MADCOR

High-throughput irradiation and characterization capabilities at UW Ion Beam Laboratory to support data-driven modeling and nuclear materials design

NSUF workshop

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- **INL**: Pengyuan Xiu, Mukesh Bachhav, Lingfeng He
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CompuTherm FOR MATERIALS DESIGN

Los Alamos NATIONAL LABORATORY

Idaho National Laboratory

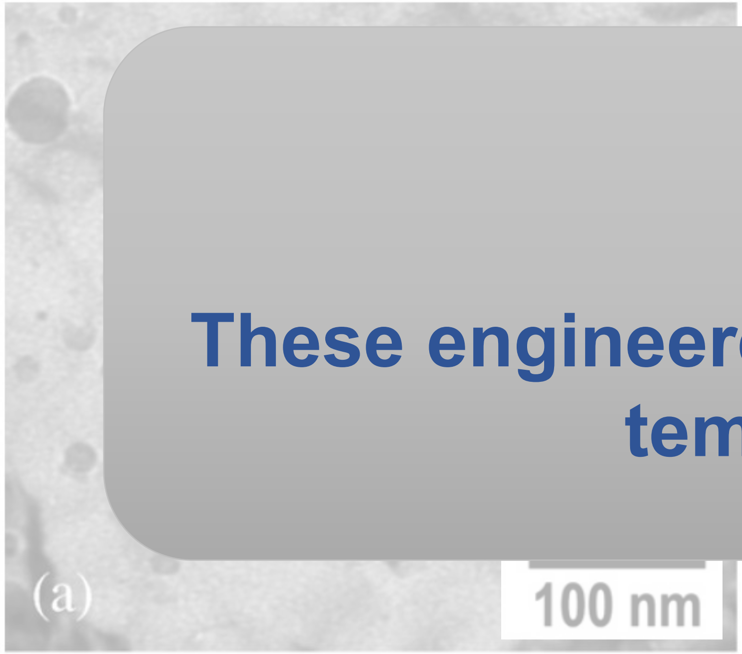
RUHR UNIVERSITÄT BOCHUM RUB

Argonne NATIONAL LABORATORY

Irradiation damage – how do we prevent it?

Typical approach to improving radiation tolerance: <u>increase sink density</u>

Oxide-dispersion strengthened (ODS) alloys



(Allen | 2008)

Nanocrystalline alloys

(Barr | 2018)

Cold-worked alloys

(Alshater | 2018)

HOWEVER

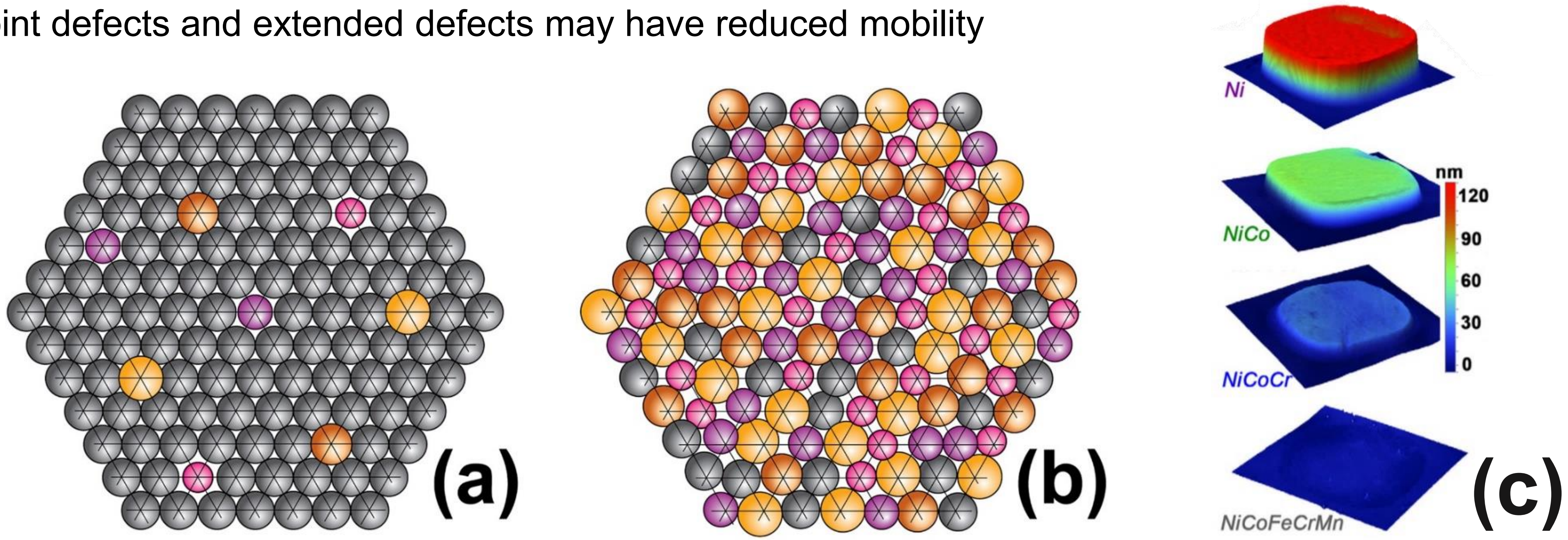
These engineered microstructures are not stable under all temperature and damage regimes

[1] N. Packan and K. Farrell, "Simulation of first wall damage: effects of the method of gas implantation," Journal of Nuclear Materials, vol. 85 & 86, pp. 677-681, 1979.
[2] C. Parkin et al., "Phase stability, mechanical properties, and ion irradiation effects in face-centered cubic FeCrMnNi compositionally complex solid-solution alloys at high temperatures," Journal of Nuclear Materials, vol. 565, 2022.
[3] L. Mansur, "Theory and experimental background on dimensional changes in irradiated alloys," Journal of Nuclear Materials, vol. 216, pp. 97-123, Oct. 1994.
[4] C. Li, X. Hu, T. Yang, N.K. Kumar, B. Wirth, and S. Zinkle, "Neutron irradiation response of a Co-free high entropy alloy," Journal of Nuclear Materials, vol. 527, pp. 151838, Dec. 2019.
[5] Y. Chen, Y. Yang, Y. Huang, T. Allen, B. Alexandreanu, and K. Natesan, "Void Swelling and Microstructure of Austenitic Stainless Steels Irradiated in the BOR-60 Reactor," US Nuclear Regulatory Commission CR-7128, Nov. 2012

Background: high-entropy alloys

Modeling studies, limited experimental work suggest radiation resistance of compositionally complex matrix

- HEA are of interest as a replacement base matrix for nuclear alloy design
- Phonon broadening in displacement cascade may slow quench, promote recombination
- Point defects and extended defects may have reduced mobility

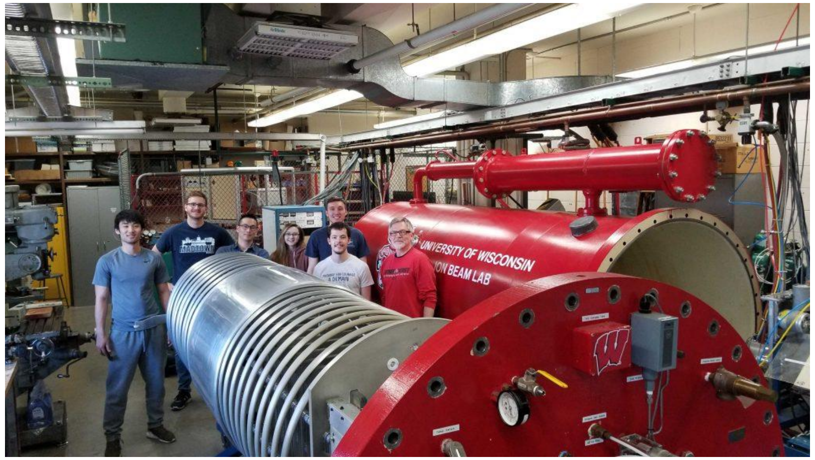


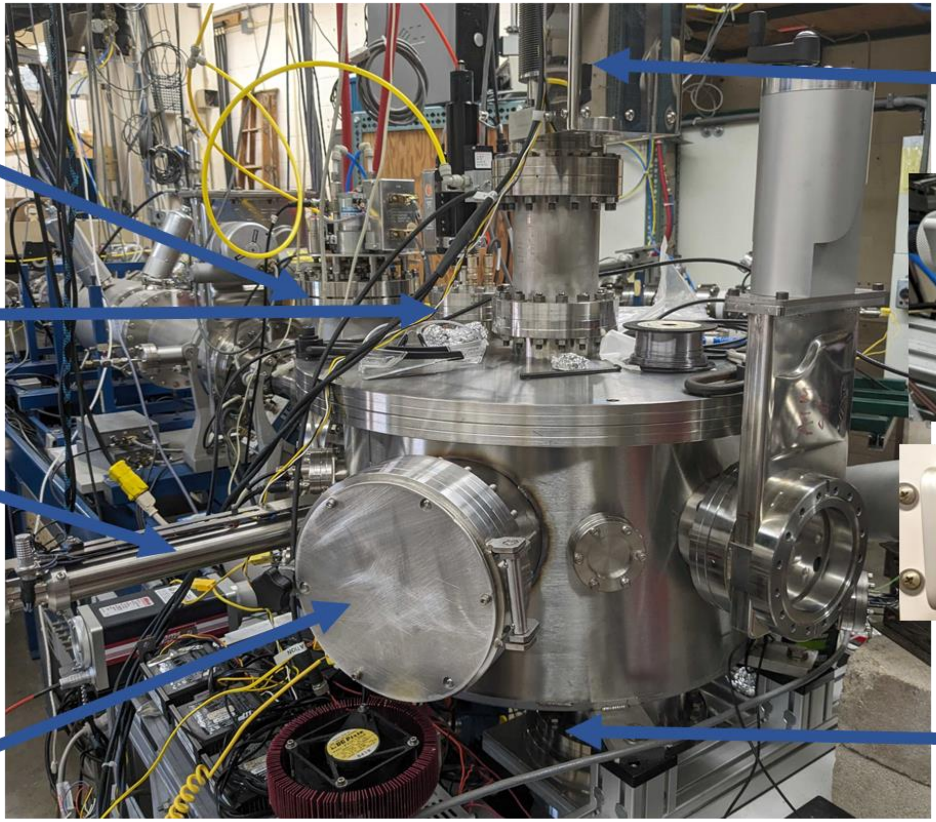
(a) Conventional Alloy, (b) High Entropy Alloy[1], (c) Swelling of increasingly complex alloys under ion irradiation[2]

Irradiation of single-phase HEA (CCA) needed to investigate benefits of compositional complexity.

1. D. Miracle, O. Senkov, A critical review of high entropy alloys and related concepts, Acta Materialia 122 (2017) 448 - 511.
2. K. Jin, C. Lu, L.M. Wang, J. Qu, W.J. Weber, Y. Zhang, H. Bei, Effects of compositional complexity on the ion-irradiation induced swelling and hardening in Ni-containing equiatomic alloys, Scripta Materialia 119 (2016) 65-70.

High-Throughput Testing: Ion irradiation at UW-IBL





Adjustable X-Y Apertures

Faraday Cup

X Linear Actuator

Pyrometer

Load Lock

Y Linear Actuator

800 L/s Turbo Pump Below

High Throughput Irradiation – independent alloys

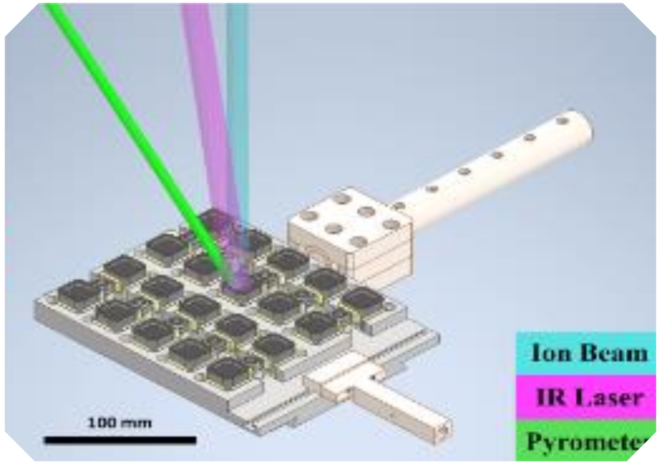
- 1.7 MV tandem Pelletron accelerator at UW-Ion Beam Lab
- 200 W near-IR laser for individual sample heating
- Dual wavelength pyrometer for closed loop temperature control

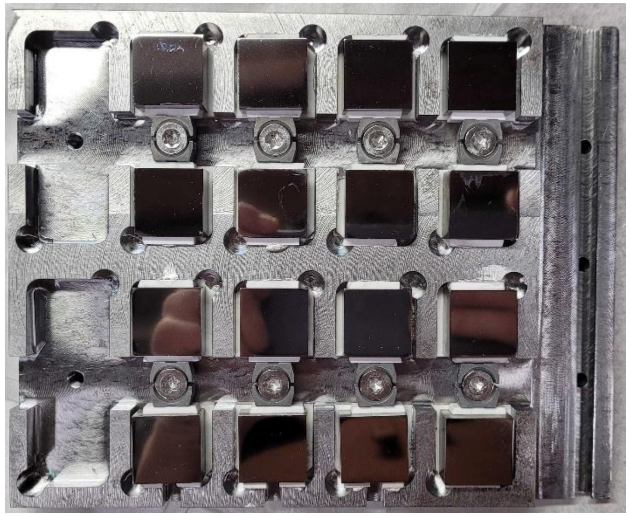
A Nuclear Science User Facility!

Total of 232 dpa at 3 temperatures in 16 hours!

Example: Completed W Irradiations

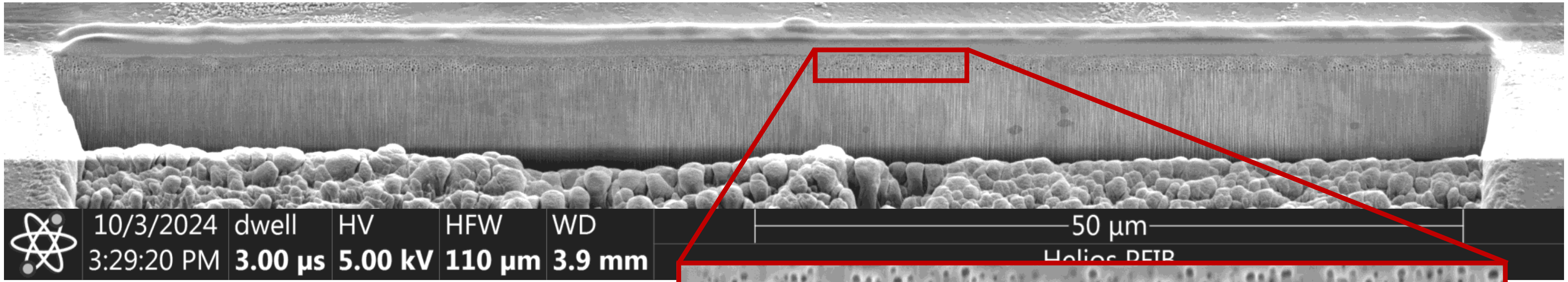
T	1 dpa	5 dpa	10 dpa	20 dpa	50 dpa
25 °C	-	-	X	-	X
450 °C	X	X	X	X	X
650 °C	X	X	X	X	X

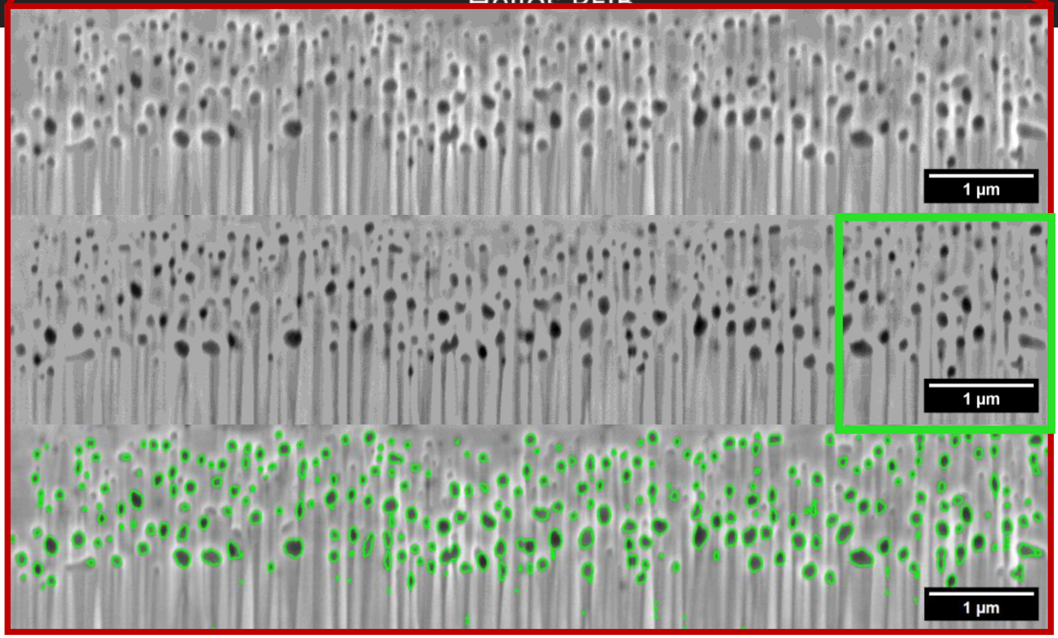


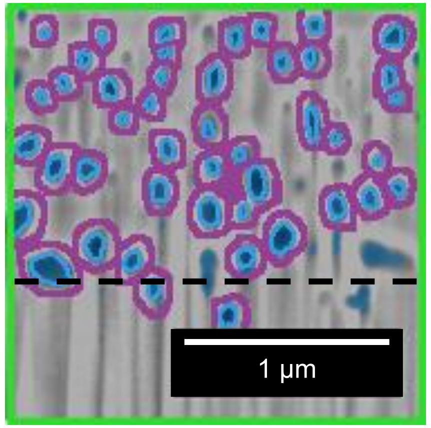


Top: UW-Madison Ion Beam Lab
Left: Triple beam in high throughput chamber
Right: Loaded 15x15mm W samples in high temp. 4140 stage

ML output: Void Swelling (case study in SS304)



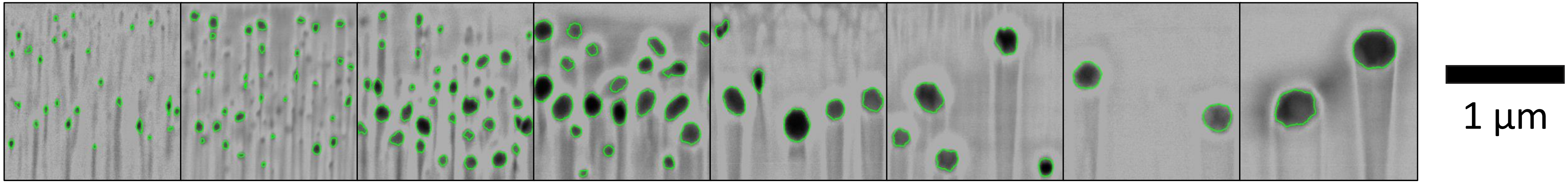




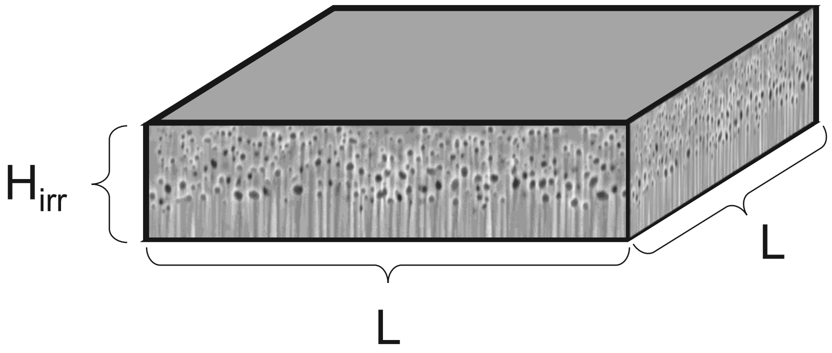
- Void recognition models were trained using 10 µm from each temperatures' <u>400 µm</u> of trenched width

Irradiation Conditions	
Ions	4.0 MeV Ni^{++}
Vacuum	~6 x 10^{-7} torr
Fluence	1.71 x 10^{17} ions/cm^2
Peak damage	225 dpa
Damage rate	0.042 dpa/s
Temperature	530 - 730 °C

ML output: Void Swelling (case study in SS304)



