Microstructural and Mechanical Characterization of a *Dispersion Strengthened* Medium Entropy Alloy Produced Using Selective Laser Melting

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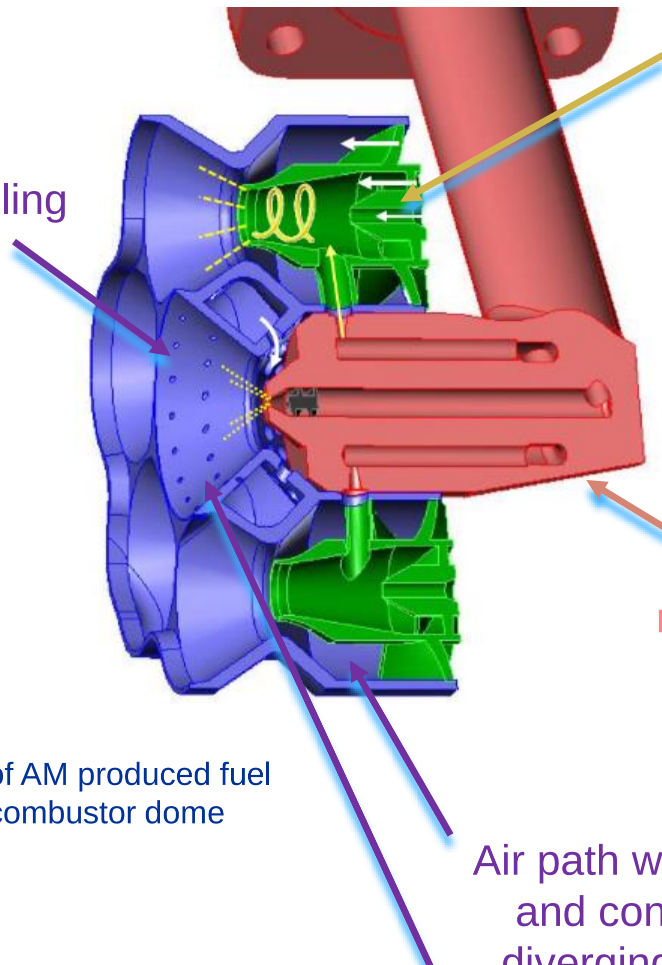
Background – NASA Application

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

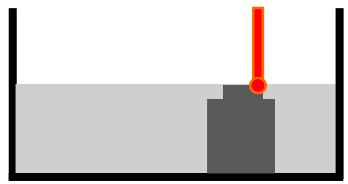
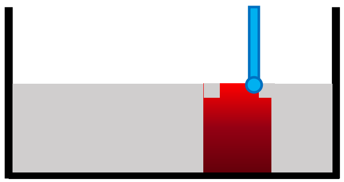
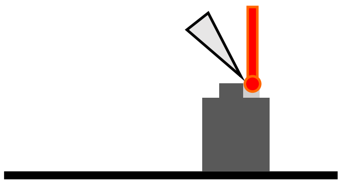
Proposed Solution: Develop a high ductility, high temperature material for an additively-manufactured (AM) combustor fuel nozzle and dome for supersonic aircraft (>1093°C (2000°F) operating temperature).

- 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

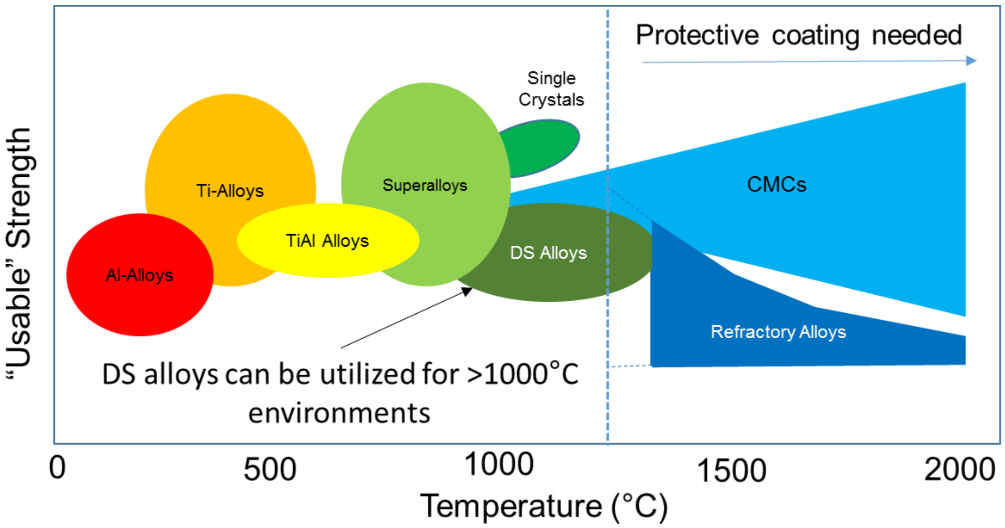
Process	Selective Laser Melting (SLM) 	Electron Beam Melting (EBM) 	Direct Energy Deposition (DED) 
Energy Source	Laser	E-Beam	Laser or E-Beam
Powder Bed	Yes	Yes	No
Power (W or kV)	50-1000 W	**30-60kV**	100-2000 W
Max Build Size (mm)	500 x 280 x 320	500 x 280 x 320	**2000 x 1500 x 750**
Material	Metallic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

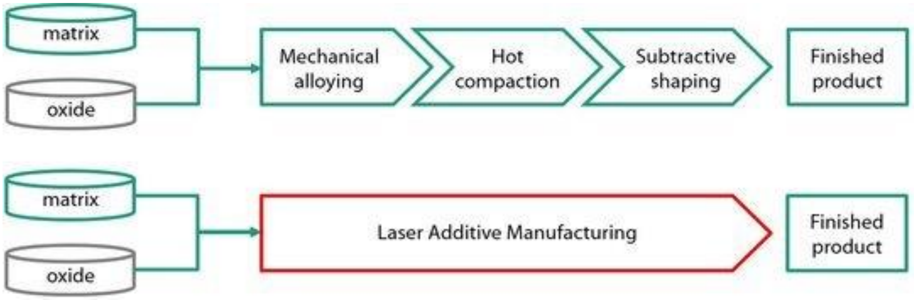
- Refractory metals
- Carbon-Carbon composites
- CMC's
- Ni-base superalloys
- **Dispersion strengthened (DS) alloys**



