Irradiation Influence on Alloys Fabricated by Powder Metallurgy and Hot Isostatic Pressing for Nuclear Applications

Janelle P. Wharry[1], Donna P. Guillen[2], David W. Gandy[3]

[1] *Purdue University*; [2] *Idaho National Laboratory;* [3] *Electric Power Research Institute*

NSUF Annual Program Review
Idaho Falls, ID & Virtual
15 April 2024

Supported by:
DOE Nuclear Science User Facilities 15-8242
Electric Power Research Institute

PURDUE UNIVERSITY

NSUF EPRI

Students Supported on this Program

Caleb D. Clement
Ph.D. 2023
Now at Westinghouse

Saquib Bin Habib
Ph.D. expected 2026

Sowmya Panuganti
M.S. 2022

Yangyang Zhao
Post-Doc 2019-2021

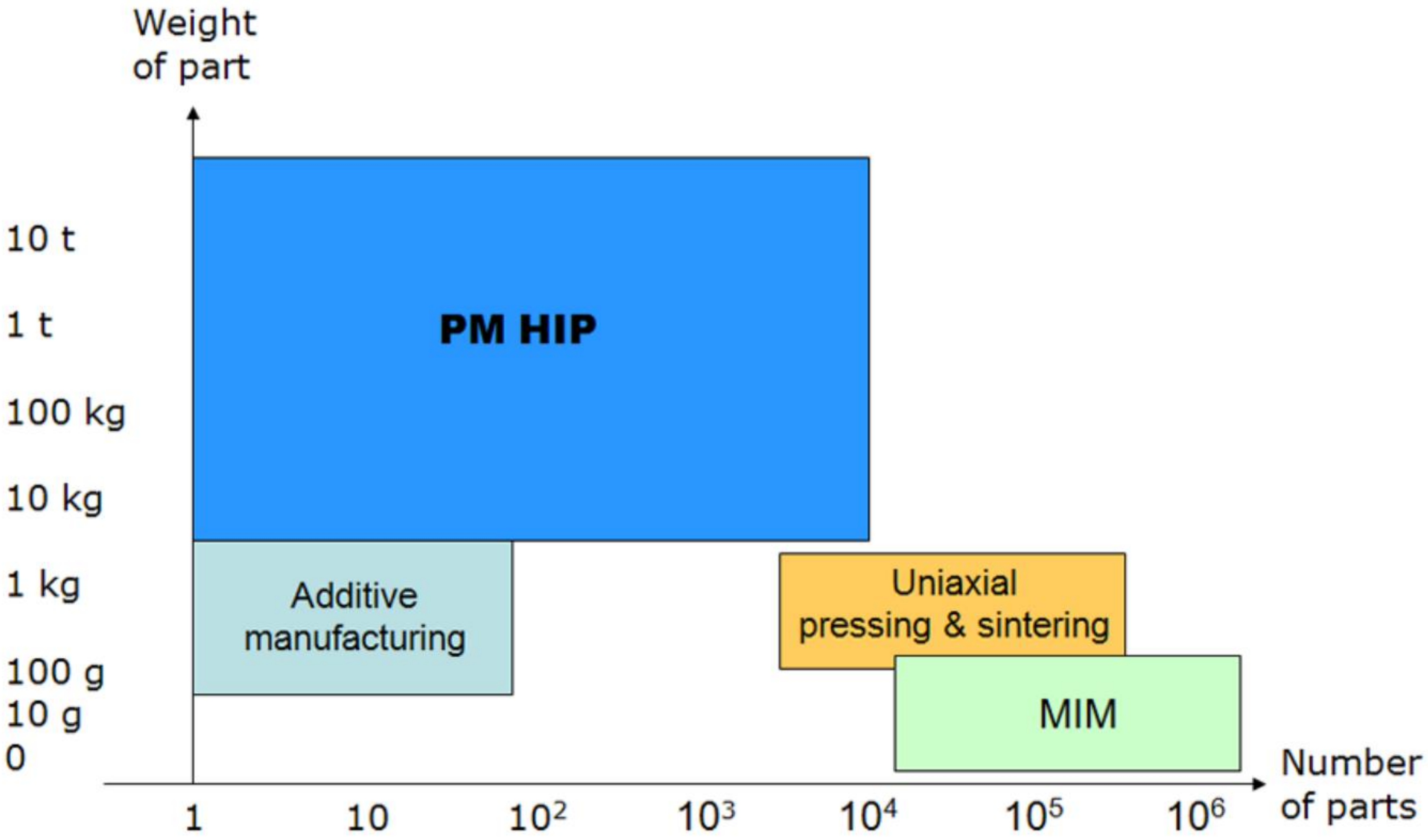
Wen Jiang
Post-Doc 2023
Now at Xi`an Jiao Tong

Objective

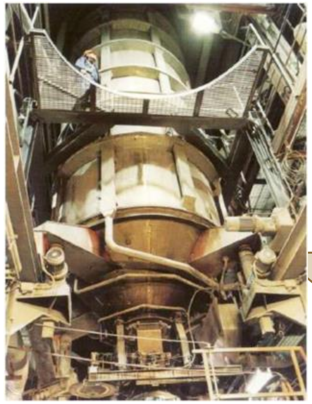
Assess the viability of using alloys manufactured by powder metallurgy with hot isostatic pressing (PM-HIP) for nuclear reactor structural components.

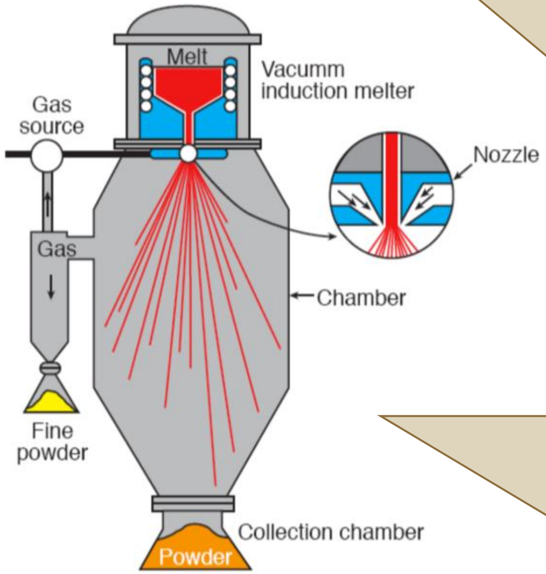
Understand irradiation effects on PM-HIP alloys through a systematic neutron irradiation campaign and post-irradiation microstructural and mechanical assessments.

PM-HIP Positioning Compared to Other Fabrication Methods



Overview of PM-HIP Process





- Powder atomization
- Sieve
- Blend

- FEM design can
- Form can
- Weld

- Fill can
- Bake out
- Seal
- HIP

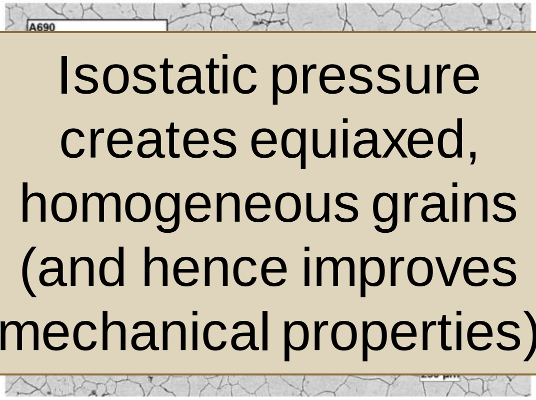
Benefits of PM-HIP

Easier to inspect, fewer quality issues

Poor quality castings

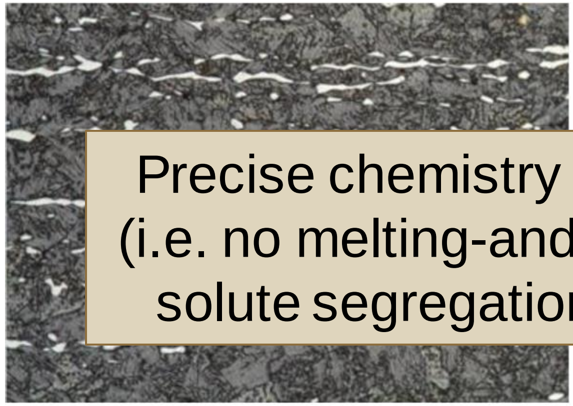
High powder compaction & densification

Voids/porosity in castings

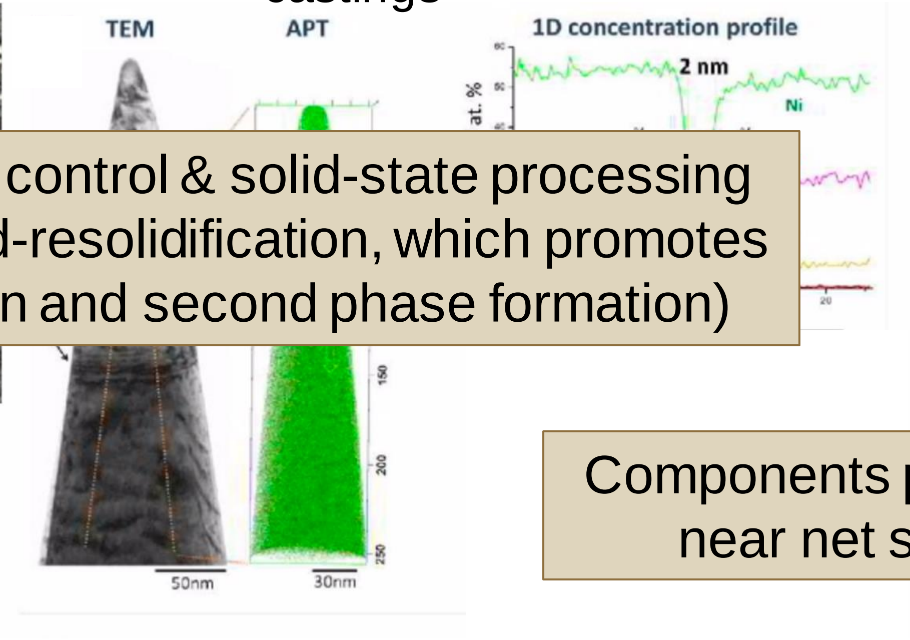


Isostatic pressure creates equiaxed, homogeneous grains (and hence improves mechanical properties)

Grain texture (Alloy 690)



Ferrite stringers in forged stainless steel plate



Precise chemistry control & solid-state processing (i.e. no melting-and-resolidification, which promotes solute segregation and second phase formation)

Solute & impurity pickup; grain boundary segregation

Components produced near net shape

NSUF Irradiation Campaign 15-8242

Jiang, et al. JNM 594 (2024) 155018

Clement, et al. MSE A 857 (2022) 144058

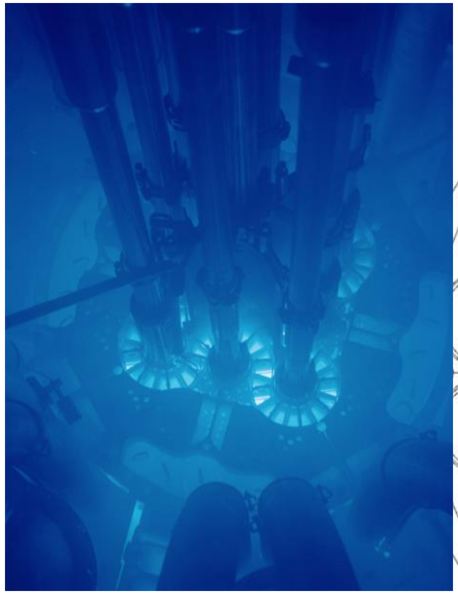
Wharry, et al. Data in Brief 48 (2023) 109092

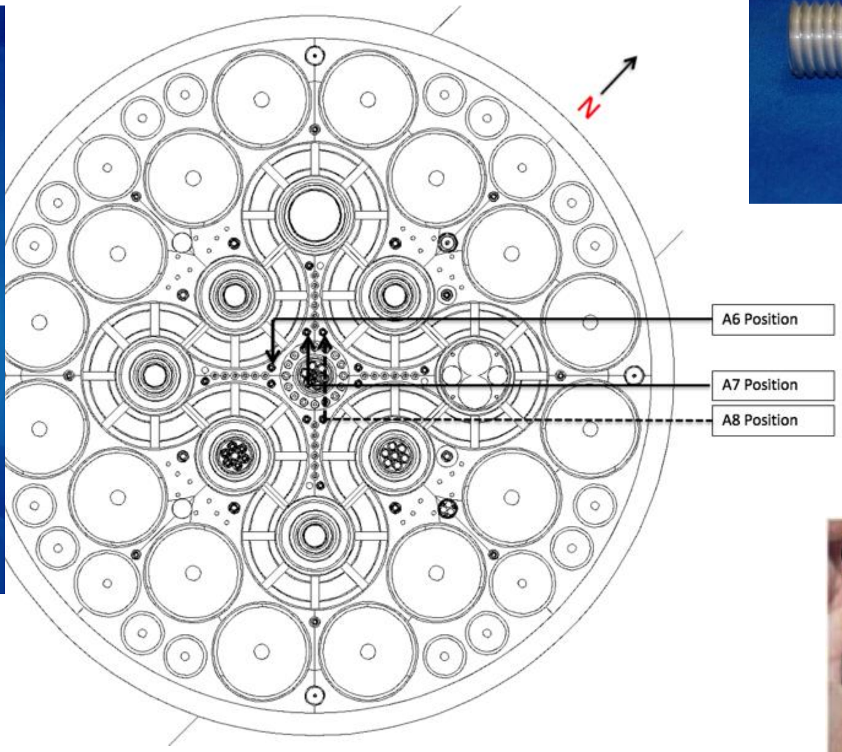
Wharry, et al. Frontiers (2023)

