GRX-810: NASA High Temperature Alloy Development for Additive Manufacturing

Lawrence Livermore National Lab Seminar
8/31/2022 - 8/31/2022

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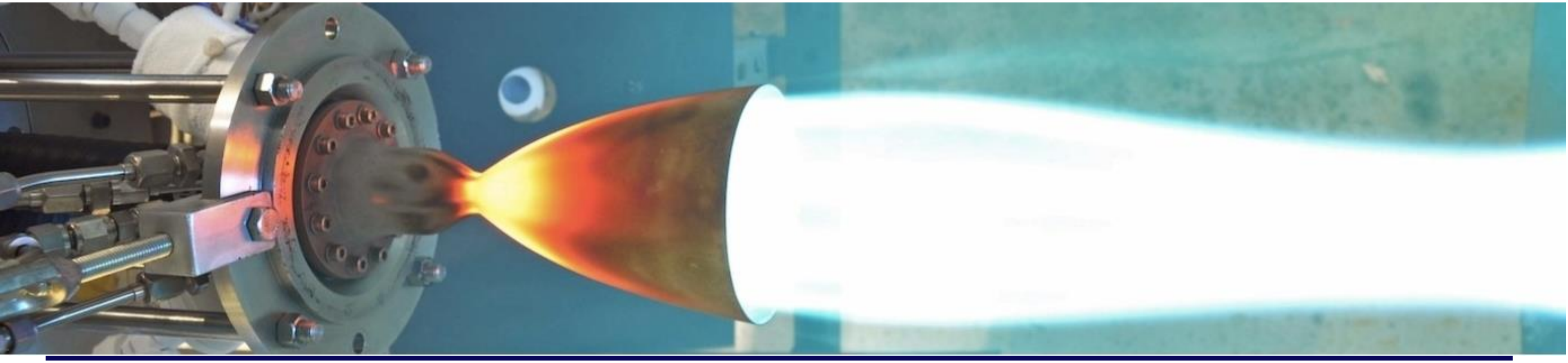
08/31/22

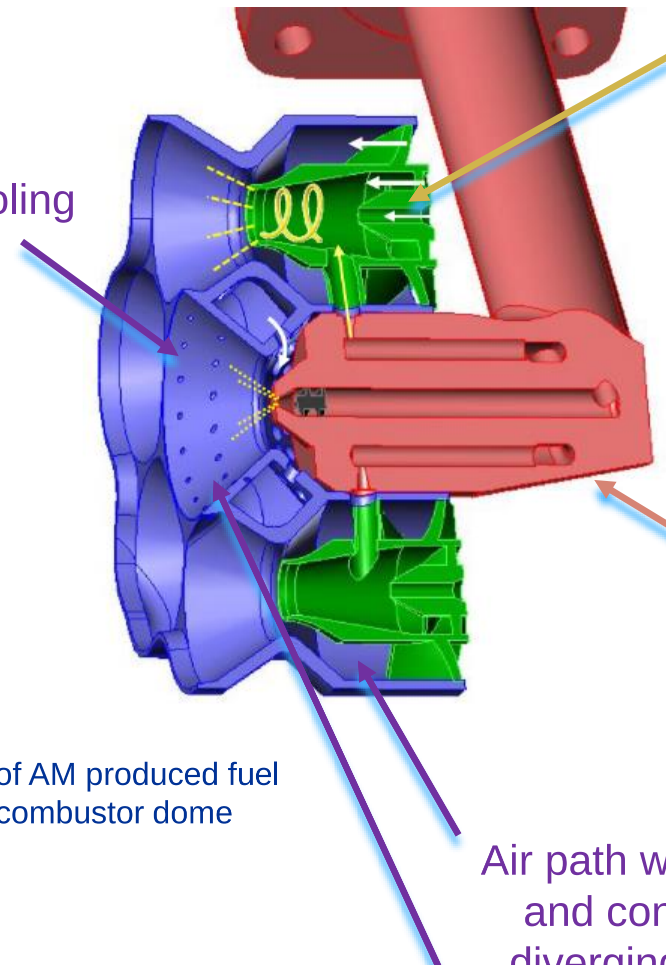


Background – NASA Application

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

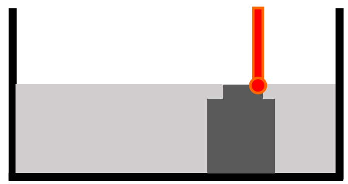
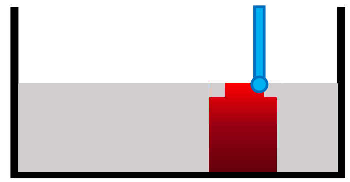
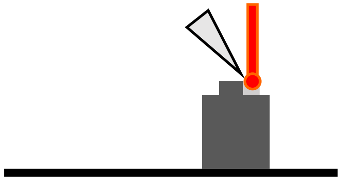
Proposed Solution: Develop a high ductility, high temperature material for an additively-manufactured (AM) combustor fuel nozzle and dome for supersonic aircraft (>1093°C (2000°F) operating temperature).

- 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

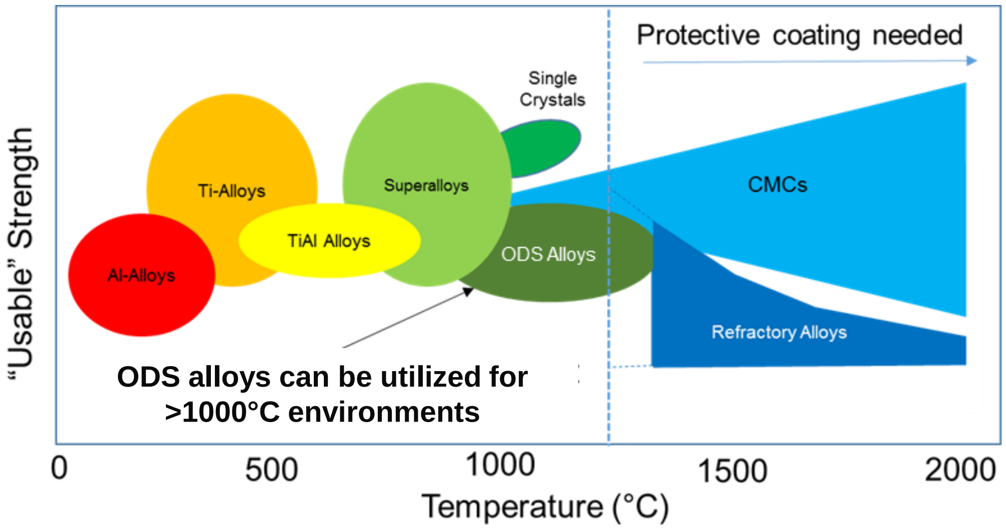
Process	Laser Powder Bed Fusion (L-PBF) 	Electron Beam Powder Bed Fusion 	Direct Energy Deposition (DED) 
Energy Source	Laser	E-Beam	Laser or E-Beam
Powder Bed	Yes	Yes	No
Power (W or kV)	50-1000 W	**30-60kV**	100-2000 W
Max Build Size (mm)	500 x 280 x 320	500 x 280 x 320	**2000 x 1500 x 750**
Material	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, L-PBF is used due to its superior dimensional accuracy.

High Temperature AM Compatible Materials

High Temperature Materials:

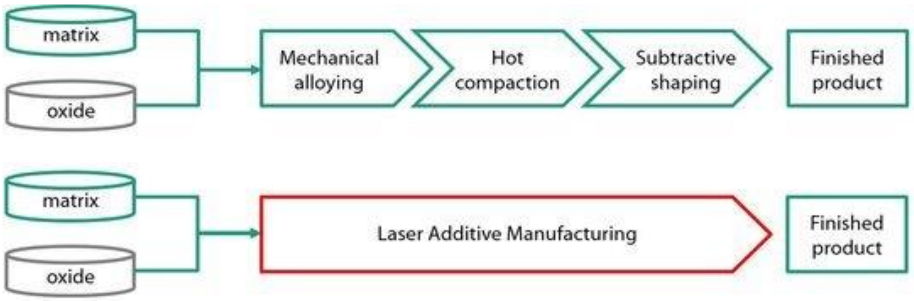
- Refractory metals
- Carbon-Carbon composites
- CMC’s
- Ni-base superalloys
- **Oxide Dispersion strengthened (ODS) alloys**



Inspired by Andy Jones. ODS alloy Development.

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

Conventional Manufacturing vs AM



Can AM improve ODS alloy manufacturability?

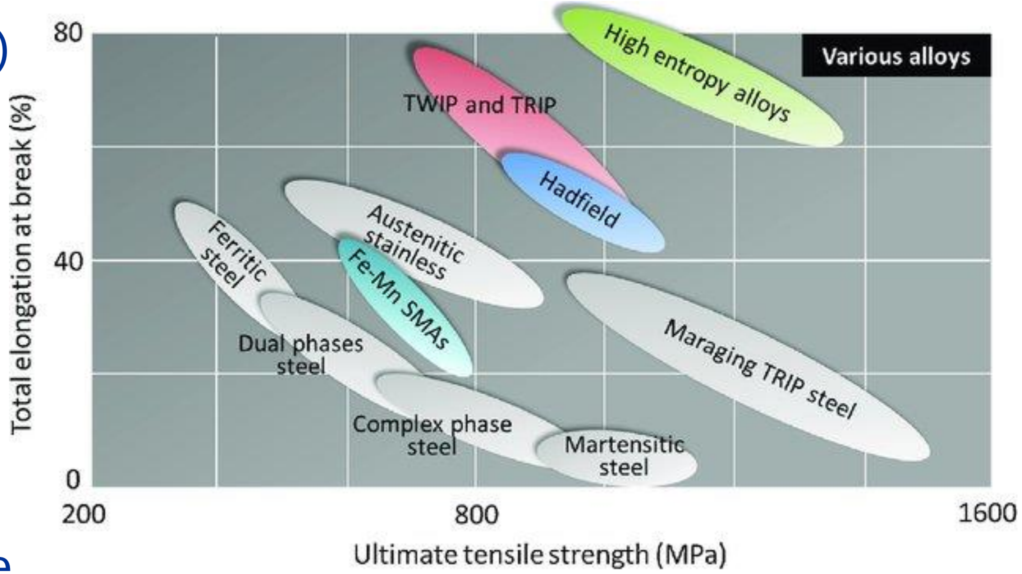
Advanced Materials and Manufacturing for High Temperature Applications

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

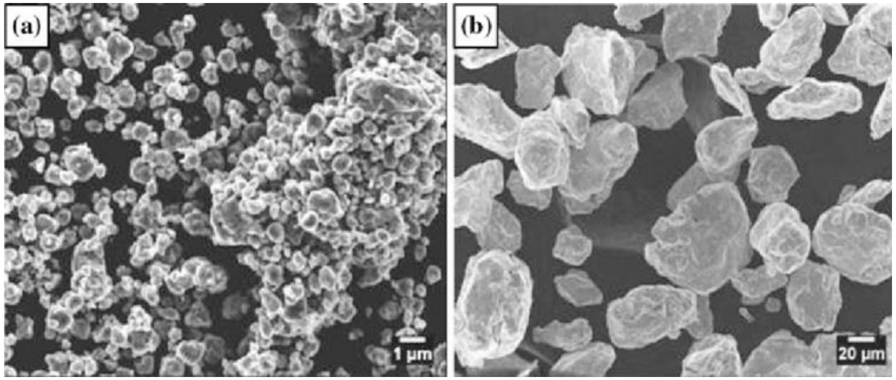
- The equiatomic NiCoCr medium-entropy alloy is particularly of interest due to its strong phase stability and mechanical properties.

