GRX-810: A 3D Printable Alloy Designed for Extreme Environments

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ASM: Superalloy - Yesterday's Wonder Metals for the Future

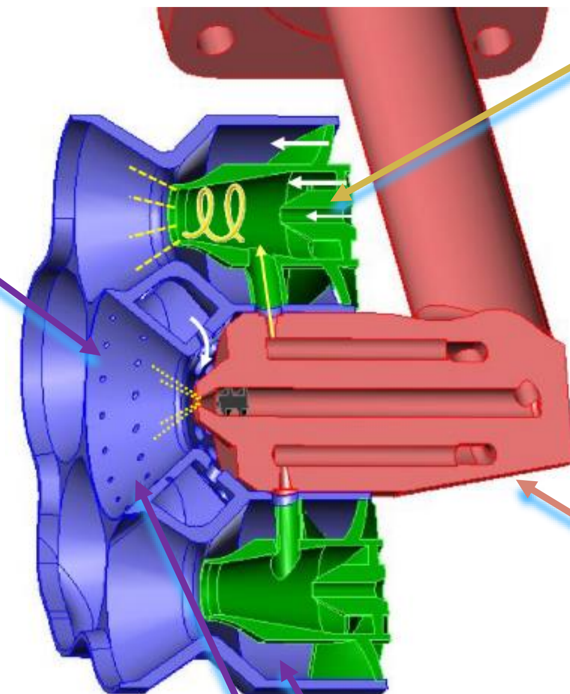
Acknowledgements: NASA's Aeronautics Research Mission Directorate (ARMD) – Transformational Tools and Technologies (TTT) Project Office and NASA's Space Technology Mission Directorate (STMD) Game Changing Development (GCD) Program under the optimized and Repeatable Components in Additive Manufacturing (ORCA) project

Background – NASA Application

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

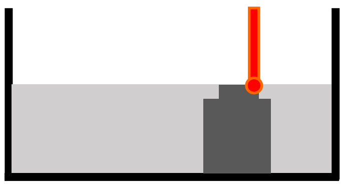
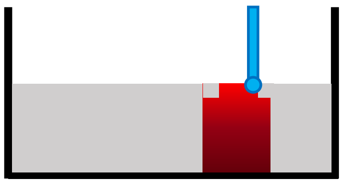
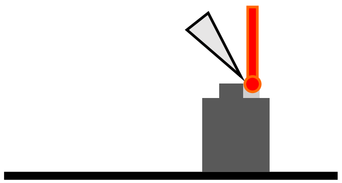
Proposed Solution: Develop a high ductility, high temperature material for an additively-manufactured (AM) combustor fuel nozzle and dome for supersonic aircraft (>1093°C (2000°F) operating temperature).

- 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

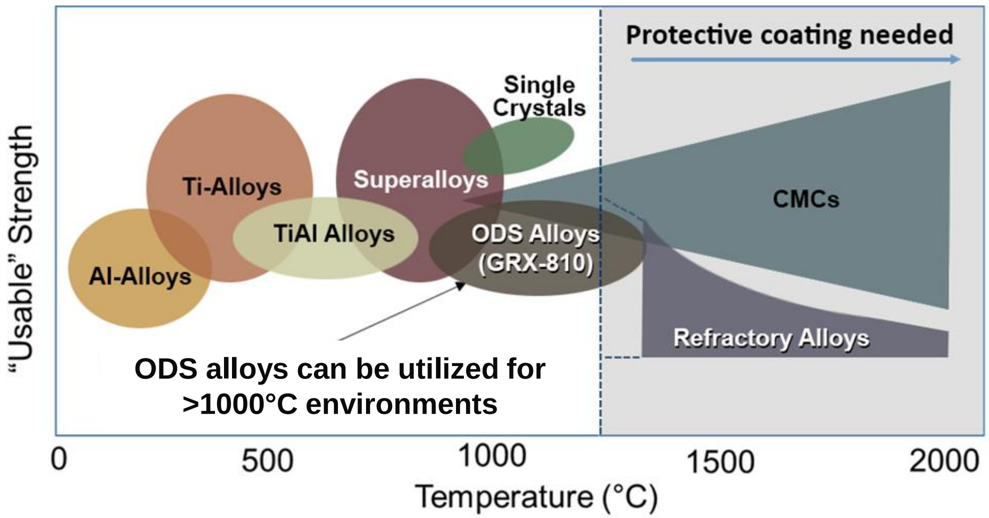
Process	Laser Powder Bed Fusion (L-PBF) 	Electron Beam Powder Bed Fusion 	Direct Energy Deposition (DED) 
Energy Source	Laser	E-Beam	Laser or E-Beam
Powder Bed	Yes	Yes	No
Power (W or kV)	50-1000 W	**30-60kV**	100-2000 W
Max Build Size (mm)	500 x 280 x 320	500 x 280 x 320	**2000 x 1500 x 750**
Material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

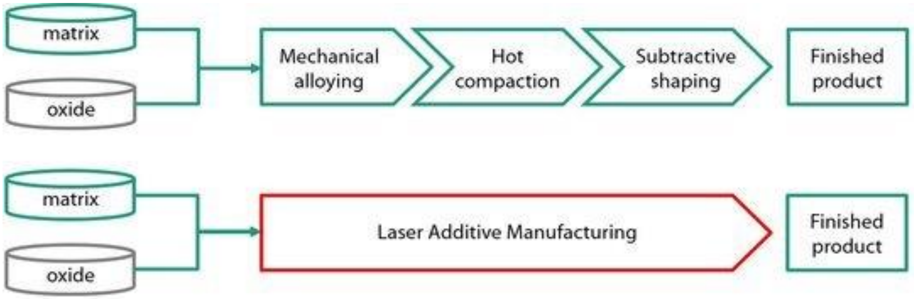
- Refractory metals
- Carbon-Carbon composites
- CMC's
- Ni-base superalloys
- **Oxide dispersion strengthened (ODS) alloys**



Inspired by Andy Jones. ODS alloy Development.

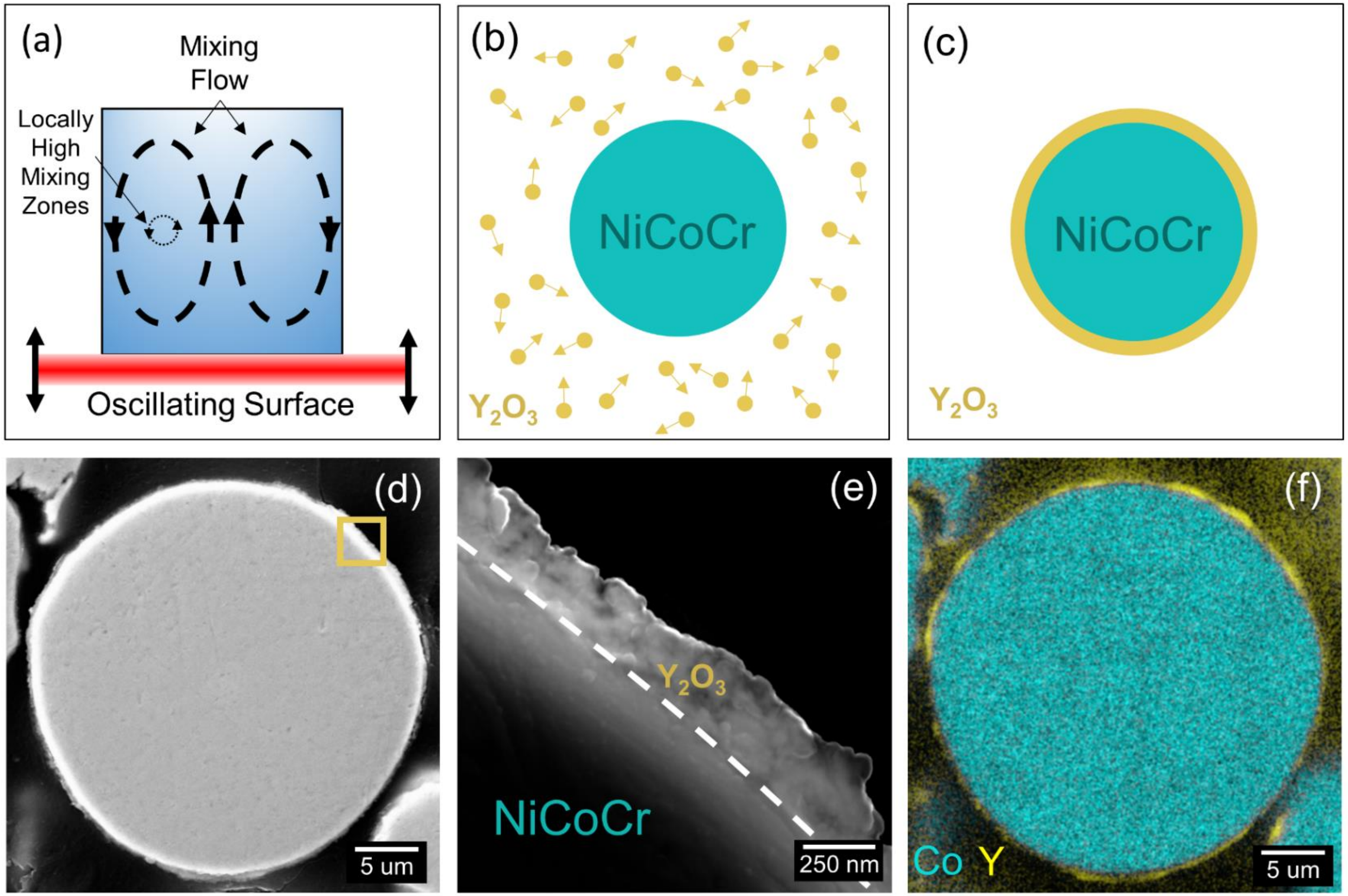
(ODS) alloys offer higher temperature capabilities compared to Ni-base superalloys. However, it has been a challenge to produce ODS alloys through conventional manufacturing methods.

Conventional Manufacturing vs AM



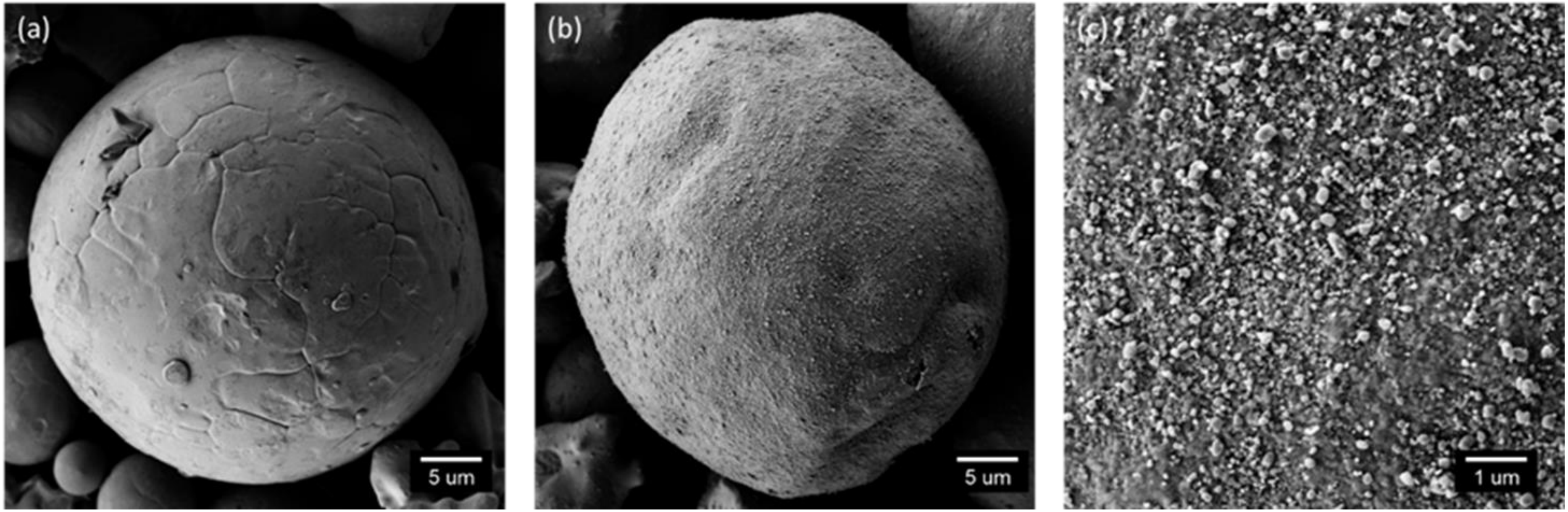
Can additive manufacturing improve ODS alloy manufacturability?

Novel Fabrication Technique for Oxide Dispersion Strengthened (ODS) Alloys



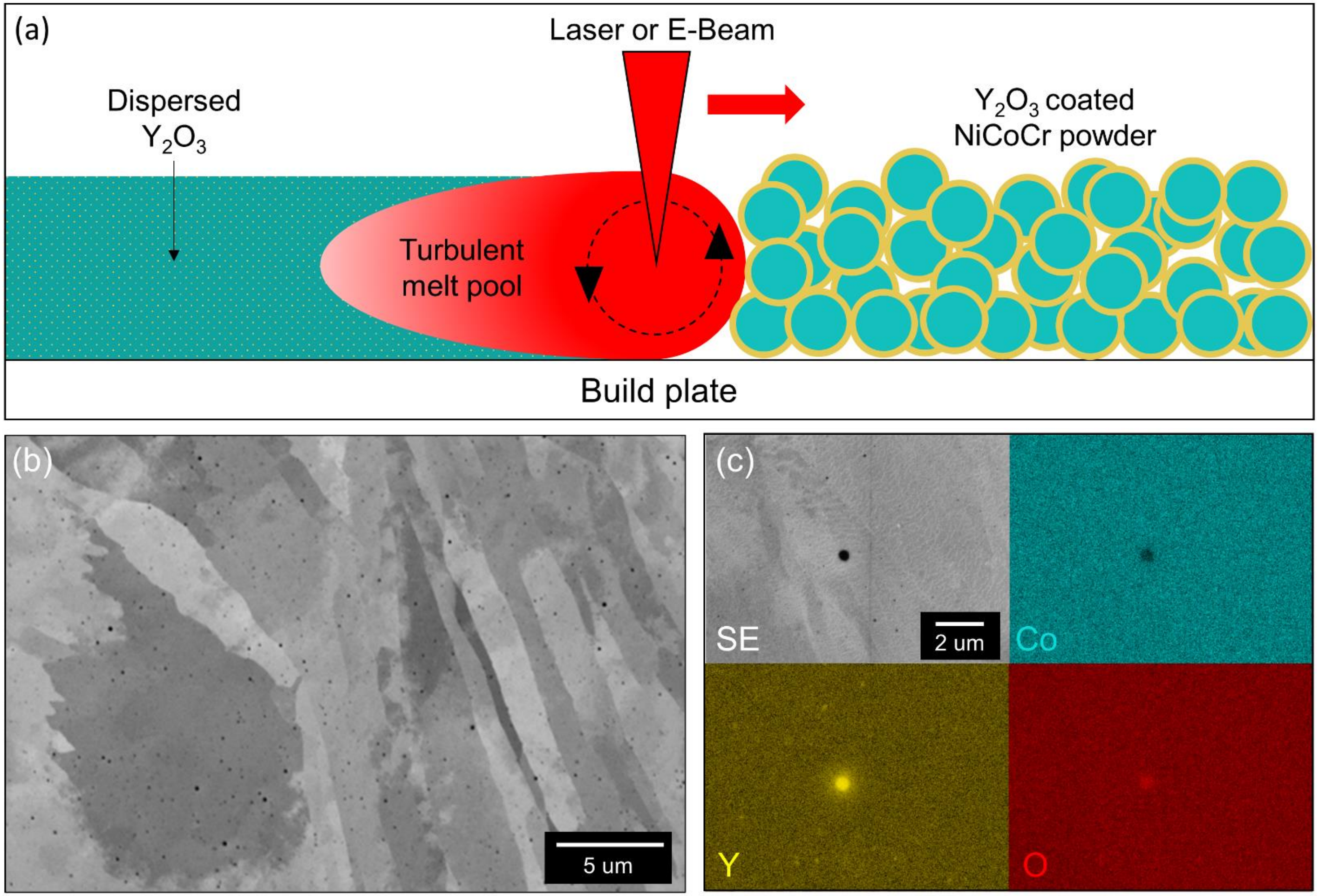
New high energy mixing technique successfully coats NiCoCr-base powders with 1 wt.% Y_2O_3.

