Producing Next Generation Superalloys Through Advanced Characterization and Manufacturing Techniques

Dr. Tim Smith

NASA Glenn Research Center

Outline

- Part 1: Enhancing the strength of Ni-base Disk Superalloys
  - Turbine Engines
  - Creep Deformation
  - Scanning Transmission Electron Microscopy
  - Phase Transformation Strengthening
- Part 2: Efficient Production of a Dispersion Strengthened Multi-Principal Element Alloy (MPEA)
  - Additive Manufacturing
  - Solid Solution Strengthening
  - Dispersion Strengthening

Part 1: Enhancing the Creep Strength of Ni-base Superalloys

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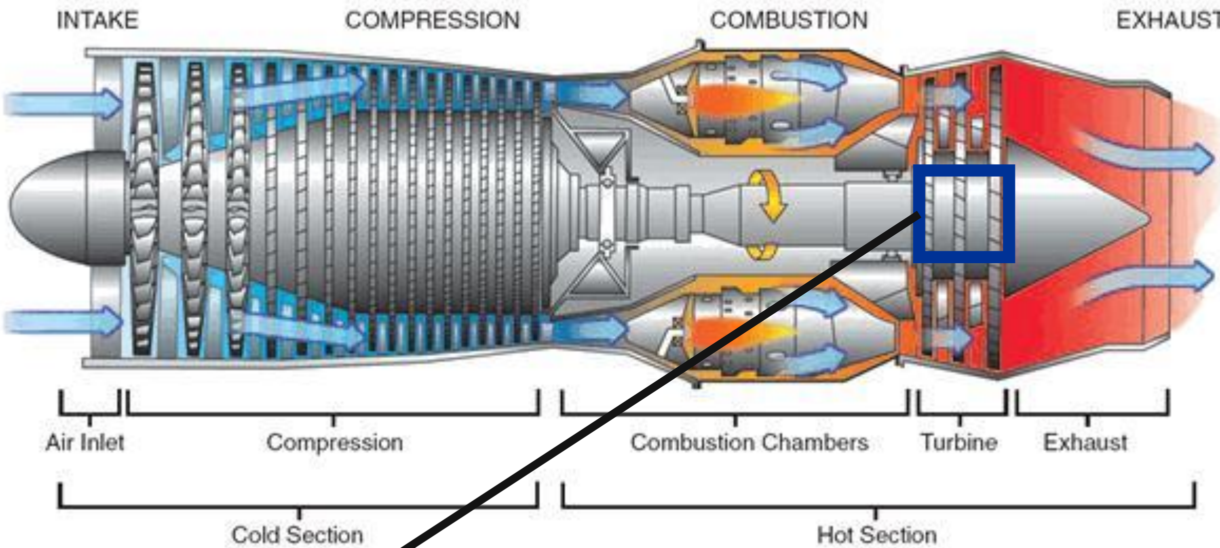
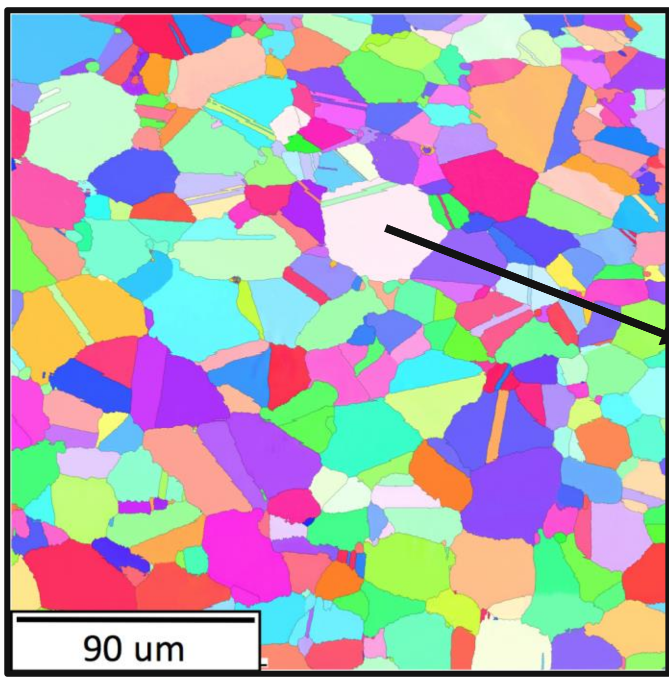
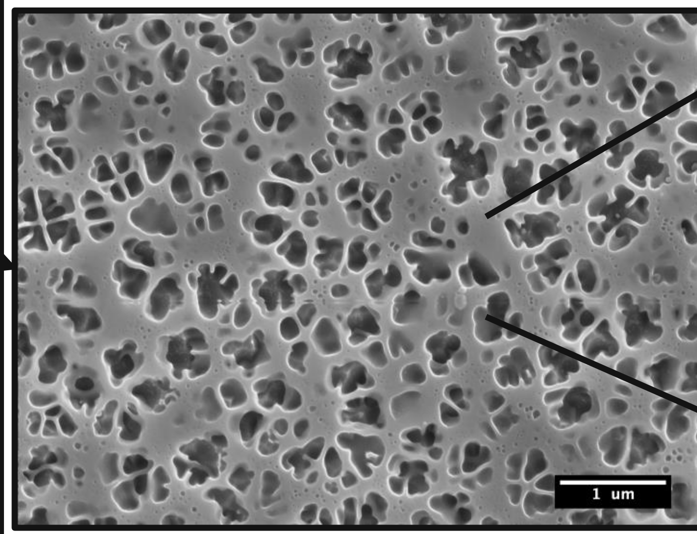
THE OHIO STATE UNIVERSITY

Center for Electron Microscopy and Analysis

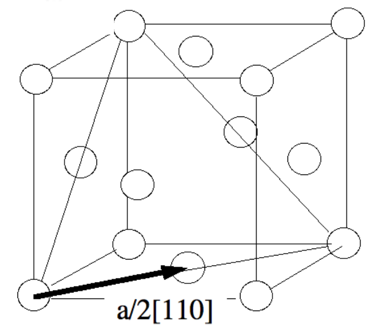
Ni-Based Superalloys for Turbine Disks



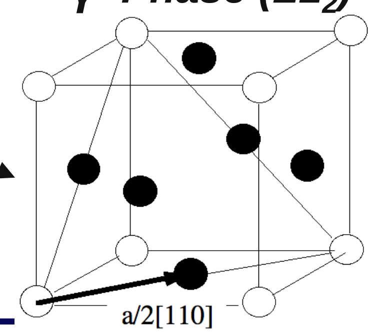




γ Phase (FCC)



γ' Phase ($L1_2$)

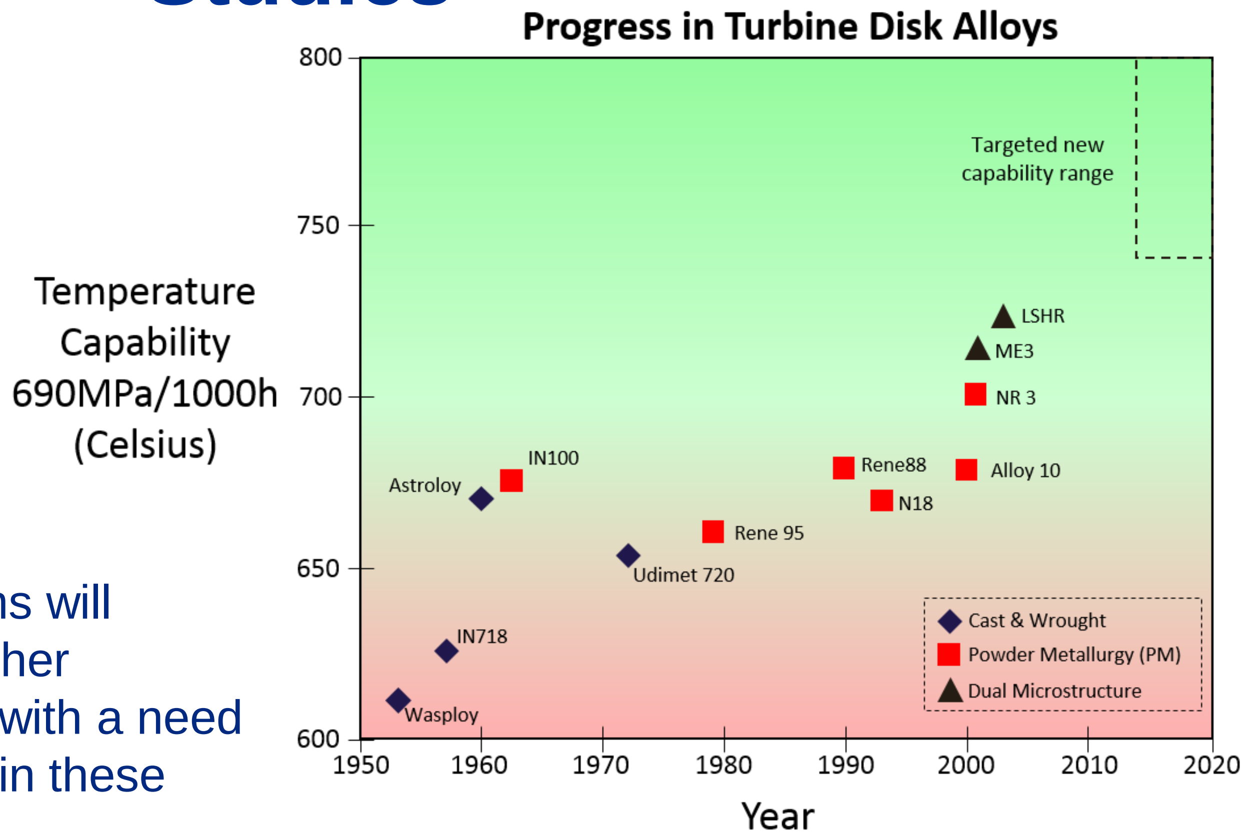


Motivation for Mechanistic Studies

- Material advancements are required to accommodate the higher compressor exit temperatures in jet turbine engines (>700°C near the rotor rim) for improved efficiency and pollution reduction.

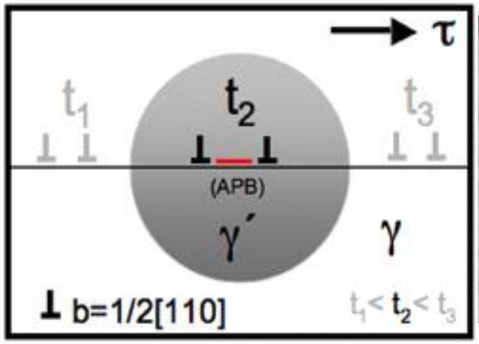
- New deformation mechanisms will become dominant at these higher operating temperatures along with a need for improved creep properties in these disk alloys.

- New understanding and materials will be needed for future advancements

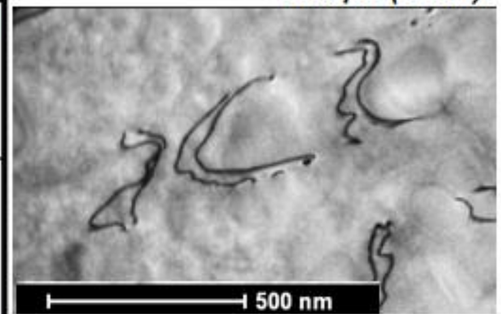


Deformation Mechanisms in Superalloys

Disk Alloys
T<700° C

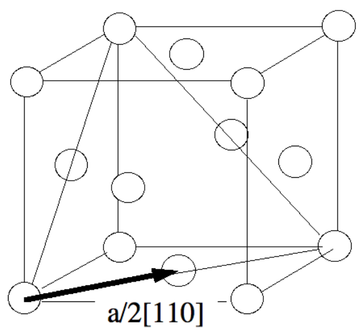


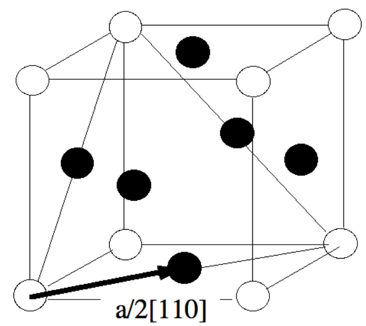
Phillips (2009)



Athermal γ' shearing by 1/2<110> dislocations

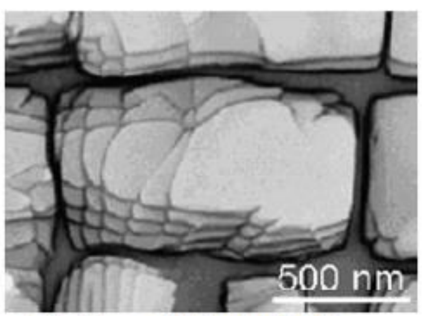
γ Phase (FCC)



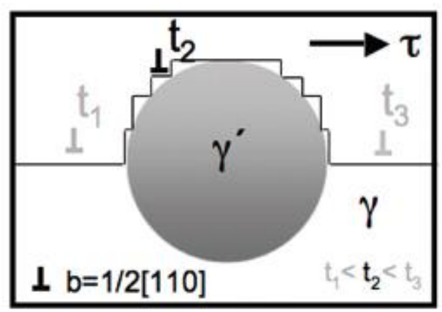


γ' Phase ($L1_2$)

Blade Alloys
T>900° C



Epishin and Link (2008)

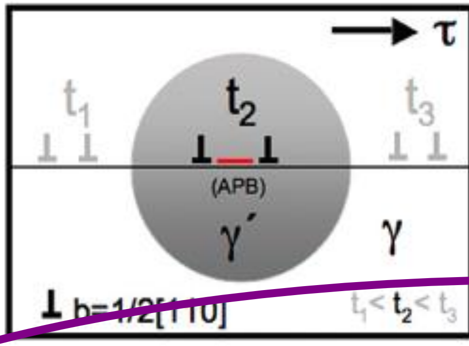


Climb By-Pass of γ′ by individual 1/2<110> dislocations

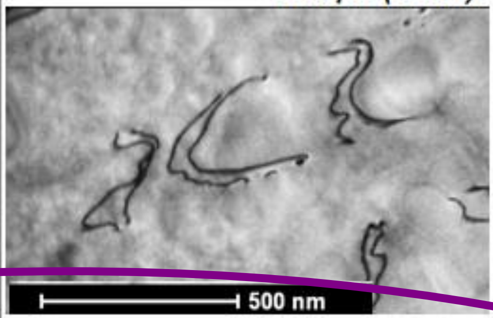
Deformation Mechanisms in Superalloys

Disk Alloys

T<700° C



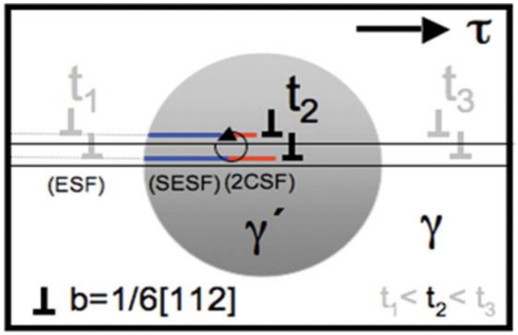
Phillips (2009)



Athermal γ' shearing by 1/2<110> dislocations

Novel mechanisms:

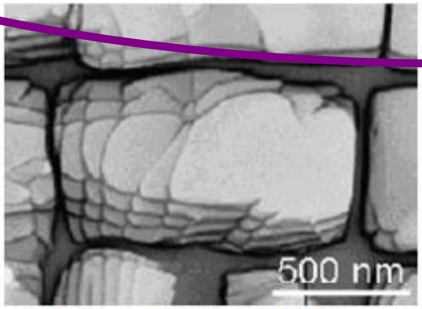
- **Stacking Fault Cutting**
- **Microtwinning**
- **Stacking Fault Ribbons**



Diffusion mediated creep deformation

Blade Alloys

T>900° C



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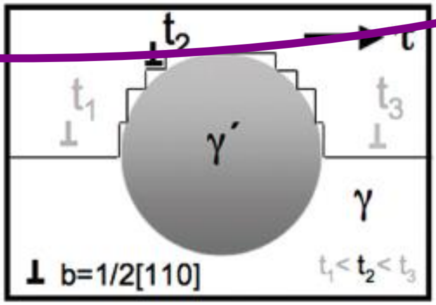
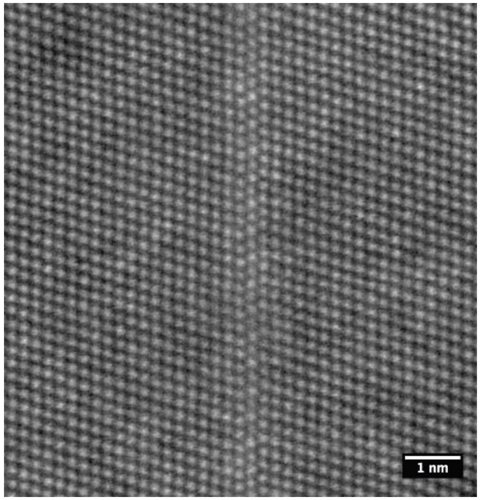
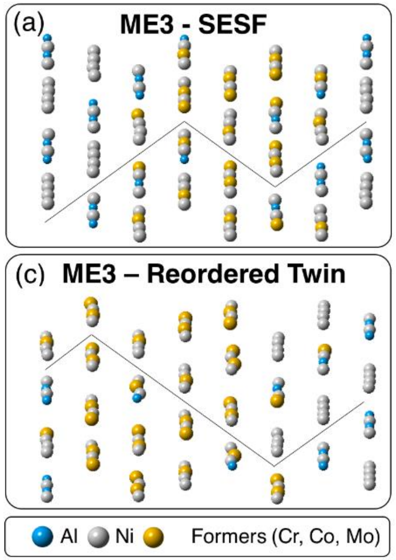


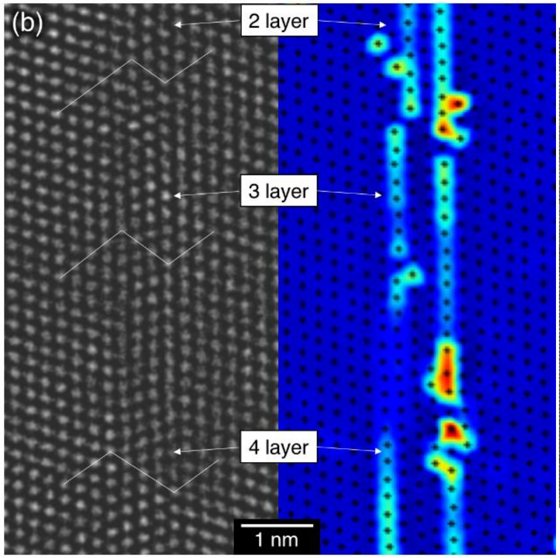
Climb By-Pass of γ′ by individual 1/2<110> dislocations

