Determining the Effects of Neutron Irradiation on the Structural Integrity of Additively Manufactured Heat Exchangers for Very Small Modular Reactor Applications

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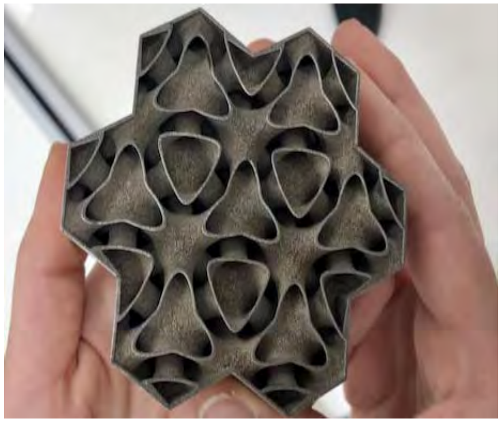
Additive Manufacturing of Heat Exchangers (HeXs)

AM enables novel geometry, materials & performance

- Part/joint consolidation
- Non-uniform cross-sectioned channels
- Asymmetric core architecture
- Fully-circular channels (as opposed to semi-circular)
- Reduced wall thicknesses
- Stream-tailored (cold/hot channel) designs
- And more



https://www.metal-am.com/

U.S. Department of Energy

Additive Manufacturing of Heat Exchangers (HeXs)

- Components for GEN IV and Small Modular Reactors (SMR) to make them simpler, smaller, more efficient, and safer

- AM can play a vital role in manufacturing geometrically complex parts for these radiation environments
    - Compact heat exchangers, manifolds, rods, etc.

- Radiation effects in AM metals are still not well understood, so more research is required to determine how these AM metals respond in nuclear environments compared to their conventionally wrought counterparts.

https://www.iaea.org/newscenter/news/small-modular-reactors-a-challenge-for-spent-fuel-management

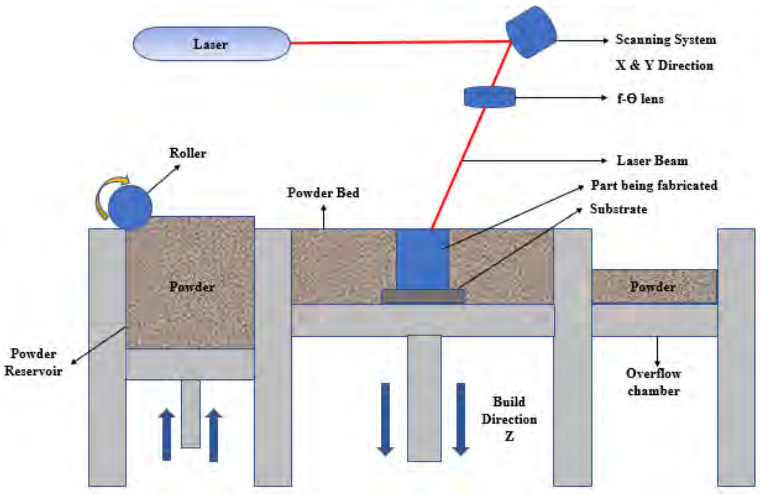
https://design-engineering.polimi.it/portfolio/additive-manufacturing-and-heat-exchange

Microreactor Program

Project Objectives

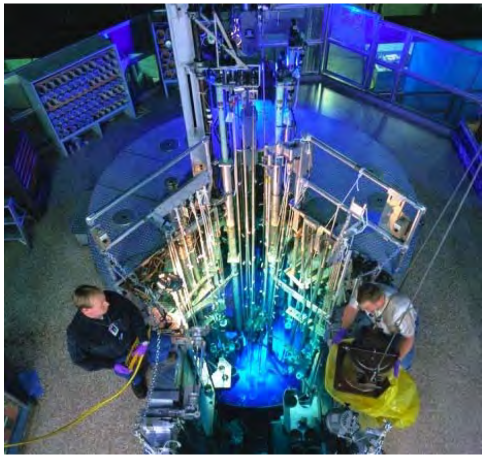
- Investigate Inconel 625 and Inconel 718 samples fabricated via laser powder bed fusion (L-PBF) with wrought control group. L-PBF samples to be in as-printed-vertical, as-printed-horizontal, and heat-treated states. For these sets of materials:
  - Determine effects of *full spectrum* neutron exposure
  - Determine effects of *fast neutron* exposure

Project Objectives



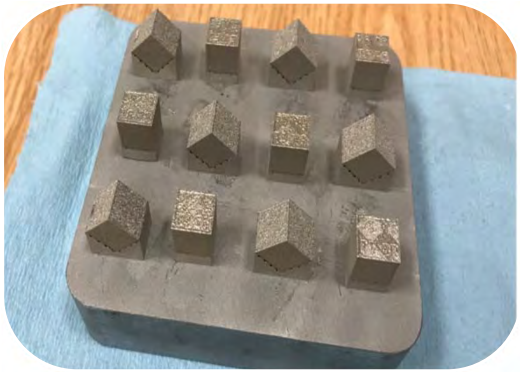
Variables:

- Manufacturing method (L-PBF vs. wrought)
- Microstructure (via heat treatment)
- Interlayer porosity orientation (print orientation)



Dosing:

- None (control group)
- Full spectrum neutrons
- Fast neutrons



Potential radiation defect mechanisms for L-PBF samples:

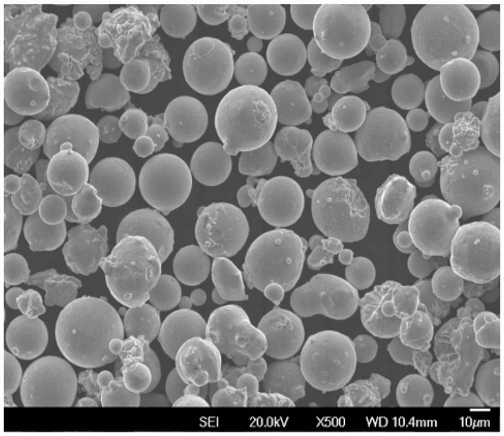
- Gas porosity
- Inclusions
- Residual stress field
- Precipitates and dislocations
- Oxygen content
- Chemical segregation

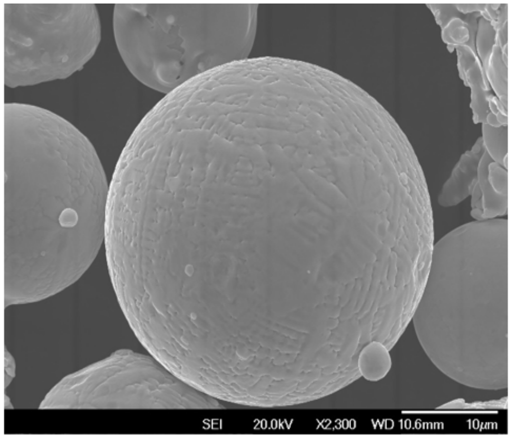
Measured Before and After Irradiation:

- Microhardness
- Microstructure

Manufacturing: L-PBF of Inconel 625

- Several 10 mm^3 cubes were printed using a Concept Laser Mlab 100R system. 1 x 1 x 0.5 mm^3 specimens were removed from cubes via EDM.
- ASTM F3056-14(2021)/UNS N06625 standard Inconel 625 powder
- Powder particle sieve down to 31 μm diameter
- Vertical and diagonal orientations
- Laser Power: 90 W
- Scan speed: 800 mm/s
- Layer thickness: 25 µm
- Hatch spacing: 60 µm