Novel Powder Coating Technique



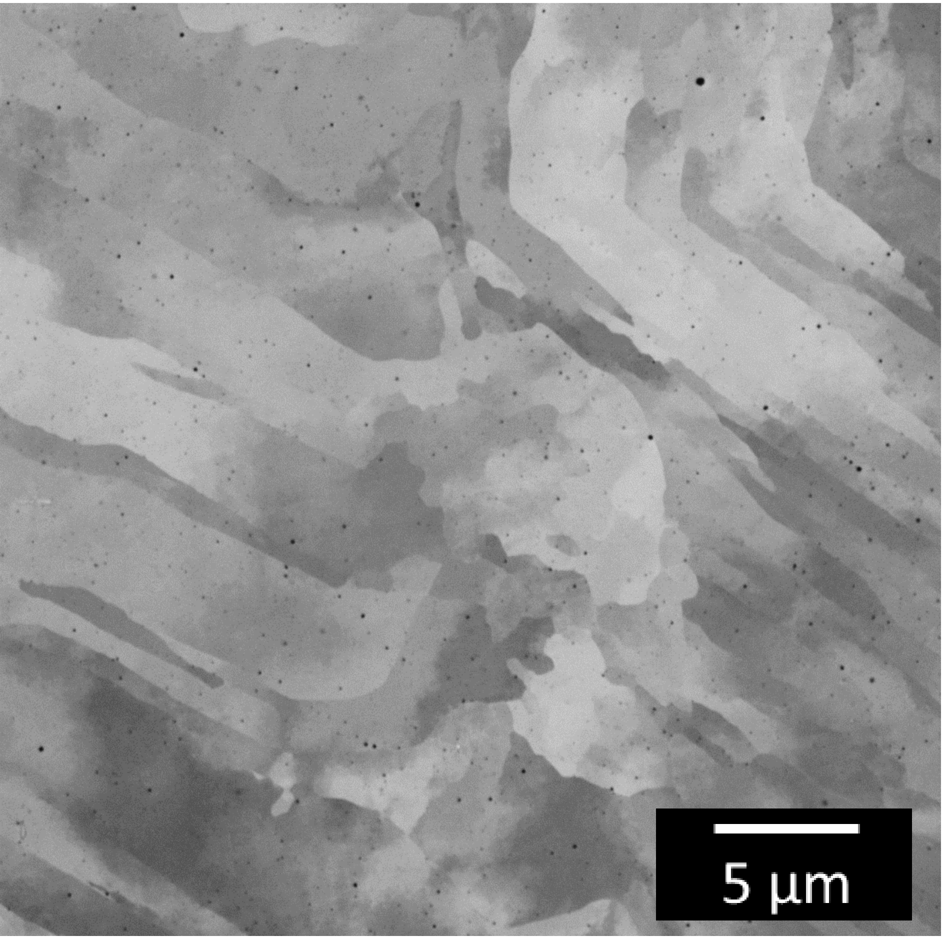
- The advanced dispersion coating (ADC) technique did not deform the metallic powder.
- The ADC technique fully coats the metallic powders with nano-scale oxides
- Both uncoated and coated powders qualitatively passed the Hall flow test.
- The technique does not affect the printability of the powder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

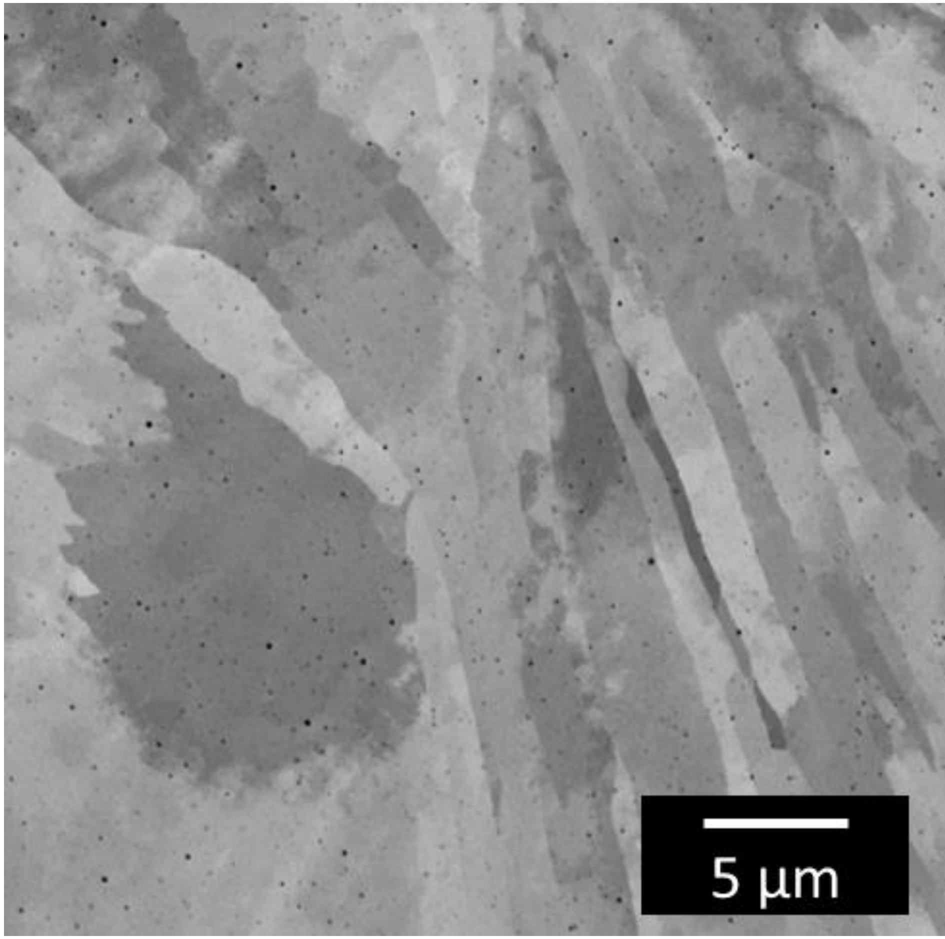
Leveraging L-PBF to Produce Oxide Dispersion Strengthened Alloys



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

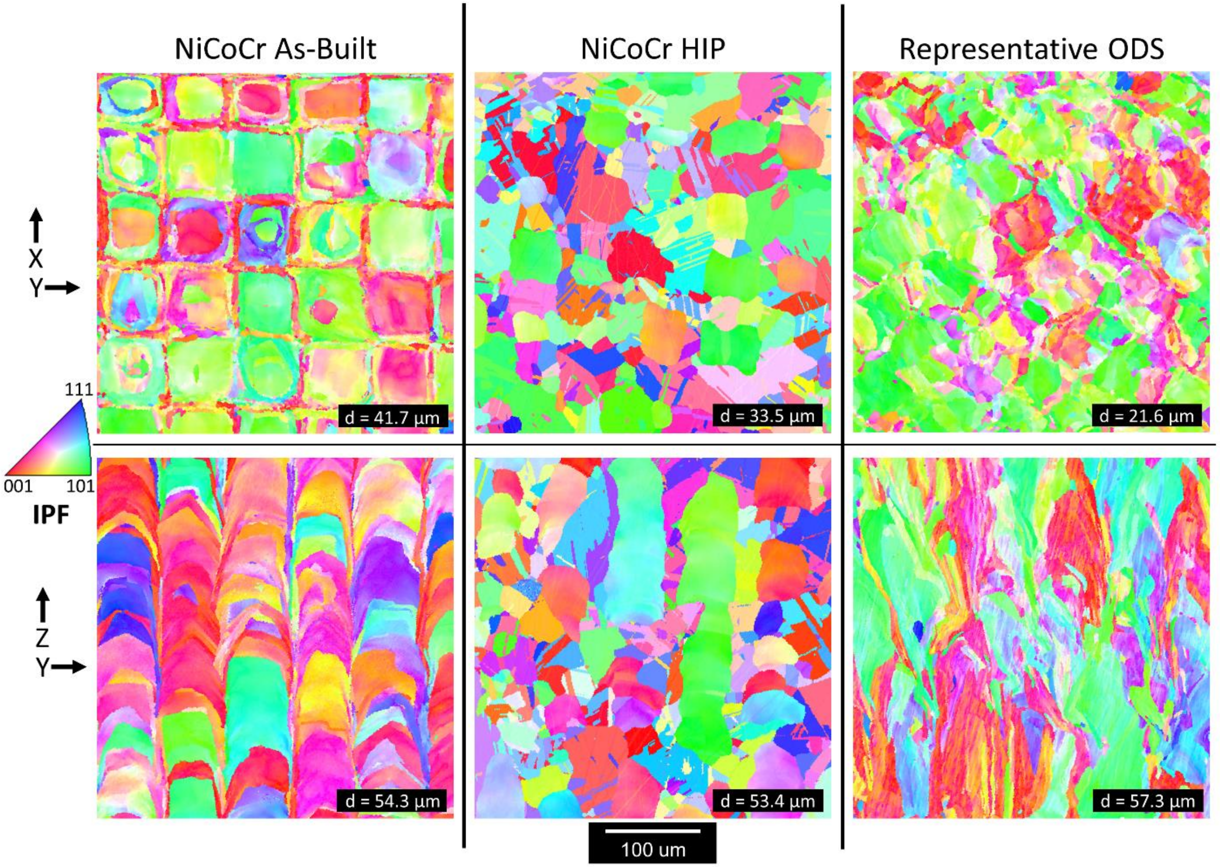
ODS-NiCoCr Microstructure





Nano-scale Y_2O_3 particles are randomly dispersed throughout microstructure.

Microstructure Analysis



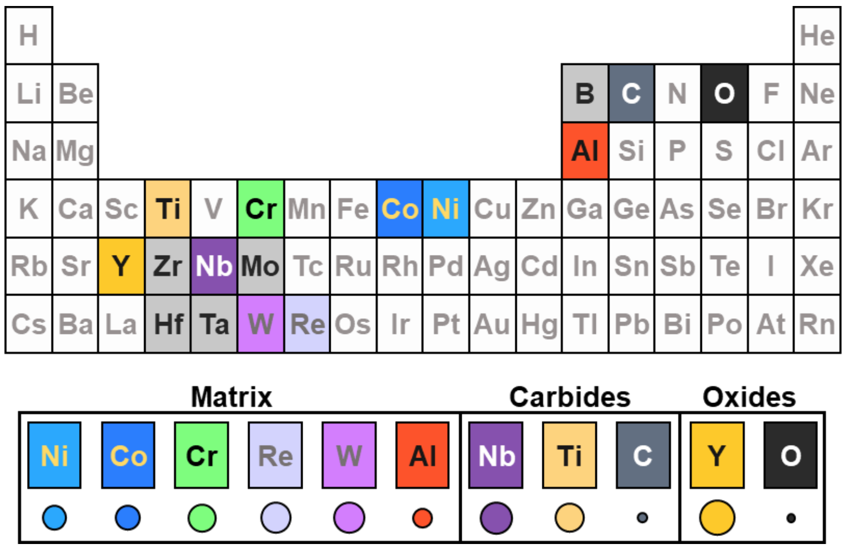
- Y_2O_3 particles have pinned the grain boundaries in the ODS AM builds.
- The HIP cycle successfully removed residual stresses for AM samples

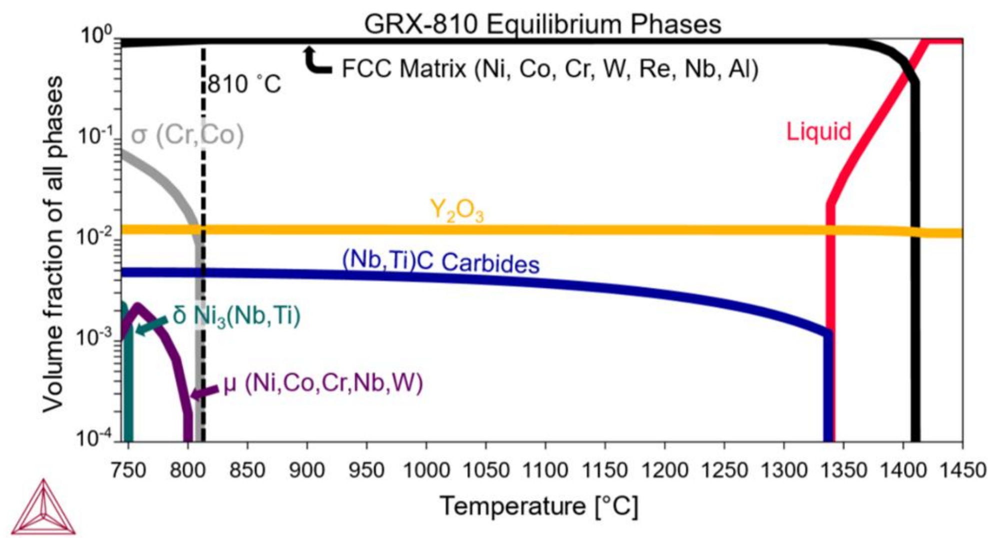
Residual Stress

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

Development of GRX-810 Composition

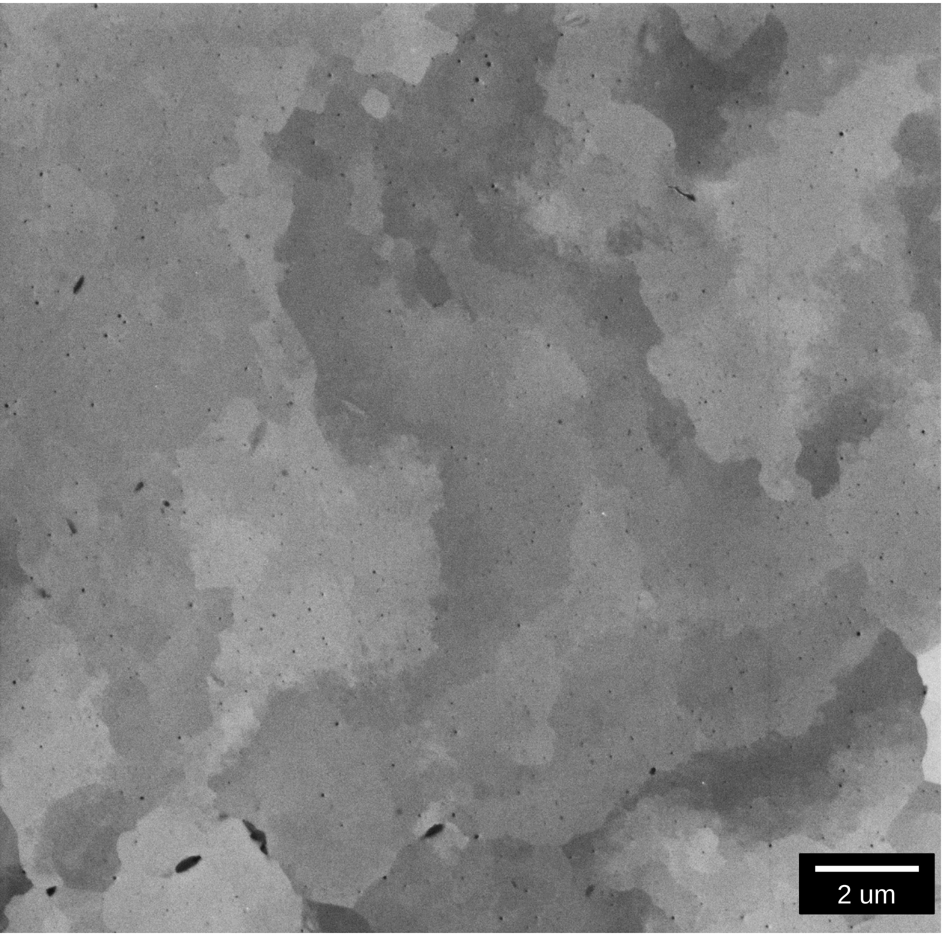
- Computational Alloy Design
- Balancing phases, properties, and manufacturability
- Final composition includes 9 elements + nano-oxides.
- Impossible to achieve through “trial and error” alloy design