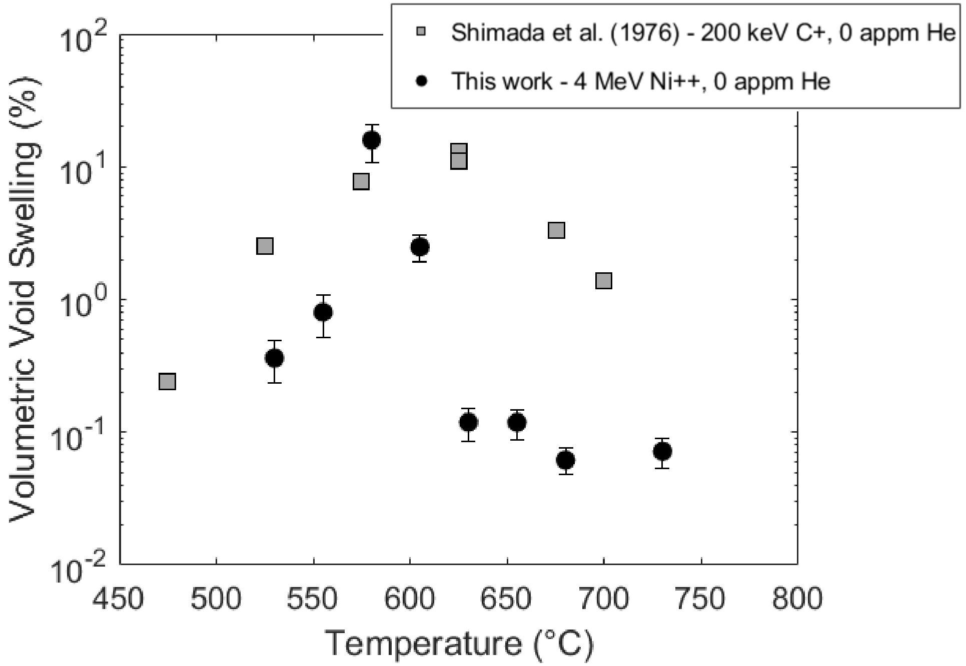
530 °C 555 °C 580 °C 605 °C 630 °C 655 °C 680 °C 730 °C



$$\frac{\Delta V}{V_o} = \left(\frac{N^2}{L^2 H_{Irr}}\right)\overline{V}_{void}$$

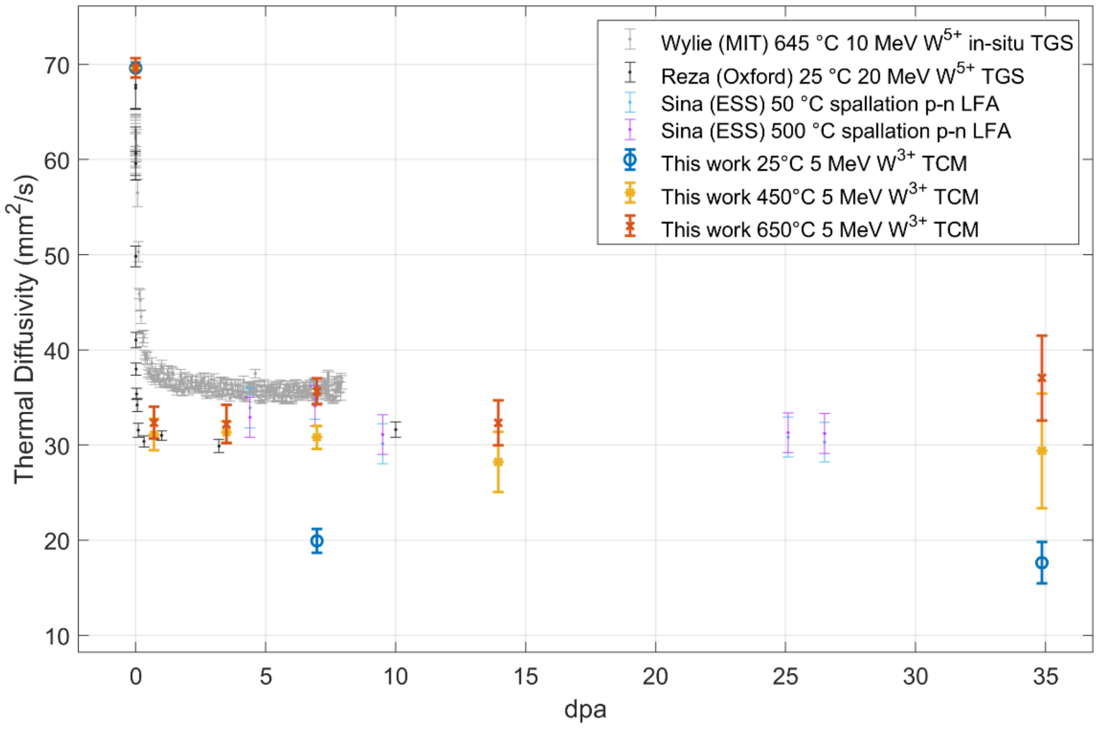
L: Width of imaged area
H_{irr}: Irradiation depth
N: Void population identified
$\overline{V}_{void}$: Average void volume

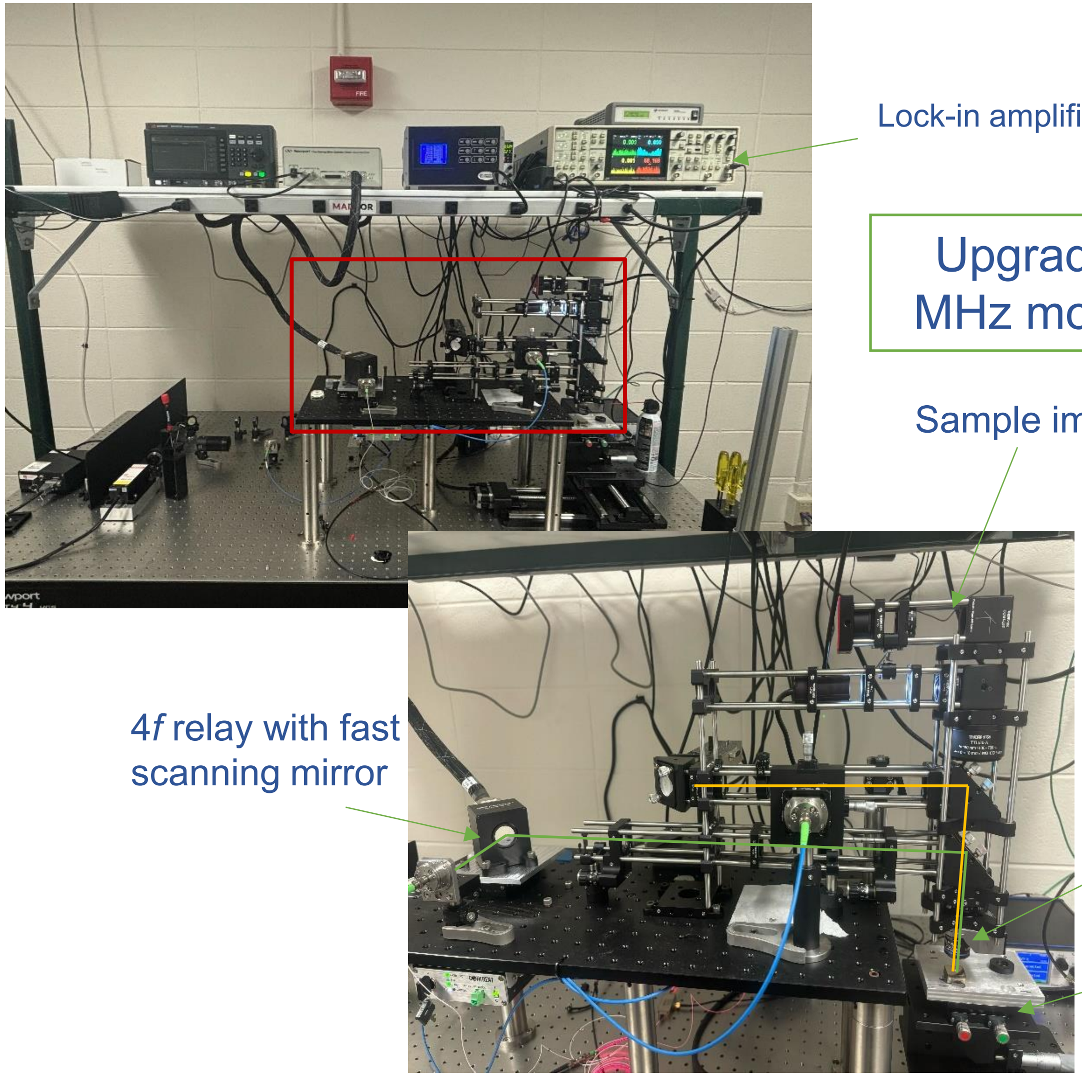
$$\overline{V}_{void} = \frac{\sum_{i=1}^{N}\left(\frac{4\pi}{3}\left(\frac{D_i}{2}\right)^3\right)}{N}$$



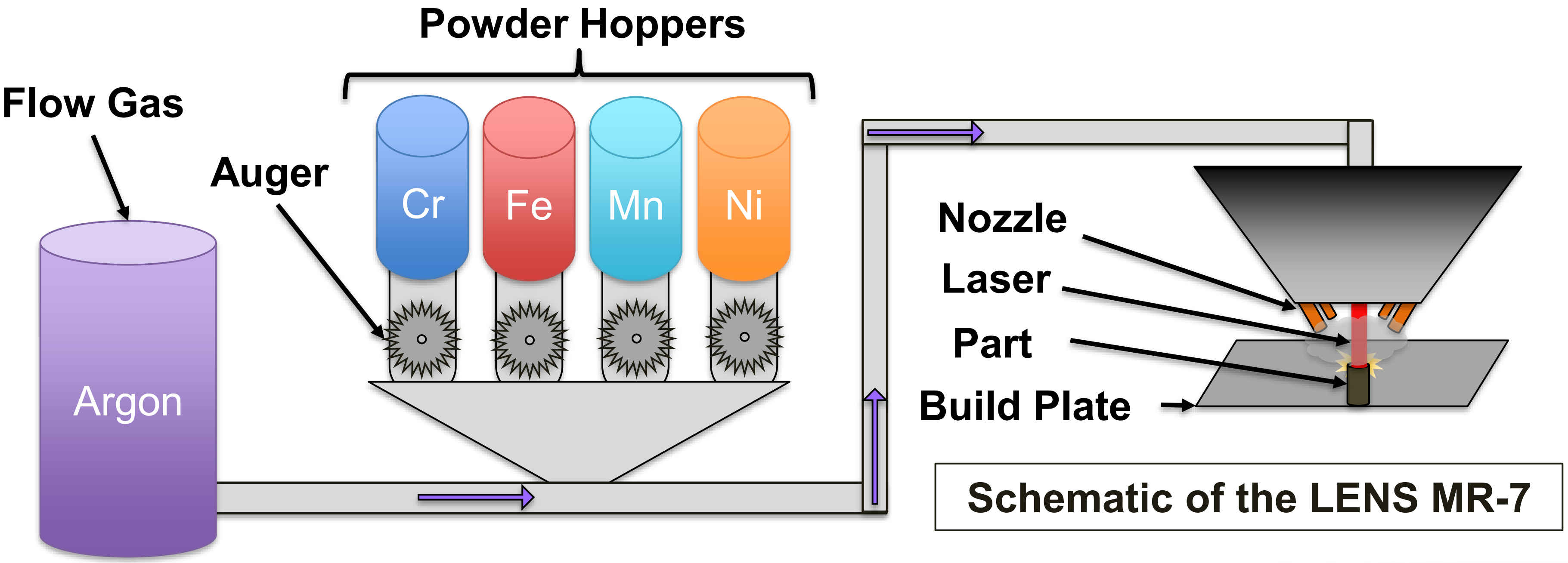
[1] E. Getto, K. Sun, A.M. Monterrosa, Z. Jiao, M.J. Hackett, G.S. Was, Void swelling and microstructure evolution at very high damage level in self-ion irradiated ferritic-martensitic steels, Journal of Nuclear Materials 480 (2016) 159–176.
[2] E. Getto, Z. Jiao, A.M. Monterrosa, K. Sun, G.S. Was, Effect of pre-implanted helium on void swelling evolution in self-ion irradiated HT9, Journal of Nuclear Materials 462 (2015) 458–469.
[3] M. Shimada, S. Nakahigashi, M. Terasawa, Swelling of Type 304 Stainless Steel Bombarded with 200 keV C+ Ions, J Nucl Sci Technol 13 (1976) 743–751.

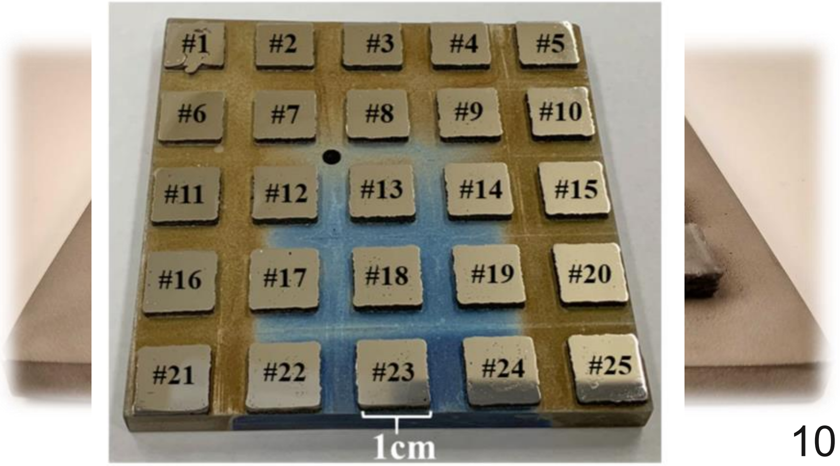
ML output: Thermal Conductivity Measurements (W case study)





In-situ Alloying Using DED: High Throughput Processing





- Elemental powders are controlled independently
- Powders are delivered to print head by argon flow gas
- Laser down optic axis melts powders

M. Moorehead, K. Bertsch, M. Niezgoda, C. Parkin, M. Elbakhshwan, K. Sridharan, C. Zhang, D. Thoma and A. Couet "High-throughput synthesis of Mo-Nb-Ta-W high-entropy alloys via additive manufacturing" Materials & Design, Vol. 187, 108358, Feb. 2020.

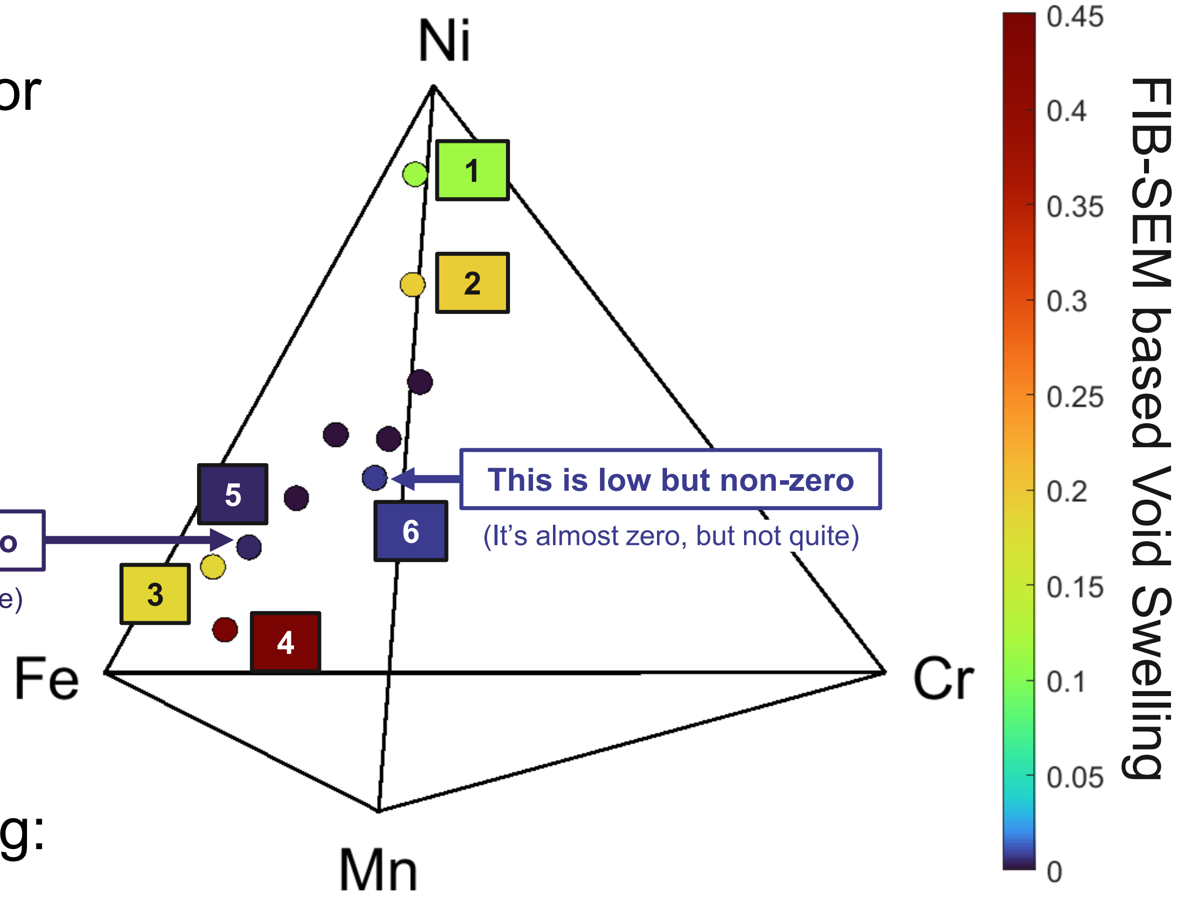
CrFeMnNi Quaternary Sample Results

Nanoscale Imaging and Analysis Laboratory

- 36 samples (3 campaigns of 12) have been performed, with characterization underway for campaigns 2 and 3

Sample	Sample Composition	Volume-Average Void Diameter (nm)	Void Population Density (nm^{-3})	FIB-SEM based Void Swelling (%)
1	$Cr_{4.0}Fe_{11.1}Mn_0Ni_{84.9}$	77 ± 0.2	$4.8 \cdot 10^{-12}$	0.11 ± 0.001
2	$Cr_{11.6}Fe_{22.3}Mn_0Ni_{66.1}$	105 ± 0.1	$3.2 \cdot 10^{-12}$	0.19 ± 0.002
3	$Cr_{5.2}Fe_{73.9}Mn_{2.3}Ni_{18.6}$	130 ± 0.5	$1.6 \cdot 10^{-12}$	0.18 ± 0.003
4	$Cr_{10.3}Fe_{76.9}Mn_{4.4}Ni_{8.4}$	149 ± 0.4	$2.6 \cdot 10$[illegible]	[illegible]
5	$Cr_{7.1}Fe_{65.1}Mn_{5.2}Ni_{22.6}$	46 ± 0.2	$9.5 \cdot 10^{-13}$	0.005 ± 0.0001
6	$Cr_{16}Fe_{38.2}Mn_{10.2}Ni_{35.6}$	81 ± 0.3	$2.9 \cdot 10^{-13}$	0.008 ± 0.0001

This is low but non-zero
(It's almost zero, but not quite)

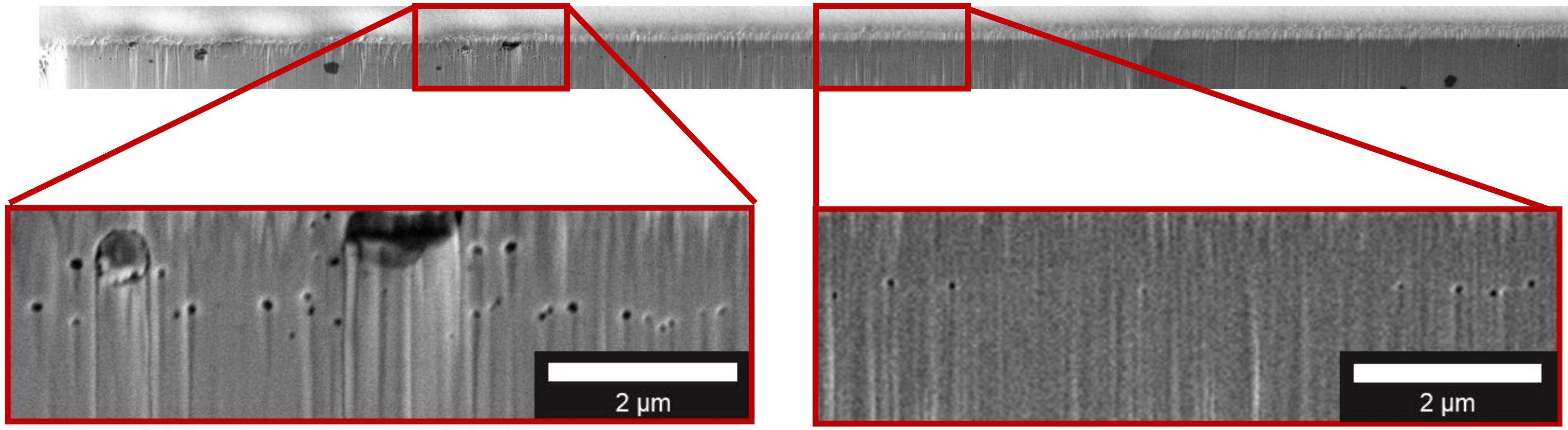


- Several samples closer to the center of the tetrahedron displayed little to no void swelling:
  - $Cr_9Fe_{40.8}Mn_{7.8}Ni_{42.2}$
  - $Cr_{9.4}Fe_{53.7}Mn_{5.8}Ni_{31.1}$
  - $Cr_{16.4}Fe_{38.2}Mn_{10.2}Ni_{35.6}$
  - $Cr_{14.2}Fe_{21.7}Mn_{11.8}Ni_{52.3}$

