Additive Manufacturing (AM) of Steels for Extreme Environments: Opportunities and Challenges

Niyanth Sridharan[1a]; **T.M. Kelsy Green[2]**; Kevin Field[1b]; Pengyuan Xiu[2]; Stephen Taller[2]; Xiang (Frank) Chen[1]; Lizhen Tan[1]; Ying Yang[1]

[1]Oak Ridge National Laboratory

[2]University of Michigan-Ann arbor

[a]Currently at Lincoln Electric, India

[b]Currently at University of Michigan-Ann Arbor

Extreme Nuclear Environments

Advanced Reactor Design Considerations for Structural Materials

- Temp. Range: 300-1,000°C
- Damage Level: >200 dpa
- Stress: ~150 MPa
- Corrosion

Advanced Reactor Radiation Effects

- Radiation hardening and embrittlement
- Phase instabilities
- Irradiation creep
- Swelling
- He embrittlement

Desired Material Features

Fine, stable precipitates

- Good long-term thermal aging
- Resist radiation embrittlement
- Creep resistance
- Resist void swelling
- High strength & good ductility

9-20% Cr Alloy Development for Nuclear Reactor Core Internal and External Components

Ingot Metallurgy

Composition, Heat Treatment, TMT Optimization

9-12% Cr FM Steels

MX: 10^{19}-10^{20} m^{-3}

9-12% Cr CNA Steels

MX: 10^{21}-10^{22} m^{-3}

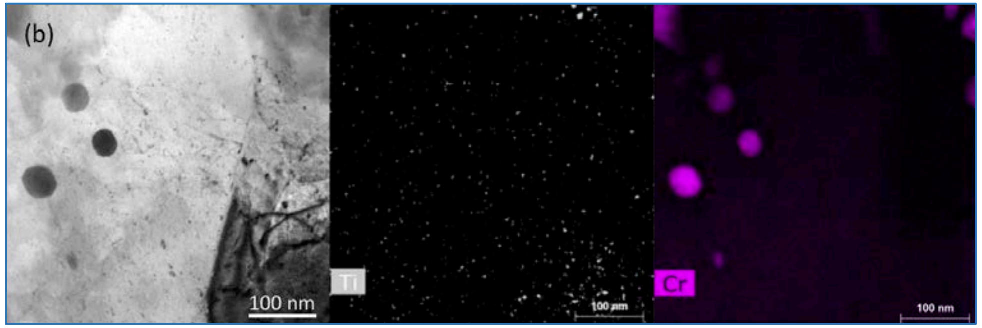
Nanoparticle Optimization

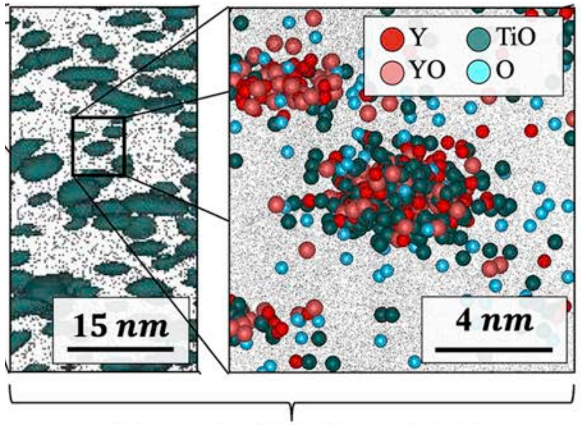
Powder Metallurgy

9-20% Cr ODS FM Steels

Oxides: 10^{21}-10^{24} m^{-3}

How can we efficiently fabricate FM steels with high nanoparticle densities?





Nanoscale (Y,Ti,O) precipitation

T.R. Allen et al. / Journal of Nuclear Materials 375 (2008) 26–37
L. Tan et al. / Journal of Nuclear Materials 509 (2018) 267-275
C.P. Massey et al. / Journal of Nuclear Materials 522 (2019) 111-122

FM=Ferritic/Martensitic, RAFM=Reduced Activation Ferritic/Martensitic, ODS=oxide dispersion strengthened

9-20 Cr% Alloy Development for Nuclear Reactor Core Internal and External Components

Ingot Metallurgy

Composition, Heat Treatment, TMT Optimization

- 9-12% Cr FM Steels — MX: 10^{19}-10^{20} m^{-3}
- 9-12% Cr CNA Steels — MX: 10^{21}-10^{22} m^{-3}

Nanoparticle Optimization

Powder Metallurgy

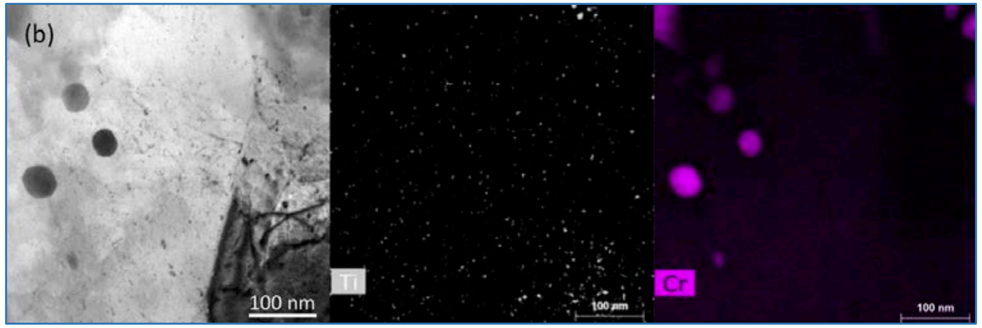
- 9-20% Cr ODS FM Steels — Oxides: 10^{21}-10^{24} m^{-3}

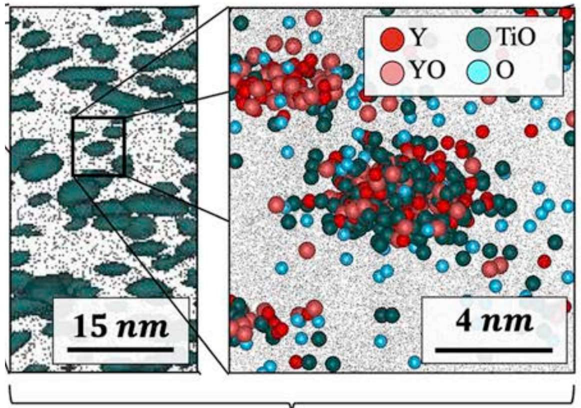
Fabrication Optimization

Additive Manufacturing

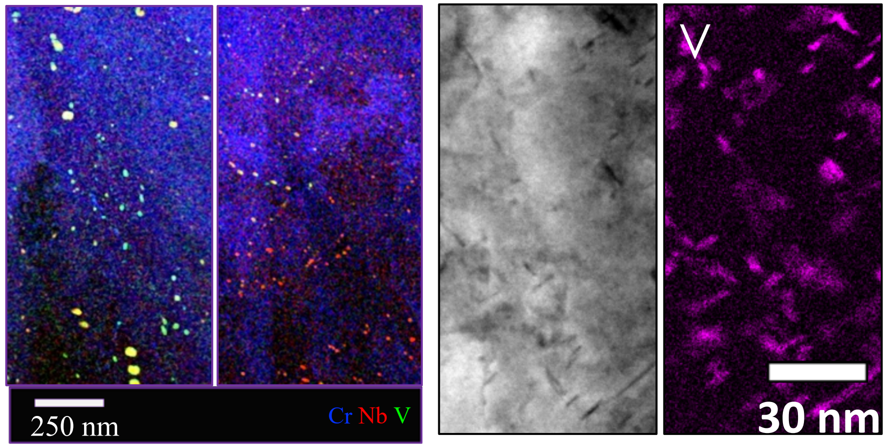
Directed Energy Deposition 9-12% Cr FM Steels

- Wire arc — MX: 10^{21} m^{-3}
- Laser blown powder — MX: 10^{23} m^{-3}





Nanoscale (Y,Ti,O) precipitation



T.R. Allen et al. / Journal of Nuclear Materials 375 (2008) 26–37
L. Tan et al. / Journal of Nuclear Materials 509 (2018) 267-275
C.P. Massey et al. / Journal of Nuclear Materials 522 (2019) 111-122

FM=Ferritic/Martensitic, RAFM=Reduced Activation Ferritic/Martensitic, ODS=oxide dispersion strengthened

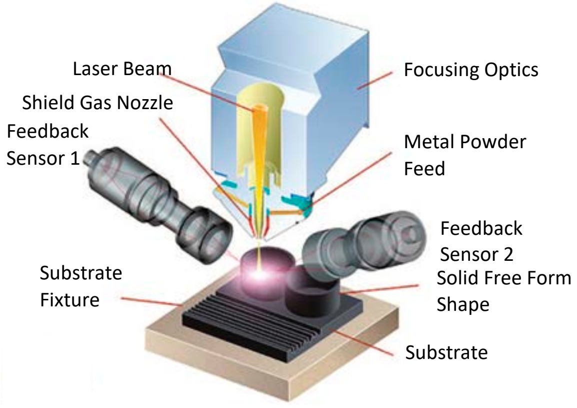
Multiple additive manufacturing techniques can be used to fabricate different reactor components

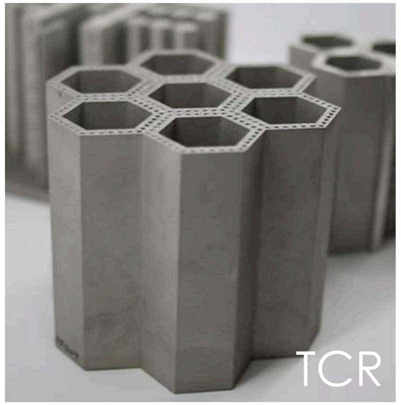
Core Internals

Complex geometries

Control Rods and cooling channels

Laser Blown Powder Additive manufacturing





Core Externals

Larger, less complex geometries

Reactor pressure vessel

Wire Arc Additive manufacturing (WAAM)

Fig. 16 – Shape welded 58 metric ton pressure vessel with dome

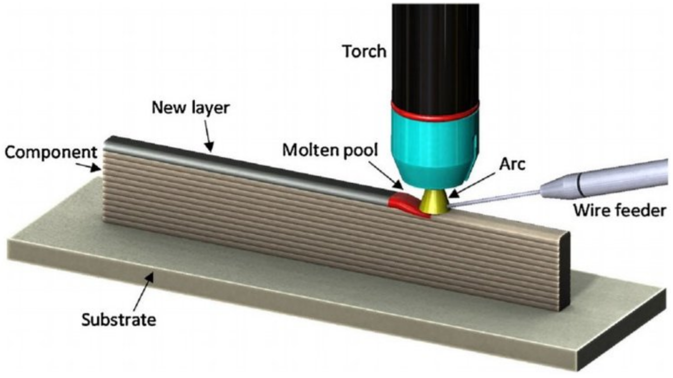


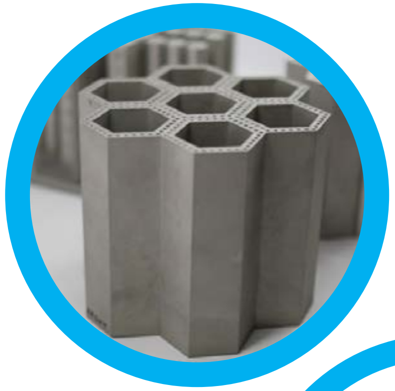
Image adapted from: Dutta et al., AMMTIAC Vol. 6, No. 2

K. Kussmaul et al., "High-Quality Large Components Shape Welded by a SAW Process," American Welding Society, vol. 62, no. 9, pp. 17-24, 1983.