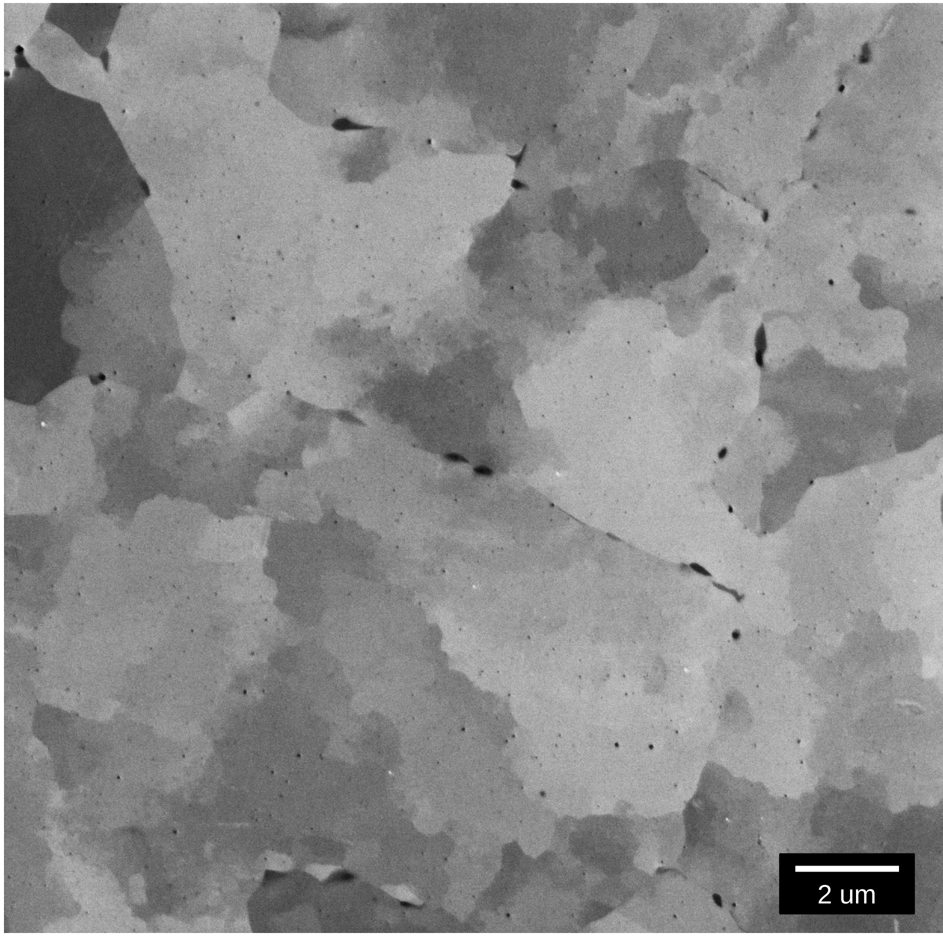




Alloy	Ni	Co	Cr	Re	Al	Ti	Nb	W	C
New composition (GRX-810)	Bal.	33	29	1.5	0.3	0.25	0.75	3.0	0.05

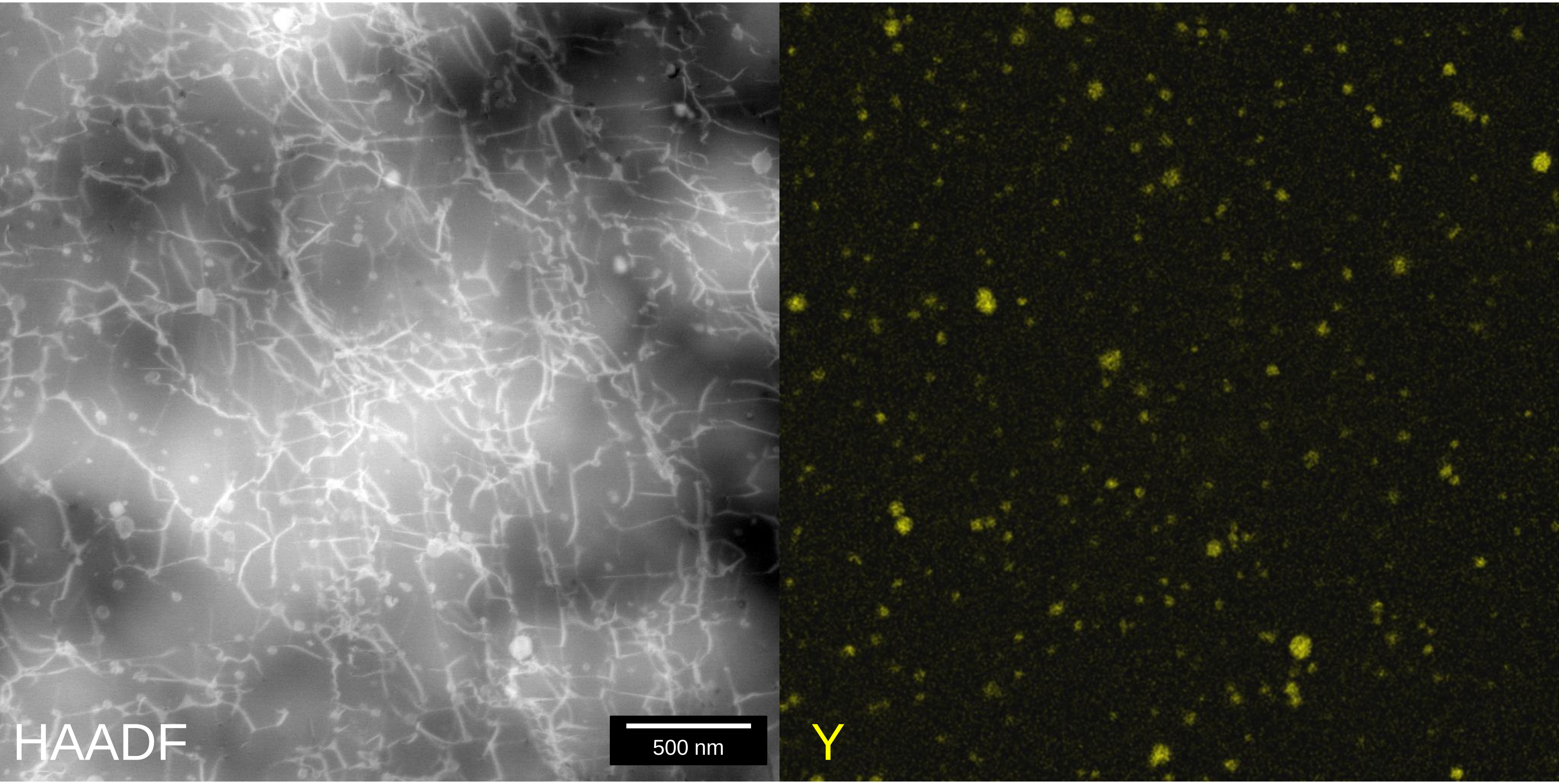
SEM – GRX-810 - HIPed





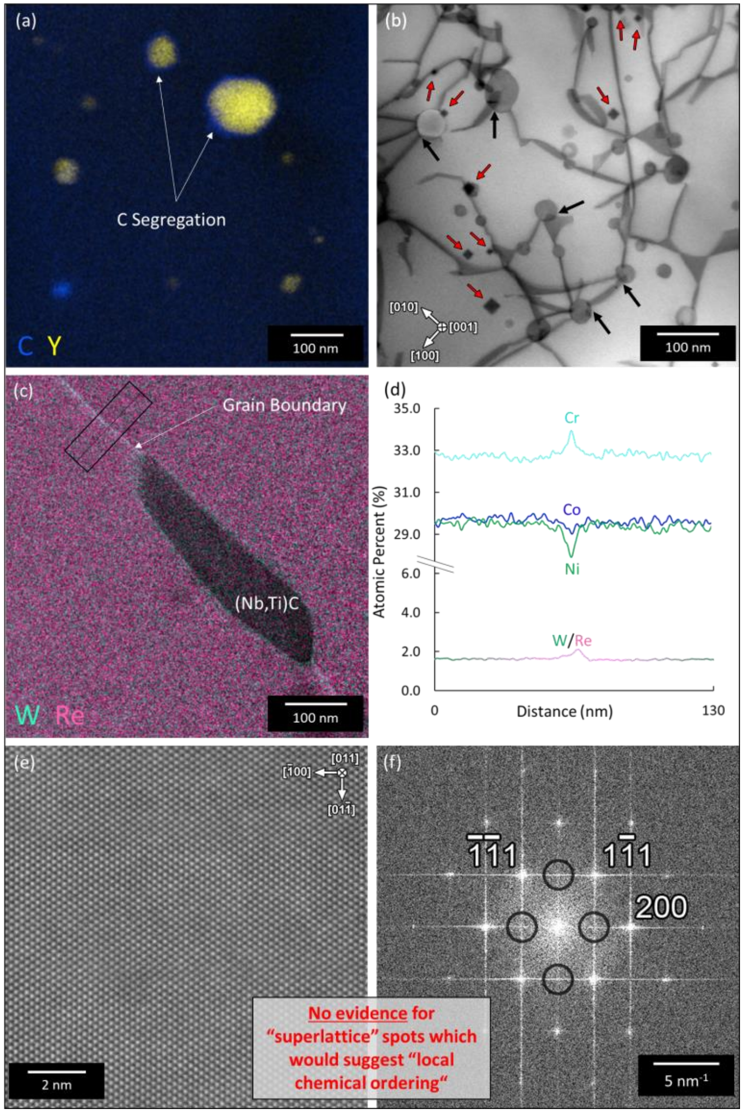
High resolution SEM reveals dispersed nano-oxides and grain boundary phases.

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.

STEM Analysis



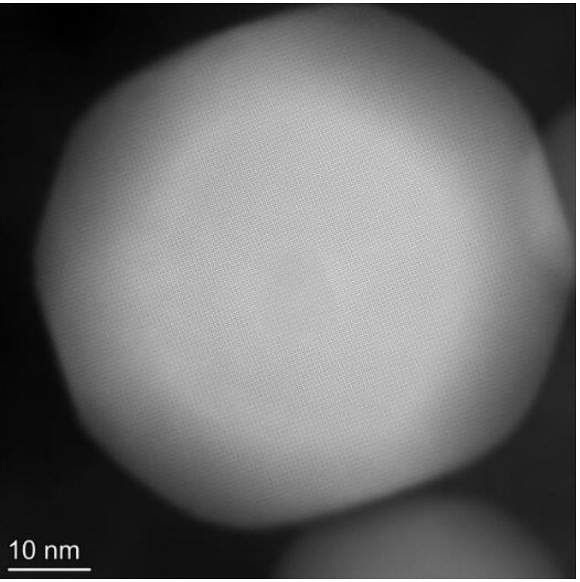
Top: STEM analysis revealed Carbon segregation at the oxide matrix interface. Top Right: Reveals dislocation oxide interactions.

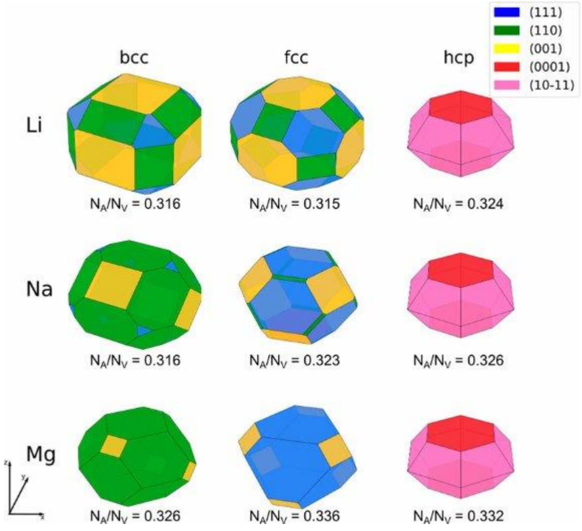
Middle: Solute segregation of W/Re/Cr along Grain Boundaries

Bottom: Diffraction from the [001] zone axis STEM image reveals that there is no local elemental ordering at the atomic level.

