Maturation of GRX-810: Mechanical Properties

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Bridging the Gap Webinar: Maturation of AM GRX-810 Superalloy

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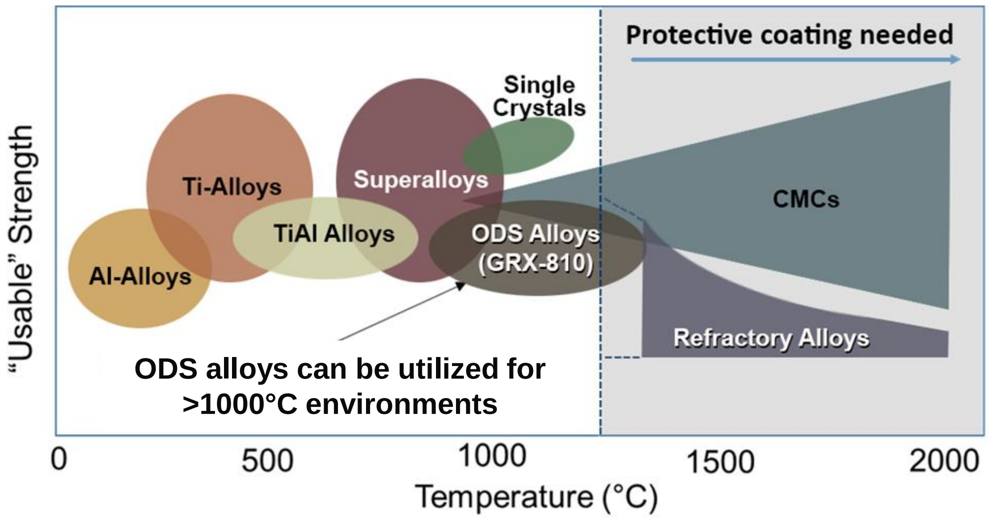
High Temperature AM Compatible Materials

High Temperature Materials:

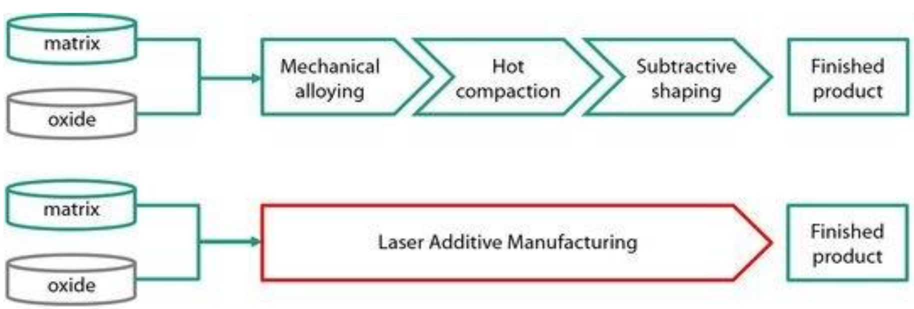
- Refractory metals
- Carbon-Carbon composites
- CMC’s
- Ni-base superalloys
- **Oxide dispersion strengthened (ODS) alloys**



Inspired by Andy Jones. ODS alloy Development.

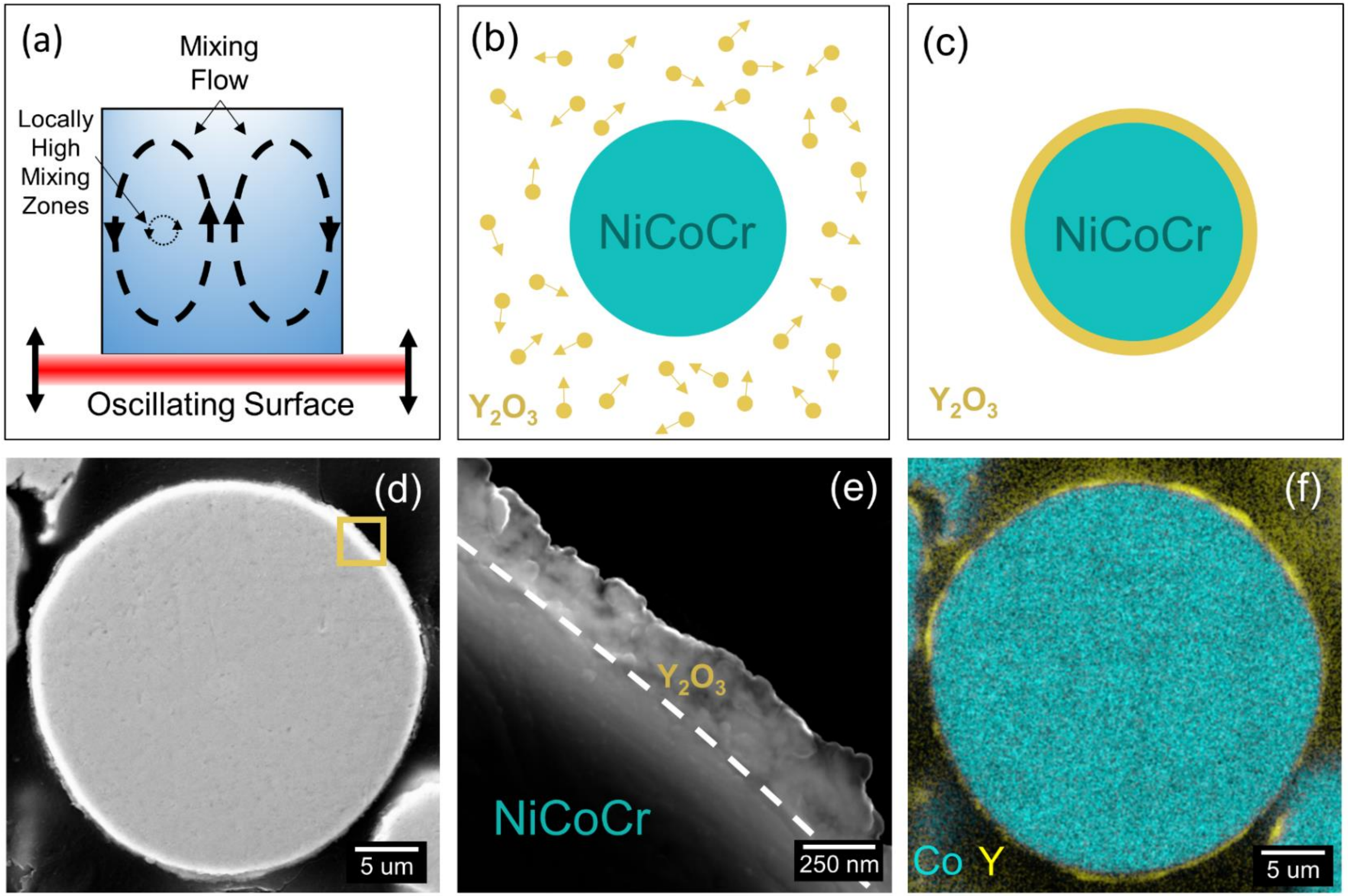
(ODS) alloys offer higher temperature capabilities compared to Ni-base superalloys. However, it has been a challenge to produce ODS alloys through conventional manufacturing methods.