- 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?**

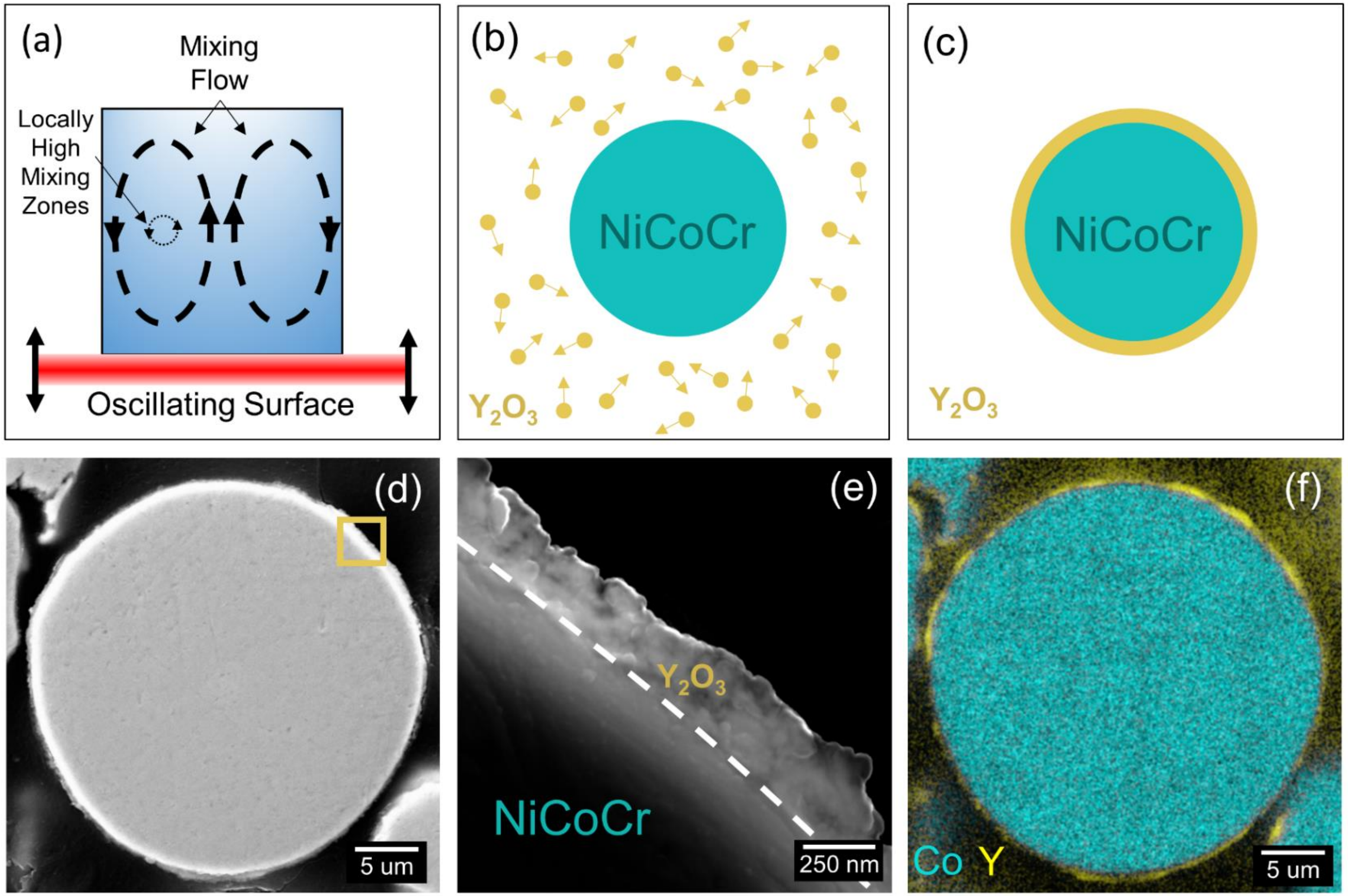


Chowdury *et al*. Materials Science and Engineering: Reports (2017)



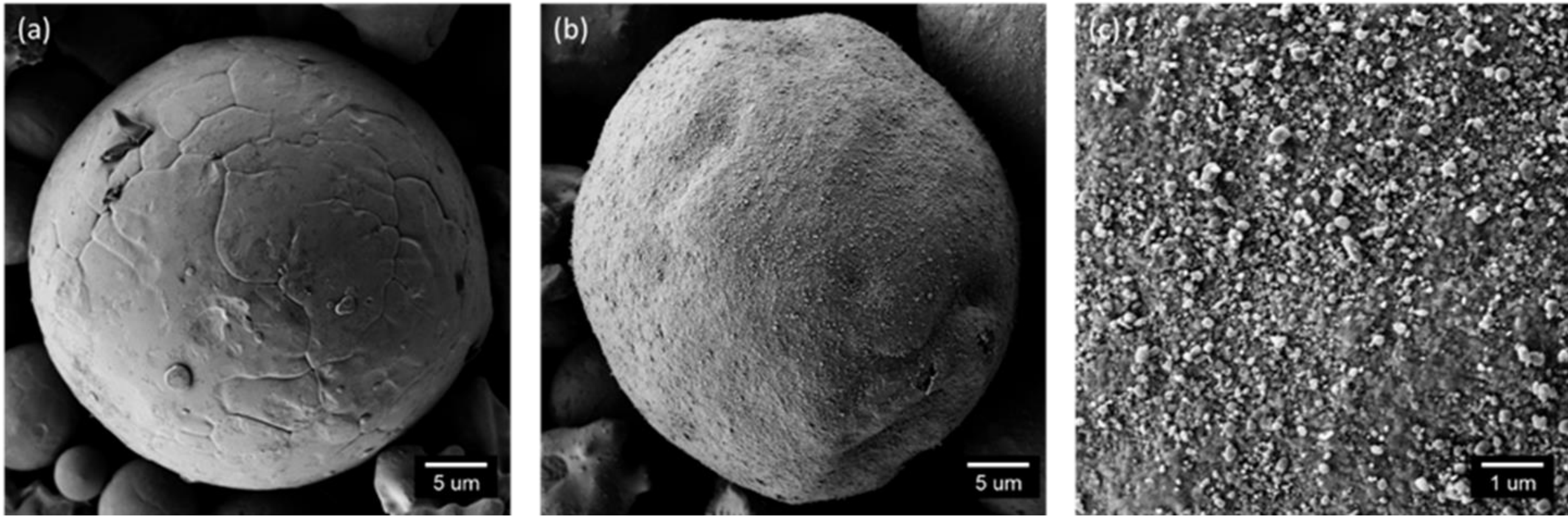
Oksiuta *et al*. Journal of Material Science (2010)

Novel Fabrication Technique for Oxide Dispersion Strengthened (ODS) Alloys



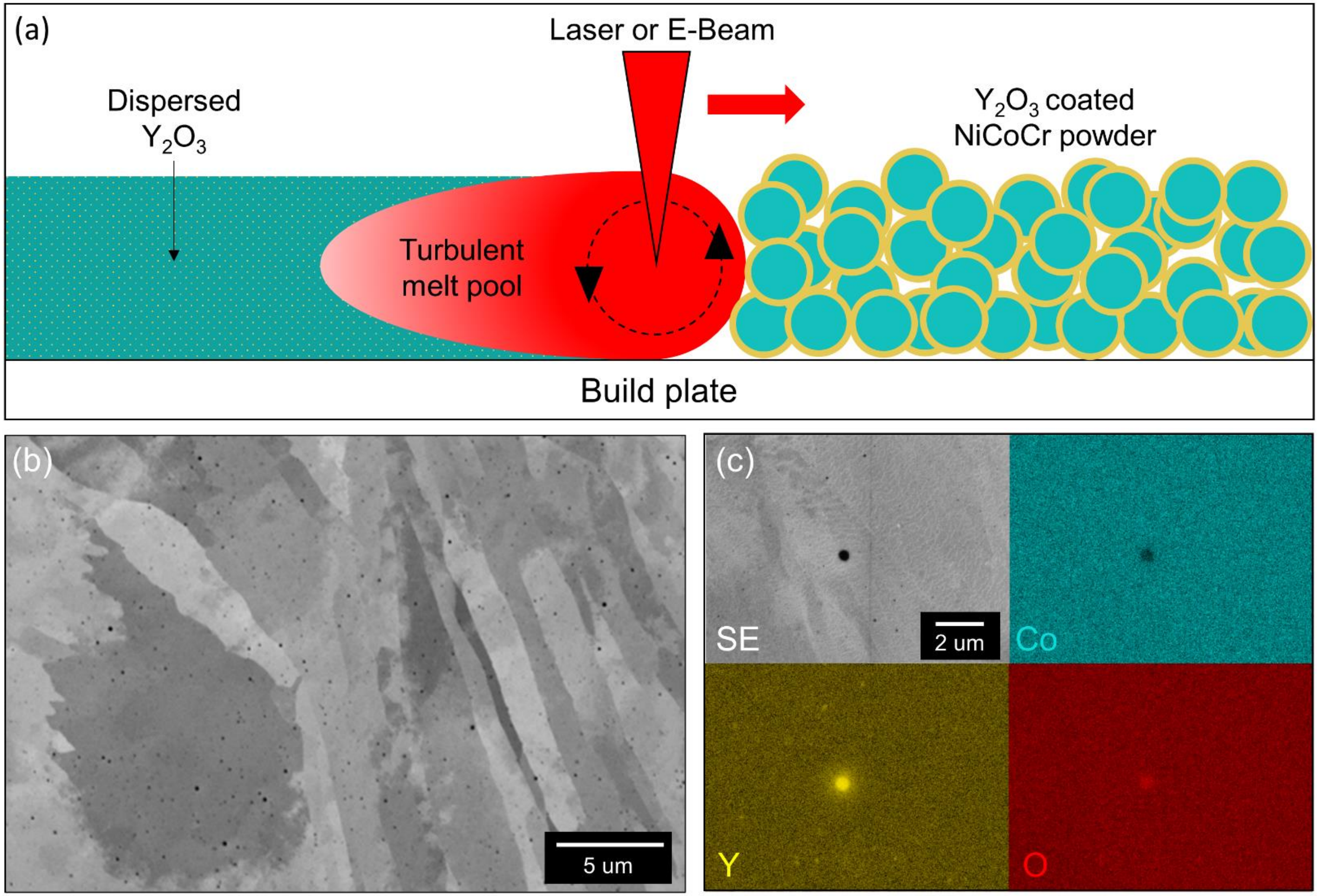
New high energy mixing technique successfully coats NiCoCr powder with 1 wt.% Y_2O_3.