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

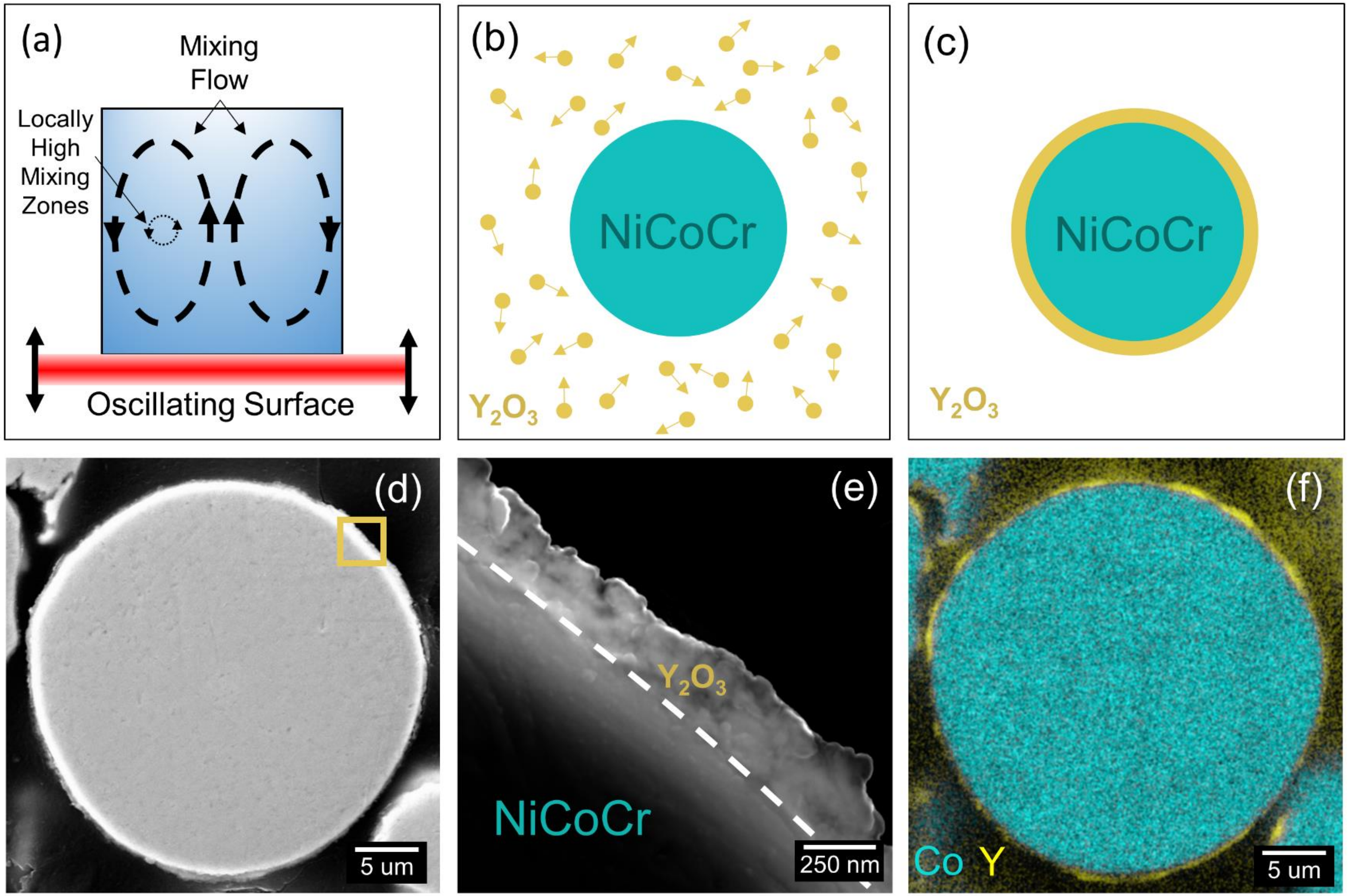


Can AM improve DS alloy manufacturability?

Methods

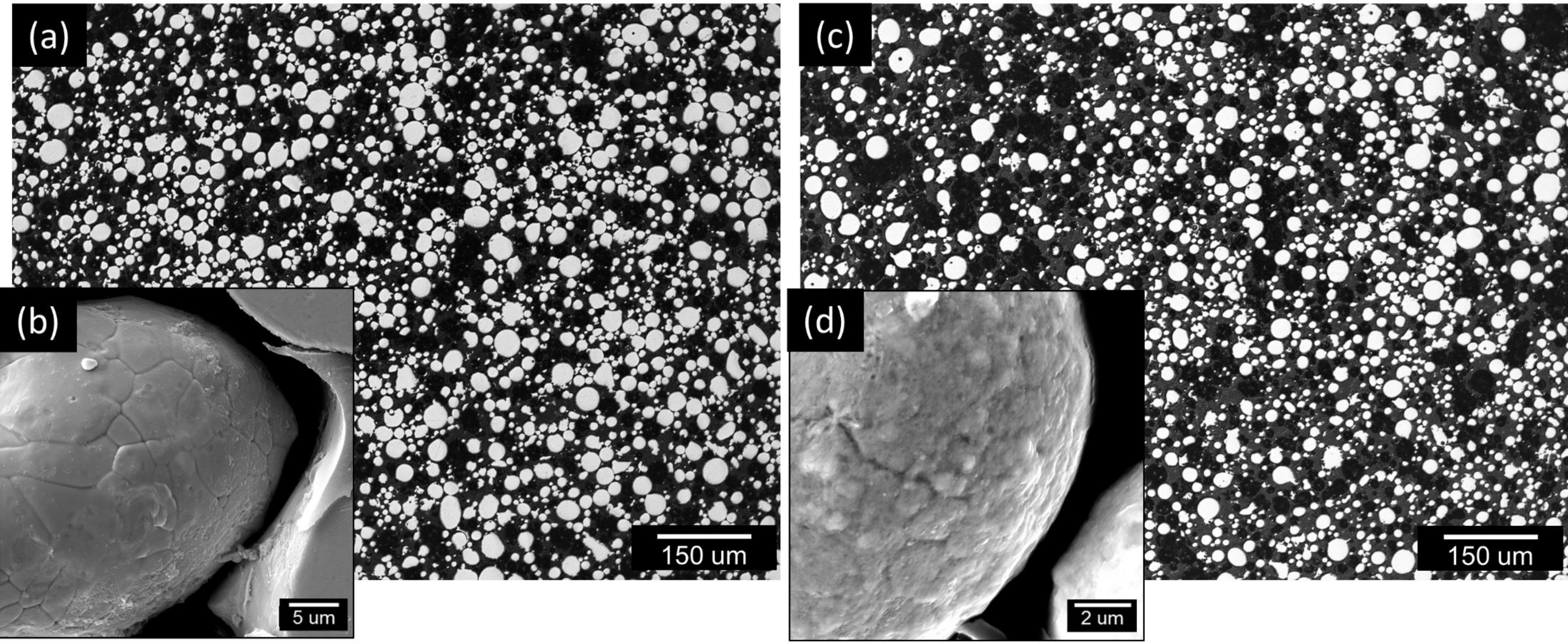
- Micron-scale (10-45um) NiCoCr powder was acquired from Praxair.
- Nano-scale (100-200nm) Yttria powder was acquired from American Elements.
- SLM Machine: EOS M100
- Powder Mixing: Resodyn LabRAM II
- Aim of study
  - Leverage SLM to produce dispersion strengthened multi-principal element alloys.
  - 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



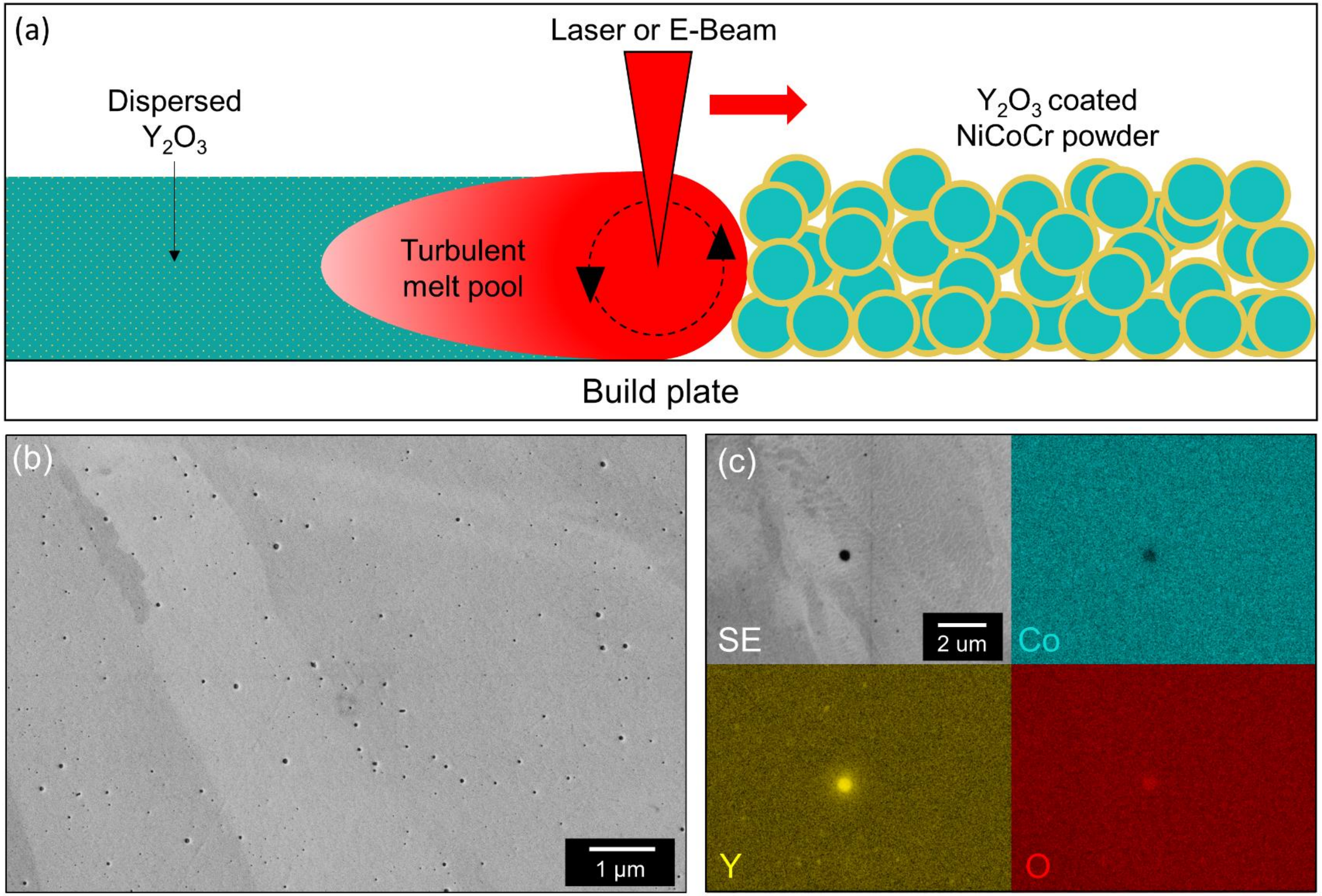
New high energy mixing technique successfully coats NiCoCr powder with 1 wt.% Yttria.