This Talk

Alloy	Process	Target Dose [dpa]	Target Temp [ºC]	Micro-structure	Tensile
SA508	PM-HIP, Forged	1	300	√	√
		1	400	√	√
Grade 91	PM-HIP, Cast	1	400	√	√
		3	400	√	√
Alloy 625	PM-HIP, Forged	1	400	√	√
		3	400	√	√
Alloy 690	PM-HIP, Forged	1	400	√	√
		3	400	√	√
316L SS	PM-HIP, Wrought	1	400	–	–
		3	400	–	√

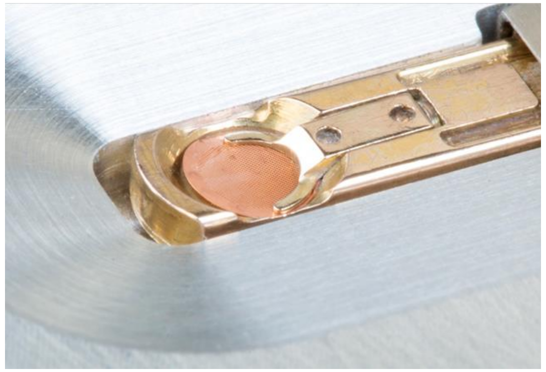
Experiment Design



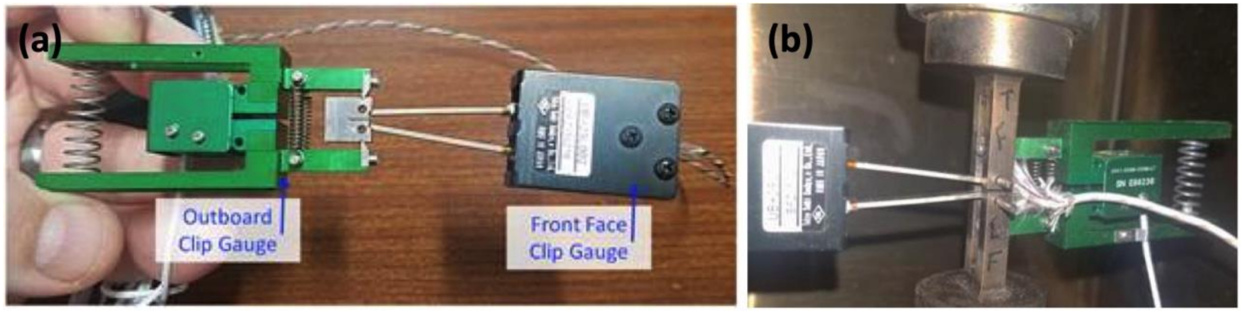


Advanced Test Reactor
Idaho National Laboratory

ASTM standard tensile bars: yield strength, modulus, % elongation



TEM discs: microstructure, nanoindentation



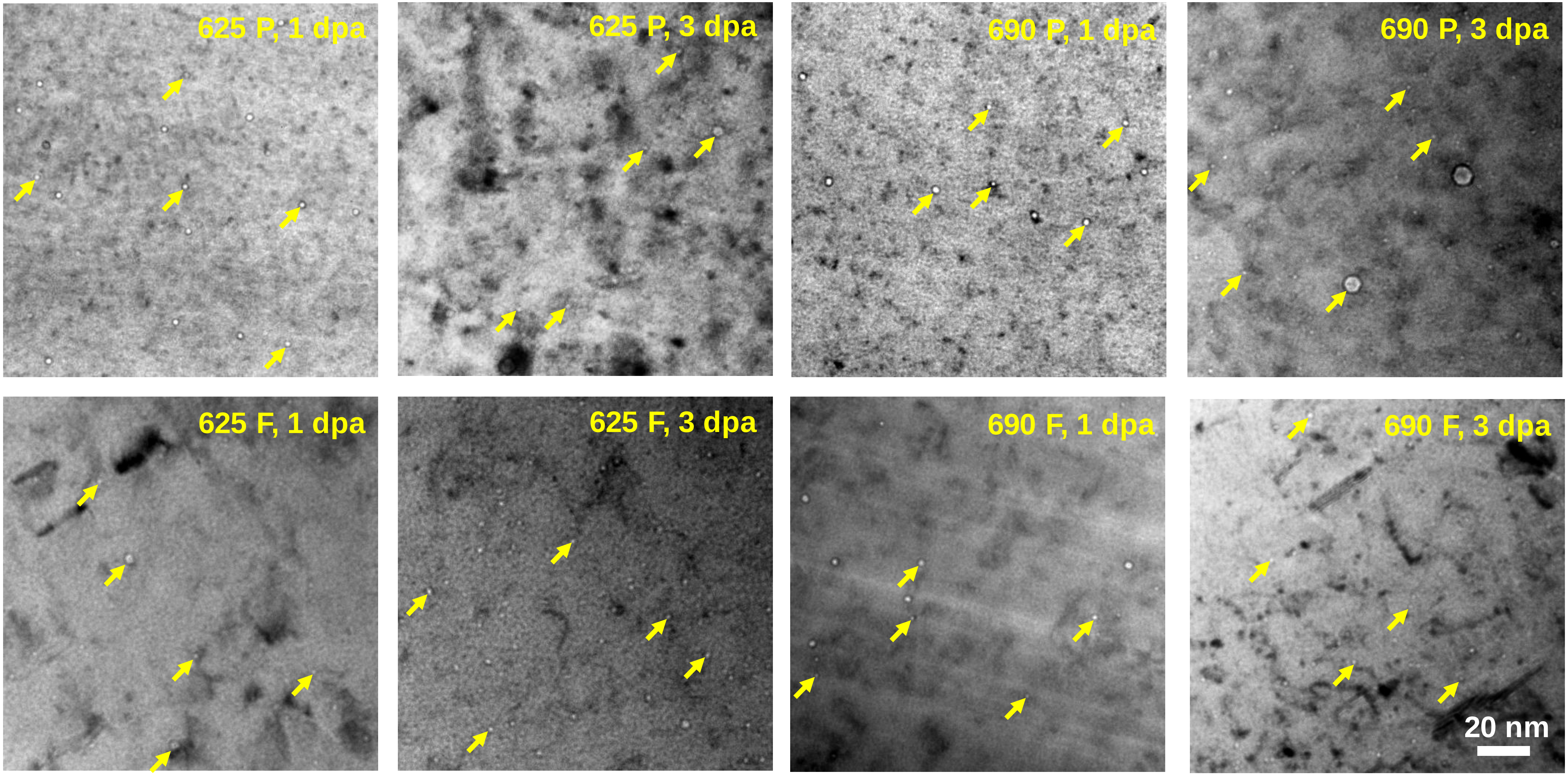
Miniature CTs: fracture toughness