Novel Powder Coating Technique



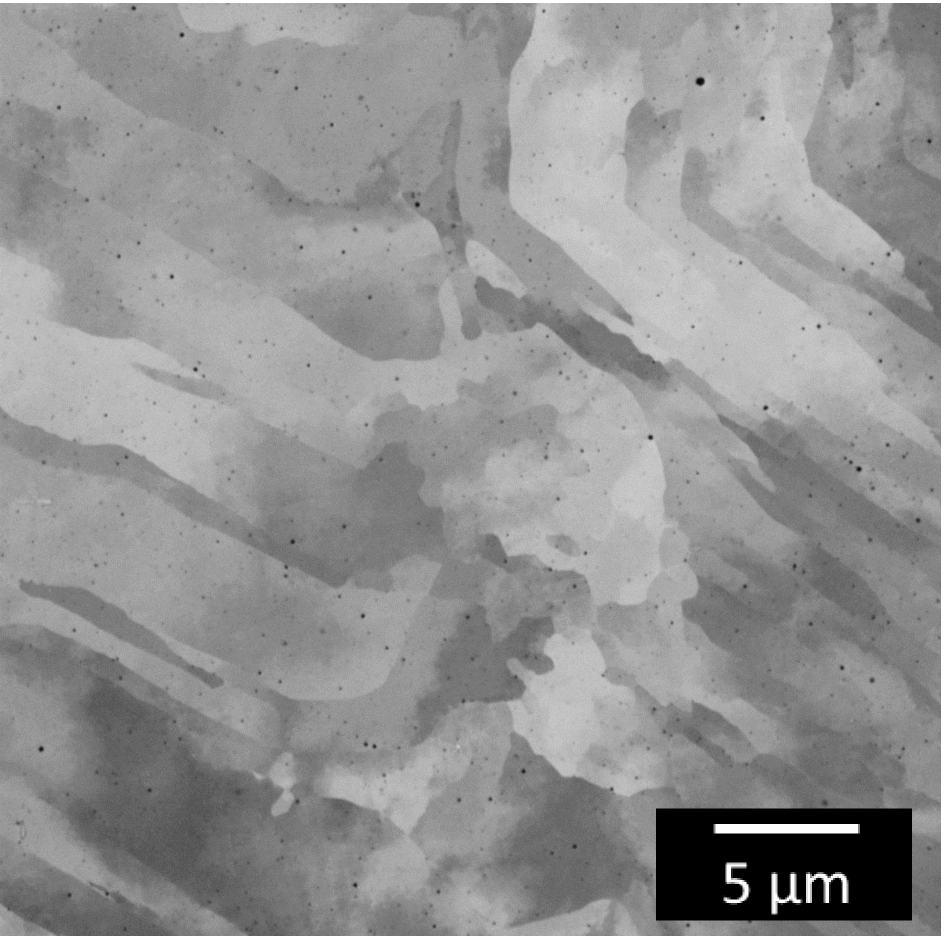
- The advanced dispersion coating (ADC) technique did not deform the metallic powder.
- The ADC technique fully coats the metallic powders with nano-scale oxides
- Both uncoated and coated powders qualitatively passed the Hall flow test.
- The technique does not affect the printability of the powder lot.

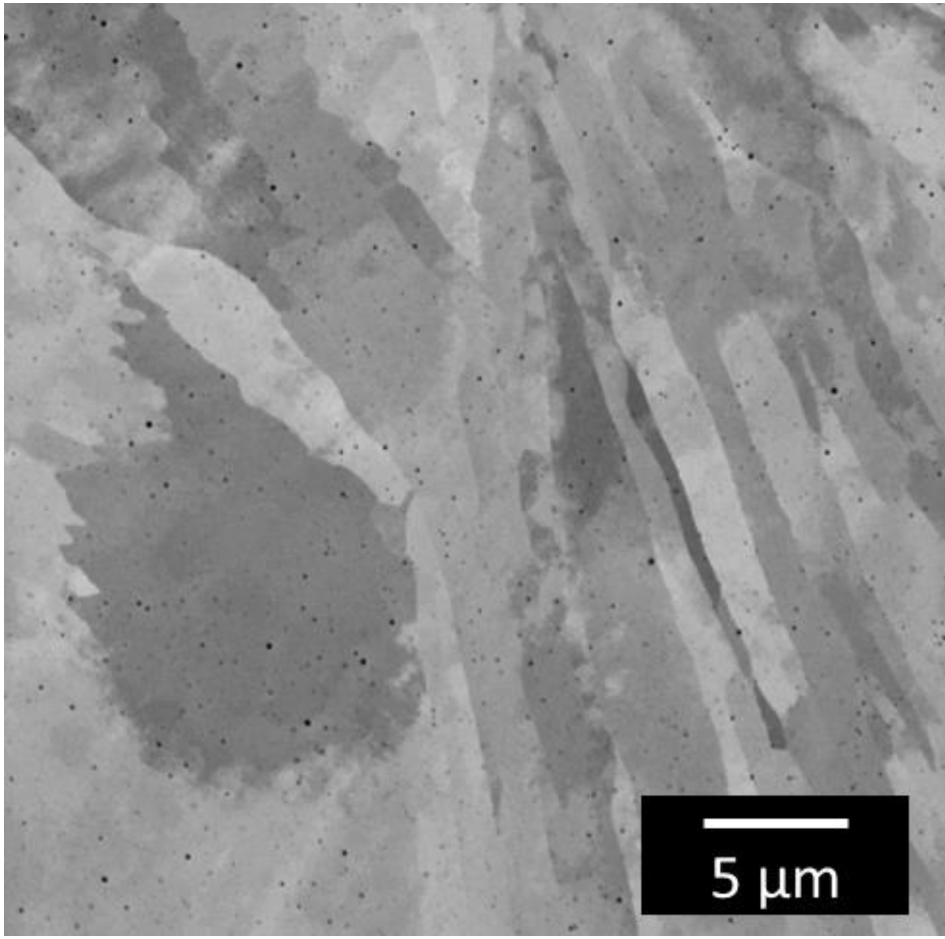
Leveraging L-PBF to Produce Oxide Dispersion Strengthened Alloys



L-PBF successfully disperses the nano-scale Y_2O_3 particles throughout the AM build

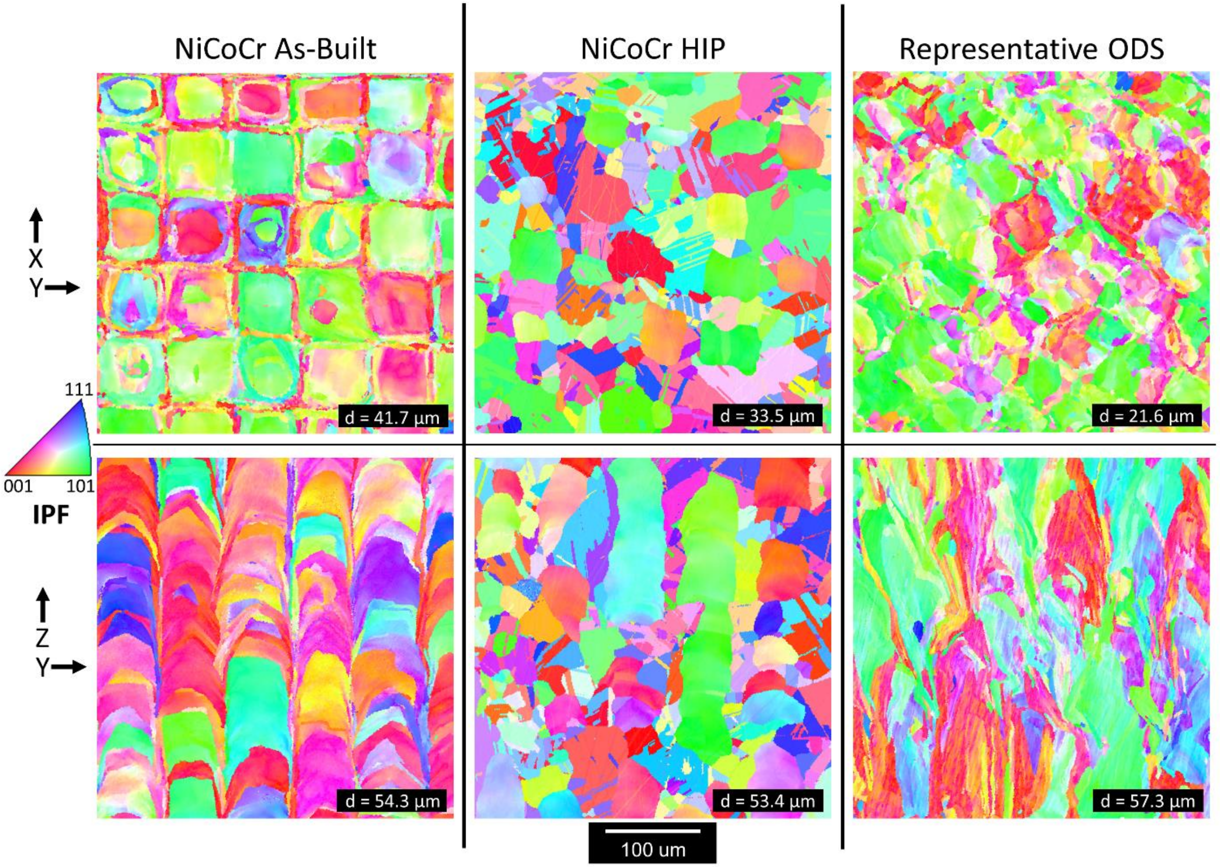
ODS-NiCoCr Microstructure





Nano-scale Y_2O_3 particles are randomly dispersed throughout microstructure.

Microstructure Analysis

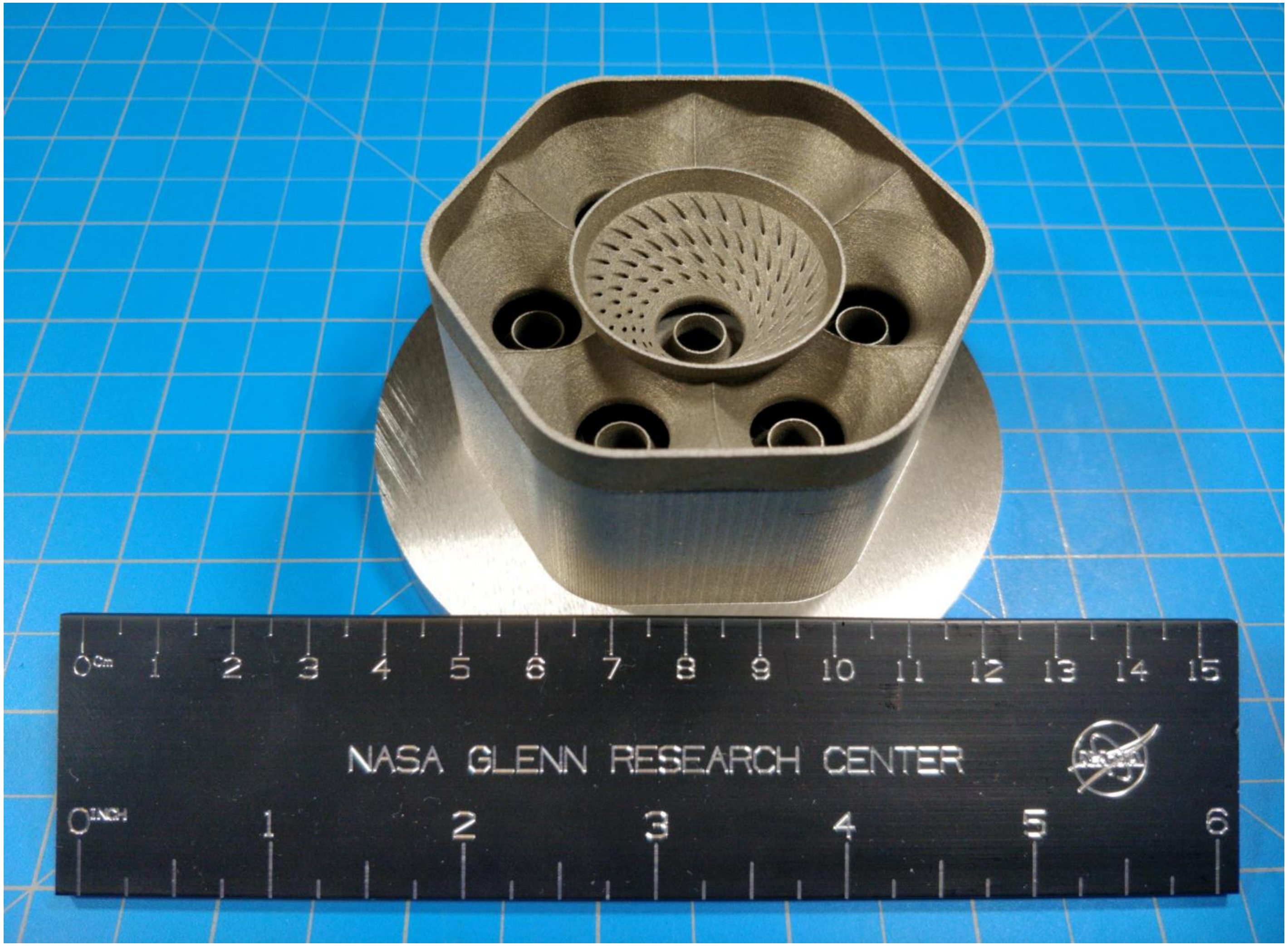


- Y_2O_3 particles have pinned the grain boundaries in the ODS AM builds.
- The HIP cycle successfully removed residual stresses for AM samples