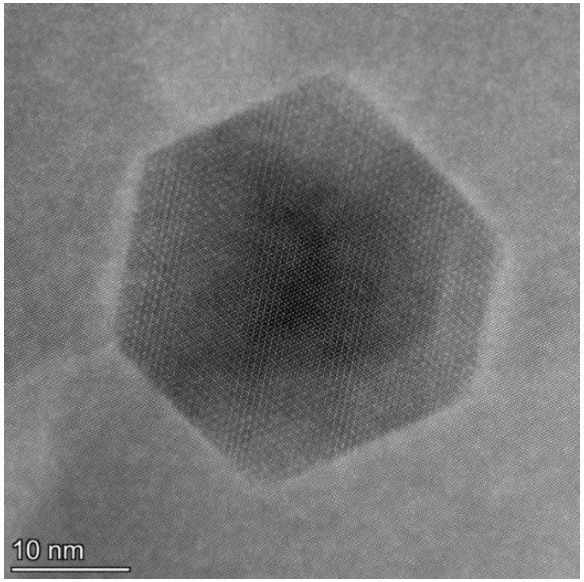
Oxide Comparison – NiCoCr and GRX-810

Cubic Y_2O_3 particle in NiCoCr-ODS





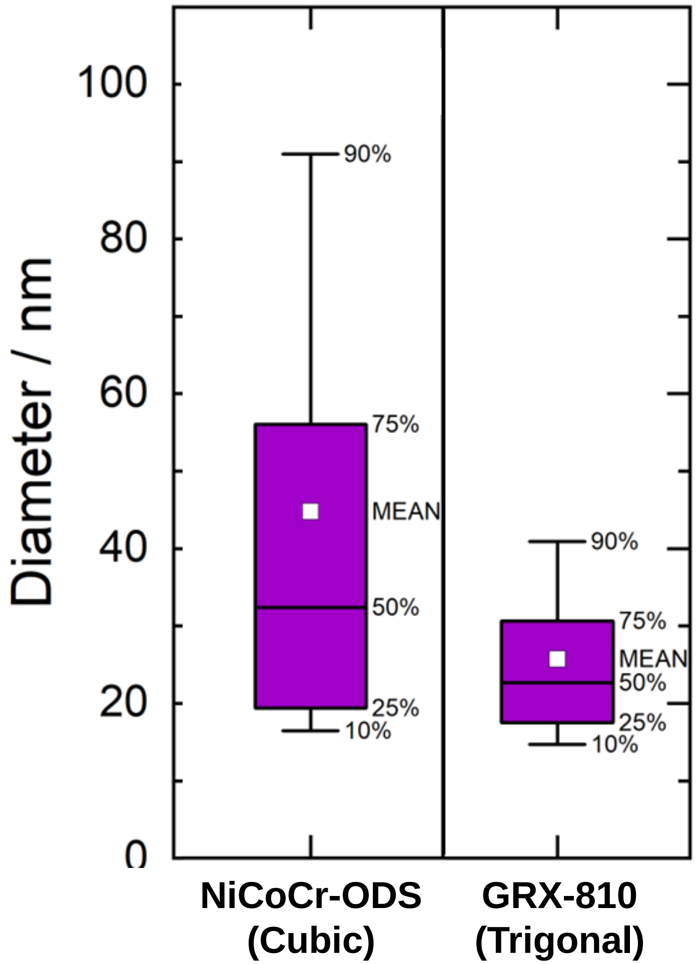
Trigonal Y_2O_3 particle in GRX-810



The oxides in GRX-810 are more faceted due to the difference in crystal structure compared to the cubic oxides in the NiCoCr-ODS material.

XRD analysis confirms the oxides were cubic prior to coating and printing.

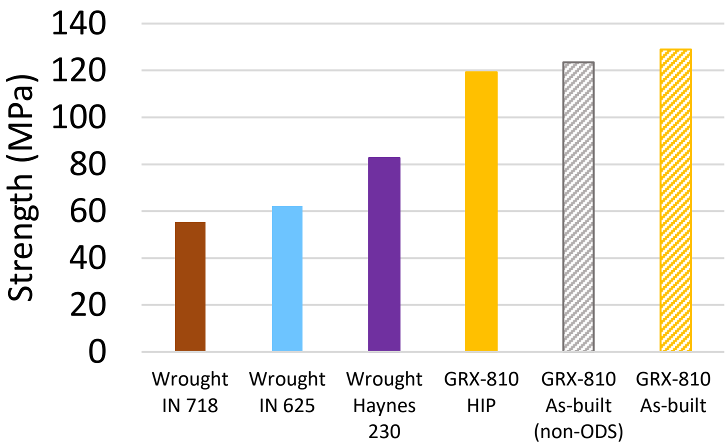
Trigonal Vs. Cubic Y_2O_3



- TEM analysis found the oxides in GRX-810 to be much finer compared to the oxides found in NiCoCr-ODS.
- The finer oxide size distribution may be a source for the significantly improved creep properties found in GRX-810 compared to NiCoCr-ODS

GRX-810 Tensile Overview

Tensile Strength Comparison 1093°C



- 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.

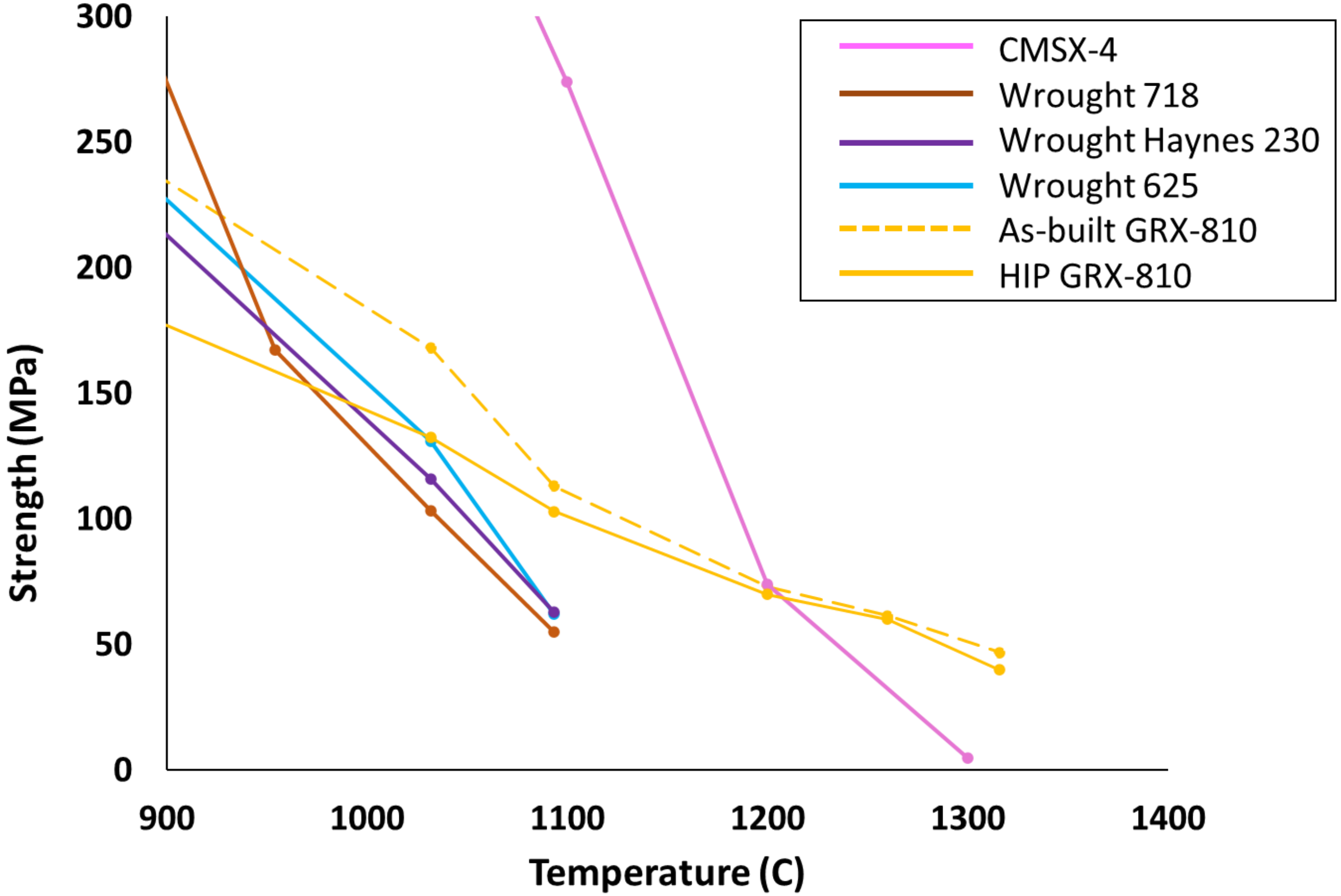
As-built GRX-810 Tensile Properties

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	710.2	527.4	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

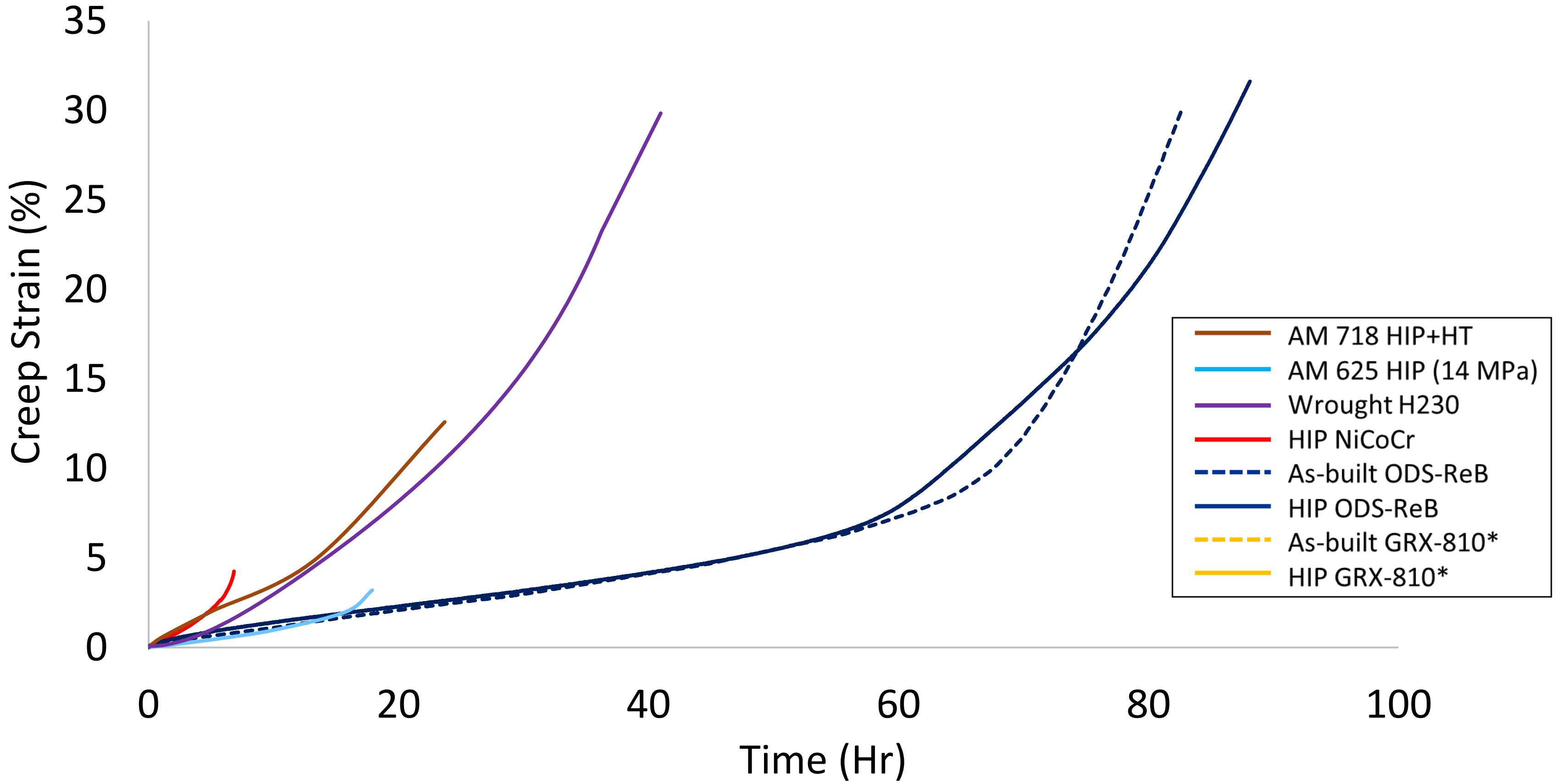
Temperature (C)	Tensile Strength (Mpa)	Yield Strength (Mpa)	Elongation
-195.6	1227.3	723.9	49
21.1	848.1	515.0	43
426.7	655.0	410.2	40
648.9	630.9	368.9	43
871.1	262.7	206.2	62
1000.0	164.1	161.3	44
1093.3	119.3	115.8	32

GRX-810 Tensile Overview

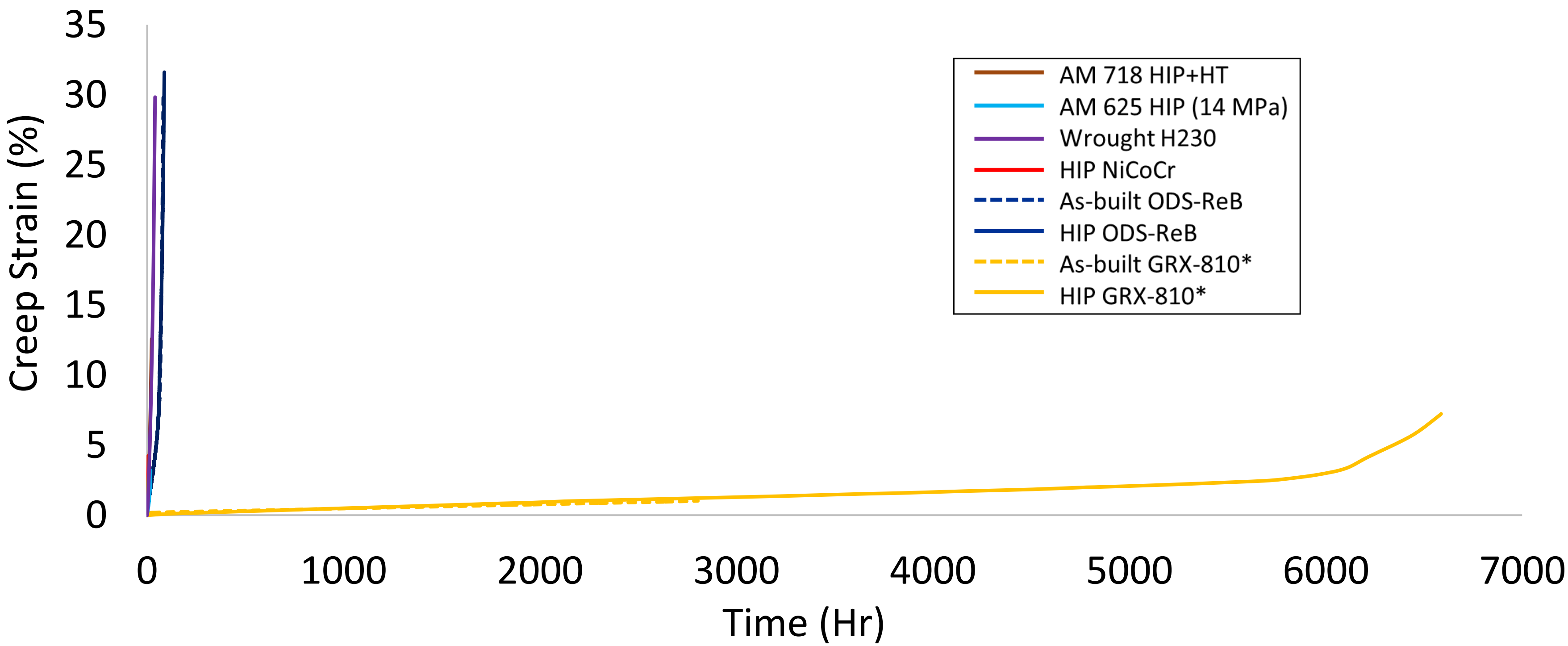


Above 900°C GRX-810 is stronger than most “printable” superalloys. Above 1200°C, GRX-810 is stronger than CMSX-4