PURDUE UNIVERSITY

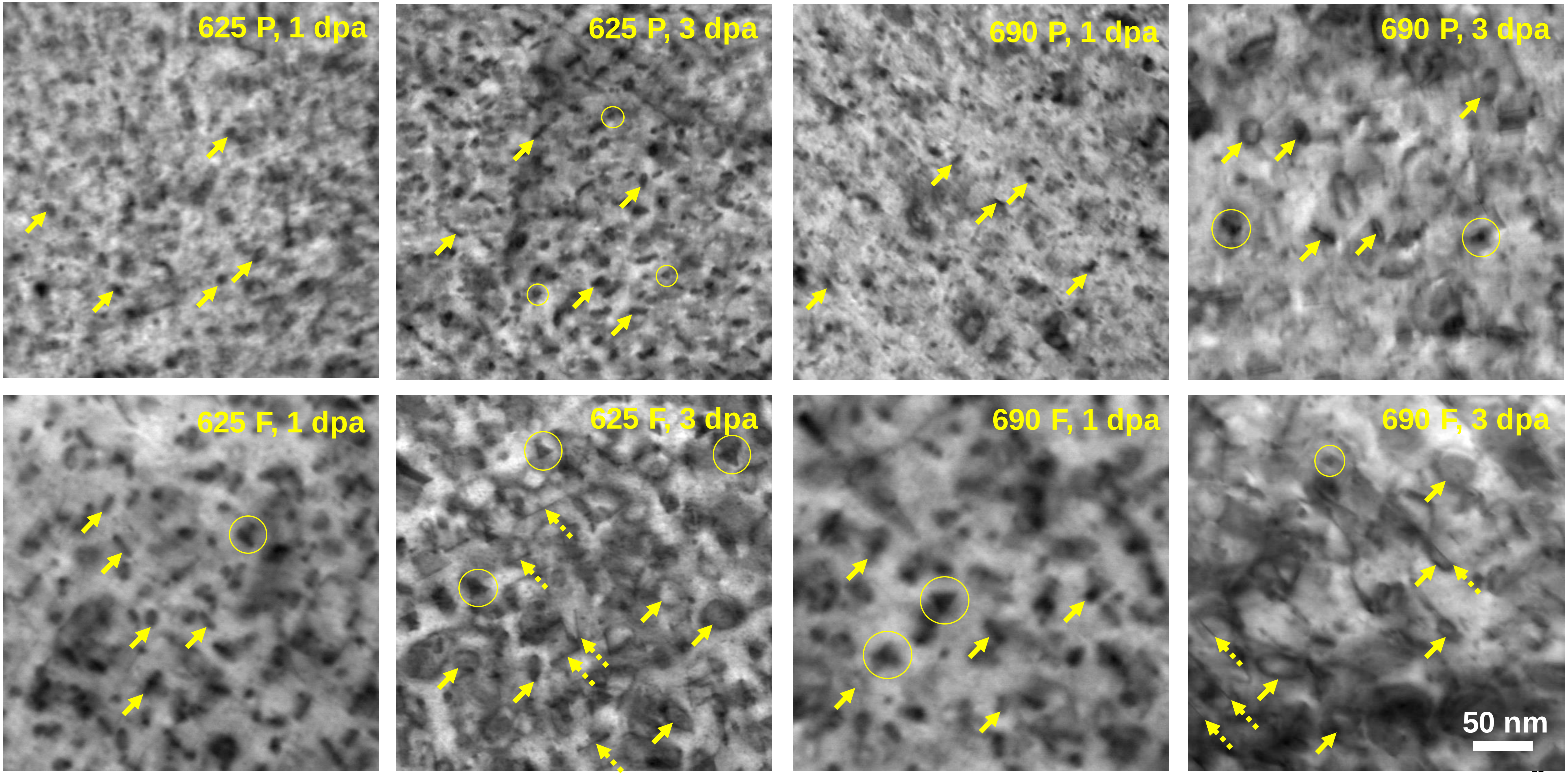
Alloy 625 & 690

Microstructure & Mechanical

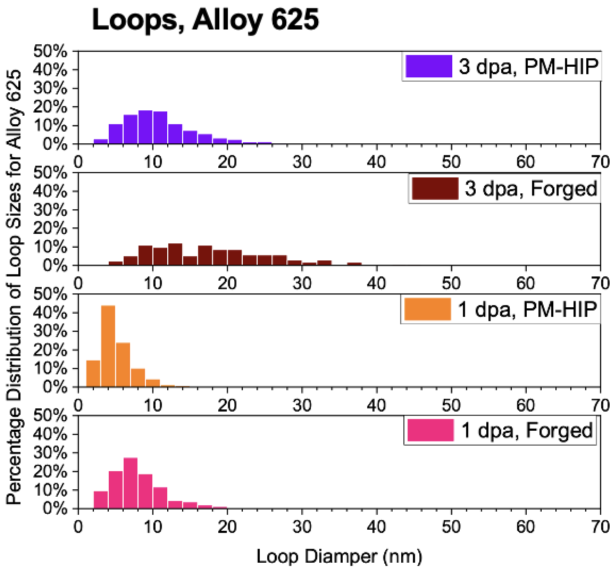
Void Evolution

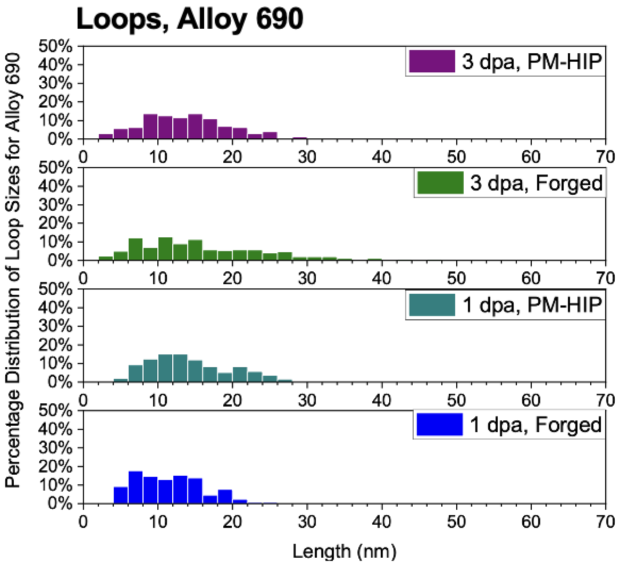


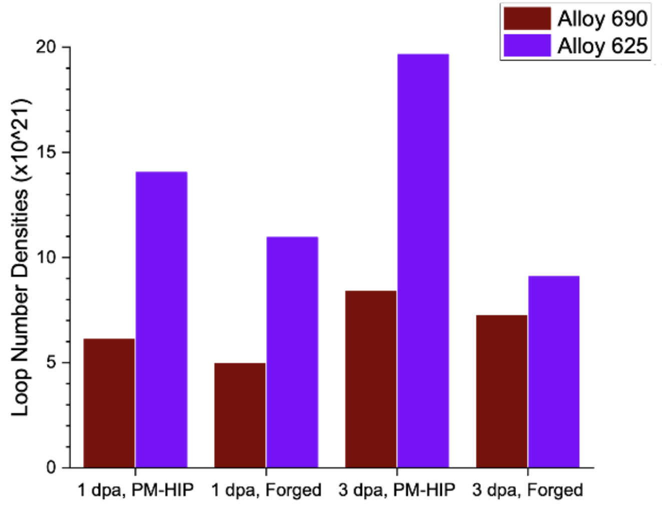
Dislocation Loop Evolution

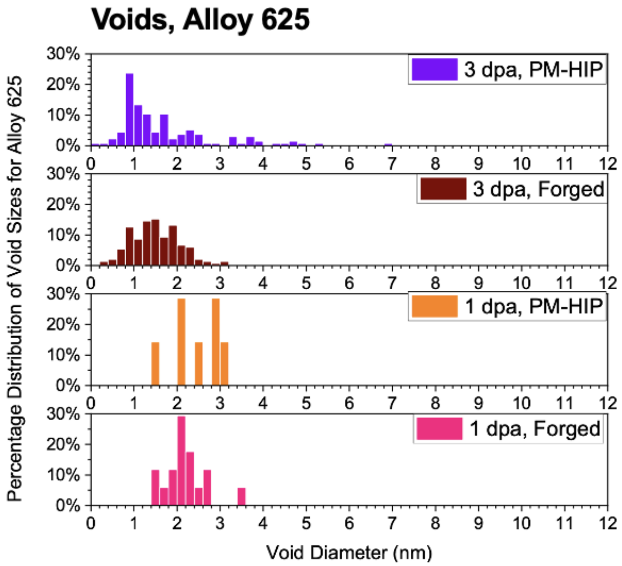


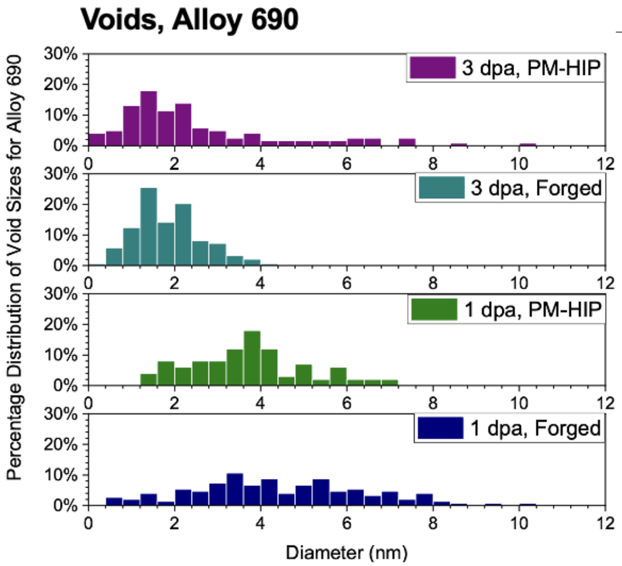
Quantitative Loop & Void Evolution

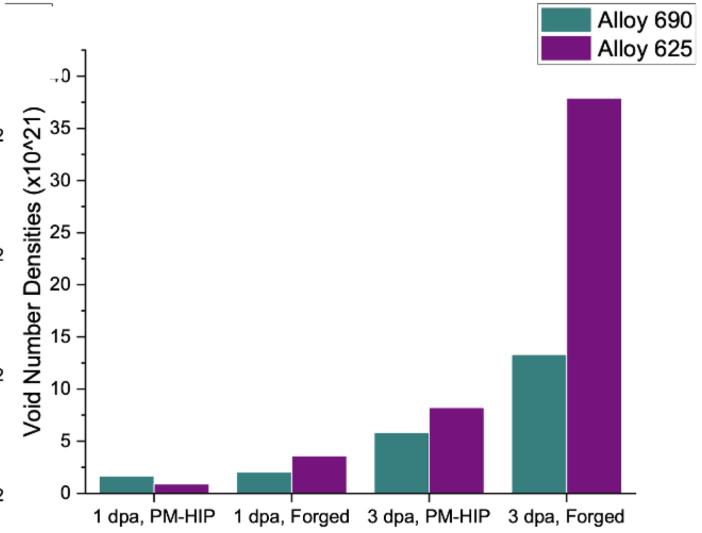




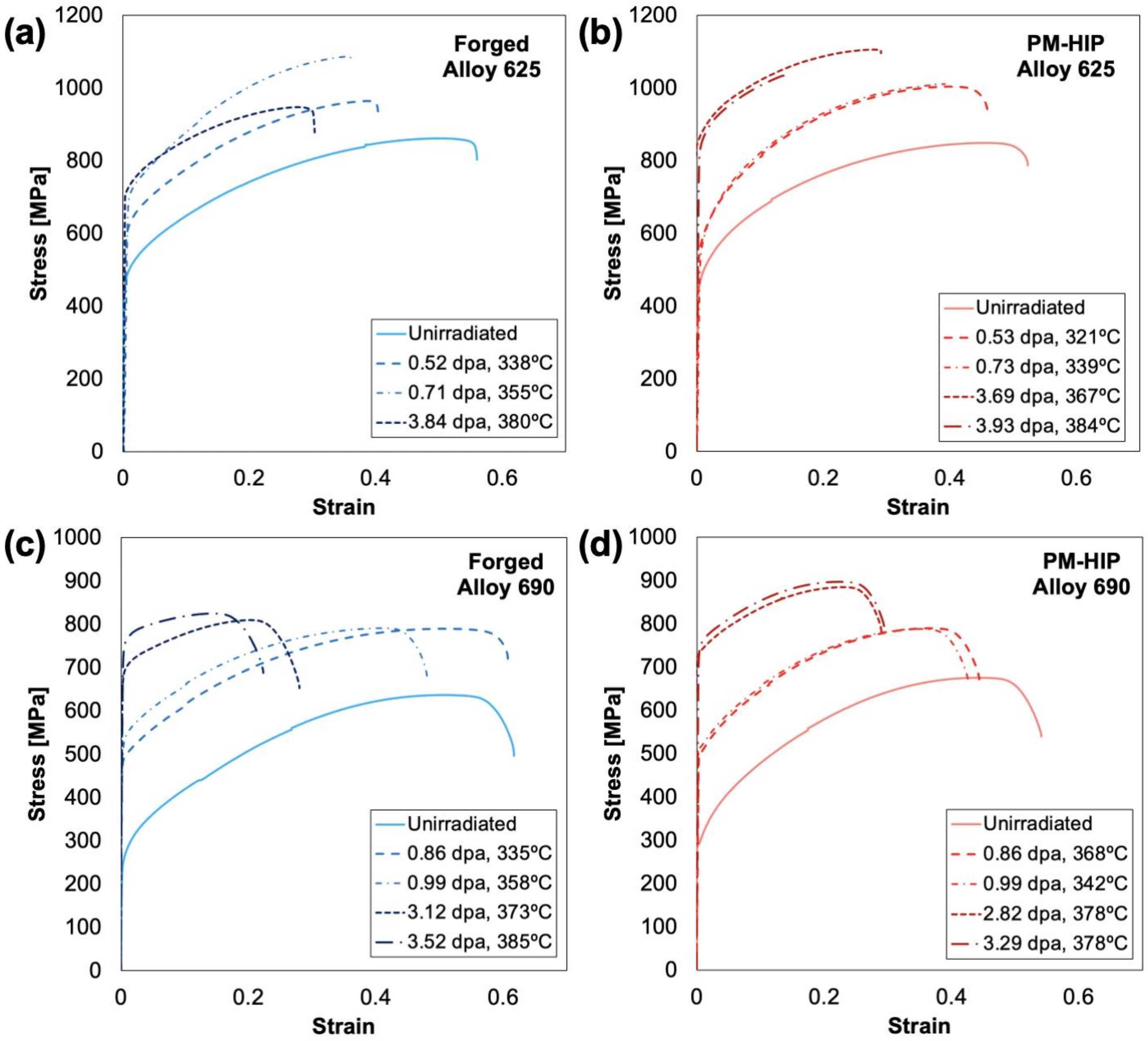


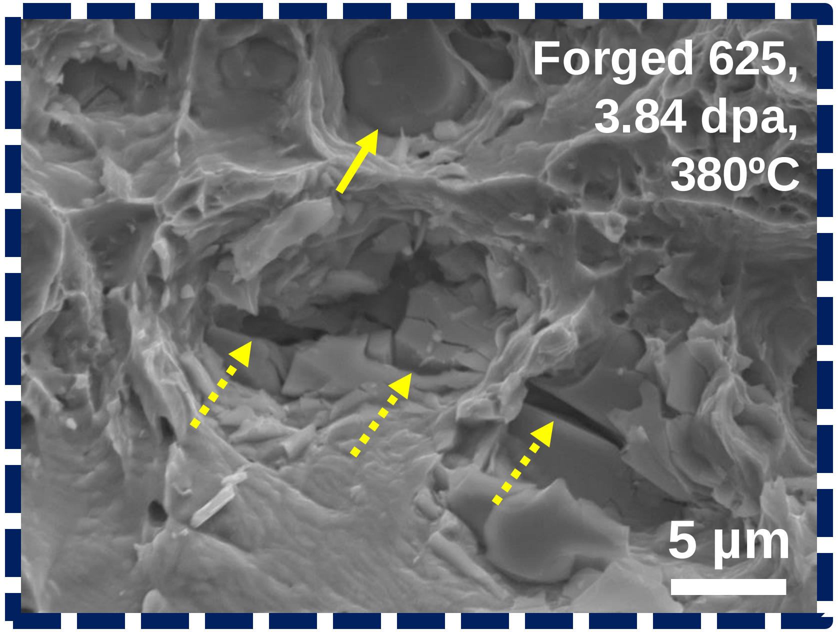


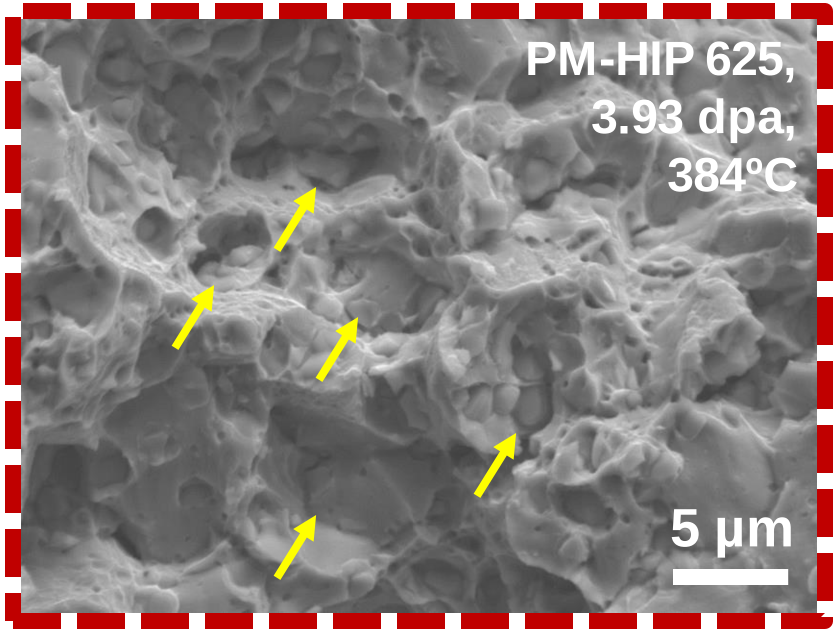




Tensile Results



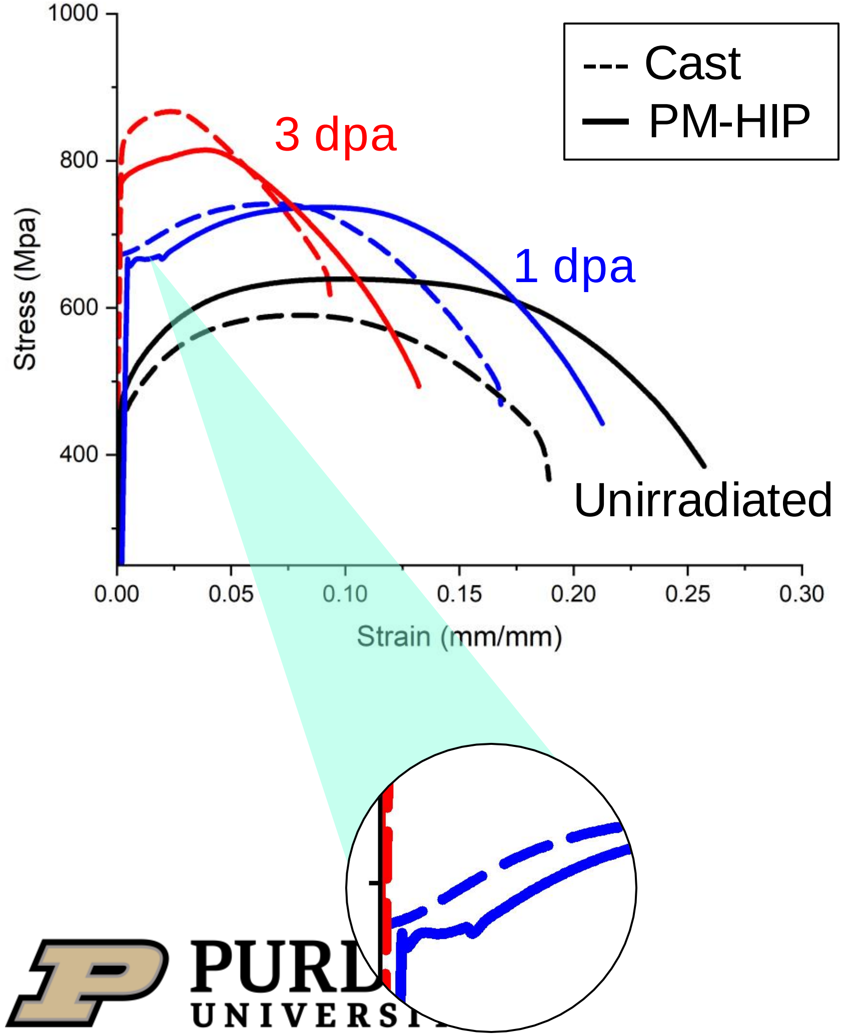