Deformation Mechanisms: Microtwinning

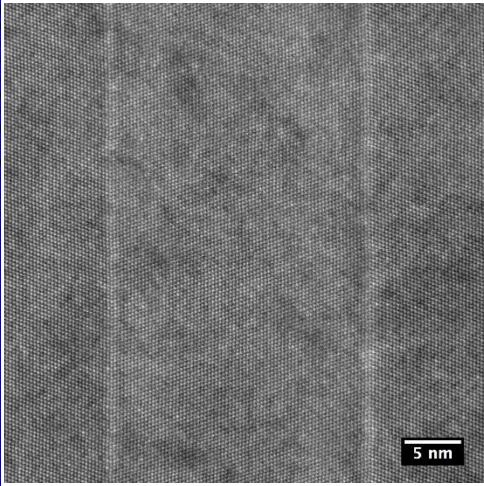
SESFs

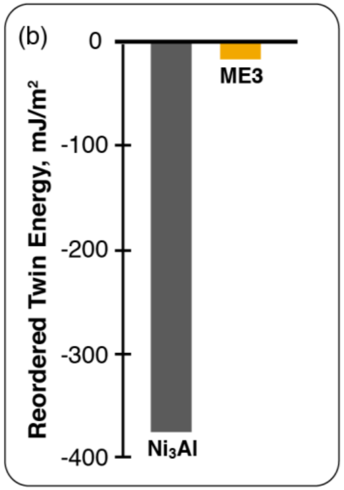






Microtwins

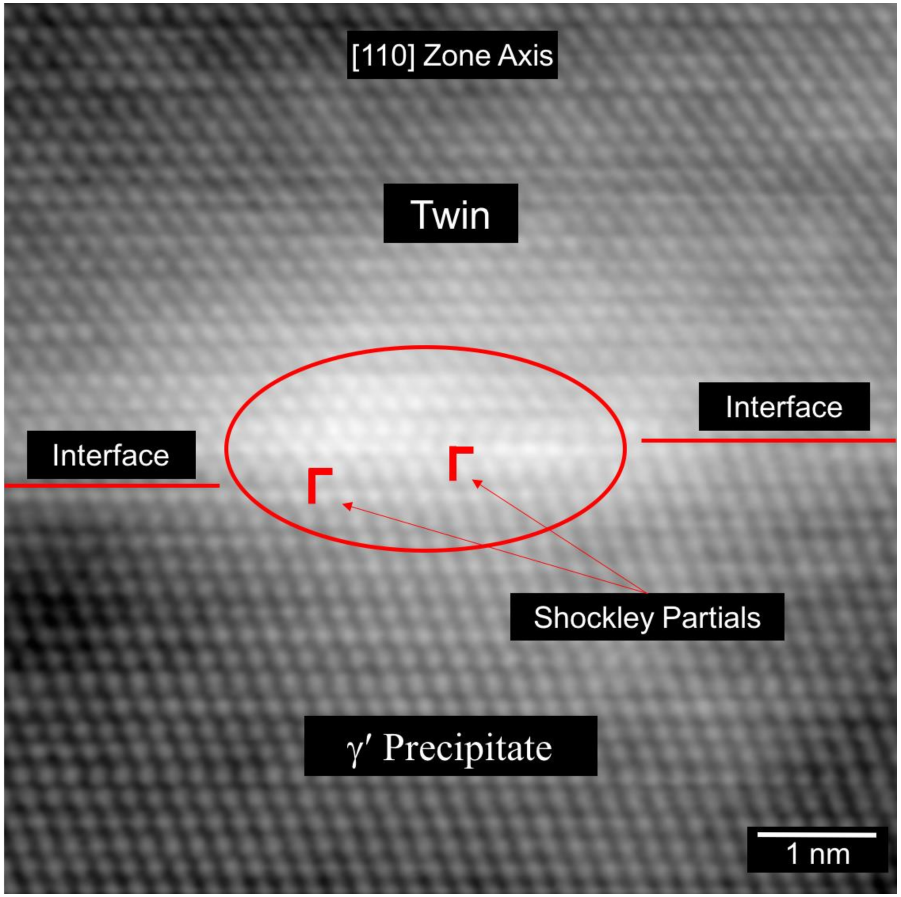


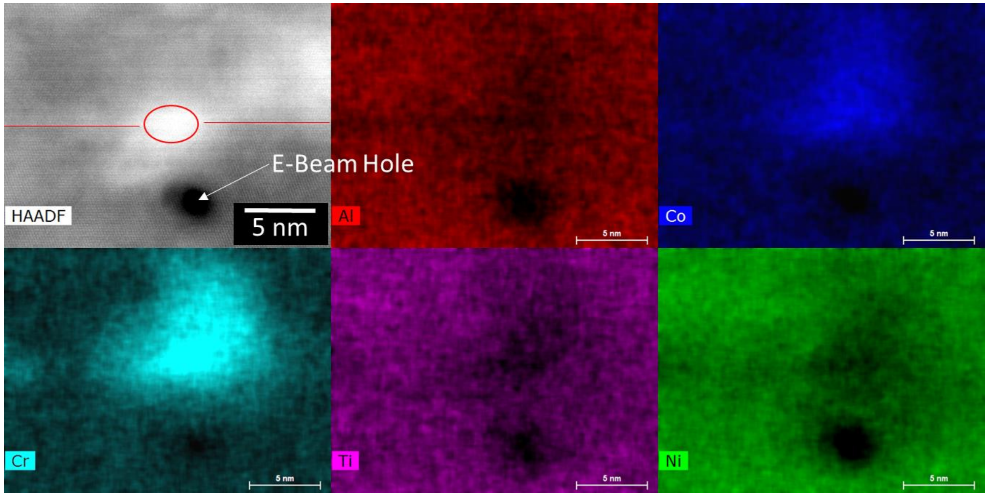


- Microtwins thicken from SESFs via additional Shockley partial pairs shearing along (111) fault planes

- Segregation of “γ former” elements strongly reduces energy penalty for twinning

Deformation Mechanisms: Microtwinning

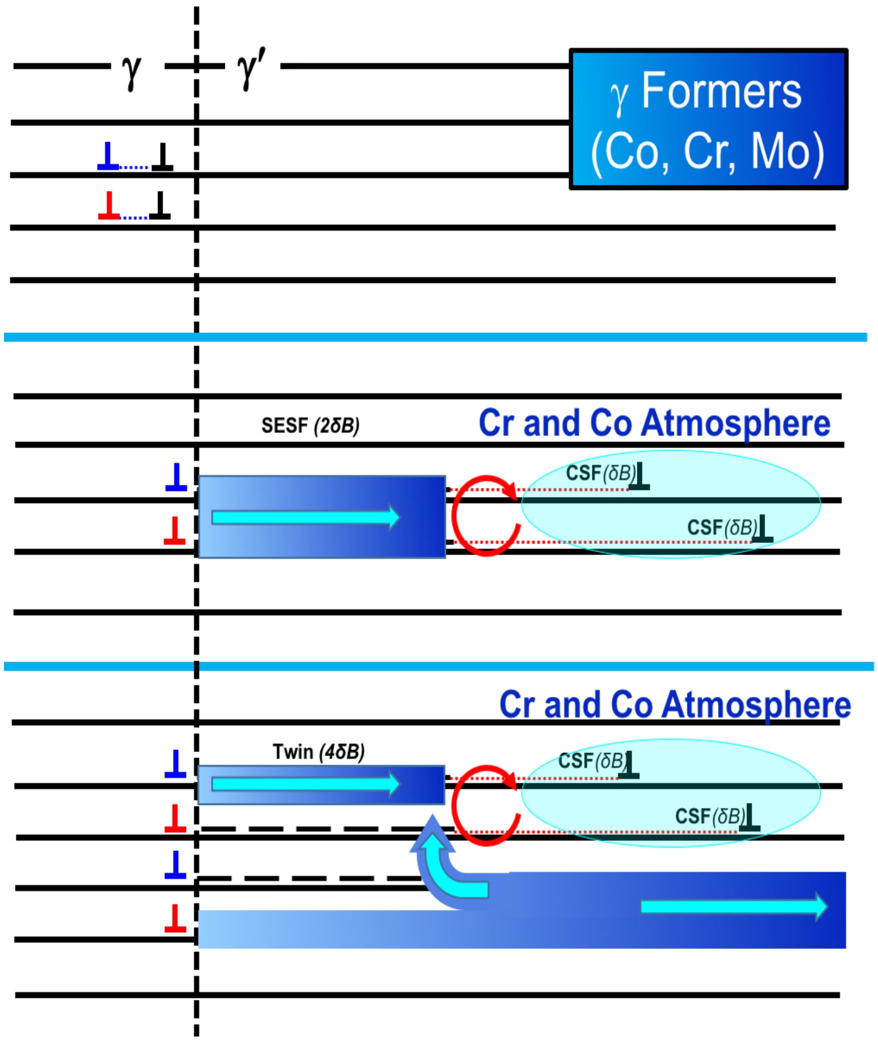


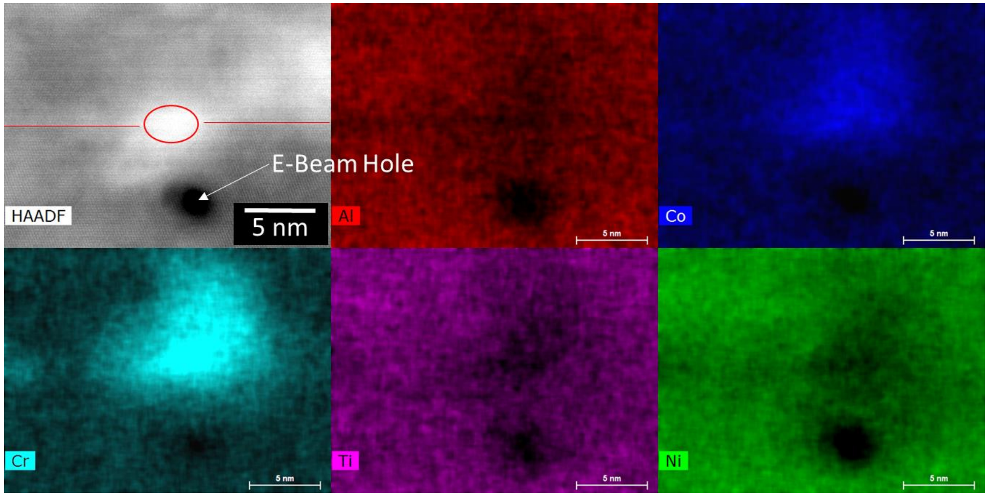


- Dissimilar matrix dislocations react at γ/γ' interface – shearing by Shockley partial pairs
- Stacking fault shearing controlled by segregation and Cottrell atmospheres
- Rate of microtwinning also limited by segregation and Cottrell atmospheres
- Can these deformation modes be mitigated/eliminated?

Smith, et al. Acta Materialia, 2017

Deformation Mechanisms: Microtwinning





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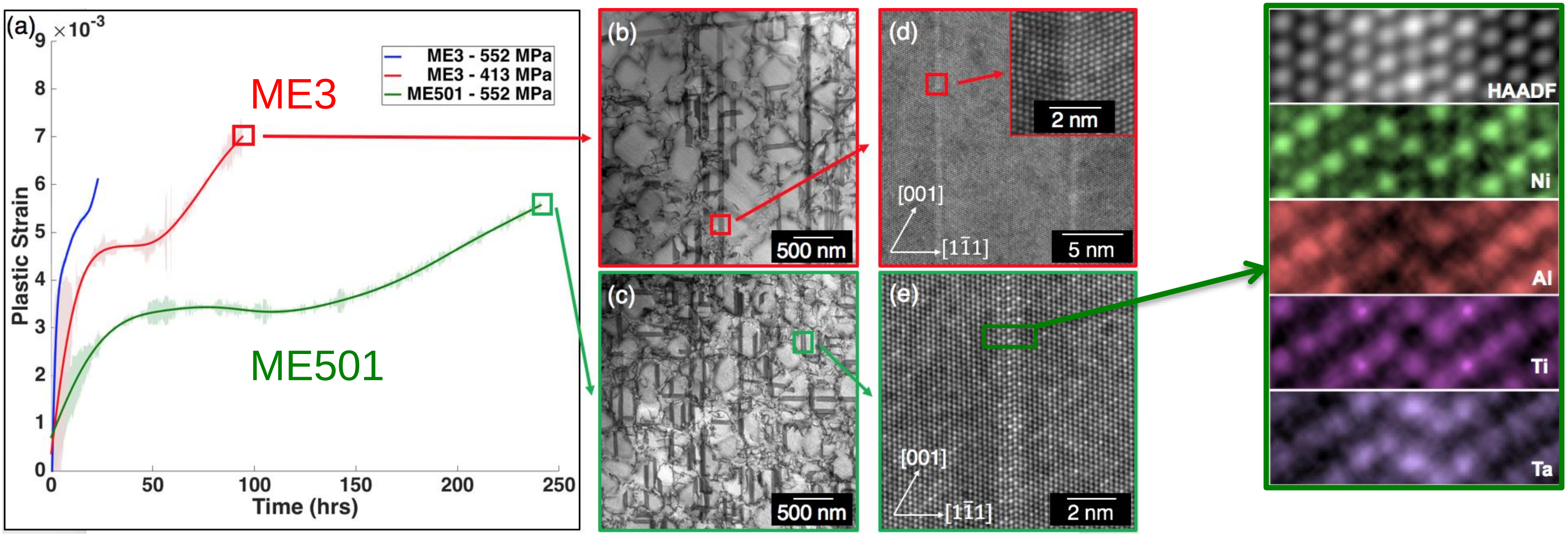
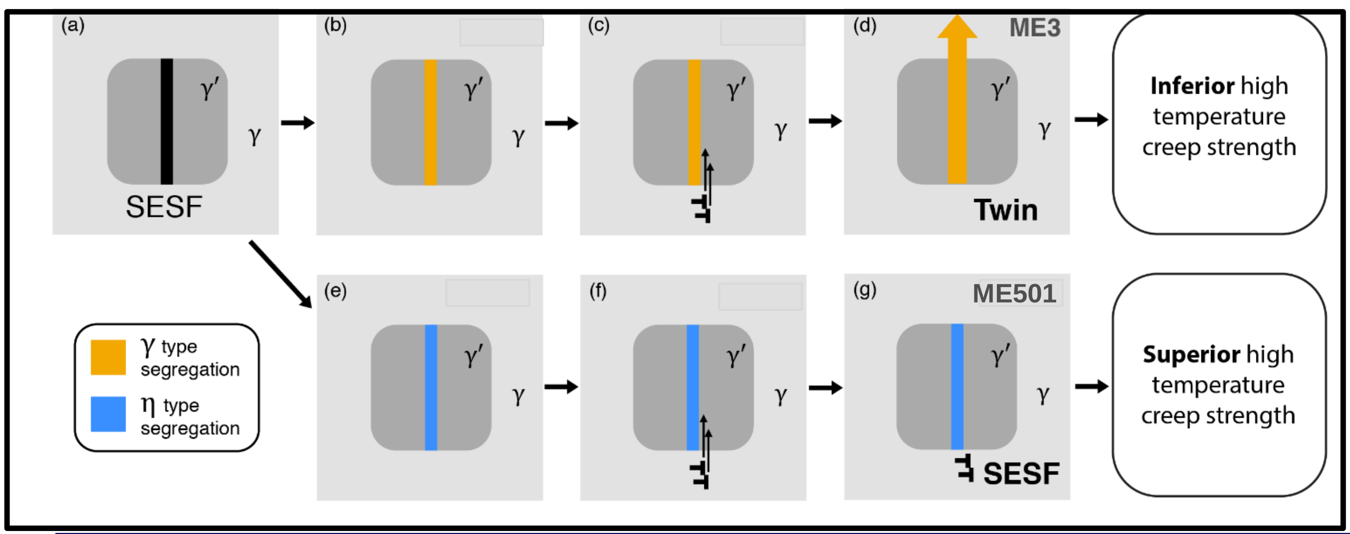
Smith, et al. Acta Materialia, 2017

Phase Transformation Strengthening



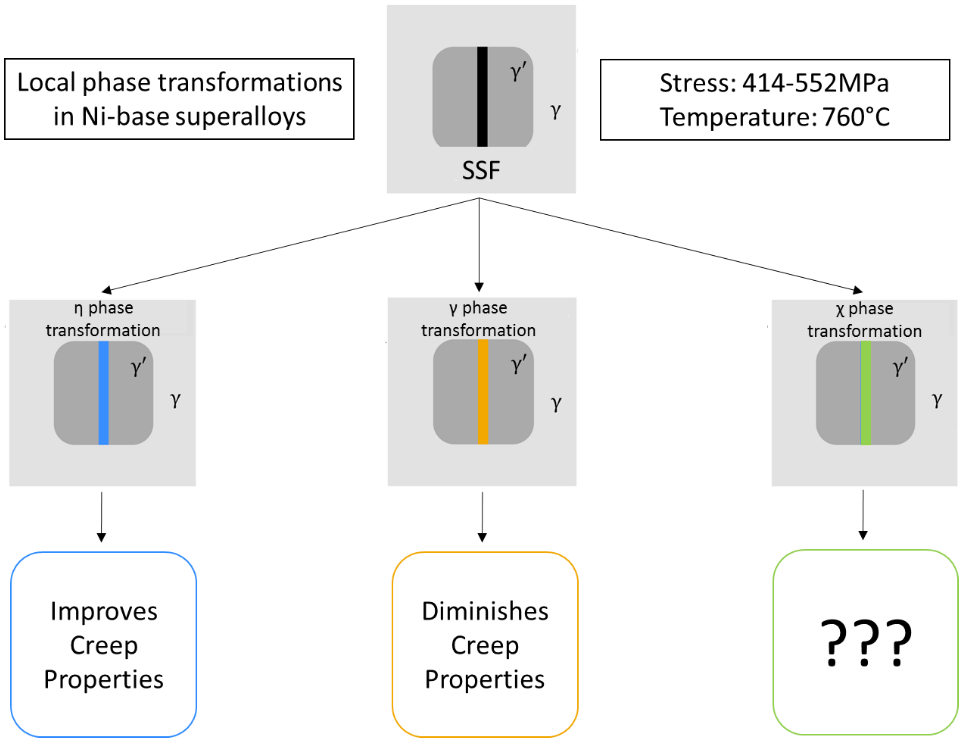


New insight into alloy effects:

- Segregation of γ formers in ME3 promotes microtwinning
- Formation of η phase at faults in ME501 inhibits microtwinning and improves creep strength

Smith, et al. Nature Communications, 2016

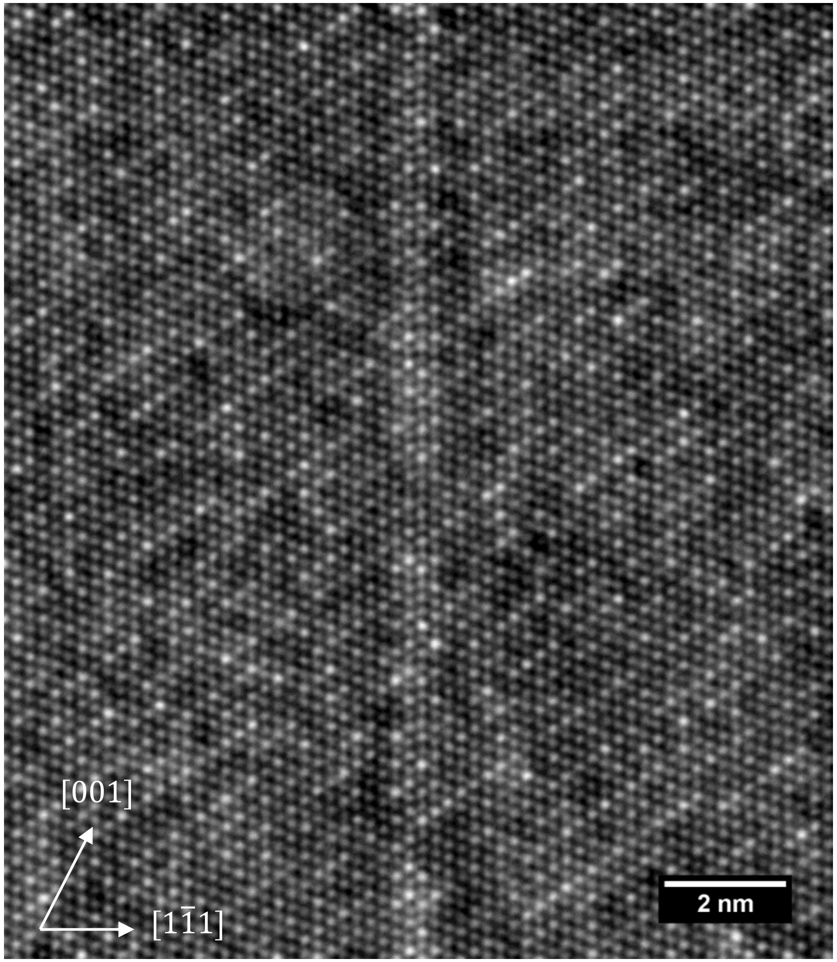
Phase Transformations along SISFs



SESFs

SESFs + SISFs

SISFs



CMSX-4 (high W content)*

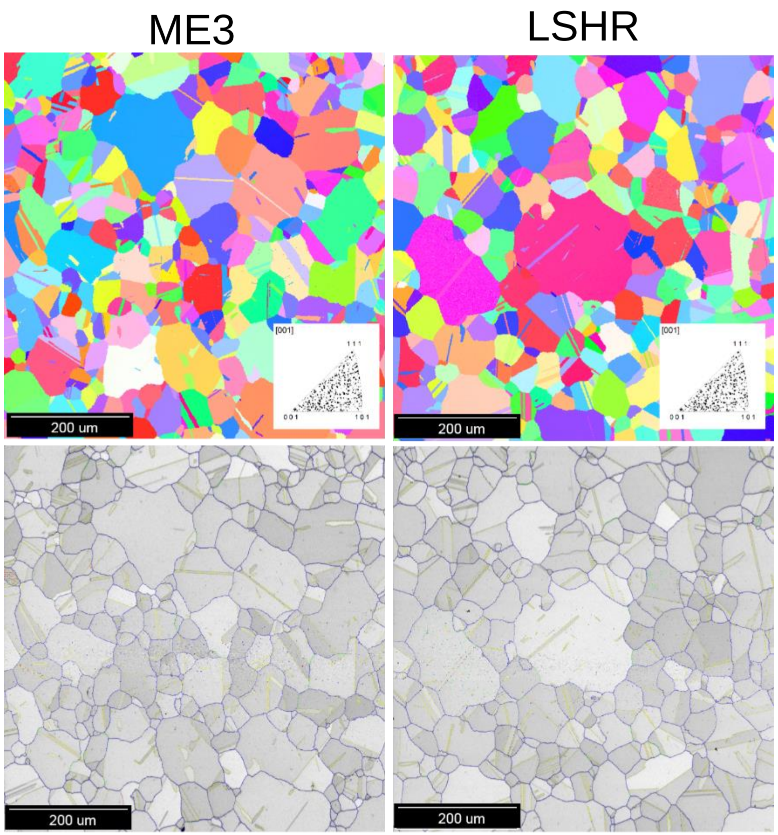
Does the observed χ (Co_3W) or γ phase transformations along SISFs have any impact on creep properties?