Novel Powder Coating Technique



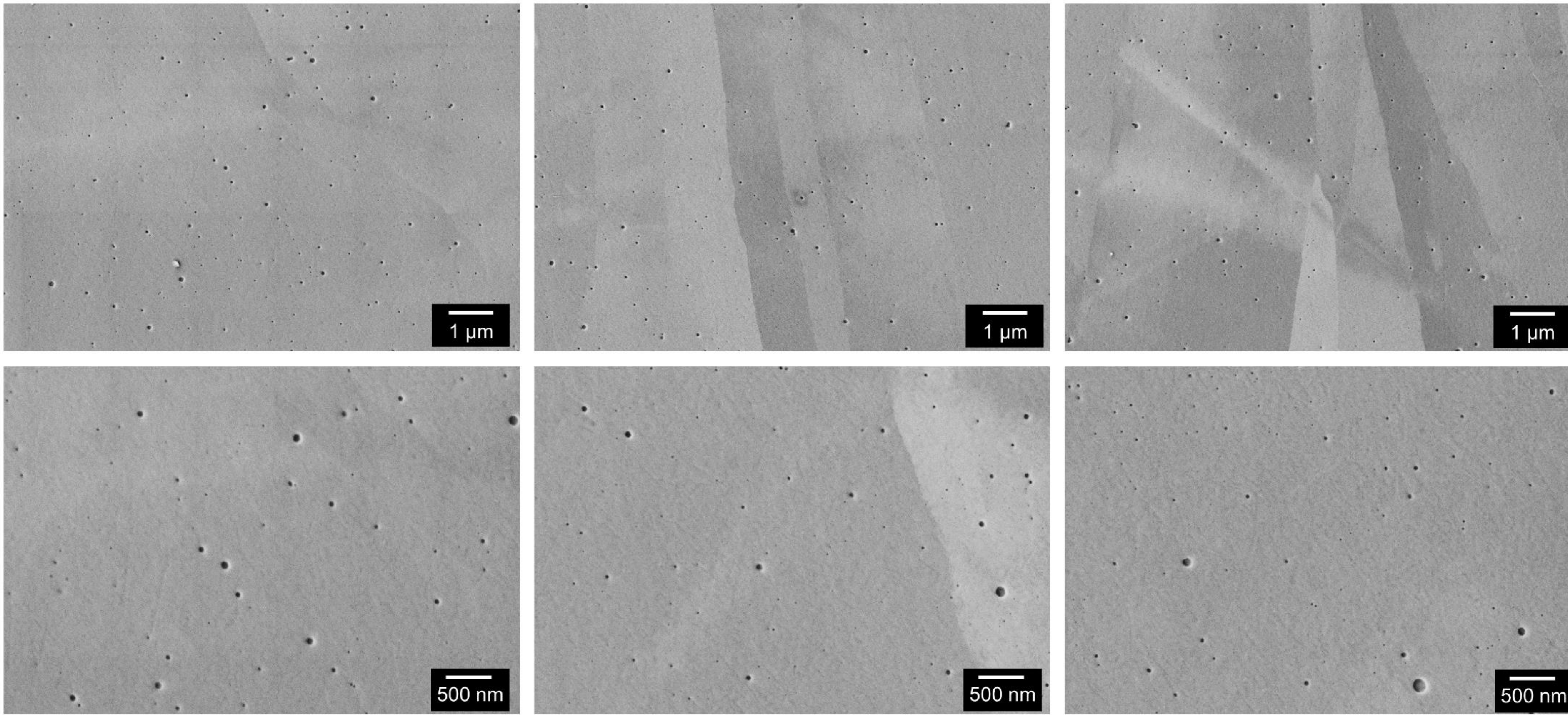
- The resonant mixing technique did not deform the NiCoCr powders.
- Both uncoated and coated powders qualitatively passed the Hall flow test.

Leveraging SLM to Produce Dispersion Strengthened Alloys



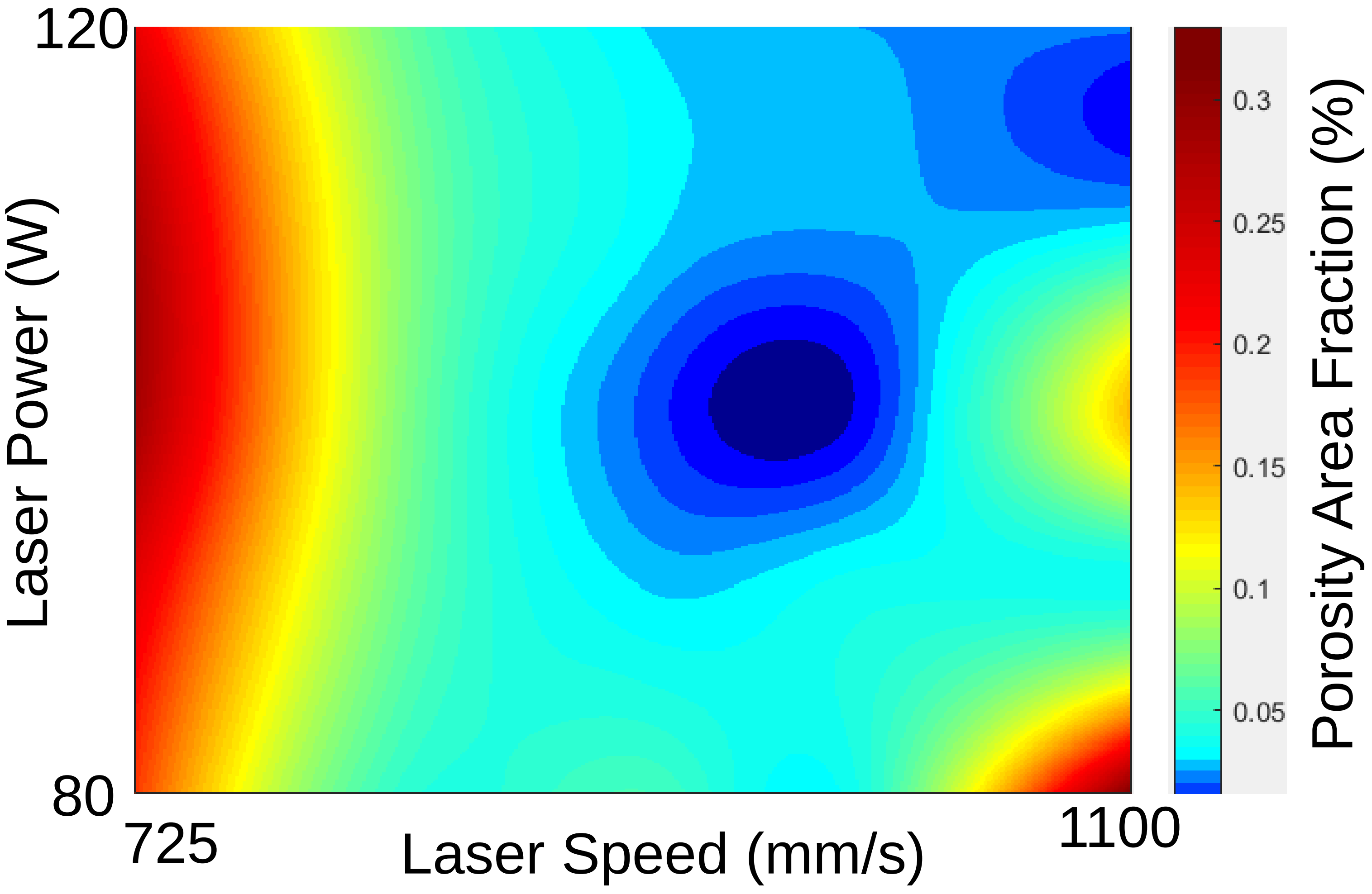
SLM successfully disperses the nano-scale Yttria particles throughout the AM build