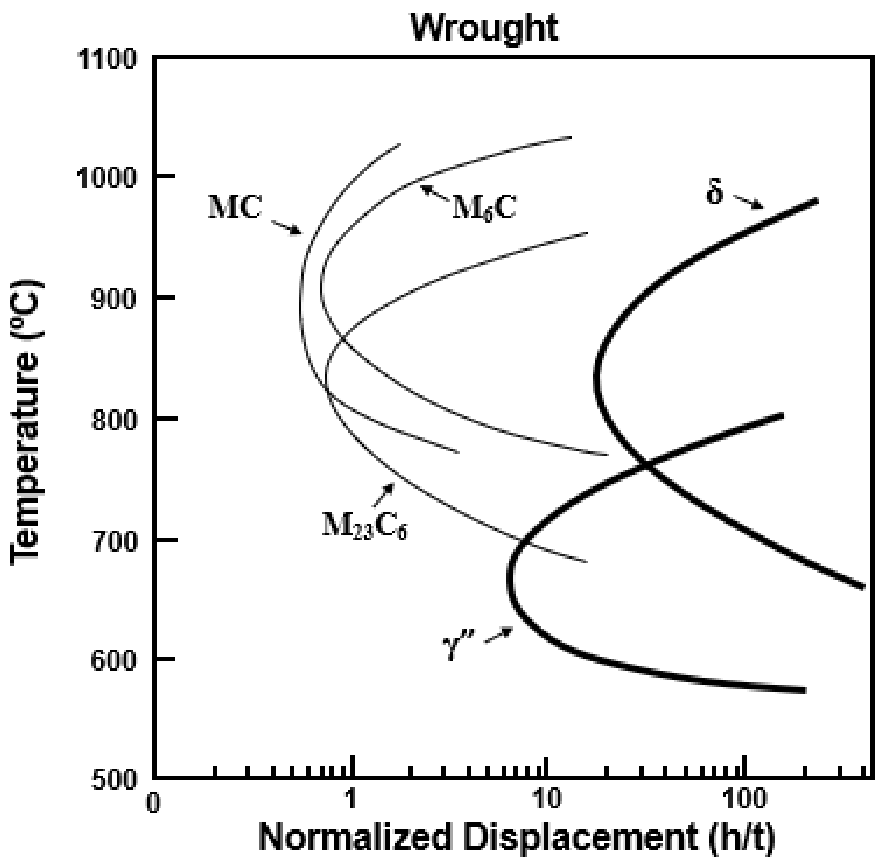


Element	UNS N06625	AM IN625 powder	Wrought IN625
Ni	Bal	Bal	Bal
Cr	20-23	21.59	22.08
Mo	8-10	9.0	8.52
Fe	<5.0	2.95	4.35
Nb+Ta	3.15-4.15	3.55	3.41
Al	<0.4	0.1	0.1
Ti	<0.4	0.1	0.1
Mn	<0.5	<0.5	<0.5
Co	<1.0	0.03	0.8
C	<0.1	0.04	0.03

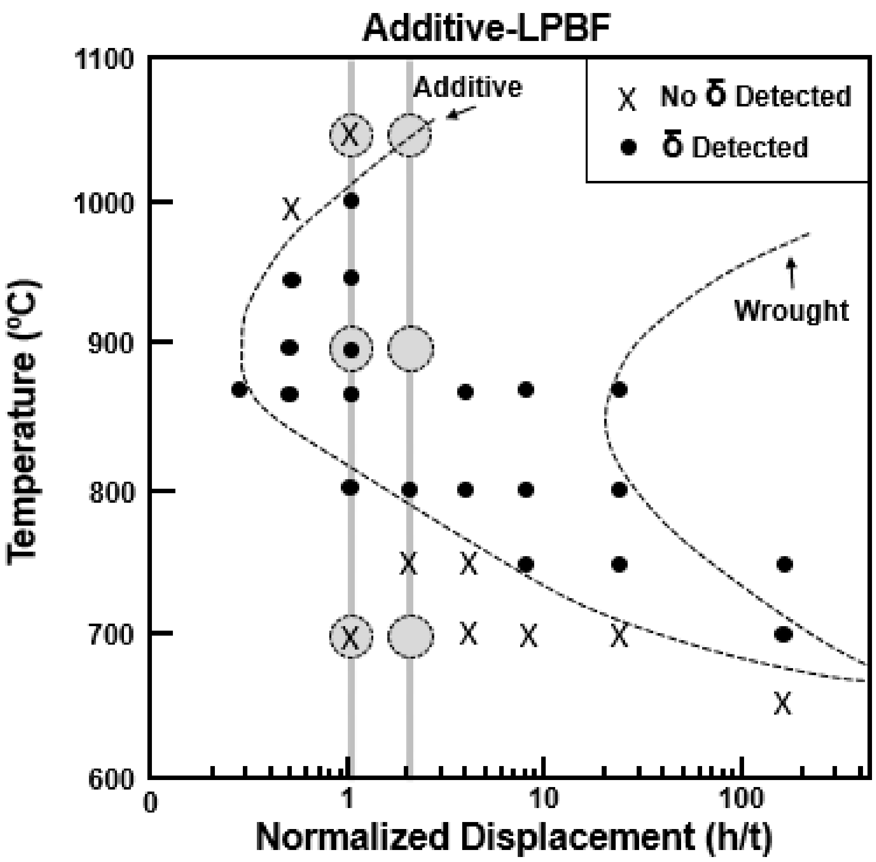
MRP Microreactor Program

Methods: Heat treatment schedules

- 700, 900, 1050 ℃ – 1 hour was selected for this study to observe effects of intermetallic phases.