Mechanical Results – 1093°C/20MPa Creep Rupture

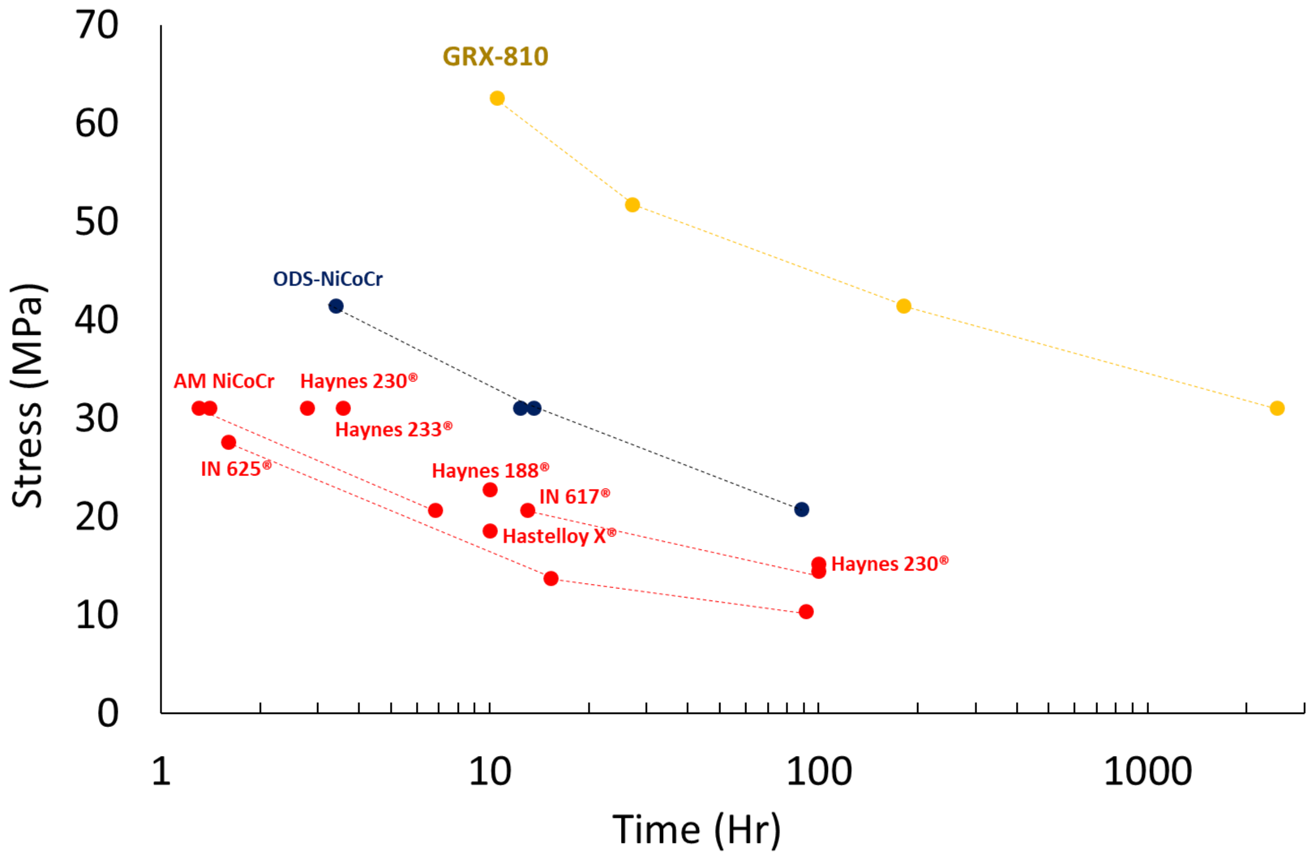


Mechanical Results – 1093°C/20MPa Creep Rupture



GRX-810 provides orders of magnitude improvements in creep rupture life at 1093°C compared to conventional superalloys 718 and 625.

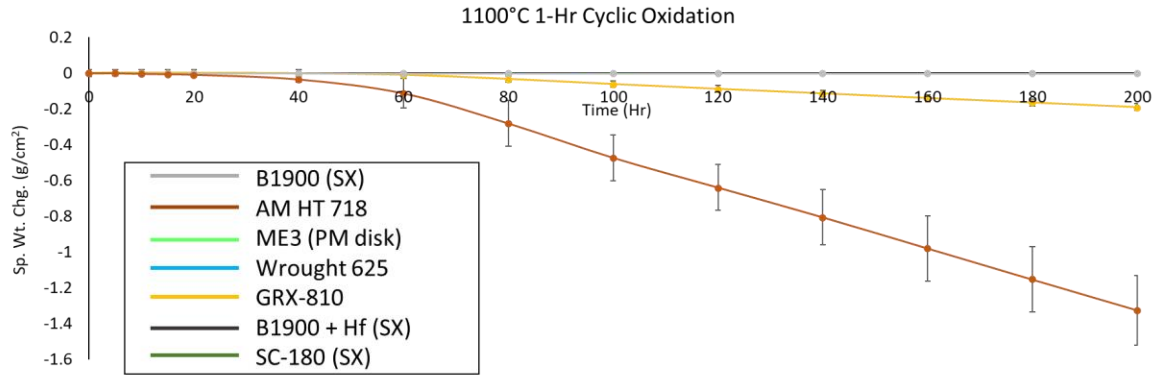
Creep Rupture Lives Comparison- 1093°C

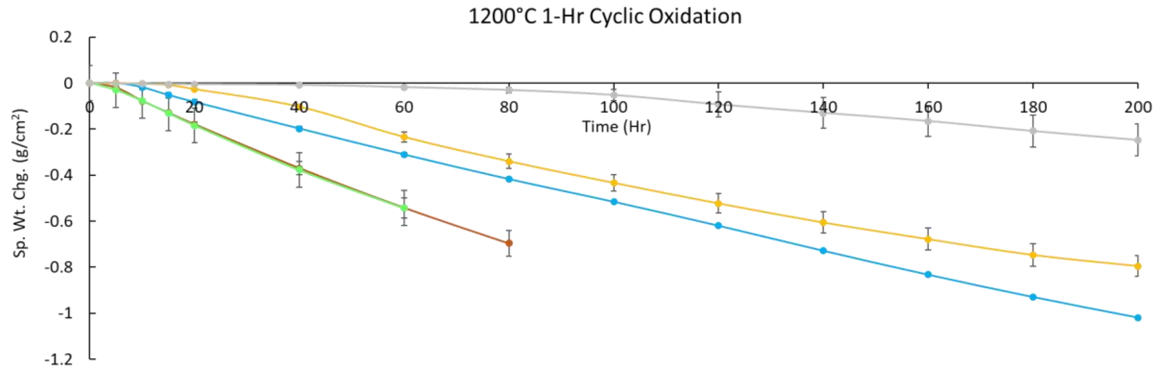


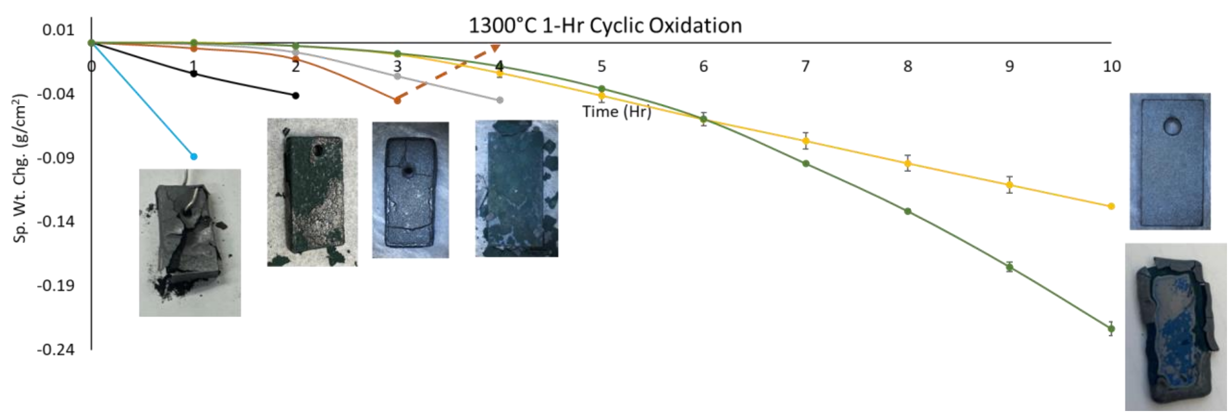
Alloy	NiCoCr	AM 718	AM 625*	Haynes 230	ODS-ReB	C-103 (Vacuum)	As-built GRX-810	HIP GRX-810
Time (Hr)	0.35	2.2	10	5	9	1170	2804	2122

Table: Time to reach 1 % Creep Strain at 20MPa. Note: Superalloy 625 test was performed at 14 MPa.

Oxidation

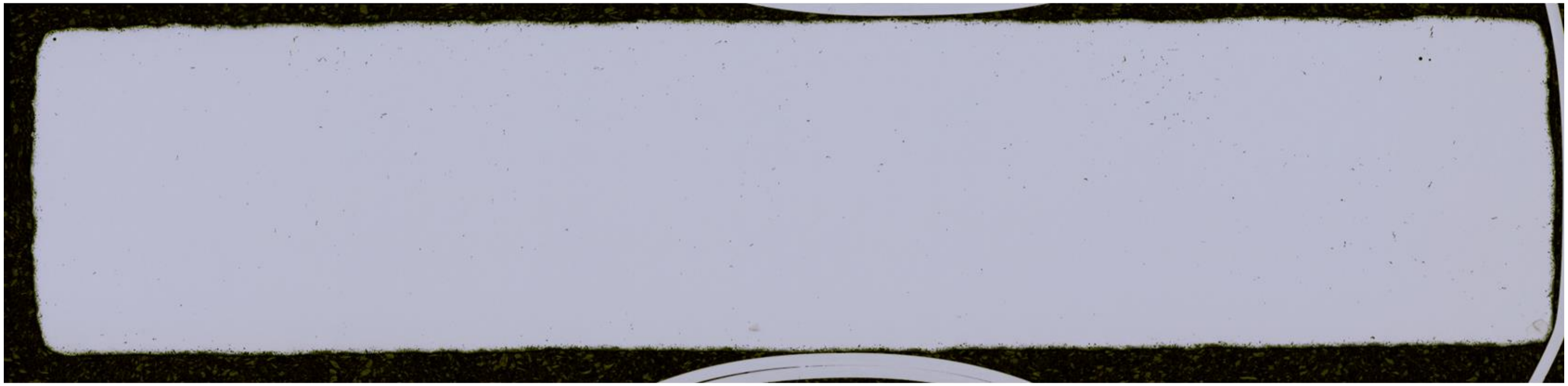

1100°C 1-Hr Cyclic Oxidation
Sp. Wt. Chg. (g/cm²)
0.2
0
-0.2
-0.4
-0.6
-0.8
-1
-1.2
-1.4
-1.6
0
20
40
60
80
100
120
140
160
180
200
Time (Hr)
B1900 (SX)
AM HT 718
ME3 (PM disk)
Wrought 625
GRX-810
B1900 + Hf (SX)
SC-180 (SX)


1200°C 1-Hr Cyclic Oxidation
Sp. Wt. Chg. (g/cm²)
0.2
0
-0.2
-0.4
-0.6
-0.8
-1
-1.2
0
20
40
60
80
100
120
140
160
180
200
Time (Hr)


1300°C 1-Hr Cyclic Oxidation
Sp. Wt. Chg. (g/cm²)
0.01
-0.04
-0.09
-0.14
-0.19
-0.24
0
1
2
3
4
5
6
7
8
9
10
Time (Hr)