Conventional Manufacturing vs AM



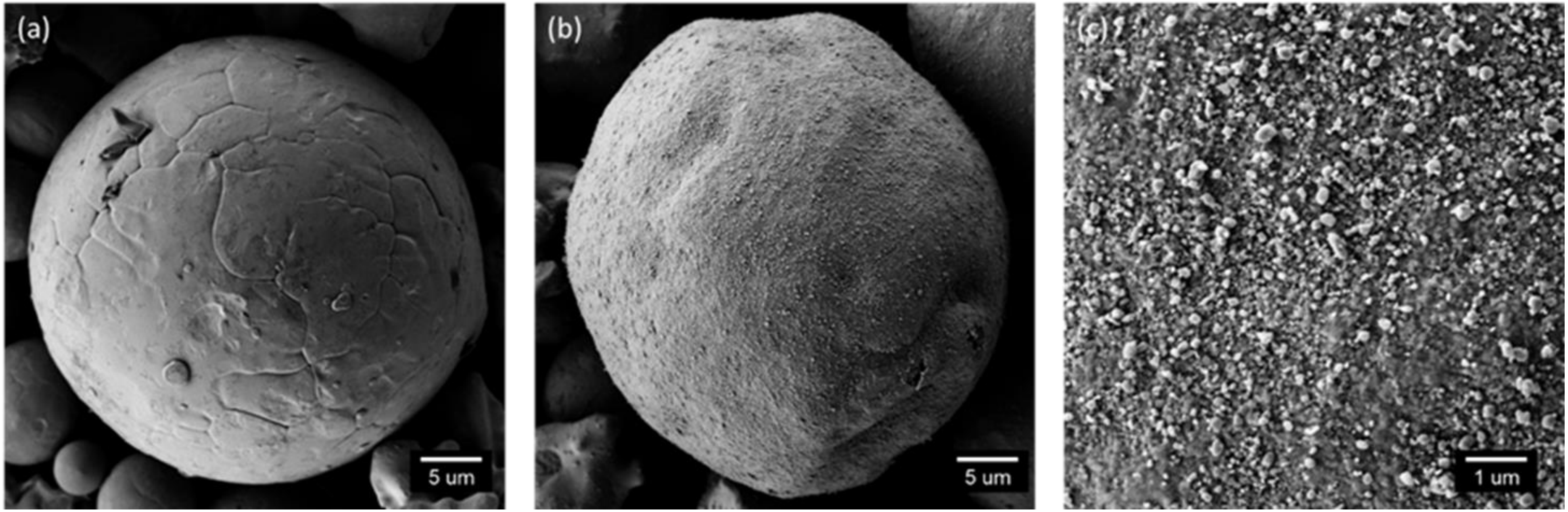
Can additive manufacturing improve ODS alloy manufacturability?

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.

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

>10^7 equilibrium calculations provided an optimized composition named GRX-810

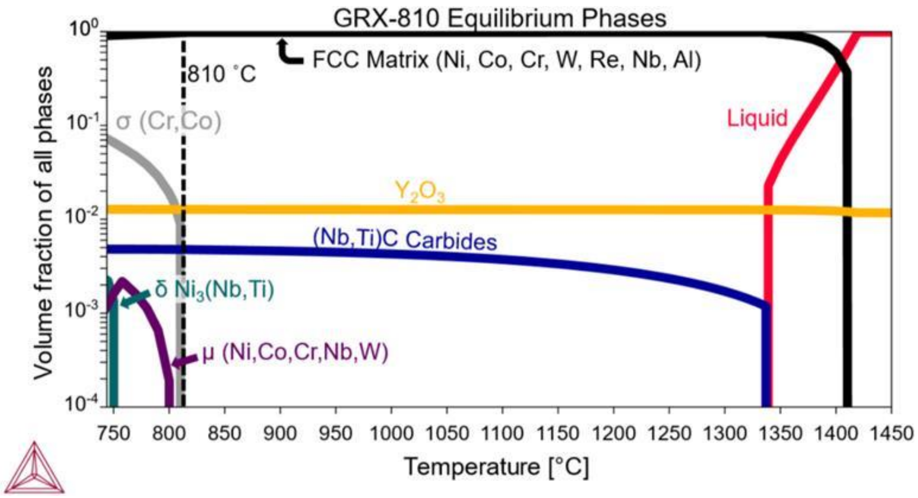
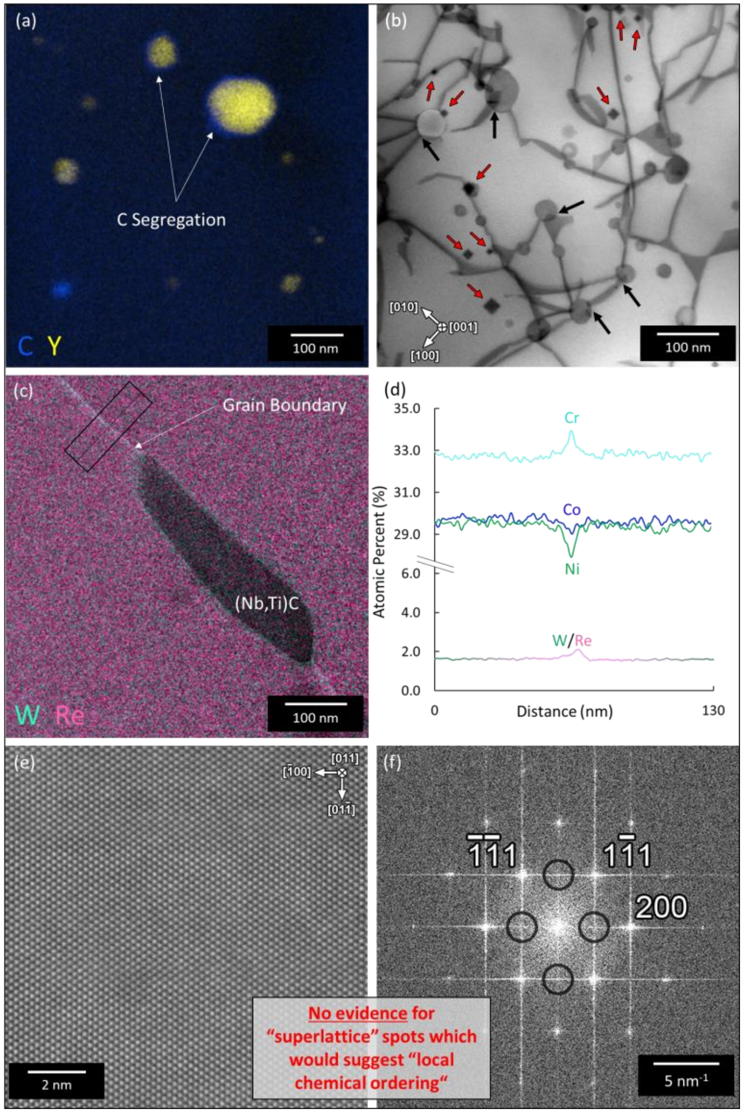


Figure: CALPHAD simulation of phase formation in new composition. No intermetallic or TCP phases are predicted.

Models calculated by C. Kantzos

	Ni	Co	Cr	Re	Al	Ti	Nb	Mo	W	Zr	C	B
Nominal Composition (GRX-810)	Bal.	33	29	1.5	**0.3**	**0.25**	**0.75**	**0**	**3**	**0**	**0.05**	0

STEM Analysis



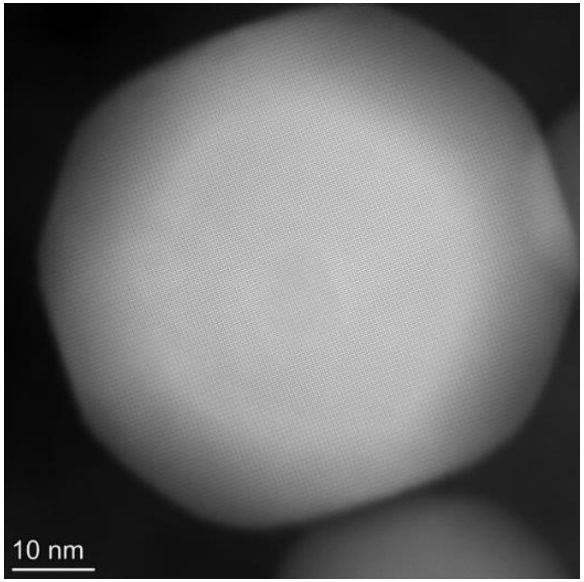
Top: STEM analysis revealed Carbon segregation at the oxide matrix interface. Top Right: Reveals dislocation oxide interactions.