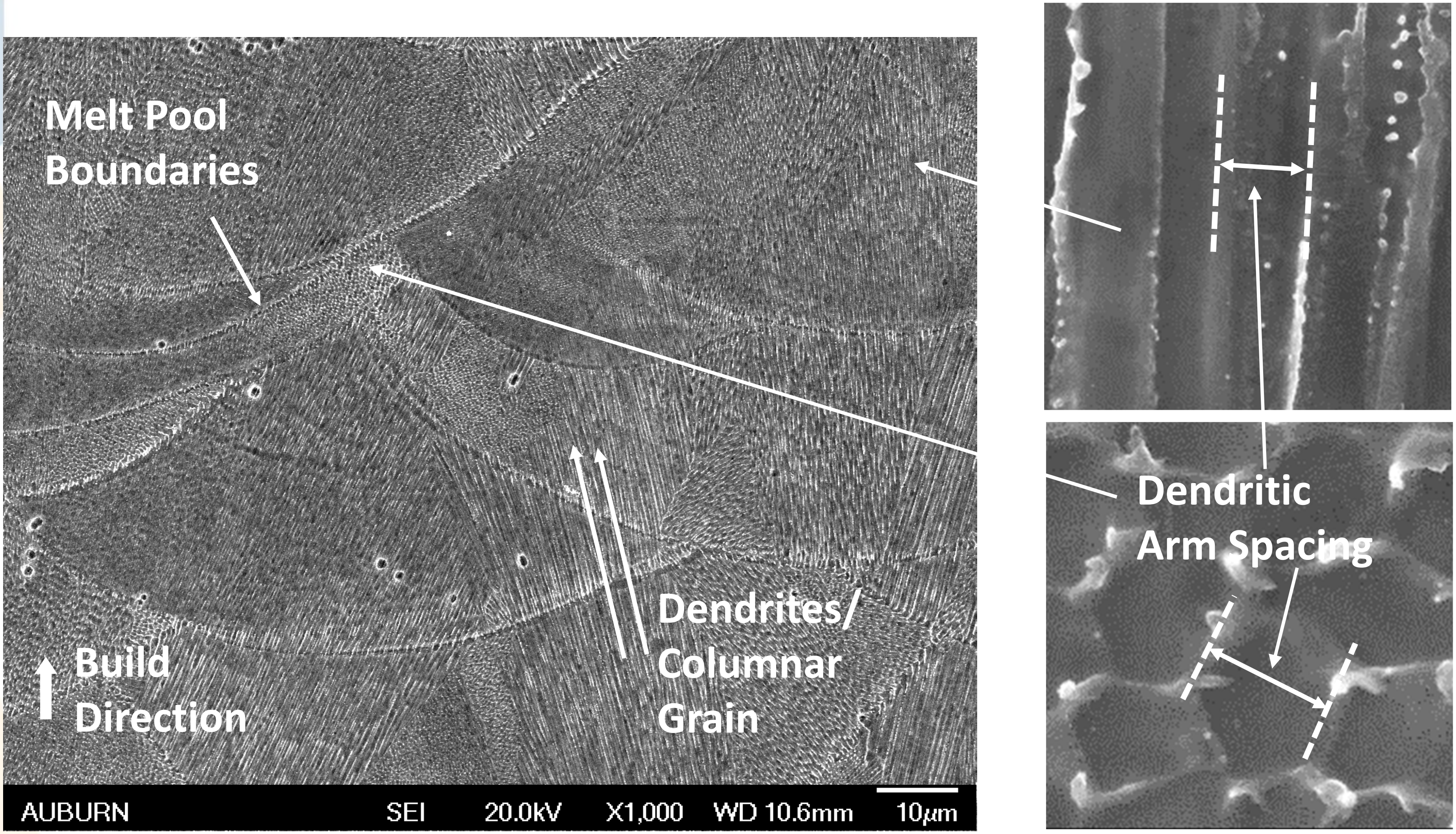


Shoemaker, Superalloys ***718*** *(625), 409 (2005)*



NIST – Stoudt, et al., Met. Trans. ***49A****, 3028 (2018)*

Results: As-built vertically printed Inconel 625 microstructure (non-irradiated)



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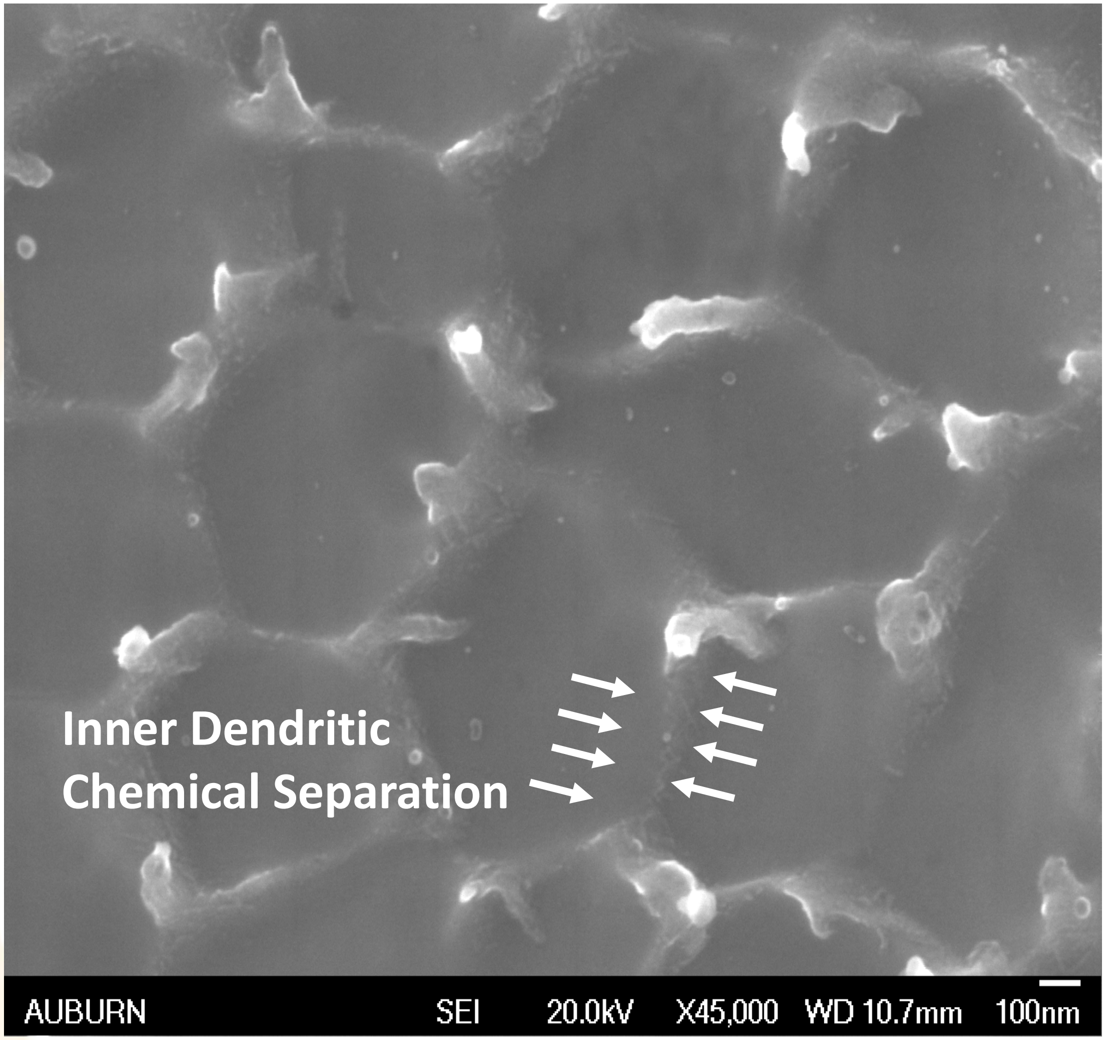
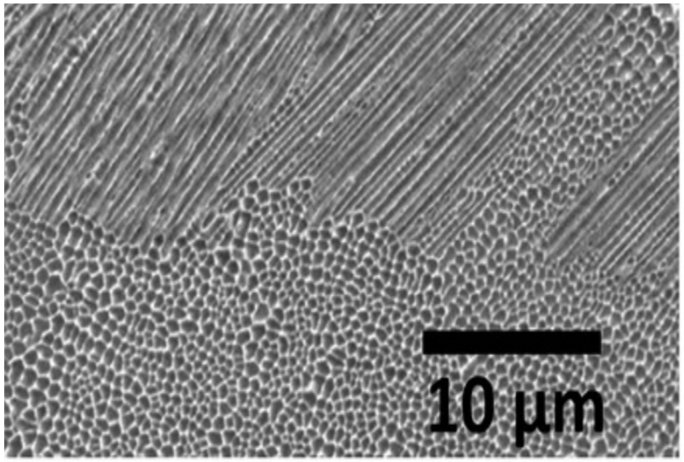
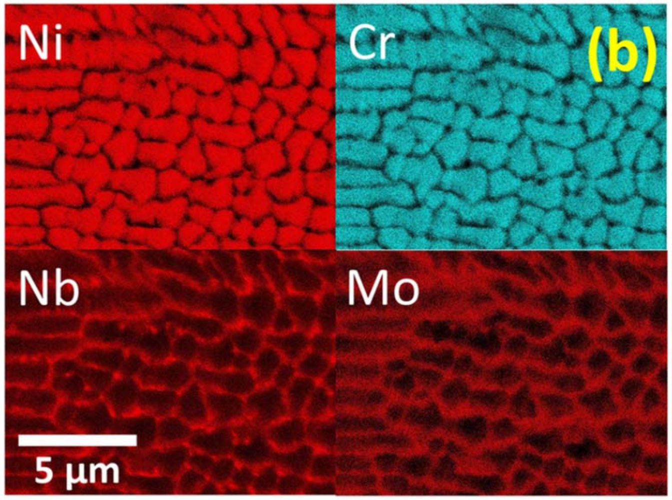