DS-MEA Microstructure

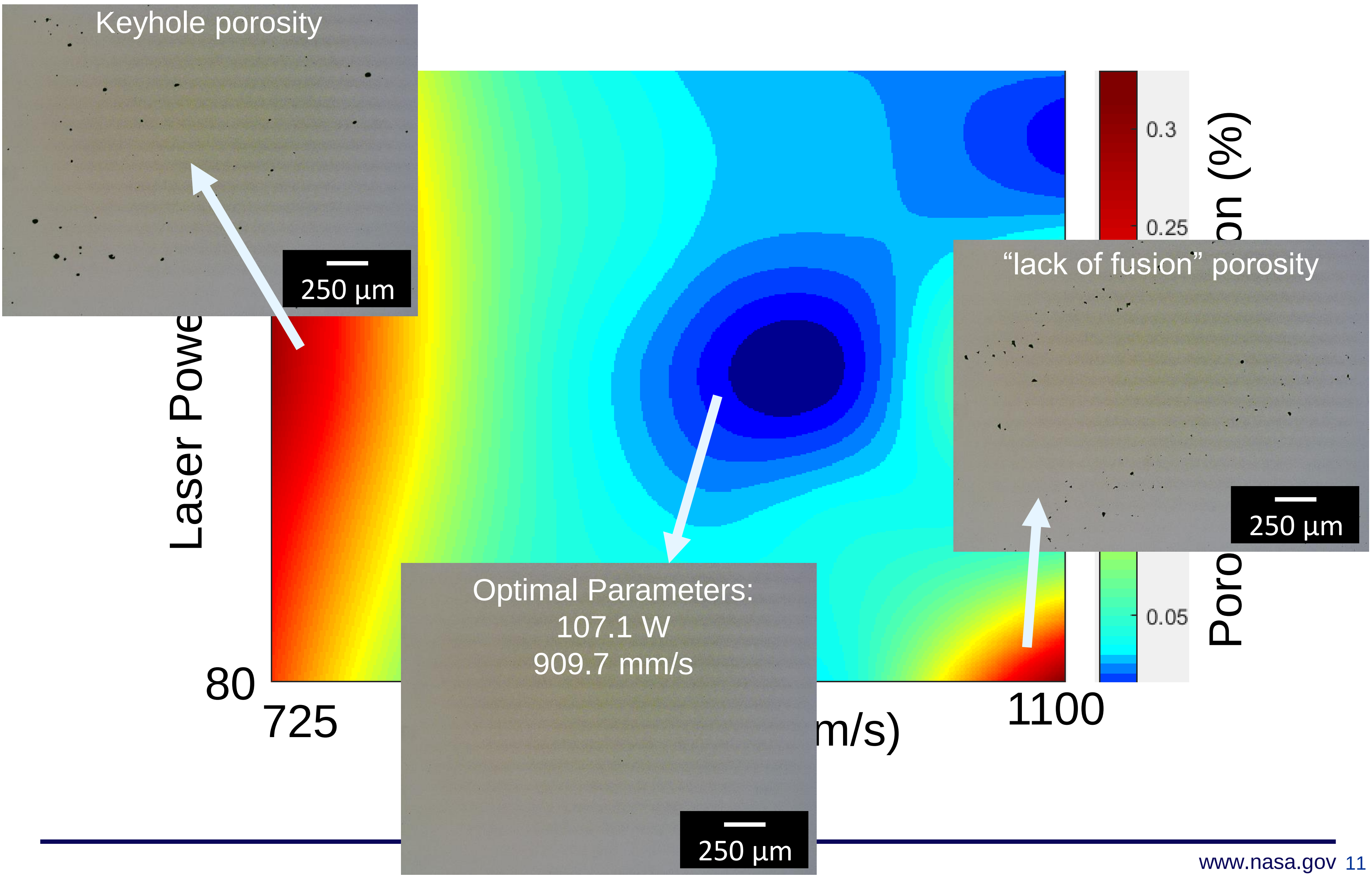


Nano-scale Y_2O_3 particles are randomly dispersed throughout microstructure.