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

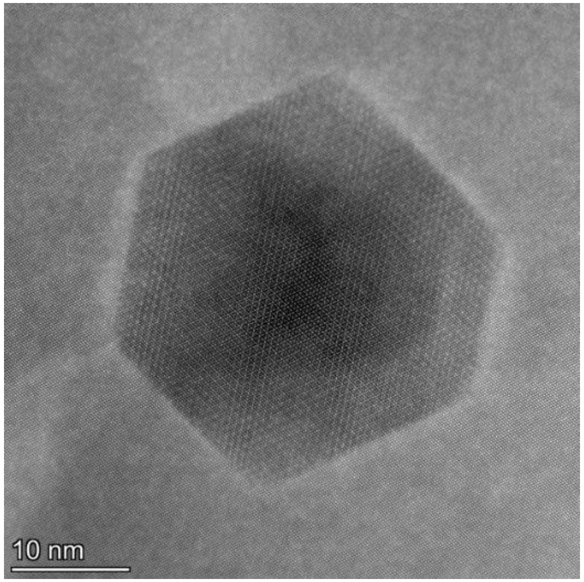
Bottom: Diffraction from the [001] zone axis STEM image reveals that there is no local elemental ordering at the atomic level.

Oxide Comparison – NiCoCr and GRX-810

Cubic Y_2O_3 particle in NiCoCr-ODS



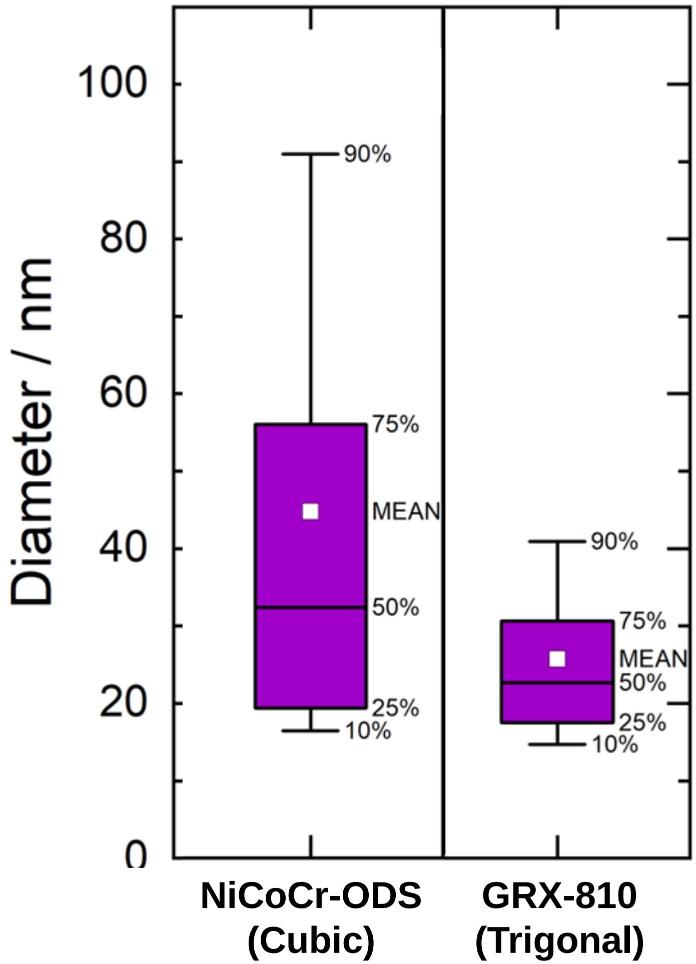
Trigonal Y_2O_3 particle in GRX-810



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.

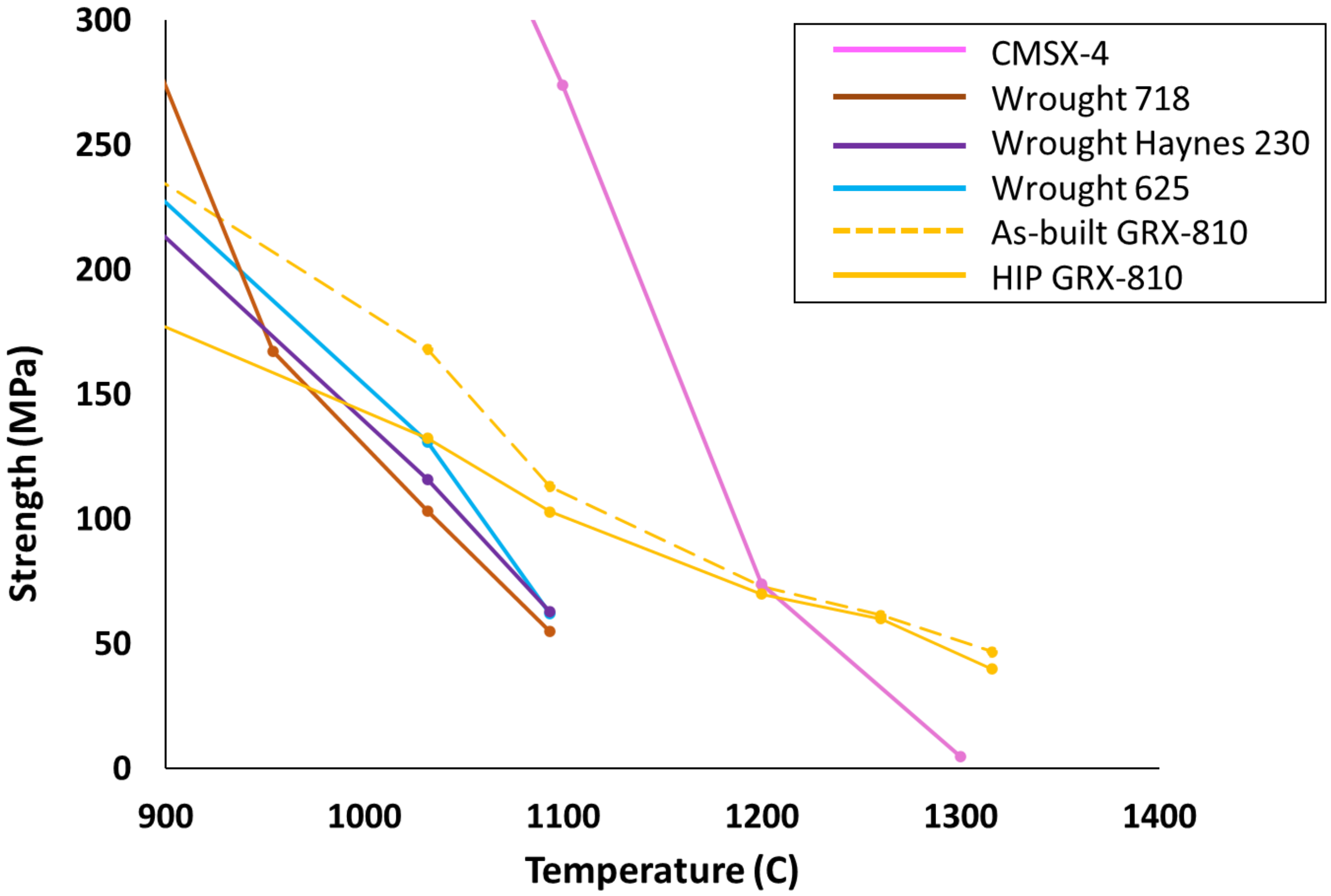
XRD analysis confirms the oxides were cubic prior to coating and printing.