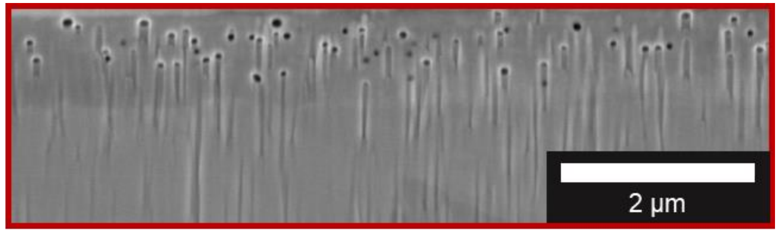
Nanoscale Imaging
and Analysis
Laboratory

CrFeMnNi Quaternary Sample Results

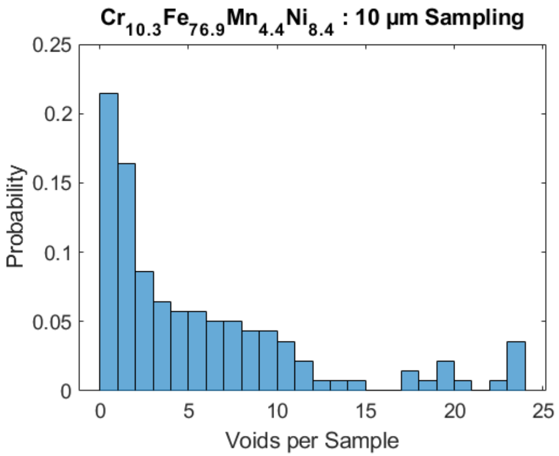
$Cr_{10.3}Fe_{76.9}Mn_{4.4}Ni_{8.4}$ – 600 °C, 250 dpa

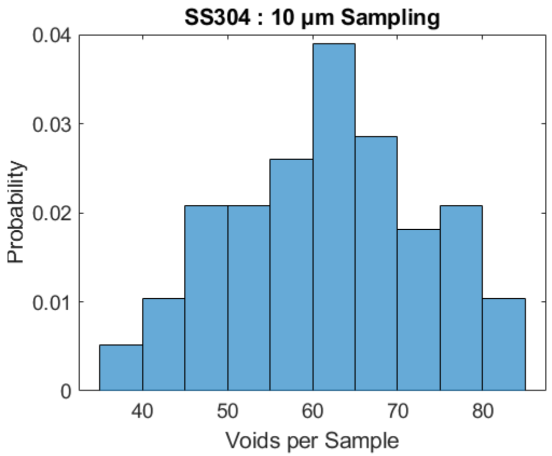


SS304 – 605 °C, 150 dpa



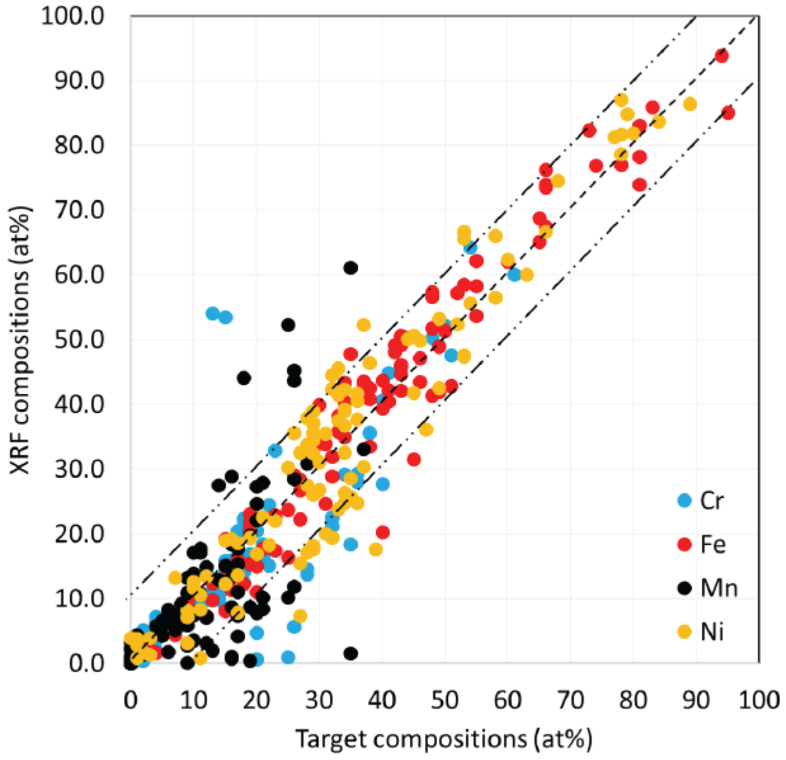
- Samples which contained void swelling would often also have regions of no void swelling *within the same grain*.
- This inconsistency needs further investigation, since especially since it presents issues with heavy sampling bias when taking TEM lamellae





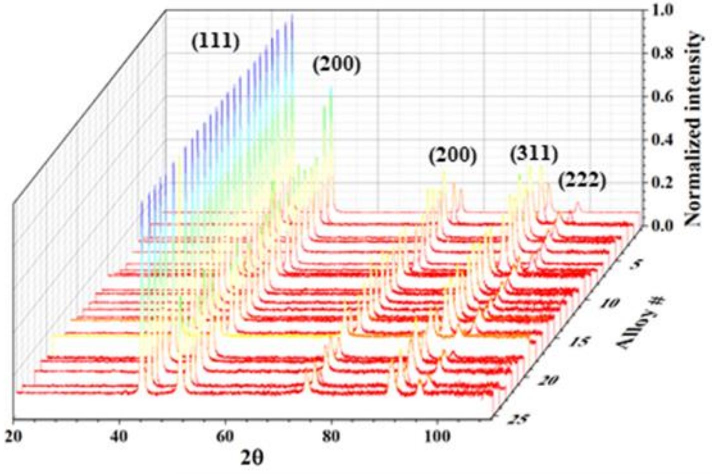
Automated Characterization for ML Featurization

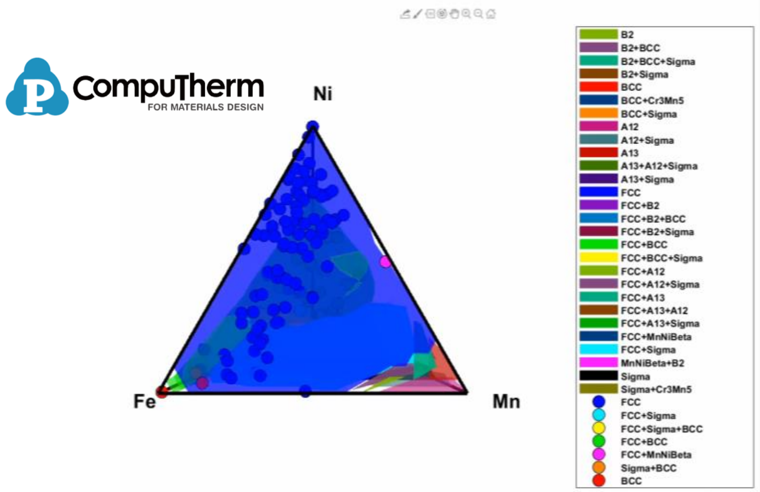
XRF measurements



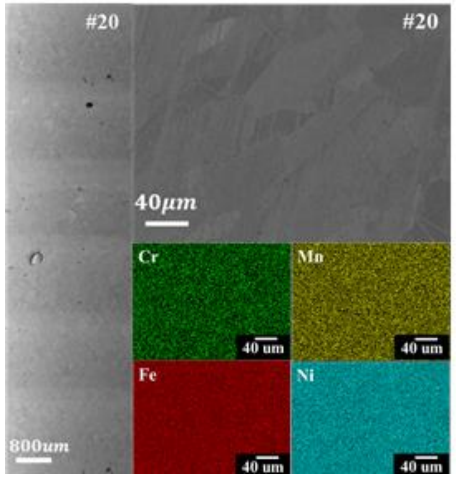
The target vs. XRF-measured compositions of the Cr-Fe-Mn-Ni alloys

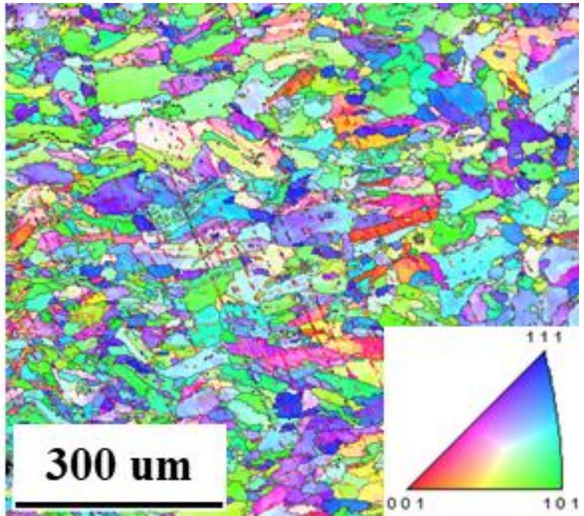
Automated XRD for microstructure analysis



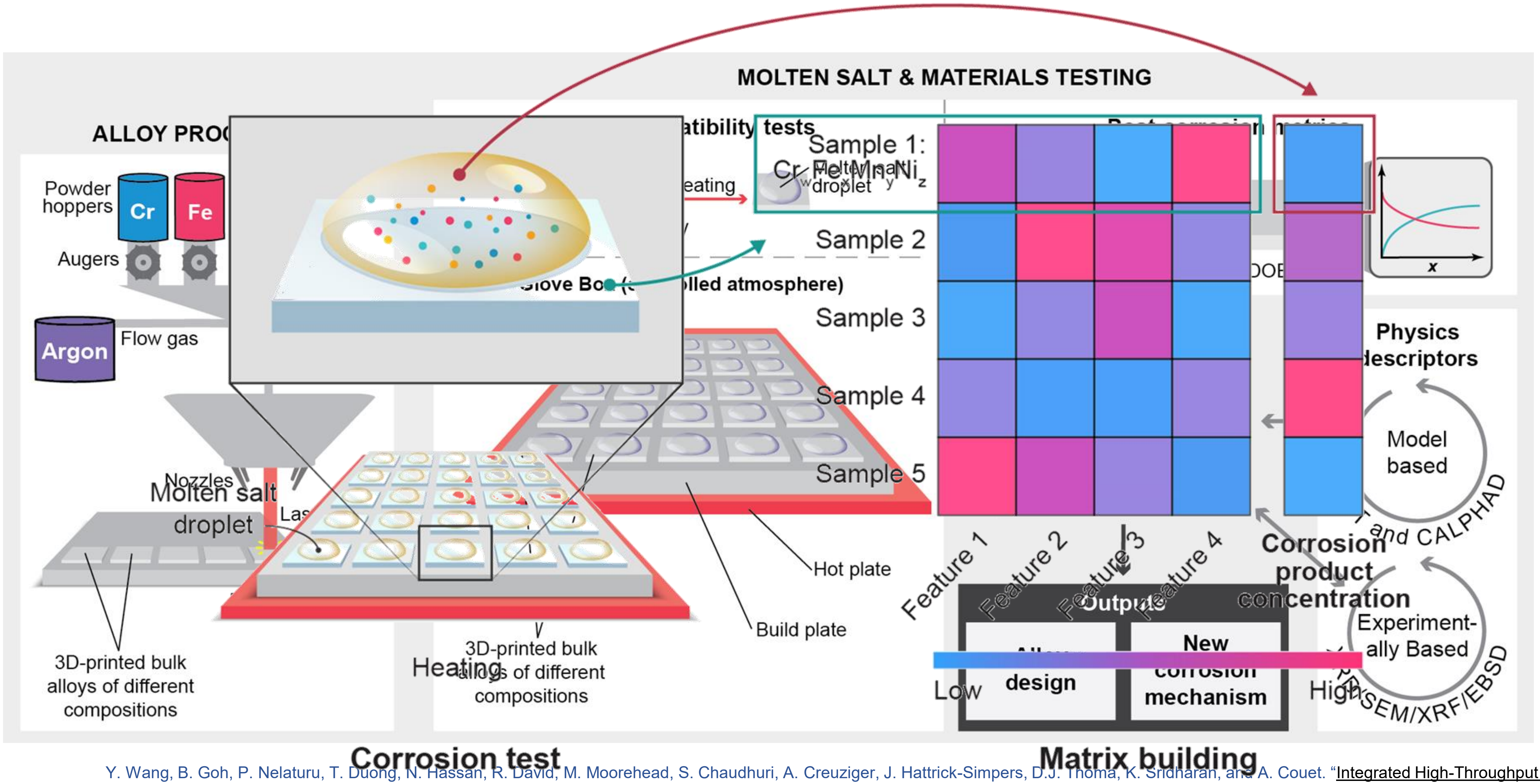


Automated SEM/EDS maps of polished surfaces





Physical Parametrization for Interpretable Machine Learning Predictions



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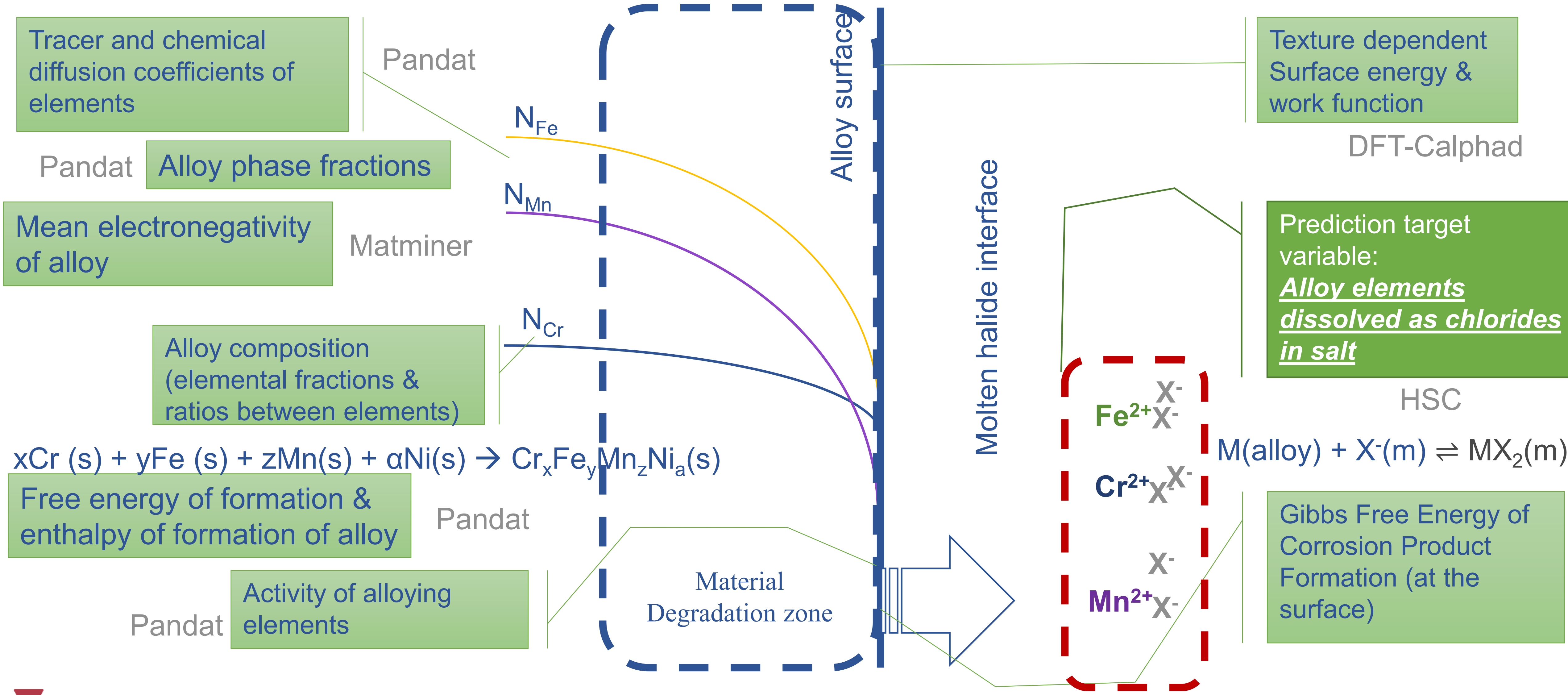
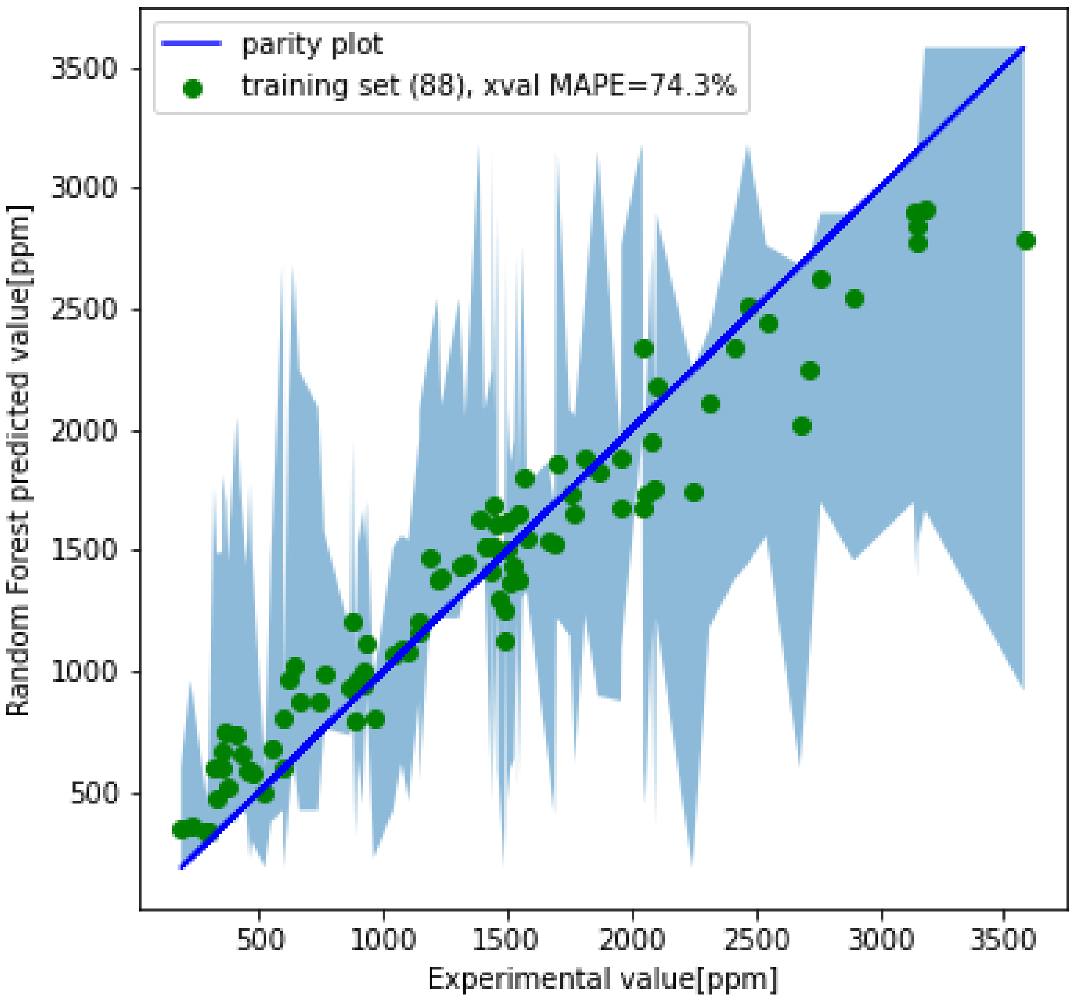
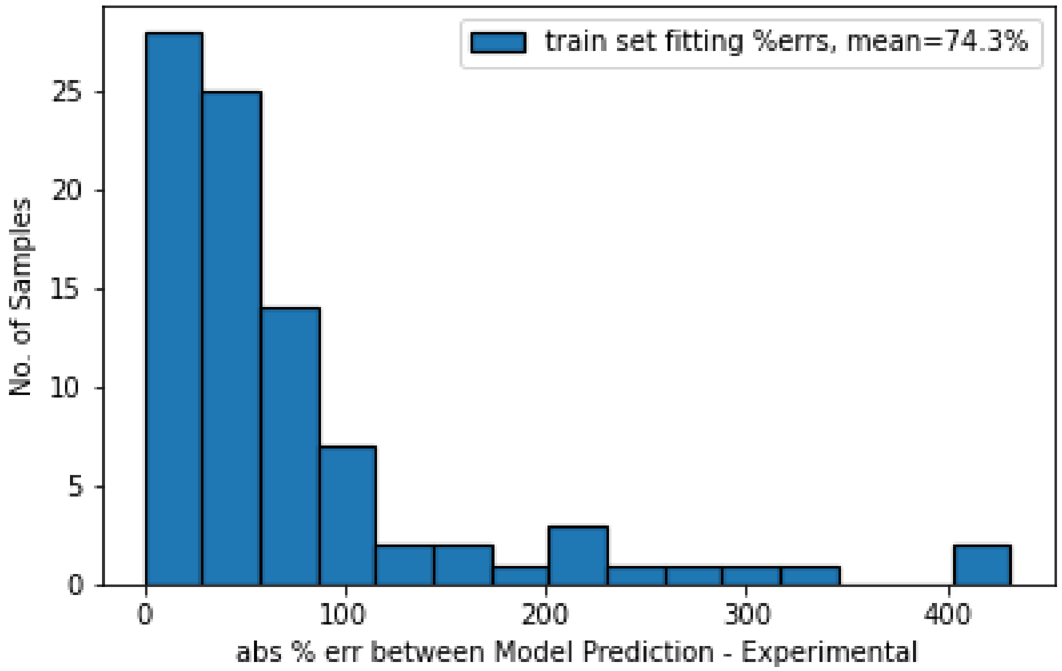
Y. Wang, B. Goh, P. Nelaturu, T. Duong, N. Hassan, R. David, M. Moorehead, S. Chaudhuri, A. Creuziger, J. Hattrick-Simpers, D.J. Thoma, K. Sridharan, and A. Couet. "Integrated High-Throughput and Machine Learning Methods to Accelerate Discovery of Molten Salt Corrosion-Resistant Alloys." **Advanced Science**. 7 May 2022.
B. Goh, Y. Wang, P. Nelaturu, H. Zhang, M. Moorehead, T. Duong, P. Priya, D. Thoma, S. Chaudhuri, J. Hattrick-Simpers, K. Sridharan, and A. Couet. "Nobility vs. mobility: Insights into molten salt corrosion mechanisms of high-entropy alloys via high-throughput experiments and machine learning." **Matter**. June 2024.