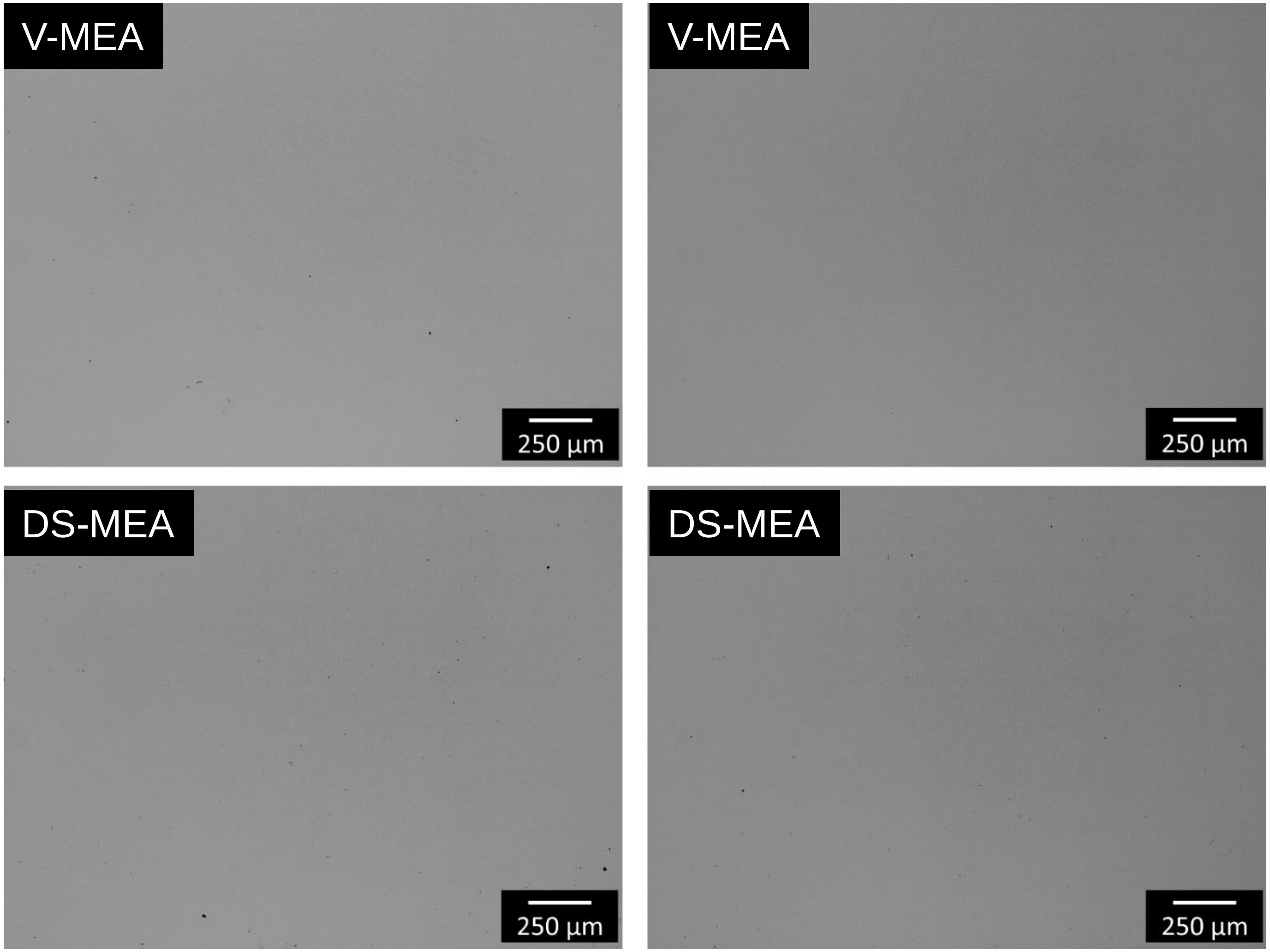
SLM Laser Parameters V-MEA



SLM Laser Parameters V-MEA

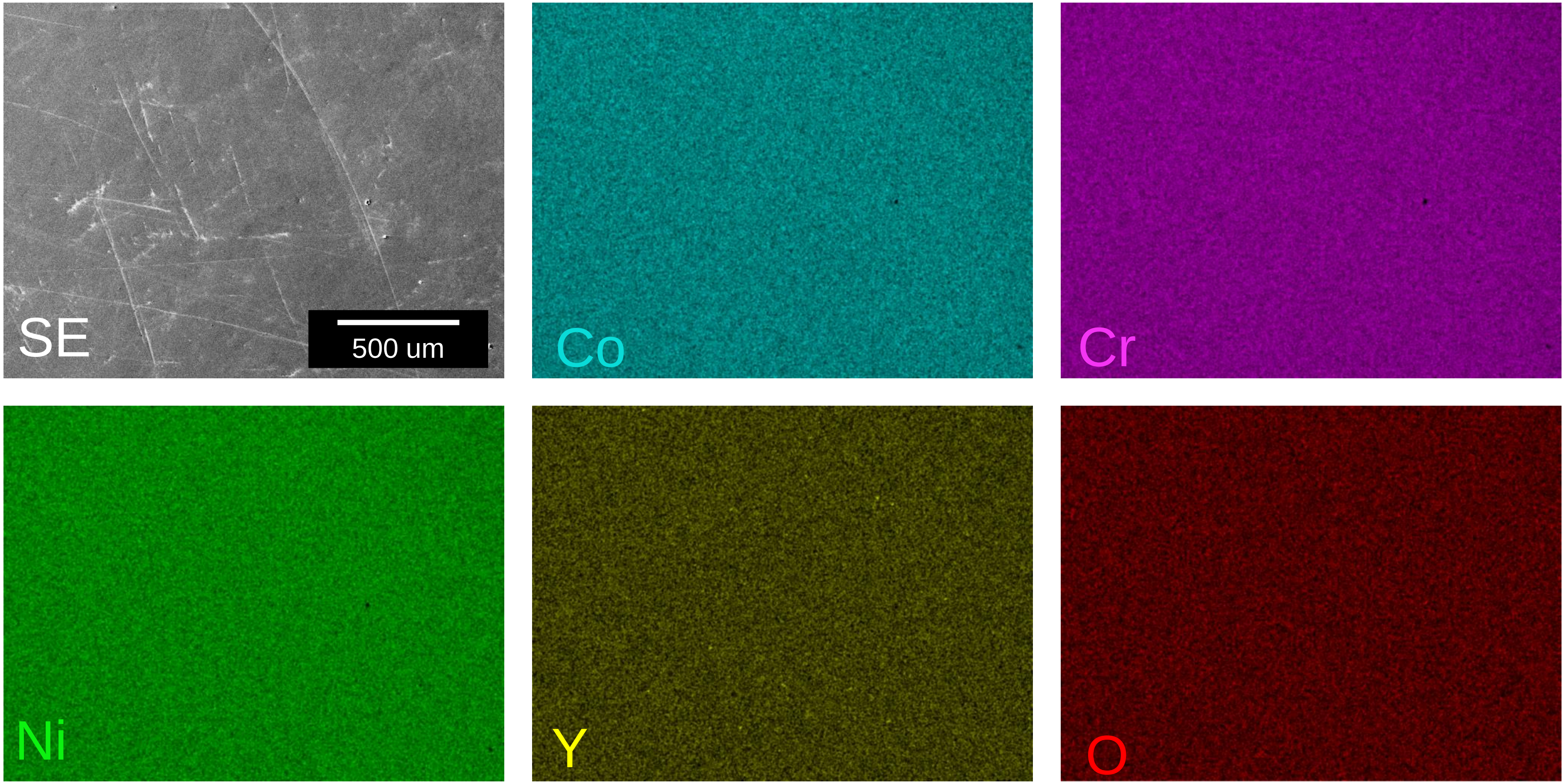


MEA Microstructures - Porosity



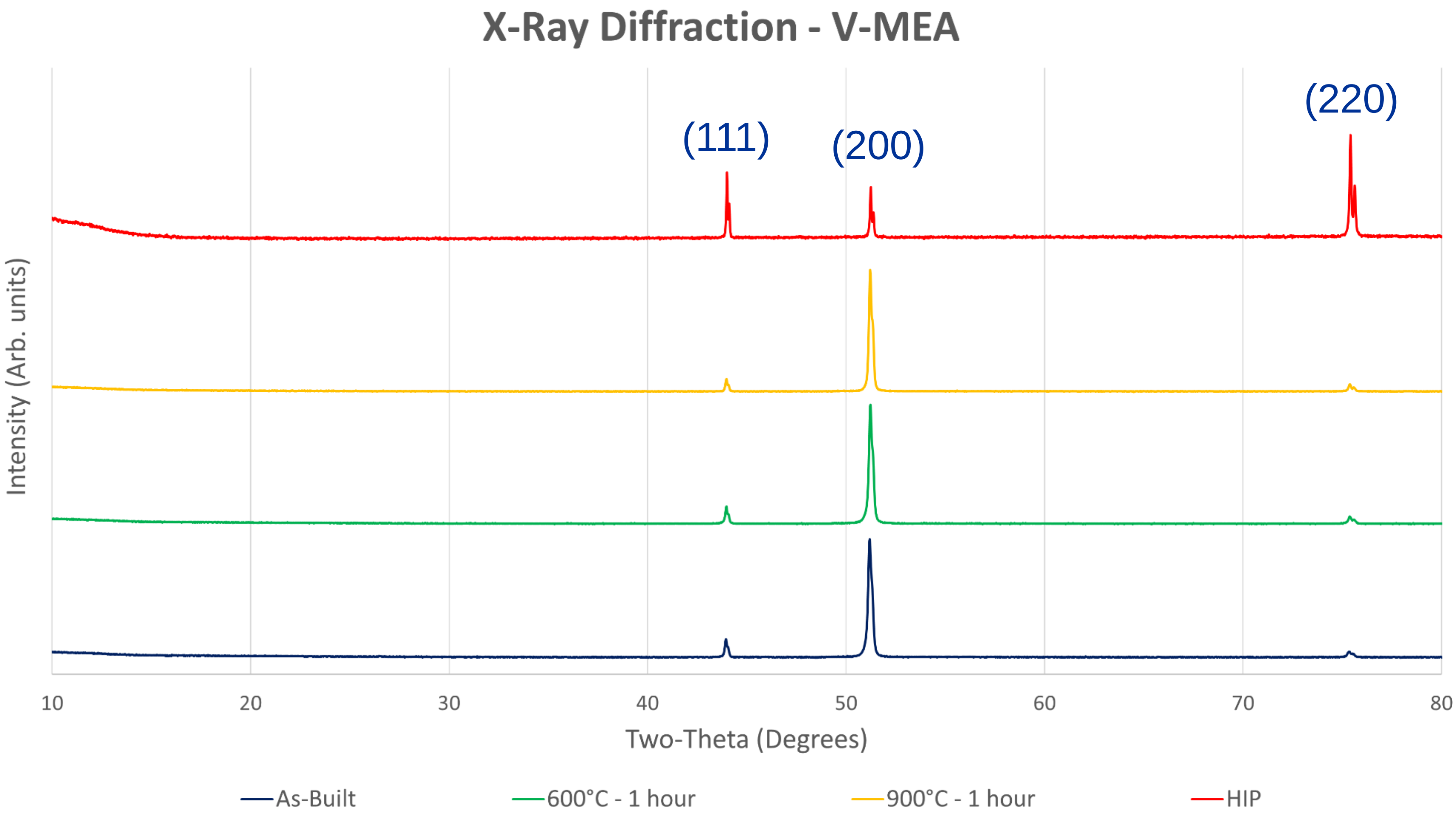
99.9% dense parts were successfully built for both the V-MEA and DS-MEA powder lots.

EDS – DS-MEA Microstructure



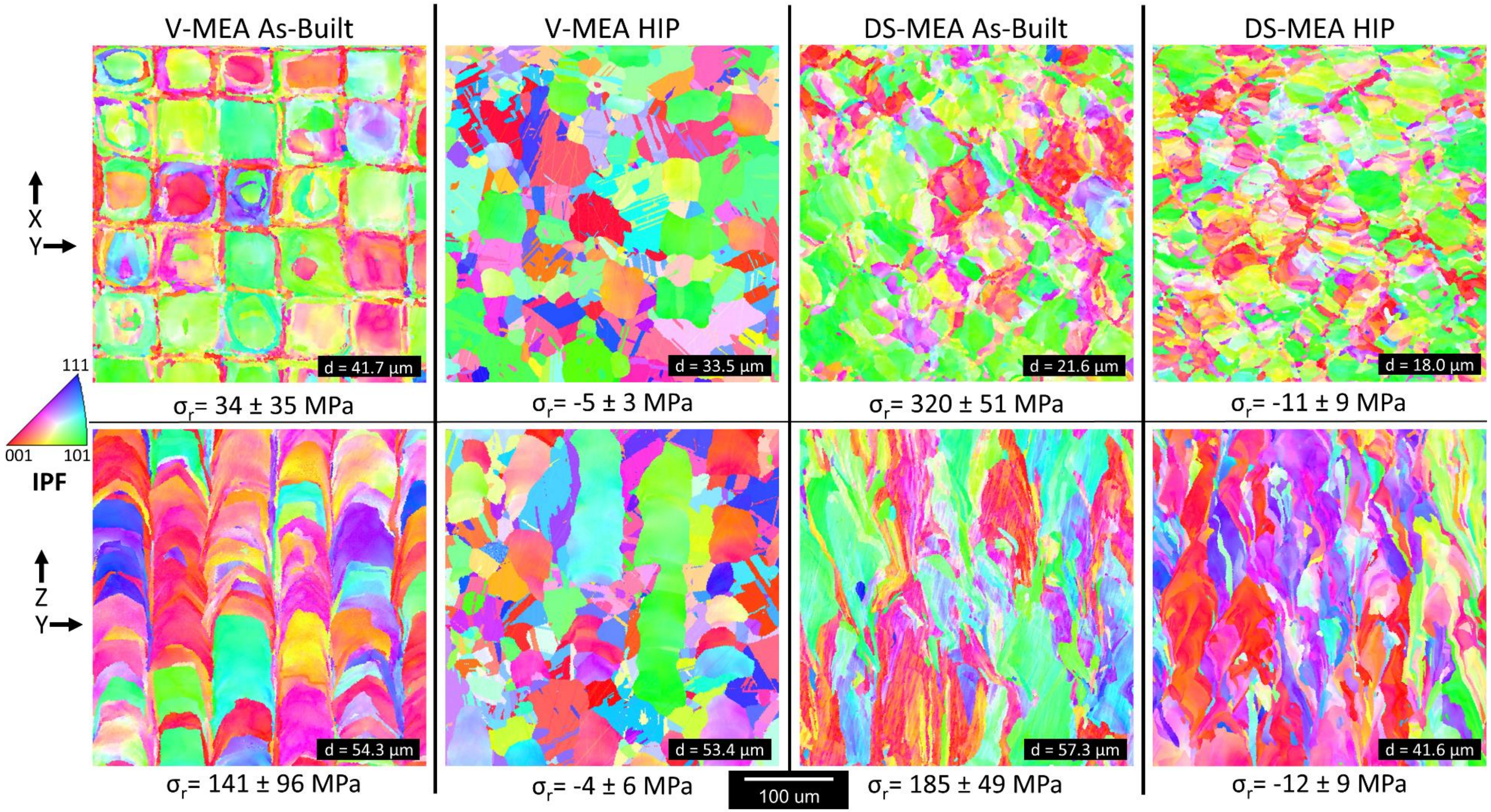
- Large (>20um) Y_2O_3 particles are not present in AM builds
- NiCoCr matrix remained a random solid solution during SLM process.