Trigonal Vs. Cubic Y_2O_3



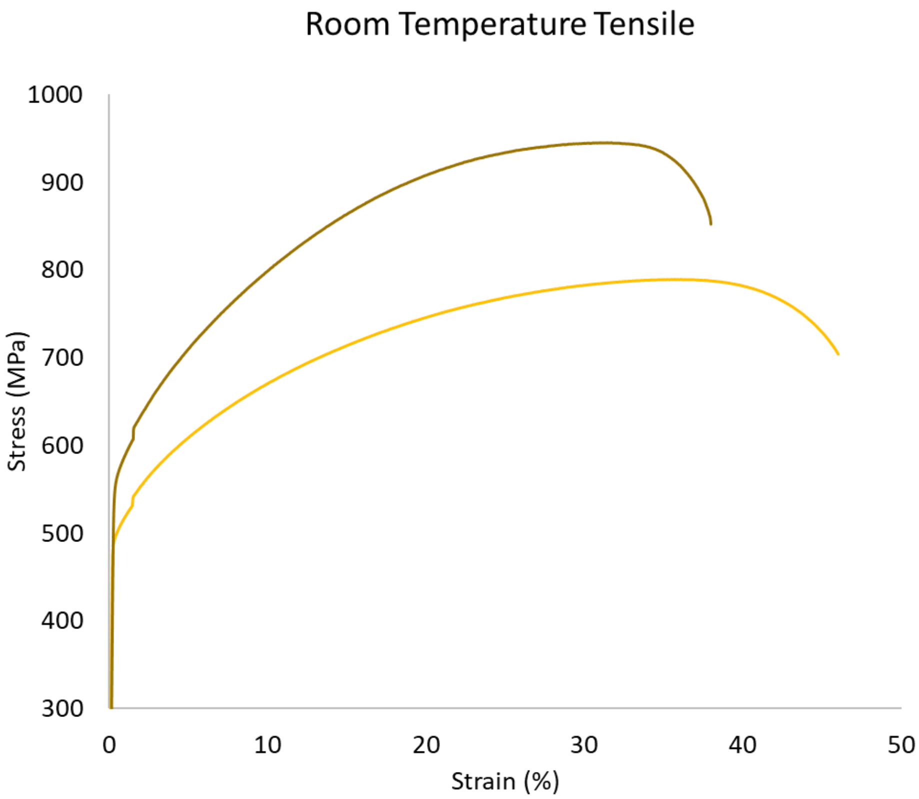
- TEM analysis found the oxides in GRX-810 to be much finer compared to the oxides found in NiCoCr-ODS.
- The finer oxide size distribution may be a source for the significantly improved creep properties found in GRX-810 compared to NiCoCr-ODS