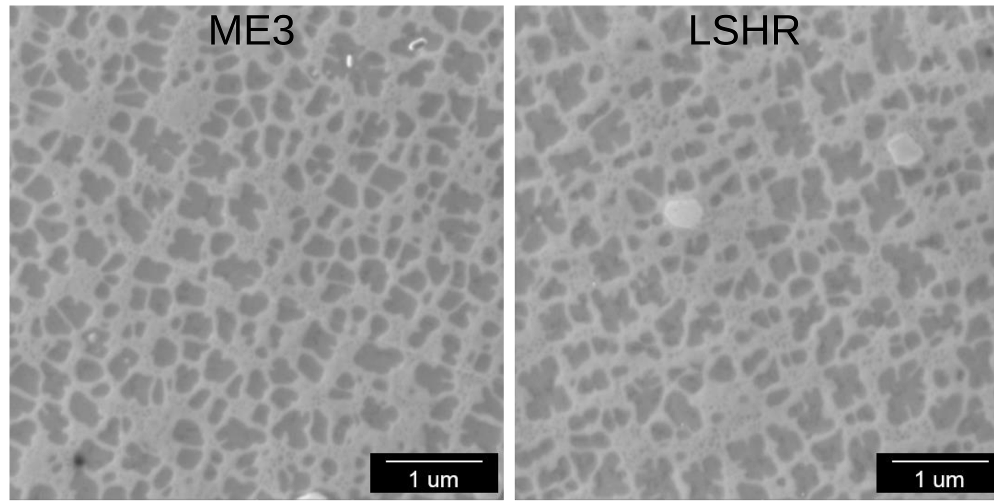
*Smith *et al.* 2018

SESF = Superlattice Extrinsic Stacking Fault SISF = Superlattice Intrinsic stacking Fault

Material Preparation

Average Alloy Composition in Weight Percent												
Alloy	Cr	Co	Al	Ti	**Nb**	**Mo**	**Ta**	**W**	Zr	B	C	Ni
LSHR	12.5	20.4	3.5	3.5	**1.5**	**2.7**	**1.5**	**4.3**	0.05	0.03	0.045	Bal
ME3	13	21	3.4	3.8	**0.8**	**3.7**	**2.4**	**2.1**	0.05	0.02	0.05	Bal



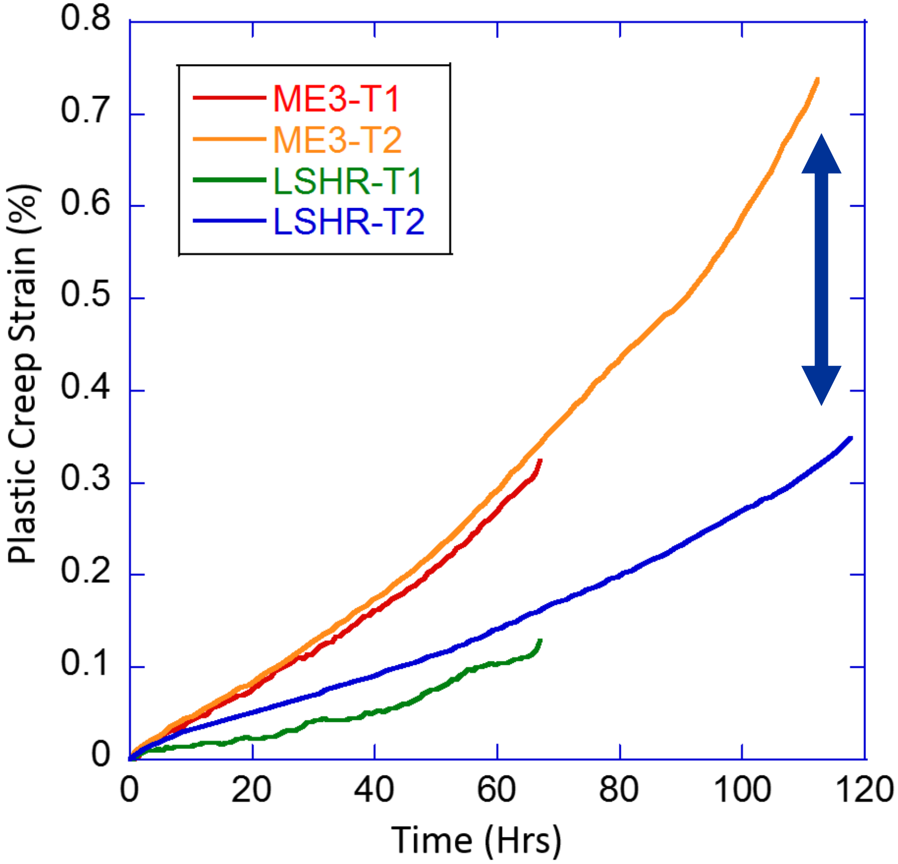


Alloy	Secondary γ' VF	Tertiary γ' VF	**Total γ' VF**	Average Secondary γ' Size	Average Tertiary γ' Size
ME3	43.97 ± .6	2.65 ± .4	**46.61 ± 1.0**	135 nm	15.4 nm
LSHR	43.52 ± 1.7	2.27 ± .1	**45.80 ± 1.8**	154 nm	15.9 nm

ME3 Average Grain Diameter = 59.2 μm
LSHR Average Grain Diameter = 59.9 μm

The two alloys are microstructurally comparable!

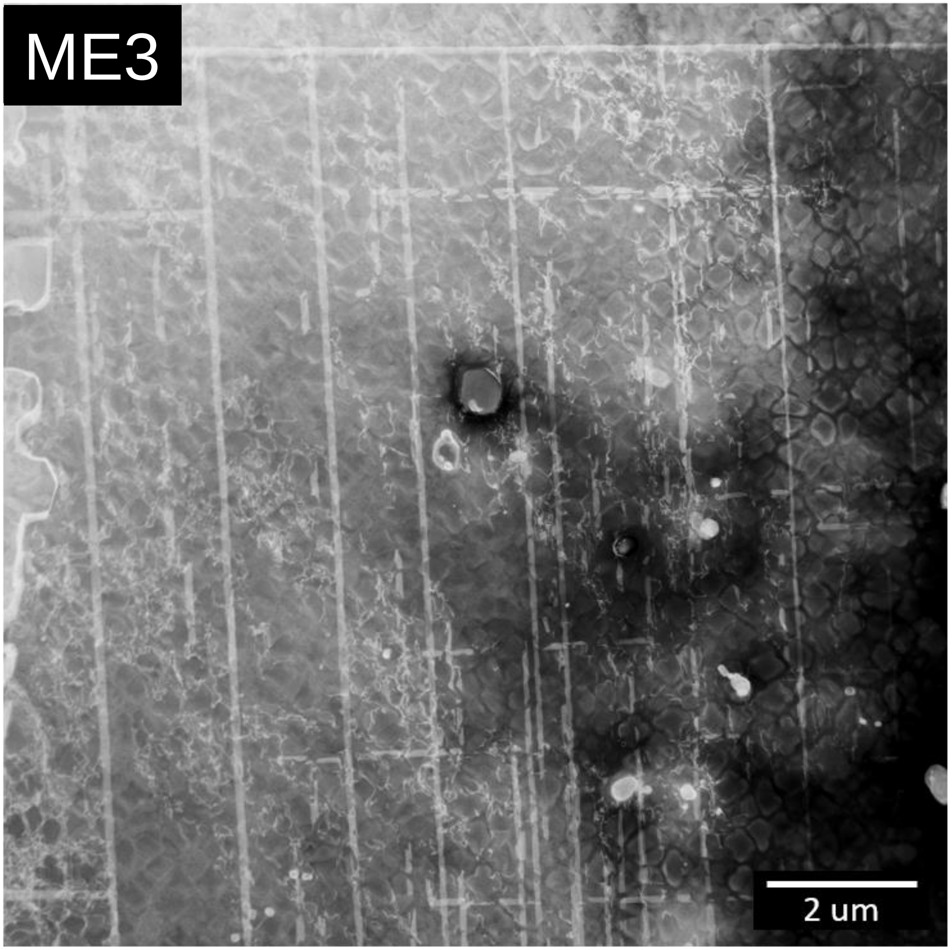
Creep Testing of ME3 and LSHR

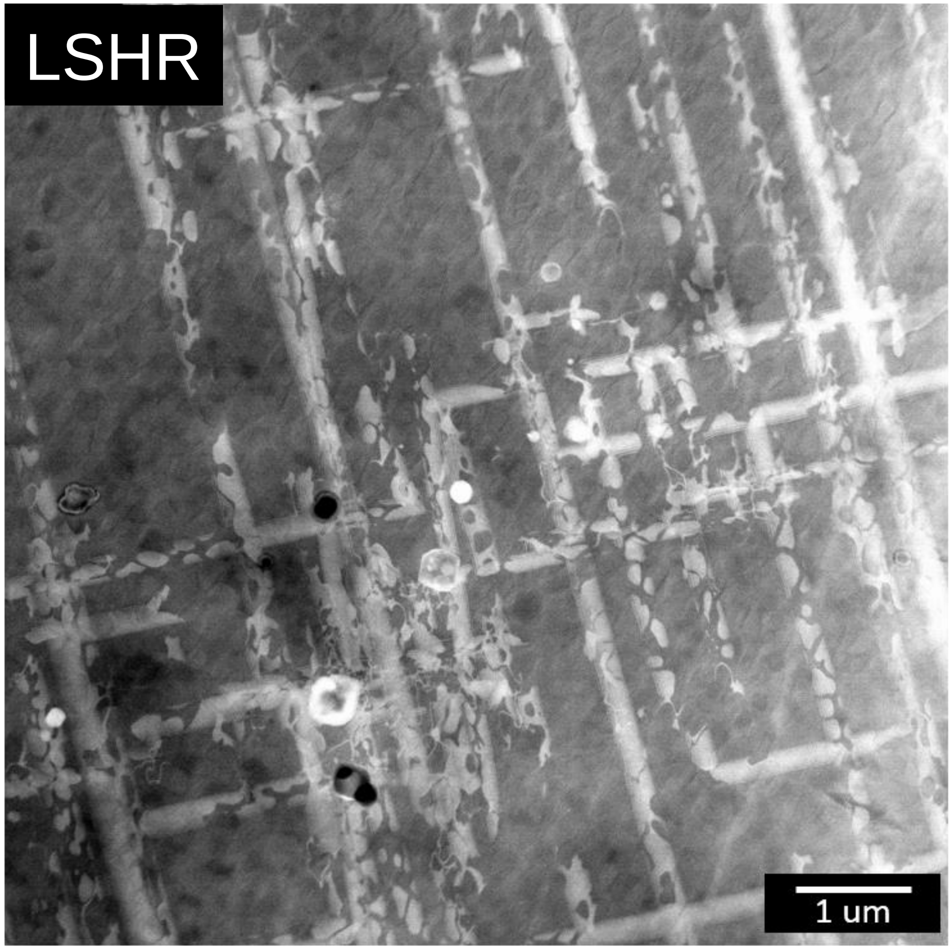


Creep tests were performed at 760°C under a stress of 552MPa

LSHR has consistently performed better in creep compared to ME3 in this temperature regime. Why?

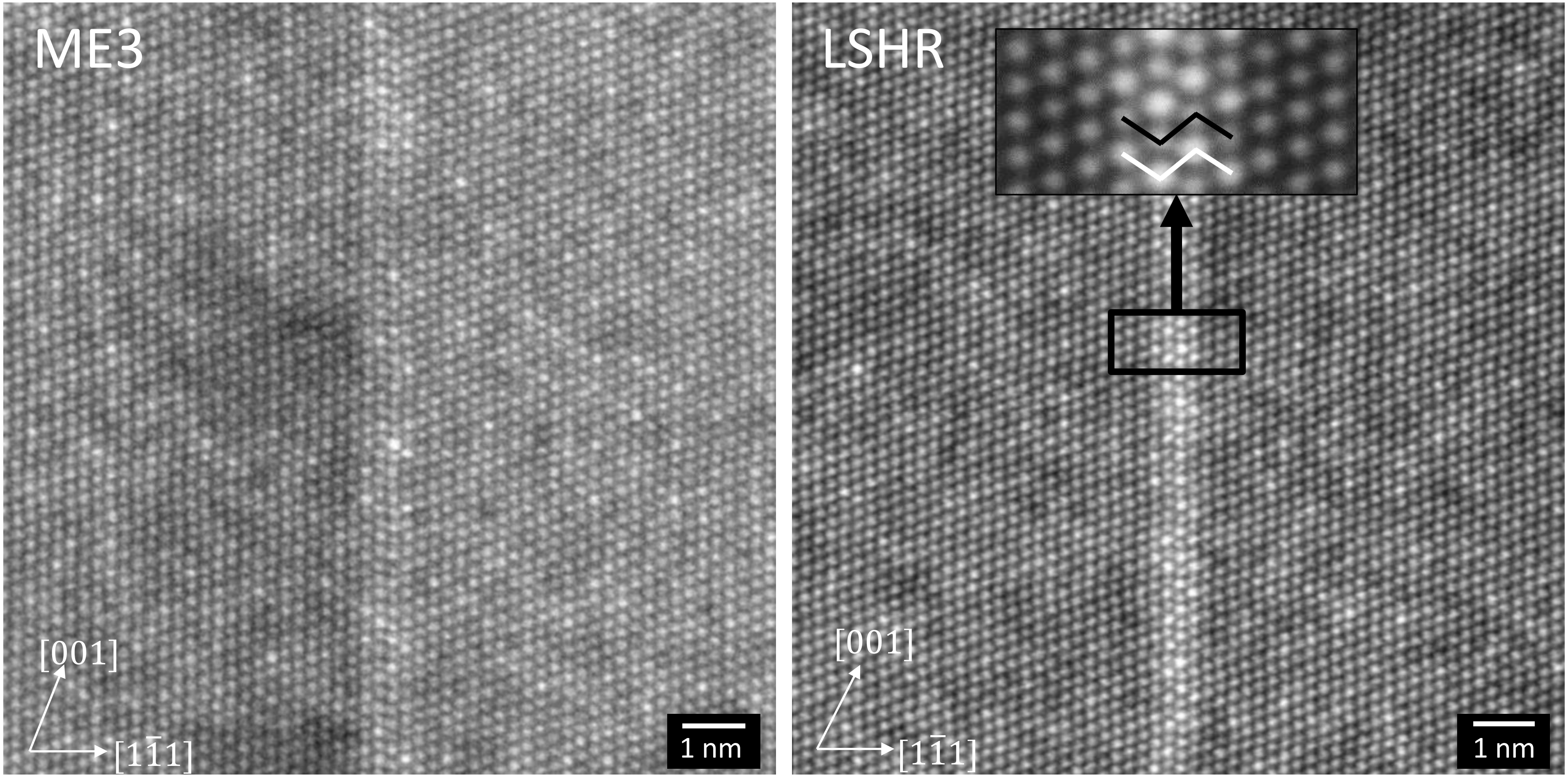
Scanning Transmission Electron Microscopy Characterization





No notable differences in active deformation modes could be discerned between the two alloys.