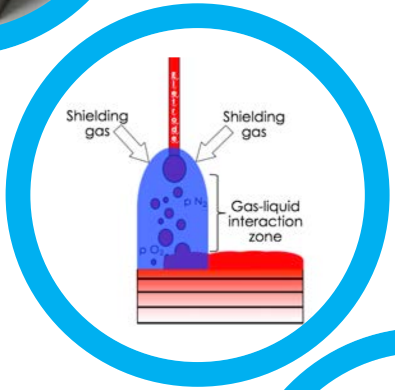
Additive Manufacturing:

Opportunities

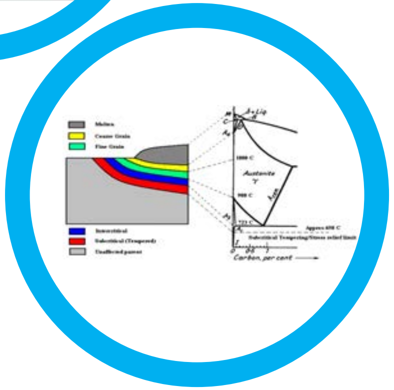
Fabricate near net shape parts



Tailor structure of materials via control of processing parameters



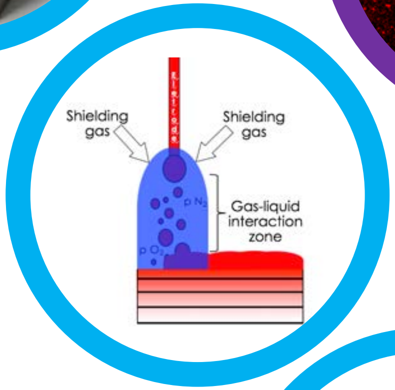
Optimize alloy composition to control solidification and thermodynamic phase pathways



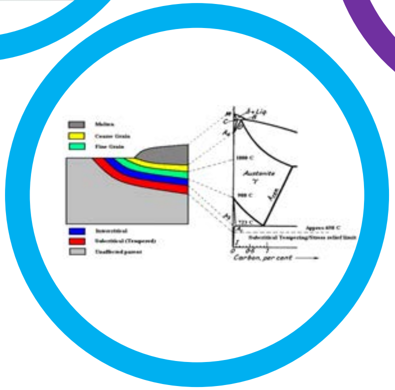
Additive Manufacturing: Challenges

Opportunities

Fabricate near net shape parts

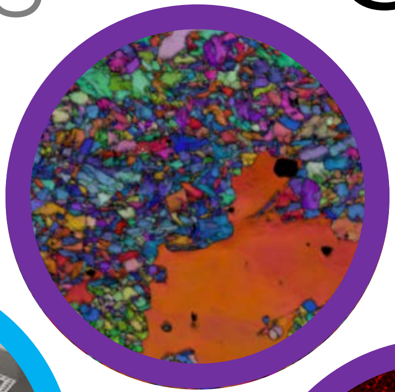


Tailor structure of materials via control of processing parameters

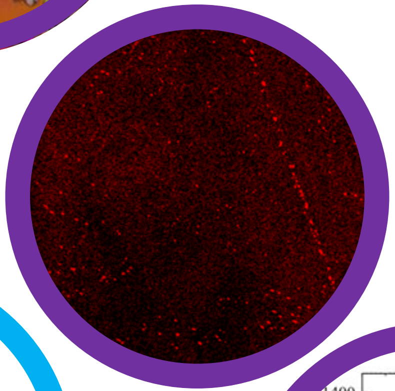


Optimize alloy composition to control solidification and thermodynamic phase pathways

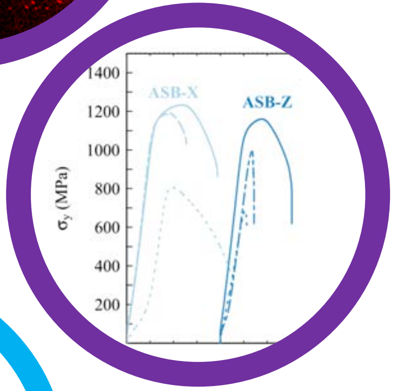
Challenges



Complex solidification processes and residual stress

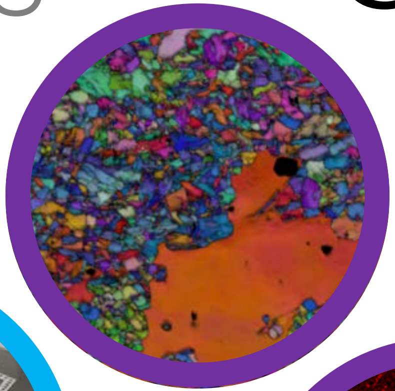


Spatial heterogeneities in microstructure



Varied mechanical properties

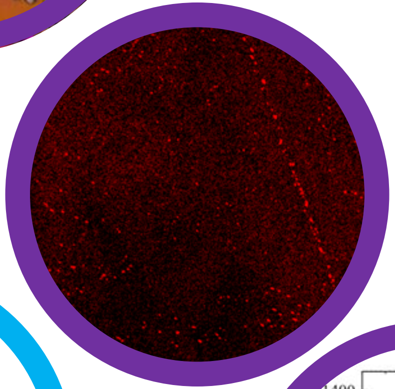
Additive Manufacturing: Challenges



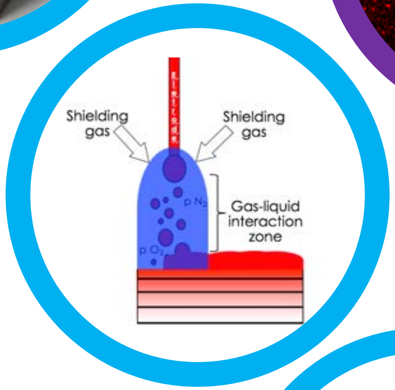
Complex solidification processes and residual stress

Opportunities

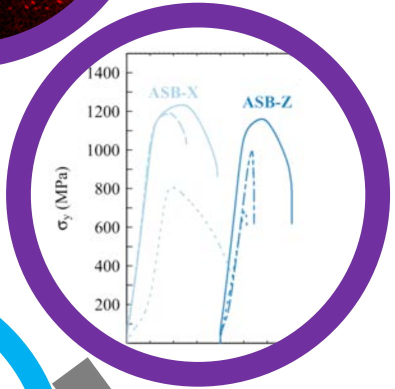
Fabricate near net shape parts



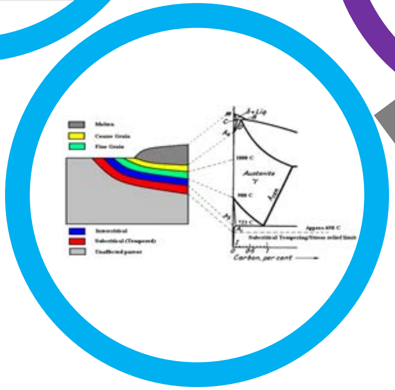
Spatial heterogeneities in microstructure



Tailor structure of materials via control of processing parameters



Varied mechanical properties



Optimize alloy composition to control solidification and thermodynamic phase pathways

Develop framework that maximizes advantages while minimizes disadvantages for FM steel fabrication with AM

Synergistic Approach Applied to Experiments

Materials Design	Materials Processing	Post Processing	Microstructure Evaluation	Mechanical Properties
δ-ferrite reduction	Power Density	δ-ferrite reduction	Prior Austenite Grain Size	Yield & Ultimate Tensile Strength
Residual Stress Reduction	Travel Speed	Residual Stress Reduction	Martensite lath and packet size	Tensile Elongation
Microstructure Homogenization	Scan Strategy	Microstructure Homogenization	Precipitate size distribution	Toughness
	Hatch Spacing		δ-ferrite content	
			Sink Strength	