77 FEATURES TO PREDICT CORROSION



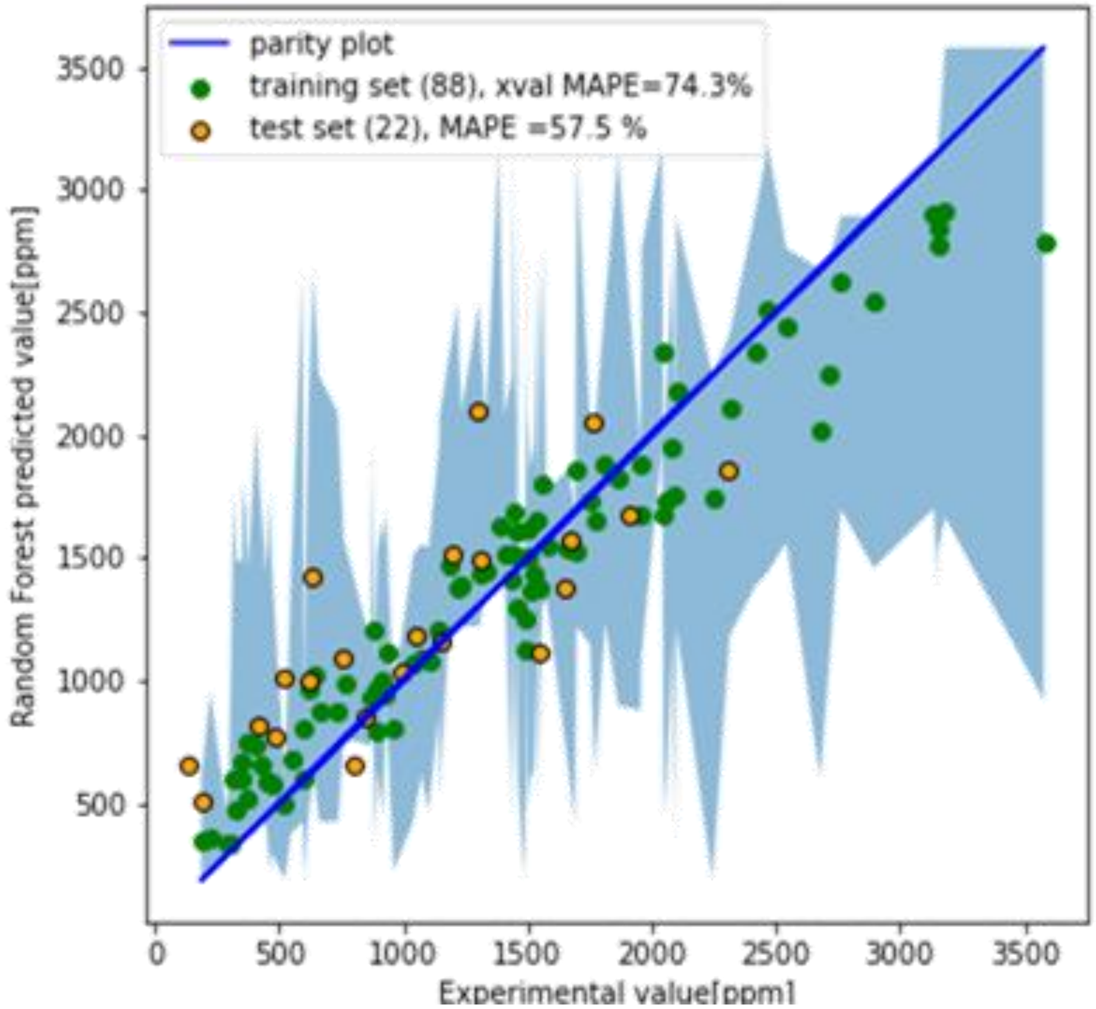
Machine Learning to Predict Corrosion Results

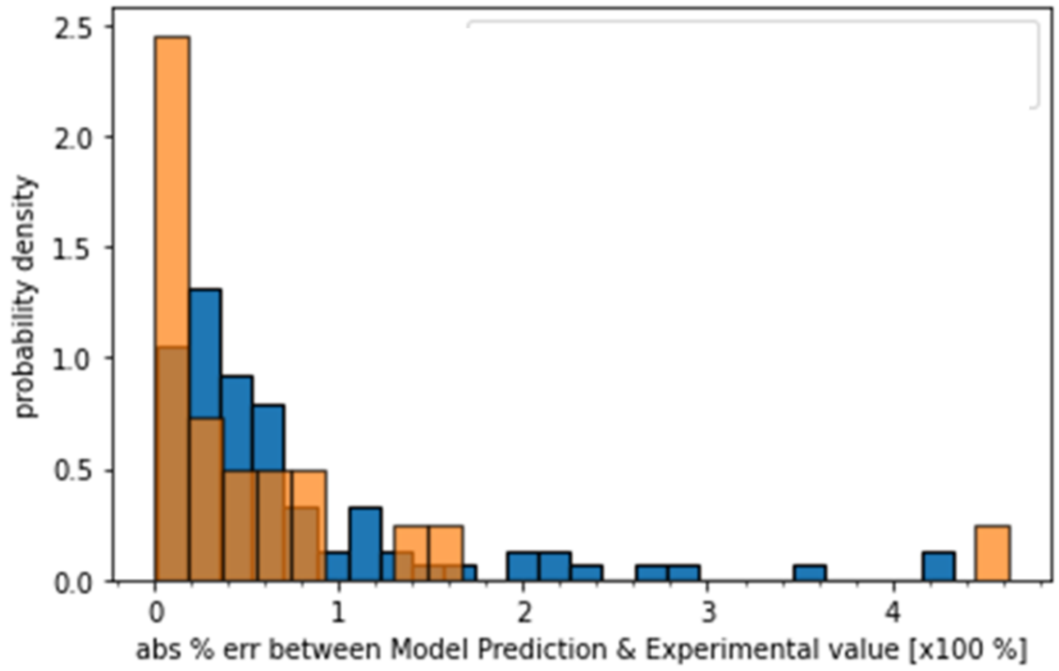




- Random Forest Regressor trained on 88 samples.
- Mean Absolute Percent Error (MAPE) was estimated by 5-fold cross-validation (~74%).
- Majority of samples well-fitted (<50% absolute error).

Machine Learning to Predict Corrosion Results



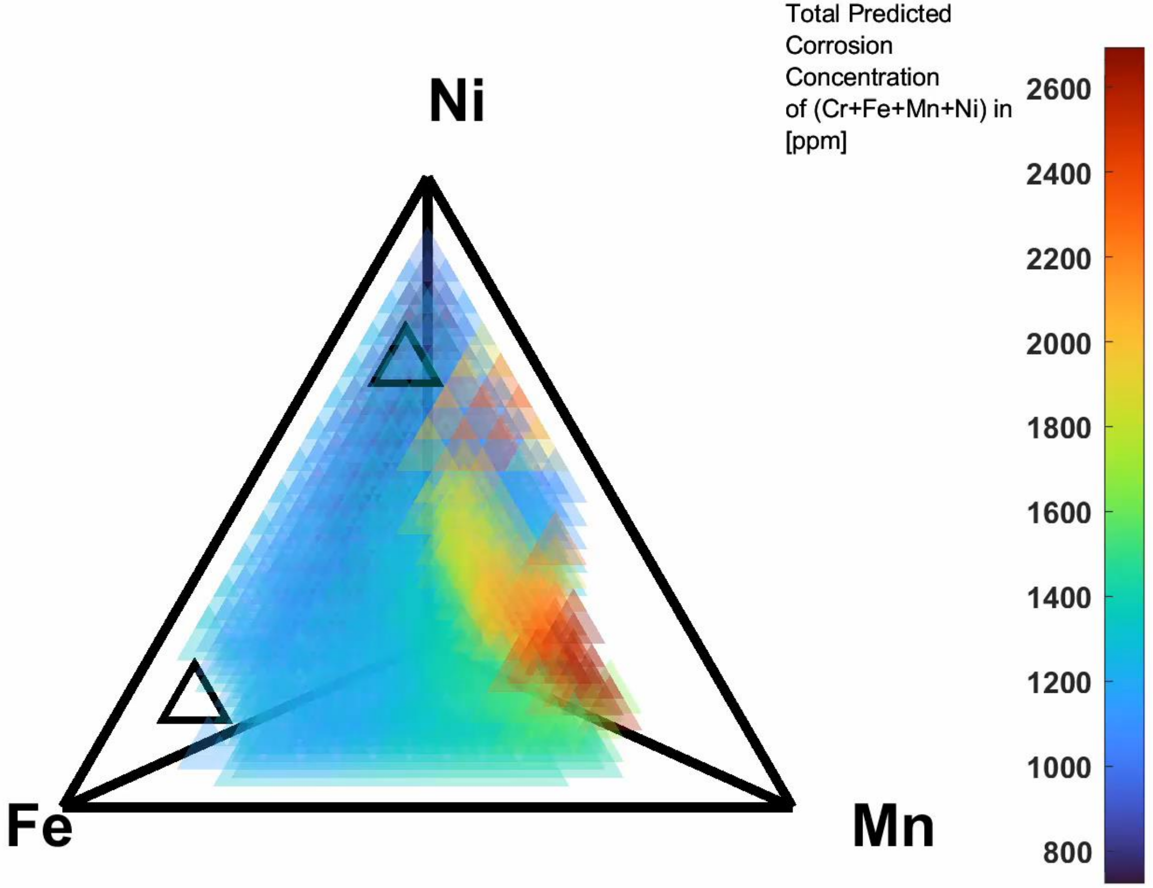


- Test set was within the training set estimation for out-of-bag samples, in fact it is improved: 74.3% down to 57.5%

→ With larger training dataset, we can likely approach experimental standard deviation of 14%

ML Model Predictions beyond compositions that were tested

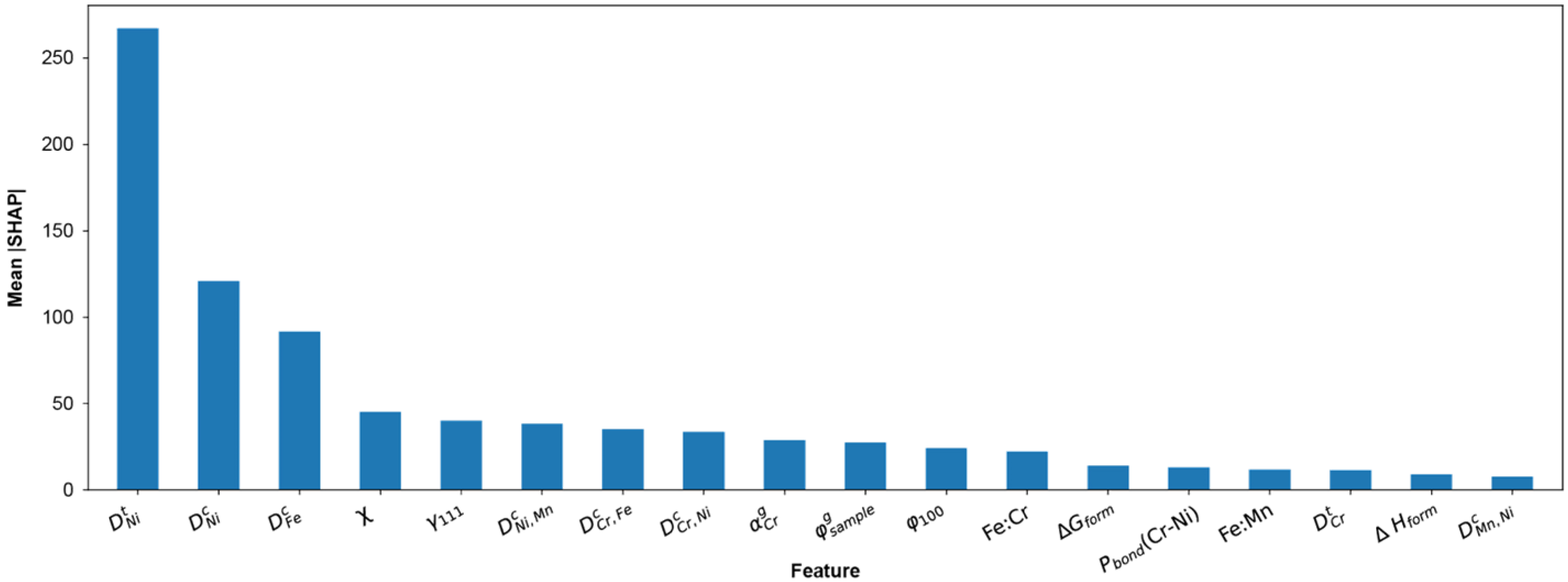
1. Grid of compositions in (Cr,Fe,Mn,Ni) set up with mesh size 5at%
2. Feature matrix generated using reduced feature space.
3. This data is inserted into the previously trained RFR to predict corrosion behavior using the same target variable
4. Results are plotted in 4D composition space.
5. SS316 – near Fe corner
6. Hastelloy N composition – near Ni corner



SS 316 is outside of RFR fitted model scope, Hastelloy N projection is in low-corrosion region.

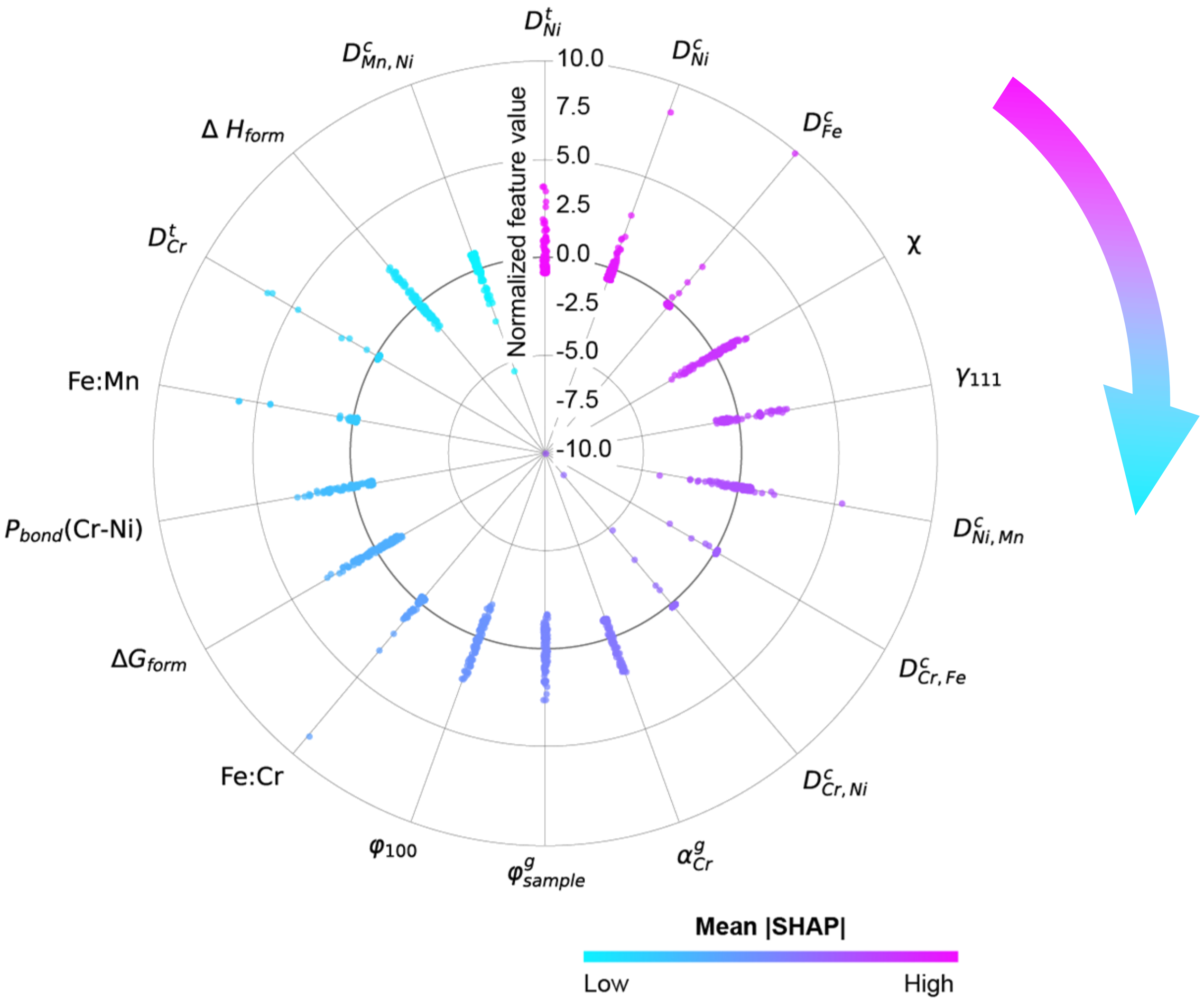
Interpretation of Machine Learning Model: SHAP Theory

- Use SHAP theory to highlight feature importance → feature reduction (X features account for 75% of each sample's ML prediction value).
- Reduction from 75 $\xrightarrow{\text{Spearman} < 94\%}$ → 52 $\xrightarrow{\text{SHAP} > 75\%}$ 18 features
- MAPE evolution 56% → 56% → 64%