Heat Treatment effect on solid solution stability



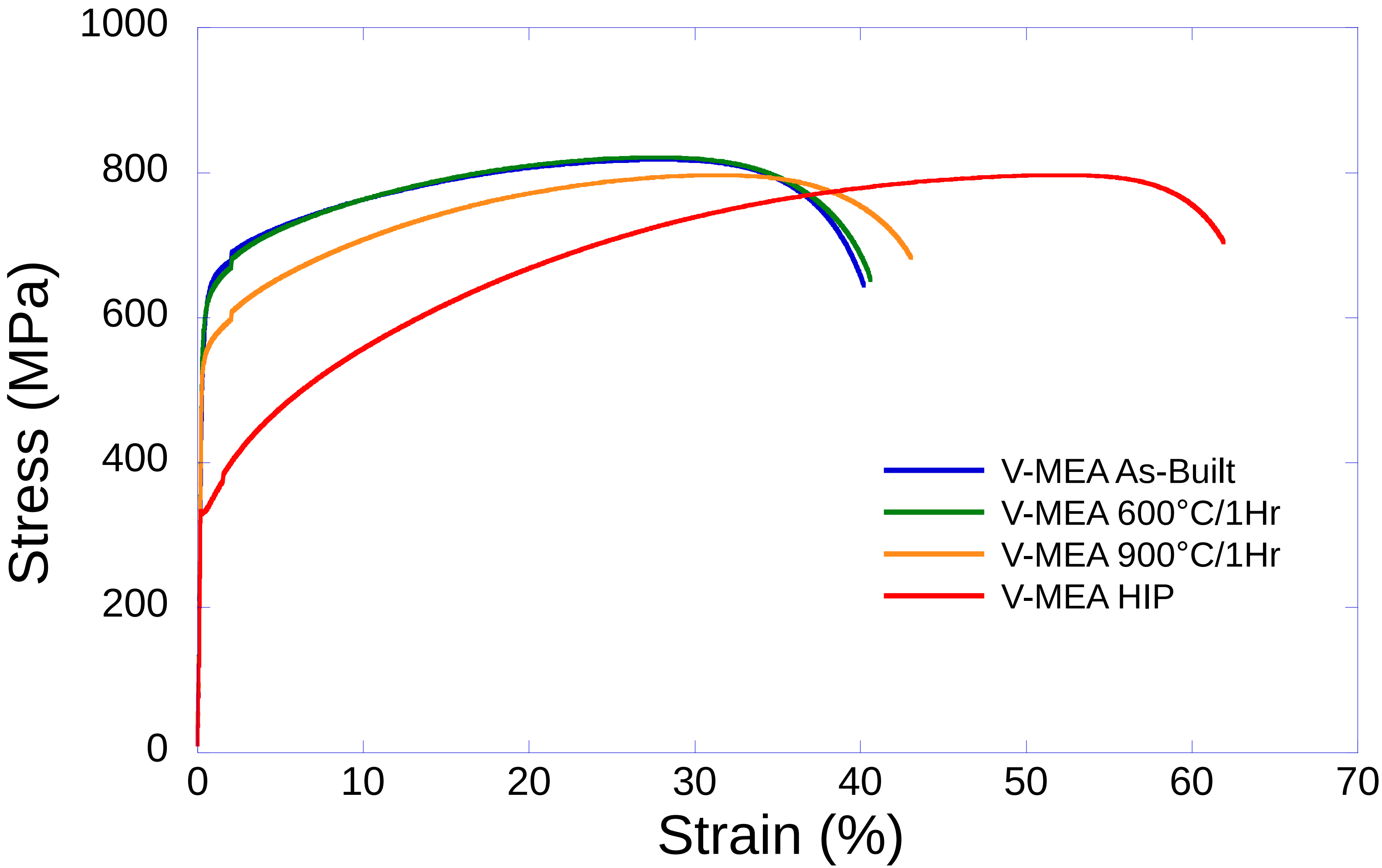
No intermetallic phases present after anneal or HIP steps.

Microstructure Analysis



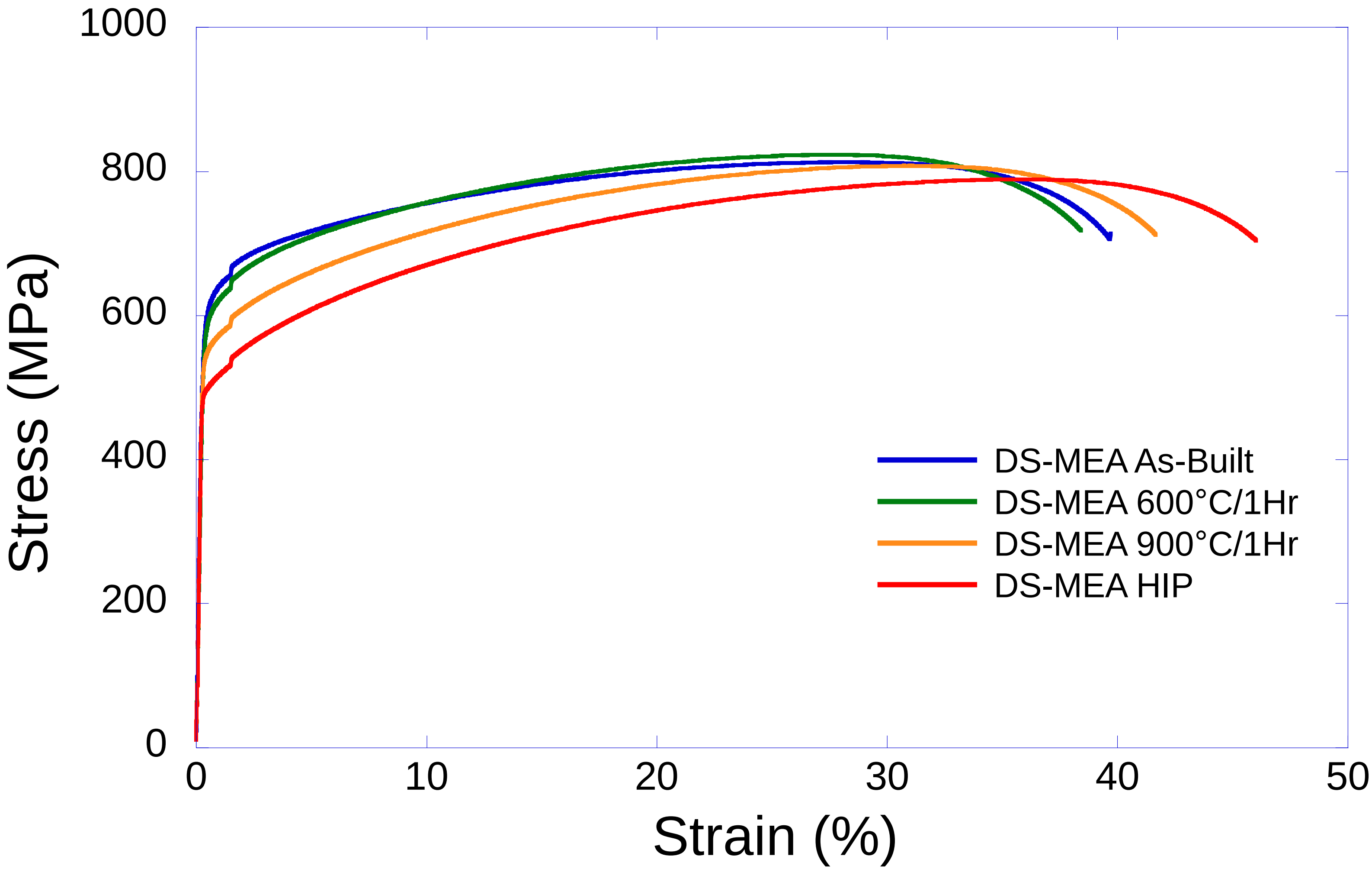
- Yttria particles have pinned the grain boundaries in the MEA-ODS builds
- The HIP cycle successfully removed residual stresses in both the V-MEA and DS-MEA builds