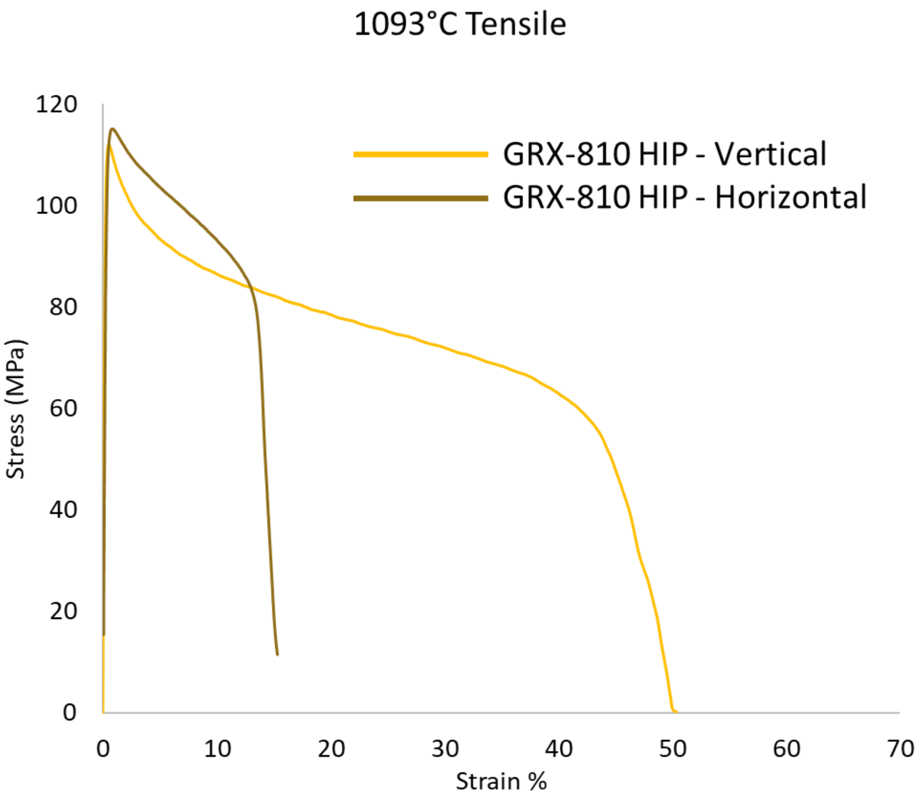
GRX-810 Tensile Overview



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

Tensile – Orientation Effects

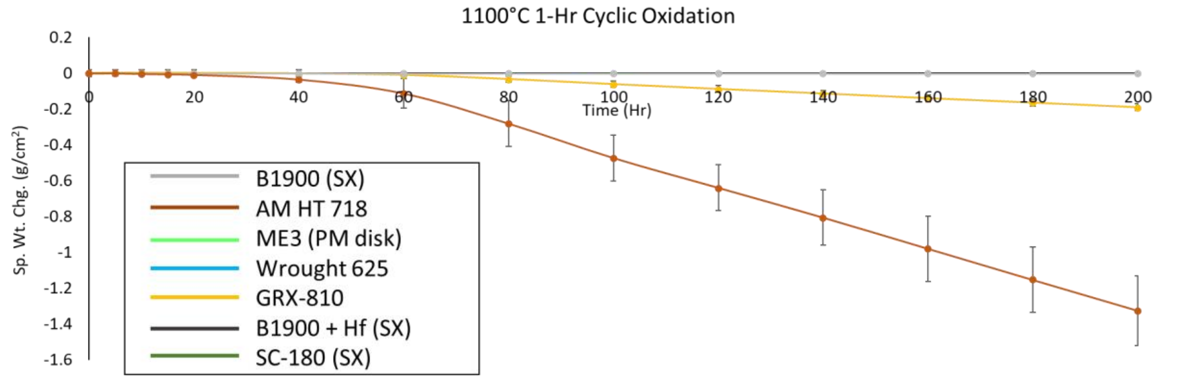


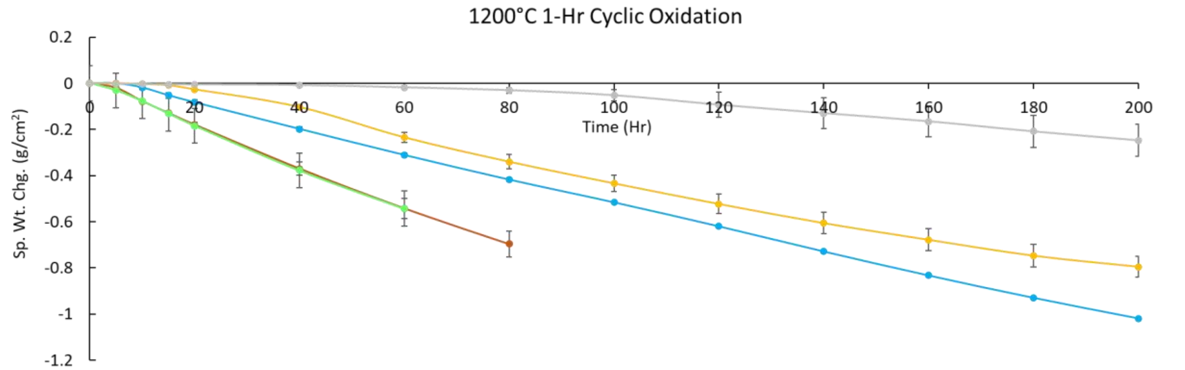


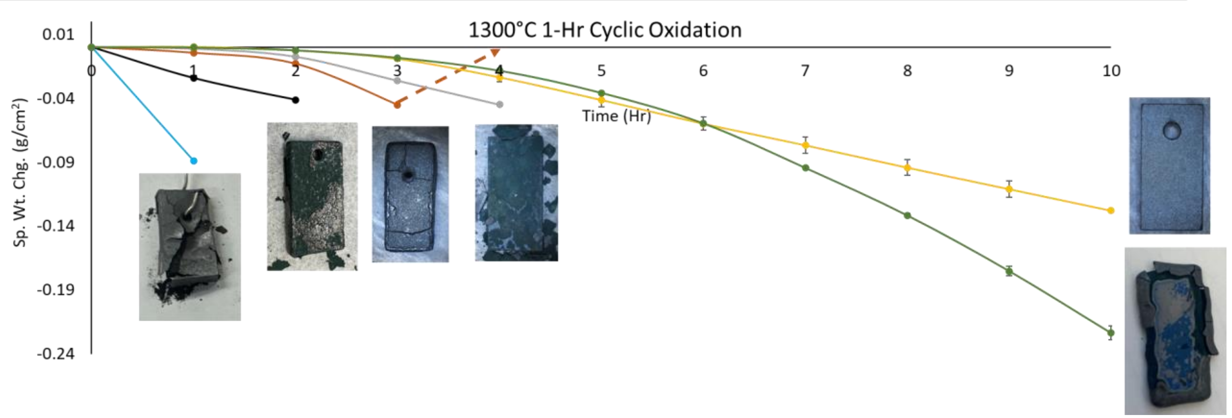
*Specimen printed on the M100

Horizontal GRX-810 exhibits higher strength compared to vertical specimen, but this difference converges at elevated temperatures.

Oxidation

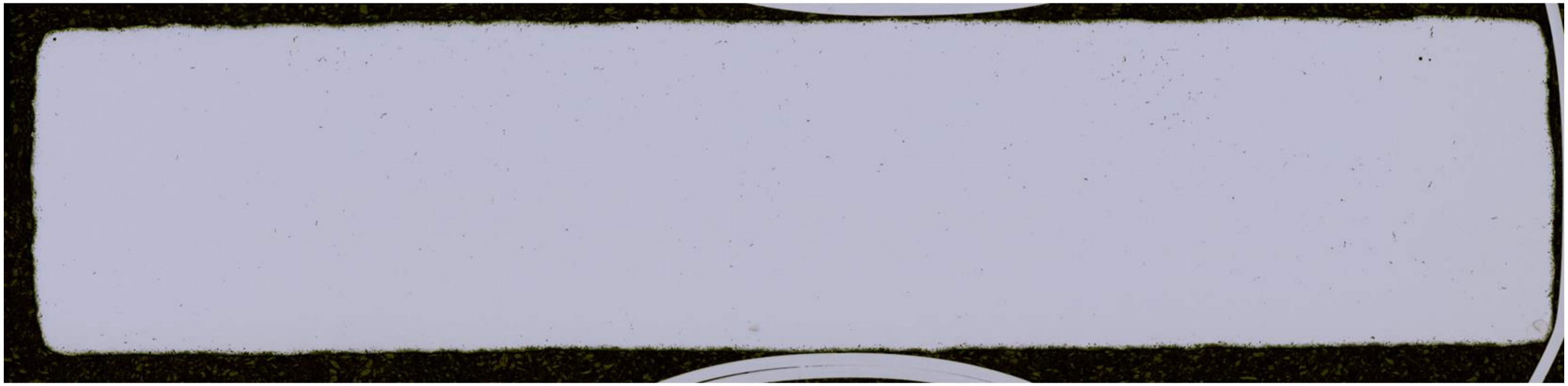

1100°C 1-Hr Cyclic Oxidation
Sp. Wt. Chg. (g/cm²)
0.2
0
-0.2
-0.4
-0.6
-0.8
-1
-1.2
-1.4
-1.6
0
20
40
60
80
100
120
140
160
180
200
Time (Hr)
B1900 (SX)
AM HT 718
ME3 (PM disk)
Wrought 625
GRX-810
B1900 + Hf (SX)
SC-180 (SX)


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)

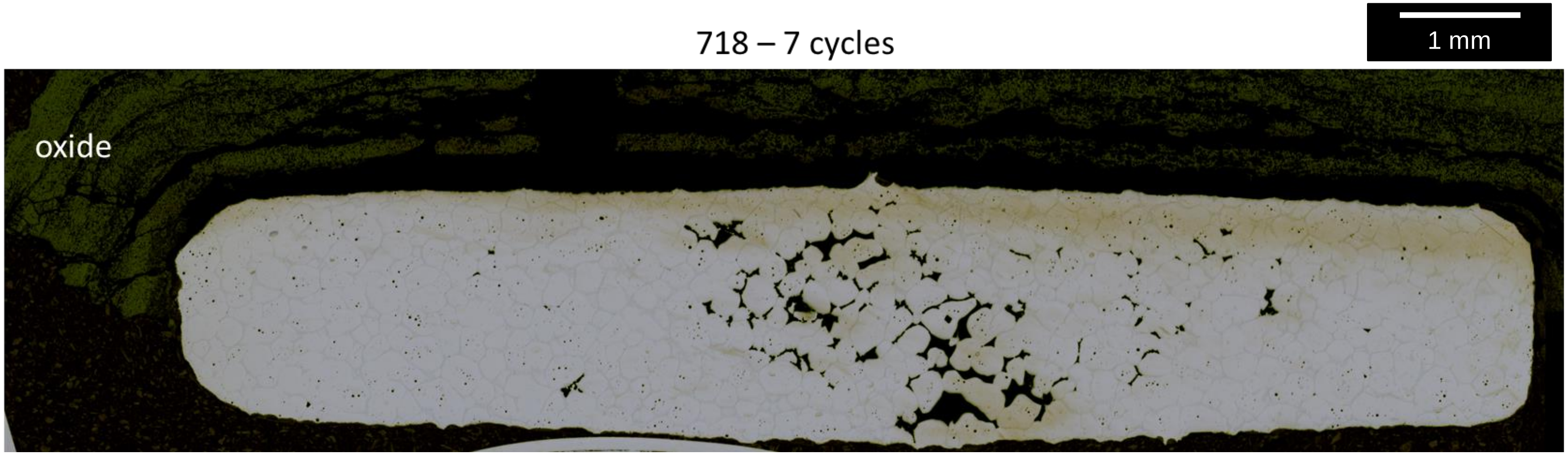

1300°C 1-Hr Cyclic Oxidation
Sp. Wt. Chg. (g/cm²)
0.01
-0.04
-0.09
-0.14
-0.19
-0.24
0
1
2
3
4
5
6
7
8
9
10
Time (Hr)