Residual Stress

Alloy	As-Built – Build direction	As-Built – 90° from build direction	HIP – Build direction	HIP – 90° from Built direction
AM NiCoCr	34 ± 35	141 ± 96	-5 ± 3	-4 ± 6
ODS-NiCoCr	320 ± 51	185 ± 49	-11 ± 9	-12 ± 9
ODS-ReB	321 ± 52	179 ± 47	-8 ± 5	-12 ± 6

Oxide Dispersion Strengthened MPEA Combustor Dome



Development of GRX-810 Composition

Model Driven MPEA Design

Goals to improve on previous NiCoCr Entropy Alloy:
1.) Maximize solid solution strengthening
2.) Maintain solid solution matrix
3.) Add grain boundary carbides
4.) Reduce freezing range to under 100°C for printability
5.) Avoid TCP and intermetallic grain boundary phases

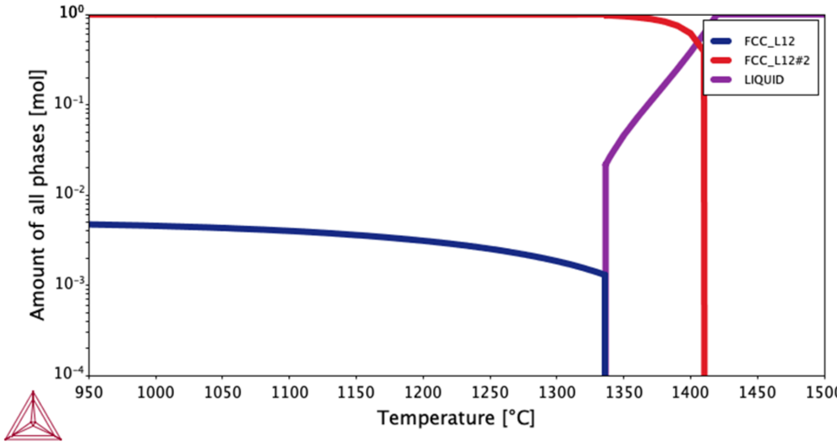


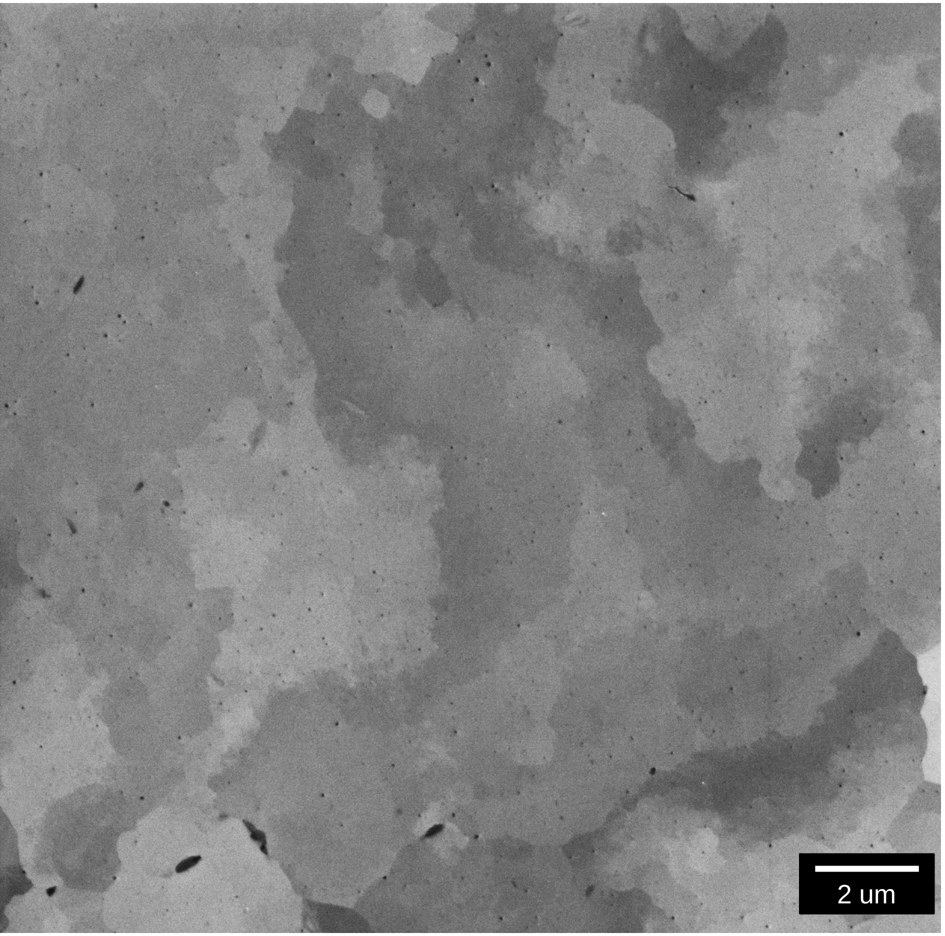
Figure: CALPHAD simulation of phase formation in new composition. No intermetallic or TCP phases are predicted.

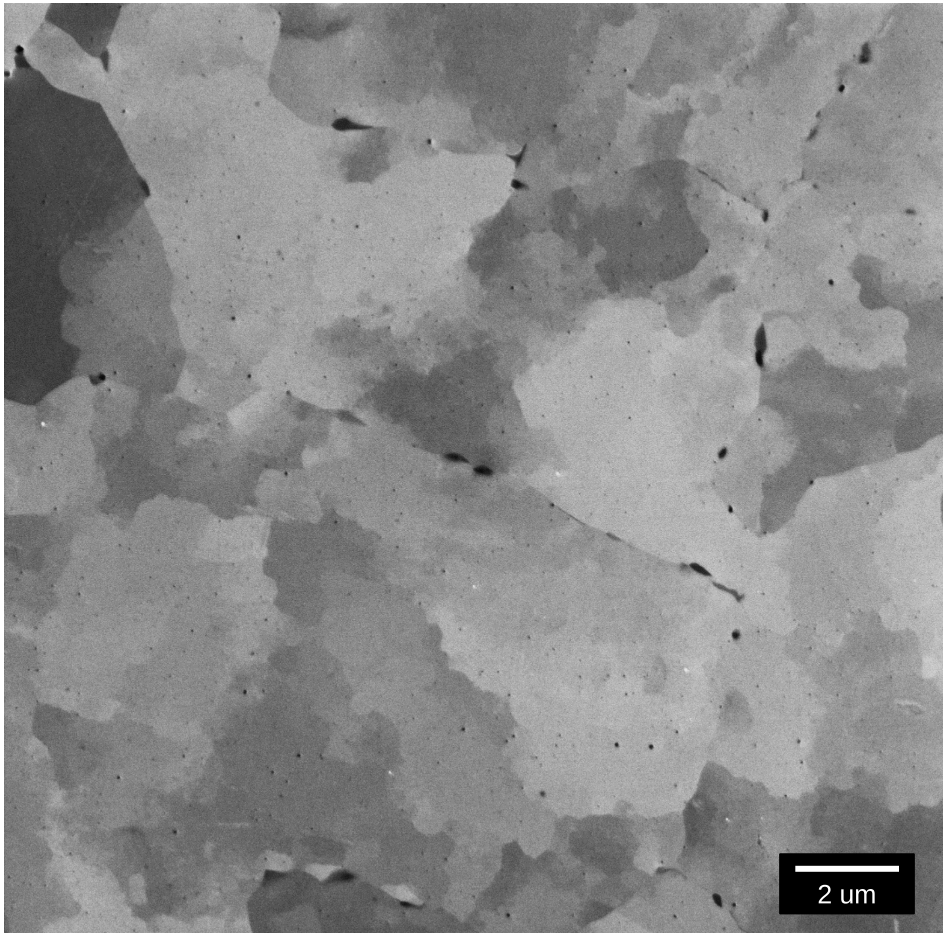
Over 50 simulations provided an optimized composition named GRX-810

Models calculated by C. Kantzos

	Ni	Co	Cr	Re	Al	Ti	Nb	Mo	W	Zr	C	B
Old Composition	Bal.	32	30	1.5								.003
New Composition (GRX-810)	Bal.	33	29	1.5	x	x	x	x	x	x	x	x