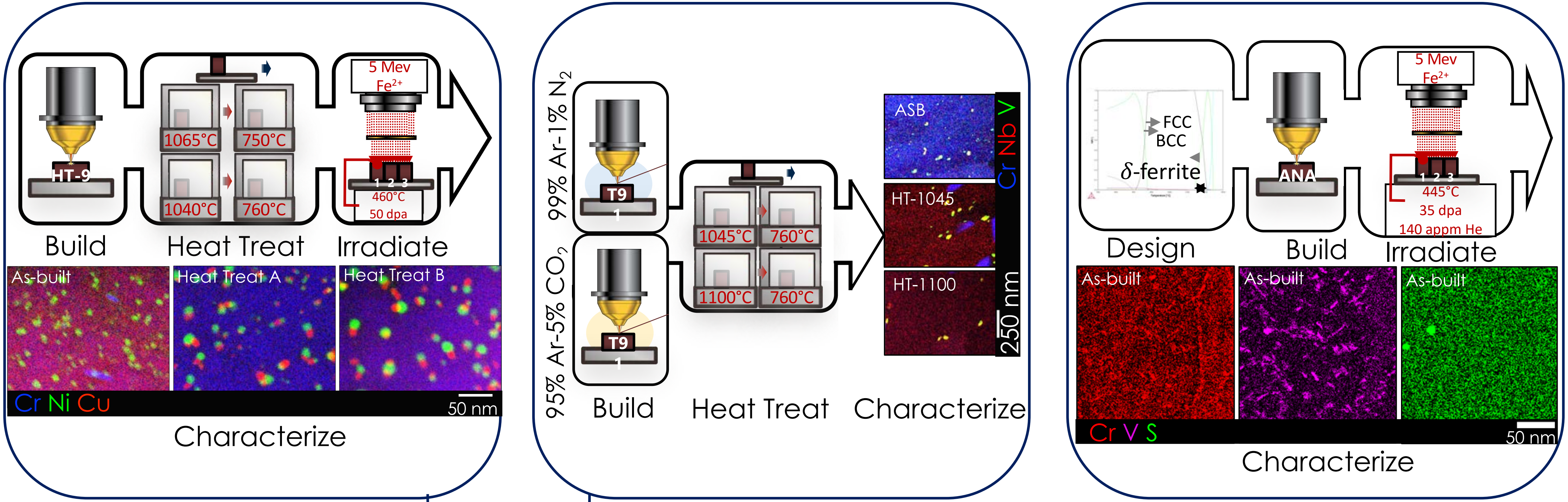
Core internals

Experiment 1:
Laser Powder Blown HT9

Core externals

Experiment 2:
Wire Arc Grade 91

Experiment 3:
Laser Powder Blown Additive Nanostructured Alloy



Assess AM techniques for FM fabrication

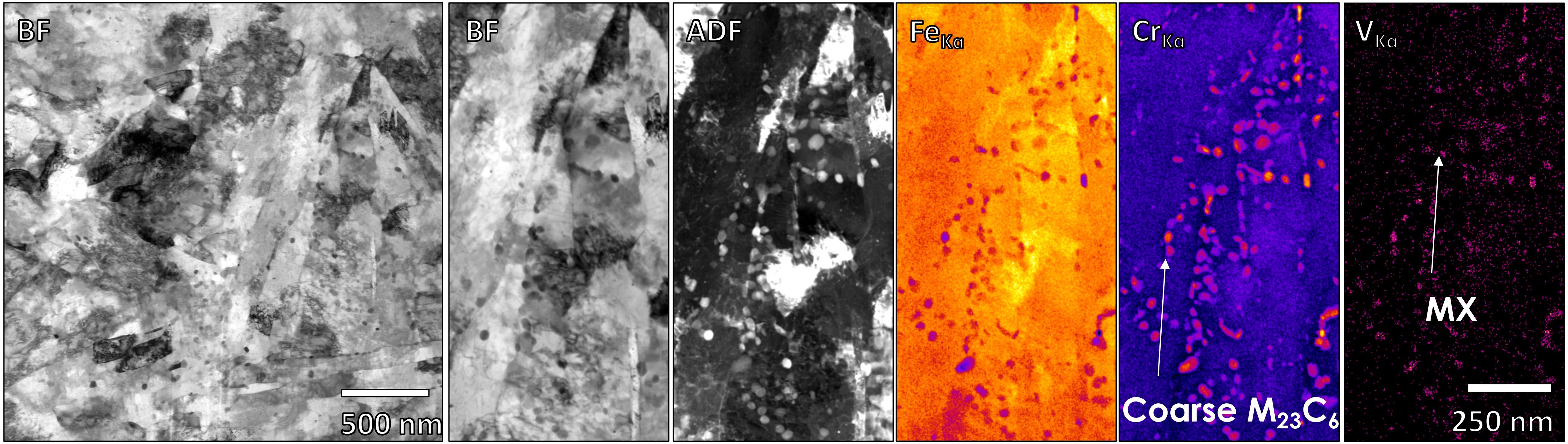
Mimic or improve upon wrought properties

Develop new alloys, monitoring techniques, & certification procedures

Laser Powder Blown HT9: ASB Microstructure

Materials Design	Materials Processing	Post Processing	**Microstructure Evaluation**	Mechanical Properties

As-Built



STEM analysis shows that the as-fabricated structure has:

fine lath size of 250 nm

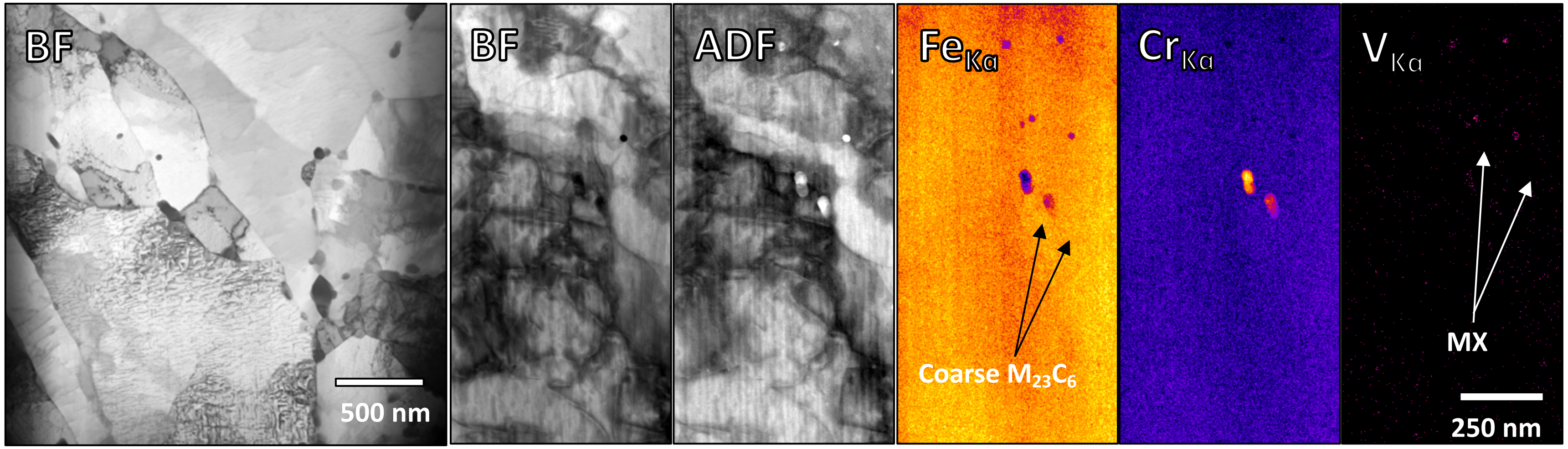
fine PAG size of 3 μm

fine precipitate structure

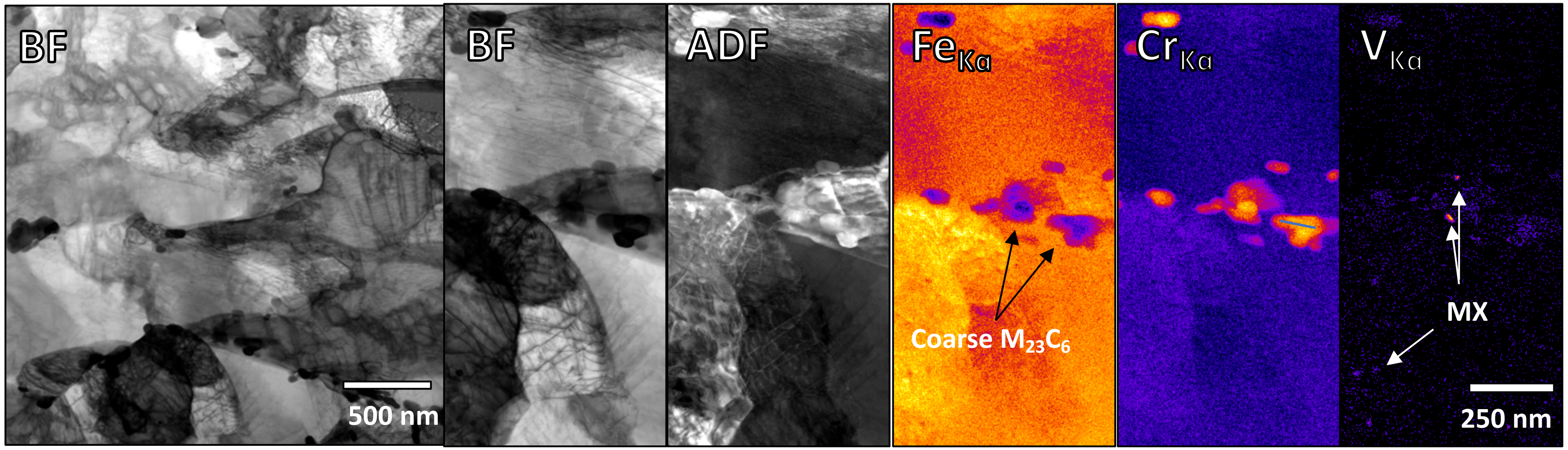
Laser Powder Blown HT9: N&T Microstructure

Materials Design	Materials Processing	**Post Processing**	**Microstructure Evaluation**	Mechanical Properties

N&T 1: 1065˚C/30 min+ 750˚C/1 hr



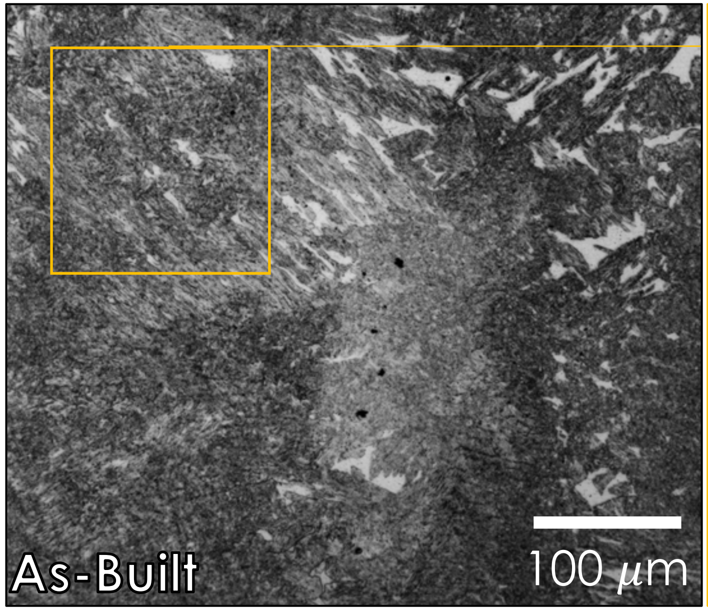
N&T 2: 1040˚C/30 min+ 760˚C/1 hr

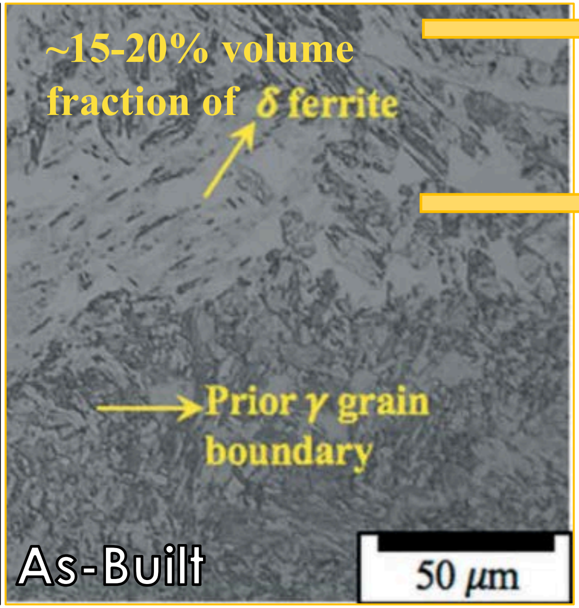


- Coarsening of on grain/lath boundary carbides after heat treatment compared to as-built microstructure
- Agglomeration of MX (assumed VN) precipitates with $M_{23}C_6$ precipitates, although some are still intra-lath