Oxidation – 1300°C

GRX-810 – 10 cycles



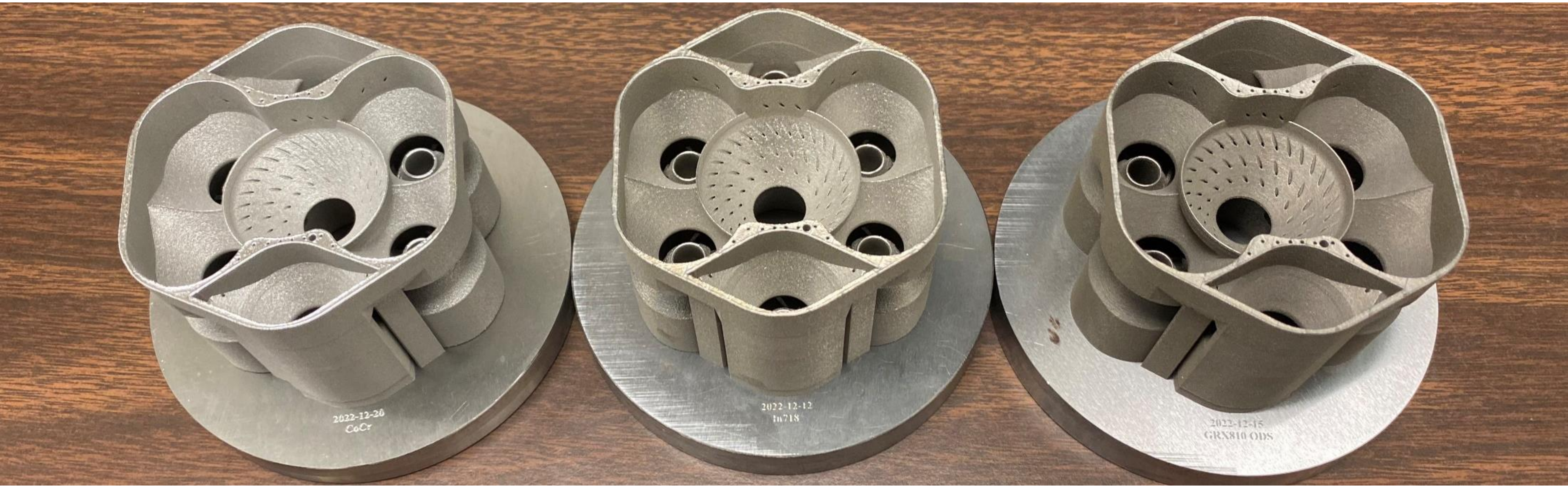
718 – 7 cycles

1 mm

The oxidation and microstructural stability of GRX-810 is notable up to 1300°C

Oxide Dispersion Strengthened Combustor Dome

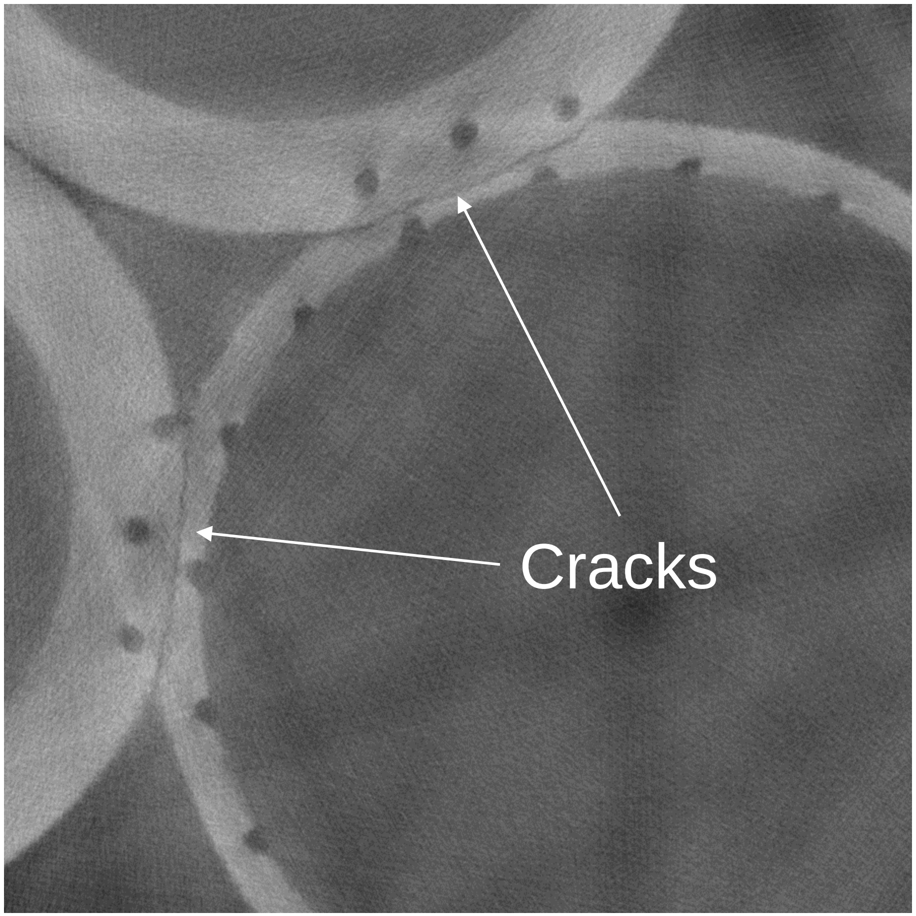
CoCr IN718 GRX-810



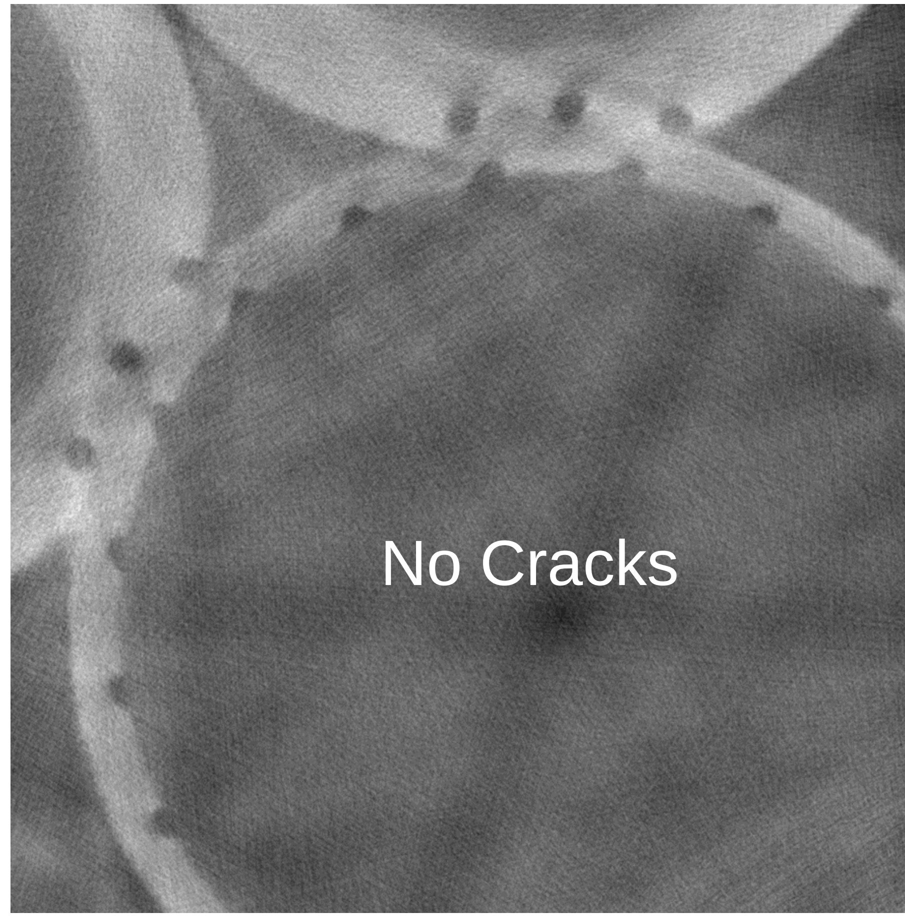
Three combustor domes were printed and tested at CE-13 using test conditions that resulted in crack issues previously.

Combustor Dome Study – CT Scan

CoCr



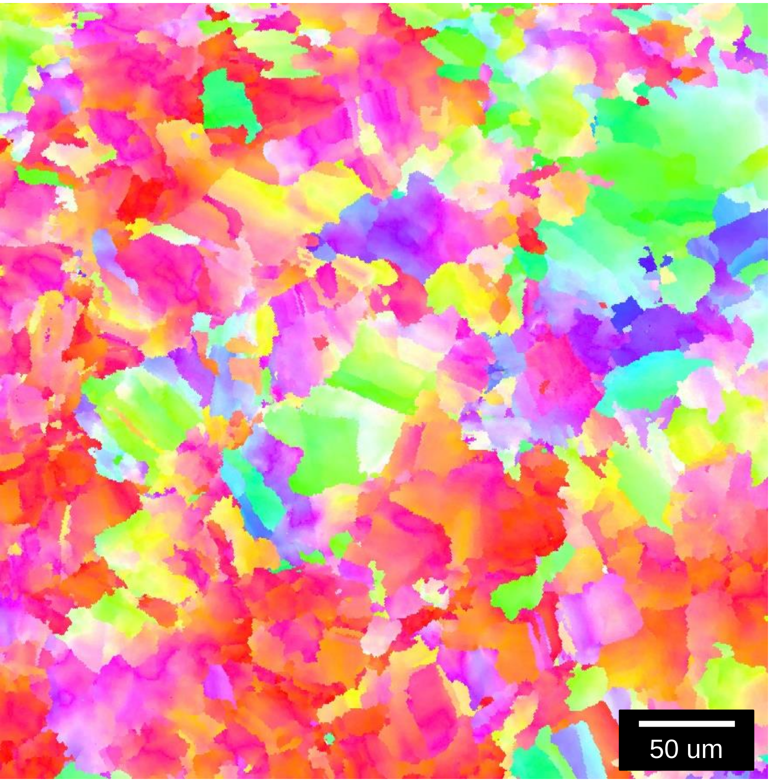
GRX-810



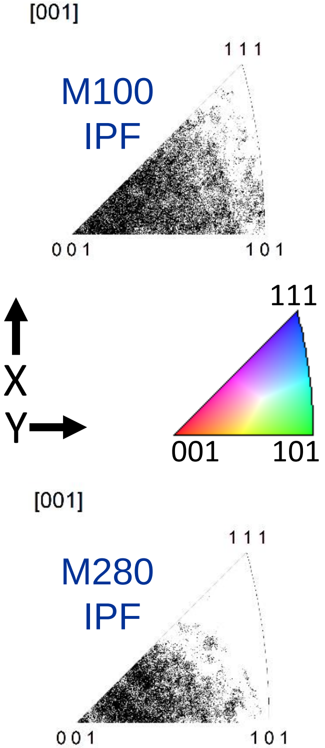
GRX-810 dome completed the test series without any cracks forming.

EBSD – As-Built

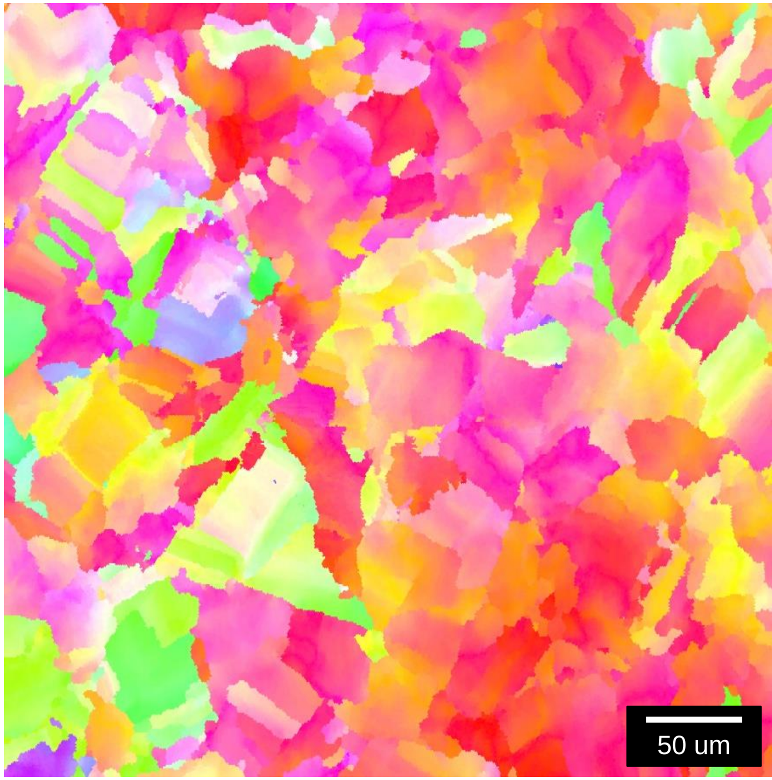
GRX-810 M100 Original



Average Grain size: 39um



GRX-810 M280

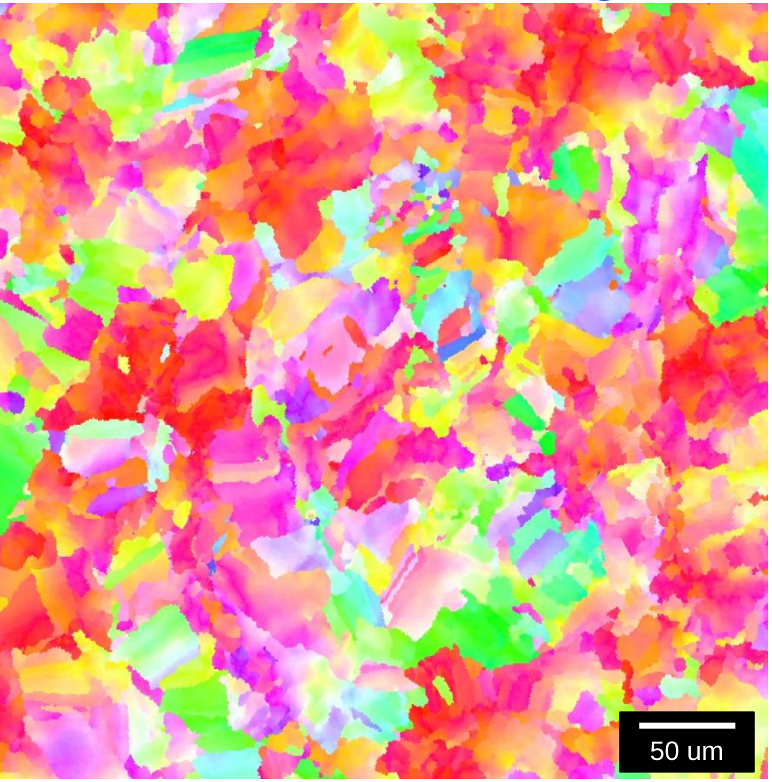


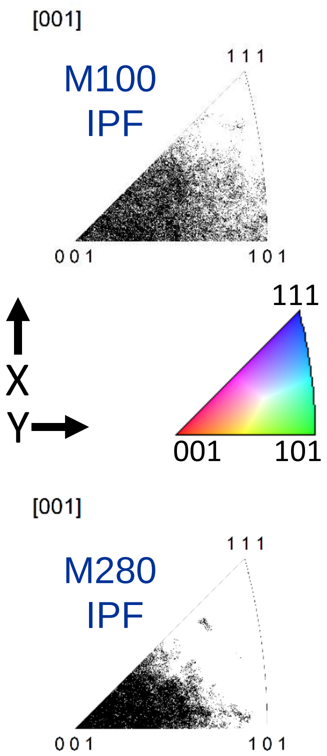
Average Grain size: 59um

Larger grain structure in M280 builds. Both exhibit an [001] texture along build axis.