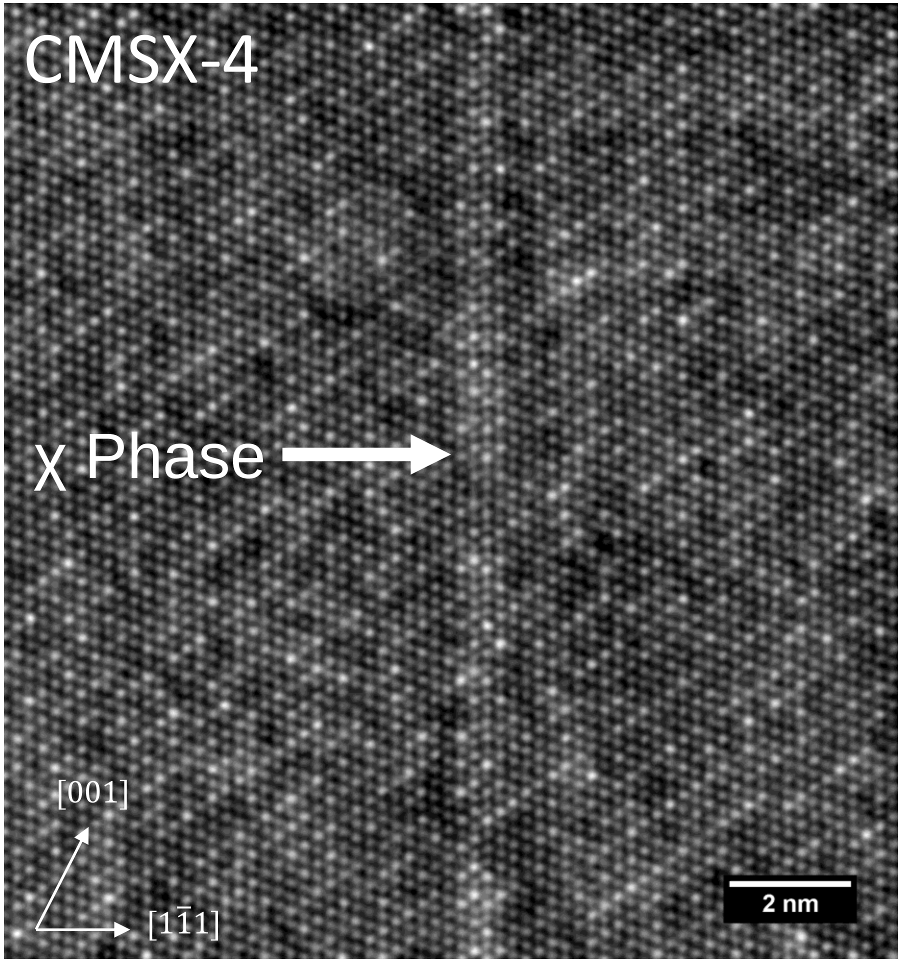
Segregation along SISFs in ME3 and LSHR

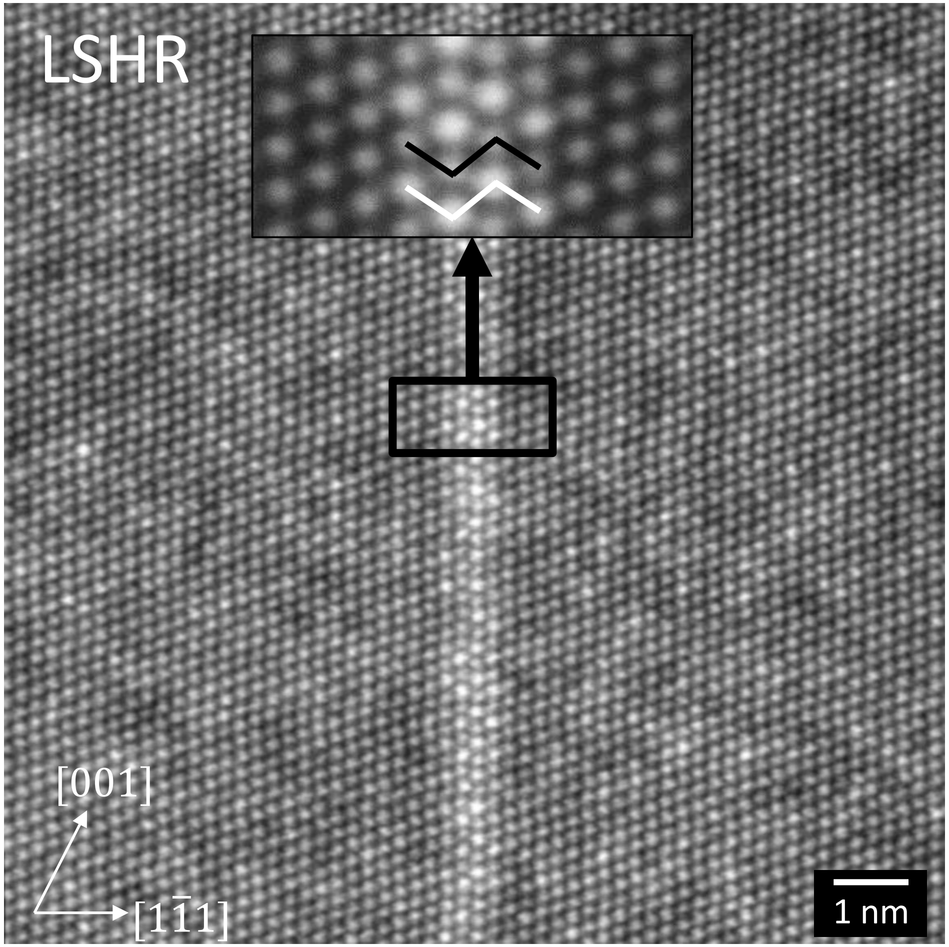


Ordered contrast exists along SISFs in LSHR but not ME3

SISF = Superlattice Intrinsic stacking Fault

Segregation along SISFs in ME3 and LSHR

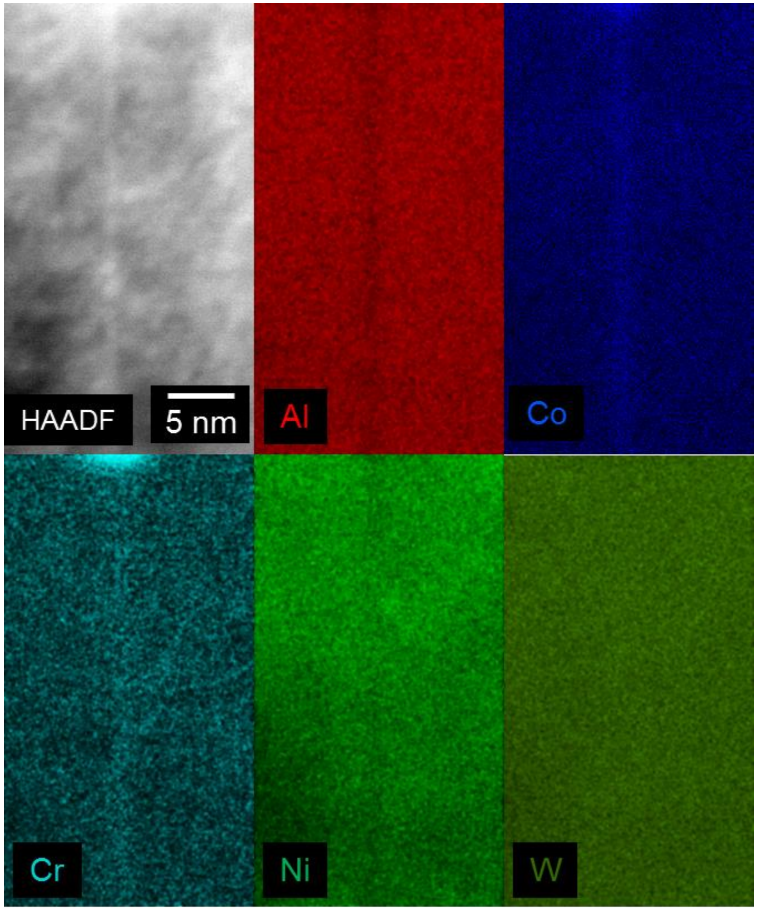


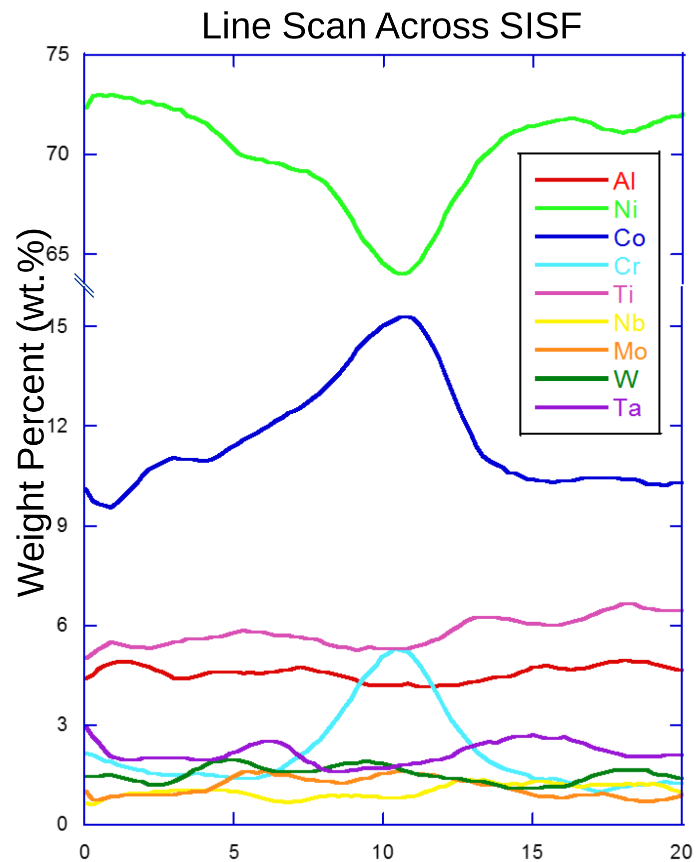


Ordered contrast exists along SISFs in LSHR but not ME3

SISF = Superlattice Intrinsic stacking Fault

Segregation along SISFs – ME3



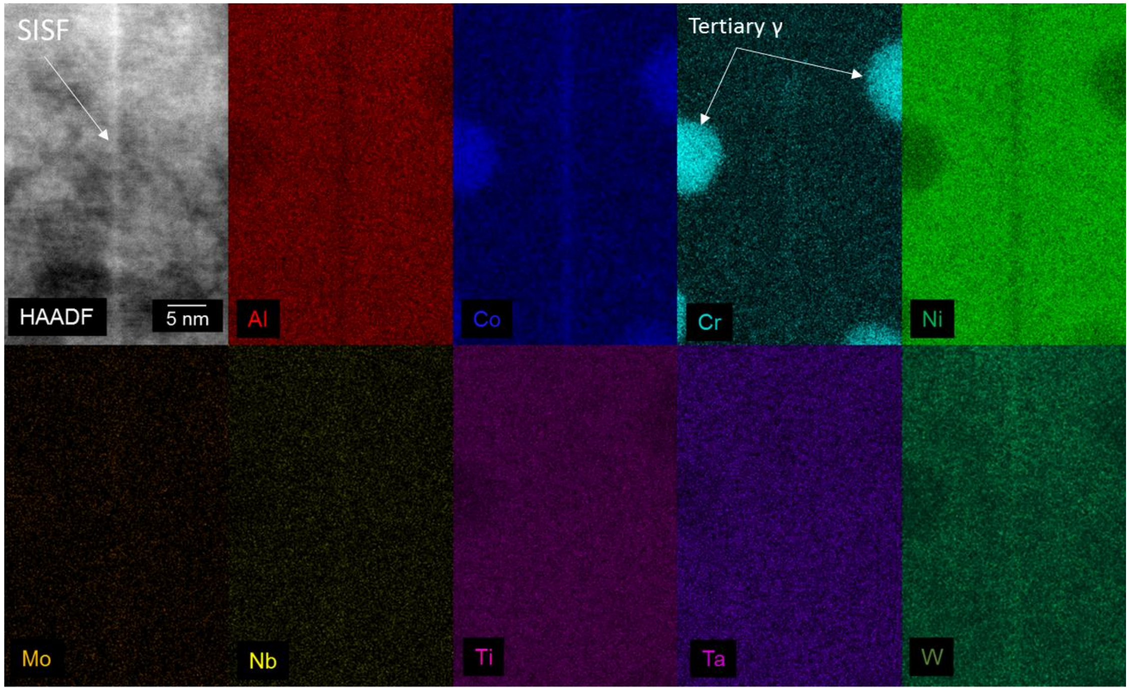


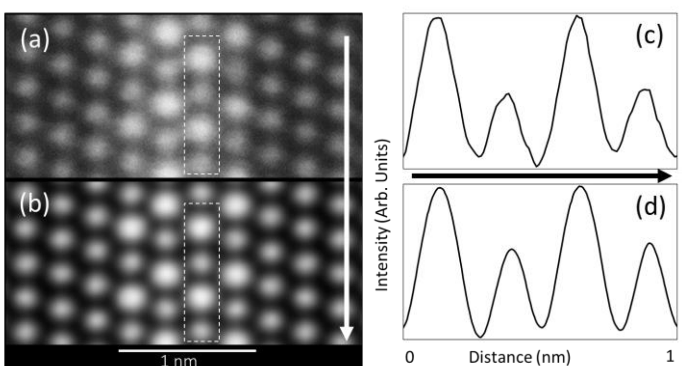
Confirmed γ phase nucleation along SISFs in ME3

Co, Cr Segregation
Ni, Al Depletion

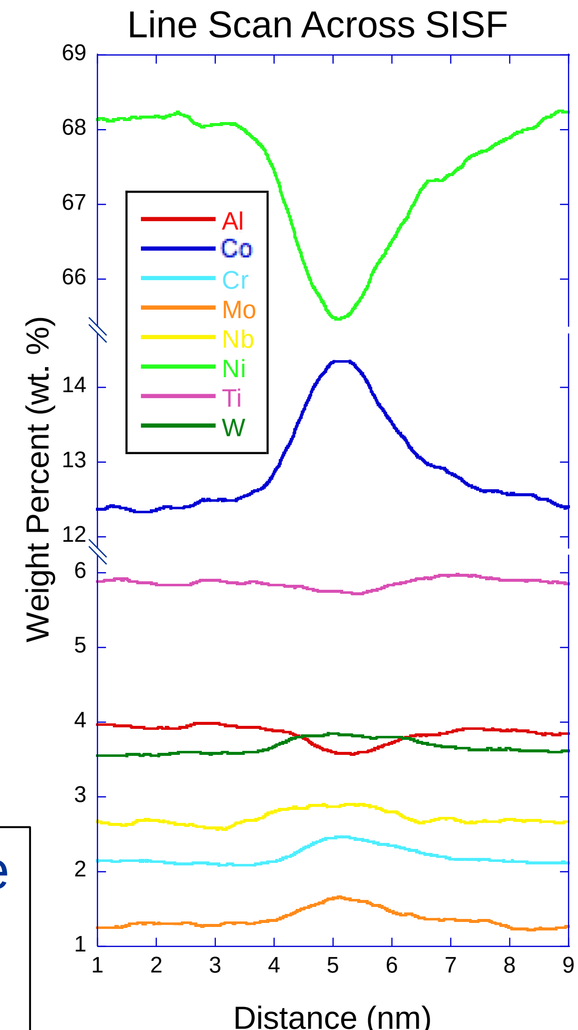
SISF = Superlattice Intrinsic stacking Fault

Segregation along SISFs - LSHR





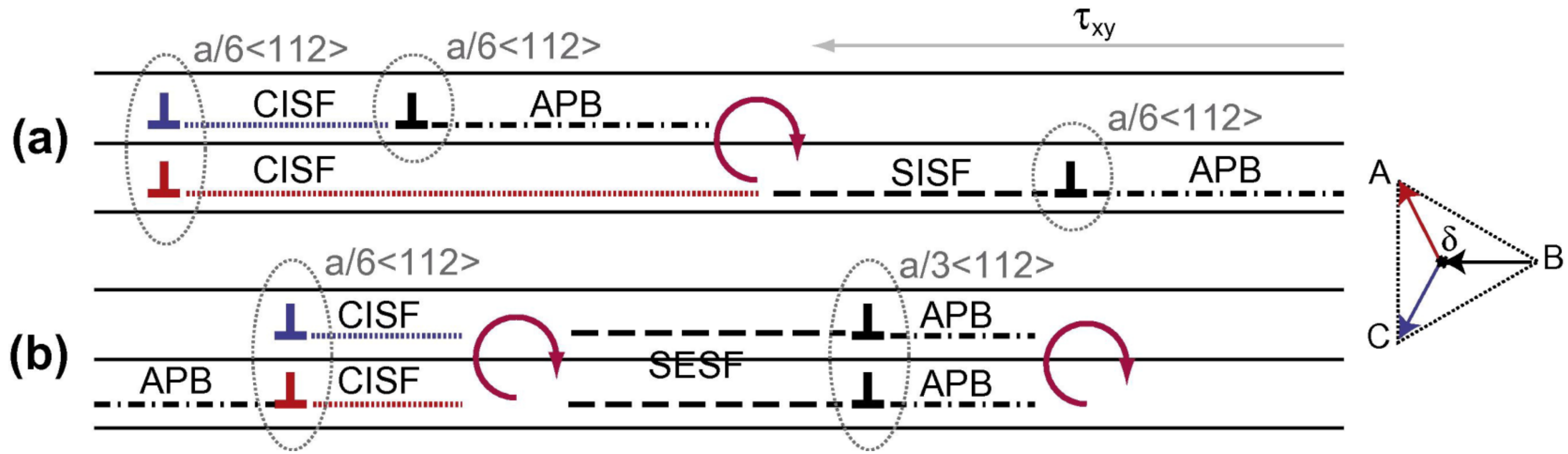
Confirmed χ phase nucleation along SISFs in LSHR



Co, W, Cr Segregation
Ni, Al Depletion

SISF = Superlattice Intrinsic stacking Fault

Stacking Fault Ribbon Formation



Vorontsov *et al*. Acta Materialia. 2012

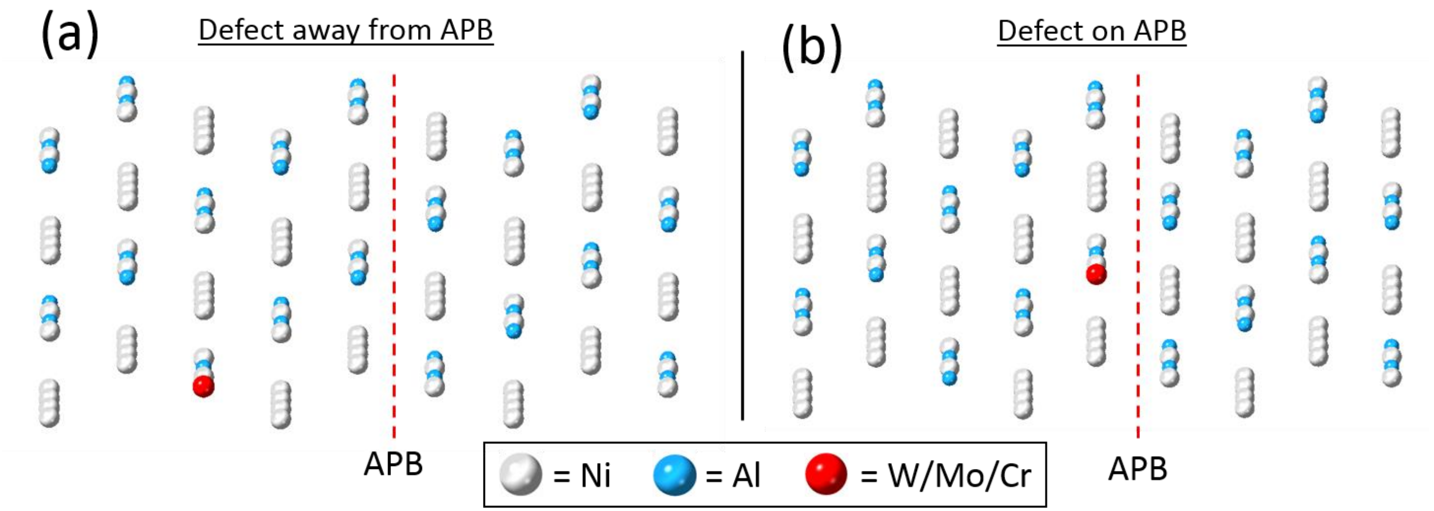
$$\frac{a}{3} < 112 > (SISF) + \frac{a}{6} < 112 > (APB) + \frac{a}{6} < 112 > (SESF) + \frac{a}{3} < 112 > = a < 112 >$$

Stacking Fault ribbons are a major source of primary creep strain in this temperature regime for single crystal superalloys

C.M.F. Rae and R.C. Reed. Acta Materialia. 2007

What effects will γ or χ phase formation along SISFs have on this shearing process?