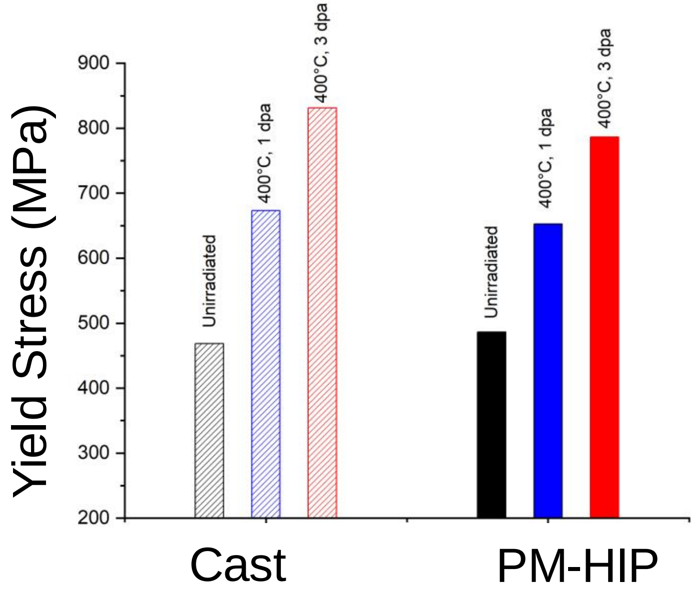


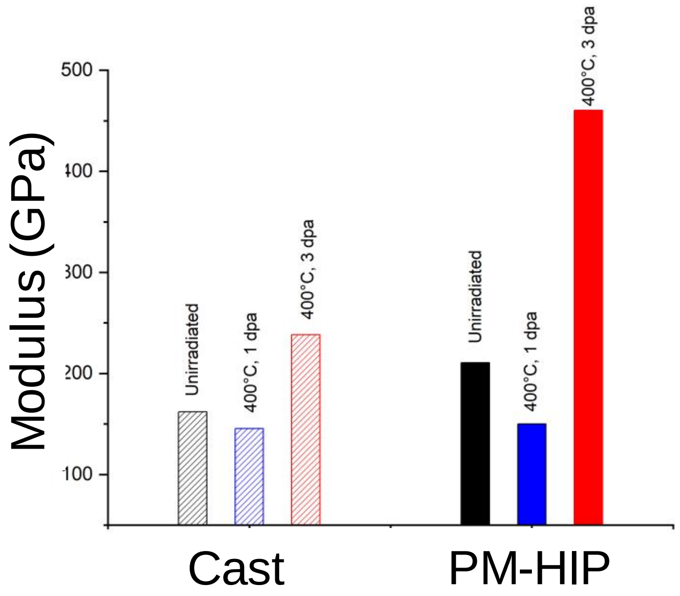
Grade 91 Ferritic Steel

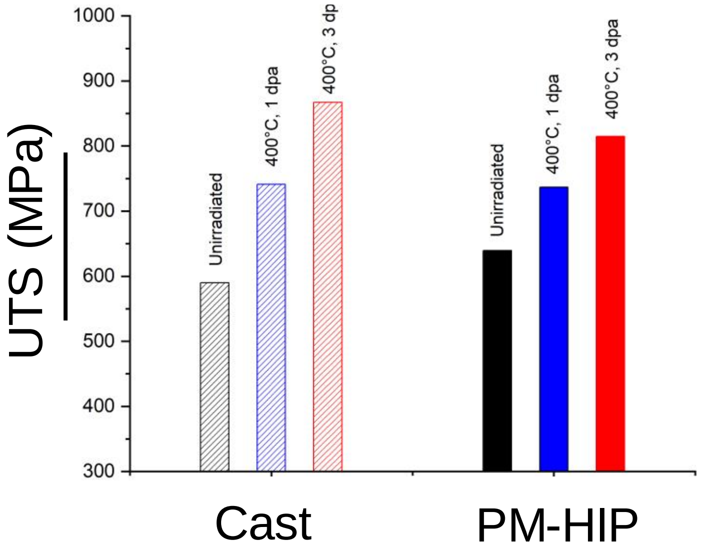
Microstructure & Mechanical

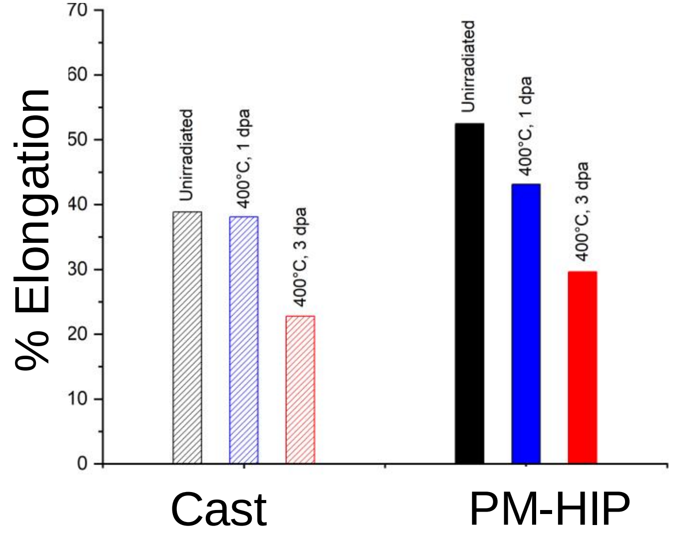
Development of Yield Point Phenomenon









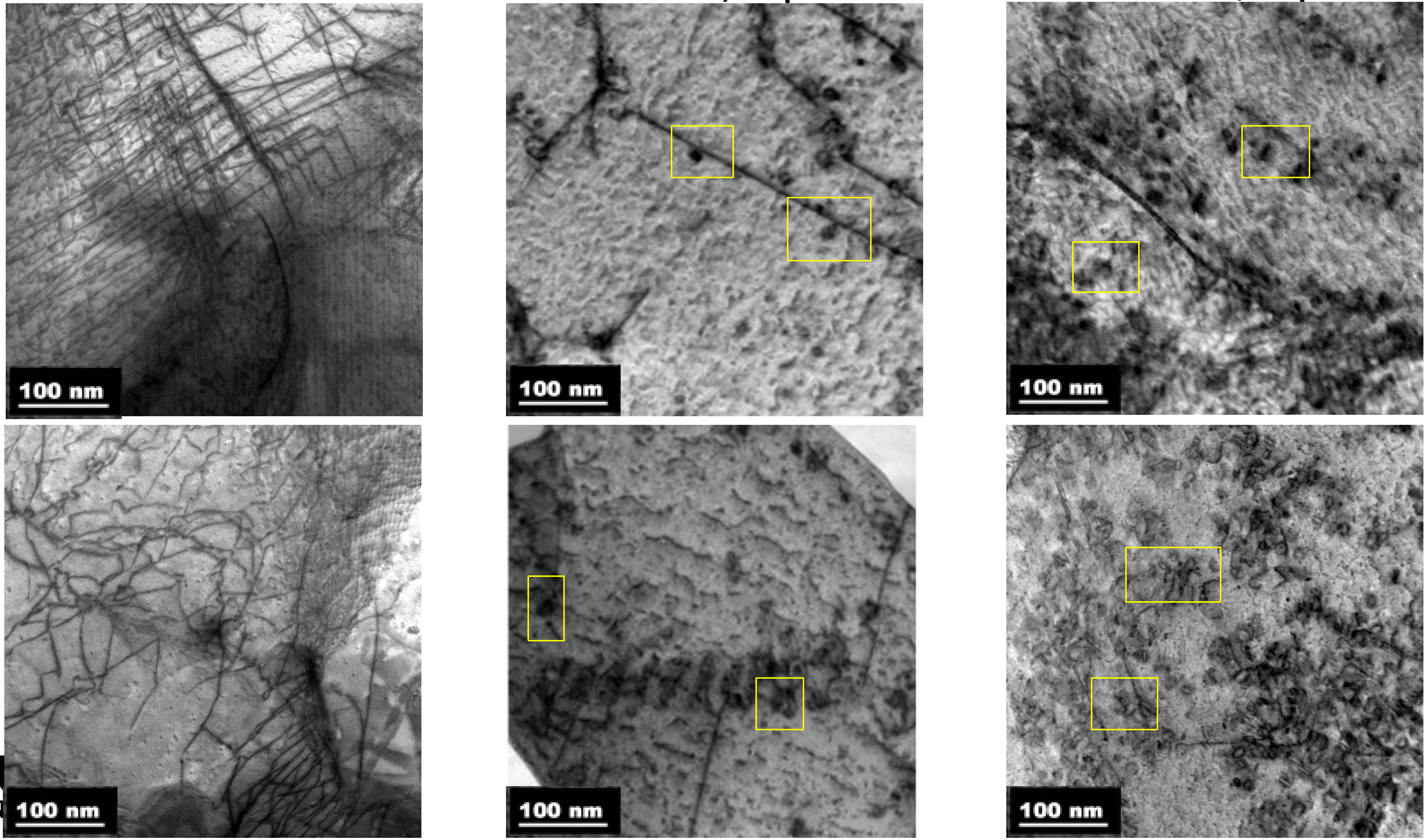


Heterogeneous Dislocation Loop Nucleation

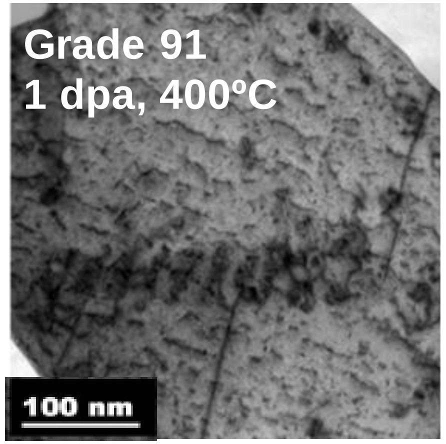
Unirradiated | 400°C , 1 dpa | 400 °C, 3 dpa

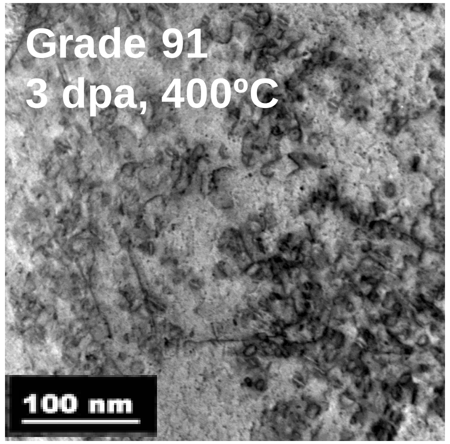
Cast

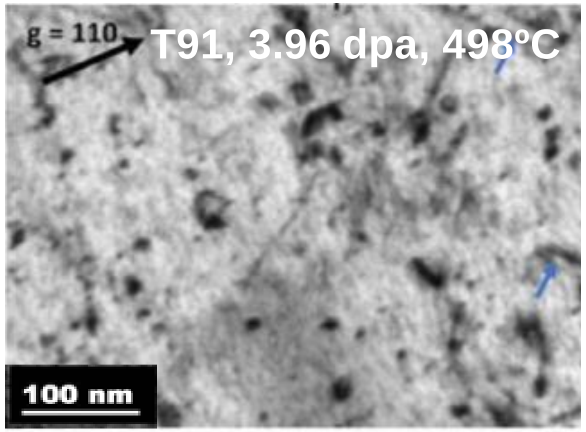
PM-HIP



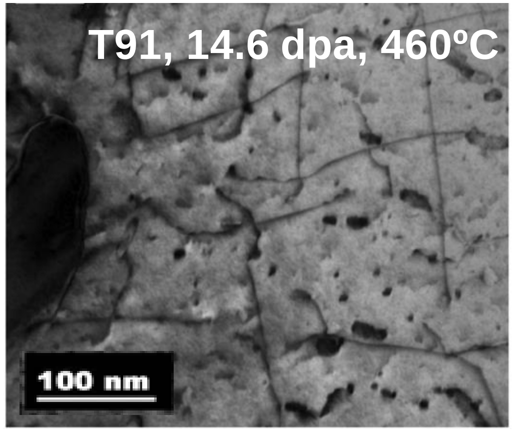
Heterogeneous Loop Nucleation is Uncommon in Ferritic Steels