Laser Powder Blown HT9: δ-Ferrite Formation

Materials Design | **Materials Processing** | **Post Processing** | **Microstructure Evaluation** | Mechanical Properties



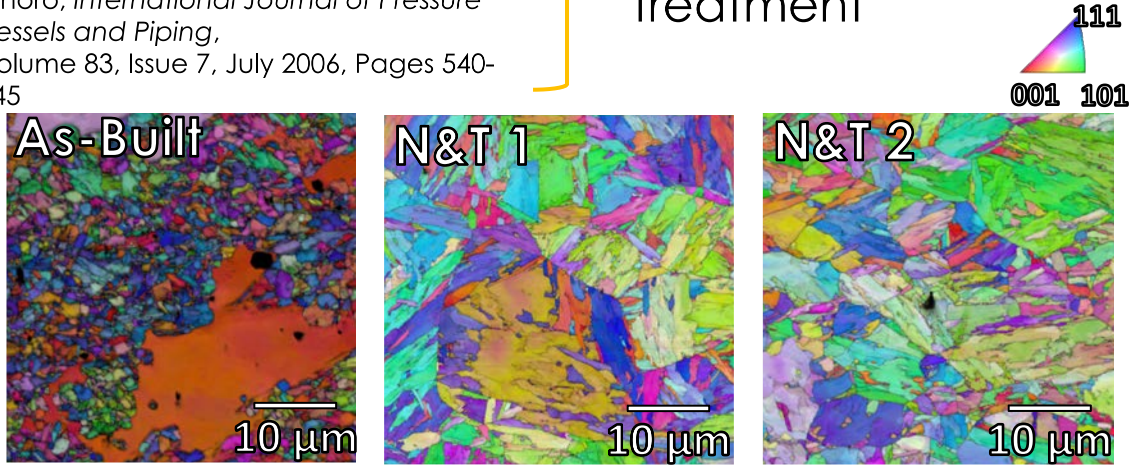


Higher than values found in welding literature

Decreases toughness

Oñoro, *International Journal of Pressure Vessels and Piping*, Volume 83, Issue 7, July 2006, Pages 540-545

No δ-ferrite found after heat treatment

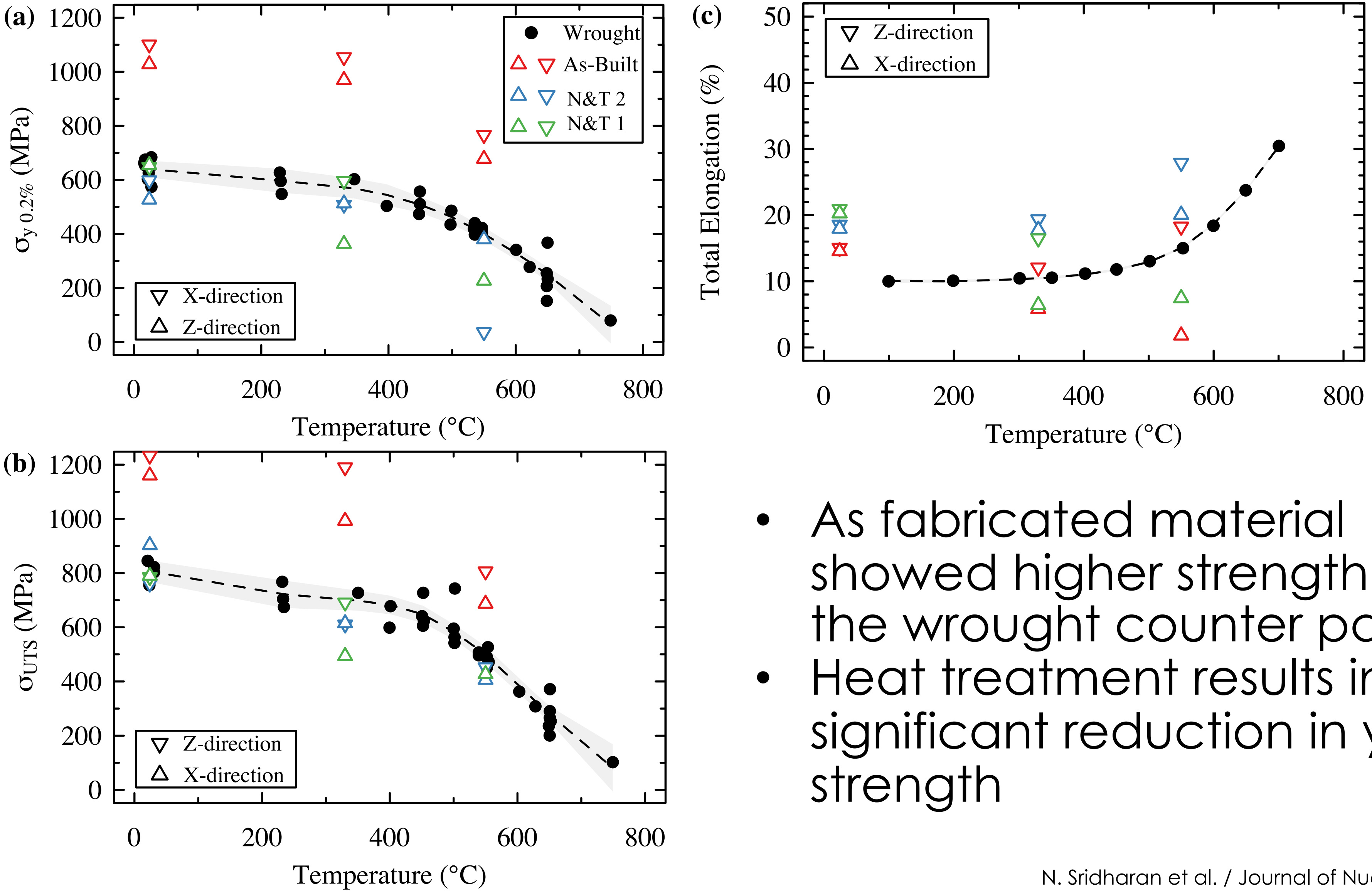


The formation of δ-ferrite in laser blown powder AM is analogous to welding literature:

- Forms due to either 1) high cooling rates during fabrication that lead to incomplete phase transformations or <u>2) during reheating of previously deposited layers</u>
- Can reduce δ-ferrite through optimization of the Cr_{Eq} & Ni_{Eq} by controlling Cr, Mo, W, C, and N contents → decrease ferrite formers elements as much as possible

Laser Powder Blown HT9: Mechanical Properties

Materials Design	**Materials Processing**	**Post Processing**	Microstructure Evaluation	**Mechanical Properties**

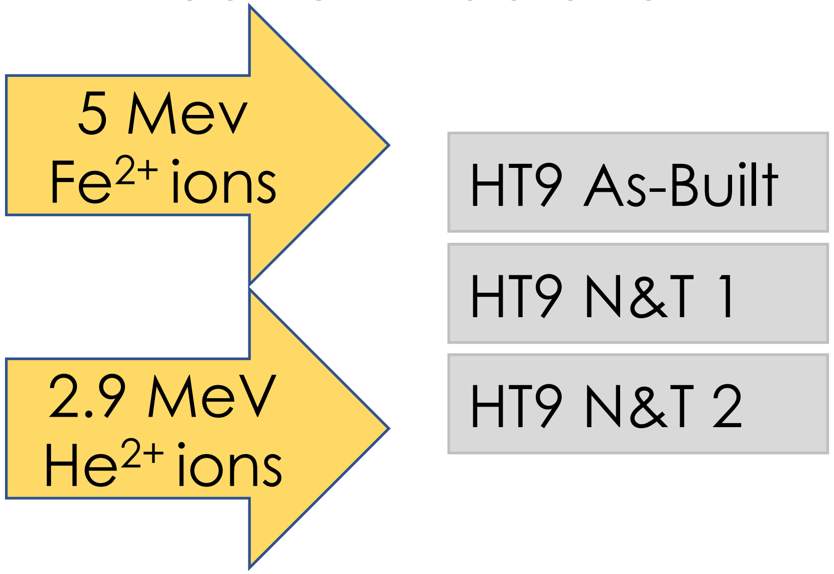


- As fabricated material showed higher strength than the wrought counter parts
- Heat treatment results in significant reduction in yield strength

N. Sridharan et al. / Journal of Nuclear Materials 521 (2019) 45-55

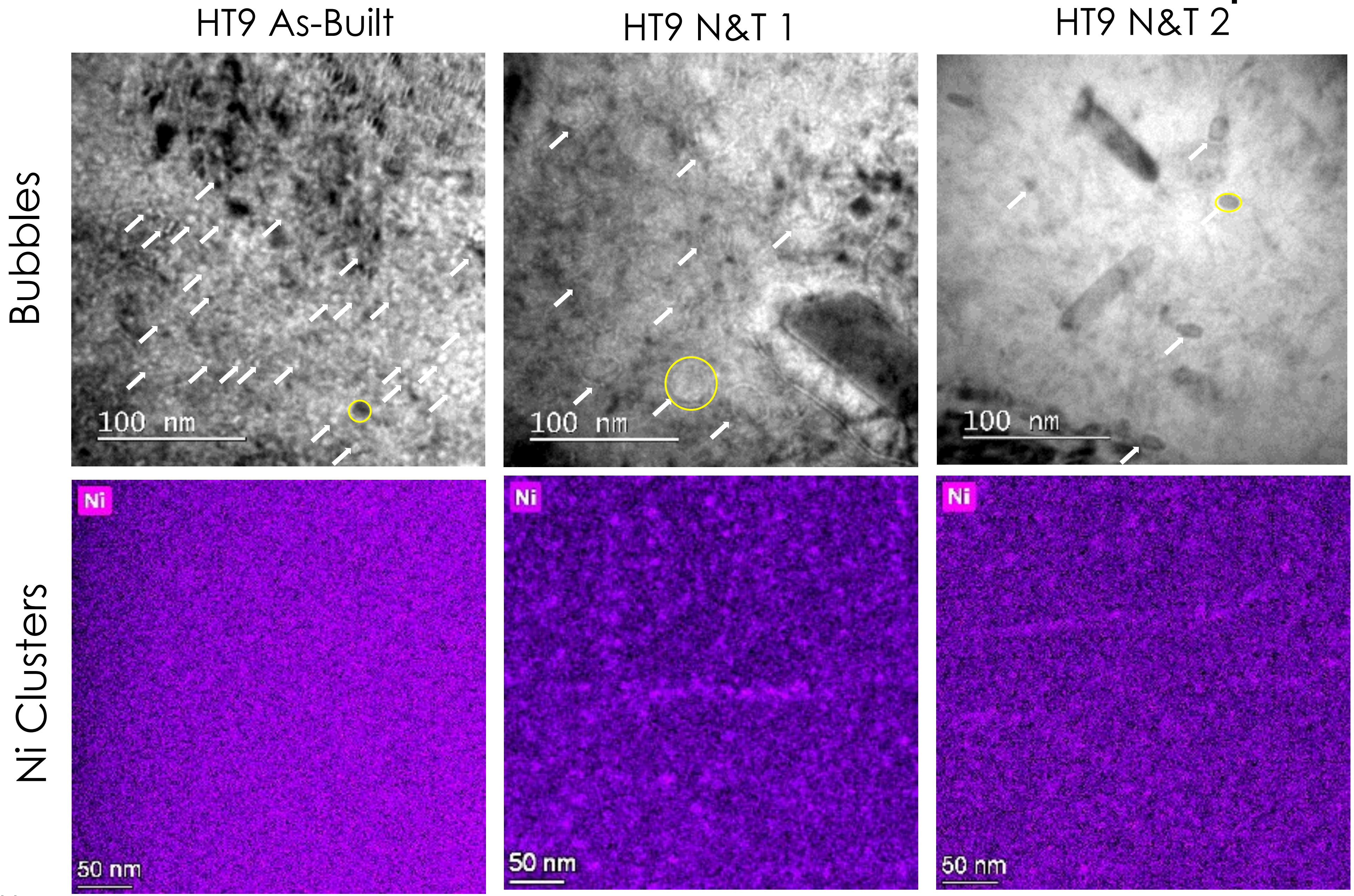
Laser Powder Blown HT9: Radiation Tolerance Assessment