Oxidation – 1300°C

GRX-810 – 10 cycles



718 – 7 cycles



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

ODS Creep Anisotropy

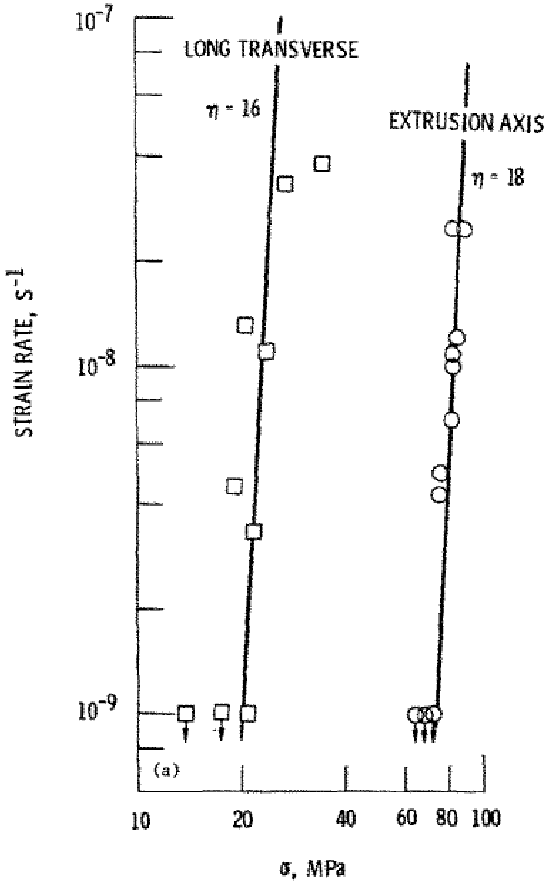


Fig. 2—Steady state creep rate of Inconel MA-754 as a function of (*a*) applied stress (σ) for testing in the extrusion direction and long transverse direction, (*b*) effective stress ($\sigma - \sigma_0$) for testing in extrusion direction, and (*c*) effective stress ($\sigma - \sigma_0$) for testing in long transverse direction.

Table III. Transverse Creep Conditions and Results

Specimen ID	Temperature (°C)	Stress (MPa)	Minimum Creep Rate (s^{-1})	Time to Rupture (h)	Elongation (Pct)	RA (Pct)
T-32	800	132	3.0×10^{-9}	287	0.7	0
T-46	800	160	1.4×10^{-9}	760	0.4	0
T-39	800	175	5.8×10^{-9}	235	0.8	0
T-37	800	182	6.1×10^{-9}	266	0.1	0
T-41	800	183	1.2×10^{-8}	124	1.3	0
T-38	800	189	1.0×10^{-7}	37	2.1	0
T-42	800	196	8.7×10^{-8}	41	2.0	0
T-44	800	206	5.7×10^{-7}	11	4.6	8
T-50	900	98	1.8×10^{-9}	327	0.5	0
T-33	900	114	6.1×10^{-9}	115	0.4	0
T-36	900	121	7.7×10^{-9}	98	0.4	0
T-40	900	124	9.2×10^{-9}	82	0.5	0
T-43	900	129	2.4×10^{-8}	59	0.9	0
T-45	900	136	1.7×10^{-8}	46	0.6	0
T-47	900	145	1.5×10^{-8}	34	0.6	0
T-48	900	160	8.0×10^{-8}	10	0.8	0
T-49	900	170	7.4×10^{-7}	3	1.3	5
T-61	1000	41	—*	>3000	—*	—*
T-60	1000	51	2.2×10^{-10}	1876	0.2	0
T-62	1000	51	8.9×10^{-10}	294	1.8	0
T-58	1000	58	3.7×10^{-9}	111	0.5	0
T-59	1000	61	2.1×10^{-9}	122	0.1	0
T-53	1000	68	3.4×10^{-9}	142	0.3	0
T-52	1000	78	—**	107	—**	0
T-51	1000	88	7.8×10^{-8}	6.7	0.5	0
T-56	1000	92	2.9×10^{-9}	33	0.4	0
T-34	1000	100	—**	18	—**	0
T-57	1000	100	1.7×10^{-8}	15.5	0.2	0
T-55	1000	107	2.6×10^{-8}	8.2	0.2	0

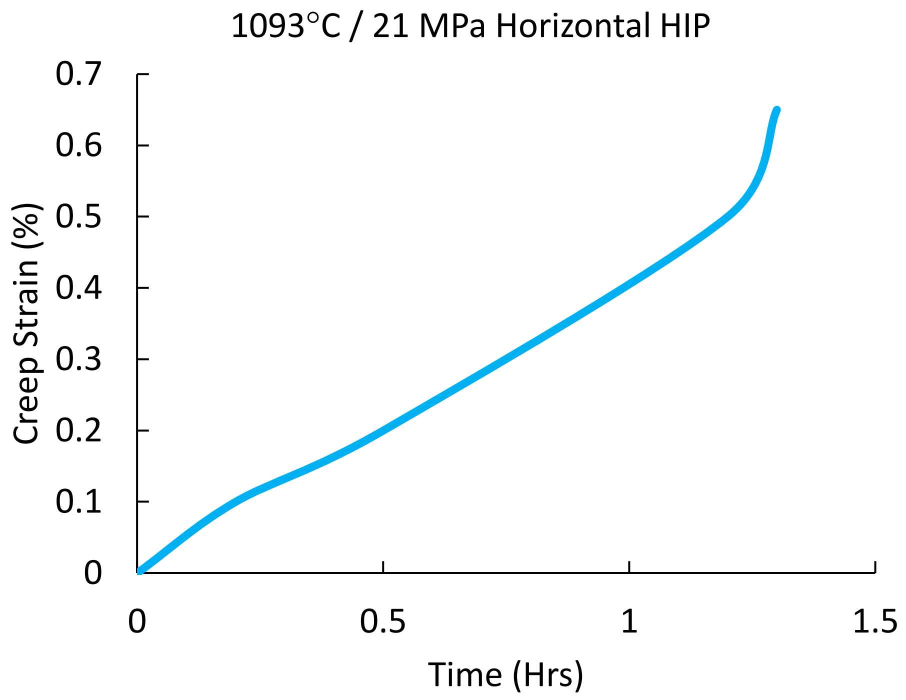
*Test terminated prior to failure.
**No strain data due to extensometer malfunction.

"The utility of the strength advantage is limited, however, by poor transverse ductility. The extremely creep-brittle nature of MA754 in the transverse orientation leads to poor defect tolerance in this direction, as confirmed by creep and fatigue crack growth tests. Ductility and defect tolerance are critical for materials that serve as pressure boundaries."