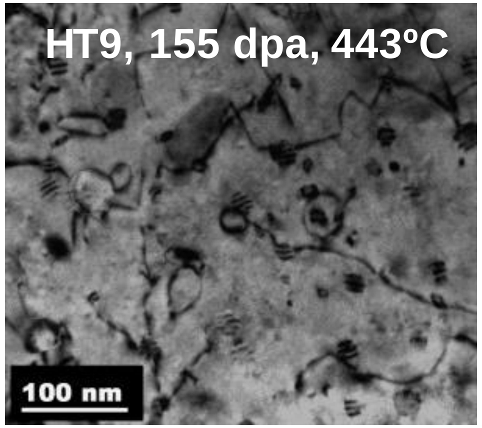




Yan, et al. JNM (2021)



Jiao, et al. JNM (2018)



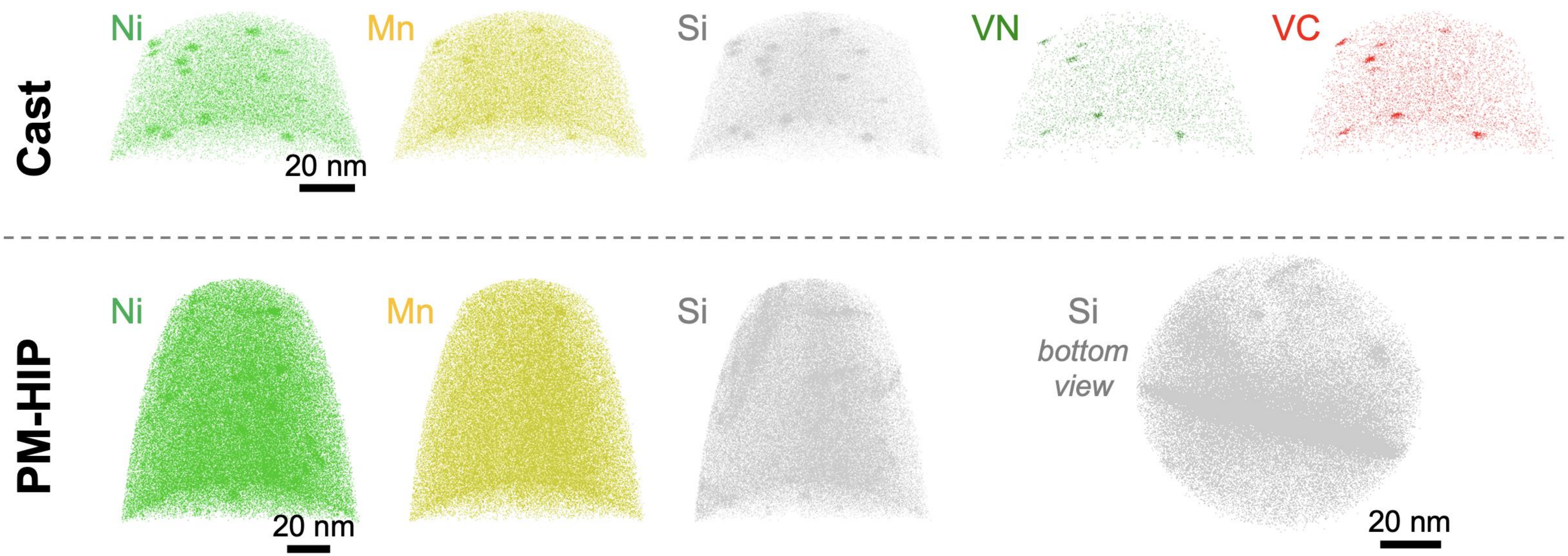
Sencer, et al. JNM (2009)

Heterogeneous Loops

Homogeneous Loops

Dislocations are annealed while loops begin to nucleate → Dislocations get pinned on loops, forming quasi subgrain → Heterogeneous loop nucleation at subgrains → After subgrain sink is exhausted, loops nucleate homogeneously

Irradiation-Induced Nanoclusters and Segregation



Alloy	Clustered Species	Cluster Radius [nm]	No. Density [10^{22} m^{-3}]	Vol Frac [%]
Cast	Ni, Mn, Si, VN, VC	2.26 ± 0.32	4.84 ± 0.97	0.28 ± 0.18
PM-HIP	Ni, Mn, Si	1.80 ± 0.35	4.89 ± 2.46	0.16 ± 0.17

Yield Drop Mechanism

Both Cast & PM-HIP

Dislocation lines are pinned at loops

+

PM-HIP Only

Si segregation to dislocations; Fe-Si bond is stronger, causing dislocation strengthening

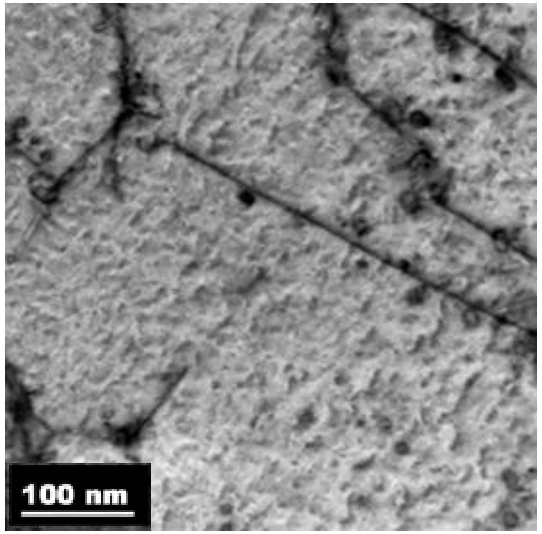
→

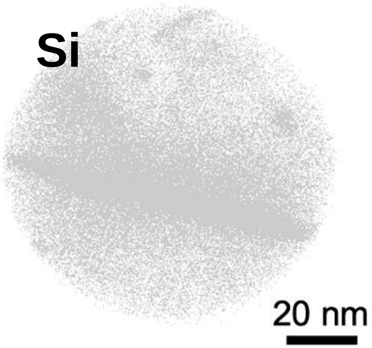
Yield drop more pronounced in PM-HIP because of these *two* factors

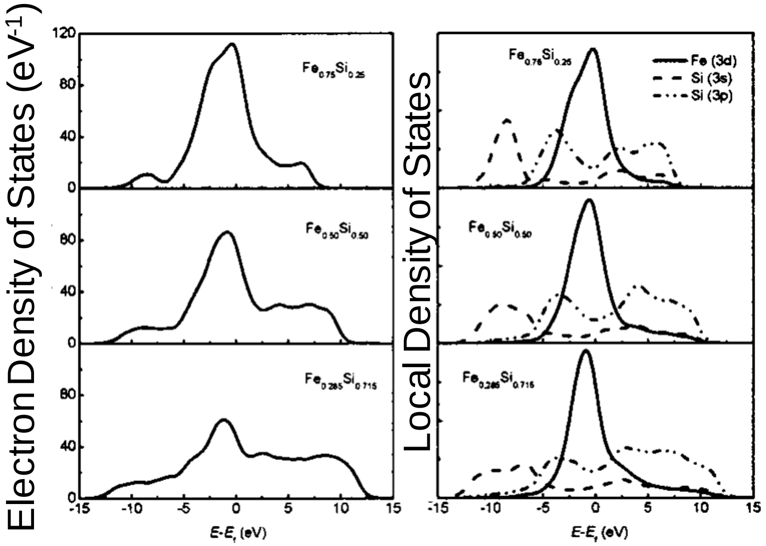
After dislocation overcomes these obstacles, relaxation occurs.

→

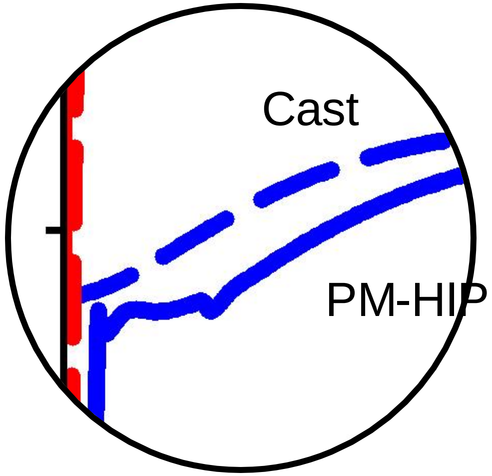
Yield drop phenomenon







Gu, et al. Phys Rev (2004)



Conclusions

- PM-HIP Ni-based Alloys 625 and 690 and Grade 91 ferritic steel exhibit superior irradiation response than their cast/forged counterparts → greater retained ductility and strain hardening capacity due to higher resistance to nucleating irradiation defects
- PM-HIP Grade 91 exhibits unique heterogeneous loop nucleation, leading to yield point phenomenon → current data gap between nucleation and high-dose irradiation studies
- **There are many challenges ahead before PM-HIP structural alloys can be fully codified… but also many opportunities!**

Metrics

- 1 Ph.D. and 1 M.S. degree granted; 1 Ph.D. in progress
- 11 peer-reviewed publications (+ at least 4 more planned/in preparation)
- 23 invited conference, seminar, or workshop presentations
- 13 contributed conference presentations
- Follow-on funding: ~$1.5M DOE-NE CINR/Infrastructure, ~$1M NRC
- 3 follow-on RTEs utilizing irradiated/library specimens (+ 2 related RTEs and 3 pending RTEs)

Follow-On RTEs

- **Awarded**
  - 23-4703: Understanding the origin of irradiation-induced yield drop phenomena in Grade 91 (D.P. Guillen)
  - 22-4415: Irradiation effects on deformation-induced phase transformation in Ni alloys (C.D. Clement)
  - 21-4280: Microstructure examination of irradiation effects on MMC neutron absorber (D.P. Guillen)

- **Related**
  - 18-1412: Irradiated microstructure evolution in cast compared to PM-HIP Alloy 625 (J.P. Wharry)
  - 15-558: Proton irradiations of alloys fabricated by PM-HIP (J.P. Wharry)

- **Pending**
  - Understanding the remarkable strain-hardening capacity of irradiated PM-HIP 316L SS (A. Chatterjee)
  - Synergetic effects of irradiation, temperature, and strain on ordering in Ni-based alloys (J.P. Wharry)
  - Inter-phase localized fracture: A new mechanism for RPV embrittlement (J.P. Wharry)

Thank You!

Acknowledgements:

Marc Albert, Matthew Anderson, Kaitlyn Baird, Jeremy Burgener, Megha Dubey, Elizabeth Getto, J. Keith Jewell, Colin Judge, Rory Kennedy, Yu Lu, Dan Ogden, Darren Pagan, Simon Pimblott, Ching-Heng Shiau, Rongjie Song, Benjamin Sutton, Yaqiao Wu

PURDUE UNIVERSITY

jwharry@purdue.edu

ASME BPVC Status: PM-HIP

Currently in the Code:

- ASME CC N-834 – 316L SS (nuclear)
- ASME CC 2770 – Grade 91 (fossil)
- ASME B31.1 CC approved – Grade 91
- ASME Section VIII CC – Div. 1 and 2 – Duplex SS (29Cr-6.5Ni-2Mo-N)
- Incorporation of ASTM A988, A989, and B834 into ASME Section II
- Section II – Appendix 5

Missing and Needs Qualification:

- Low alloy steel (A508 equivalency) – Material specifications, Section III code case
- Ni-based alloys – Code cases for Alloy 600M, 625, 690, 718
- Longer-term needs – Grade 91, 316H SS, Alloy 617, hardfacing alloys (composite PM-HIP)