Zhang, et al., Scripta Mater 131, 98 (2017)

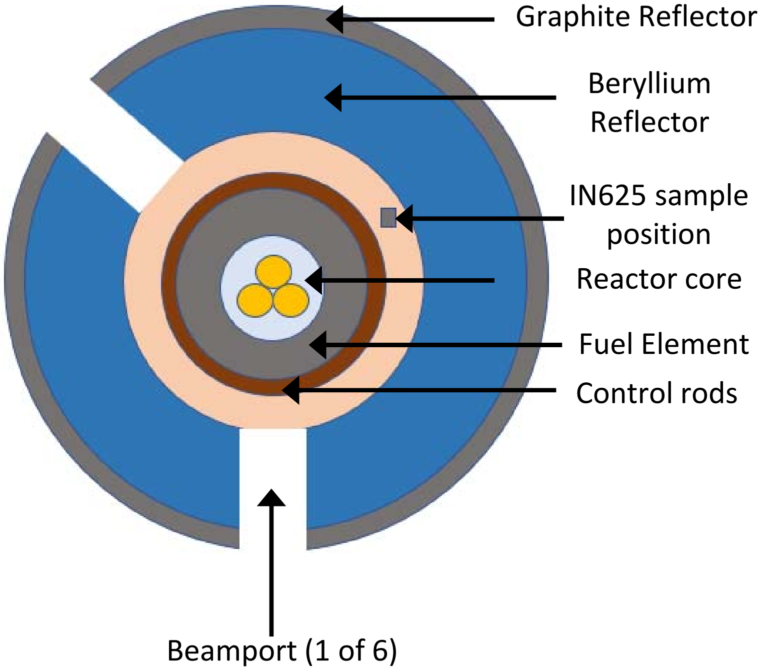


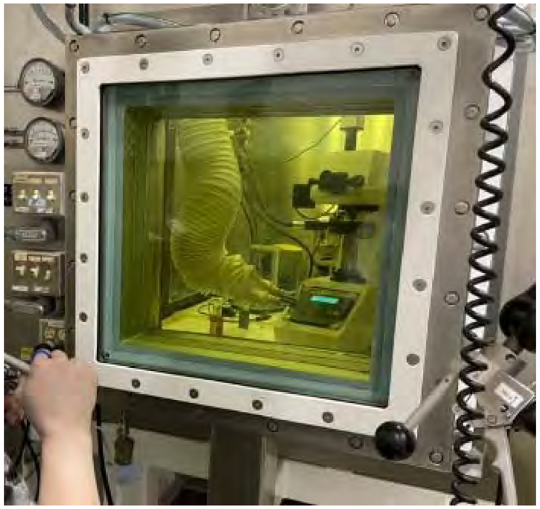


Issue for precipitate formation

Method: Full spectrum neutron irradiation

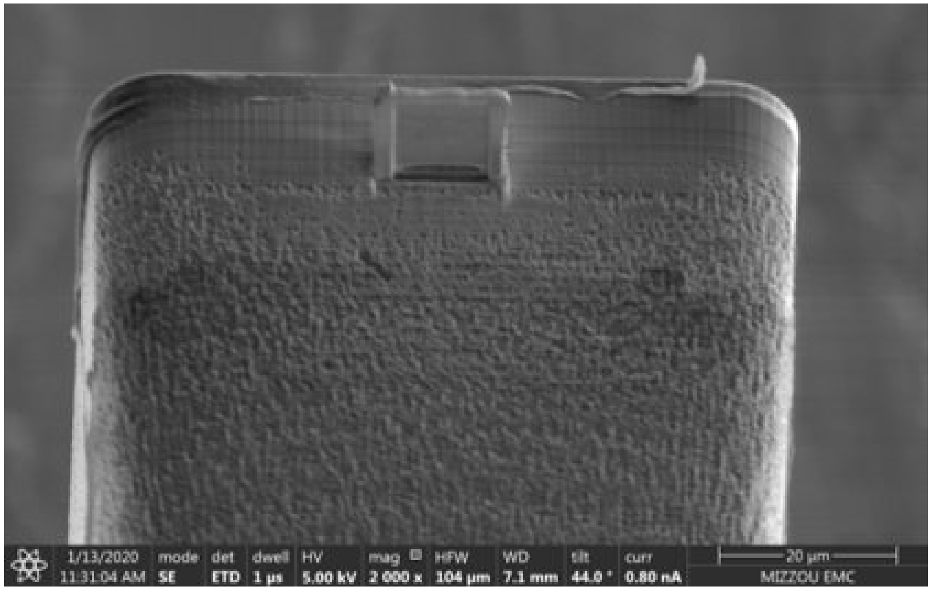
- University of Missouri Research Reactor (MURR) with 10 MW capacity (highest for any academic research reactor)
- Irradiation time: 310 hours
- Flux: 6.61 x 10^{13} n/cm^2/s (at radiation position in reflector)
- Total fluence: 7.37 x 10^{19} neutrons/cm^2 (approx. 90% thermal and 10% fast neutrons)
- **DPA: approx. in range 0.01-0.11**
- 1 inch diameter cannister in the reflector
- 12 samples (4 vertical AM, 4 diagonal AM, 4 wrought) (different conditions: as built, 700, 900, and 1050 °C)
- Activation: approx. 176 Ci upon removal from reactor, approx. 4.54 Ci after 120 days.
- Testing of radioactive samples were performed in the hot cell