Horizontal creep ductility has plagued ODS alloys for the last few decades

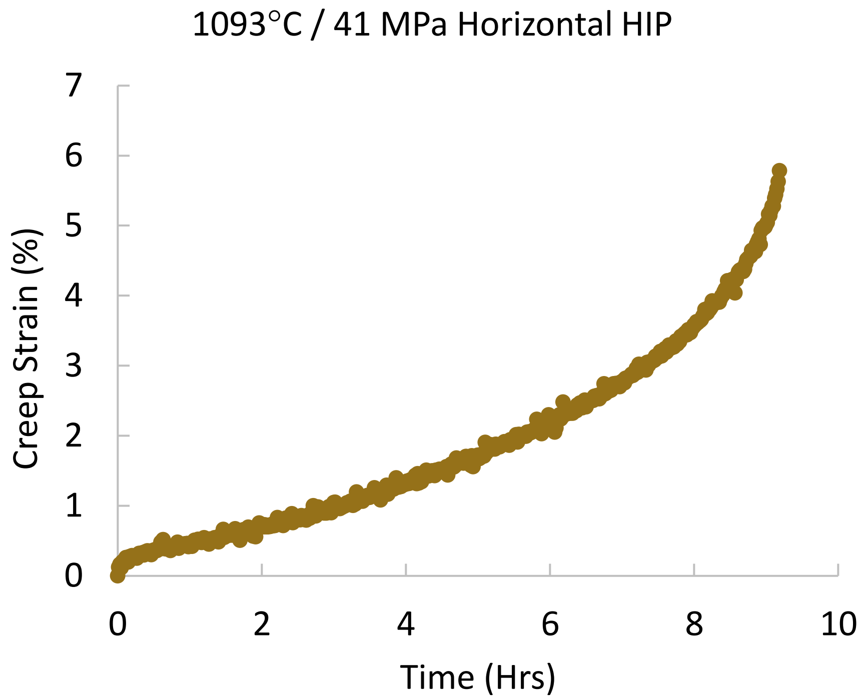
GRX-810 Creep Update

NiCoCr-ODS



Severe creep ductility in NiCoCr-ODS

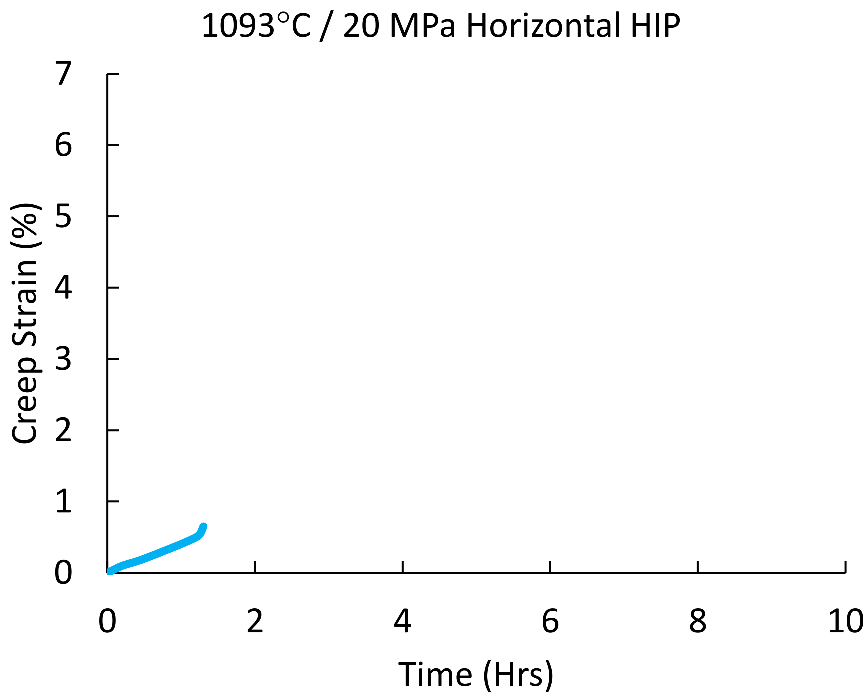
GRX-810



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

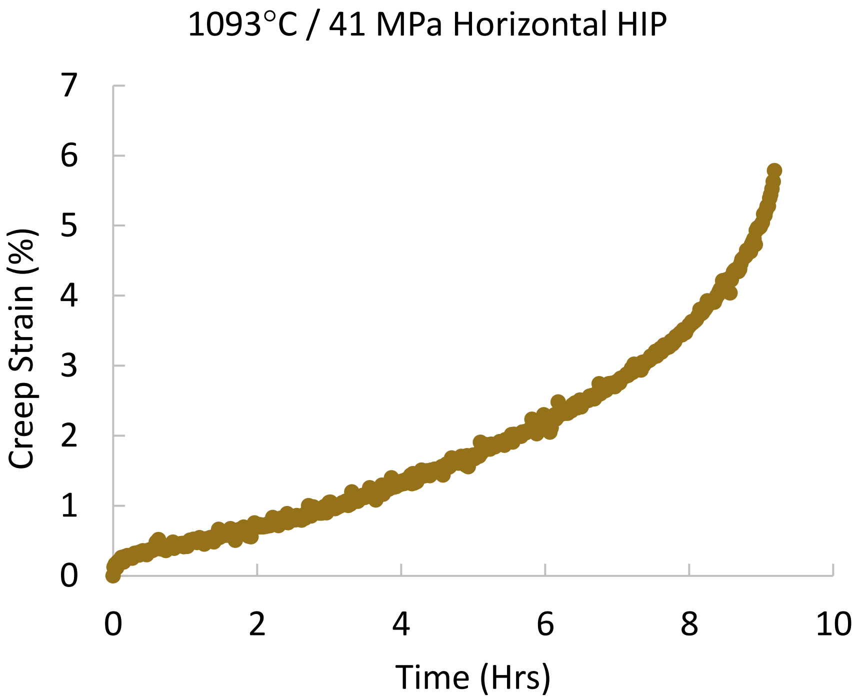
GRX-810 Creep Update

NiCoCr-ODS – 20 MPa



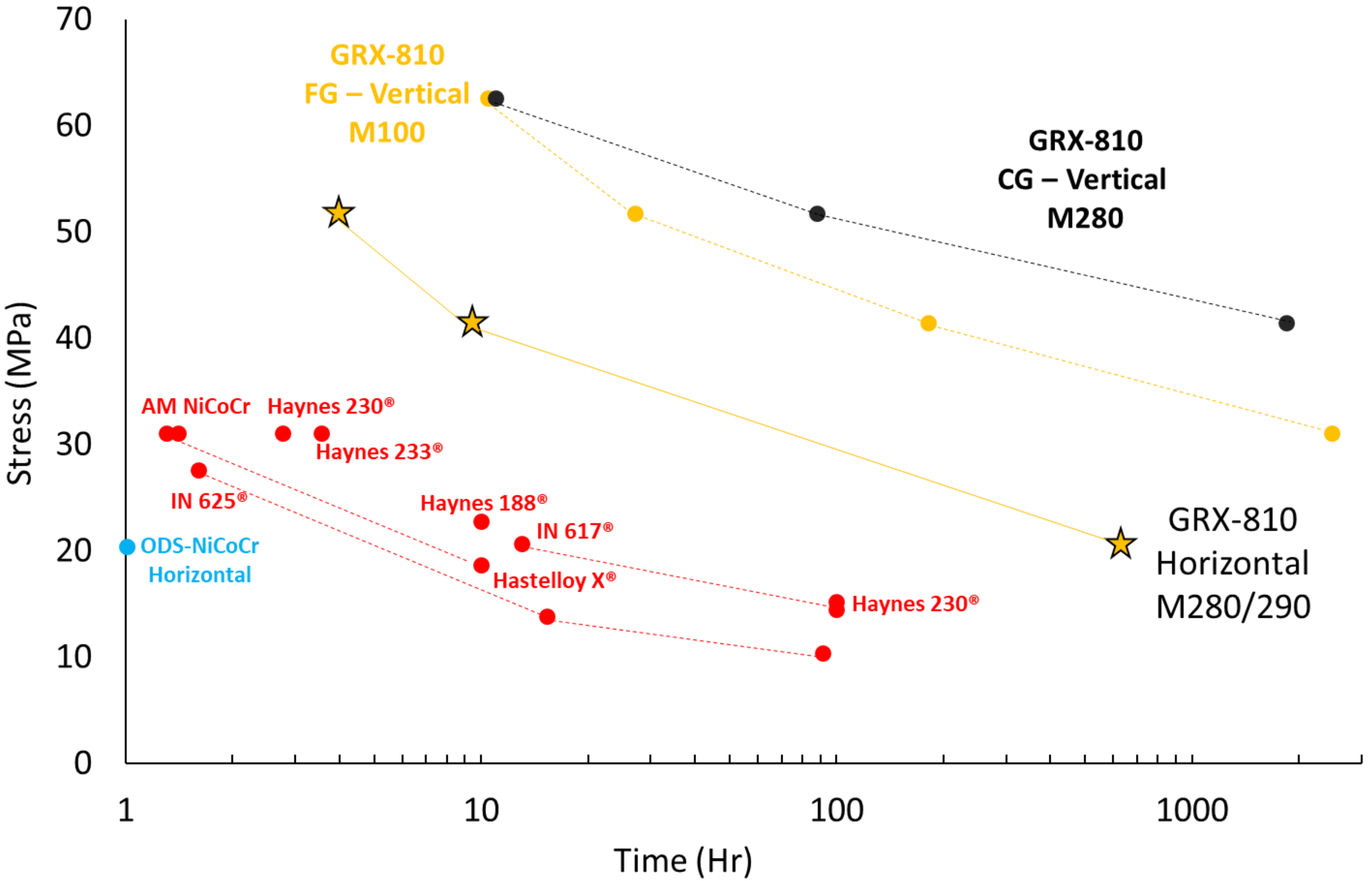
Severe creep ductility in NiCoCr-ODS

GRX-810 – 41 MPa



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

GRX-810 Creep Overview



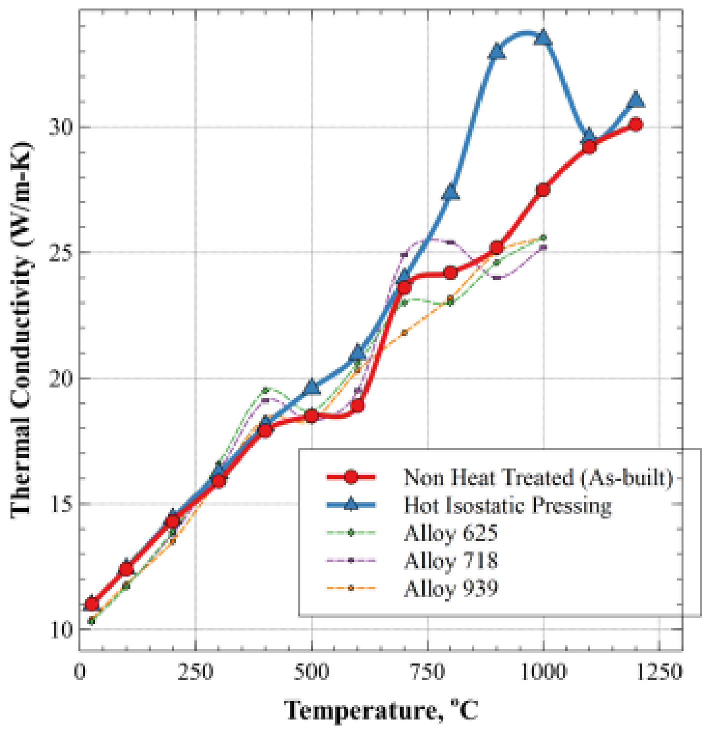
GRX-810 Long-Term Creep

Temperature: 1093°C
Load Frame Material: IN713
Time: 5000 hrs.
Stress: 21 Mpa
Sample Strain ≈ 1%

Load frame failed before GRX-810 creep sample

We have started printing GRX-810 grips to test GRX-810 in temperatures above 1100°C

GRX-810 Thermal Properties



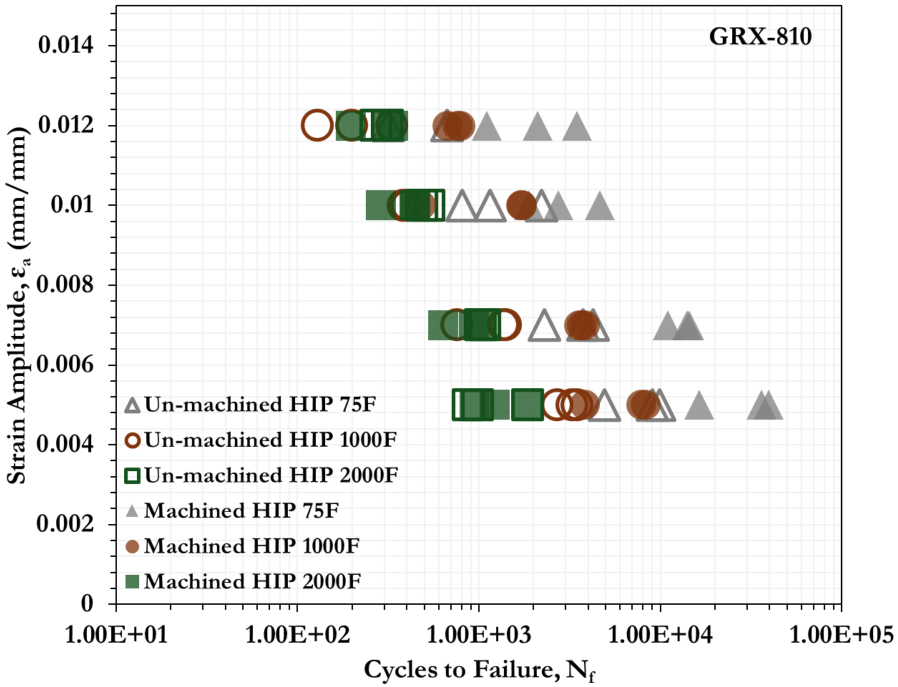
Temperature (°C)	CTE-P-28 [04-0037QY]	CTE-A-25 [04-0037QZ]