PURDUE UNIVERSITY

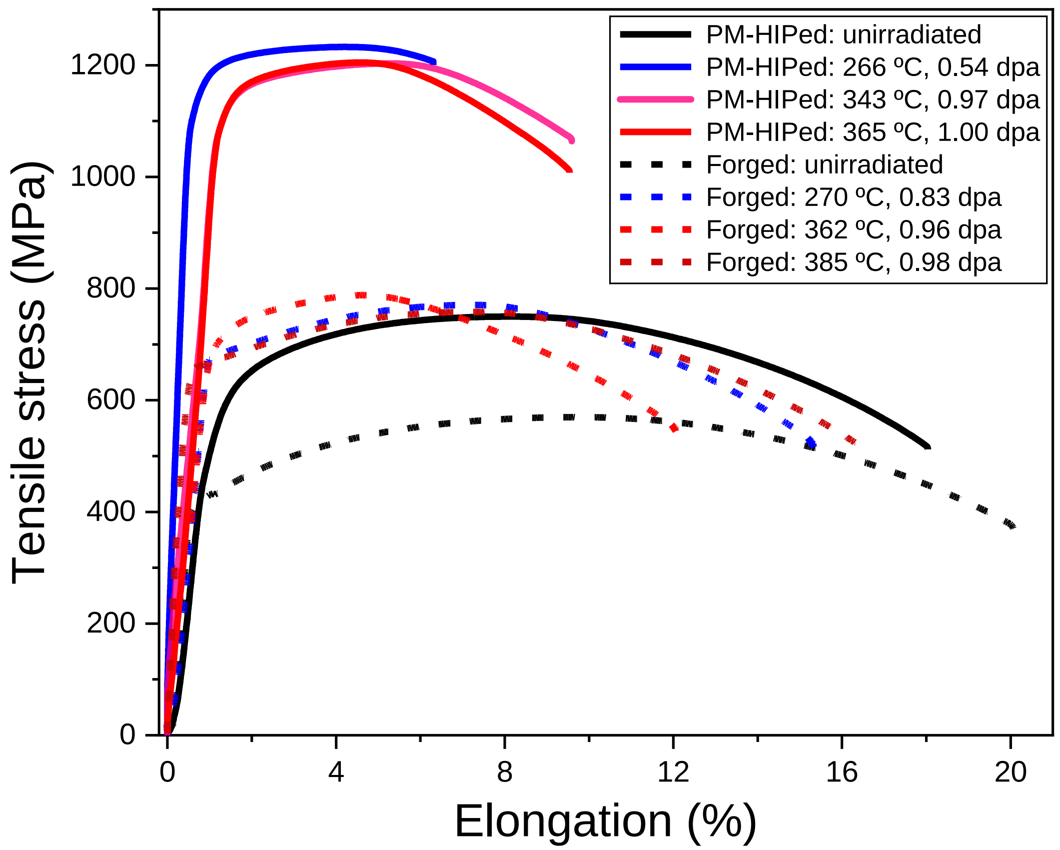
Gaps in PM-HIP Code-Qualification

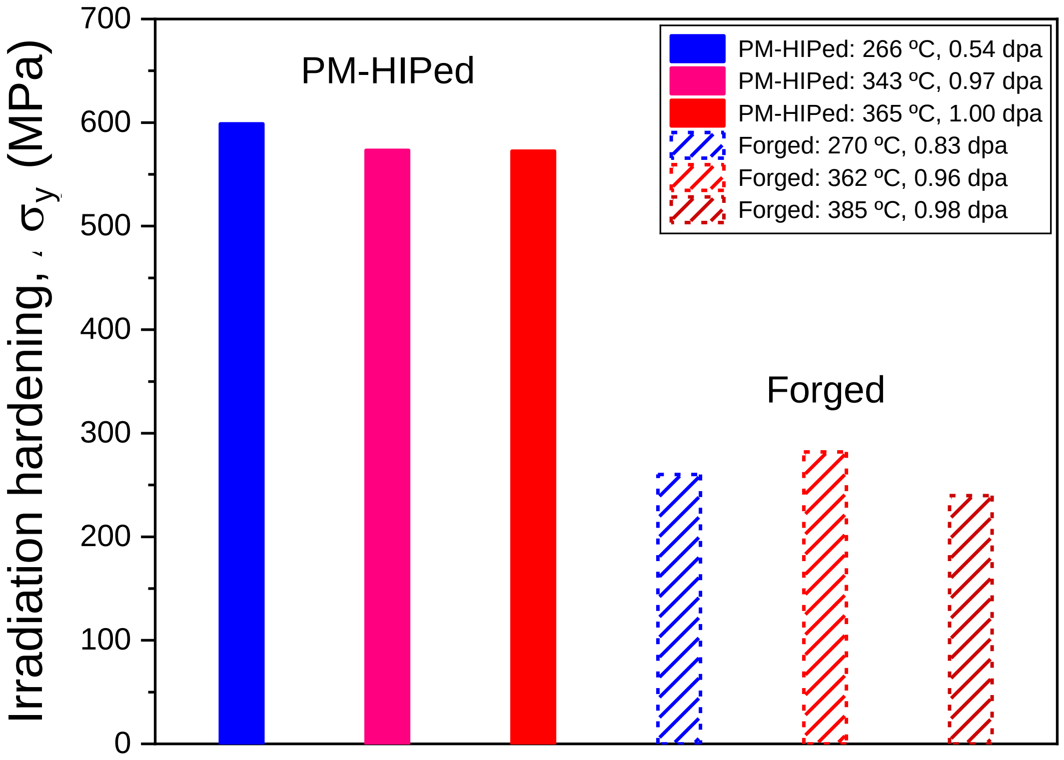
- Material Standards – Additional ASTM specifications need to be developed for Ni-base alloys and low alloy steels (A508 equivalent)
- Environmental Data – SCC for Ni-base alloys
- Fracture Toughness – Needed for low alloy steels
- **Irradiation Performance**
- Creep – Necessary for Division V applications
- Low Alloy Steels – Welding acceptability needs to be confirmed

SA508 Low-Alloy Steel

Microstructure & Mechanical

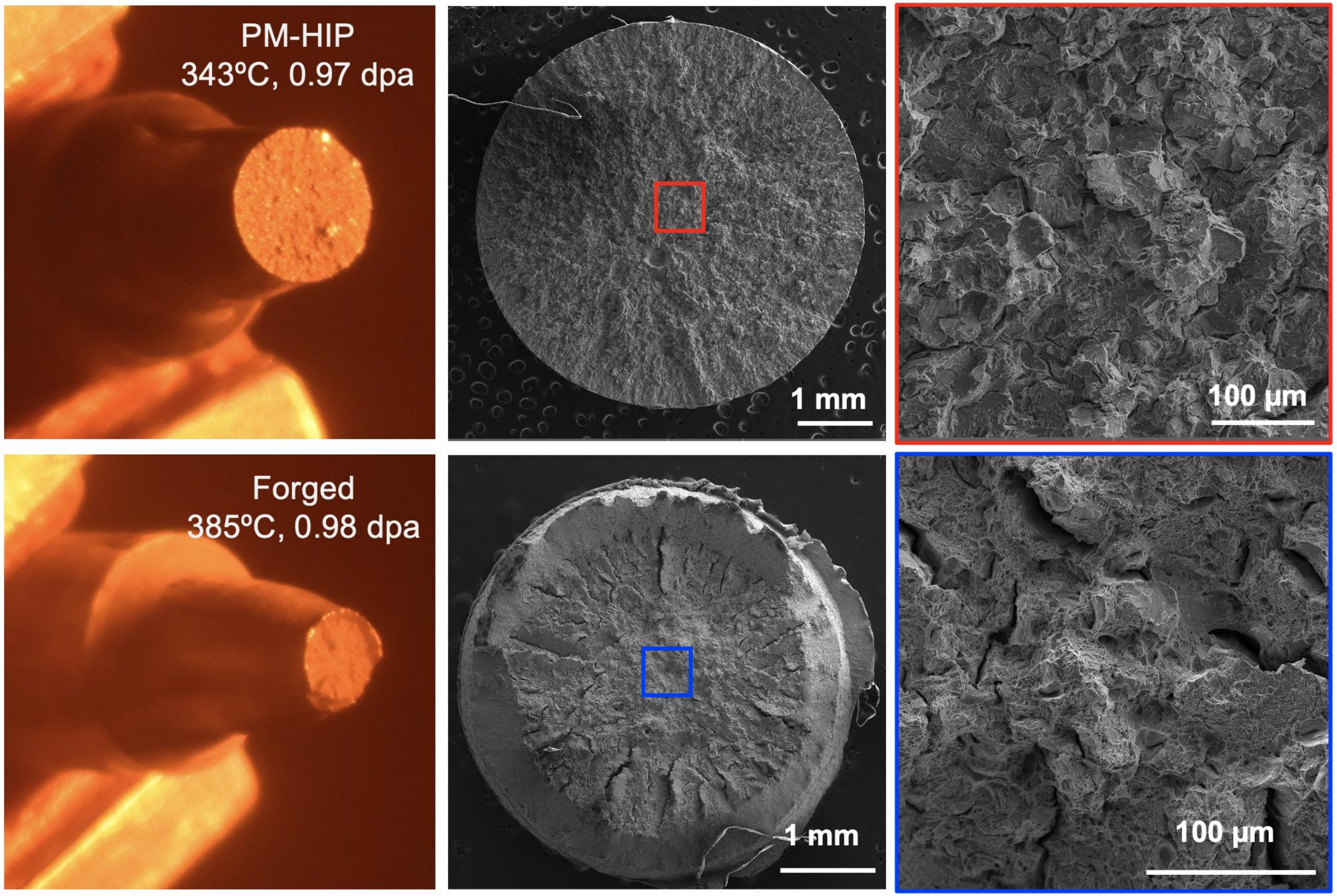
Irradiation Effects on Tensile Properties