Density Functional Theory Measurements

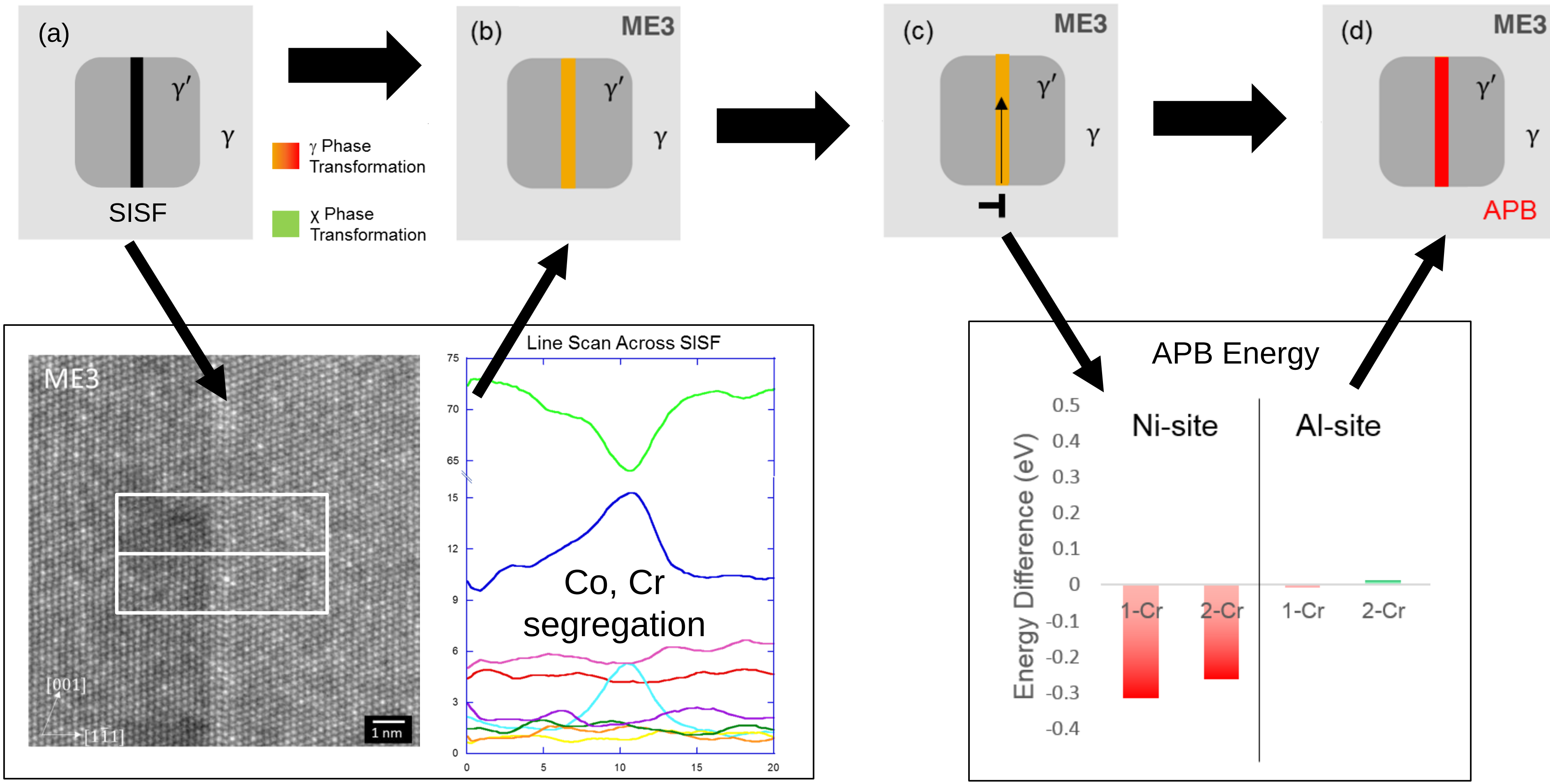


Ni_3Al γ' cells with an APB were created to explore the effect SISF segregation has on the formation of the trailing APB.

Relaxed energies were compared when a W, Mo, or Cr atom were away from the APB or on the APB.

APB = Anti-Phase Boundary

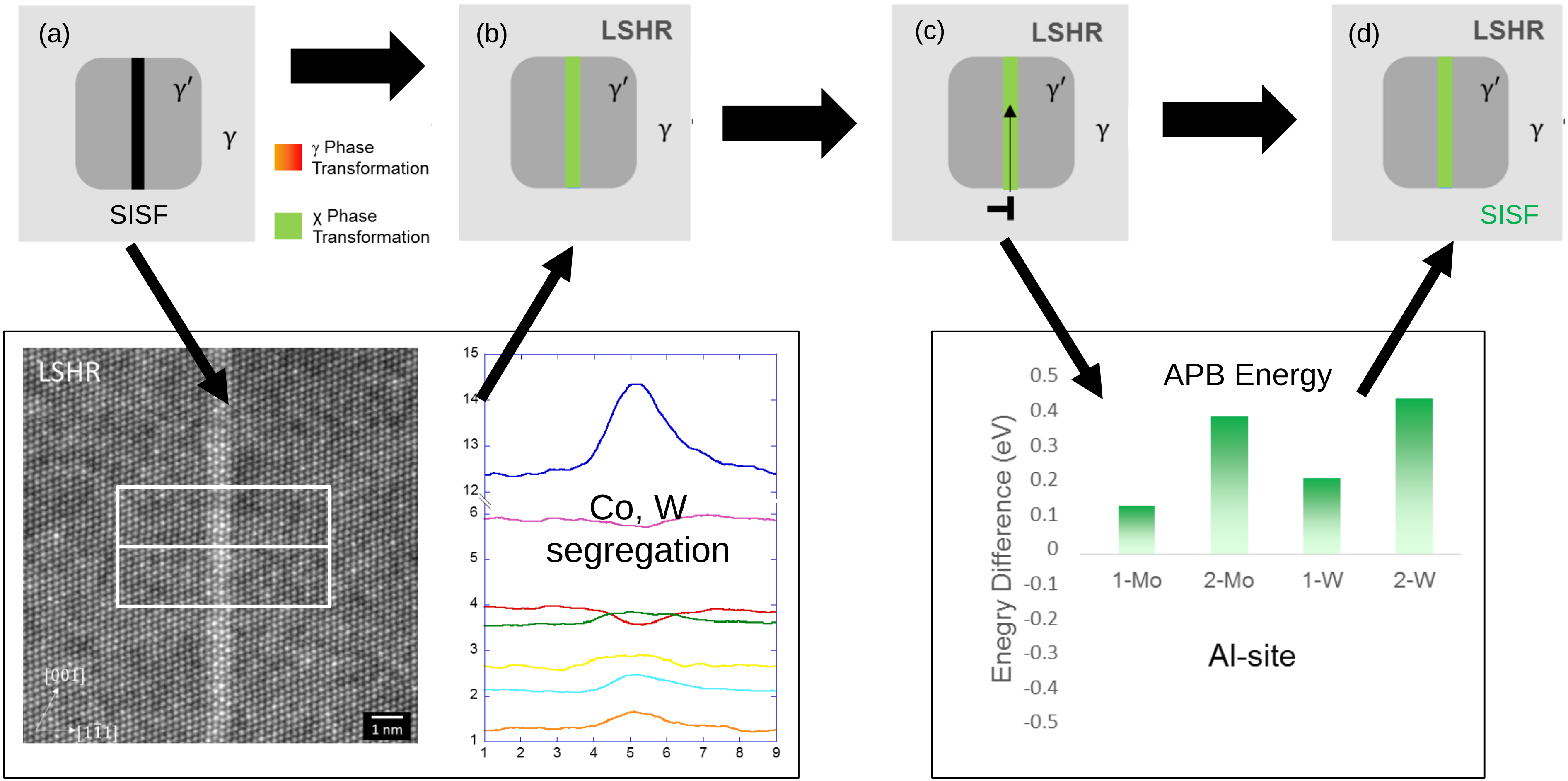
Phase Transformation Softening – γ Phase



γ phase formation along SISF promotes stacking fault ribbon shear

SISF = Superlattice Intrinsic stacking Fault

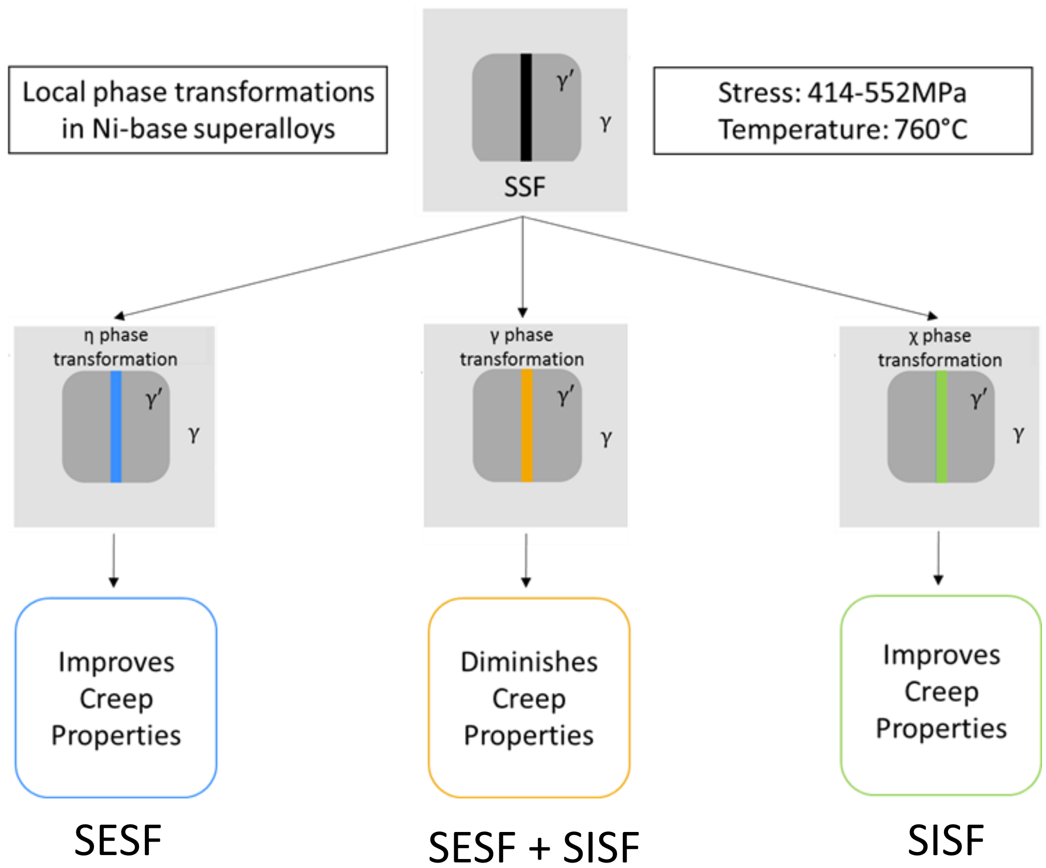
Phase Transformation Strengthening – χ phase



χ phase formation along SISF inhibits stacking fault ribbon shear

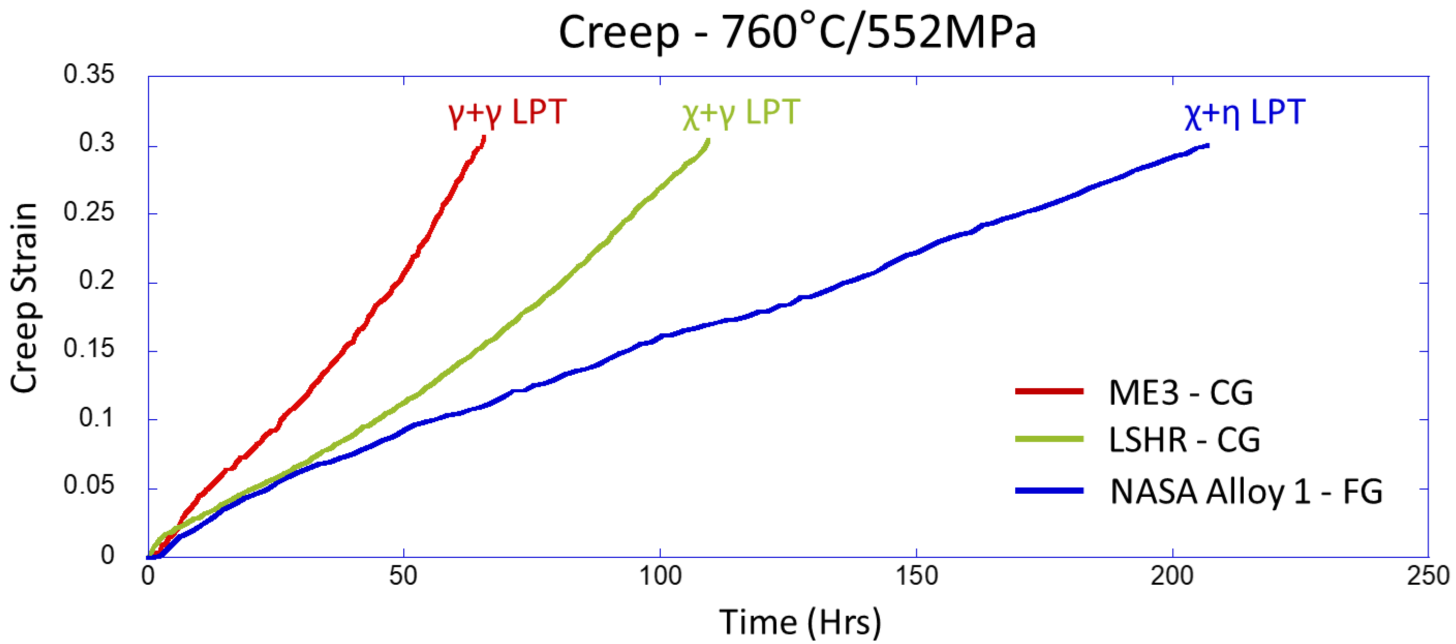
SISF = Superlattice Intrinsic stacking Fault

Phase Transformation Strengthened Superalloys



Can the η and χ phase transformation strengthening mechanisms be combined into a single alloy without precipitating bulk topologically close packed (TCP) phases?

NASA Alloy 1



Conclusion: NASA Alloy 1 presents significantly better creep properties over current state of the art alloys through phase transformation strengthening

Part 2: Efficient Production of a High Performance Dispersion Strengthened, Multi-principal element alloy

TM Smith[1], AC Thompson[2], TP Gabb[1], RB Rogers[1], MJ Kulis[1], KM Tacina[1]

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[2]Vantage Partners, 3000 Aerospace Pkwy, Brook Park, OH 44142, USA

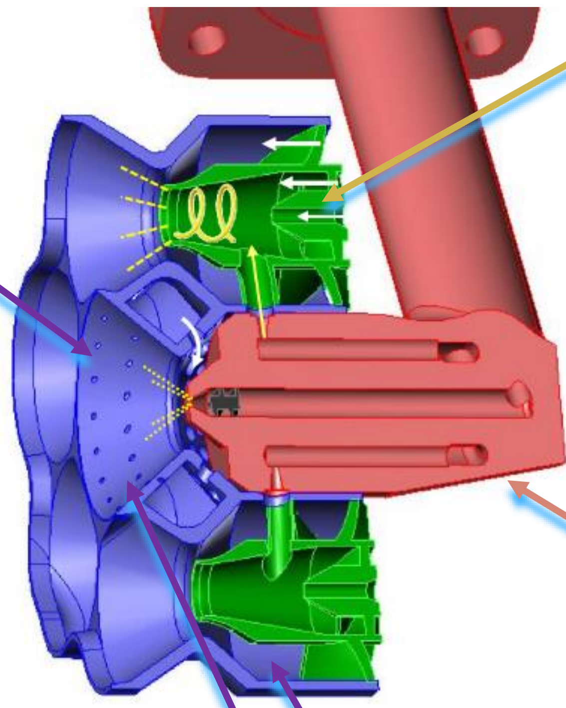
Support provided by NASA's Aeronautics Research Mission Directorate (ARMD) Transformational Tools and Technologies (TTT) Project Office

Background – NASA Application

Problem: Conventional materials and processing techniques limit the design of combustor domes used in jet turbine engines.

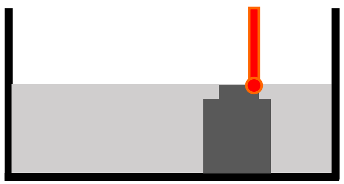
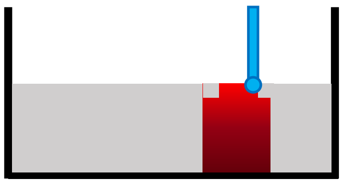
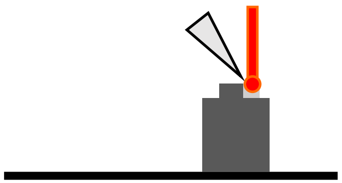
Proposed Solution: Develop a high ductility, high temperature material for an additively-manufactured (AM) combustor fuel nozzle and dome for supersonic aircraft (>1093°C (2000°F) operating temperature).

- Lead to several improvements to the turbine combustor design ultimately reducing NOx pollution and lowering weight.
- May enable lean-front-end small-core combustors.



Schematic of AM produced fuel injector/combustor dome

Metallic Additive Manufacturing

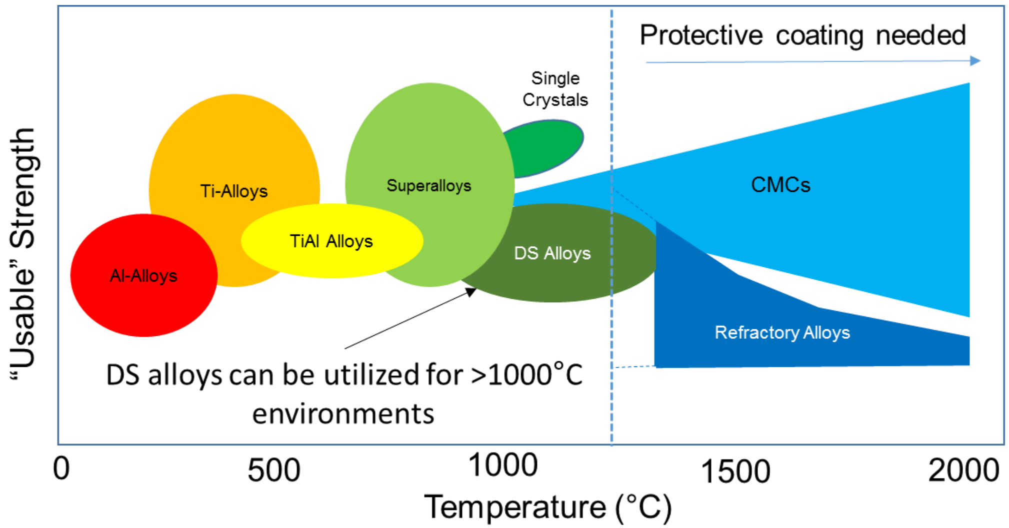
Process	Selective Laser Melting (SLM) 	Electron Beam Melting (EBM) 	Direct Energy Deposition (DED) 
Energy Source	Laser	E-Beam	Laser or E-Beam
Powder Bed	Yes	Yes	No
Power (W or kV)	50-1000 W	**30-60kV**	100-2000 W
Max Build Size (mm)	500 x 280 x 320	500 x 280 x 320	**2000 x 1500 x 750**
Material	Metallic Powder	Metallic Powder	Metallic Powder or Wire
Dimensional Accuracy	**<0.04 mm**	0.04-0.2 mm	0.5 mm (powder) 1.0 mm (wire)

- 3D printing or additive manufacturing (AM) has shown promise in realizing a new design space for aerospace applications.
- Each AM technique has a set of pros and cons associated with them.
- Instead of producing well known cast and wrought alloys with AM. We should look at AM as a new opportunity to produce materials that are currently difficult to create.
- For this study, SLM is used due to it’s superior dimensional accuracy.

High Temperature AM Compatible Materials

High Temperature Materials:

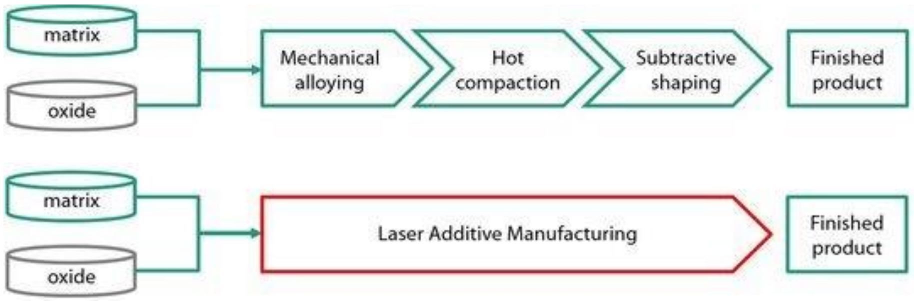
- Refractory metals
- Carbon-Carbon composites
- CMC's
- Ni-base superalloys
- **Dispersion strengthened (DS) alloys**



Inspired by Andy Jones. ODS alloy Development.