Mechanical Tests V-MEA



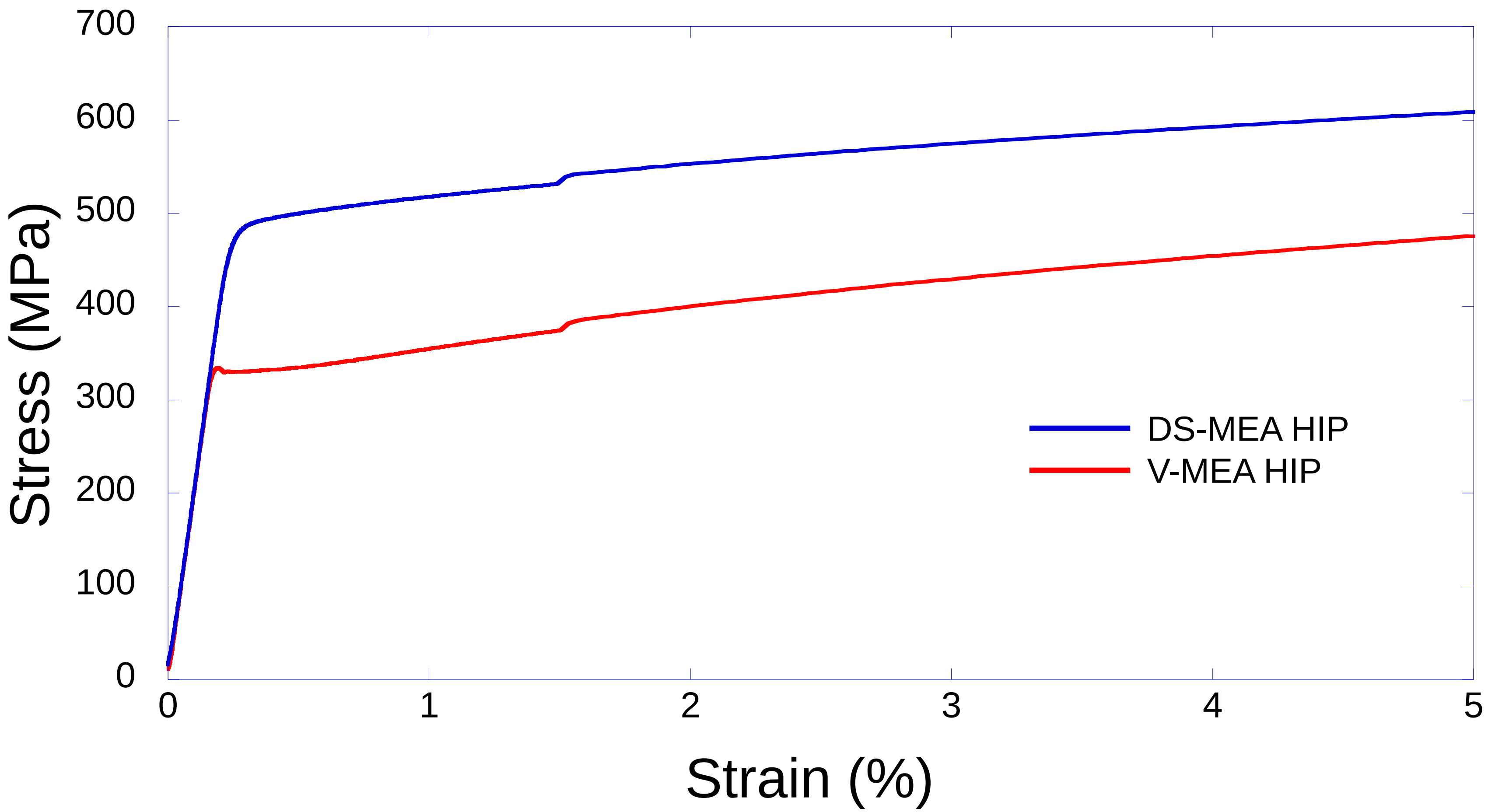
Significant reduction in yield strength associated with experience of extreme temperature for V-HEA specimen

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.

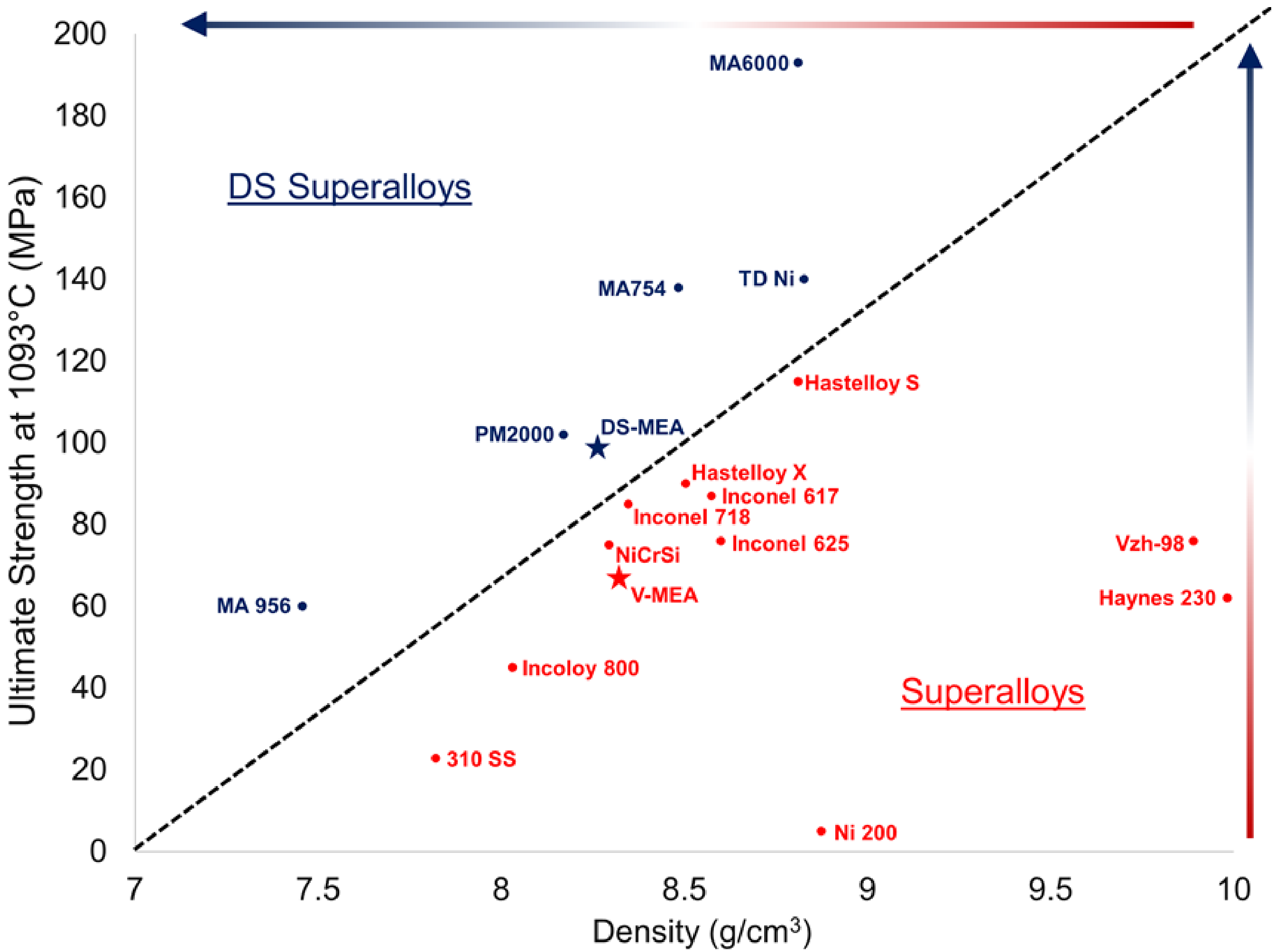
1093°C Mechanical Properties

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

Future Work

- Explore the NiCoCr chemistry for improved strength and creep properties.
- Optimize the dispersion strengthening parameters (As-built grain structure, oxide volume fraction, heat treatments, etc.)
- Model the dynamic melt pool in SLM for ceramic coated metallic particles.
- Test the AM combustor dome