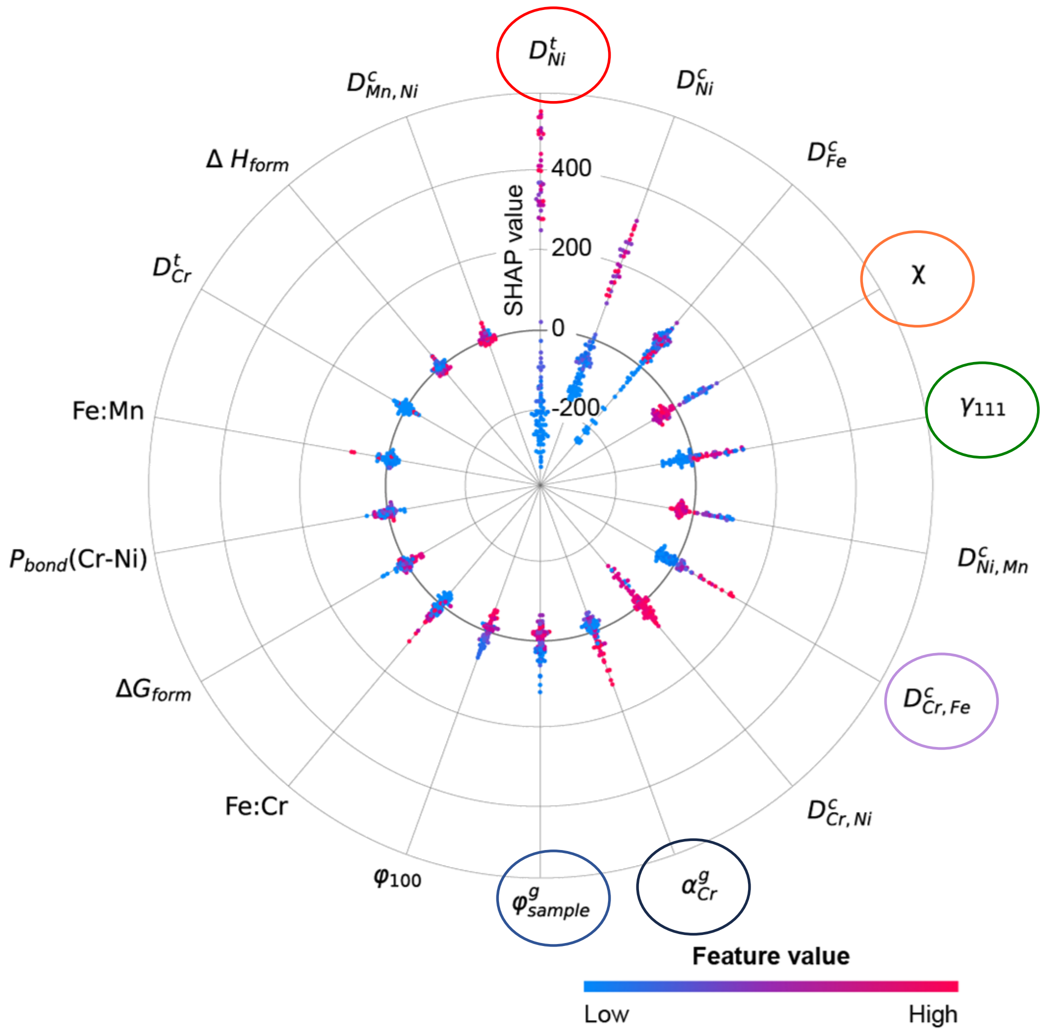
Interpretation of Machine Learning Model

- Verification that these features are well distributed:



INTERPRETATION OF MACHINE LEARNING MODEL

- Analysis of each feature's importance on the corrosion rate:



Some interpretations of the ML model **<u>were expected</u>**:

- ↗ a_{Cr} → ↗ corrosion rate
- ↗ Surface energy → ↗ to corrosion rate
- ↗ D_{Cr} in Fe →↗ to corrosion rate
- ↗ Electronegativity → ↘ to corrosion rate
- ↗ Work function → ↘ to corrosion rate

- But the **<u>most important feature</u>** was not expected:

- ↗ Ni diffusion coefficient → ↗ ↗ corrosion rate

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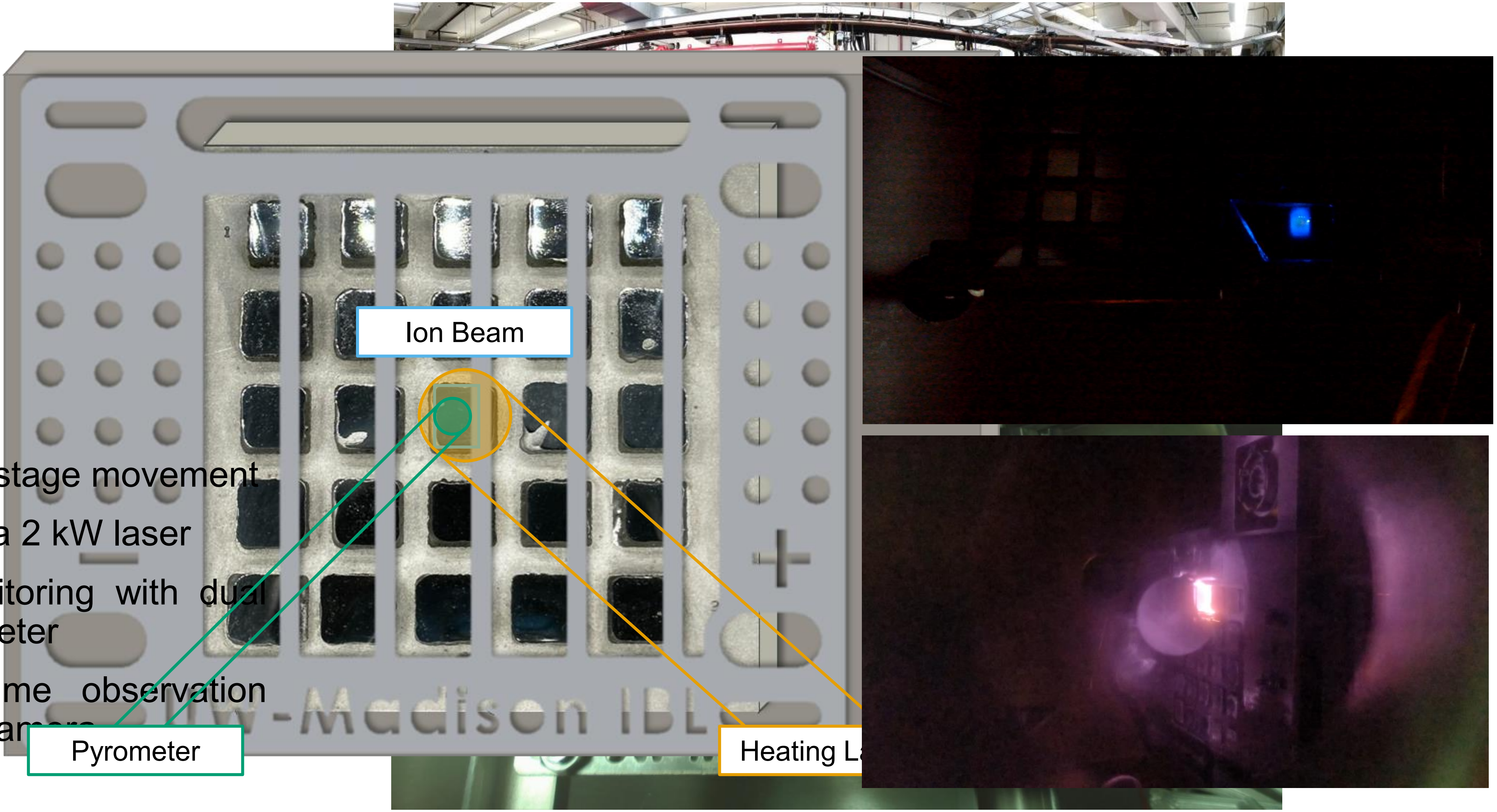
Question Time

Thank You

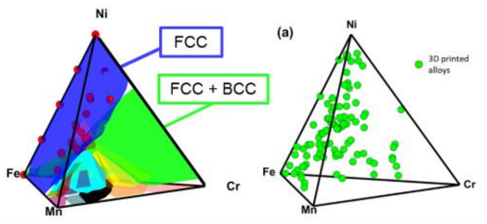
High-Throughput Irradiation – UW Ion Beam Lab

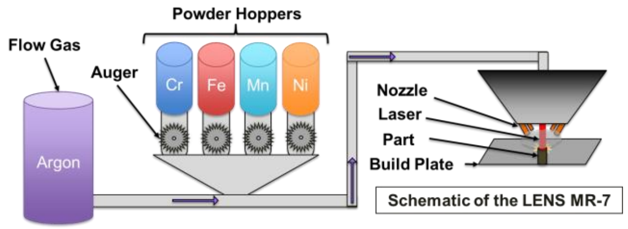


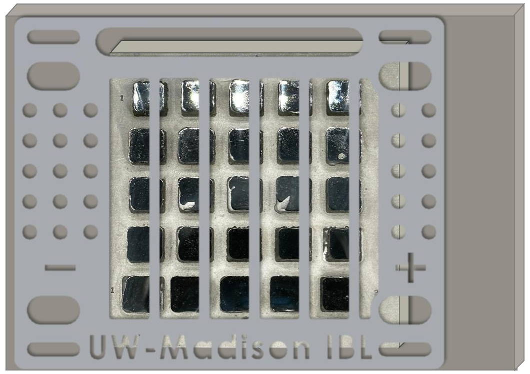
- 150mm x 200mm stage movement
- Sample heating via 2 kW laser
- Temperature monitoring with dual wavelength pyrometer
- Irradiation real time observation from in-chamber camera

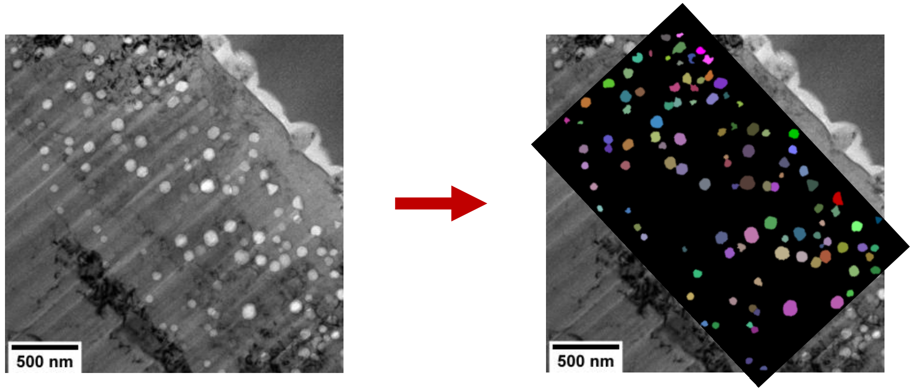
Current work, continuing work

- Additively manufactured 100 CrFeMnNi and 50 CrFeMoNi MPEAs
- Developed a high-throughput beamline setup for rapid irradiation
- Employed rapid characterization techniques to increase data acquisition and processing rates
- Corrosion testing will begin following irradiation testing and characterization
- Quantifiable data from this work will serve as the basis for training a machine learning model for future alloy design







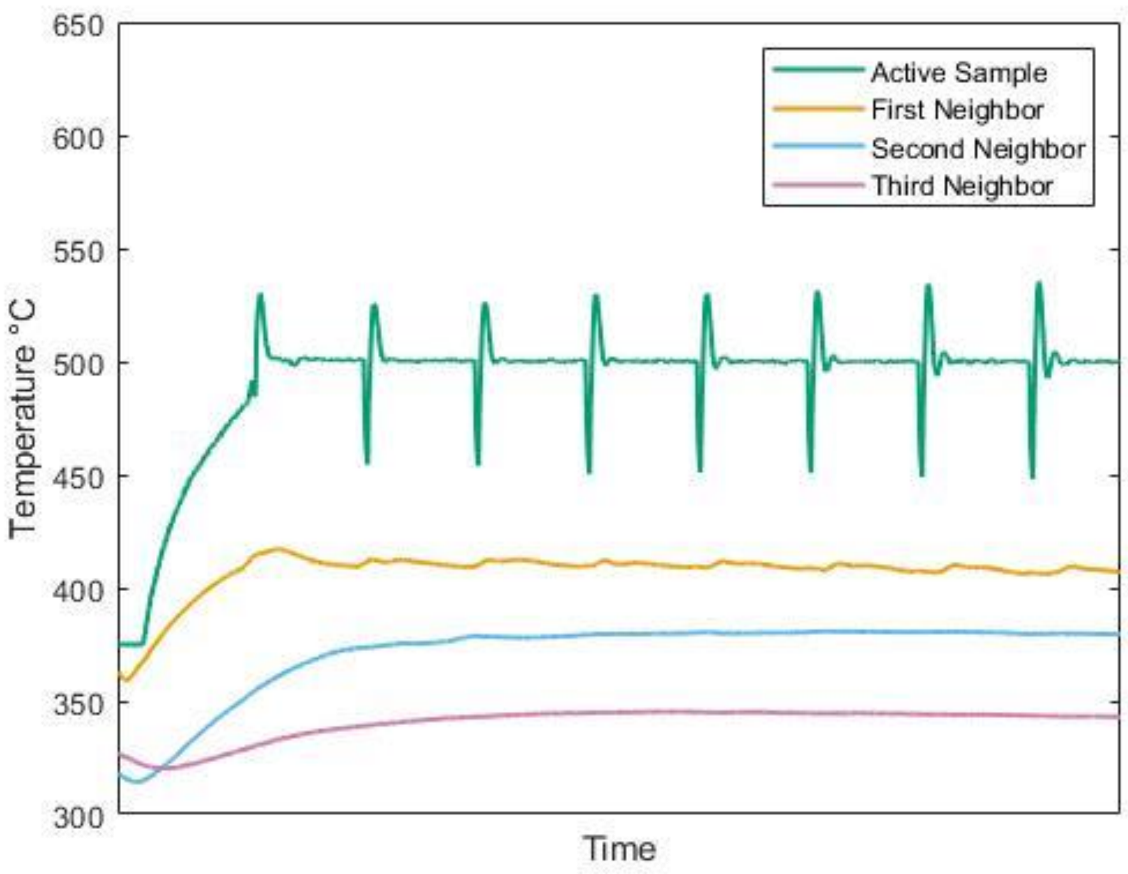


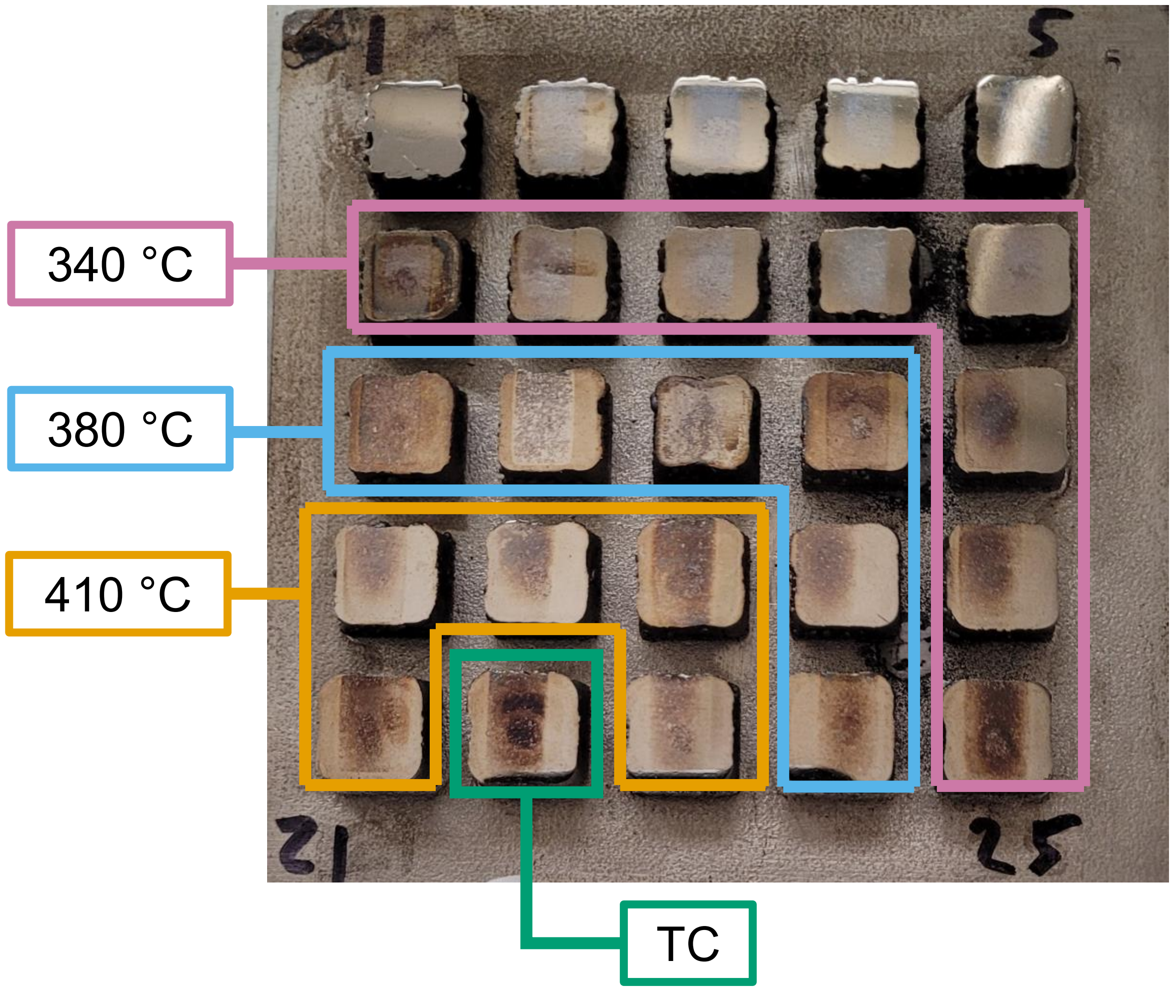
[1] M. Moorehead, et al., "High-throughput ion irradiation of additively manufactured compositionally complex alloys," Journal of Nuclear Materials, vol. 547, pp. 152782, Apr. 2021.
[2] C. Parkin, M. Moorehead, M. Elbakhshwan, X. Zhang, P. Xiu, L. He, M. Bachhav, K. Sridharan, and A. Couet, "Phase stability, mechanical properties, and ion irradiation effects in face-centered cubic FeCrMnNi compositionally complex solid-solution alloys at high temperatures," Journal of Nuclear Materials, vol. 565, pp. 153733, 2022.

List of publications from UW-MaDCoR with RTE/NSUF (since 2019)

1. Z. Yu, M. Bachhav, F. Teng, L. He, M. Dubey, and A. Couet. "STEM/EDS and APT study on the microstructure and microchemistry of neutron irradiated ZIRLOTM." **Journal of Nuclear Materials**. January 2023
2. C. Parkin, M. Moorehead, M. Elbakhshwan, X. Zhang, P. Xiu, L. He, M. Bachhav, K. Sridharan, and A. Couet. "Phase stability, mechanical properties, and ion irradiation effects in face-centered cubic CrFeMnNi compositionally complex solid-solution alloys at high temperatures." **Journal of Nuclear Materials**, pp. 153733. 17 Apr. 2022.
3. F. Pellemoine, K. Ammigan, S. Bidhar, A. Couet, T. Ishida, S. Makimura, M. Moorehead, and K. Yonehara. "Novel materials to improve High Power Target reliability." **Snowmass**, 2022
4. A. Couet "Integrated high-throughput research in extreme environments targeted toward nuclear structural materials discovery" **Journal of Nuclear Materials**, Vol. 559,Pp. 153425, Feb. 2022
5. M. Moorehead, B. Queylat, H. Zhang, K. Kriewaldt and A. Couet "Development of a versatile, high-temperature, high-throughput ion irradiation system", **Nuclear Instruments and Methods in Physics Research section A**, Oct. 2021
6. Z. Yu, M. Bachhav, F. Teng, L. He and A. Couet" Nanoscale redistribution of alloying elements in high-burnup AXIOM-2 (X2®) and their effects on in-reactor corrosion" **Corrosion Science**, Vol.195, pp 109658, September 2021
7. M. Moorehead, P. Nelaturu, M. Elbakhshwan, C. Parkin, C. Zhang, K. Sridharan, D. Thoma and A. Couet "High-Throughput Ion Irradiation of Additively Manufactured Compositionally Complex Alloys" **Journal of Nuclear Materials**, 152782, Jan. 2021
8. Z. Yu, J. Werden, N. Capps, K. Linton and A. Couet " (S)TEM/EDS study of native precipitates and irradiation induced Nb-rich platelets in high-burnup M5® " **Journal of Nuclear Materials**, 152667, Dec. 2020
9. C. Parkin, M. Moorehead, M. Elbakhshwan, J. Hu, W-Y. Chen, M. Li, L, He, K. Sridharan and A. Couet "In situ microstructural evolution in face-centered and body-centered cubic complex concentrated solid-solution alloys under heavy ion irradiation" **Acta Materiala**, Vol. 198, Pp. 85-99, Oct. 2020.
10. Z. Yu, T. Kim, M. Bachhav, X. Liu, L. He and A. Couet "Effect of proton pre-irradiation on corrosion of Zr-0.5Nb model alloys with different Nb distributions" **Corrosion Science**, Vol. 173, 108790, August. 2020
11. A. Kareer, J.C. Waite, B. Li, A.Couet, D.E.J.Armstrong and A.J. Wilkinson "Short communication: 'Low activation, refractory, high entropy alloys for nuclear applications" **Journal of Nuclear Materials**, Vol. 526, 151744, December 2019.
12. Zefeng Yu, Chenyu Zhang, Paul M.Voyles, Lingfeng He, XiangLiu, KellyNygren and Adrien Couet "Microstructure and microchemistry study of irradiation-induced precipitates in proton irradiated ZrNb alloys" **Acta Materialia**, Vol. 178, pp. 228-240, October 2019.
13. Z. Yu, A. Couet and M. Bachhav "Irradiation-induced Nb redistribution of ZrNb alloy: An APT study" **Journal of Nuclear Materials**, vol. 516, 1 April 2019, pp. 100-110.