(DS) alloys offer higher temperature capabilities compared to Ni-base superalloys. However, it has been a challenge to produce DS alloys through conventional manufacturing methods.

Conventional Manufacturing vs AM



Can AM improve DS alloy manufacturability?

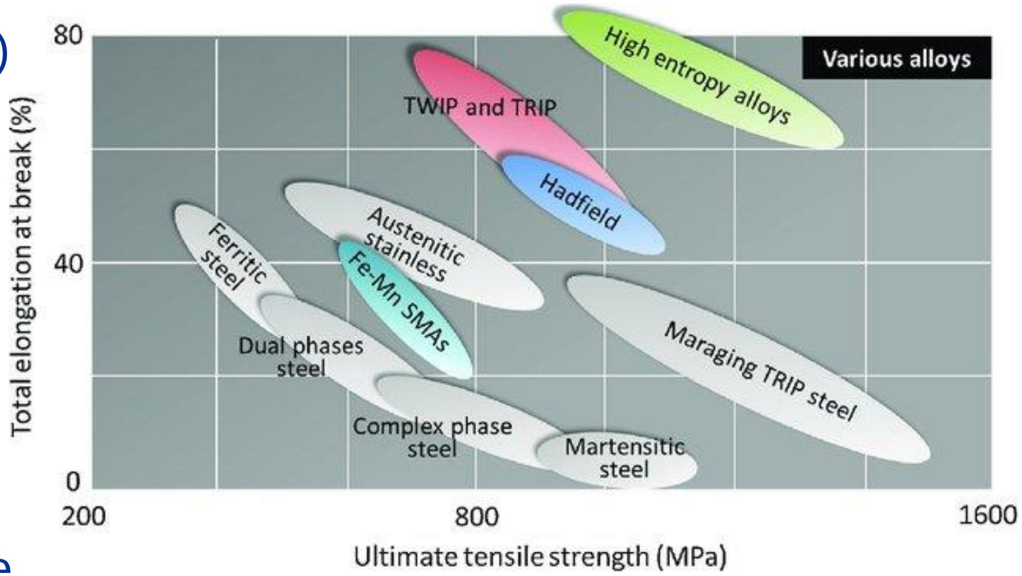
Advanced Materials and Manufacturing for High Temperature Applications

- Multi-principal element alloys (MPEAs) or "High-entropy alloys" overcome the strength - ductility trade off.

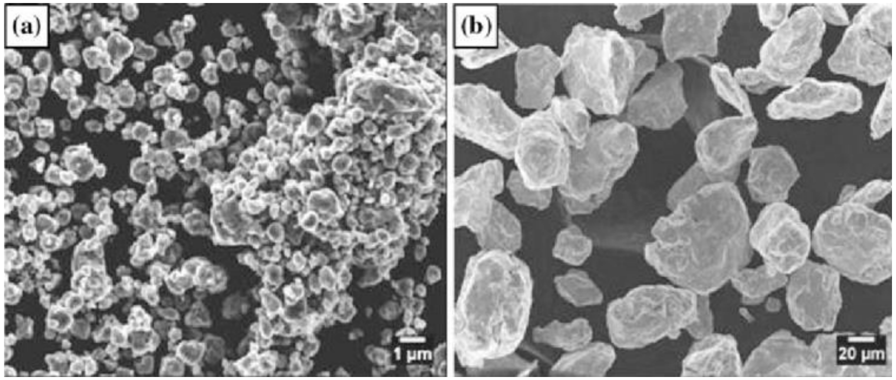
- The equiatomic NiCoCr medium-entropy alloy (MEA) is particularly of interest due to it's strong phase stability and mechanical properties.

- Single phase solid solution MPEAs are promising AM materials due to minor differences between their liquidus and solidus temperatures. This limits dendritic segregation, solidification cracking, and residual stress.

- **Can strengthening oxide particles be incorporated into the AM build without mechanical alloying?**



Chowdury *et al*. Materials Science and Engineering: Reports (2017)

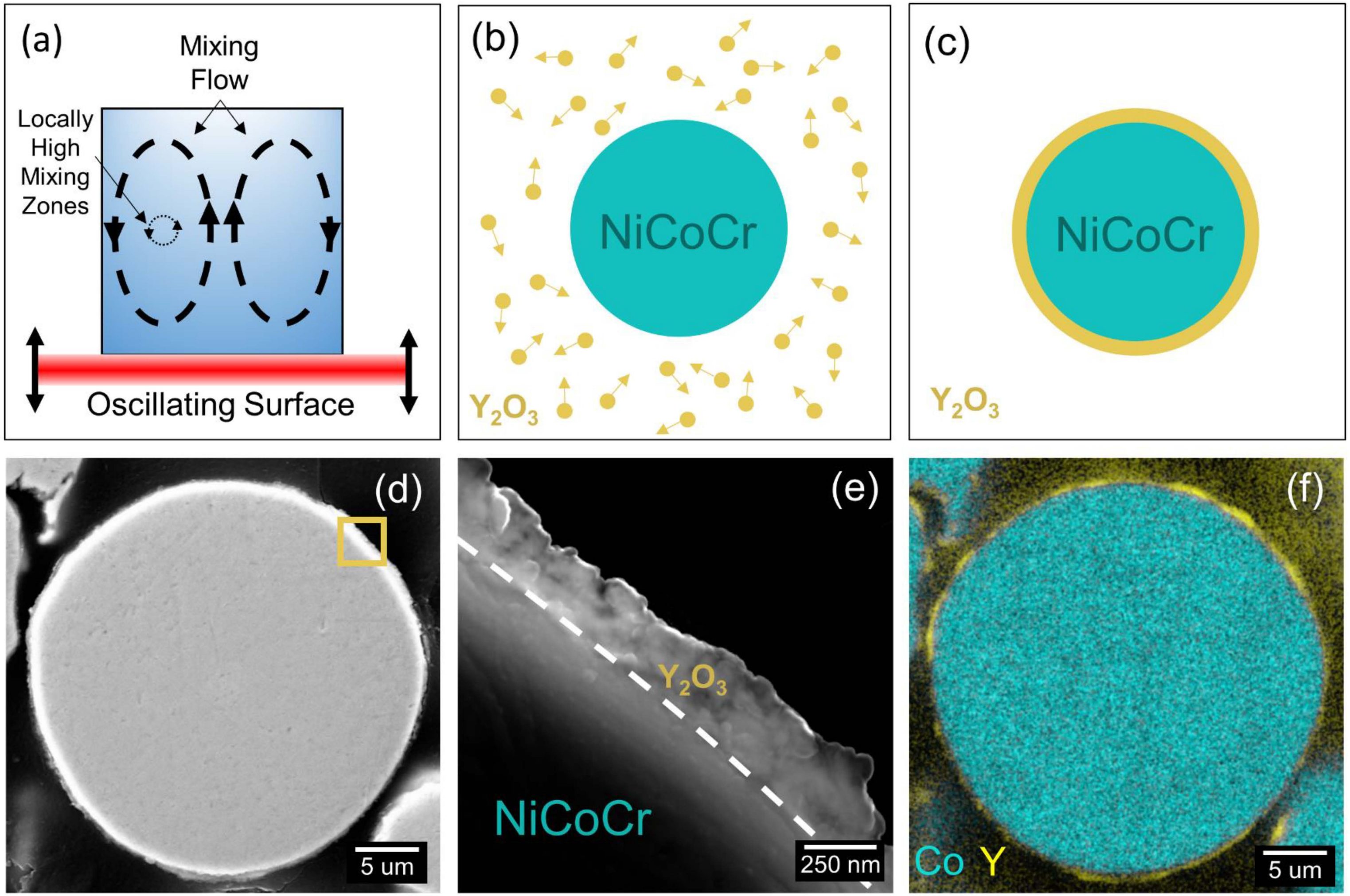


Oksiuta *et al*. Journal of Material Science (2010)

Methods

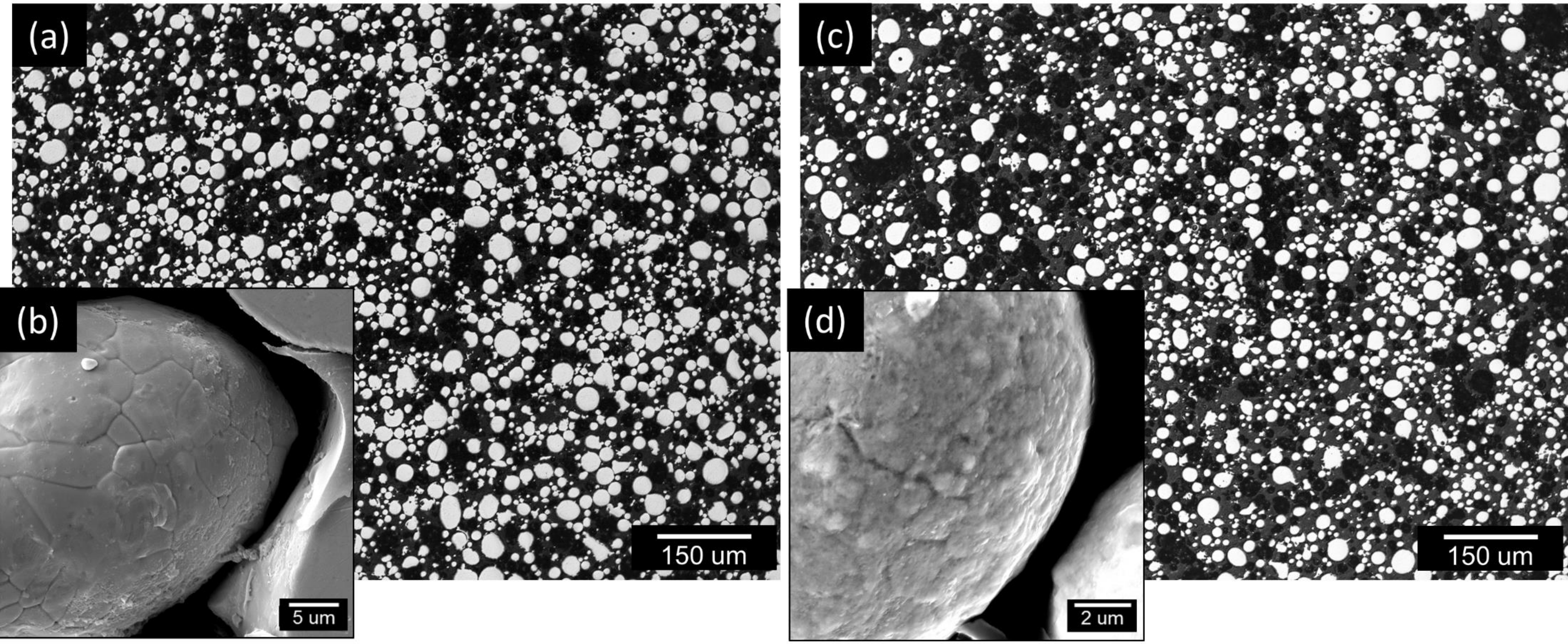
- Micron-scale (10-45um) NiCoCr powder was acquired from Praxair.
- Nano-scale (100-200nm) Yttria powder was acquired from American Elements.
- SLM Machine: EOS M100
- Powder Mixing: Resodyn LabRAM II
- Aim of study
  - Leverage SLM to produce dispersion strengthened multi-principal element alloys.
  - Determine optimal SLM laser parameters for both baseline (V-MEA) and dispersion strengthened (DS-MEA) builds.
  - Produce 99.9% dense vertical test specimen for microstructural and mechanical analysis using both V-MEA DS-MEA NiCoCr.
  - Explore heat treatment effects on mechanical performance
  - Produce a high temperature capable 3D printed combustor dome.

Novel Powder Coating Technique



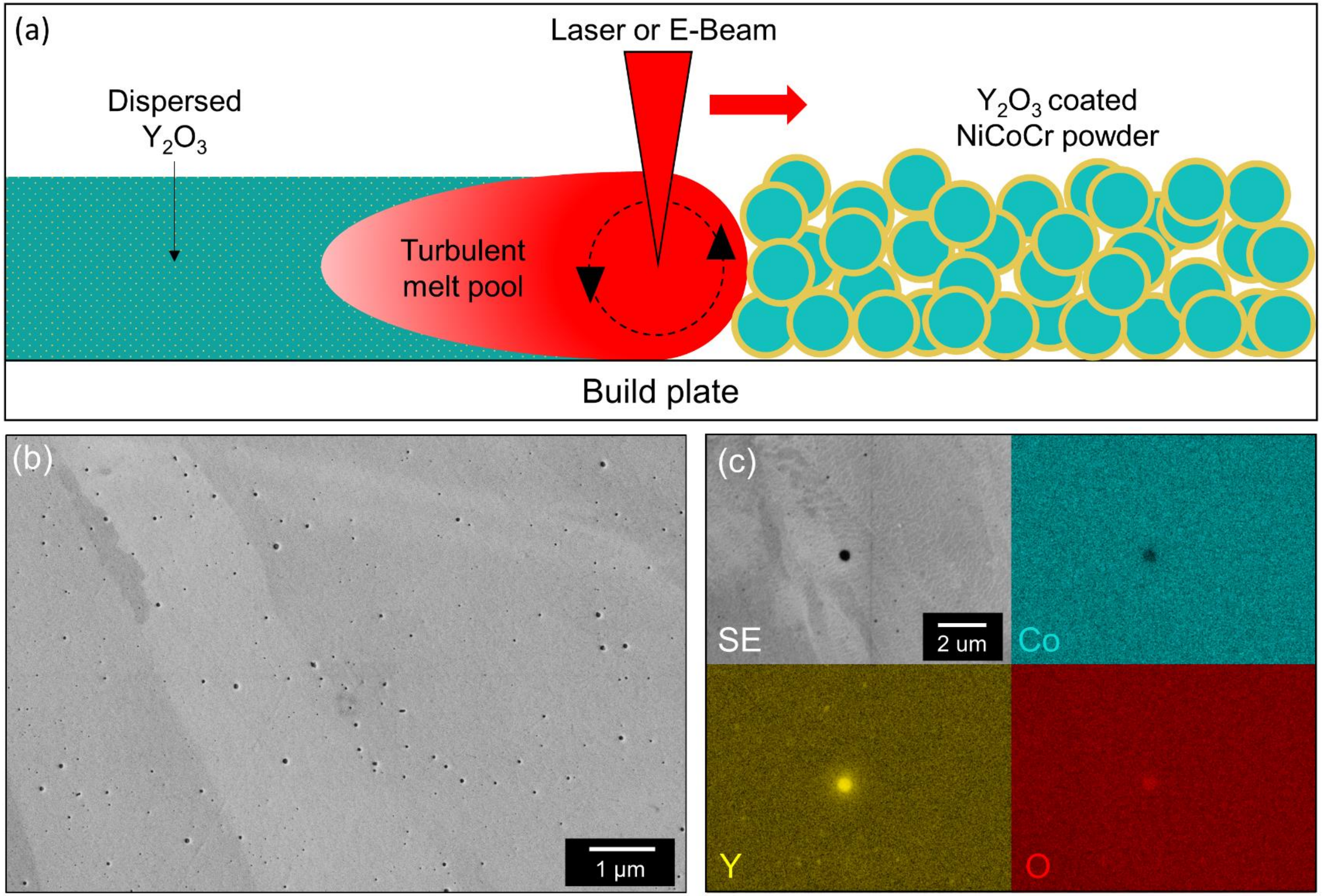
New high energy mixing technique successfully coats NiCoCr powder with 1 wt.% Yttria.

Novel Powder Coating Technique



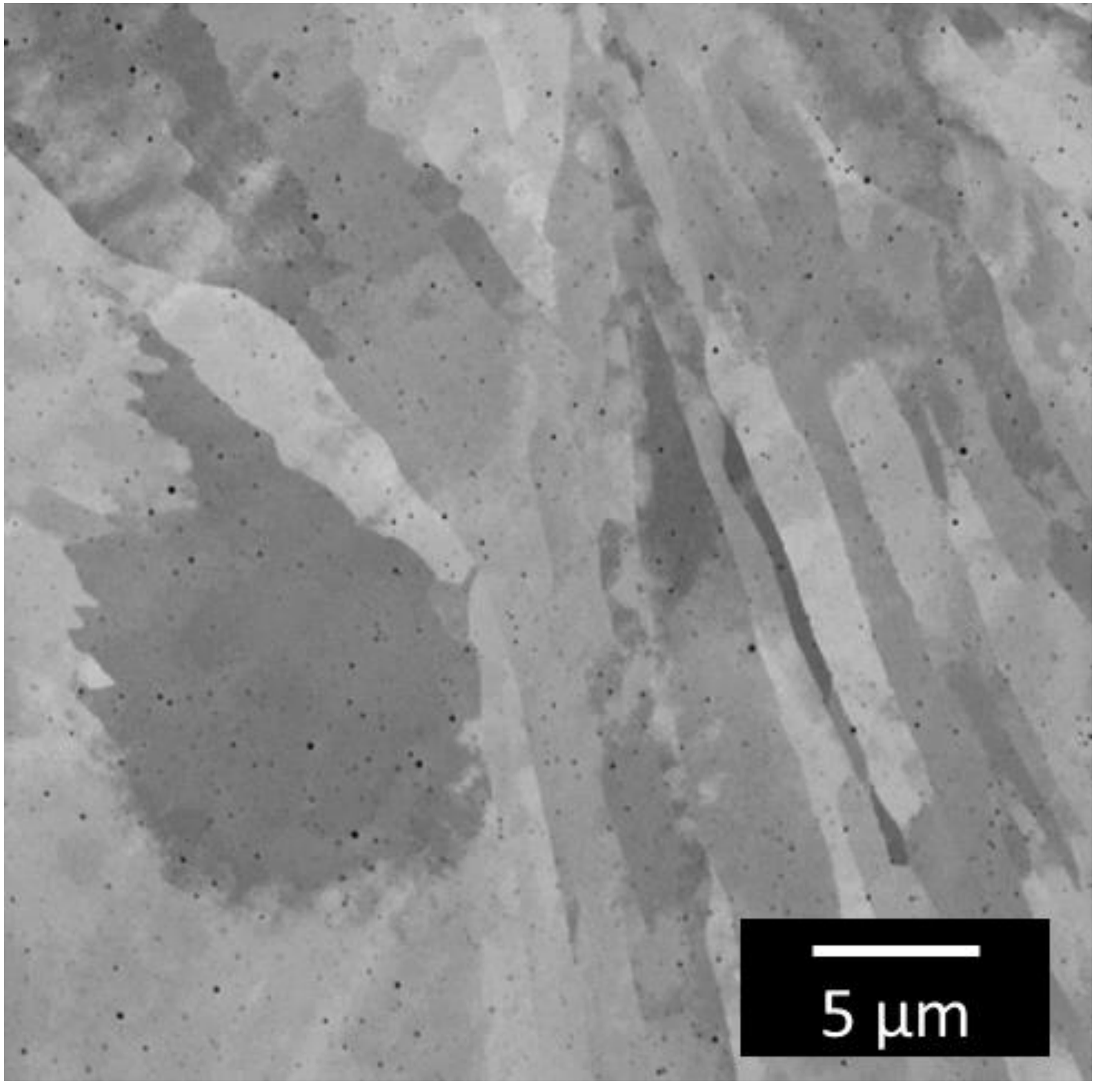
- The resonant mixing technique did not deform the NiCoCr powders.
- Both uncoated and coated powders qualitatively passed the Hall flow test.