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

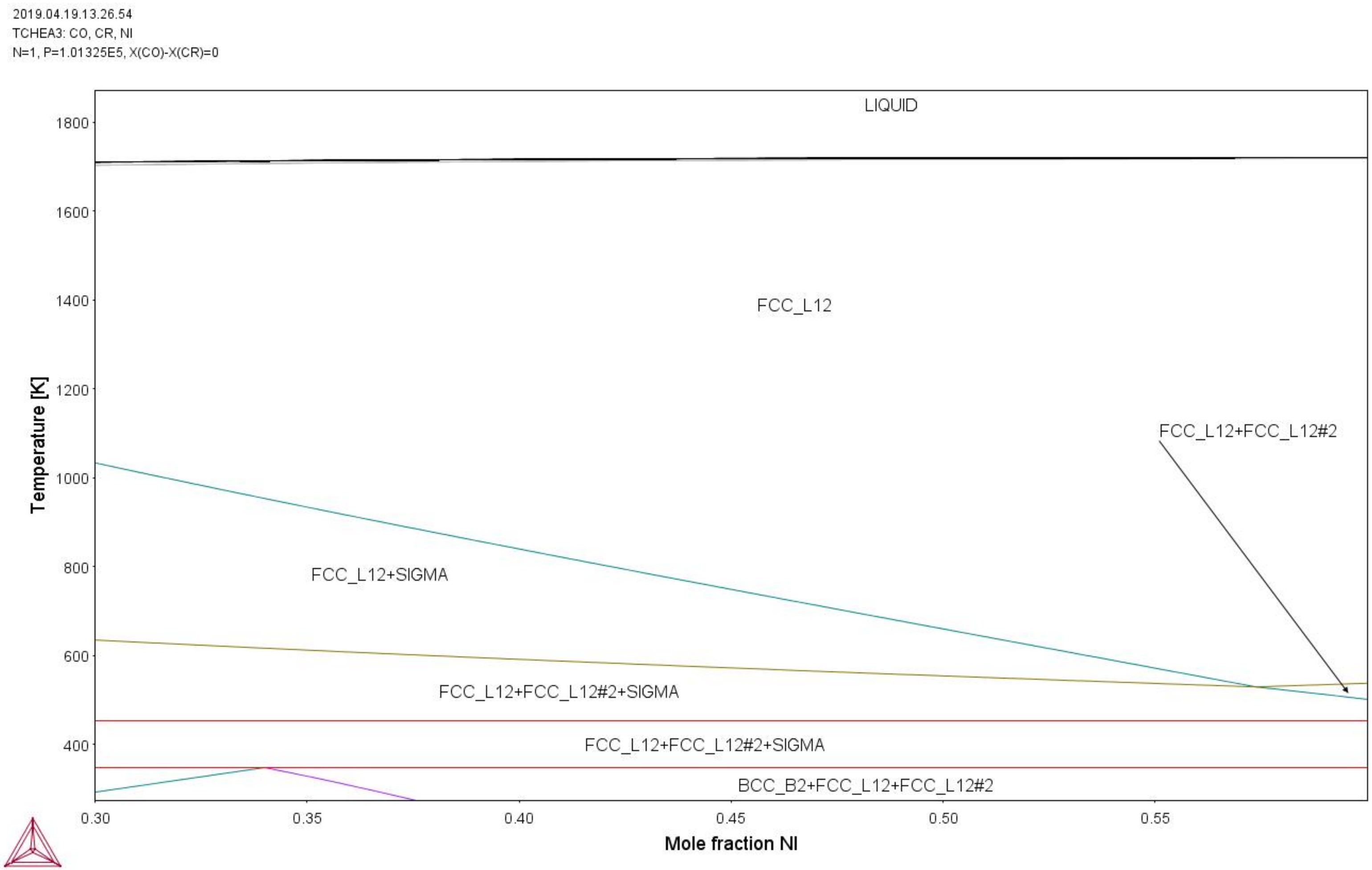
Acknowledgments

Questions?

- ASG
- Dave Ellis
- Henry de Groh
- Quynhgiao Nguyen
- Joy Buehler
- Bob Carter
- Pete Bonacuse
- David Scannapieco
- Cheryl Bowman

Extra Slides

NiCoCr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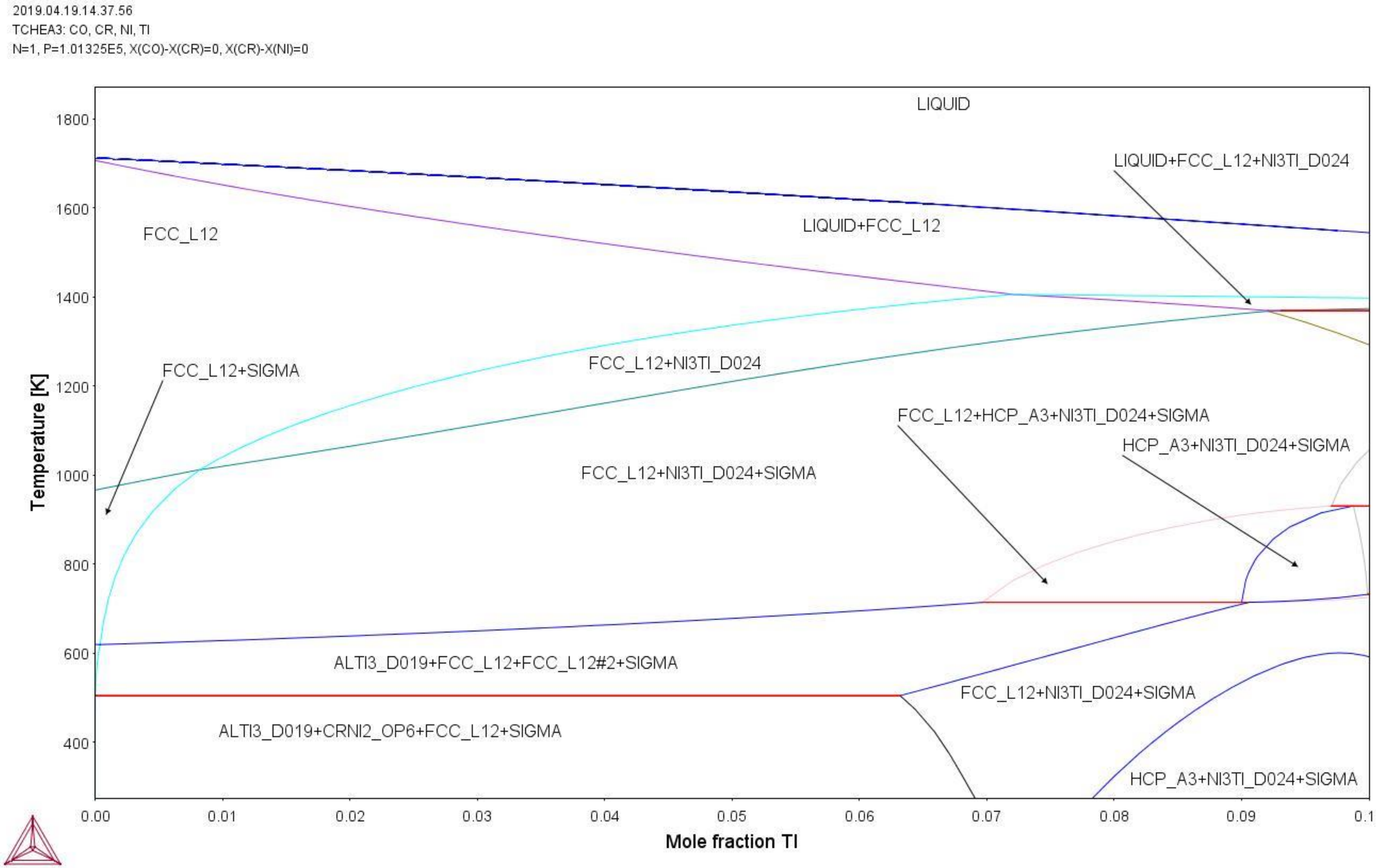
TCHEA3: CO, CR, NI
N=1, P=1.01325E5, X(CO)-X(CR)=0
LIQUID
FCC_L12
FCC_L12+FCC_L12#2
FCC_L12+SIGMA
FCC_L12+FCC_L12#2+SIGMA
FCC_L12+FCC_L12#2+SIGMA
BCC_B2+FCC_L12+FCC_L12#2
Temperature [K]
1800
1600
1400
1200
1000
800
600
400
0.30
0.35
0.40
0.45
0.50
0.55
Mole fraction NI

NiCoCr-Ti


2019.04.19.14.37.56
TCHEA3: CO, CR, NI, TI
N=1, P=1.01325E5, X(CO)-X(CR)=0, X(CR)-X(NI)=0
LIQUID
LIQUID+FCC_L12+NI3TI_D024
LIQUID+FCC_L12
FCC_L12
FCC_L12+SIGMA
FCC_L12+NI3TI_D024
FCC_L12+HCP_A3+NI3TI_D024+SIGMA
HCP_A3+NI3TI_D024+SIGMA
FCC_L12+NI3TI_D024+SIGMA
ALTI3_D019+FCC_L12+FCC_L12#2+SIGMA
FCC_L12+NI3TI_D024+SIGMA
ALTI3_D019+CRNI2_OP6+FCC_L12+SIGMA
HCP_A3+NI3TI_D024+SIGMA
Temperature [K]
1800
1600
1400
1200
1000
800
600
400
0.00
0.01
0.02
0.03
0.04
0.05
0.06
0.07
0.08
0.09
0.10
Mole fraction TI