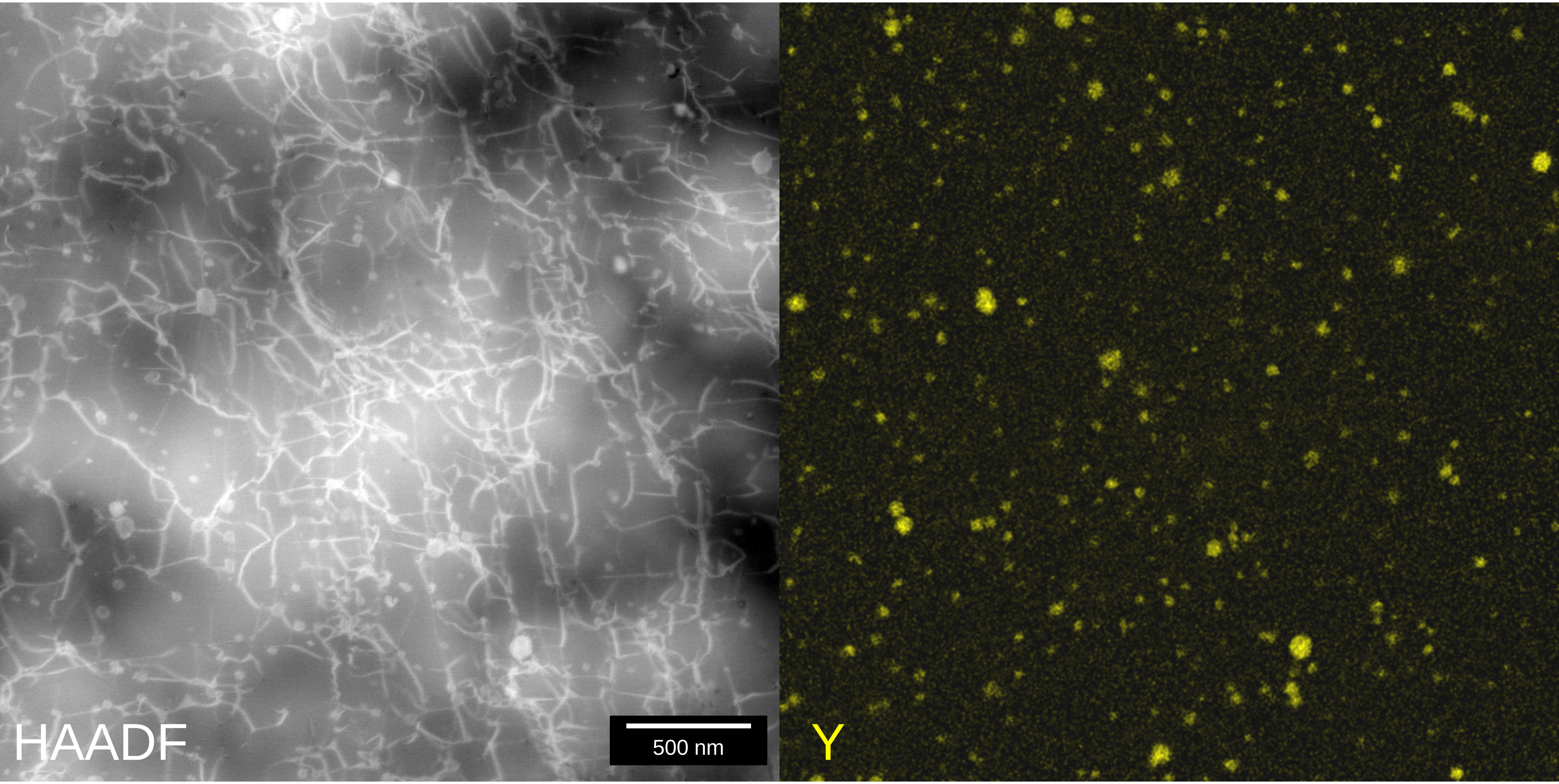
SEM – GRX-810





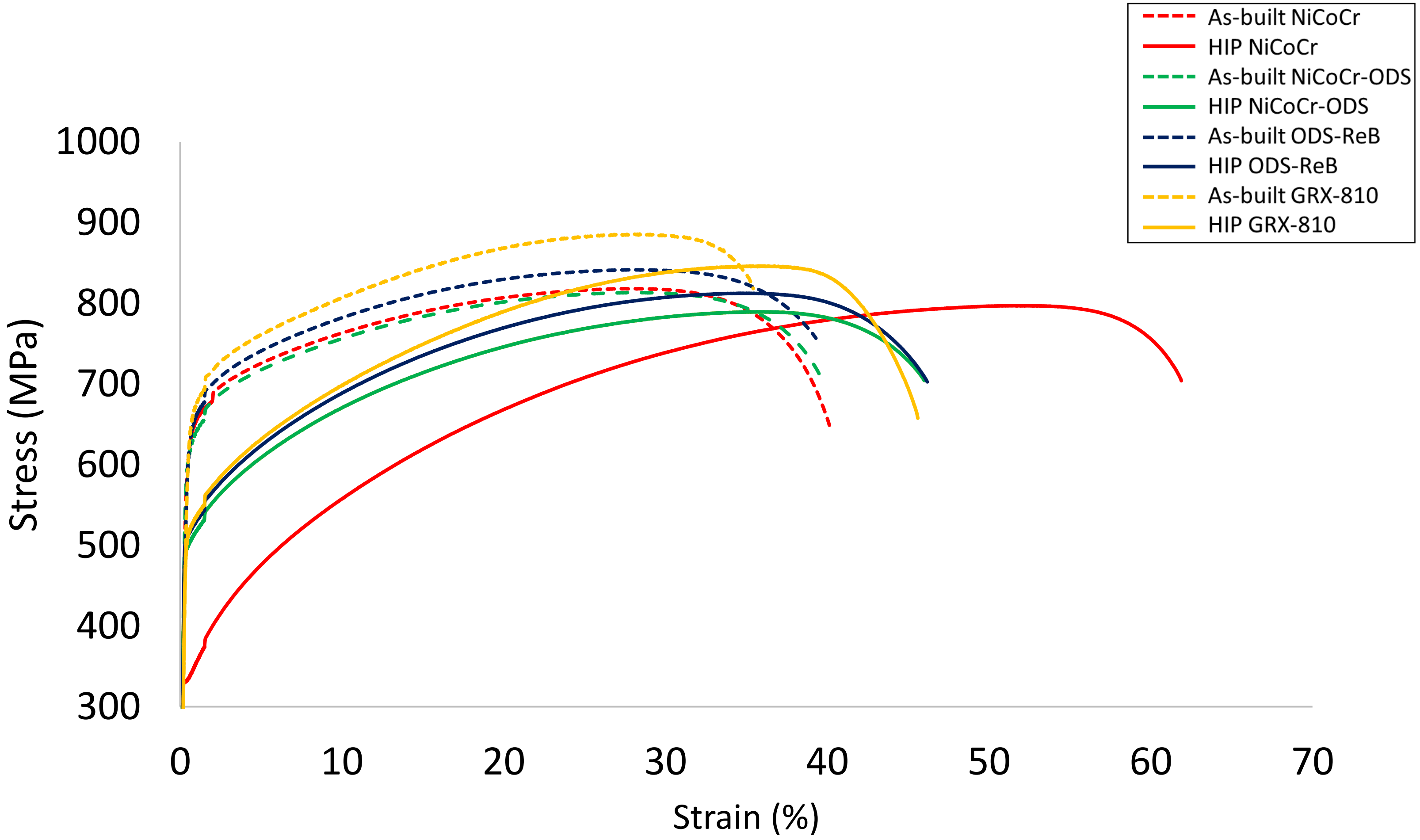
High resolution SEM reveals dispersed nano-oxides and grain boundary phases.

STEM-EDS



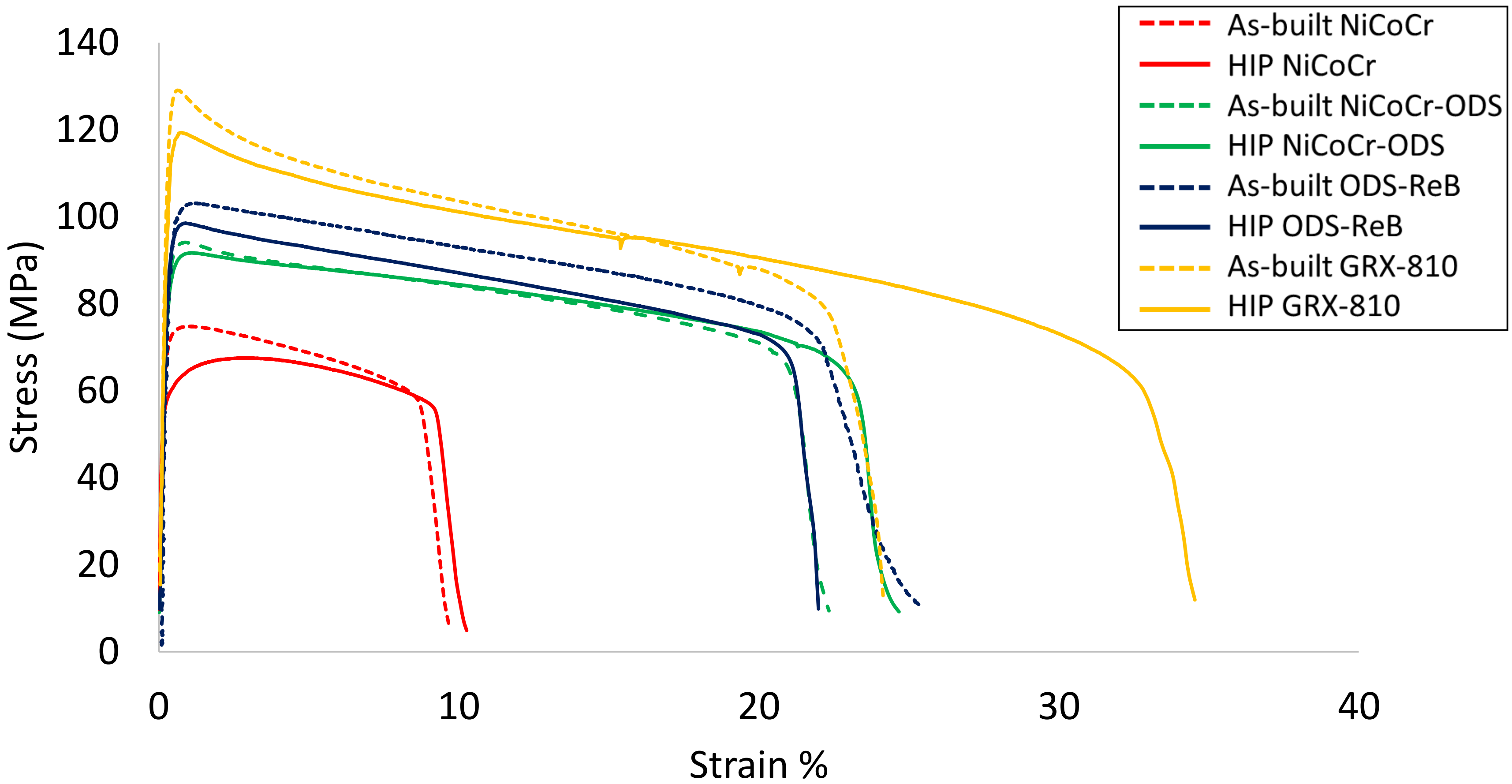
STEM-EDS analysis confirms high density of nano-scale Y_2O_3 particles throughout bulk. No oxide agglomeration was present. Most other elements did not react with oxides (Nb and Ti were exceptions).

Mechanical Results – Room Temperature Tensile



- **GRX-810 presents higher ultimate tensile strength at room temperature while maintaining the base materials high ductility.**

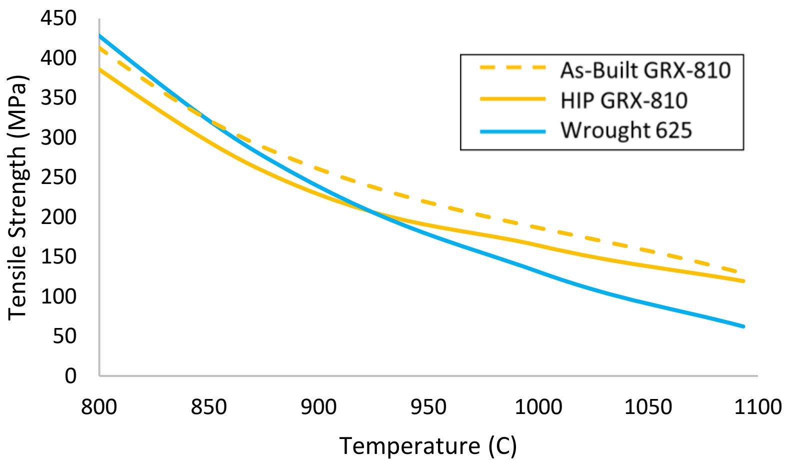
Mechanical Results – 1093°C Tensile



- **GRX-810 provides almost double the tensile strength and over 2x the ductility compared to AM NiCoCr.**

GRX-810 Tensile Overview

Tensile Strength Comparison



- GRX-810 begins to perform better than conventional alloys (625/718) around 850°C

- GRX-810 possesses good ductility at all temperatures tested – including cryogenic temperatures.