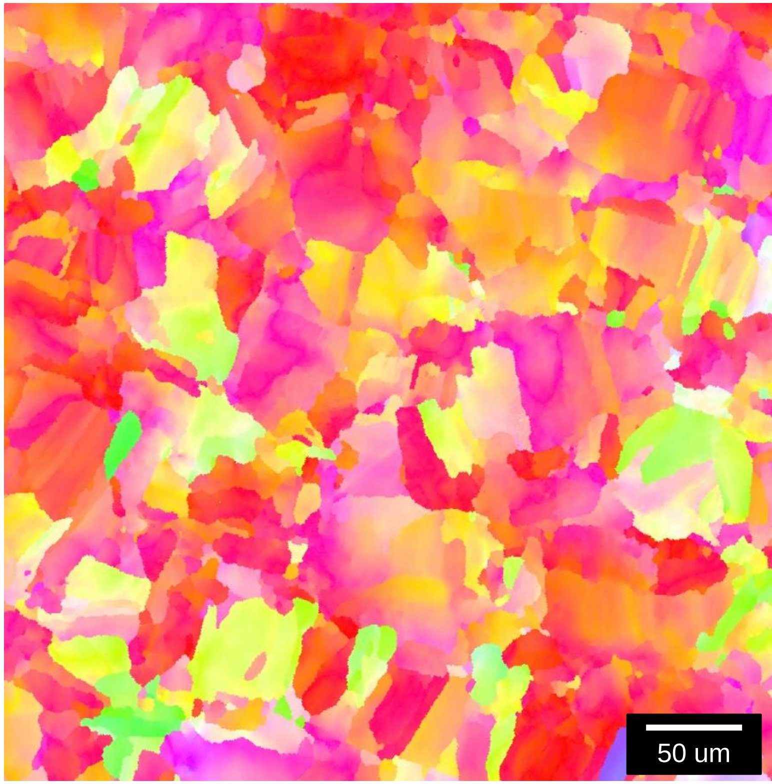
EBSD – HIP

GRX-810 M100 Original





GRX-810 M280



Average Grain size: 34um

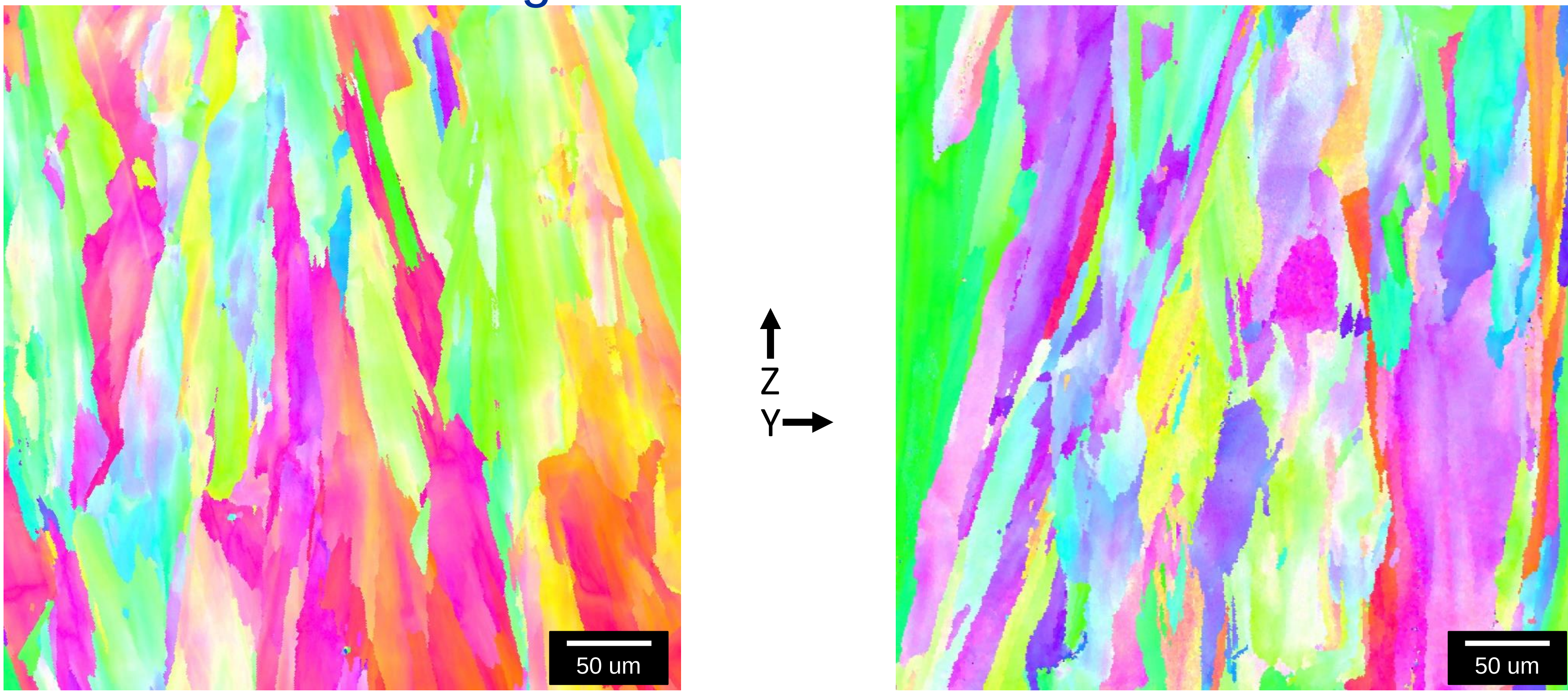
Average Grain size: 45um

Grain structure larger in M280 builds. Both exhibit an [001] texture along build axis. M280 build did not recrystallize confirming a stable ODS microstructure.

EBSD – As-Built

GRX-810 M100 Original

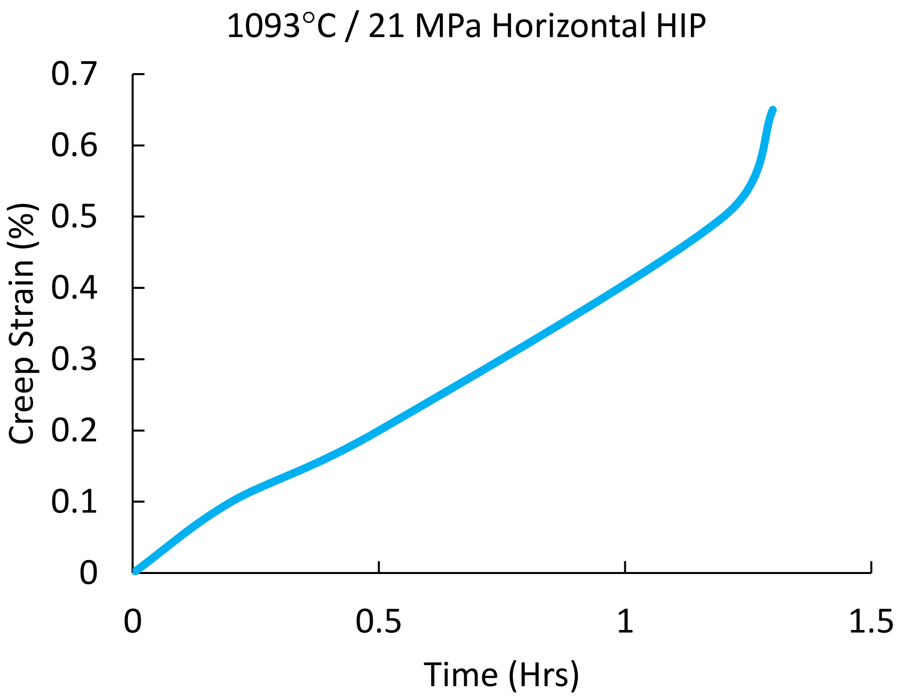
GRX-810 M280



Average grain size between 70-80um for both samples. This suggests that the M280 builds may exhibit less anisotropy compared to the original GRX-810 samples

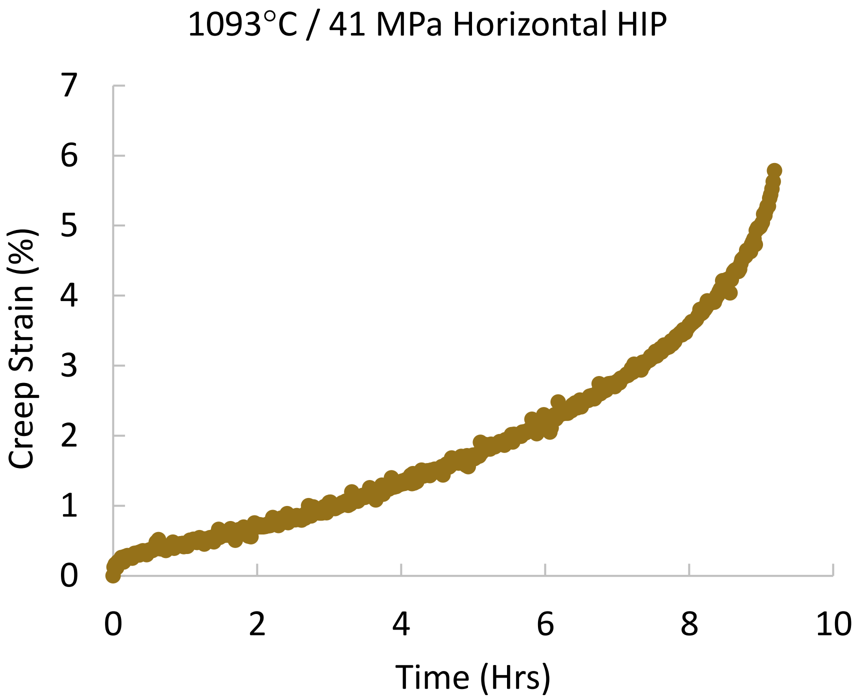
GRX-810 Creep Update

NiCoCr-ODS



Severe creep ductility in NiCoCr-ODS

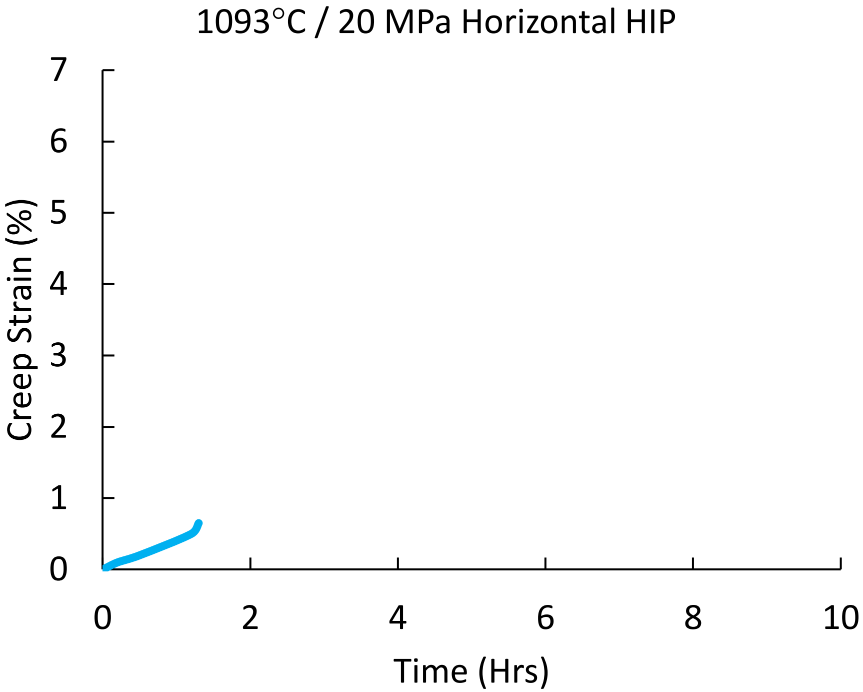
GRX-810



GRX-810 overcomes the severe creep ductility issue that limited the application space for ODS alloys.

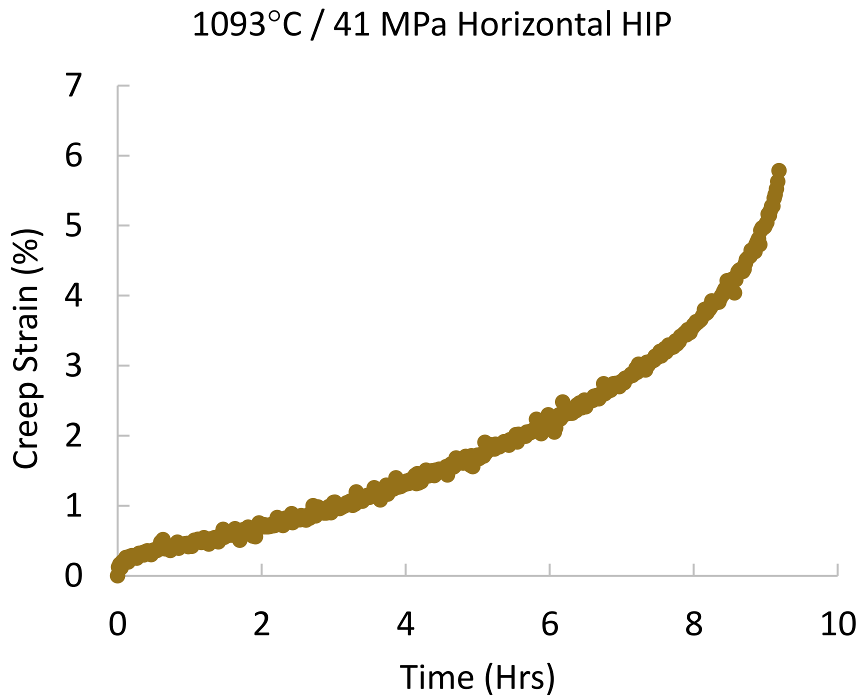
GRX-810 Creep Update

NiCoCr-ODS – 20 MPa



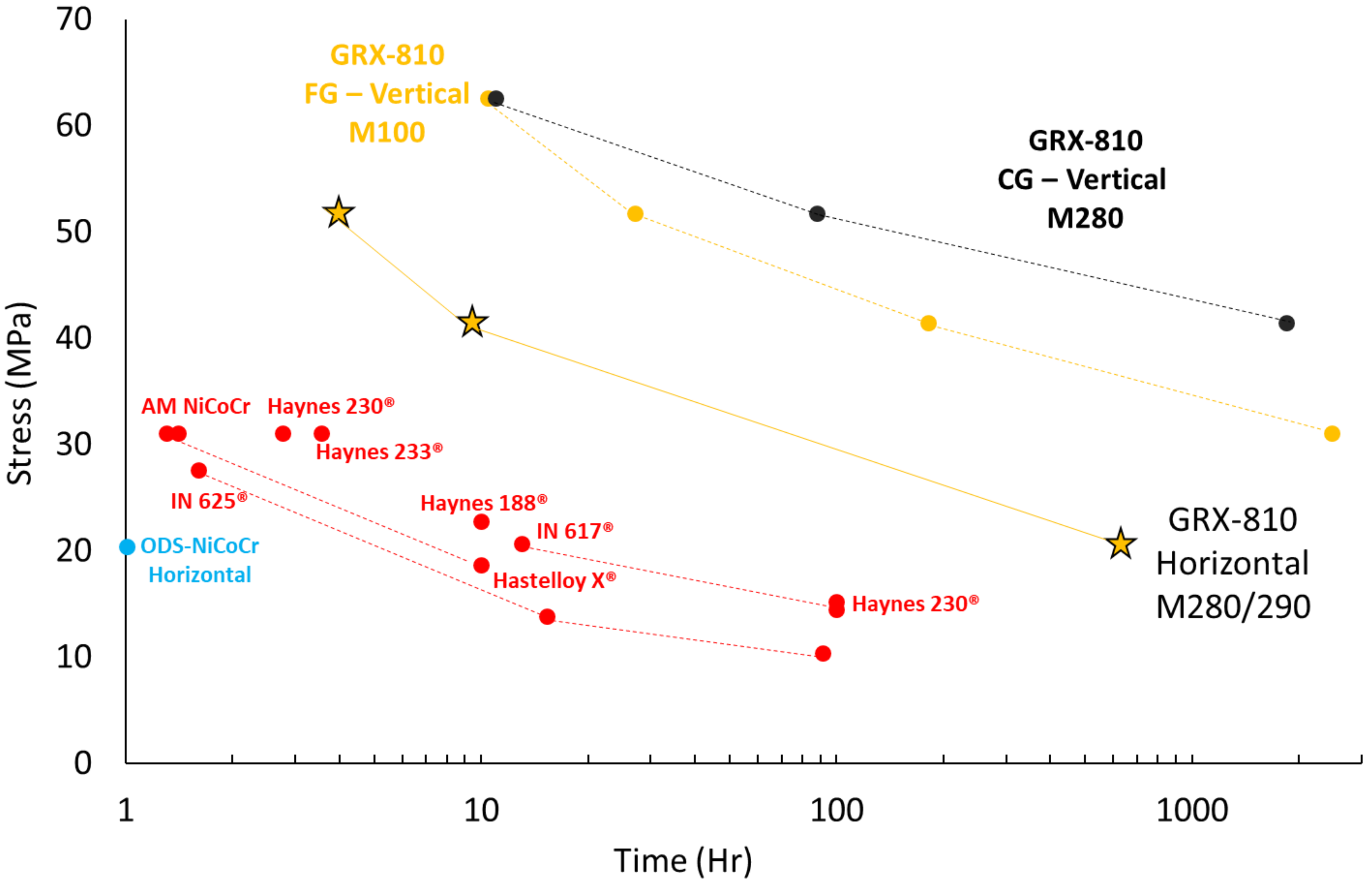
Severe creep ductility in NiCoCr-ODS

GRX-810 – 41 MPa

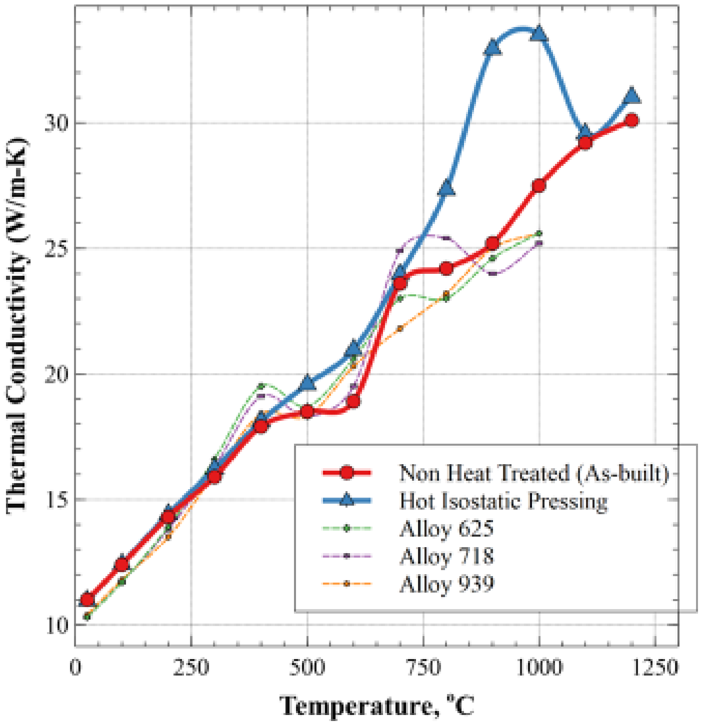


GRX-810 overcomes the severe creep ductility issue that limited the application space for ODS alloys.