100	11.8	10.6
200	12.9	12.3
300	13.5	13.0
400	13.9	13.5
500	14.3	13.9
600	14.7	13.9
700	15.4	14.4
800	16.0	15.0
900	16.5	15.5
1000	17.2	16.1
1100	17.9	16.8
1200	18.5	17.4
1300	19.0	17.9

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

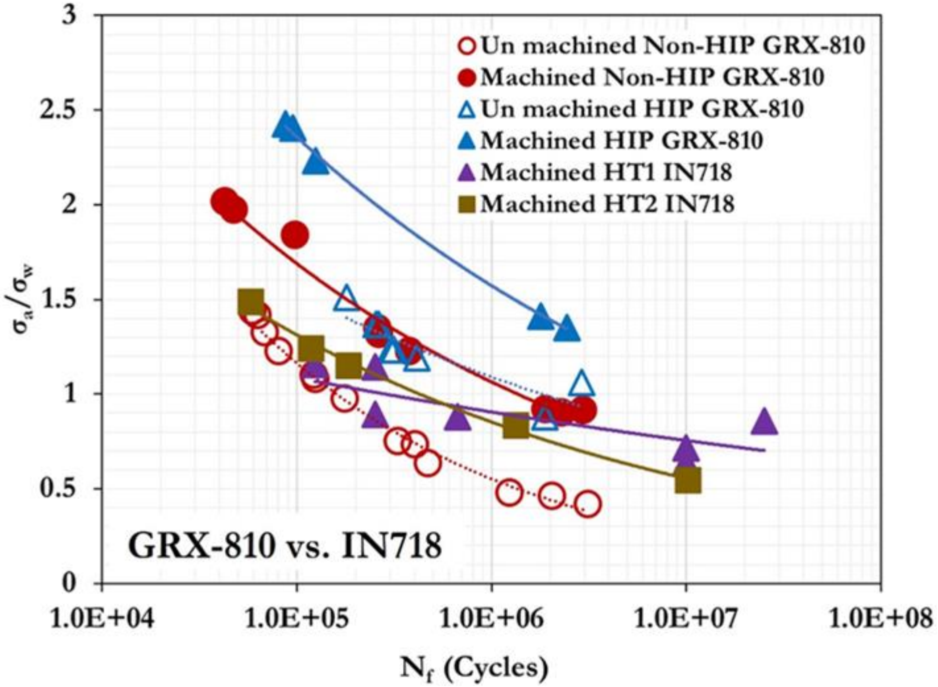
GRX-810 thermal properties comparable to other Ni-based superalloys.

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

Looking Forward

- NASA plans to release a preliminary property data sheet over GRX-810 in the next few weeks.
- High temperature mechanical testing of GRX-810 continues to reveal surprising and superior results compared to previous ODS alloys produced within this project and conventionally manufactured high temperature alloys.
- GRX-810 appears to overcome the severe creep ductility issue that has limited the use of ODS alloys the last few decades.
- Moving forward: Explore DED and other additive methods for GRX-810 production.