High-Throughput Troubleshooting

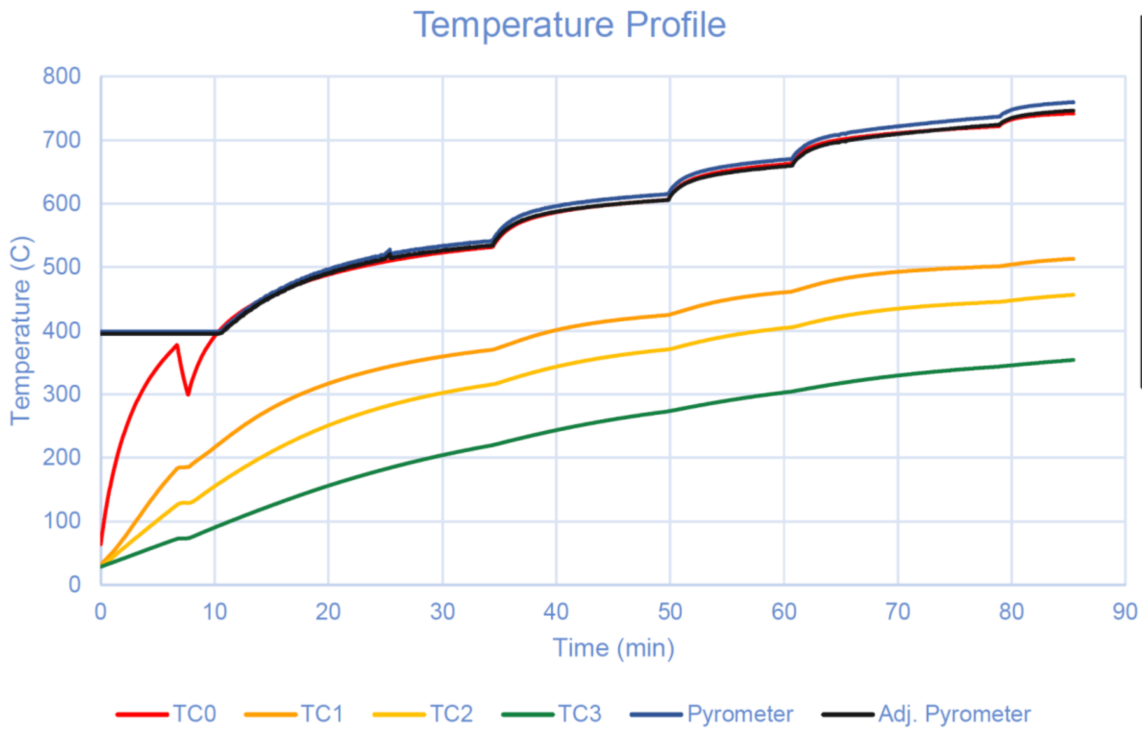


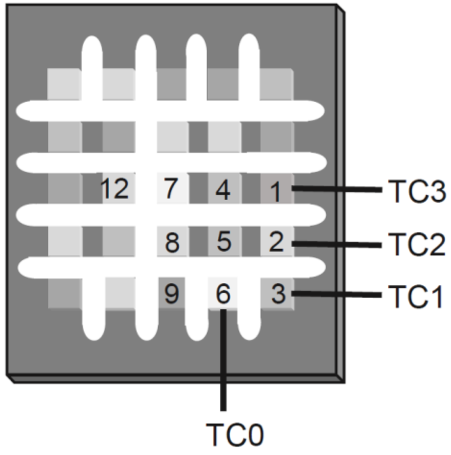


Heat Test

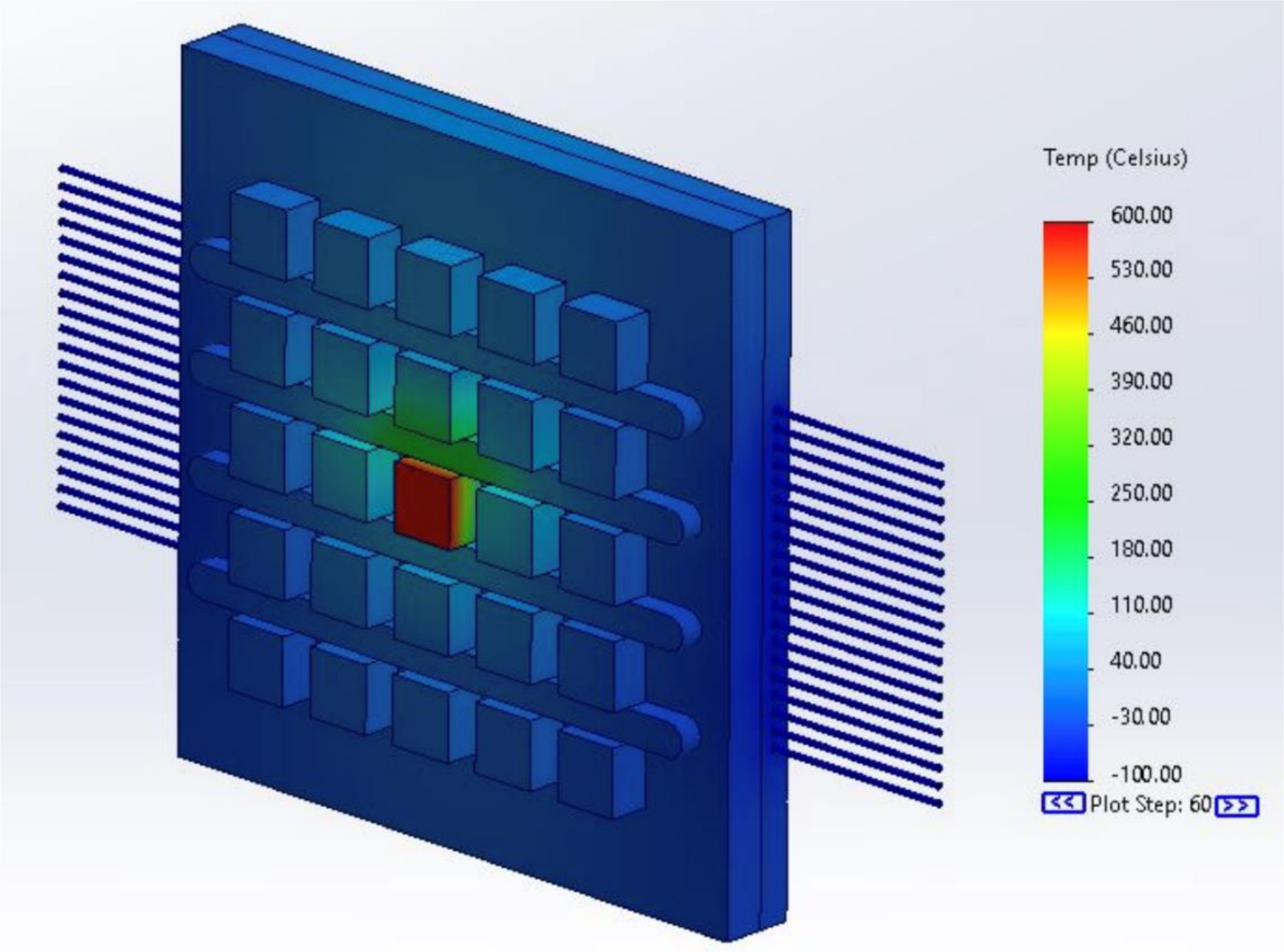
Results

- Heating sample 6 (TC0) (3/11)
- Error < 5°C above 440°C
- Each bump is LP = [40, 50, 60, 70, 80]%
- [New adj. Pyro]=0.7434*[Pyro]+116





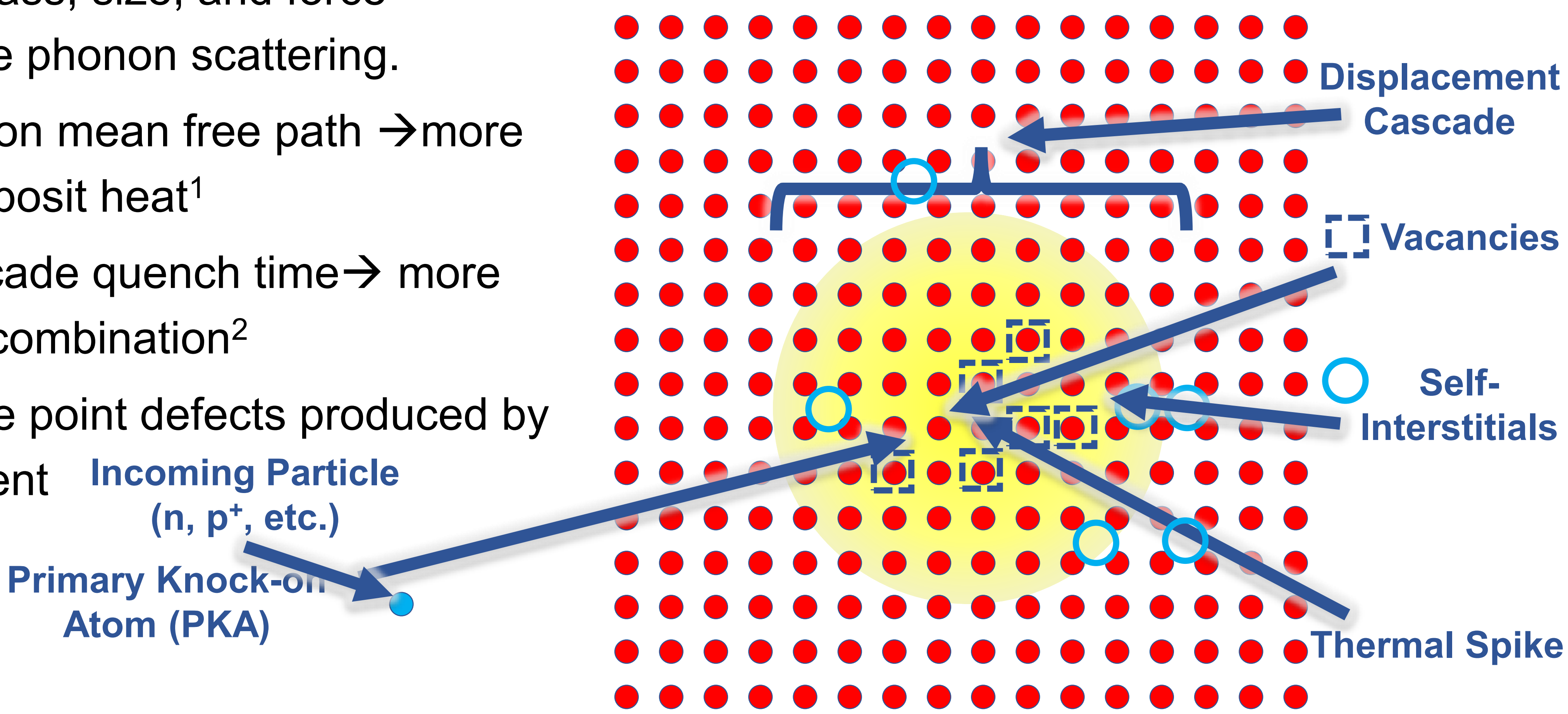
Heat Test


Temp (Celsius)
600.00
530.00
460.00
390.00
320.00
250.00
180.00
110.00
40.00
-30.00
-100.00
Plot Step: 60

CCA irradiation resistance hypothesis

Variable atomic mass, size, and force constants increase phonon scattering.

- Lower phonon mean free path →more confined deposit heat[1]
- Longer cascade quench time→ more athermal recombination[2]
- Fewer stable point defects produced by cascade event

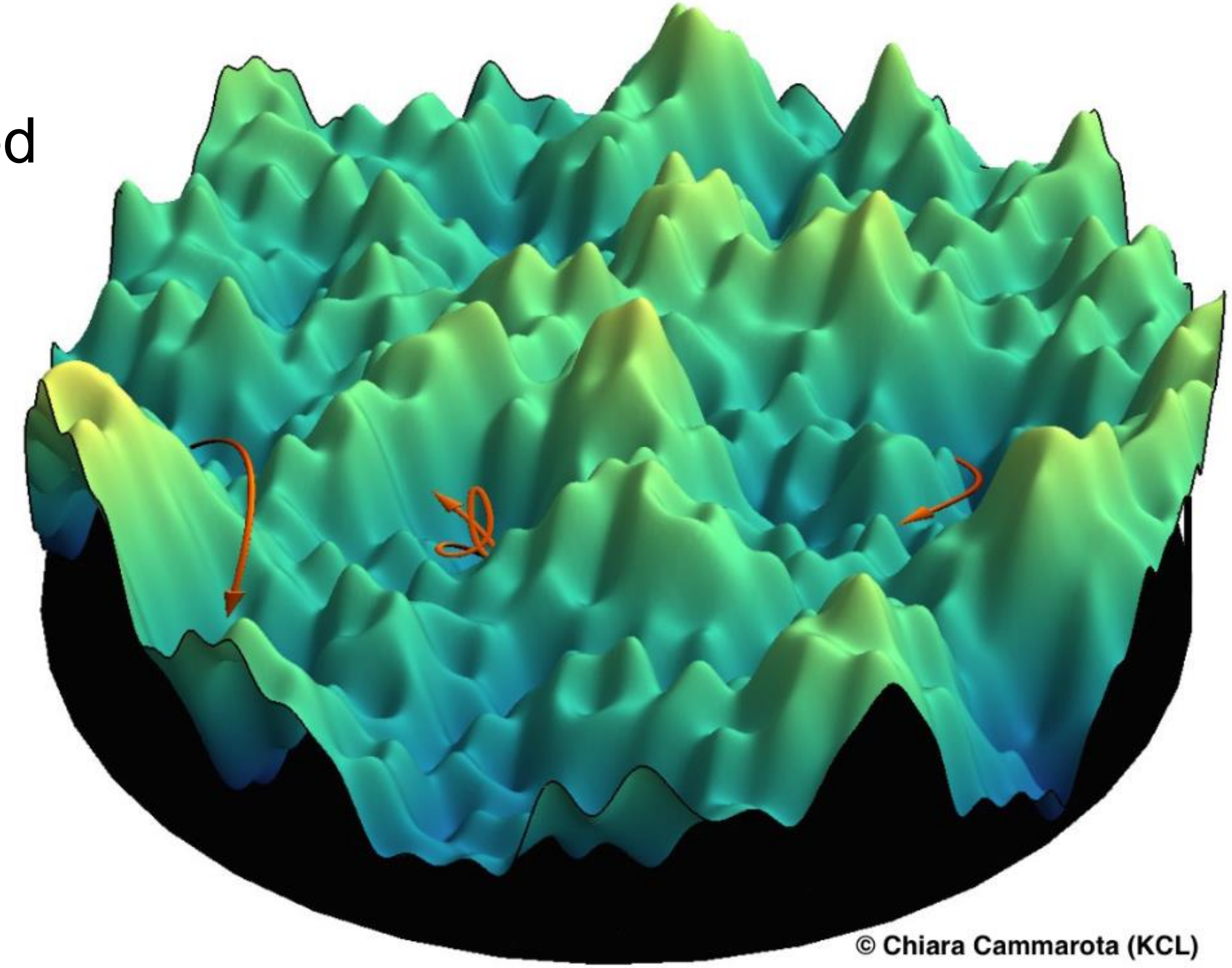


1. F. Granberg, K. Nordlund, M. Ullah, K. Jin, C. Lu, H. Bei, L.M. Wang, F. Djurabekova, W.J. Weber, Y. Zhang, Mechanism of Radiation Damage Reduction in Equiatomic Multicomponent Single Phase Alloys, Physical Review Letters (2015).
2. L.K. Béland, G.D. Samolyuk, R.E. Stoller, A. Caro, Lattice thermal conductivity of multi-component alloys, Journal of Alloys and Compounds 648 (2015) 408 413
3. Animation by Michael Moorehead

CCA irradiation resistance hypothesis

Complex energy landscape and distorted lattice reduce point defect / cluster mobility

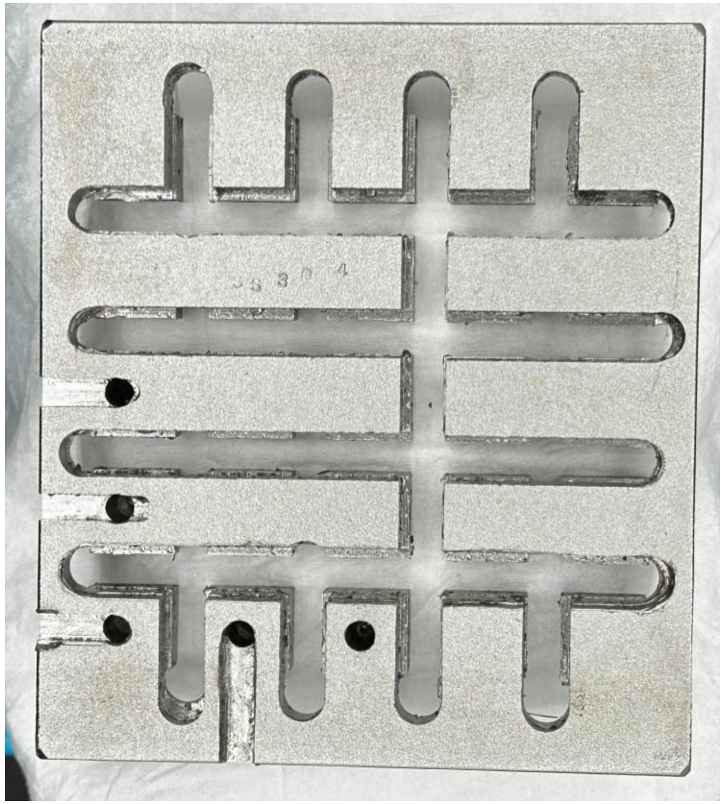
- Increased average migration energy for atoms[1]
- Immobile interstitial loops slow vacancy saturation[2]

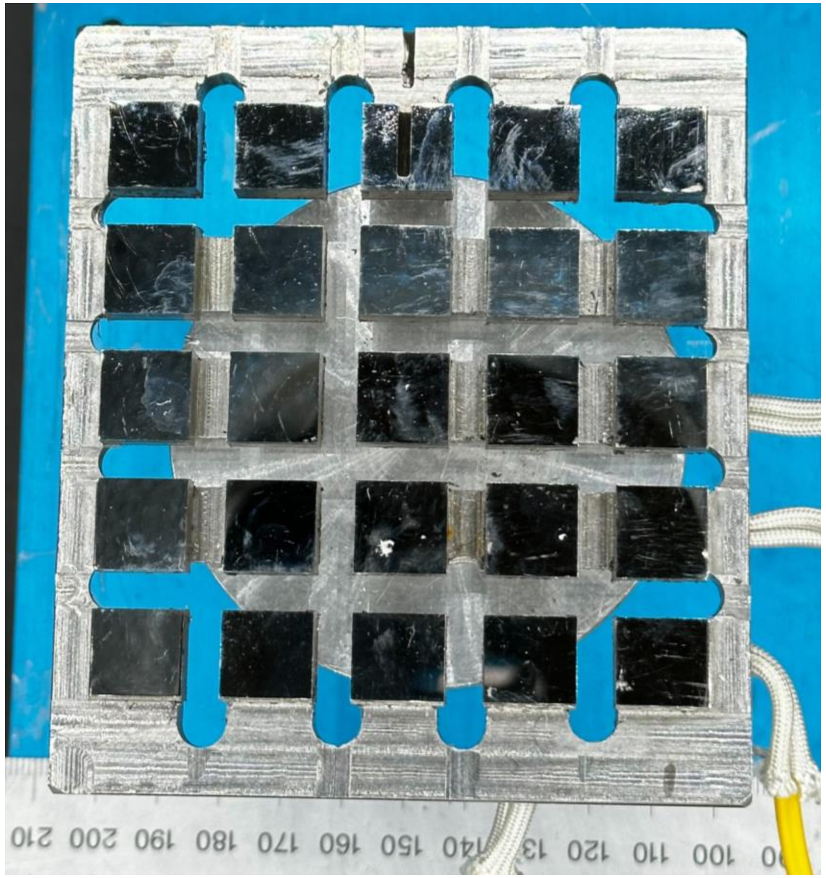


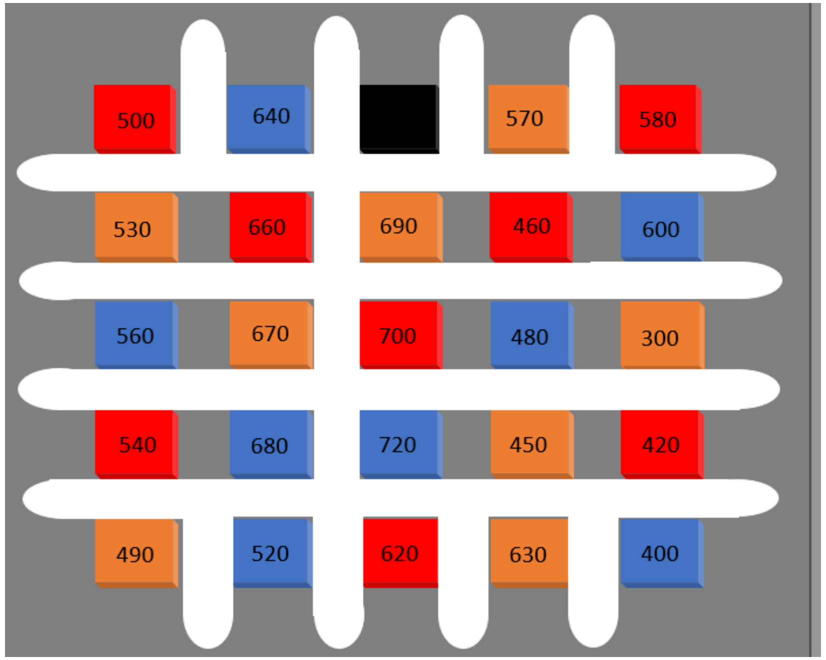
1. J.-W. Yeh, Physical Metallurgy of High-Entropy Alloys, JOM 67(10) (2015) 2254-2261.
2. C. Lu, L. Niu, N. Chen, K. Jin, T. Yang, P. Xiu, Y. Zhang, F. Gao, H. Bei, S. Shi, M.-R. He, I.M. Robertson, W.J. Weber, L. Wang, Enhancing radiation tolerance by controlling defect mobility and migration pathways in multicomponent single-phase alloys, Nature Communications 7 (2016) 13564.