Michigan Ion Beam Laboratory
Dual Ion Irradiation



Dose rate: 10^{-4} dpa/s
Total dose: 16.6 dpa
He appm/dpa: 4
Irradiation Temperature: 445°C

Laser Powder Blown HT9: Radiation Response

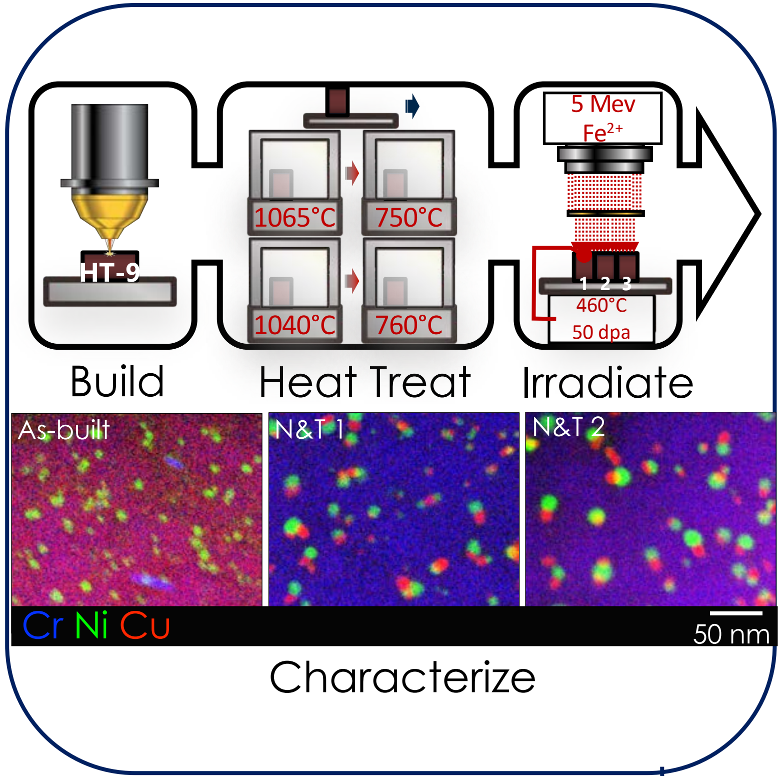


Courtesy of Pengyuan Xiu

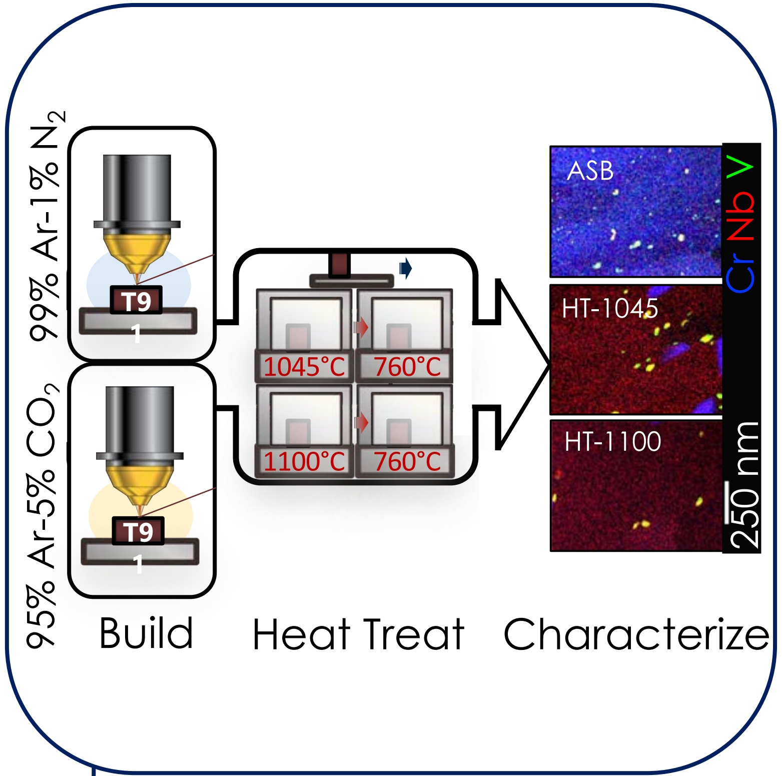
Core internals

Core externals

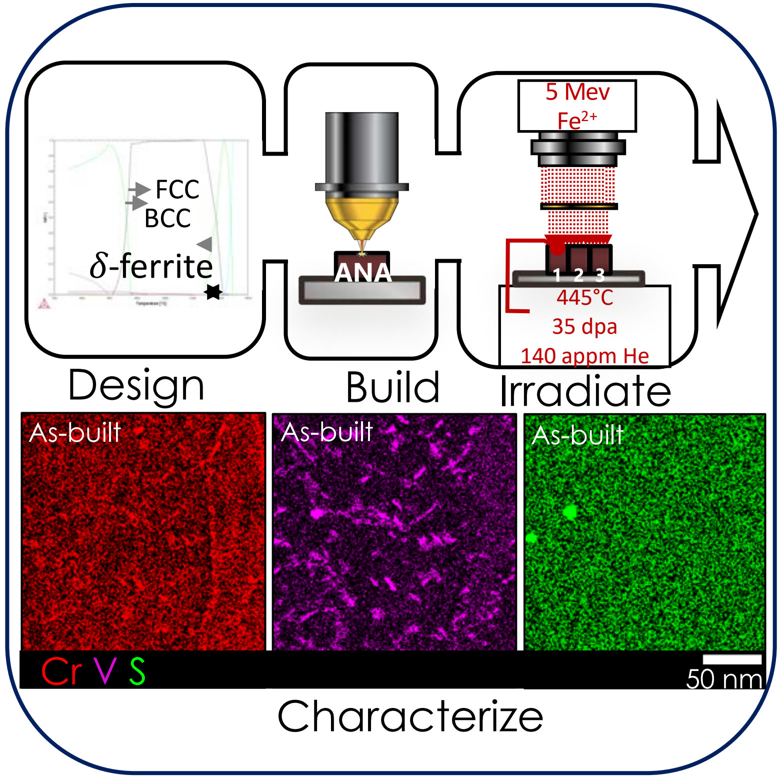
Experiment 1:
Laser Powder Blown HT9



Experiment 2:
Wire Arc Grade 91



Experiment 3:
Laser Powder Blown Additive
Nanostructured Alloy



Assess AM techniques for FM fabrication

Mimic or improve upon wrought properties

Develop new alloys, monitoring techniques, & certification procedures

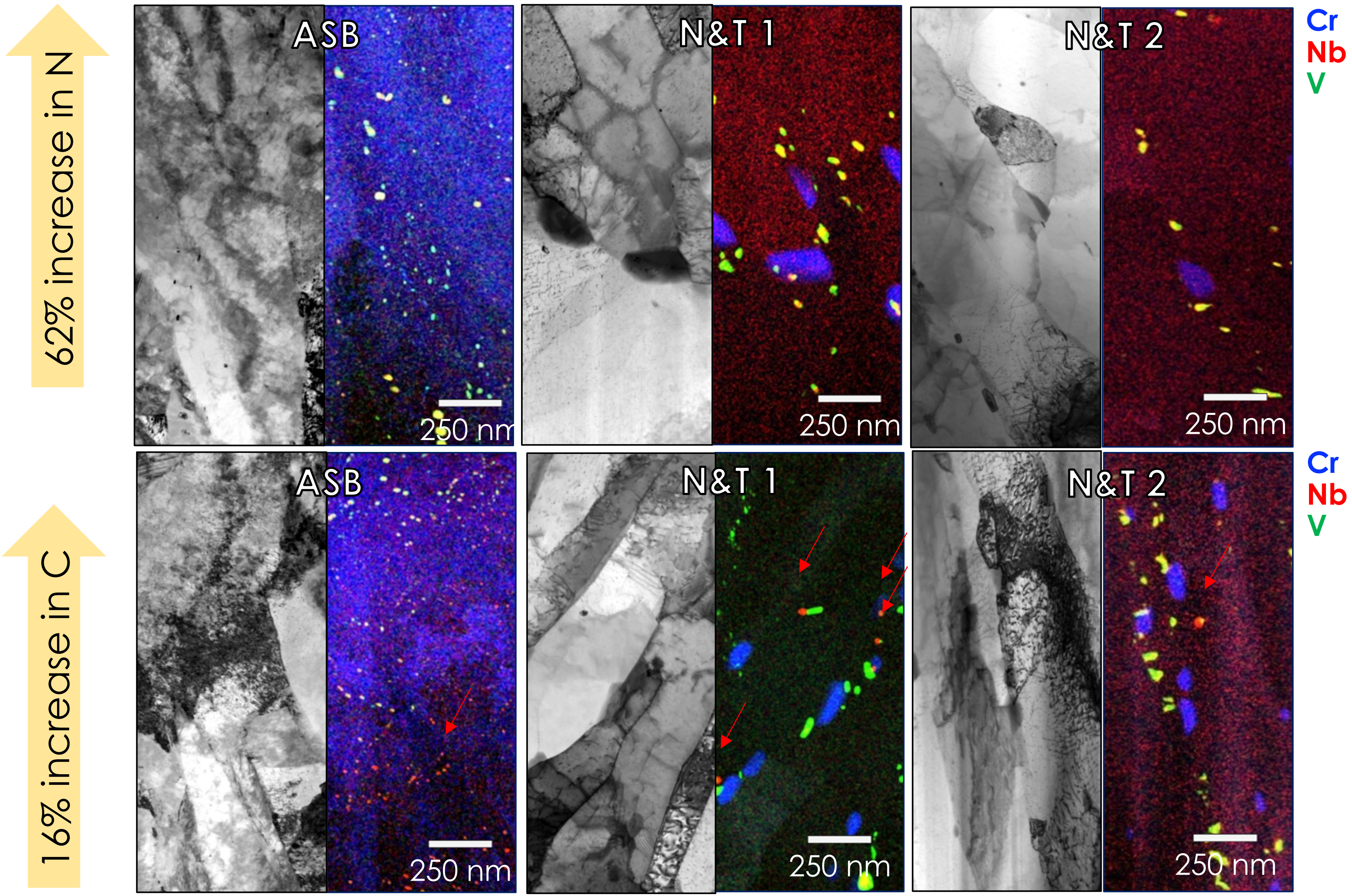
WAAM G. 91

Control MX precipitate structure with C and N additions using different shielding gases