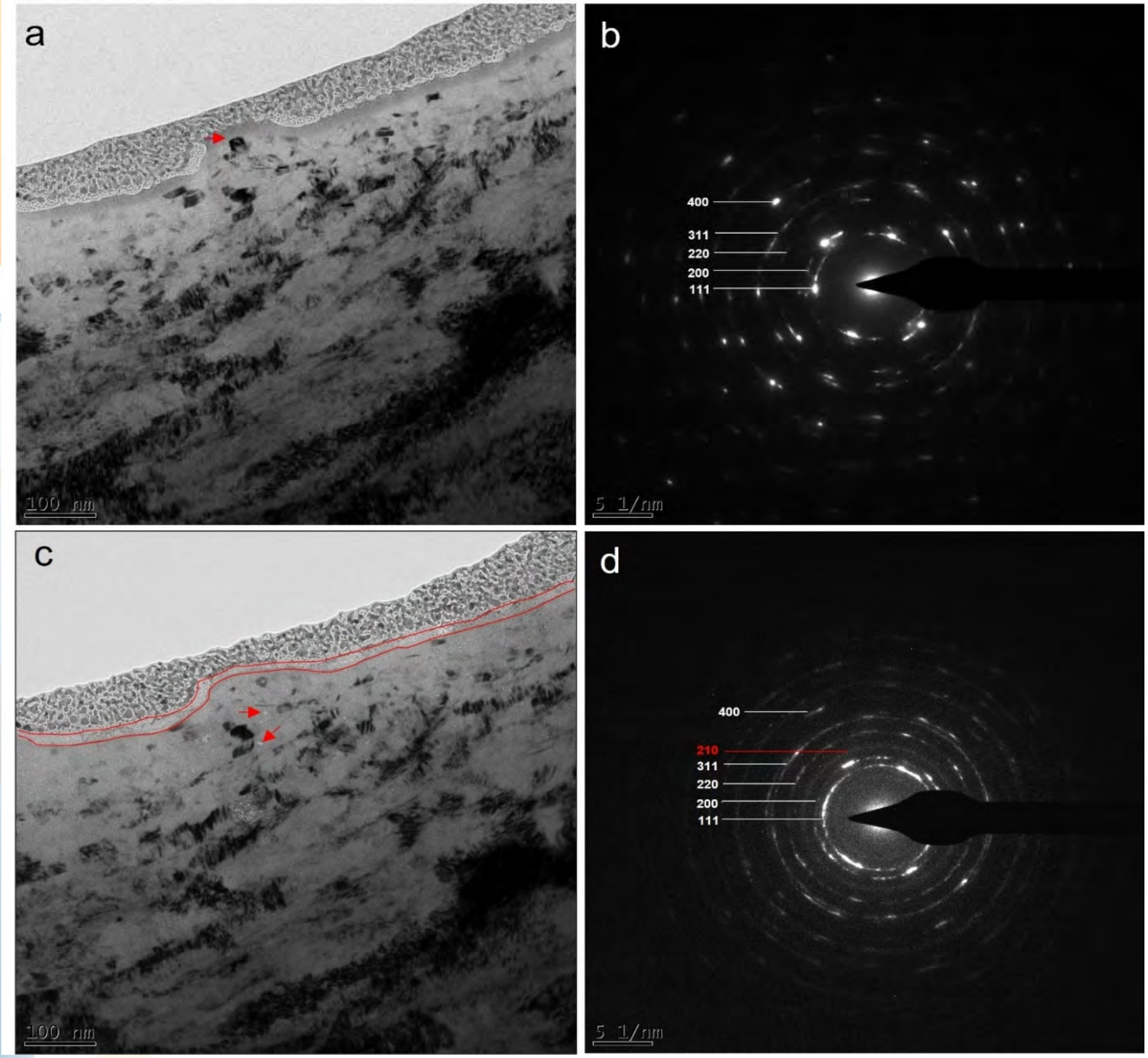
Method: Lamella creation

- By reducing the amount of material that becomes activated in a reactor setting, you can decrease activity personnel are exposed to during post-irradiation processing of the materials.
- Lift-out grid purpose is to hold the lamella
- Lamella approximately 15 µm by 8 µm and less than 100 nm thick
- Lamellae are extremely delicate
- Grid made of silicon, chosen for its low neutron activation
- Grids are approximately 3 mm by 2 mm and 100 µm thick



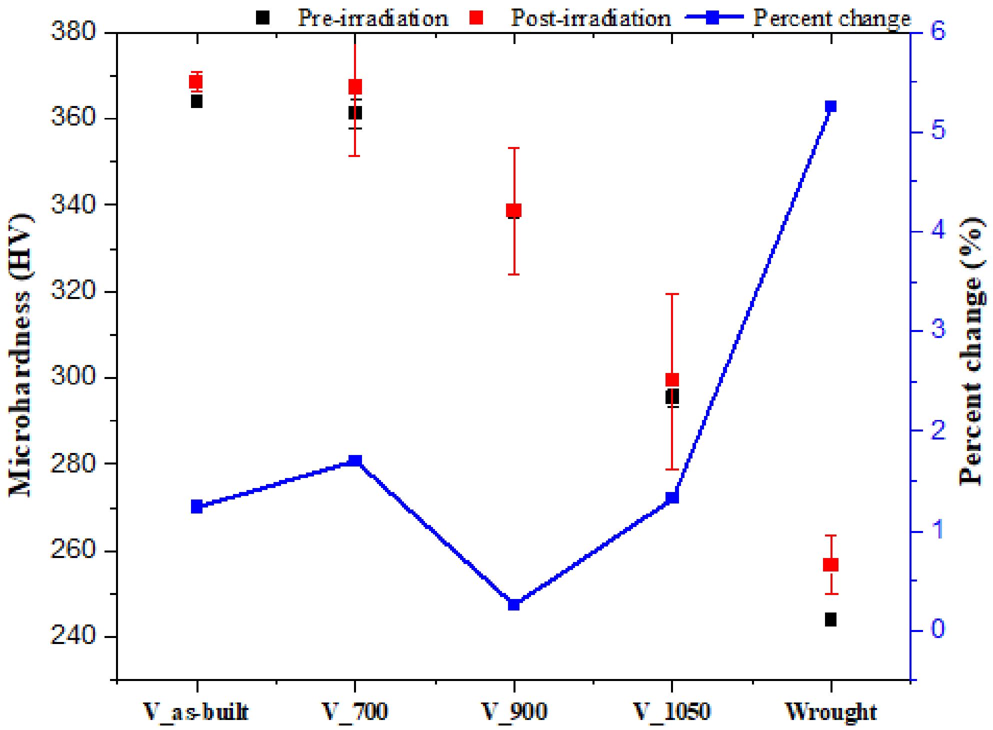
MRP Microreactor Program

Results: Post-irradiation TEM analysis of AM IN625

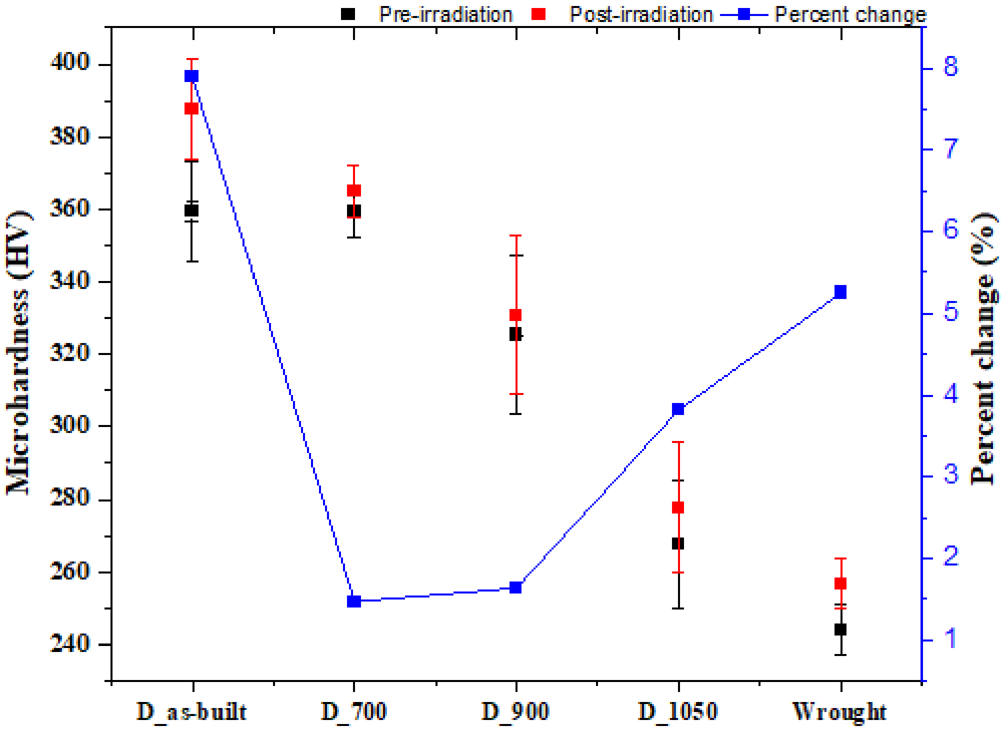


The kinetic input from neutron bombardment enabled the similarly oriented dendrite structure to begin recrystallizing into new grains of random orientation.