Sample

- SS304 plate machined to have trenches to promote thermal isolation between samples

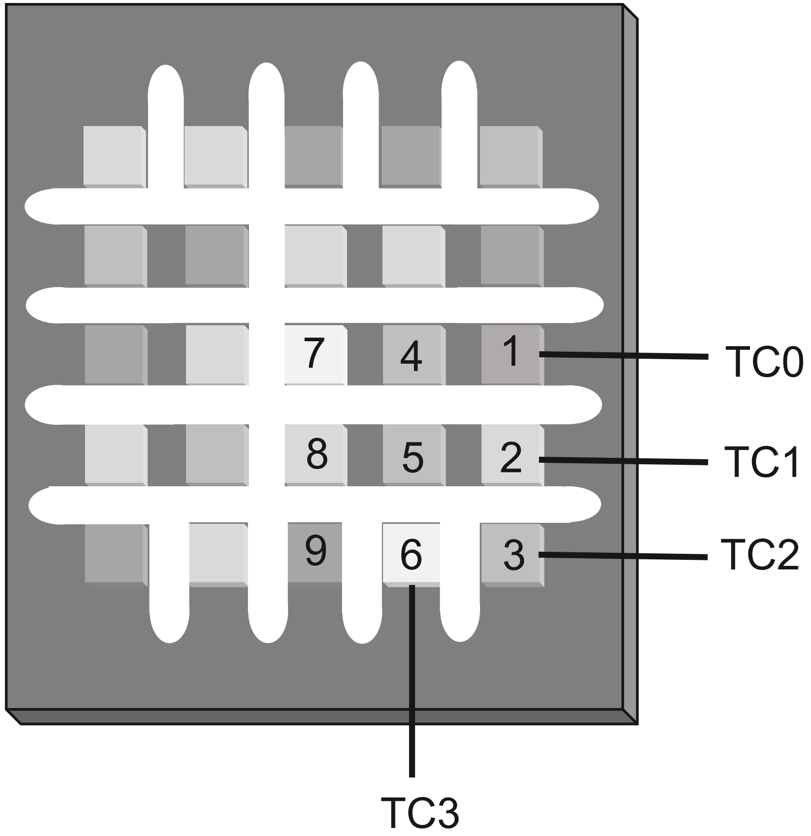






Heat Test

- TCs placed in samples 1, 2, 3, 6 [TC0, TC1, TC2, TC3]
  - ~<1 mm below the surface
- 5 tests:
  - 4 tests to keep each TC at 600°C until steady state for >30 min.
  - 1 test setting pyrometer at 600°C on sample 7 until steady state for >30 min
- TC2 (sample 3) had bad connection – readings not accurate



Heat Test

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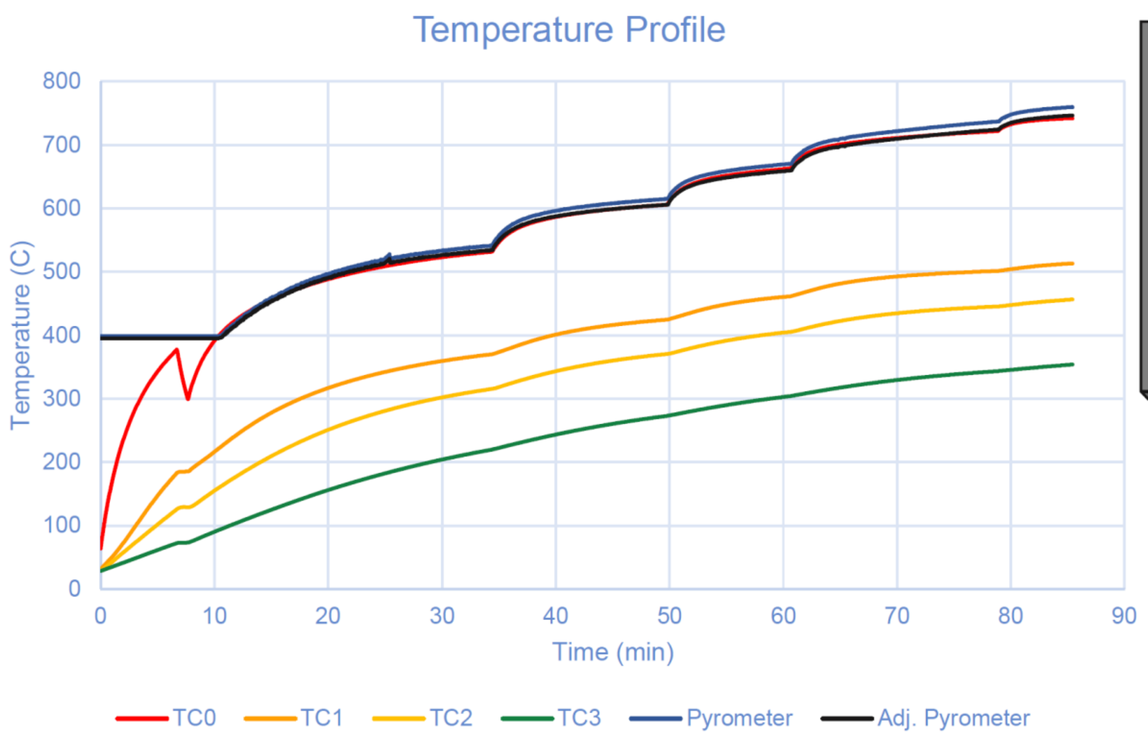
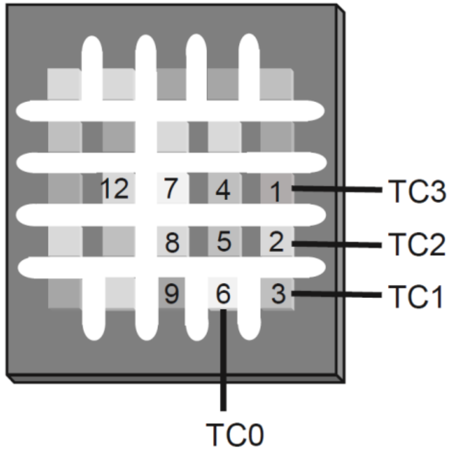
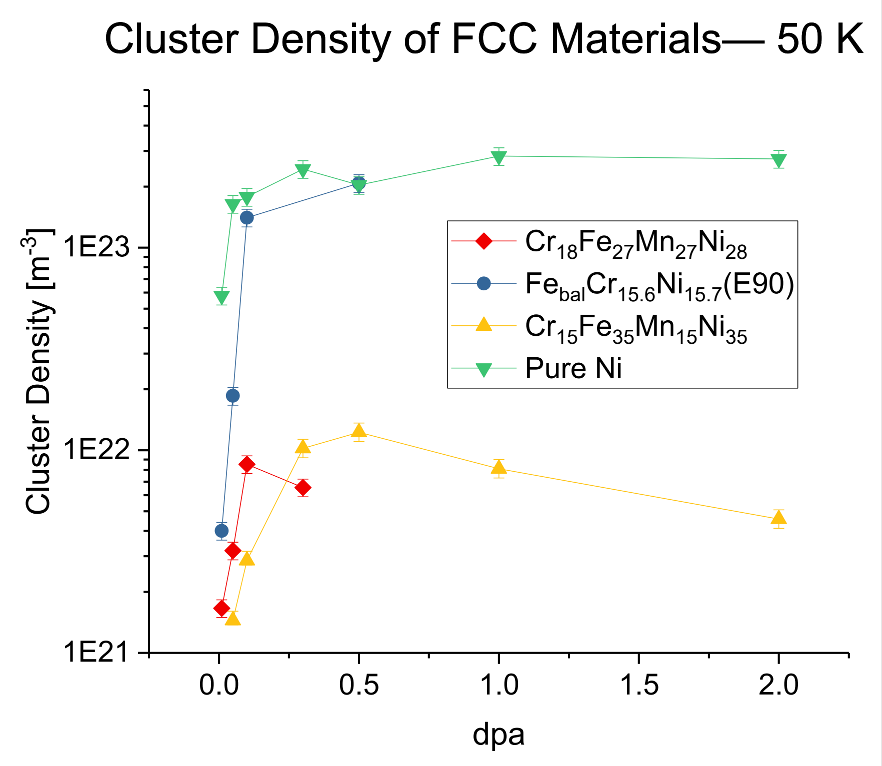
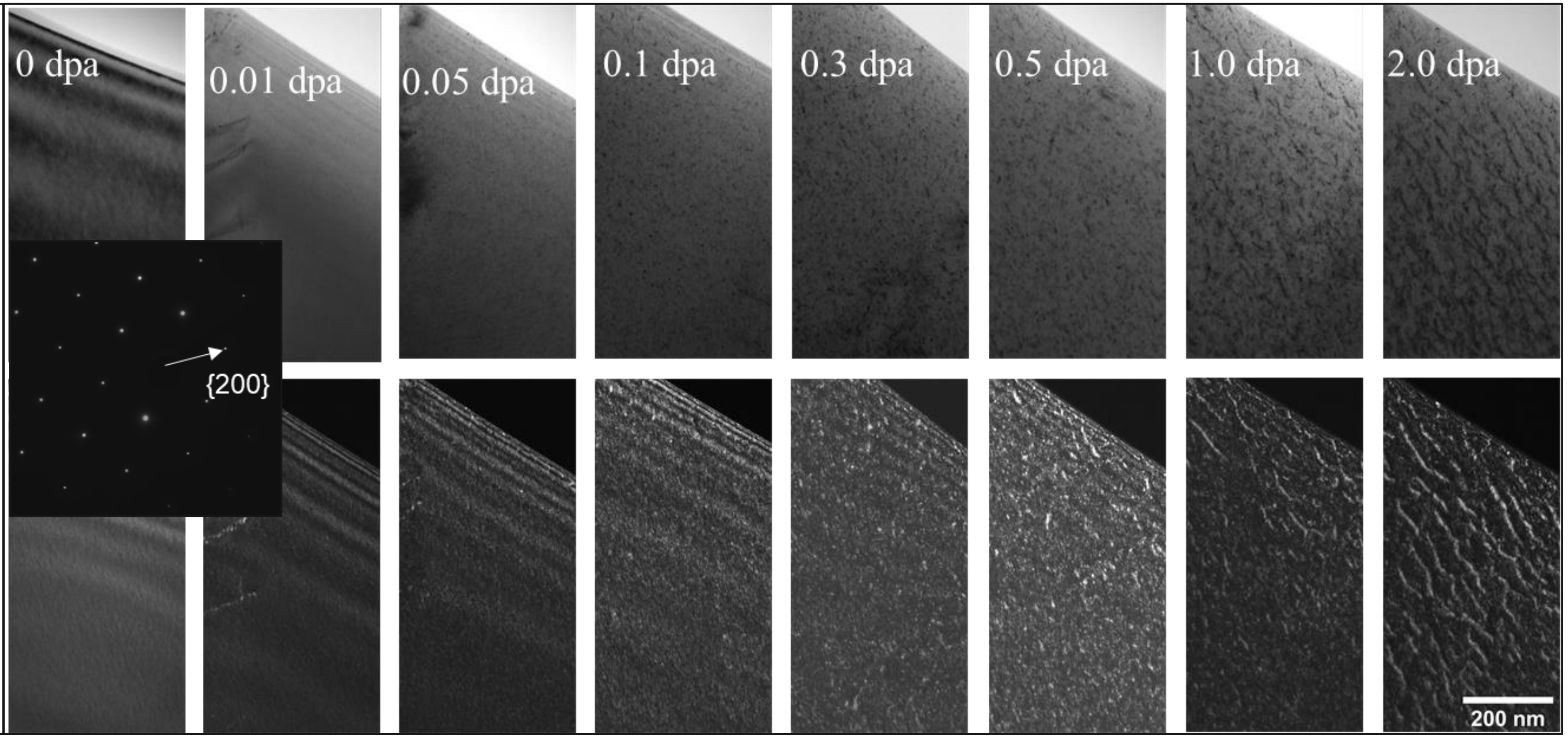




In-situ single-beam irradiation - 1.0 MeV Kr^{2+} at 50 K

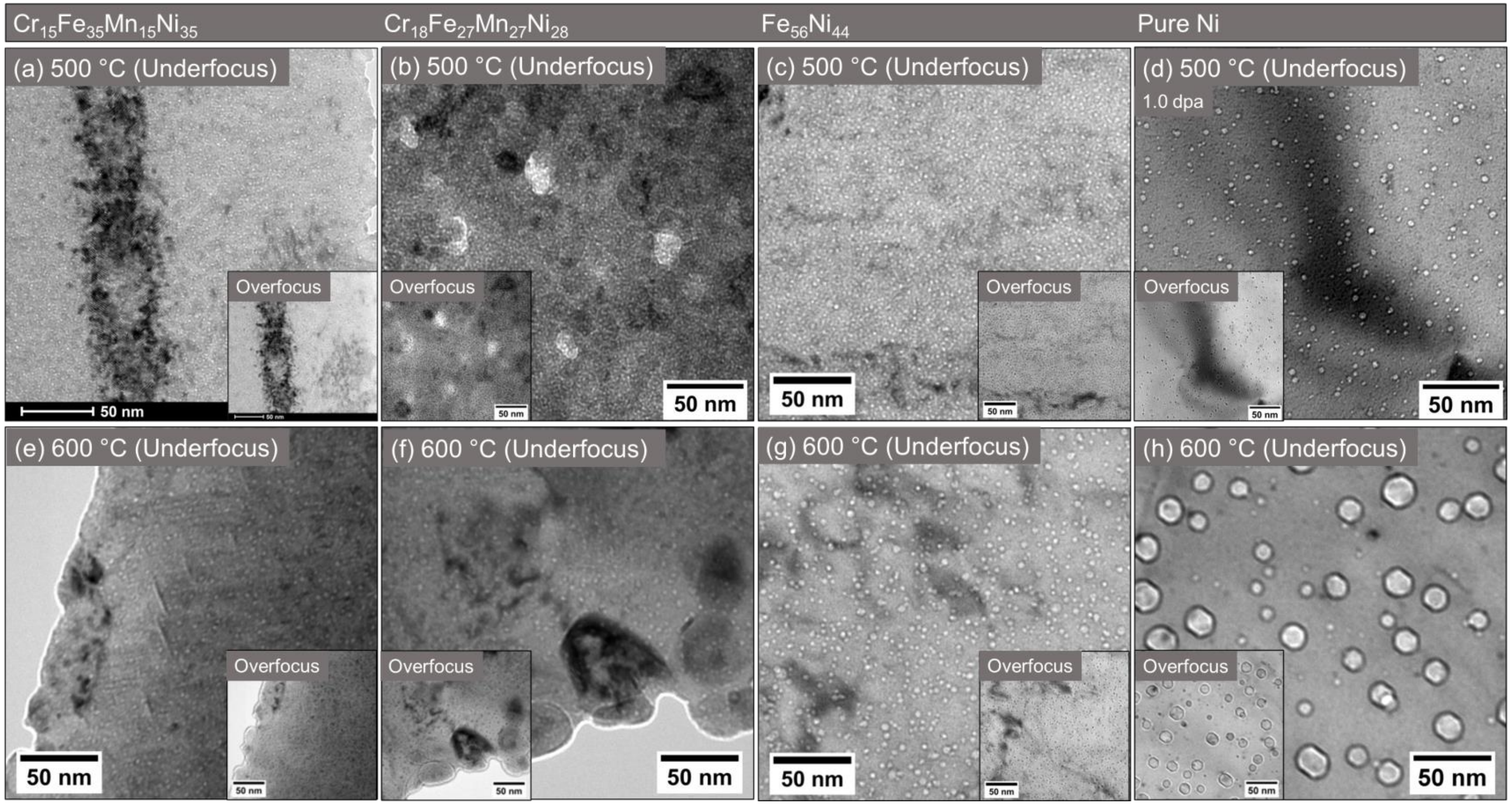


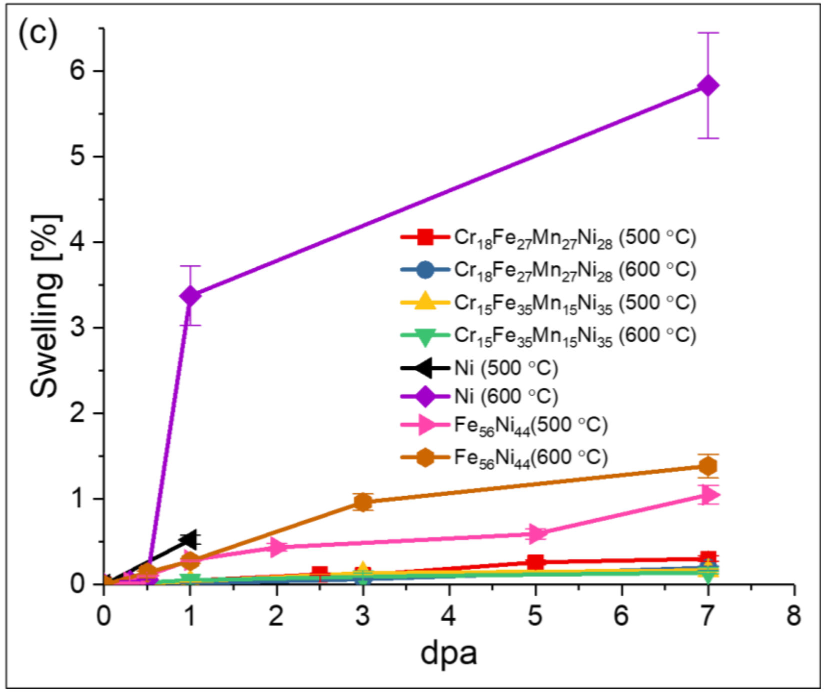


- $Cr_{15}Fe_{35}Mn_{15}Ni_{35}$ near <110> zone axis shown as example. Clusters measured as black/white dots in bright-field and WBDF.
- **Compositional complexity reduced defect cluster density at 50 K** → reduced primary defect production compared to E90 and pure Ni.