Materials Design	**Materials Processing**	**Post Processing**	**Microstructure Evaluation**	Mechanical Properties

Grade 91 with N addition
99%Ar+1%N_2 shielding gas

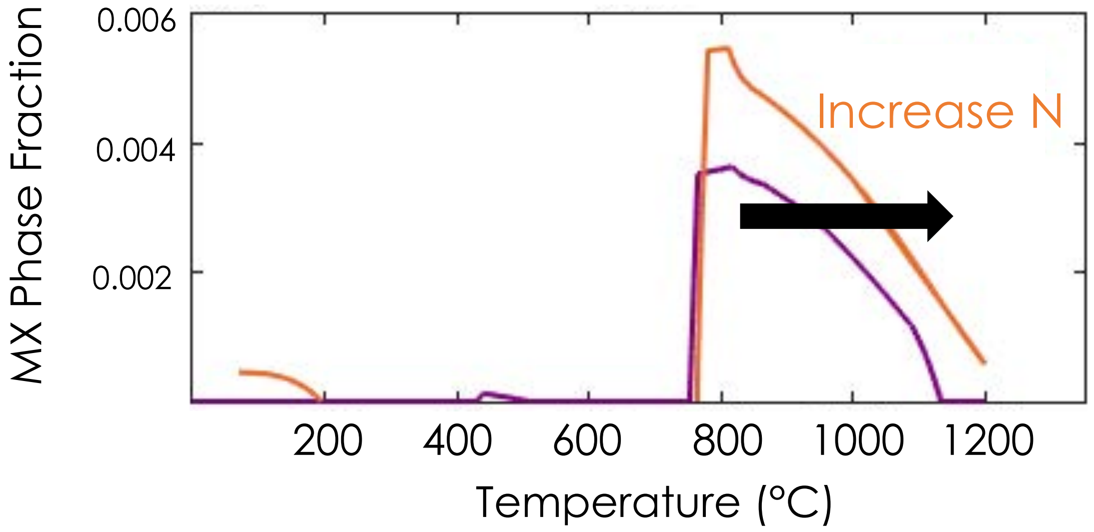
Grade 91 with C addition
95%Ar+5%CO_2 shielding gas



WAAM G. 91

Control MX precipitate structure with C and N additions using different shielding gases

Materials Design	Materials Processing	**Post Processing**	**Microstructure Evaluation**	Mechanical Properties



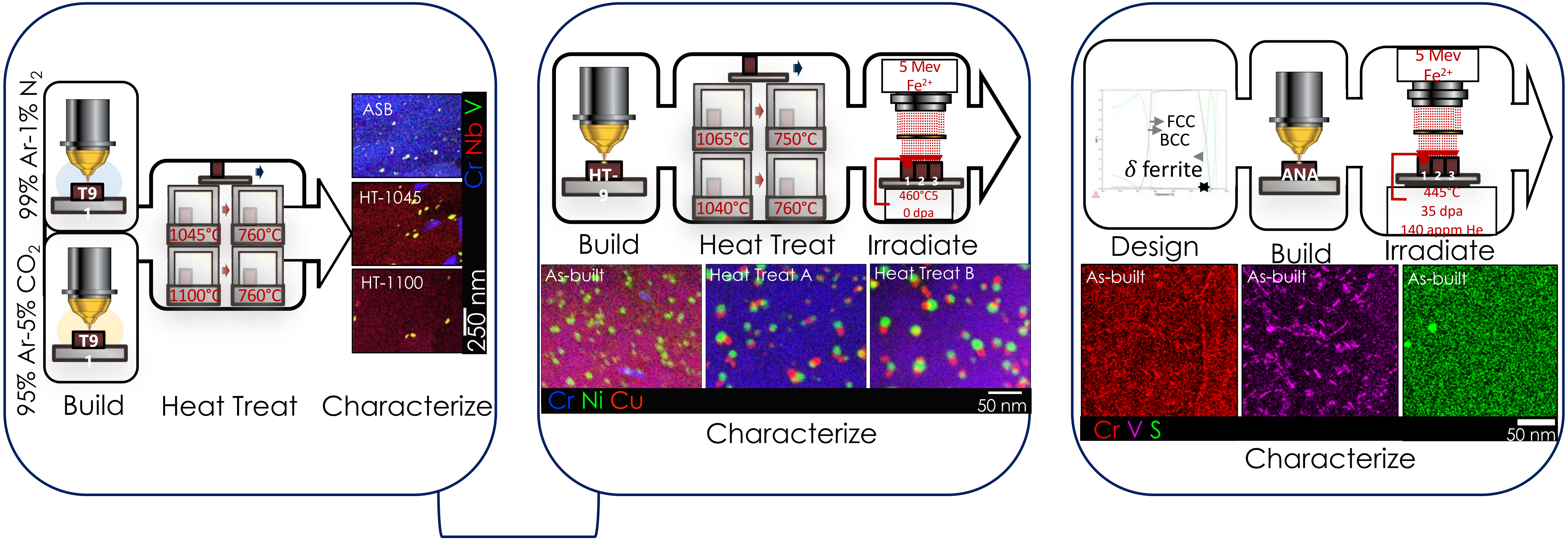
Shielding Gas Used	MX Stability Range
99%Ar+1%N_2	765->1,200°C
95%Ar+5%CO_2	755-1,130°C

- Proved ability to control MX precipitate chemistry and stability with shielding gas composition

Experiment 1:
Wire Arc Grade 91

Experiment 2:
Powder HT9

Experiment 3:
Powder Additive Nanostructured Alloy (ANA)



Results from Grade 91 and HT9 led to an effort to develop new materials, manufacturing processes, and certification strategies for AM

Develop new alloys, monitoring techniques, & certification procedures

Laser Blown Powder ANA

Materials Design	Materials Processing	~~Post Processing~~	Microstructure Evaluation	Mechanical Properties

CALPHAD based design

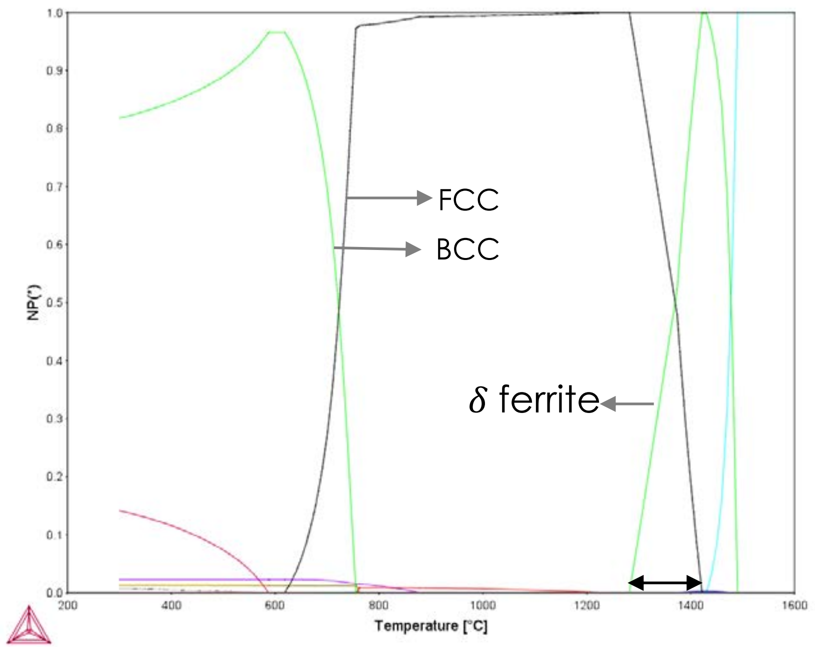
Design composition to control phase and thermodynamic pathways during AM

Wider intercritical temperature →

Increase solvus temperature of MX →

← Decrease stable δ-ferrite window

← Higher A_{c1} temperature



Results courtesy of Ying. Yang, ORNL

Alloy (wt.%)	**Fe**	**Cr**	**Mn**	**Si**	**Ta**	**V**	**C**	**Mo**	**N**
ANA powder	Bal.	8.5-9.5	2.5-3.5	0.4	0.1	0.3-0.6	0.08-0.15	0.3-0.6	0.1