Results: Post-irradiation microhardness



Vertical



Diagonal

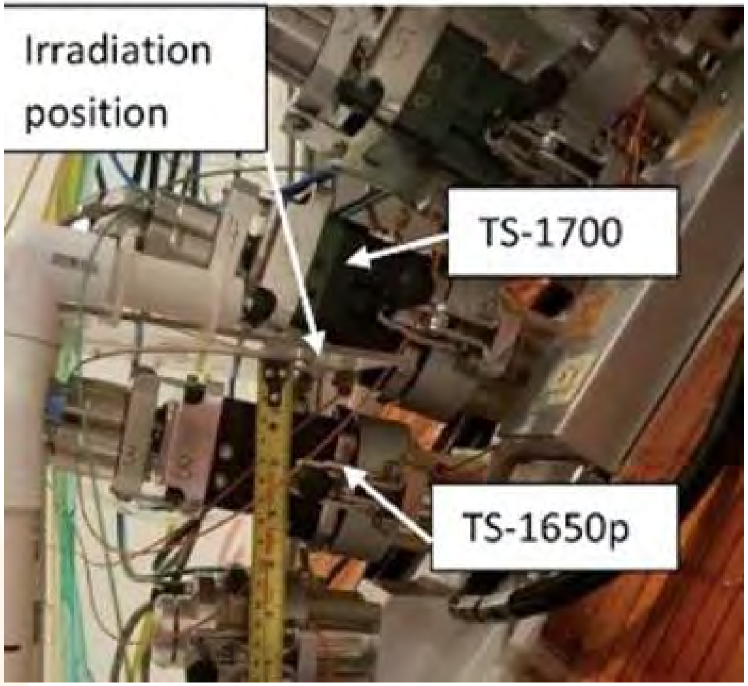
Radiation hardening was more prominent in wrought sample compared to most of the AM Inconel 625 samples.

Methods: Full spectrum vs fast neutron

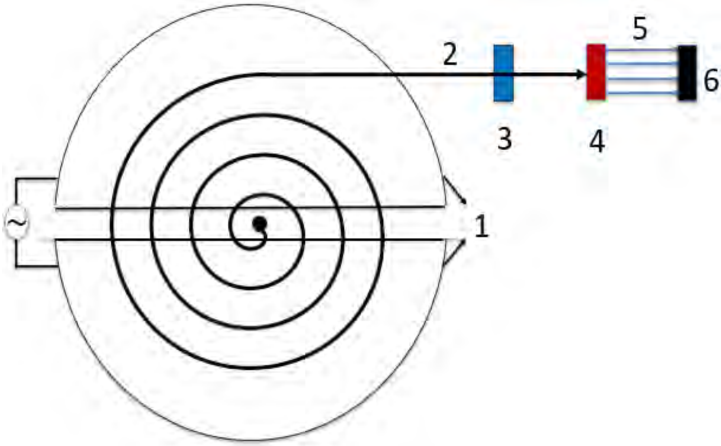
Reactor based neutrons	Fast neutrons
Thermal neutrons can move through the crystalline material without displacing any host atoms.	Fast neutrons greatly disturb the periodic structure of a crystalline material due to displacement damage effects.
Thermal neutrons have high absorption cross section and low scattering	Fast neutrons have high scattering cross section and low absorption
Higher potential to create isotopes in the alloy resulting in higher radioactivity	Lower potential to create isotopes in the alloy resulting in lower radioactivity

Methods: Fast neutron irradiation

- Samples used in the fast neutron irradiation experiment were picked from the same batch as those used in the full spectrum neutron irradiation experiment for continuity.
- Fast neutron irradiation was carried out in the vault of a PET cyclotron at MURR.
- Four batches of IN625 and IN718 were irradiated for 7, 17, 20, and 25 weeks.
- Fluence experienced after 7 and 17 weeks were fluence of 2.74 x 10^{15} and 6.61 x 10^{15} neutrons/cm^2, respectively.