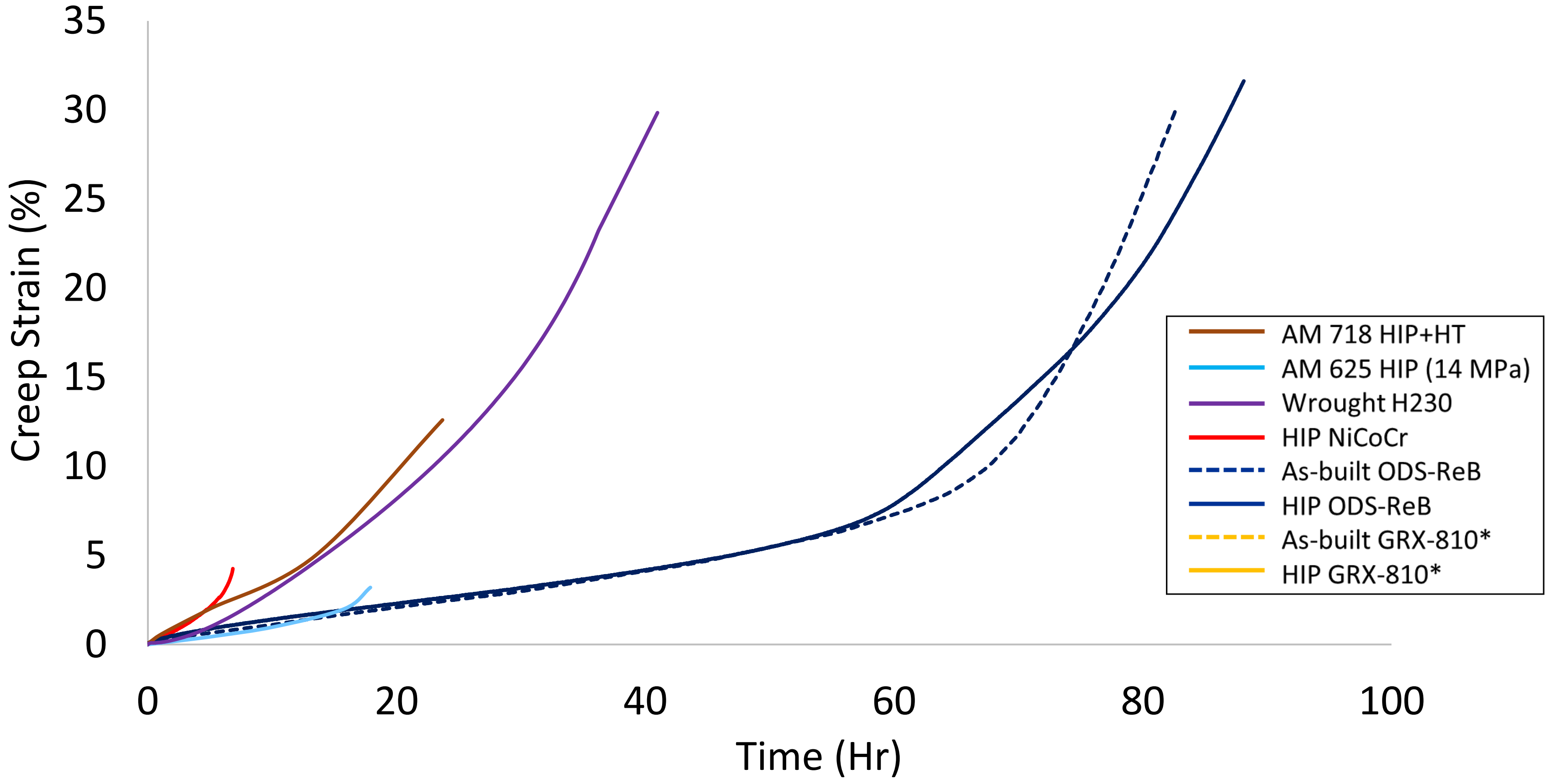
As-built GRX-810 Tensile Properties

Temperature (C)	Tensile Strength (Mpa)	Yield Strength (Mpa)	Elongation
-195.6	1303.1	910.1	39.6
21.1	882.5	641.2	33
426.7	655.0	410.2	33.3
648.9	675.7	479.2	32.1
871.1	292.3	249.6	56.1
1093.3	128.9	127.6	22

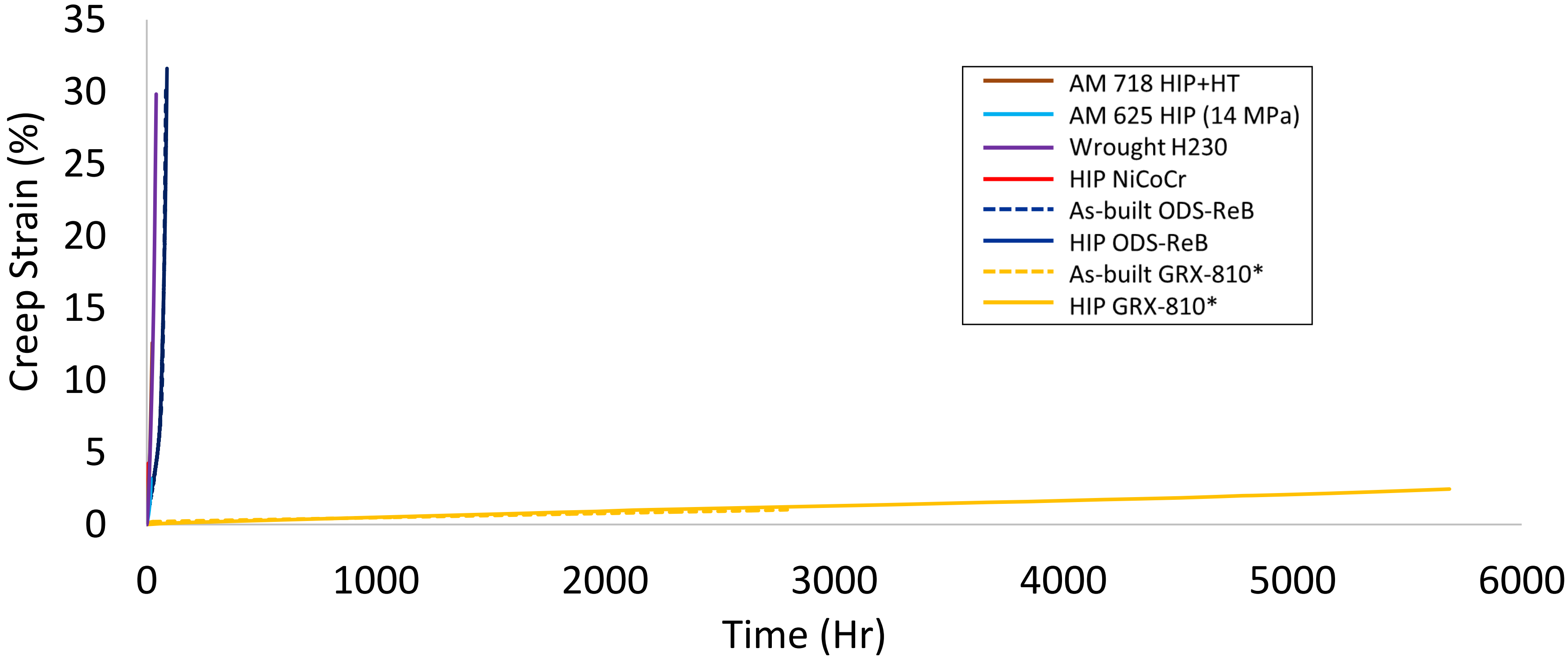
HIPed GRX-810 Tensile Properties

Temperature (C)	Tensile Strength (Mpa)	Yield Strength (Mpa)	Elongation
-195.6	1227.3	723.9	49
21.1	848.1	515.0	43
426.7	655.0	410.2	40
648.9	630.9	368.9	43
871.1	262.7	206.2	62
1000.0	164.1	161.3	44
1093.3	119.3	115.8	32