GRX-810 Creep Overview



GRX-810 Thermal Properties



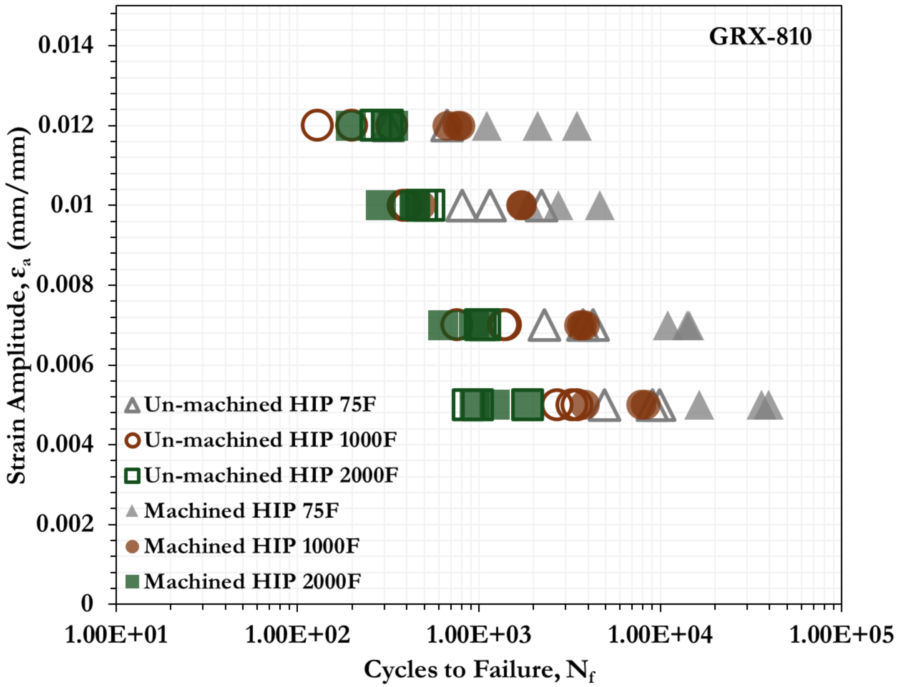
Temperature (°C)	CTE-P-28 [04-0037QY]	CTE-A-25 [04-0037QZ]
100	11.8	10.6
200	12.9	12.3
300	13.5	13.0
400	13.9	13.5
500	14.3	13.9
600	14.7	13.9
700	15.4	14.4
800	16.0	15.0
900	16.5	15.5
1000	17.2	16.1
1100	17.9	16.8
1200	18.5	17.4
1300	19.0	17.9

Table No. 6: Coefficient of Thermal Expansion from 25°C to indicated temperature ($x10^{-6}$ m/m/°C)

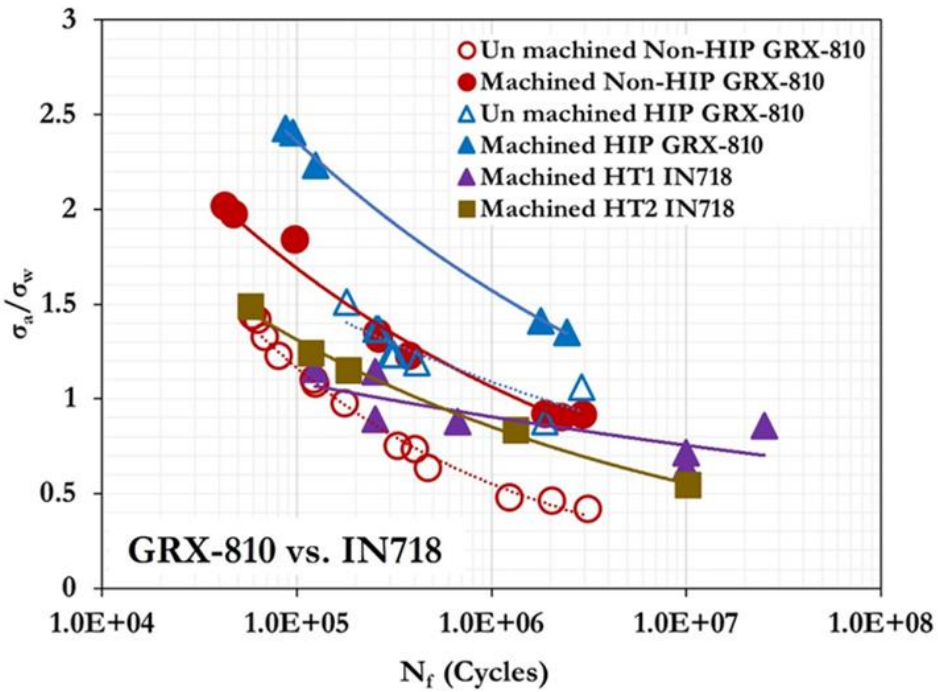
GRX-810 thermal properties comparable to other Ni-based superalloys.

Fatigue

Low Cycle Fatigue



High Cycle Fatigue



HCF testing performed at NCAME (Auburn University)
Dr. Alireza Jam

The fatigue resistance of GRX-810 appears to be comparable or better than current AM superalloys

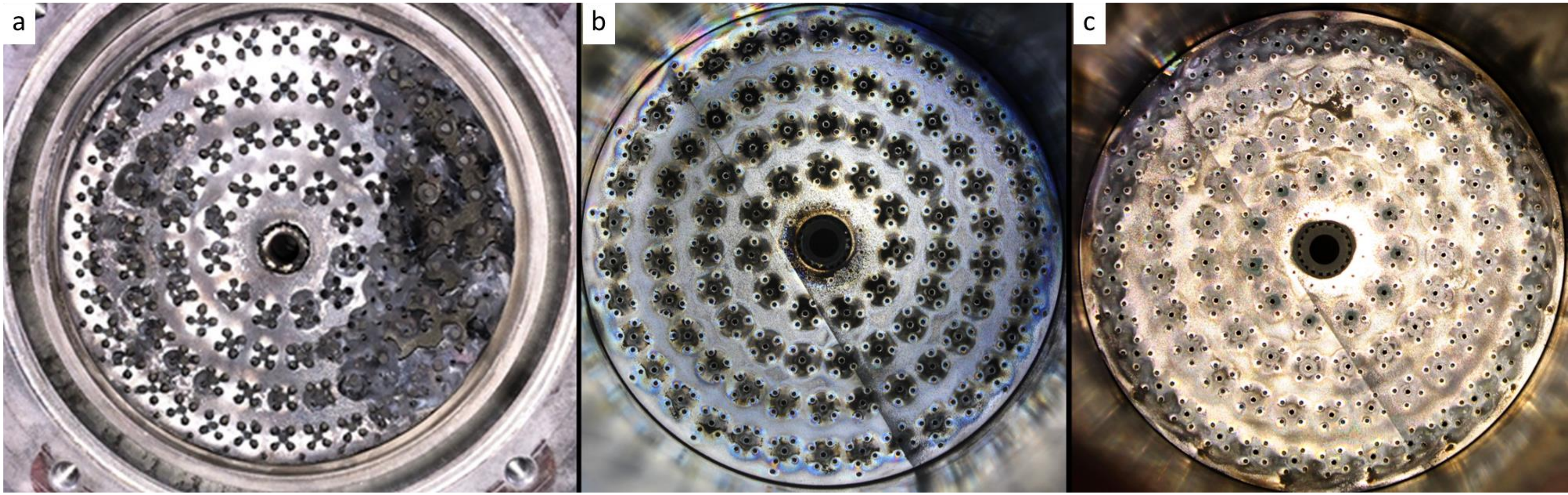
GRX-810 - Scale Up and Hot-fire Test

- 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 18kg per hour using the same machines.
- Successfully printed GRX-810 on larger EOS M280 and DED machines. Have begun component testing at MSFC (see below).

Left: Successful hot-fire test of a liquid oxygen/methane (LOX/LCH4) GRX-810 injector and nozzle.

Hot-Fire Test

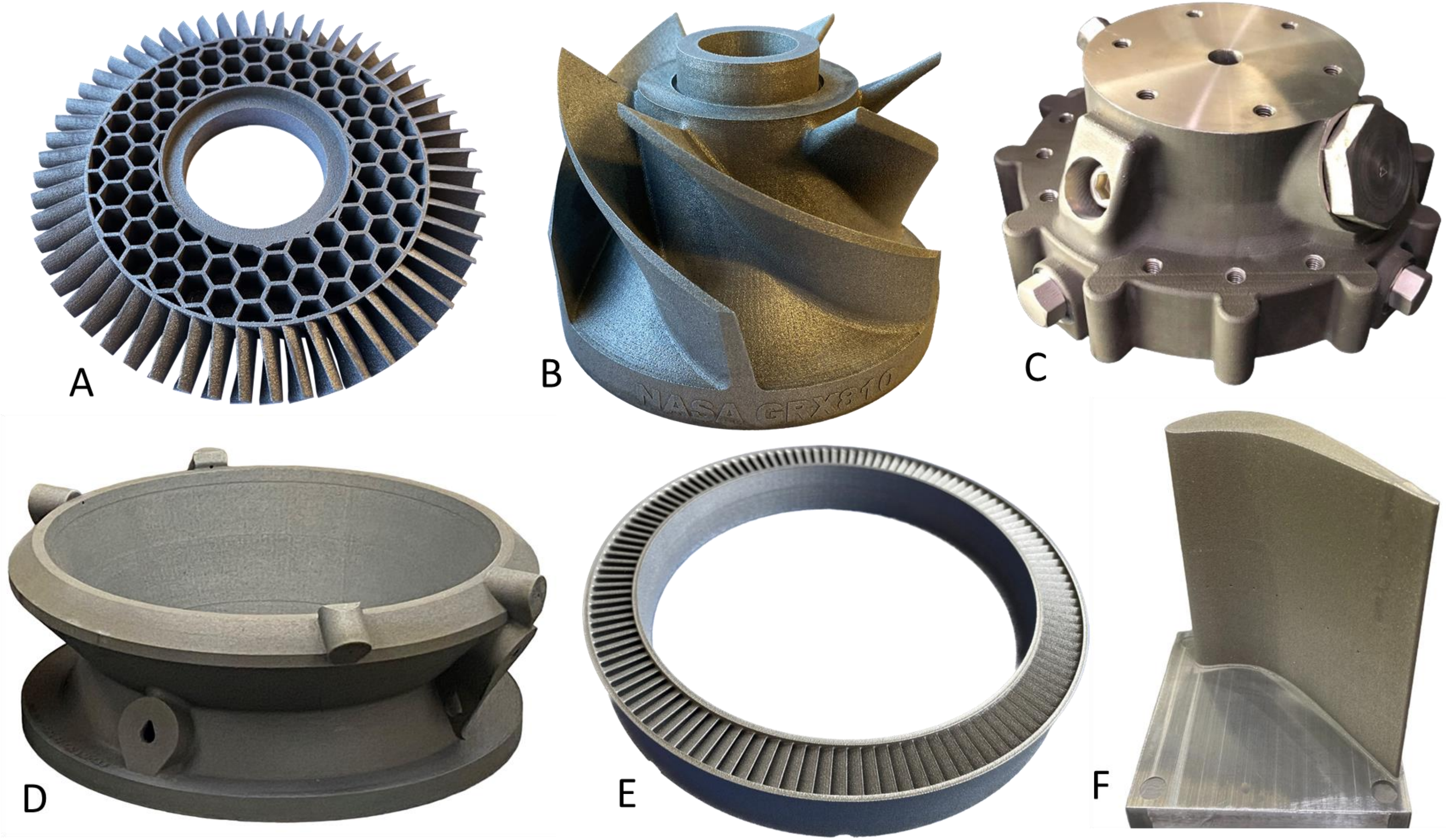
Hot-Fire Test Result



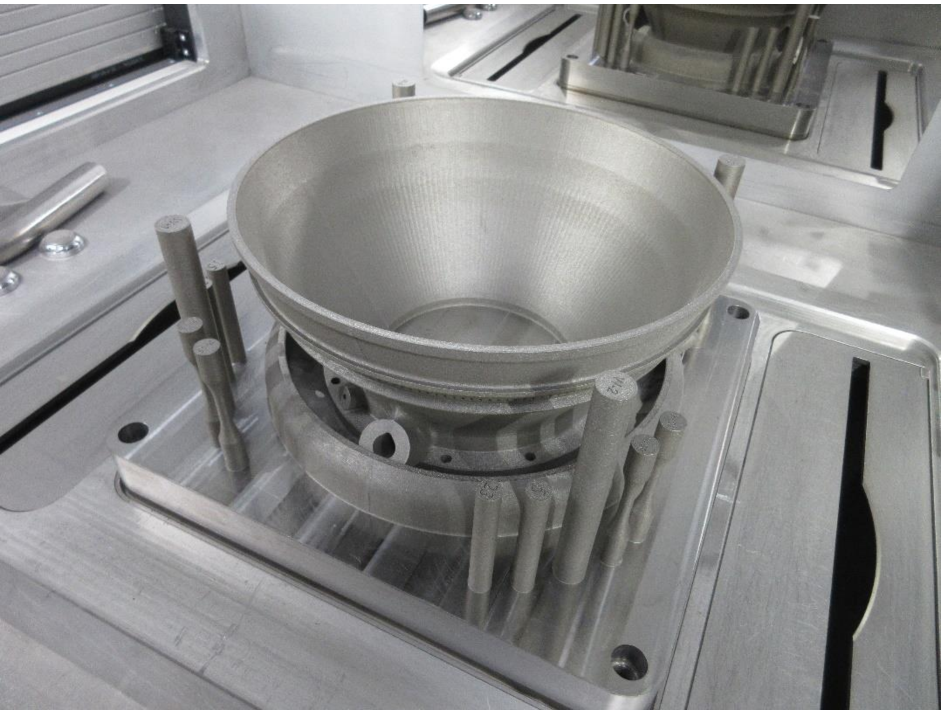
Superalloy 625: 10 tests   GRX-810: 13 tests   GRX-810: 84 tests

GRX-810 appears to provide 5-10x improvement in injector durability compared to Superalloy 625.

GRX-810 Components



M400-4 GRX-810 Nozzle



GRX-810 has been successfully printed on multiple L-PBF AM machines. Large ODS components can now be 3D printed.

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 direct energy deposition (blown powder) AM production of ODS parts and test all important properties such as crack growth, emissivity etc. for GRX-810.

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