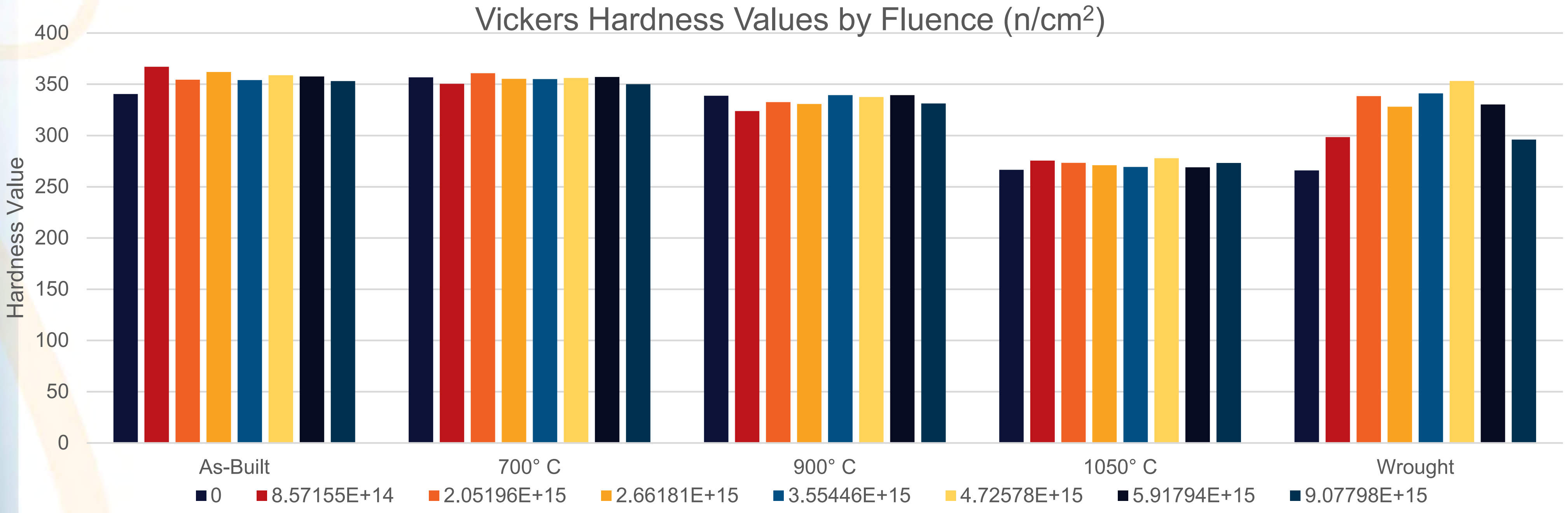


1. Dees
2. Accelerated proton
3. Charge filter
4. Fluorine target
5. Fast neutrons emitted
6. IN625/IN718 samples



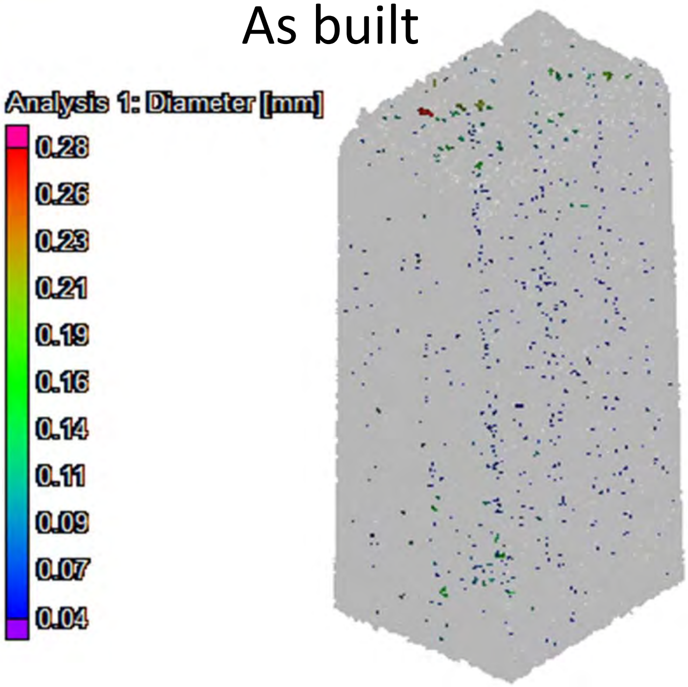
Results: Inconel 625 fast neutron irradiation

- Hardness values of the AM samples did not change significantly while the values for the untreated, conventionally wrought sample, changed noticeably throughout the 25-week experiment.
- The hardness of the wrought sample increased by over 15% and then decreased back to the initial values.

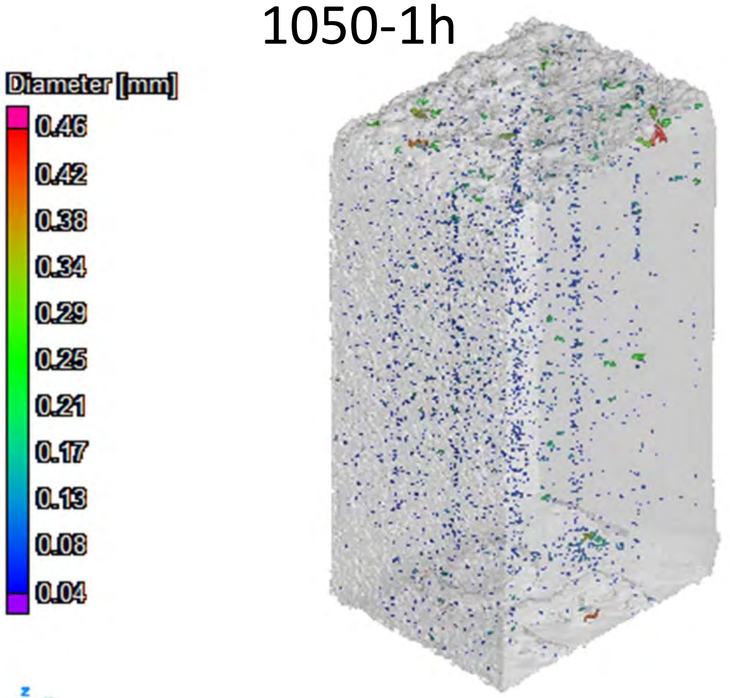


Results: Porosity comparison

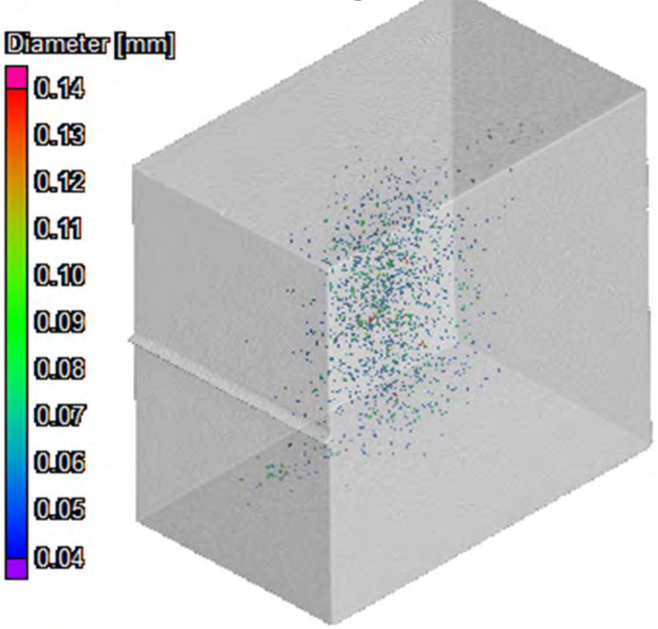
As built



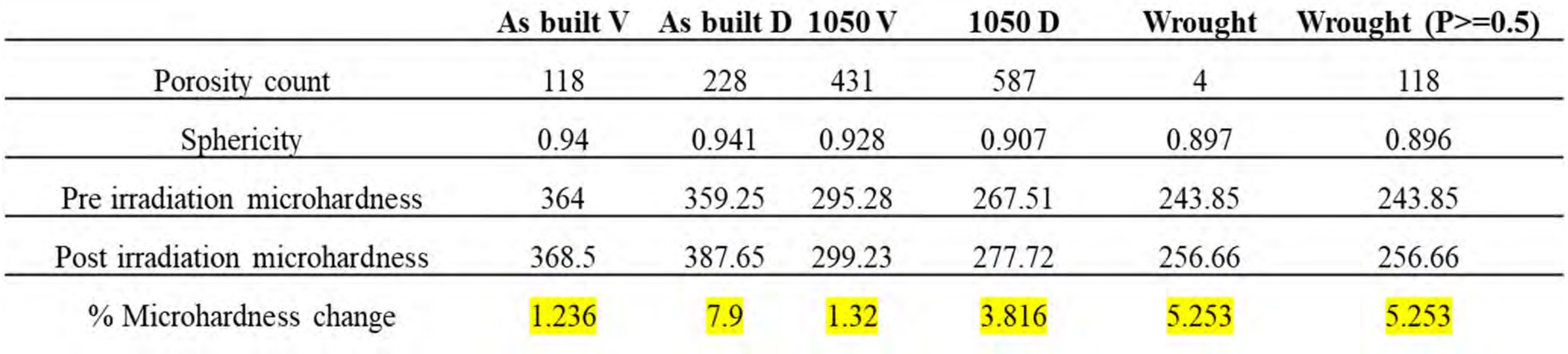
1050-1h



Wrought



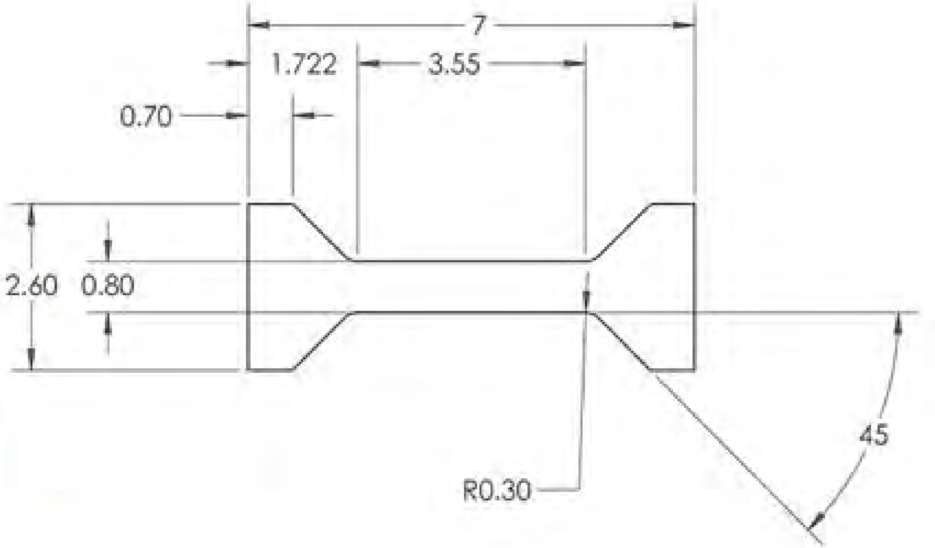
Preliminary Results: Porosity effects on radiation hardening for L-PBF Inconel 625