Mechanical Results – 1093°C/20MPa Creep Rupture

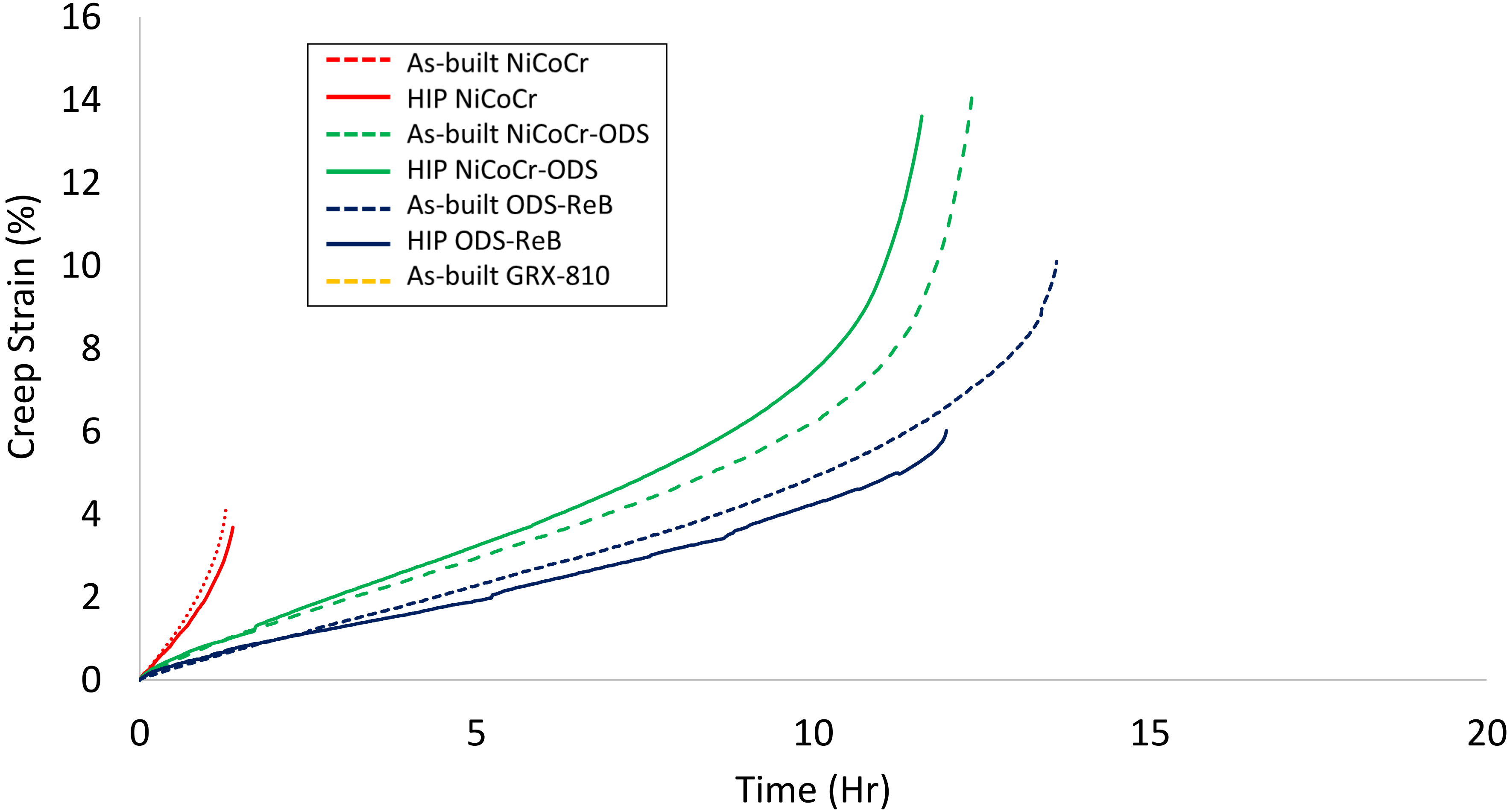


Mechanical Results – 1093°C/20MPa Creep Rupture

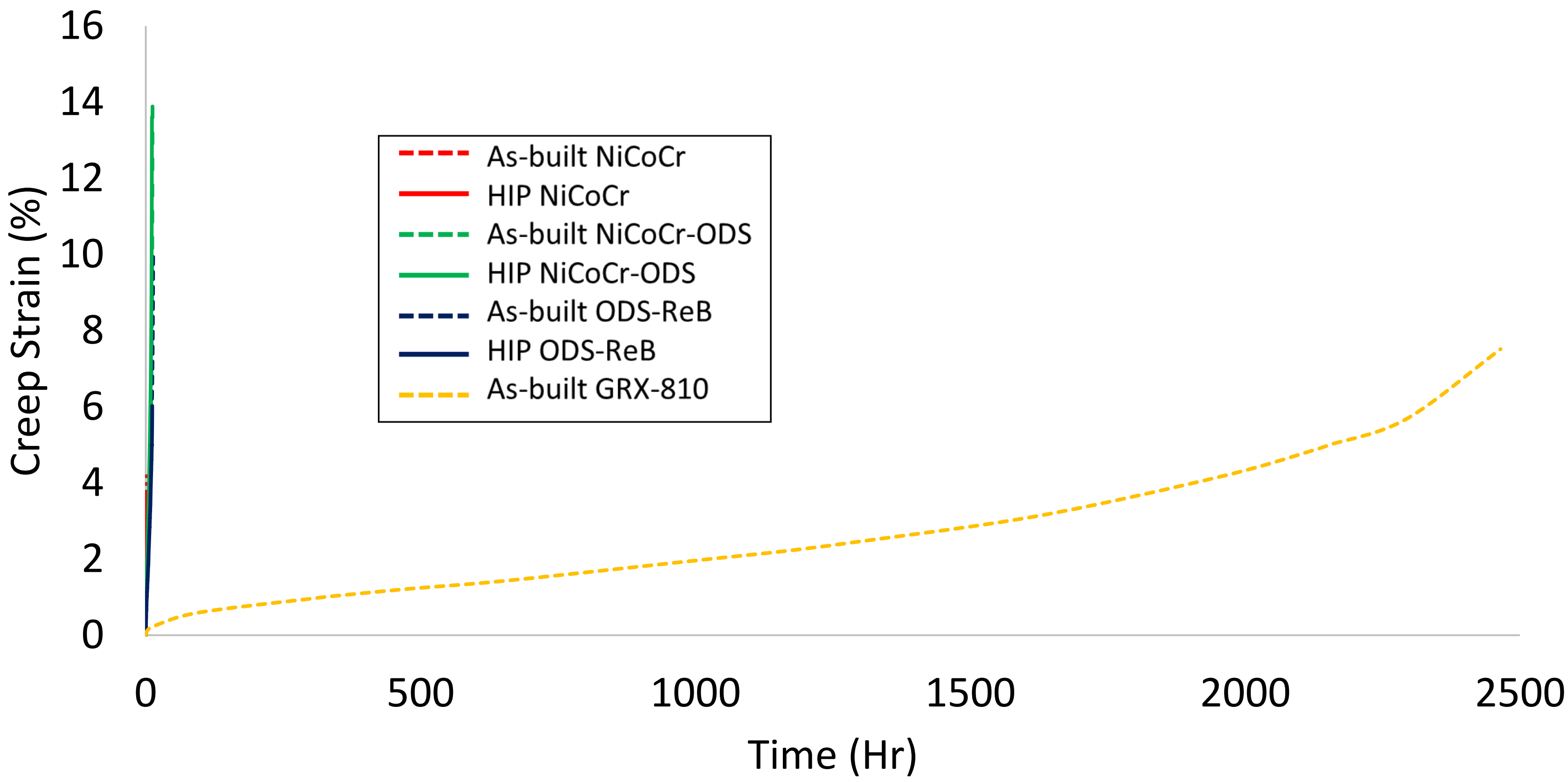


- ***The as-built and HIP GRX-810 ODS tests are still running ATM (2% strain currently).**
- GRX-810 is providing orders of magnitude improvements in creep rupture life at 1093°C compared to conventional superalloys 718 and 625.
- AM 718/625 built and tested by Henry DeGroh and Chris Kantzos.

Mechanical Results – 1093°C/31MPa Creep Rupture

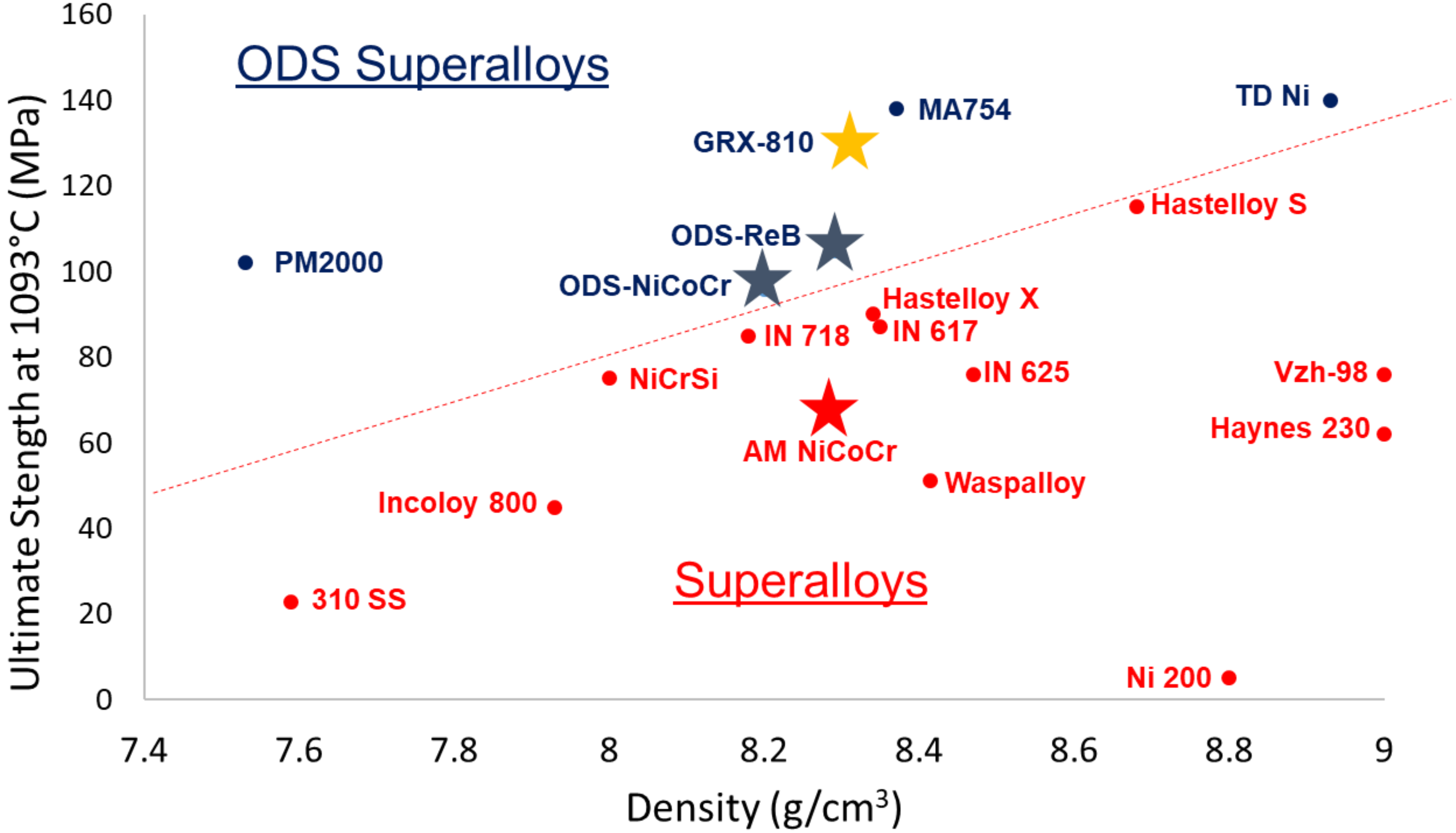


Mechanical Results – 1093°C/31MPa Creep Rupture



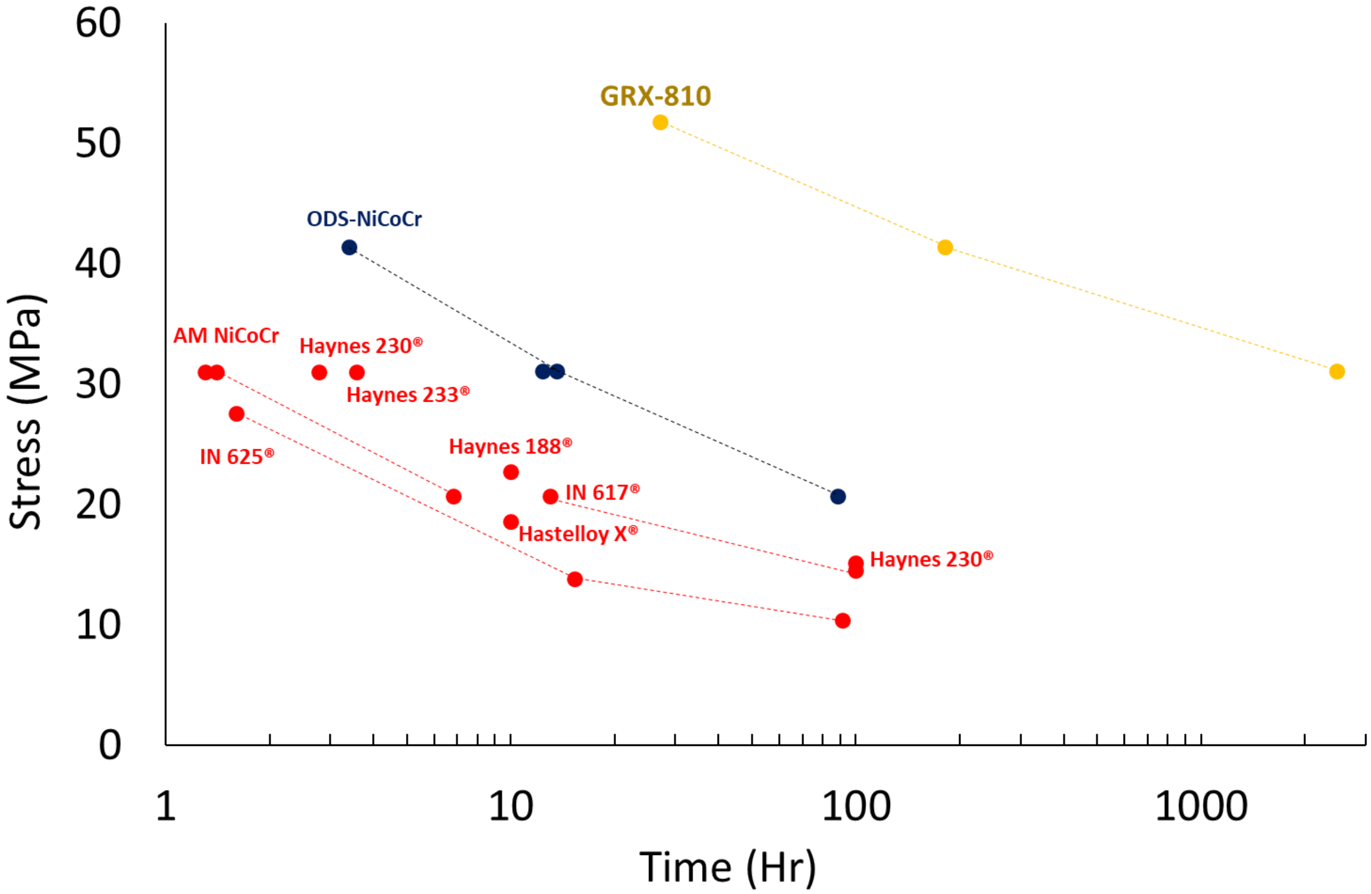
- **GRX-810 provided almost a 2000x improvement over AM NiCoCr in creep rupture life.**