Leveraging SLM to Produce Dispersion Strengthened Alloys



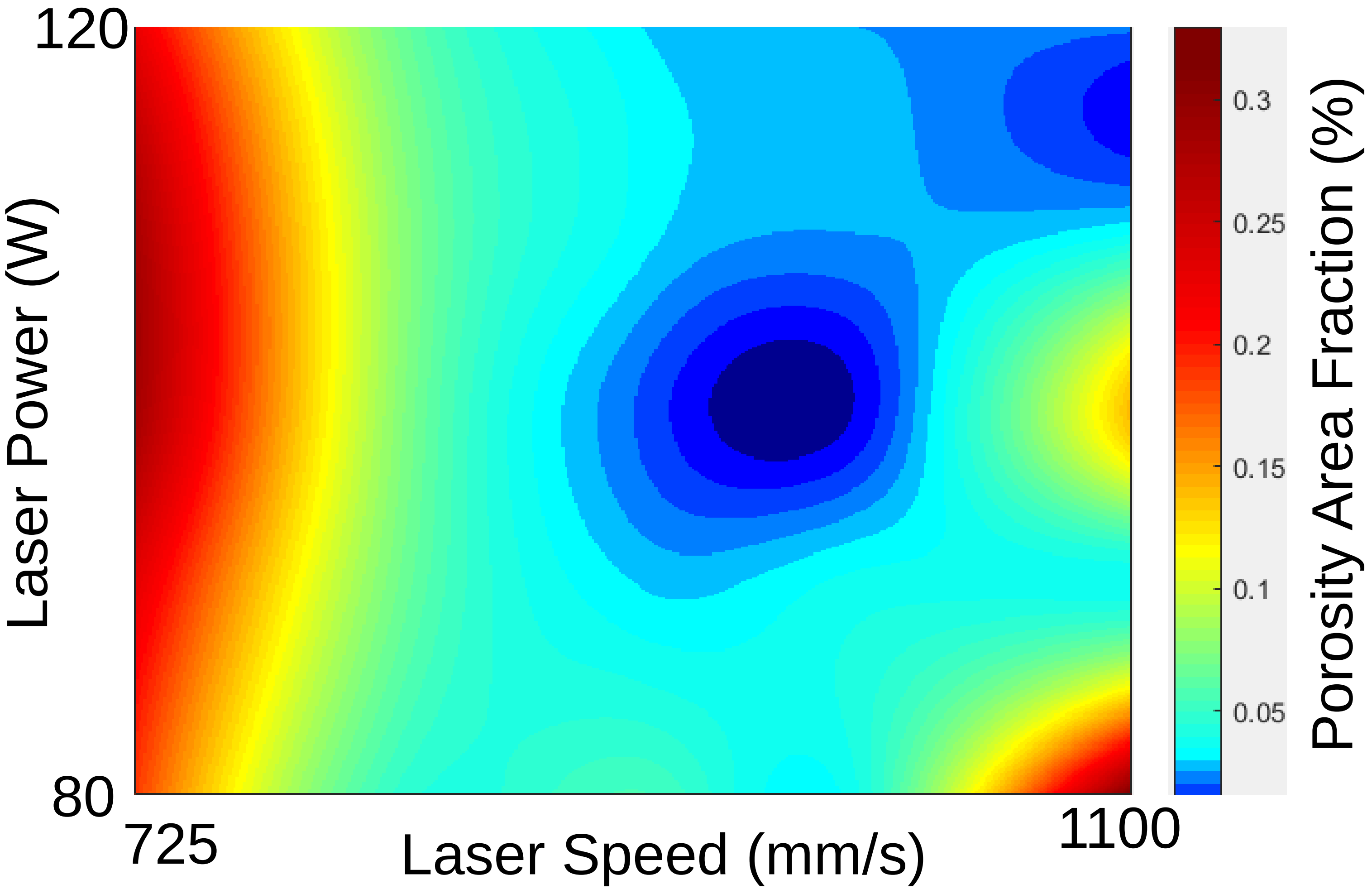
SLM successfully disperses the nano-scale Yttria particles throughout the AM build

DS-MEA Microstructure

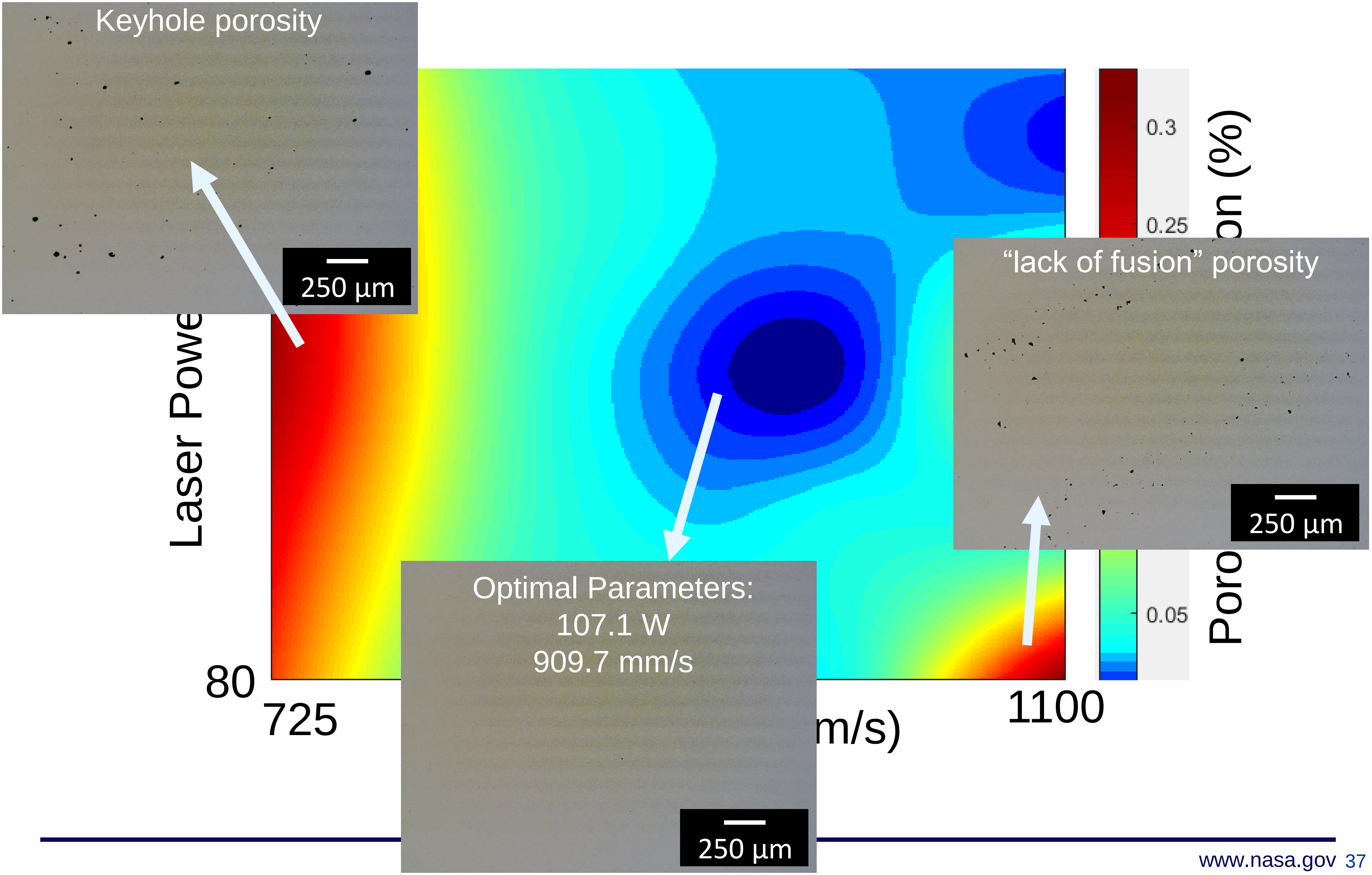


Nano-scale Y_2O_3 particles are randomly dispersed throughout microstructure.

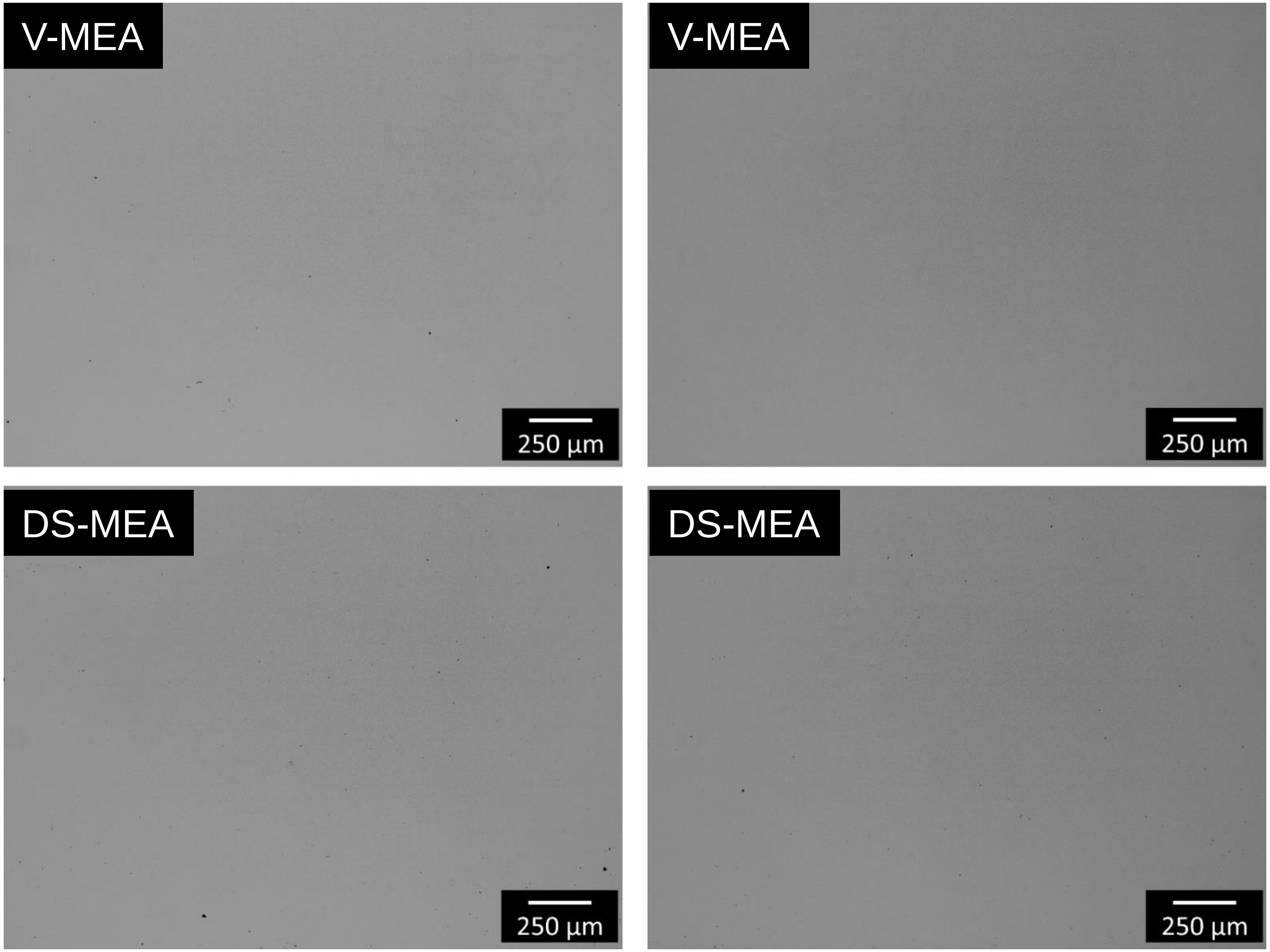
SLM Laser Parameters V-MEA



SLM Laser Parameters V-MEA

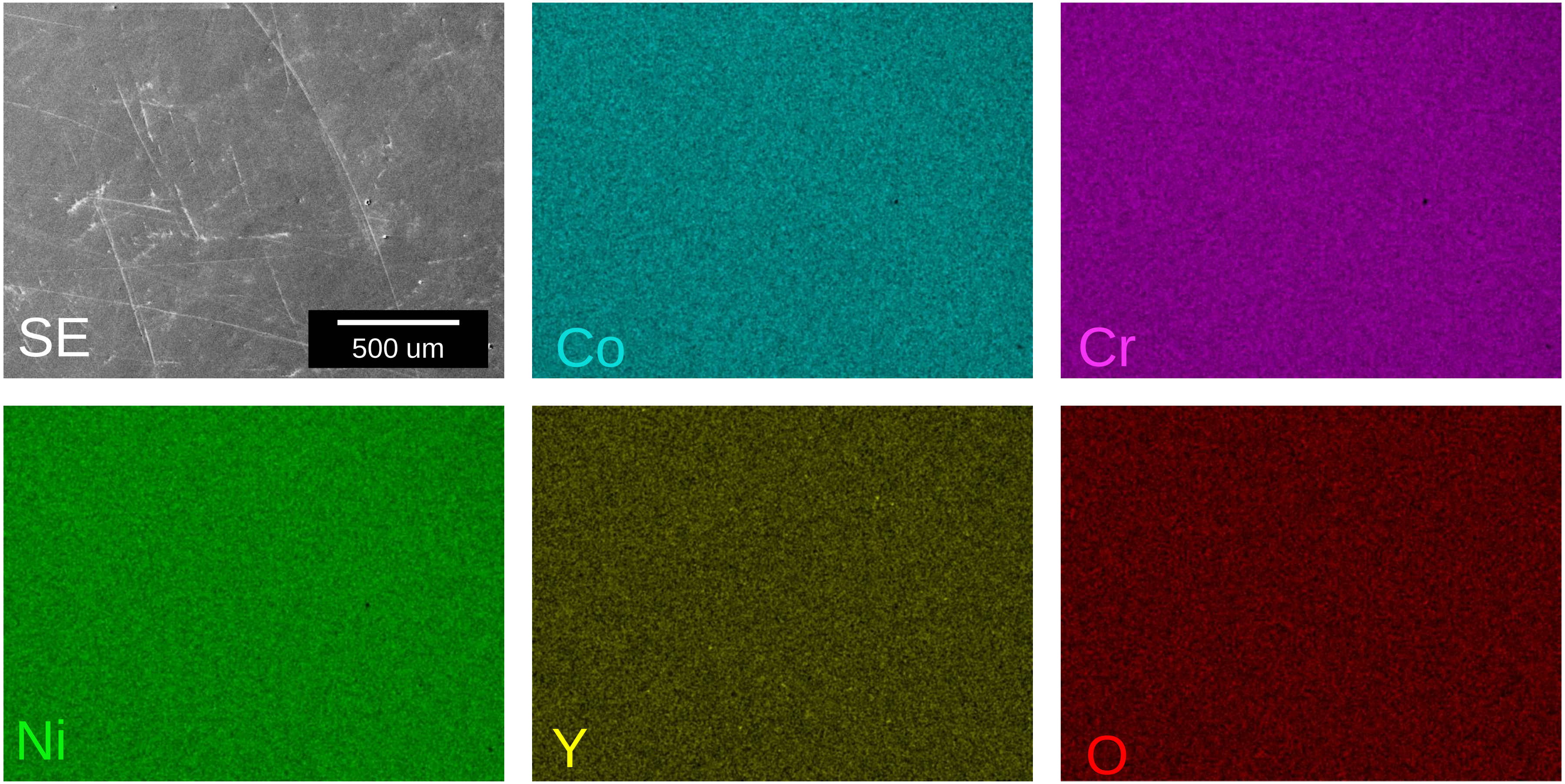


MEA Microstructures - Porosity



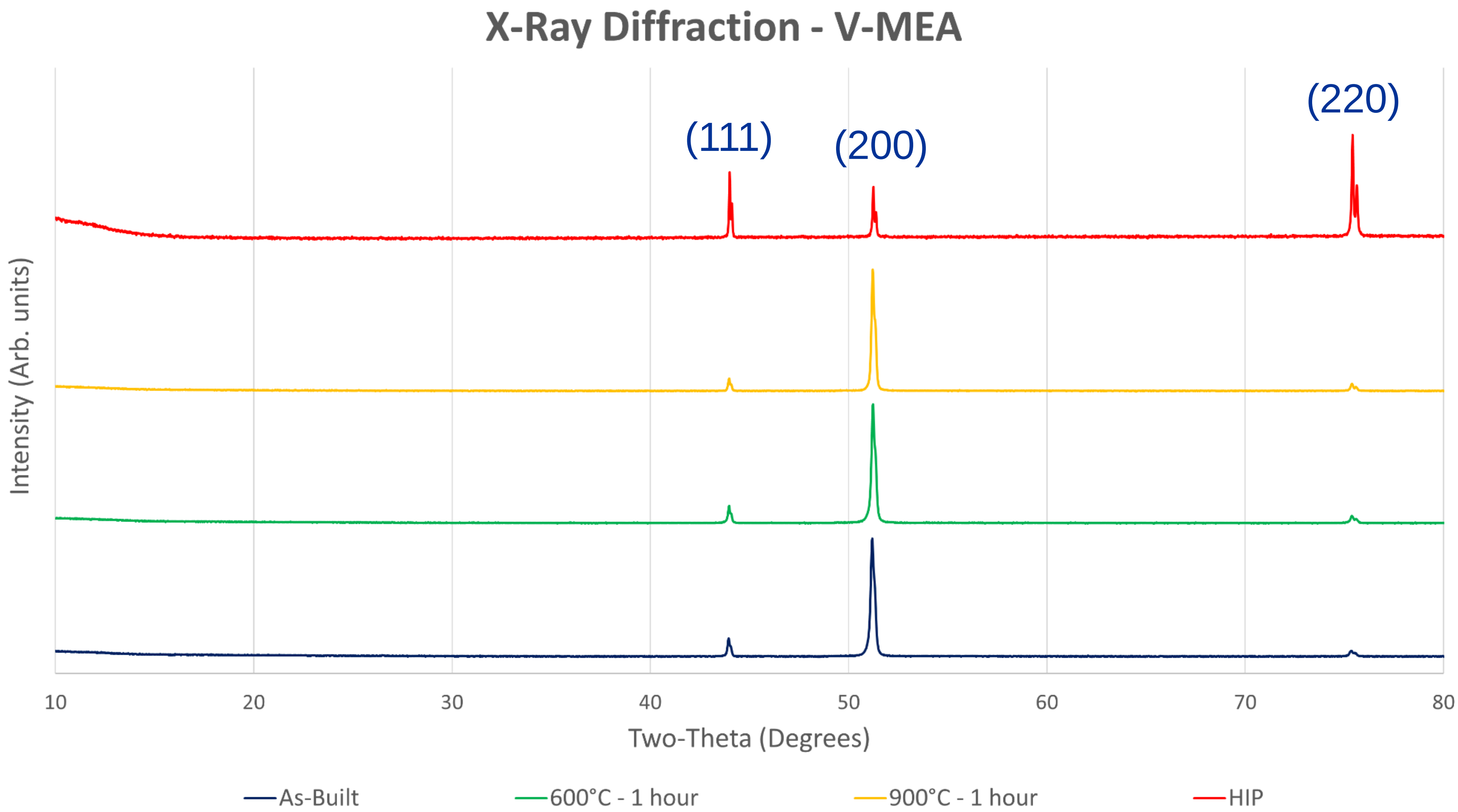
99.9% dense parts were successfully built for both the V-MEA and DS-MEA powder lots.