	As built V	As built D	1050 V	1050 D	Wrought	Wrought (P>=0.5)
Porosity count	118	228	431	587	4	118
Sphericity	0.94	0.941	0.928	0.907	0.897	0.896
Pre irradiation microhardness	364	359.25	295.28	267.51	243.85	243.85
Post irradiation microhardness	368.5	387.65	299.23	277.72	256.66	256.66
% Microhardness change	1.236	7.9	1.32	3.816	5.253	5.253

- Sphericity is used to describe the geometric deviation of a pore from a sphere.
- Sphericity of pores ranged between 0.89-0.94. This indicate pores were gas pores and not lack of fusion pores. LOF is more irregular in shape.
- A correlation between microhardness % change and sphericity/count appears

New Experiments Planned



- SEM/FIB will be in MURR in 1-2 months
- We have a hot cell equipped with a 1.6 kN high-temperature tensile/creep testing (up to 1600ºC)
- Plan to do full spectrum and ion irradiation dosing experiments

Conclusions

- Vertically oriented, as-built, L-PBF sample experienced lower radiation induced hardening (1.2 %) compared to wrought sample (5.253 %) after being exposed to a full neutron spectrum in a 10 MW reactor with an approximate fluence of 7.37 x 1019 neutrons/cm². There was major influence of build orientation in the as-built condition and this could affect how we design AM HeXs.
- Under the same fast neutron fluence, Inconel 718 showed stronger structural integrity towards radiation hardening defect compared to Inconel 625. Different heat treatment temperatures resulted in different levels of radiation hardening or softening in both Inconel 625 and Inconel 718
- Porosity level and sphericity show a correlation with nuclear hardening.
- L-PBF Inconel 625 has complex precipitate phases which may play a role in radiation hardening/softening.
- Cobalt, which is ~0.1% of Inconel 625, was confirmed to increase radioactivity substantially. Cobalt-60, the main radioactive cobalt isotope in the samples, has a half-life of 5.27 years, meaning it takes a long time to decay to a safe level.
- Fast neutron dosing may be a viable accelerated testing method for nuclear damage on metals with less radioactive materials handling. More research into this is being conducted.

Final Work

- All irradiated Inconel 625 and Inconel 718 specimens will undergo SEM to determine microstructural changes due to full-spectrum and fast nuclear dosing.
  - Mizzou has just moved an SEM/FIB system into MURR and it will be ready by May.

Publications

1. O'Donnell, V., Keya, T., Romans, A., Harvill, G., Andurkar, M., Prorok, B.C., Thompson, S.M., Gahl, J., Diagnostic Technique Characterizing Neutron Irradiation Effects on Additively Manufactured Inconel 625 Eliminating the Need for Remote Handling, Nuclear Technology, Vol. 209 (2), pp. 254-260 (2023). DOI: 10.1080/00295450.2022.2120321
2. O'Donnell, V., He, X., Harvill, G., Andurkar, M., Prorok, B.C., Thompson, S.M., Gahl, J., Comparison of neutron radiation response of additively manufactured versus conventionally wrought production methods in Inconel 625, Submitted July 2022.
3. O'Donnell, V., Keya, T., Romans, A., Harvill, G., Andurkar, M., Prorok, B.C., Thompson, S.M., Gahl, J., Novel Method for Fast Neutron Irradiation of Materials via a Positron Emission Tomography Isotope Producing Cyclotron, Submitted June 2022.
4. Andurkar, M., O’Donnell, V., Keya, T., Prorok, B.C., Gahl, J., Thompson, S.M., Thermal and Fast Neutron Irradiation Effects on Additively Manufactured and Wrought Inconel 625, ASME 2023 18th International Manufacturing Science and Engineering Conference (MSEC2023), 12-16 June 2023, New Brunswick, New Jersey.
5. Andurkar, M, Prorok, B.C., Thompson, S.M., Effect of Build Orientation on Residual Stress and Microstructure in Inconel 625 Fabricated via Laser Powder, 33rd International Solid Freeform Fabrication Symposium (SFF), The Minerals, Metals & Materials Society: TMS, University of Texas at Austin, pp. 1800-1810, 25-27 August, Austin, Texas, USA (2022).
6. Andurkar, M., O’Donnell, V., Keya, T., Prorok, B.C., Gahl, J., Thompson, S.M., Effects of Fast Neutron Irradiation on the Microhardness of Inconel 625 and Inconel 718 Materials Fabricated via Laser Powder Bed Fusion, 33rd International Solid Freeform Fabrication Symposium (SFF), pp. 603-613, 25-27 August, Austin, Texas, USA (2022).
7. O’Donnell, V. Keya, T., Romans, A., Harvill, G., Andurkar, M., Prorok, B.C., Thompson, S.M., Gahl, J., Novel Method of Characterization of Particle Beam Using Nano-Indentation, ANS Winter Meeting and Technology Expo, 14th International Topical Meeting on Nuclear Applications of Accelerators, American Nuclear Society, pp. 110-114, 30 November – 3 December, Washington, District of Columbia, USA (2021).
8. Andurkar, M., O’Donnell, V., Gahl, J., Prorok, B.C., Keya, T., Harvill, G., Thompson, S.M., Effects of Build Orientation and Heat Treatment on Neutron Irradiation Hardening in Inconel 625 Fabricated via Laser Powder Bed Fusion, 32nd International Solid Freeform Fabrication Symposium (SFF), pp. 1026-1035, 2-4 August, Virtual Event (2021). OSTI ID: 1865846.
9. Andurkar, M., Suzuki, T., Prorok, B.C., Gahl, J., Thompson, S.M., Residual Stress Measurements via X-Ray Diffraction Cos-α Method on Various Heat-Treated Inconel 625 Specimens Fabricated via Laser-Powder Bed Fusion, 32nd International Solid Freeform Fabrication Symposium (SFF), pp. 1048-1060, 2-4 August, Virtual Event (2021).
10. Keya, T., O’Donnell, V., Lieben, J., Romans, A., Harvill, G., Andurkar, M., Gahl, J., Thompson, S.M., Prorok, B.C., Effects of Heat Treatment and Fast Neutron Irradiation on the Microstructure and Microhardness of Inconel 625 Fabricated via Laser-Powder Bed Fusion, 32nd International Solid Freeform Fabrication Symposium (SFF), pp. 1036-1047, 2-4 August, Virtual Event (2021).

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- Questions?