Tensile Strength vs Density Comparison



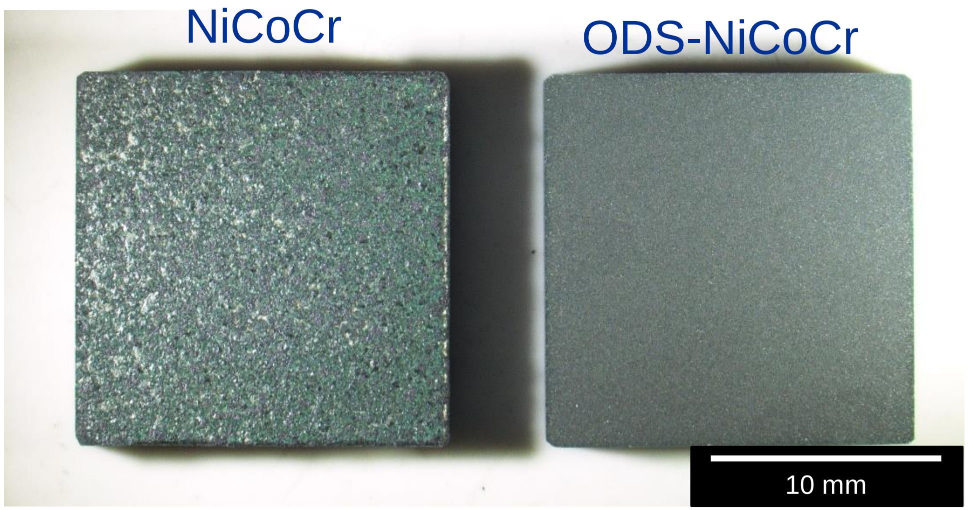
Scatter plot confirms the successful production of a ODS alloys using AM

Creep Rupture Lives Comparison- 1093°C



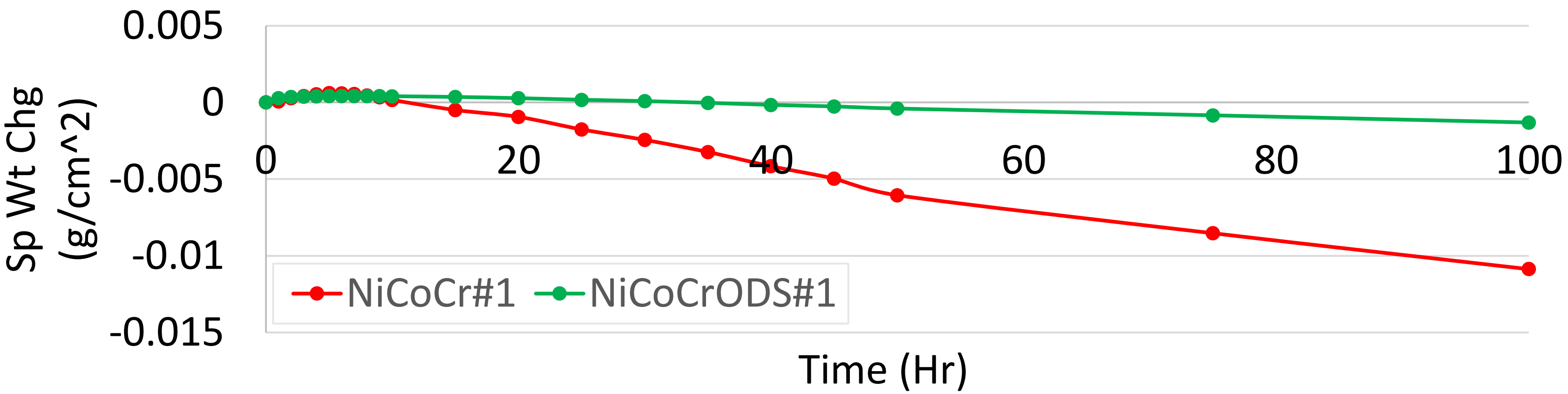
Alloy GRX-810 provides magnitudes of order better creep strength compared to conventional superalloys at 1093°C.

Oxidation – 1100°C

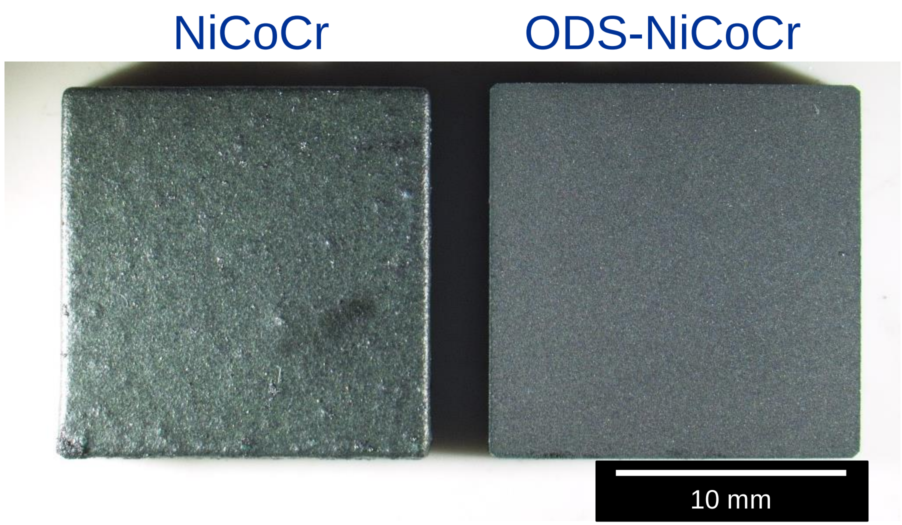


Left: Surface oxidation between NiCoCr and ODS-NiCoCr

Below: Mass change between ODS and non-ODS NiCoCr at 1100°C

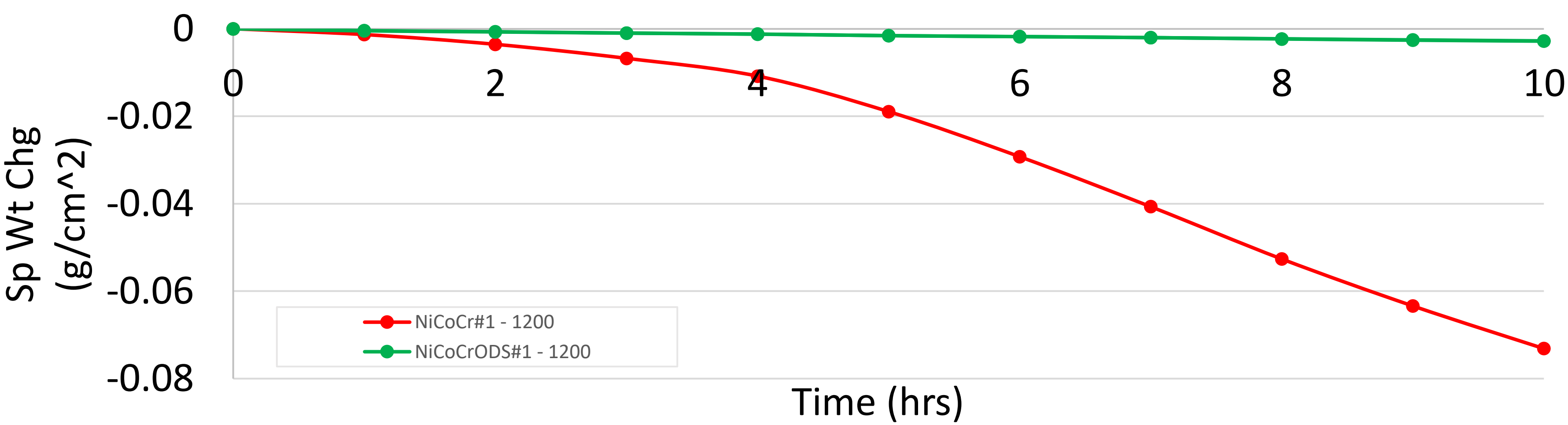


Oxidation – 1200°C



Left: Surface oxidation between NiCoCr and ODS-NiCoCr after 100hr-1100°C and 10hr-1200°C

Below: Mass change between ODS and non-ODS NiCoCr at 1200°C



GRX-810 - Scale Up

- Most work presented was coated with a coating rate of 1Kg per hour using two in-house lab-scale mixers.
- We have optimized the mixing parameters and improved the coating rate to 10kg per hour using the same machines.
- Recently, mix parameter trials were performed at Resodyn on their pilot scale mixer.
  - Improved coating rate to 150kg per hour
  - Can possibly improve to 300kg per hour
  - Larger mixers or continuous mixing are possible scale up routes.
- GRX-810 print parameters were recently optimized on an EOS-M280, improving the build rate five-fold.
- 1700lbs of LPBF and DED size cut GRX-810 powder was recently purchased for faster evaluation and component testing. Should arrive early September.

Conclusions

Novel Fabrication Technique for Oxide Dispersion Strengthened (ODS) Alloys:

- Collaboration between designers and material scientists produced the catalyst for this development.
- AM can be leveraged to economically produce oxide dispersion strengthened alloys that until now had been cost prohibitive.
- The advanced dispersion coating technique can successfully coat metallic powders with nano-scale ceramics.
- We believe this new manufacturing technique combined with MPEAs opens a new alloy design space for future high temperature alloys

GRX-810 Development and Results:

- Thermodynamic models correctly predicted a stable solid solution matrix phase for GRX-810.
- SEM and TEM characterization confirms a uniform dispersion of nano-scale oxides throughout the alloy GRX-810 build
- High temperature mechanical testing of GRX-810 reveals surprising and superior results compared to previous ODS alloys produced within this project and conventionally manufactured high temperature alloys.
- Moving forward: explore DED AM production of ODS parts and test all important properties such as fatigue, flammability, oxidation, etc. for GRX-810

Acknowledgments

Questions?

- NASA GRC ASG
- Dave Ellis
- Henry de Groh
- Quynhgiao Nguyen
- Paul Gradl
- Bob Carter
- Pete Bonacuse
- Chris Kantzos
- Cheryl Bowman
- Tim Gabb

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