1. C. Parkin, M. Moorehead, M. Elbakhshwan, J. Hu, W.-Y. Chen, M. Li, L. He, K. Sridharan, A. Couet, In situ microstructural evolution in face-centered and body-centered cubic complex concentrated solid-solution alloys under heavy ion irradiation, Acta Materialia 198 (2020) 85-99.

CCAs compared to Ni and $Fe_{56}Ni_{44}$ at maximum dpa

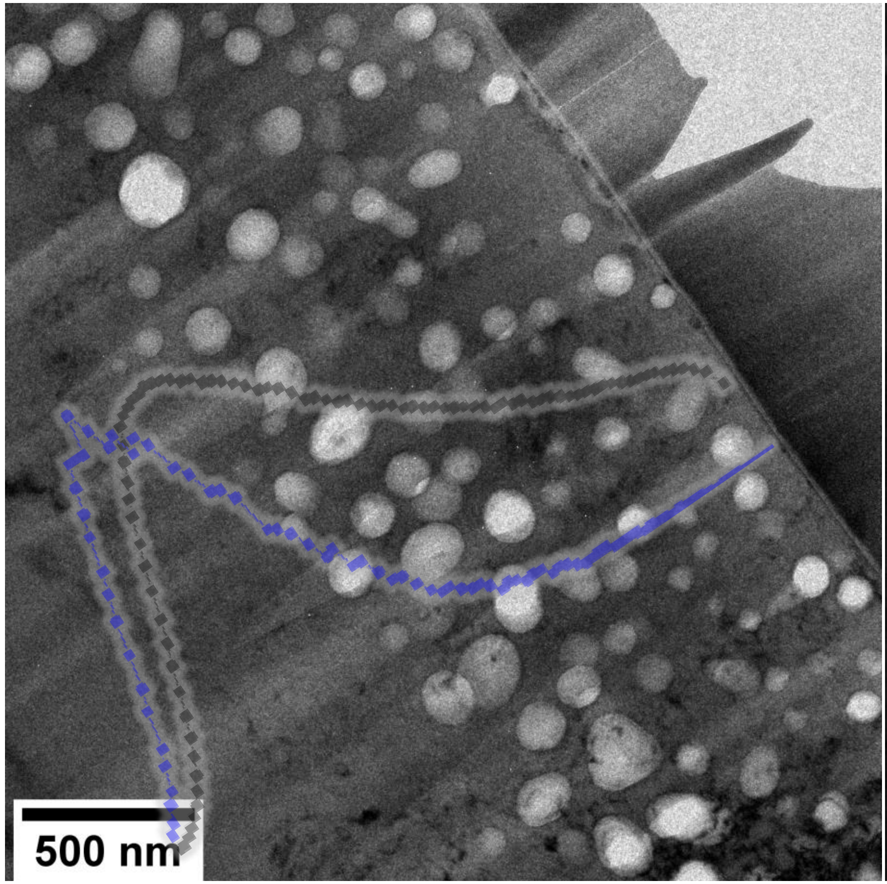




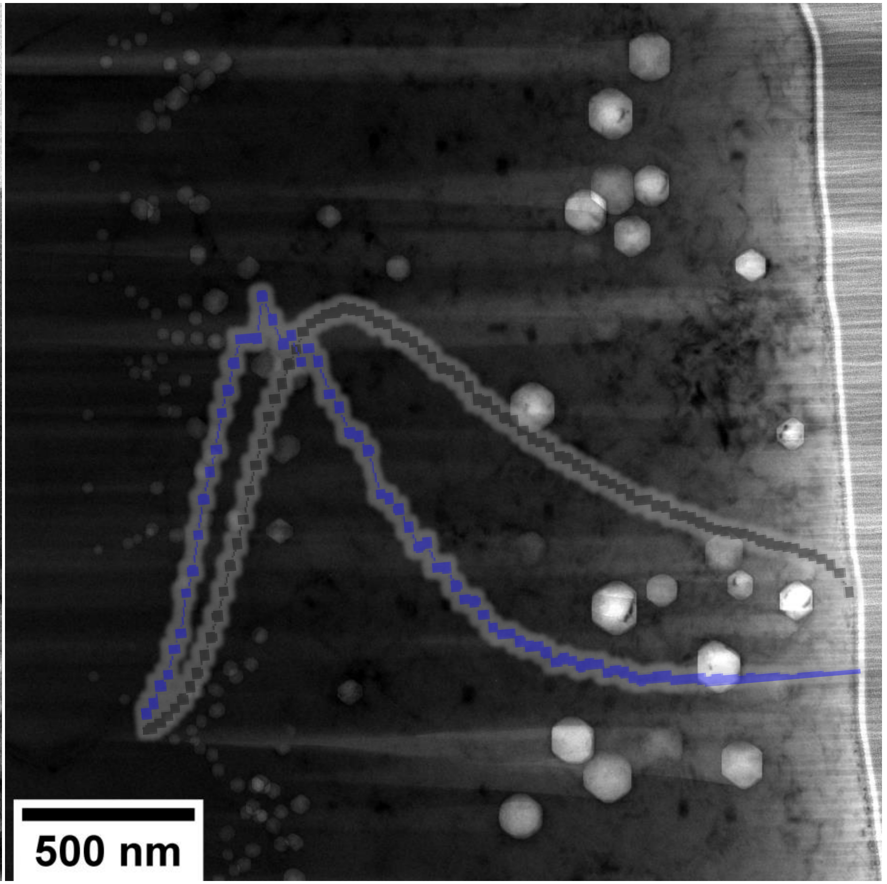
- Trapping effect of vacancies on He leads to higher bubble density at 500 °C.
- $Cr_{18}Fe_{27}Mn_{27}Ni_{28}$ swells more at 500 °C, $Cr_{15}Fe_{35}Mn_{15}Ni_{35}$ about the same.
- **More swelling in $Fe_{56}Ni_{44}$ and Ni than CCA at both temperatures**.

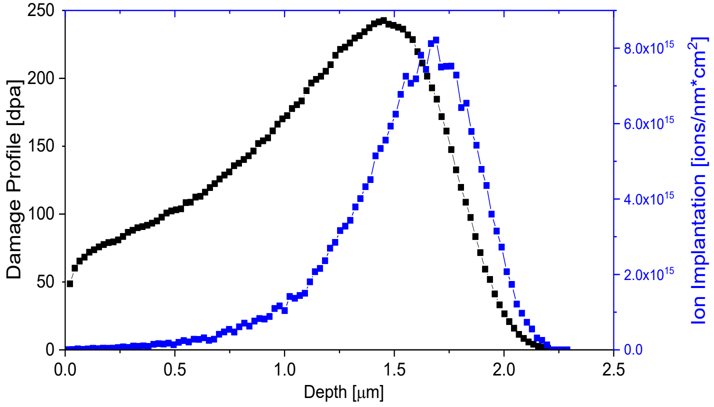
Ion Irradiation 500ºC / 150 dpa – $Cr_{15}Fe_{35}Mn_{15}Ni_{35}$

SS316H



$Cr_{15}Fe_{35}Mn_{15}Ni_{35}$



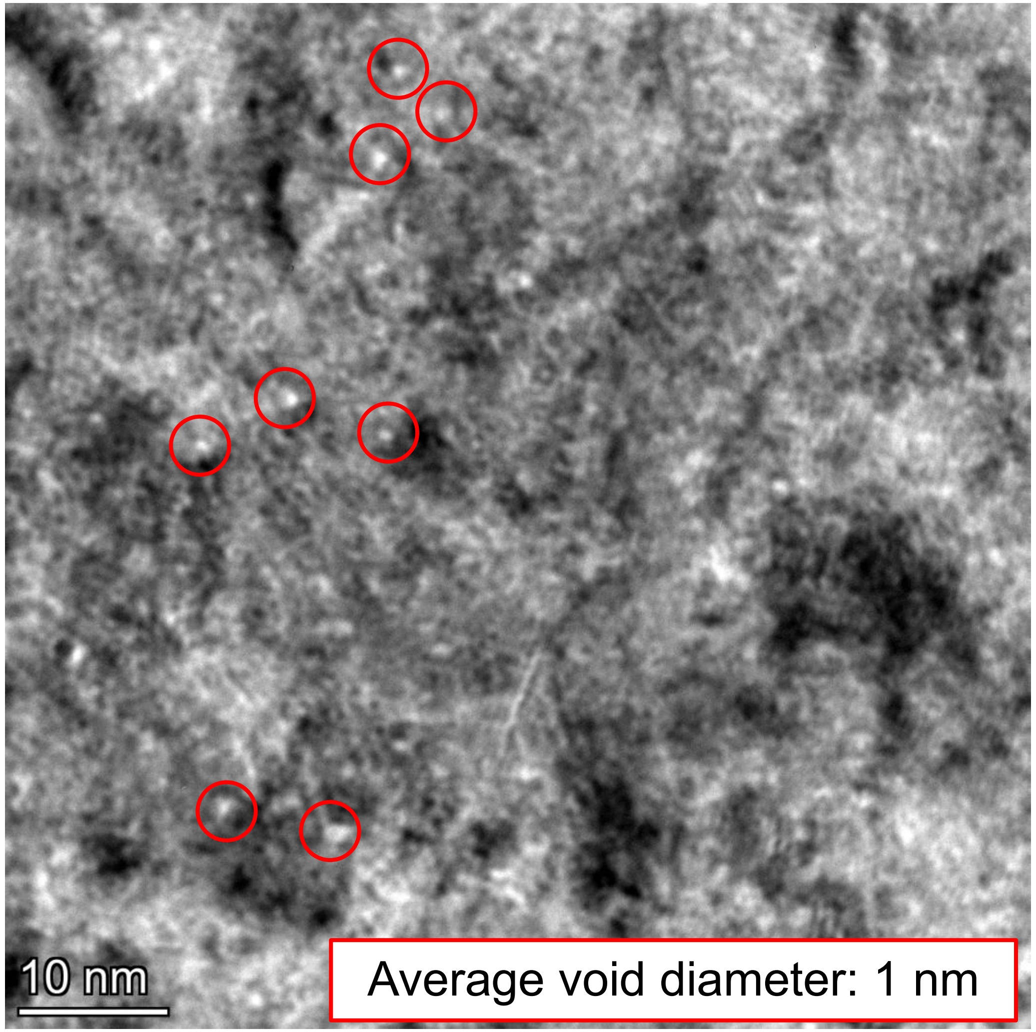


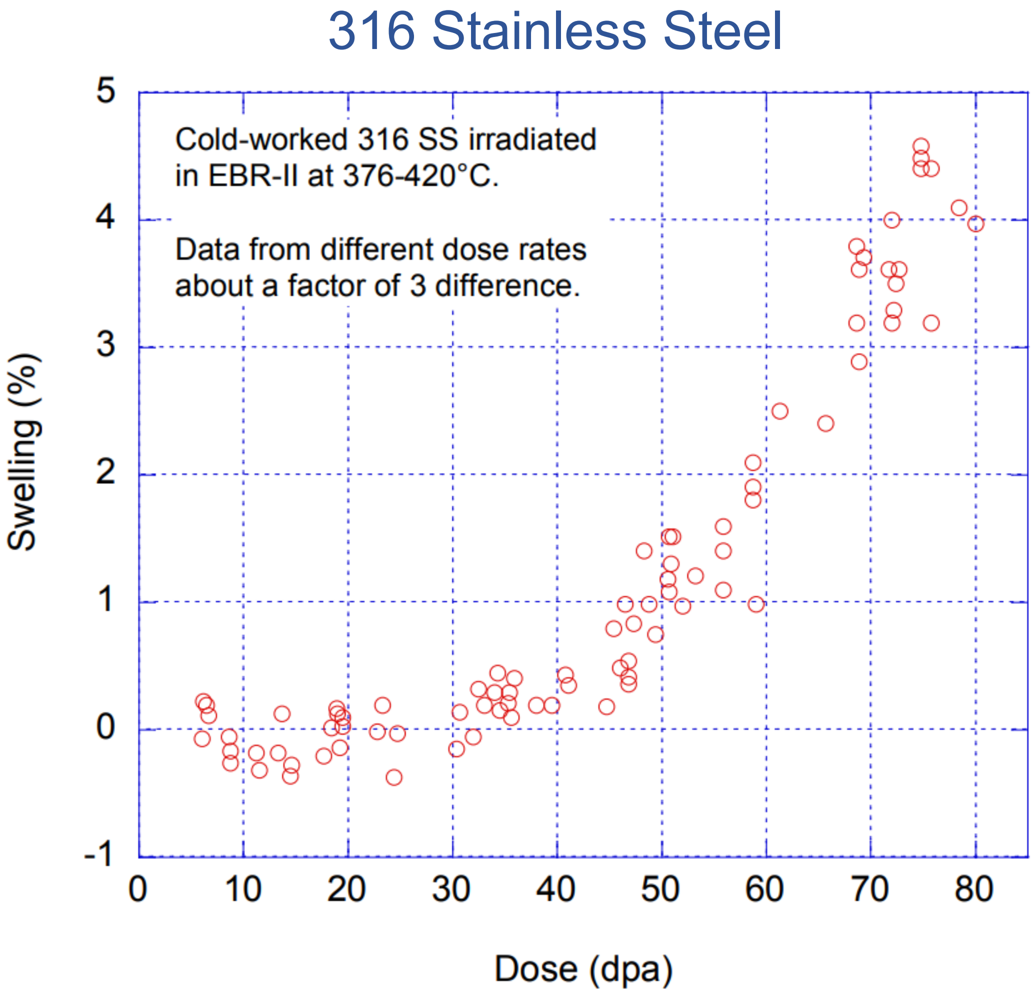
500 ºC, 150 dpa plateau

5 MeV Ni++ Ions

[1] Parkin, C. A. (2022). Microstructural Evolution of Compositionally Complex Alloys Under Heavy Ion Irradiation (29326352) [Doctoral Dissertation, University of Wisconsin - Madison]. Madison ProQuest Dissertations Publishing.

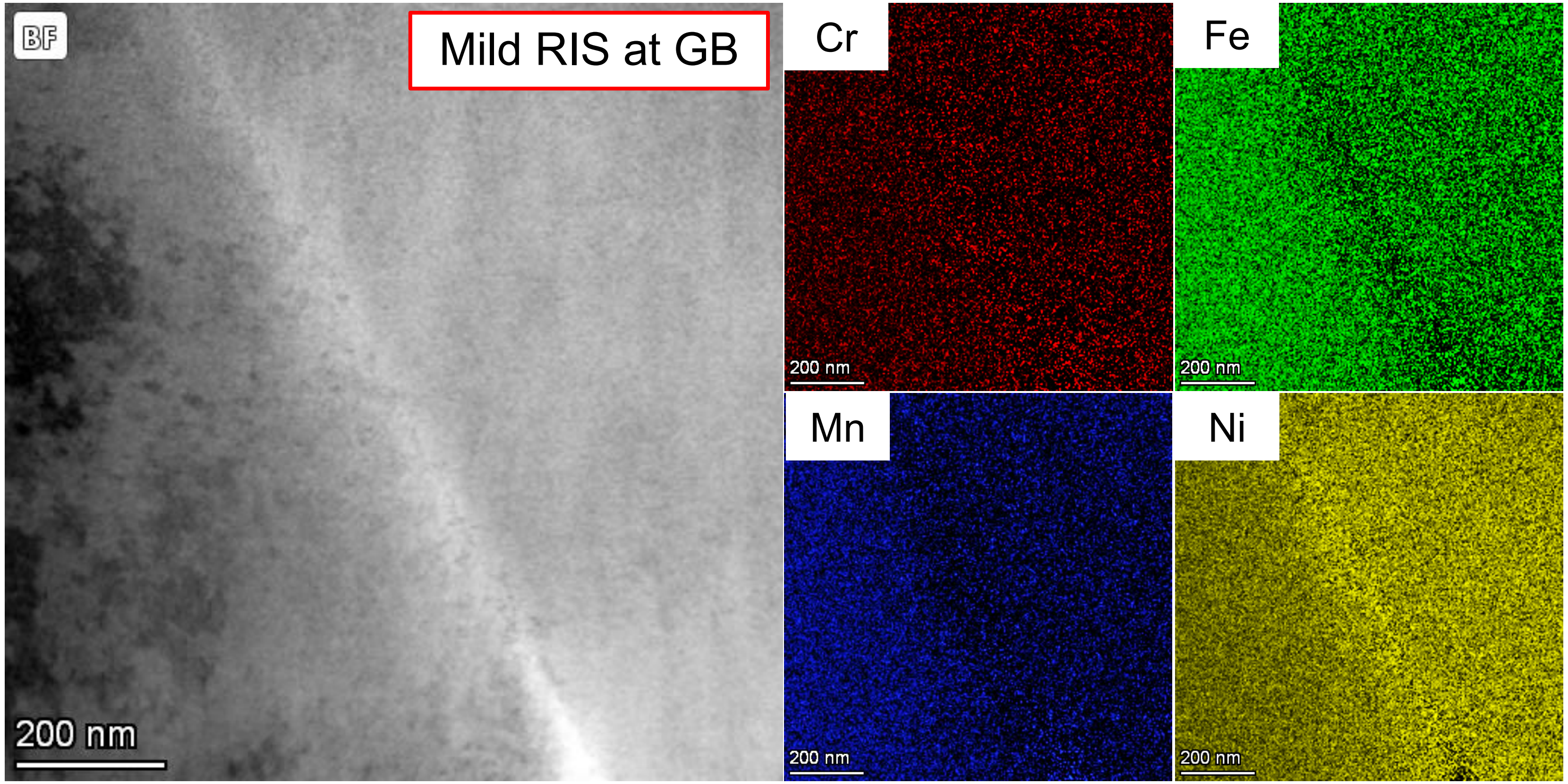
Neutron Irradiation 6 dpa and 500ºC – $Cr_{10}Fe_{30}Mn_{30}Ni_{30}$



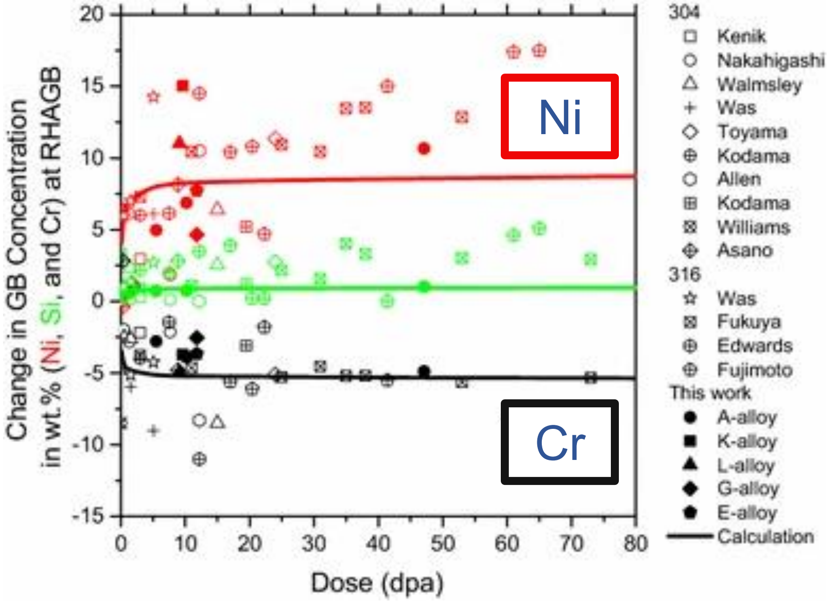


[1] Y. Chen, Y. Yang, Y. Huang, T. Allen, B. Alexandreanu, and K. Natesan, "Void Swelling and Microstructure of Austenitic Stainless Steels Irradiated in the BOR-60 Reactor," US Nuclear Regulatory Commission CR-7128, Nov. 2012
[2] Allen, T. R., H. Tsai, R. S. Daum, D. L. Porter, J. I. Cole, T. Yoshitake, N. Akasaka, T. Donomae, S. Mizuta, J. Ohta, K. Dohi, and H. Kusanagi, "Effects of Irradiation on the Swelling and Mechanical Properties of 316 Stainless Steel," 11th Int. Conf. on Environmental Degradation of Materials in Nuclear Power Systems, Stevenson, WA, August 10-14, 2003.

Neutron Irradiation 6 dpa and 300ºC – $Cr_{10}Fe_{30}Mn_{30}Ni_{30}$



Stainless Steels



[1] L. Tan, R. Stoller, K. Field, Y. Yang, H. Nam, D. Morgan, B. Wirth, M. Gussev, and J. Busby, "Microstructural evolution of Type 304 and 316 stainless steels under neutron irradiation at LWR relevant conditions," JOM, vol. 68, pp. 517-529, Dec. 2015.