed saw
  - Electro-polisher
  - Ion mill
  - High accuracy balance

UW – IBL: Post Irradiation Examination (PIE) Capabilities

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

UW – IBL: Post Irradiation Examination (PIE) Capabilities

- For sample characterization in non-rad area (after sample prep in rad-area)

FEI Helios G4 UX Plasma FIB/FE SEM

FEI Titan Cs-corrected scanning transmission electron microscope

Hysitron TI 950 TriboIndenter