EDS – DS-MEA Microstructure



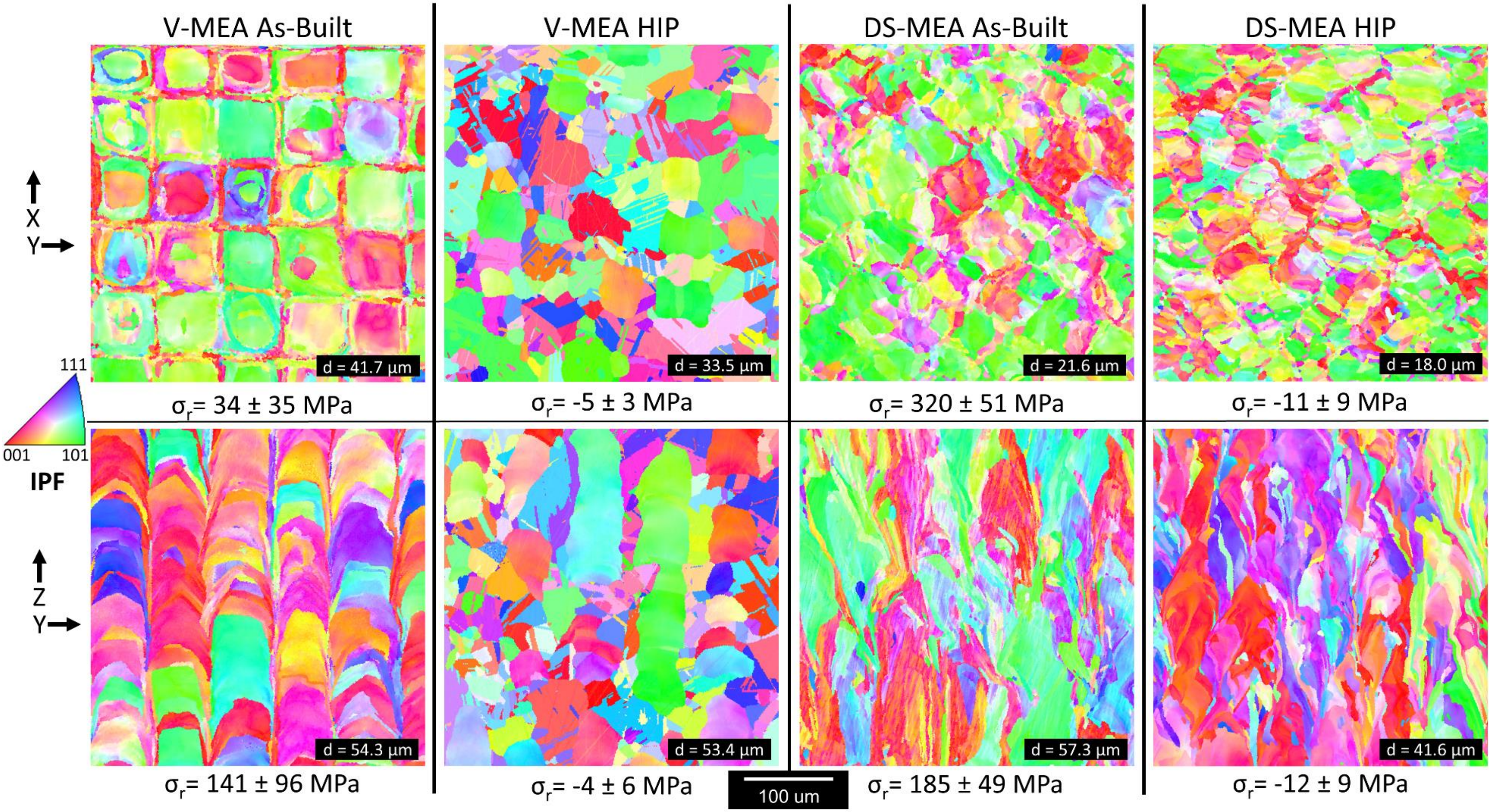
- Large (>20um) Y_2O_3 particles are not present in AM builds
- NiCoCr matrix remained a random solid solution during SLM process.

Heat Treatment effect on solid solution stability



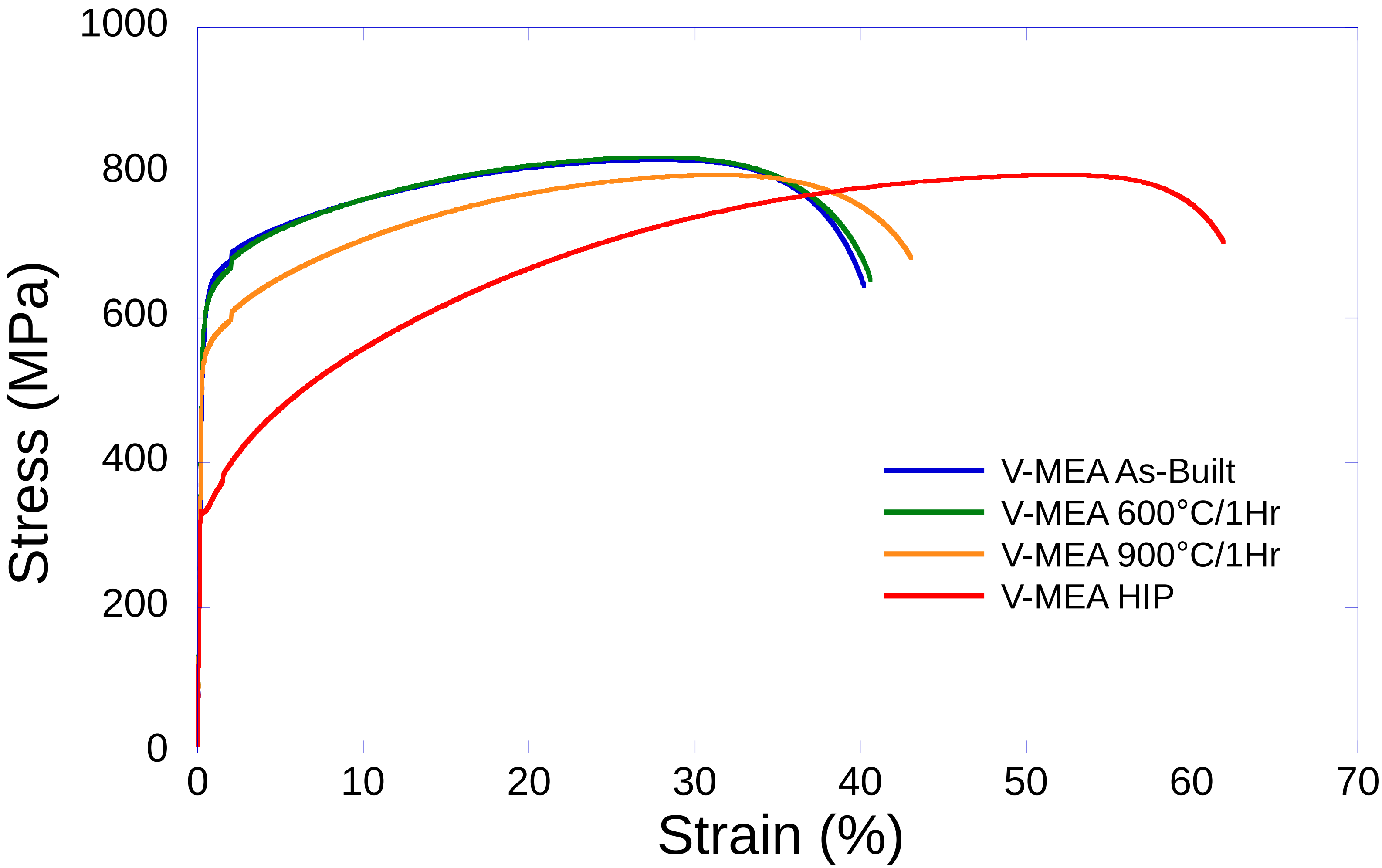
No intermetallic phases present after anneal or HIP steps.

Microstructure Analysis



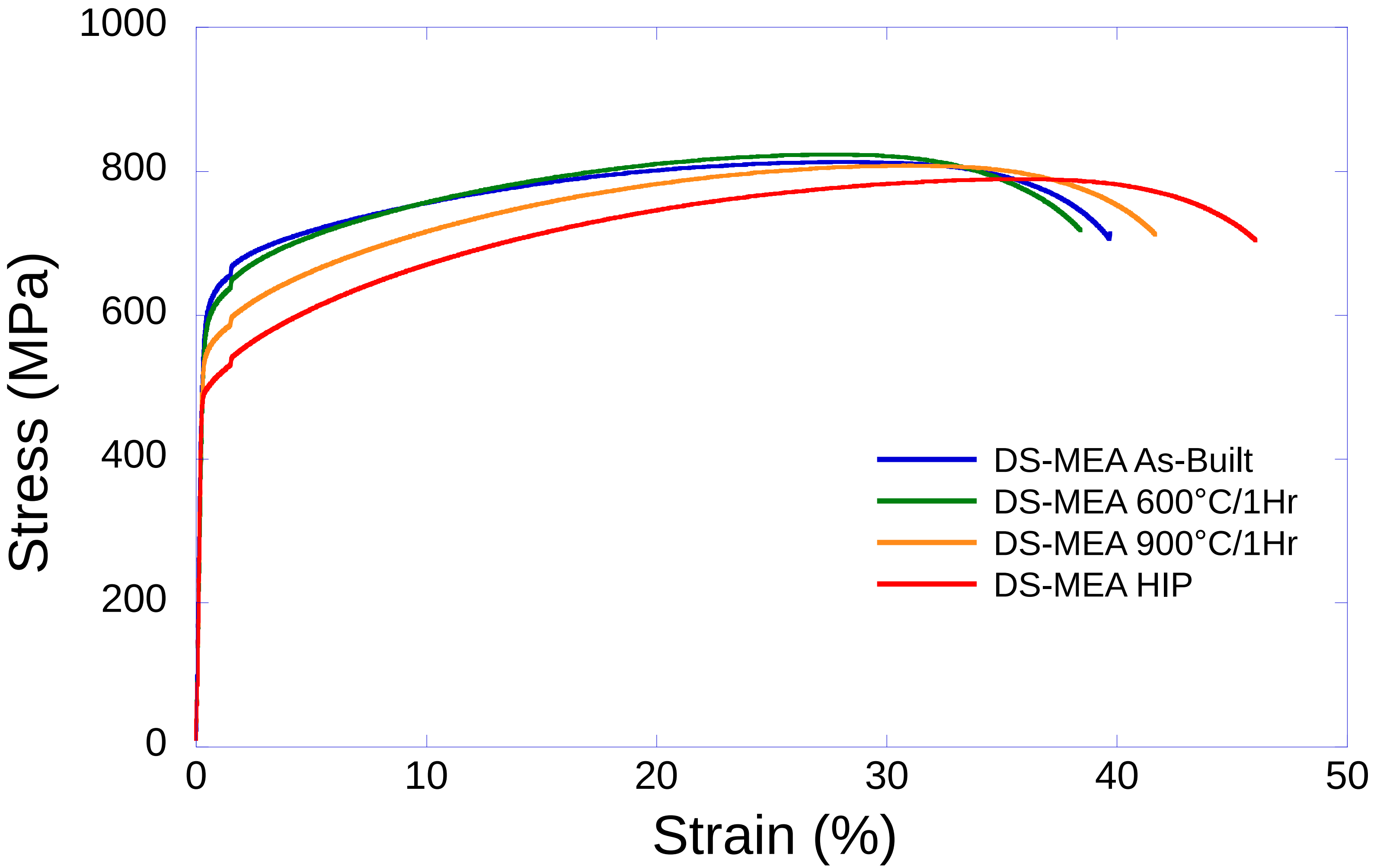
- Yttria particles have pinned the grain boundaries in the MEA-ODS builds
- The HIP cycle successfully removed residual stresses in both the V-MEA and DS-MEA builds

Mechanical Tests V-MEA



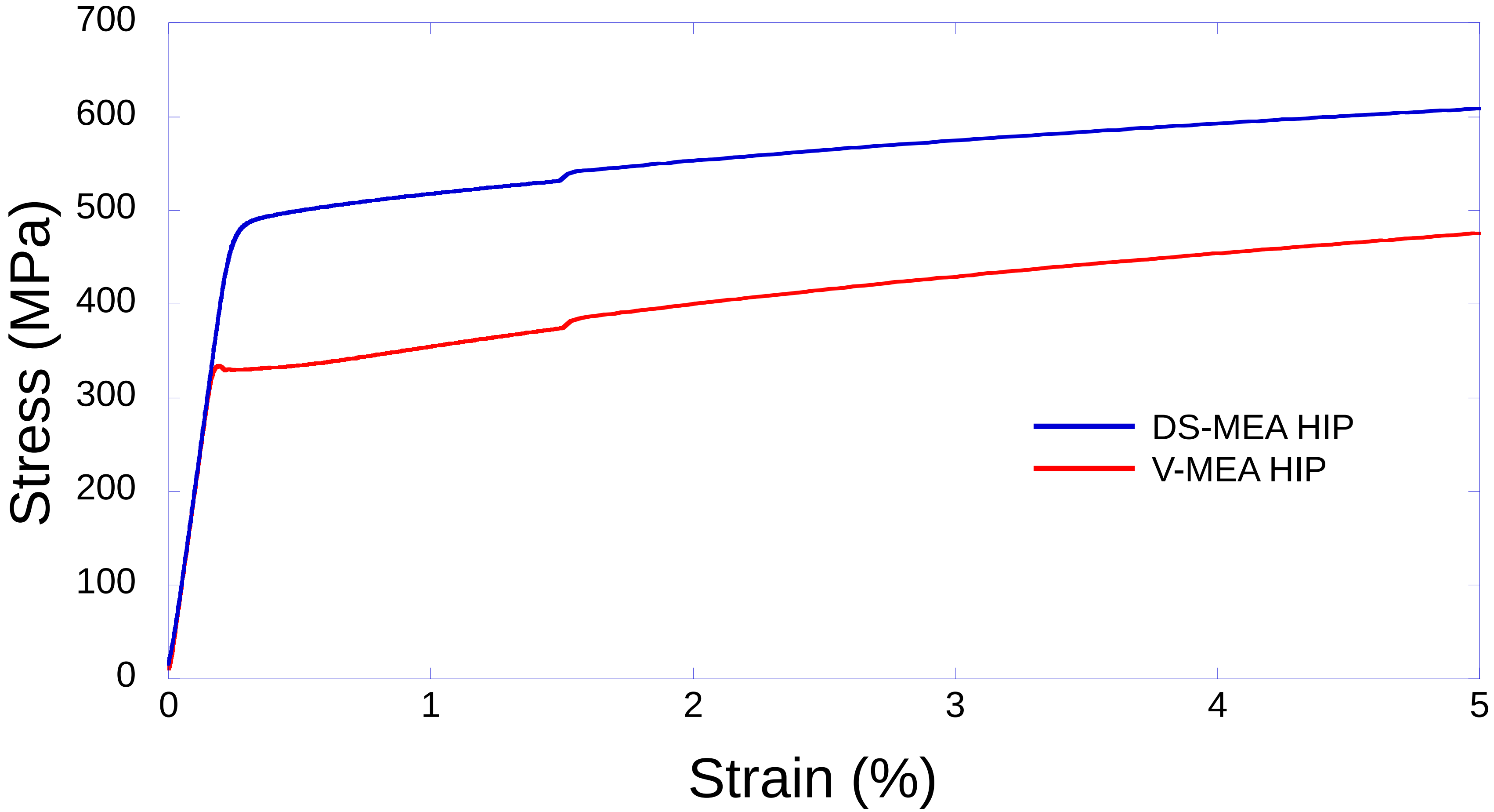
Significant reduction in yield strength associated with experience of extreme temperature for V-HEA specimen

Mechanical Tests DS-MEA



DS-MEA specimen much less sensitive to extreme environments.

Yield Strength Curve Comparison



DS-MEA specimen exhibited 50% improvement in yield strength over V-MEA after HIP.

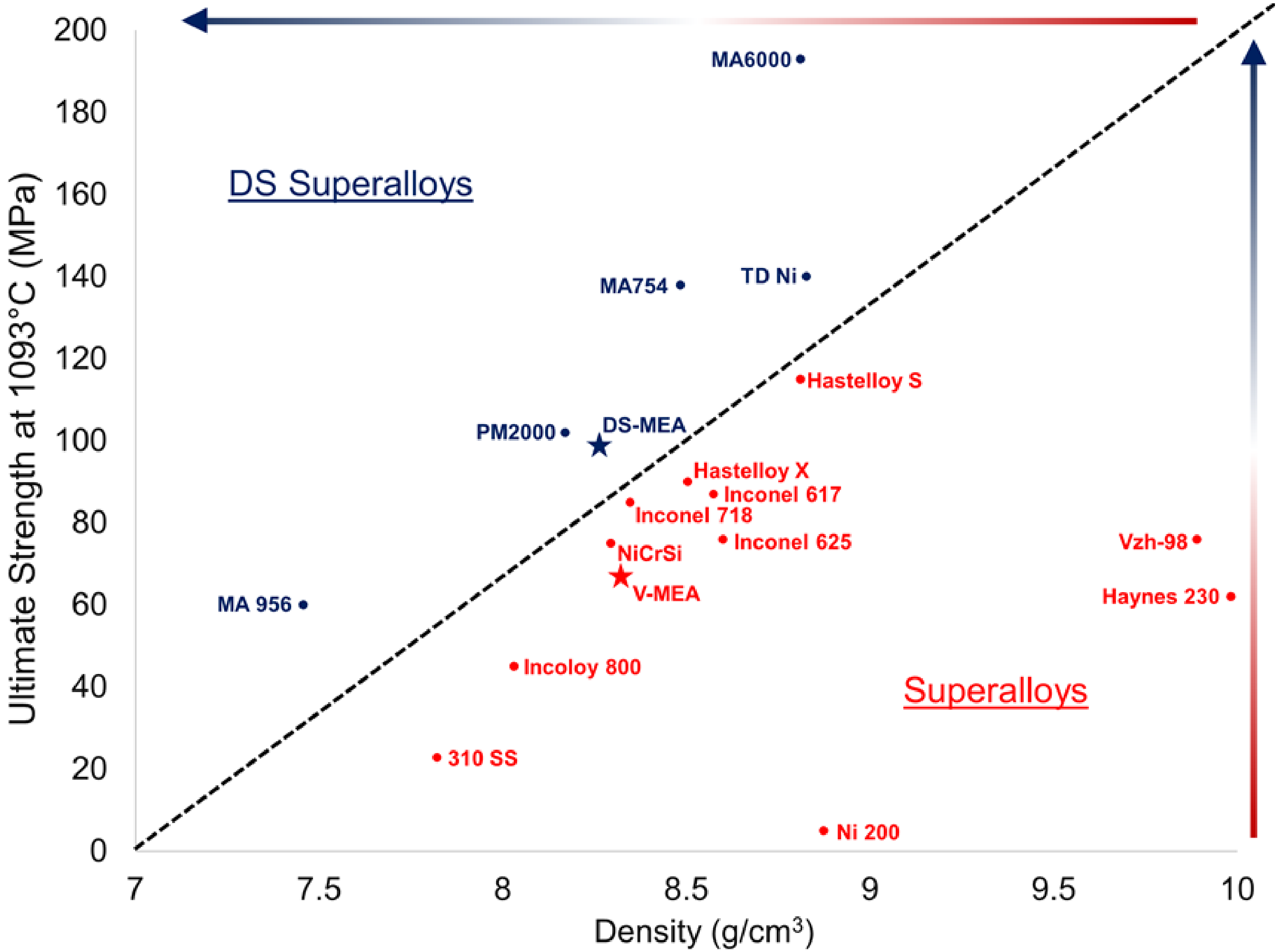
1093°C Mechanical Properties

Alloy	Yield Strength (MPa)	Ultimate Strength (MPa)	Elongation (%)	Reduction of Area (%)
V-MEA As-Built	52	68	6.5	7
V-MEA HIP	46	68	8	8.5
DS-MEA As-Built	71	96	20	22
DS-MEA HIP	66	90	19	27

DS-MEA alloys possessed significantly improved high temperature properties over the baseline V-MEA samples.

This includes a >40% increase in strength and a 3x improvement in ductility

Tensile Strength vs Density Comparison



Scatter plot confirms the successful production of a DS alloy using AM

Conclusions

- SLM can be leverage to economically produce dispersion strengthened alloys that until now had been cost prohibitive.
- Multi-principle elements alloys show promise as AM compatible materials
- The incorporation of oxides into the MEA produced a more thermally stable microstructure.
- The DS alloy exhibited improved mechanical properties over the baseline alloy.
- We believe this new manufacturing technique combined with MPEAs opens up a new alloy design space for future high temperature alloys

Acknowledgments

Questions?

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