Laser Blown Powder ANA

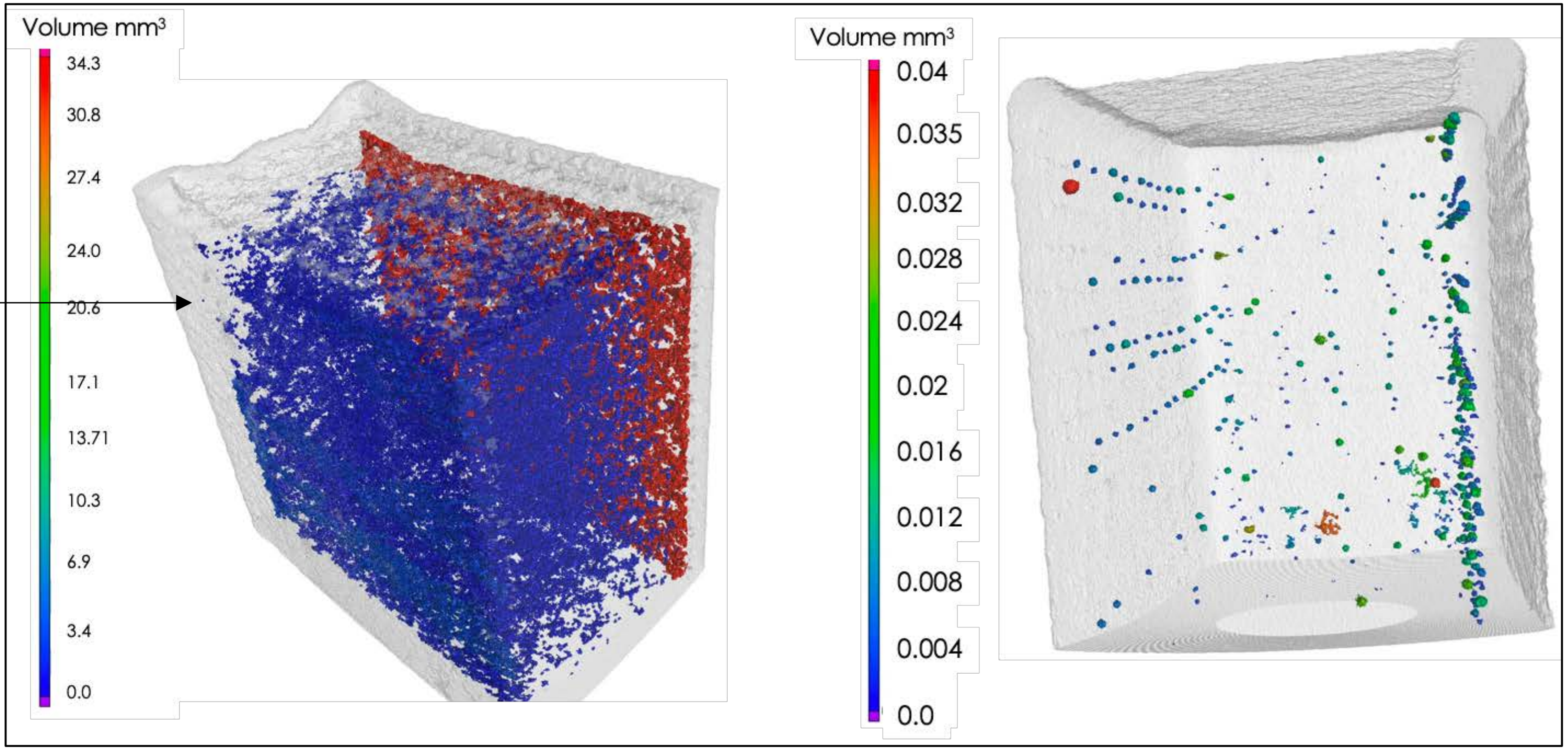
Materials Design	**Materials Processing**	Microstructure Evaluation	Mechanical Properties

Ex-situ CT measurements

Low energy density

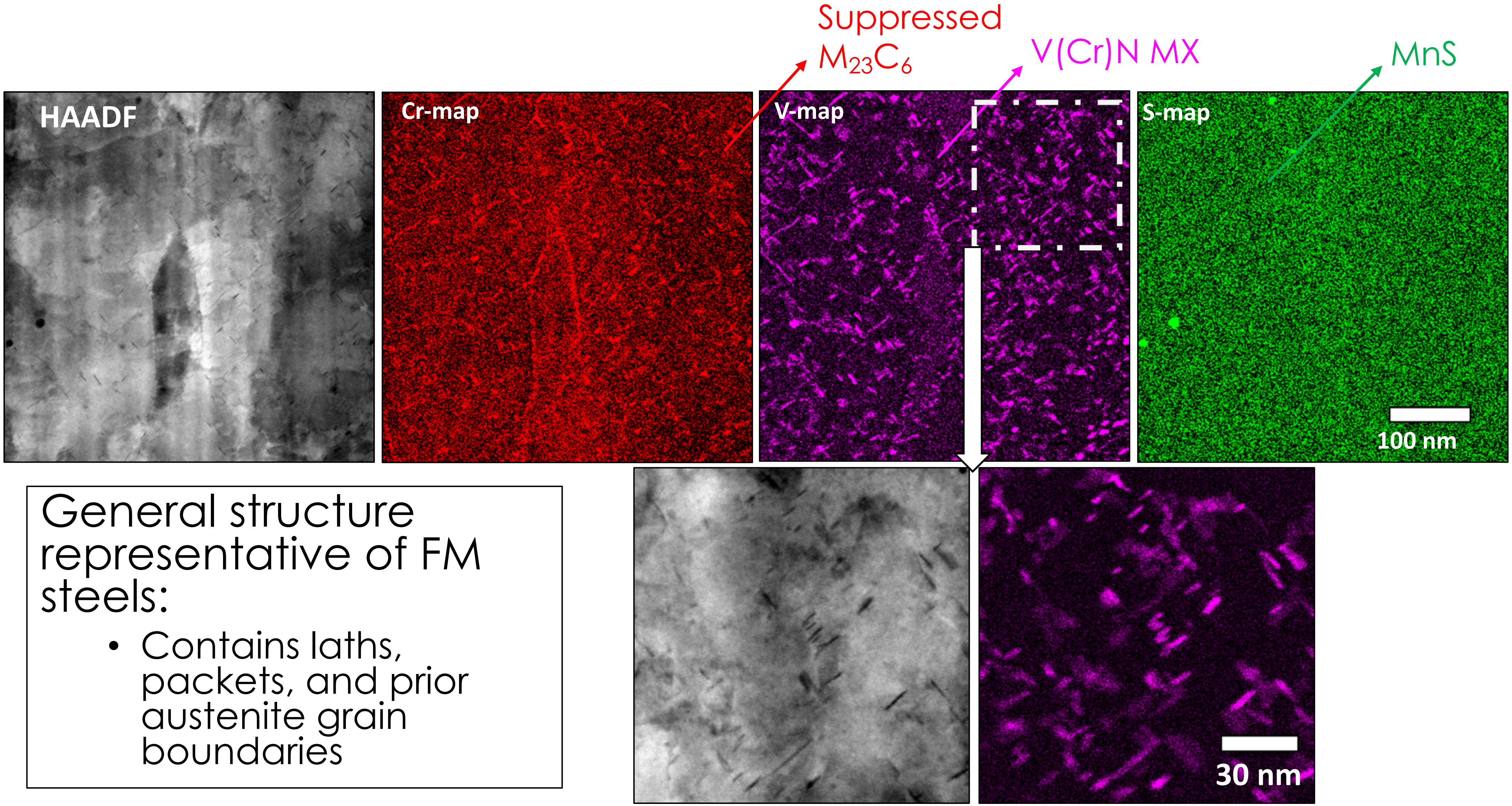
High energy density

Extensive porosity and interlayer lack of fusion observed in the builds fabricated with low energy density primarily due to the insufficient melting



Laser Blown Powder ANA

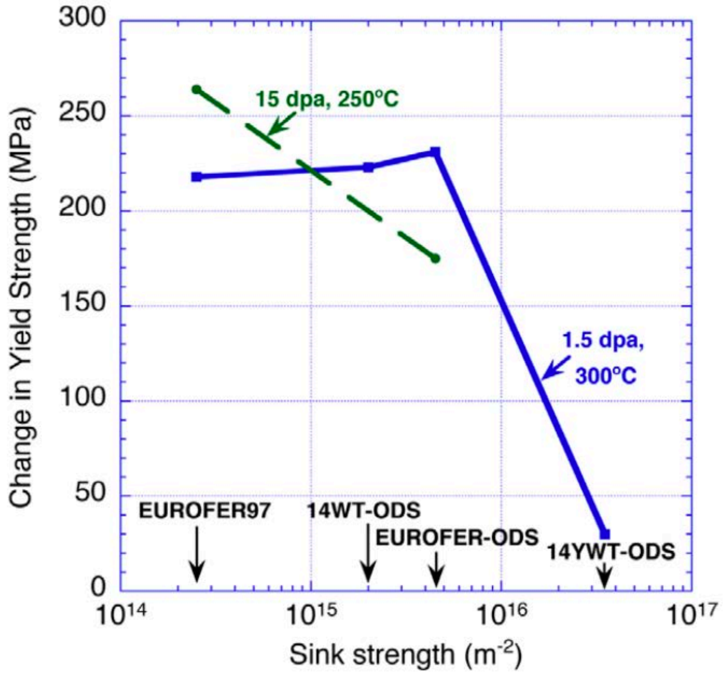
Materials Design	Materials Processing	Post Processing	**Microstructure Evaluation**	Mechanical Properties

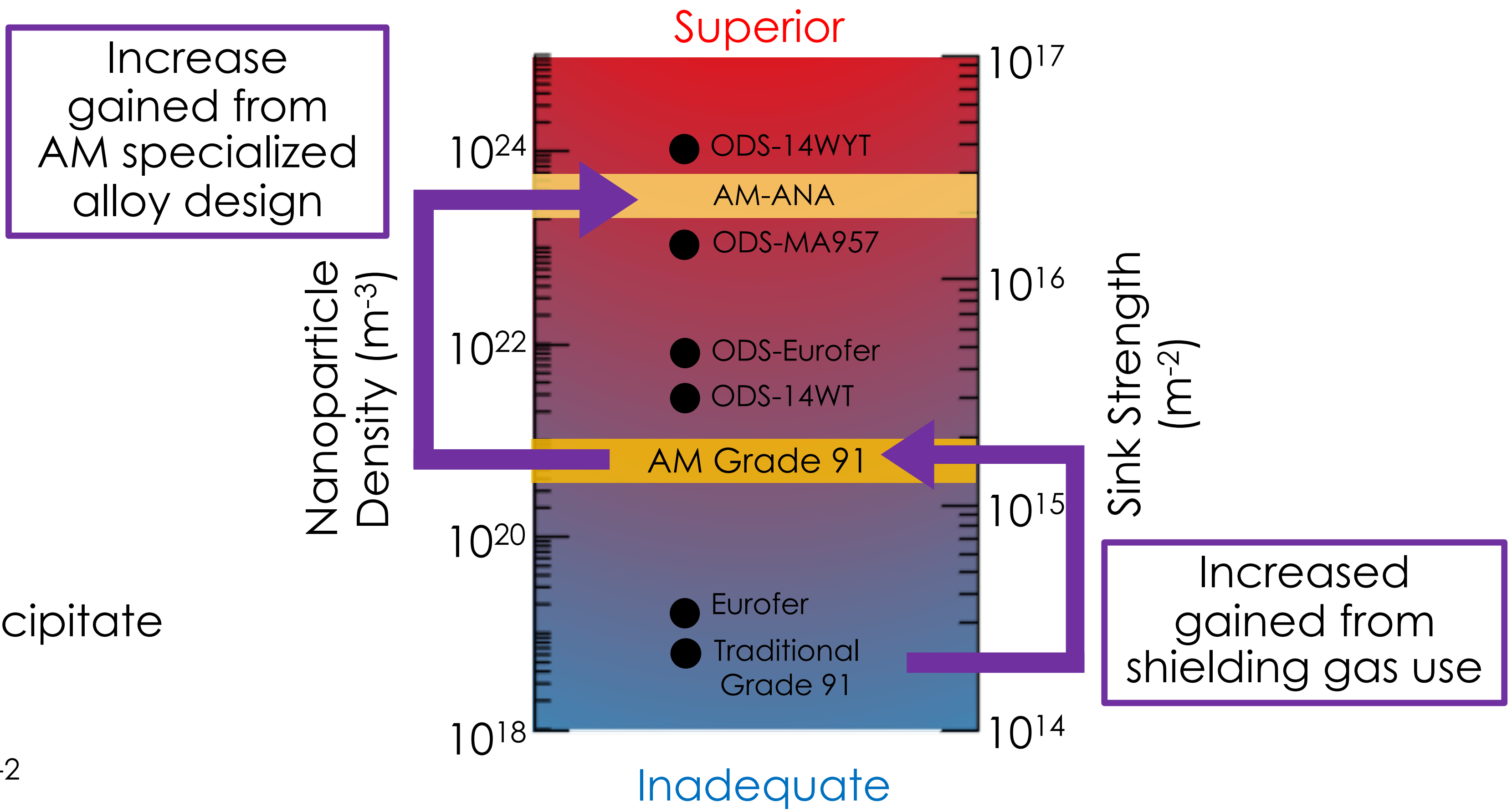


General structure representative of FM steels:

- Contains laths, packets, and prior austenite grain boundaries

Laser Powder Blown ANA: Sink Strength (Preliminary Radiation Tolerance Assessment)





- Sink strength is proportional to precipitate number density and
- precipitate size
- Critical sink strength value: 10^{16} m^{-2}

Zinkle, Nucl. Fusion 53 (2013) 104024

AM allows for rapid new FM alloy design

Summary

Successfully fabricated FM steels with DED-AM techniques
Improved upon wrought G. 91 and HT9 microstructure and mechanical properties

Developed a new FM alloy with an ultra high density of defect sinks
Utilized computational thermodynamics and AM processing

Created novel AM monitoring techniques and certification procedures

Developing rapid assessment of radiation tolerance of AM features

Conclusion

AM allows for rapid new alloy design with desired features and rapid testing

Kelsy Green:
tmkgreen@umich.edu

Pengyuan Xiu:
xiupy@umich.edu

Niyanth Sridharan:
Niyanth_Sridharan@LincolnElectric.in

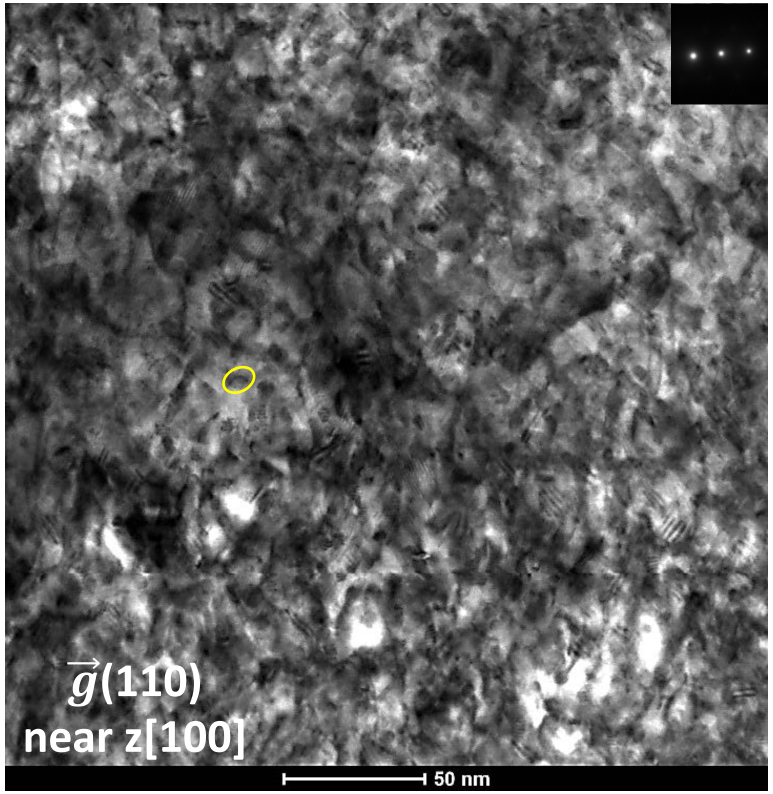
Kevin Field:
kgfield@umich.edu

Roadmap Going Forward

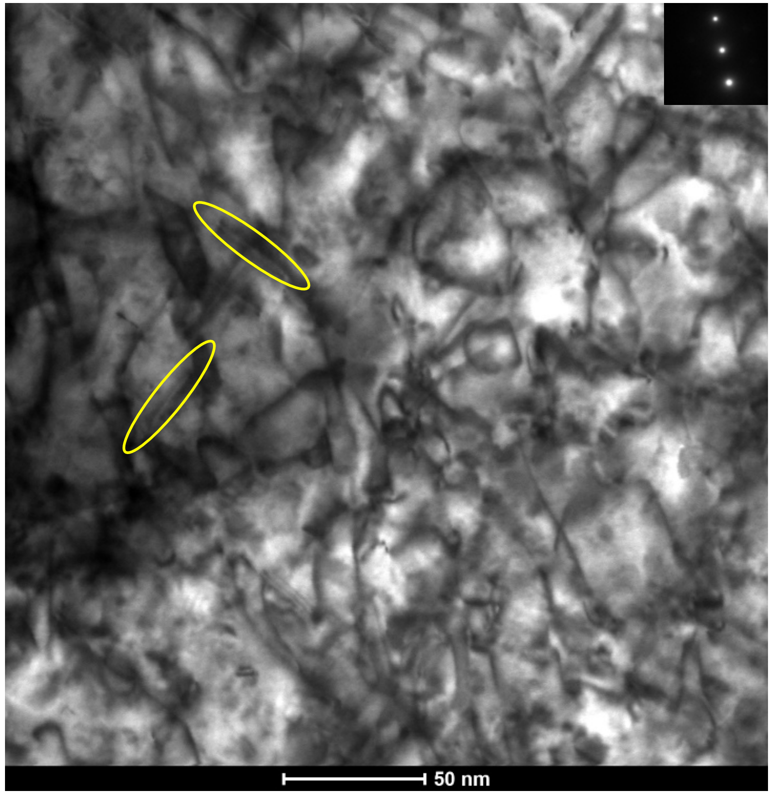
	Computational Materials Design	Monitoring Techniques	Performance Evaluation	Radiation Response
Activity	• Computational thermodynamics • AM process modeling (computational kinetics)	• In-situ monitoring of melt pool and layer development • Data collection and analysis • Ex-situ build integrity measurements	• Microstructure characterization • Mechanical properties determination	• In-situ and ex-situ bulk irradiation to rapidly assess feasibility of new alloys in radiation environments
Outcome	New materials	New qualification standards and manufacturing processes	Processing-properties correlation	Processing-radiation tolerance correlation

Laser Powder Blown HT9: Radiation Response

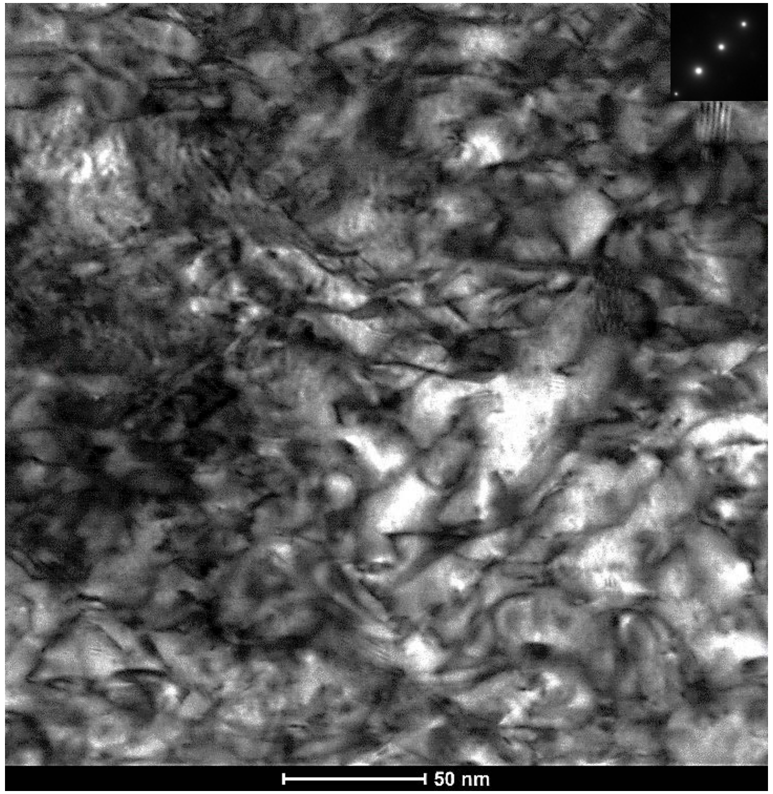
HT9 As-Built



HT9 N&T 1



HT9 N&T 2



HT9 as-built specimen has smaller microstructure features such as bubbles, Ni clusters, and dislocation loops

Courtesy of Pengyuan Xiu

Design criteria for alloys for AM: Improve toughness, creep strength, sink strength and control residual stress

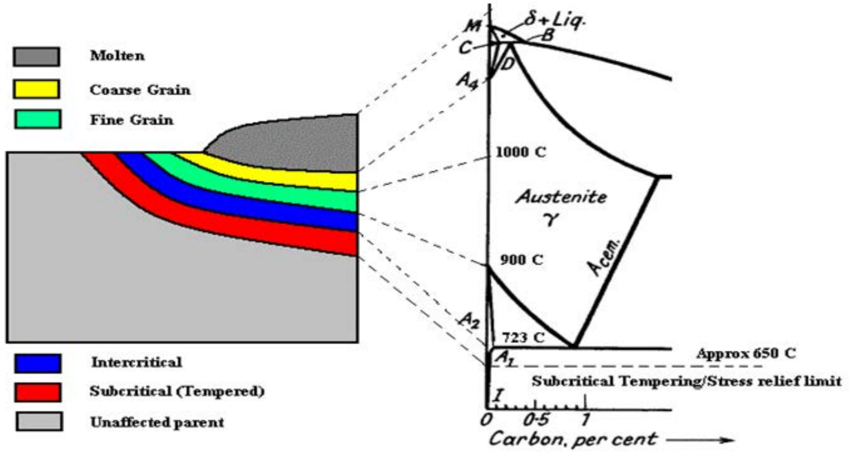
Improve toughness in the as fabricated condition

- *Welding literature reports that we can reduce δ ferrite by optimizing the Cr_{Eq} & Ni_{Eq}*

Cr_{Eq}=Cr+1.5Si+Mo+5V+0.5Nb+075W-Ni-0.5Mn-30C-30N-0.3Cu-Co

- *Control and Cr, Mo, W, C and N*

Increase sink strength and creep strength by increasing fraction of precipitates



Controlling residual stress

- *Alberry and Jones used the concept of L filler materials to develop materials to promote compressive residual stresses welds*