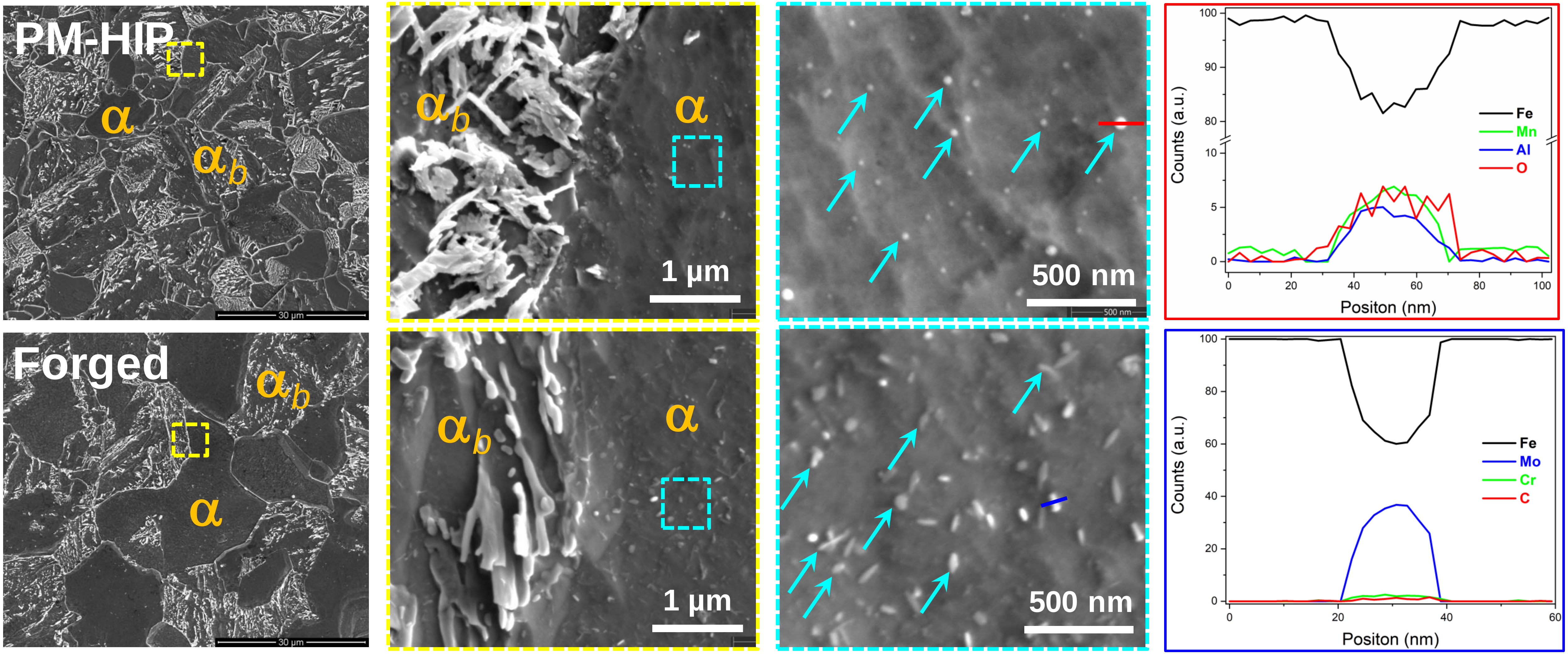


PURDUE UNIVERSITY

Fractography

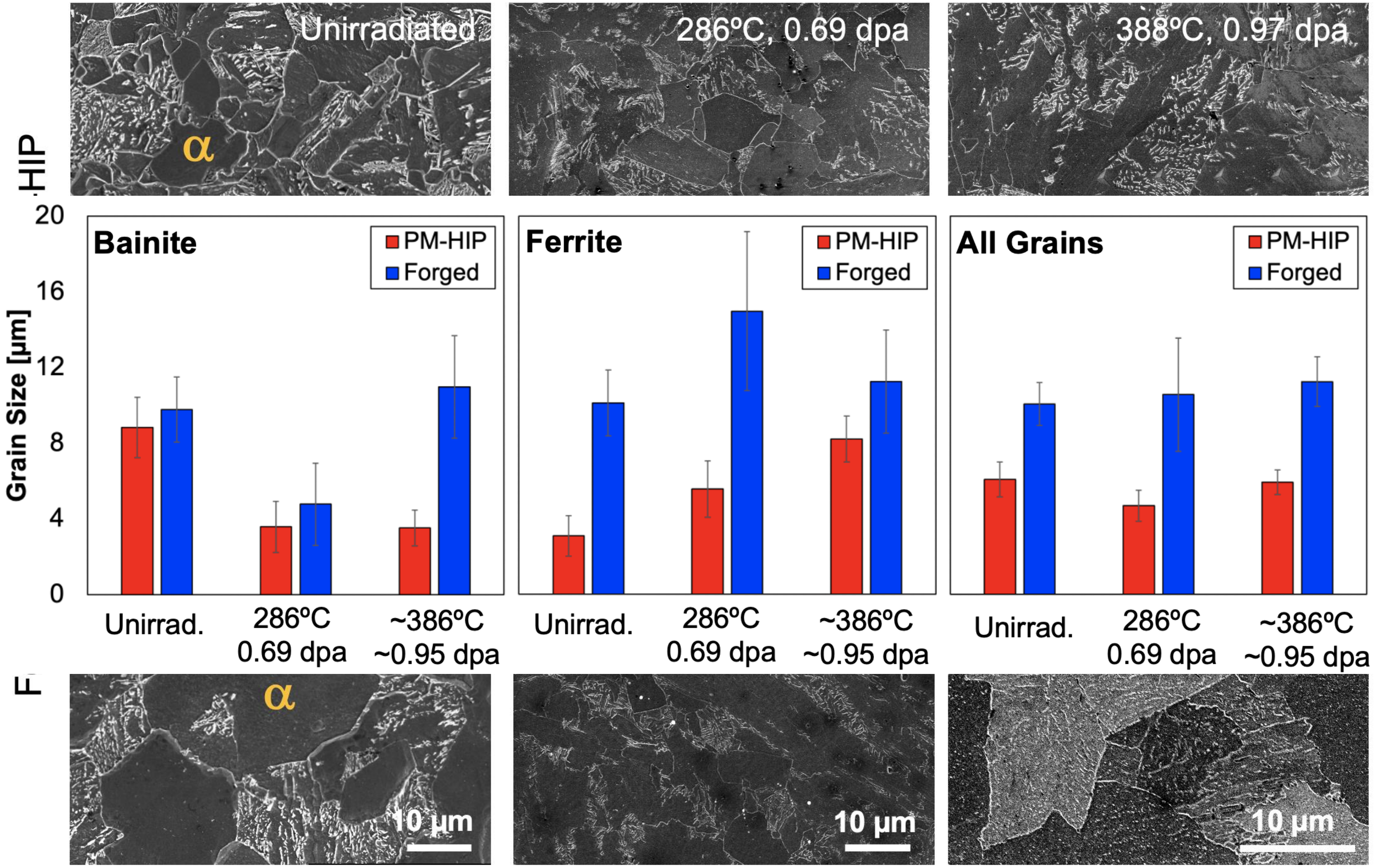


Initial Grain Structure Comparison

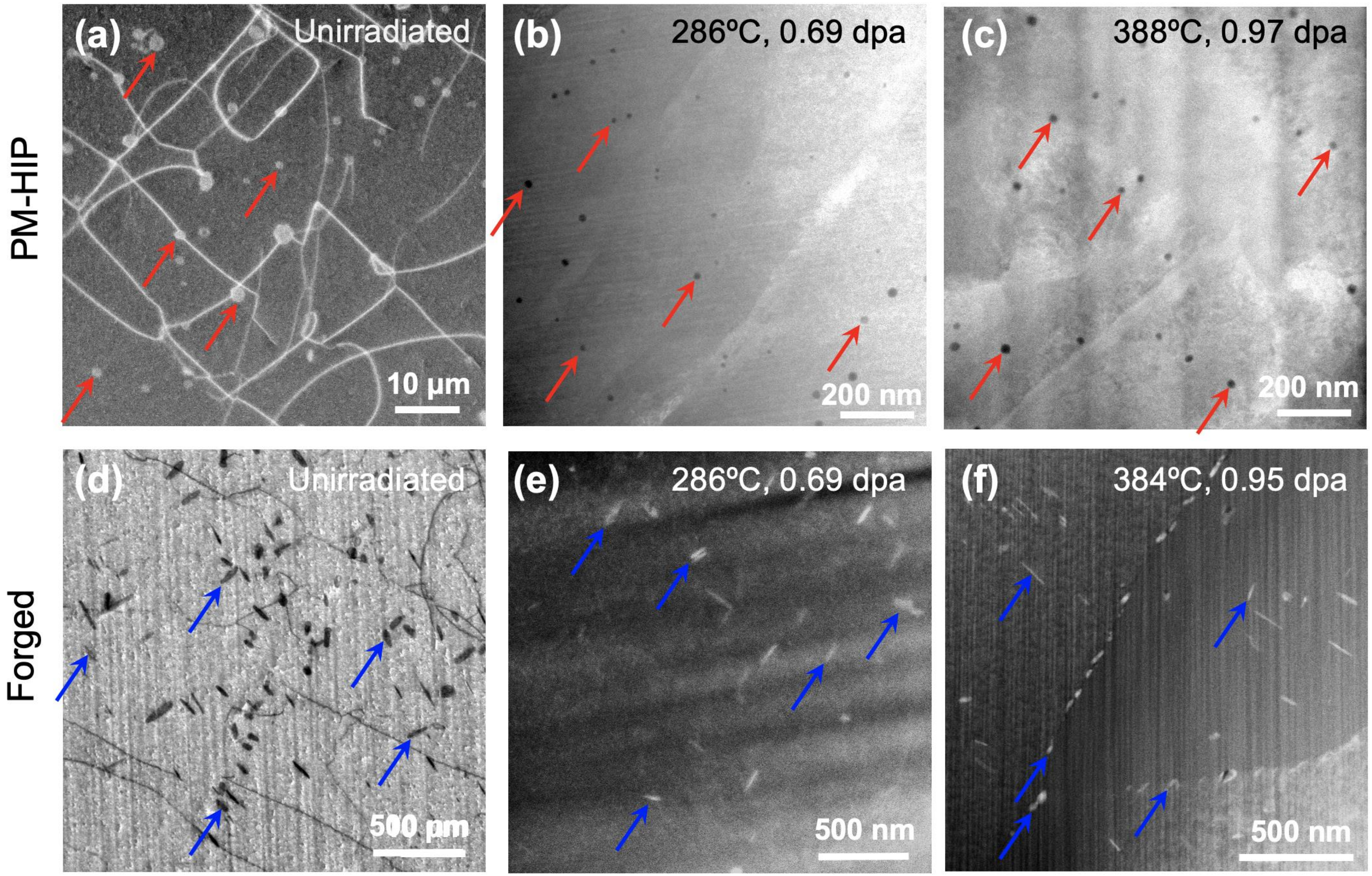


PURDUE UNIVERSITY

Phase Stability Under Irradiation

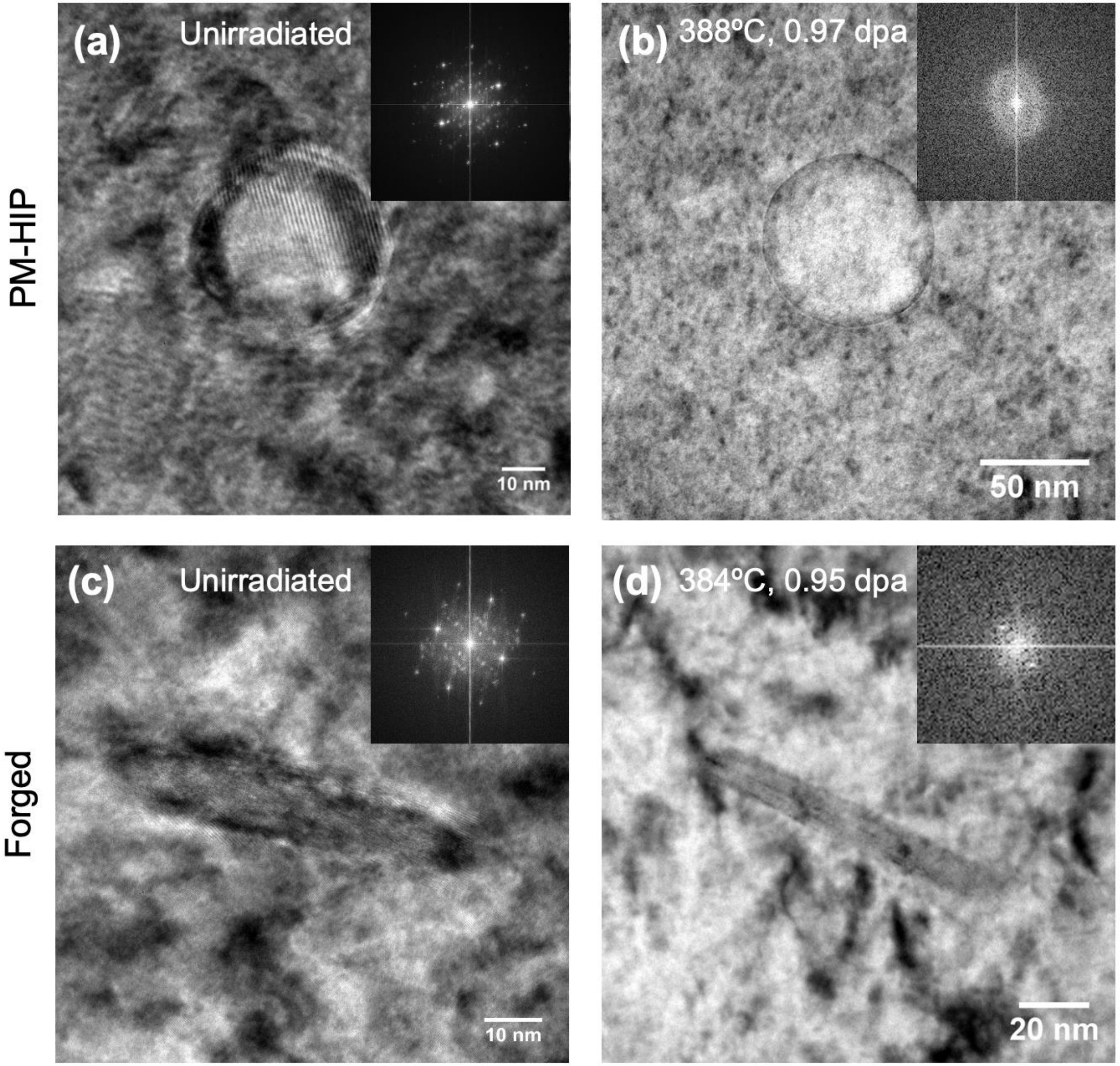


Precipitate Morphological Evolution

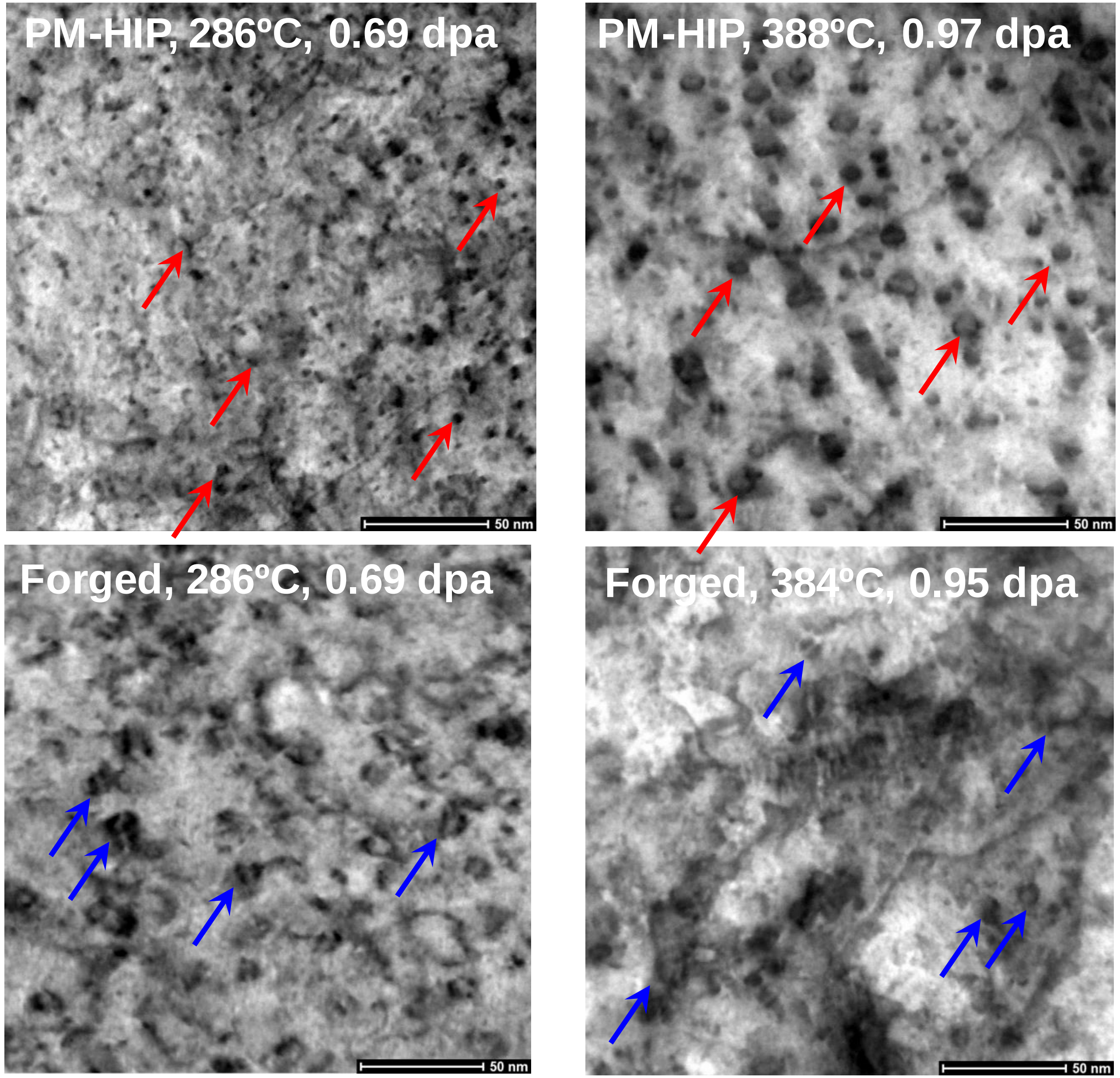


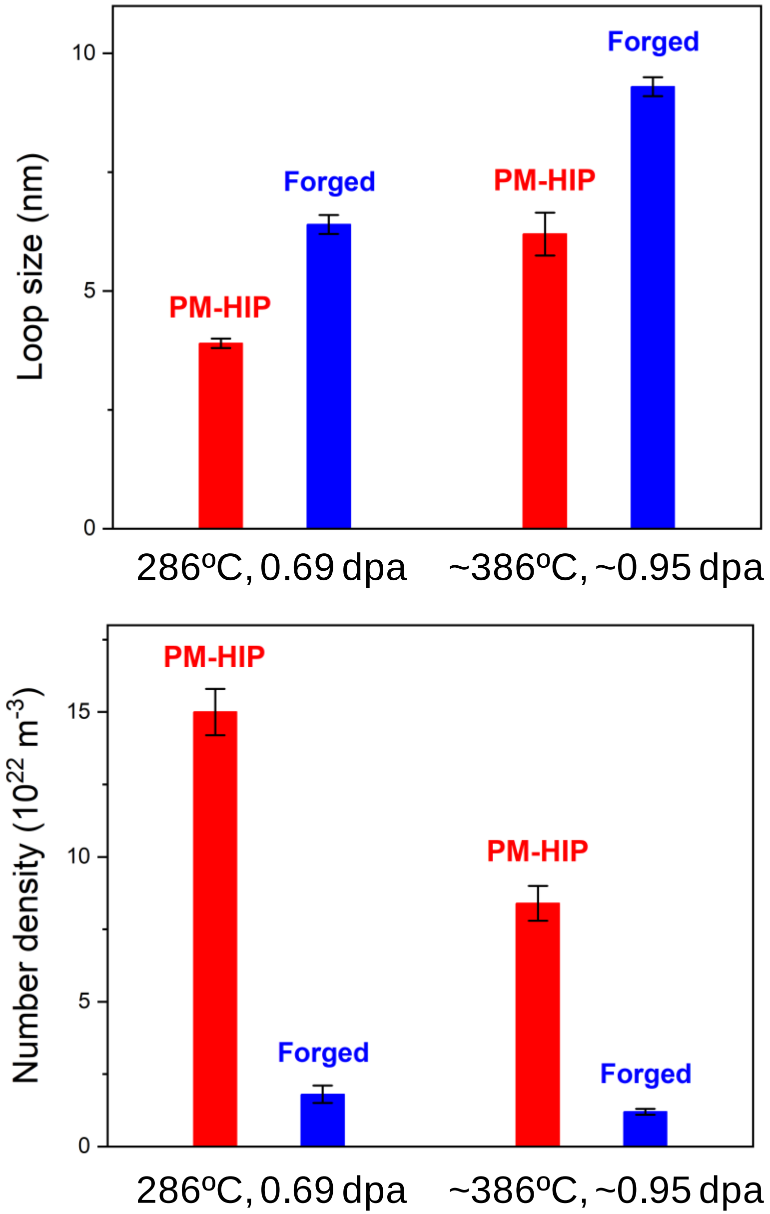
PURDUE UNIVERSITY

Precipitate Phase Evolution Under Irradiation

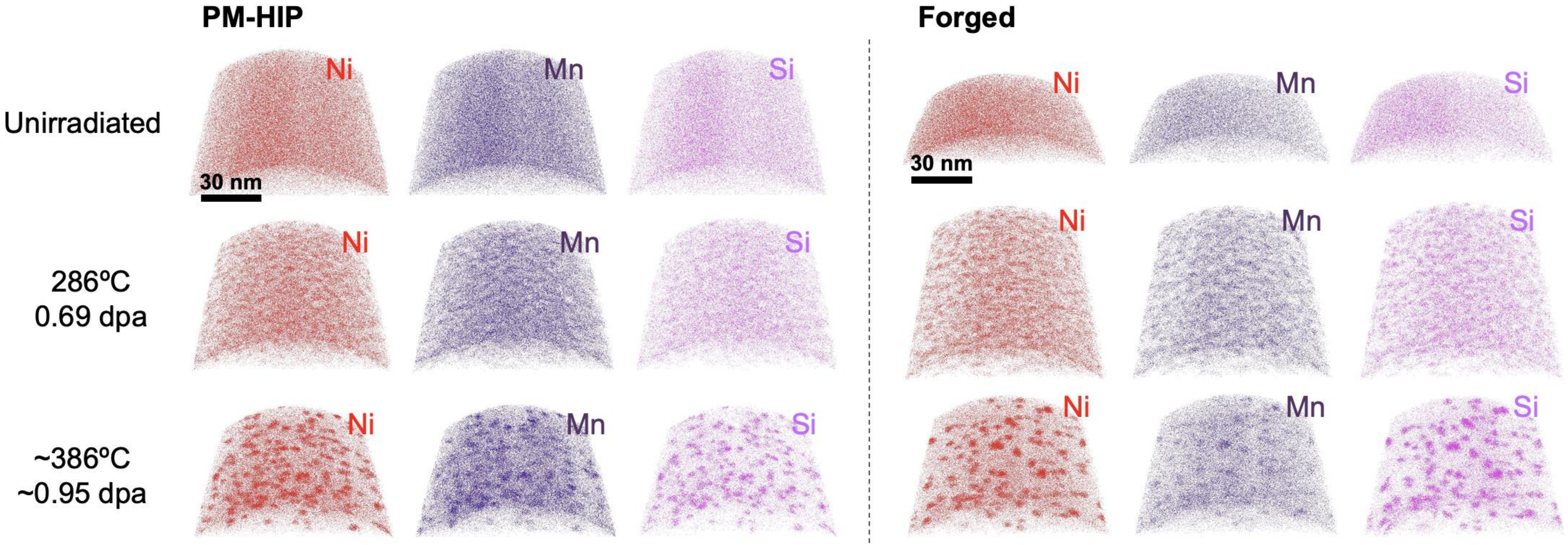


Dislocation Loop Evolution

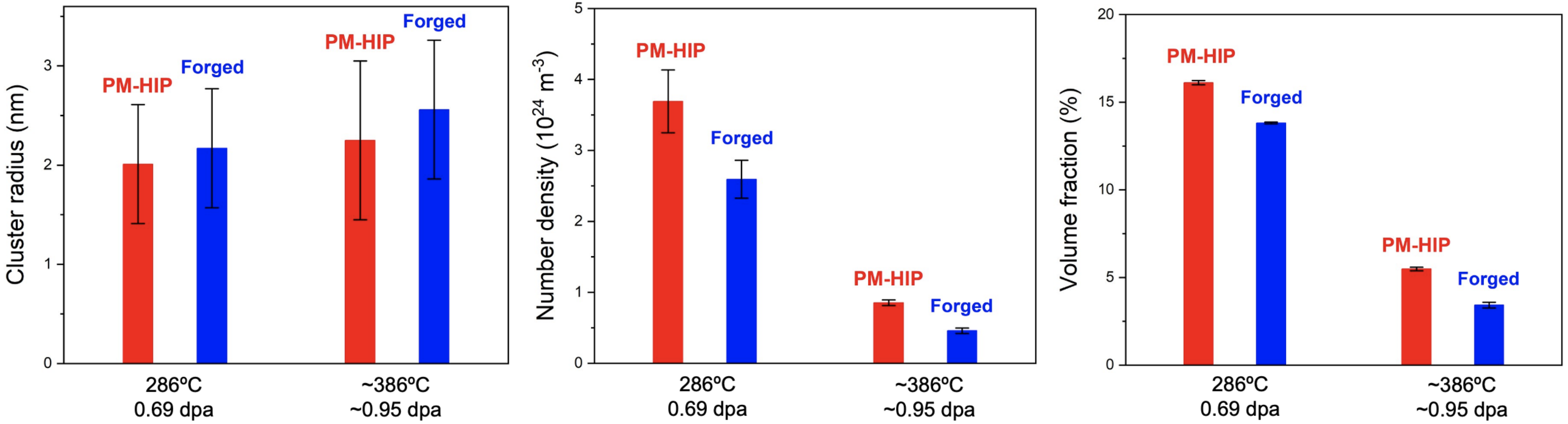




Nanocluster Evolution Under Irradiation

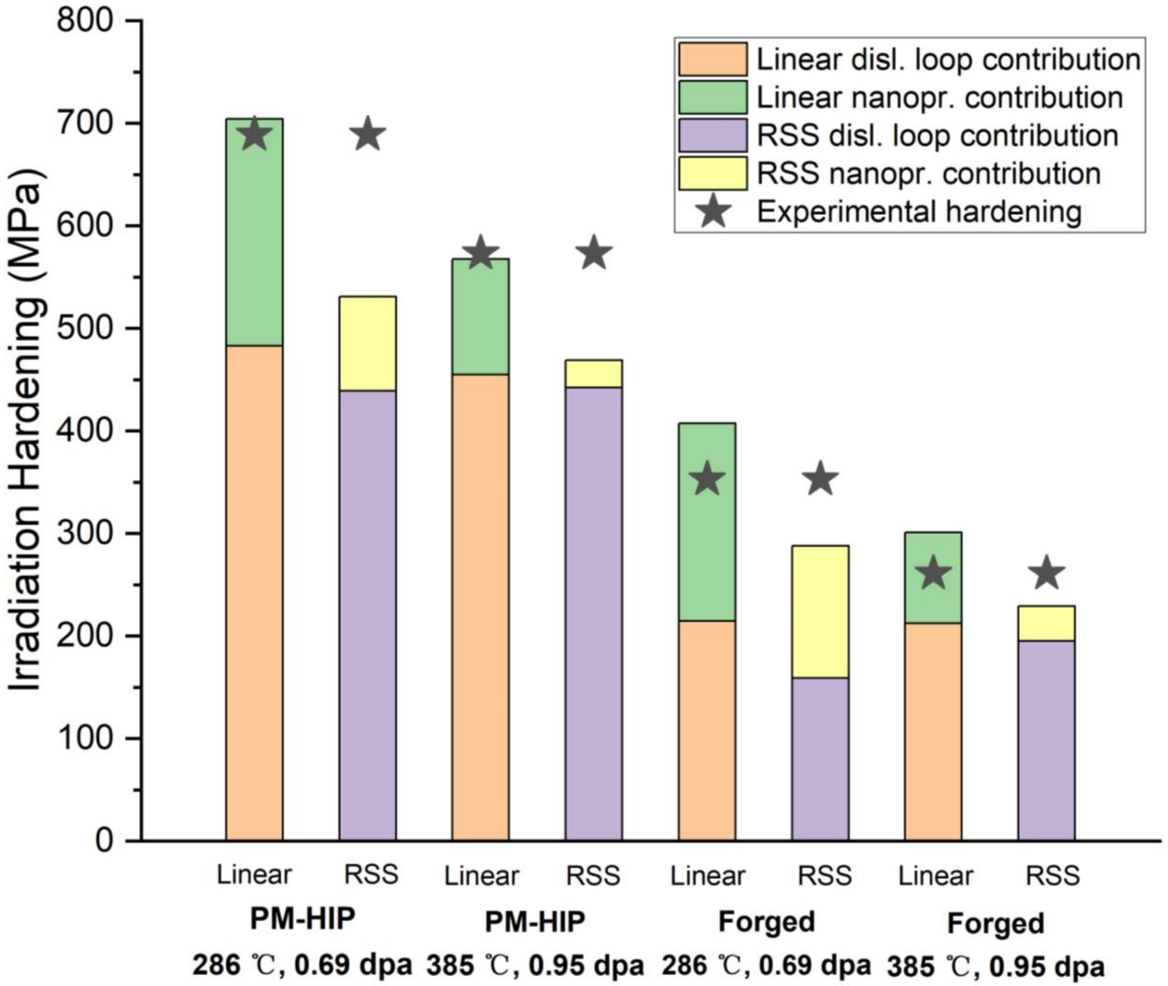


Nanocluster Evolution Under Irradiation



PURDUE UNIVERSITY

Dispersed Barrier Hardening Explains Structure-Property Relationship



PM-HIP
Linear good fit, RSS underestimates → Loops and nanoclusters have comparable strengthening effect

Forged
Experimental measurements ~halfway between linear and RSS → Loops are dominant obstacle

SA508 Materials

Alloy	C	Si	Mn	Ni	Cr	Mo	V	P	S	Fe
PM-HIP	0.01	0.21	1.39	0.79	0.18	0.37	-	0.002	0.005	Bal.
Forged	0.02	0.31	0.46	0.50	0.21	0.26	0.01	0.003	0.007	Bal.
ASTM	<0.25	0.15-0.40	1.20-1.50	0.40-1.00	<0.25	0.45-0.60	<0.05	<0.025	<0.025	Bal.

HIP: 103 MPa, 1121^{o}C, 4 hr

Heat Treatments (Both PM-HIP and Forged):

- Solution anneal 1121^{o}C, 2 hr, water quench
- Normalization 899^{o}C, 10 hr, water quench
- Tempering 649^{o}C, 10 hr, air cooling

316L Stainless Steel

Mechanical Behavior

Tensile Testing & Nanoindentation