Additive Manufacturing of Oxide Dispersion Strengthened (ODS) Nickel-Based Superalloys

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Objective

- Attempt acoustic mixing as an alternative to mechanical alloying for mixing powders for additive manufacturing
- Investigate SLM (selective laser melting) parameters to successfully print ODS Ni-based alloys
- Quantify mechanical properties of 3D printed test samples for future use as printed engine parts for future launch vehicles

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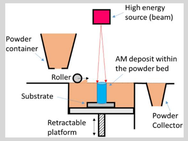
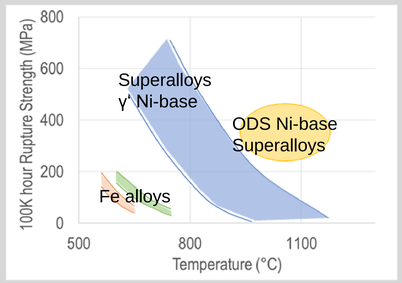


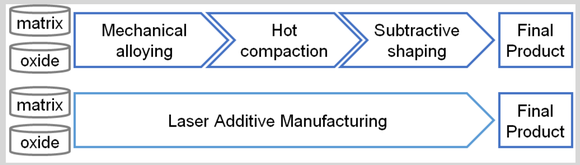
[4] Kok et.al. 2018

Background

There is an ever pressing need to increase high temperature tolerance of materials for hot sections of vehicles.



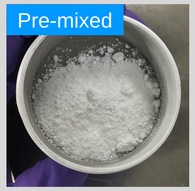
- Ni-based superalloys
  - Superior strength at higher temperatures
  - Limited by solubility of γ' phase[1]
- ODS Ni-based superalloys
  - Dispersion strengthening at high temperatures[2]
  - Retain γ' strengthening at intermediate temperatures[3]
  - Researched in the 1970-80s
    - Limited to manufacturing feasibility struggles
    - Required lots of working to get columnar grains[1,2,3]
    - Additional heat treatments to coarsen grains[2,3]
- Additive manufacturing
  - Aerospace industry was one of the first to develop and produce parts that compete with or exceed the properties of parts made by traditional techniques[4]
  - Exhibits columnar grains
  - Complex, near-net-shape manufacturing of intricate components[4]



Design of Experiment

Acoustic Mixer

Pre-mixed



Time	Acceleration: Low	Medium	High
Short	X	X	X
Medium	X	X	X
Long	X	X	X

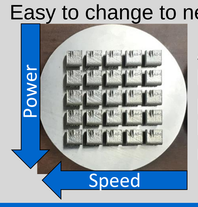
Post-mixed

	NiCoCr + Y2O3	NiCr + Y2O3	NiCr + more Y2O3
Mechanically Alloyed		X	
Acoustically Mixed	X	X	X

Outcomes
- Direct comparison of mixing techniques
- Evaluate success of method on varying compositions

Additive Manufacturing

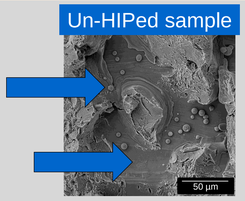
Selective Laser Melting (SLM) on EOS M100 3D-printer
- Requires small amounts of powder
- Easy to change to new chemistries



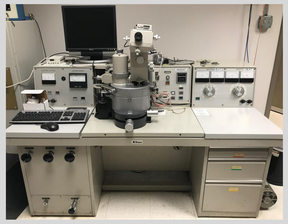
After optimizing laser speed and power, print test bars for mechanical testing

Hot Isostatic Press (HIP)

Un-HIPed sample



- Removal of lack-of-fusion defects
  - process induced porosity
  - un-melted powder
  - layer gaps
- Remove residual stresses[4]
- Microstructure coarsening[4]



Creep Testing

Time	Temperature: 1000 °C	1100 °C	1200 °C
Short	X	X	X
Medium	X	X	X
Long	X	X	X

Tensile Testing

	Temperature: 1000 °C	1100 °C	1200 °C
As-built	X	X	X
HIPed	X	X	X
Heat Treated	X	X	X

Hardness Testing

	Temperature: 25 °C	600 °C	800 °C	1000 °C
1 kg	X	X	X	X

Powder Processing

Consolidating and Heat Treatments

Mechanical Qualification

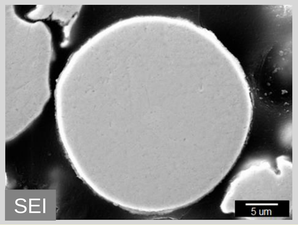
Performance Metrics

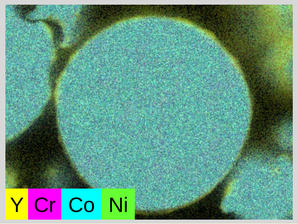
Scanning Electron Microscope (SEM)

Compare powders to previous successful powder images

- Chemistry and oxide volume fraction
- Oxide distribution on metal particles
- Shape of mixed powder

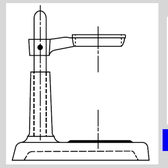
Circularity of powder

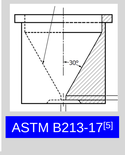




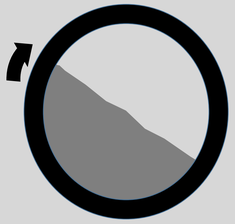
Rheology Measurement Tools

- Flowability, multi-flow, and packing of powders
- Hall flowmeter, Revolution by Mercury Scientific





ASTM B213-17[5]



Other Powder Metrics

- XRD, chemistry and phase identification
- Morphology and size distribution (Malvern and Horiba)

Powder Performance

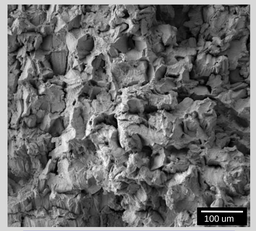
Optical and Electron Microscopy

As-built, HIPed, and heat treated samples

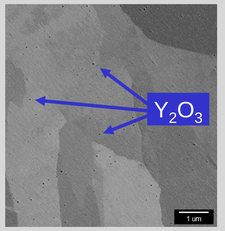
Optical:
- Porosity/cracks

SEM:
- Fractography
- Oxide distribution
- Crystallography, texture

TEM:
- Dislocation interactions
- Nano-particle phase and chemistry analysis



Fractography sample

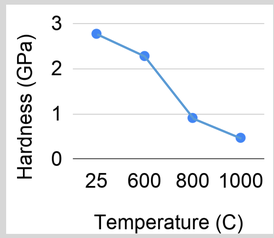


Oxide distribution

Hot Hardness

- Short time to do large number of analyses
- Small sample size requirements
- Correlates with tensile

Method	Time	Sample Size
Hot Hardness	50/day	
Tensile	2/day	
Creep	1/week	



Other Printed Sample Metrics

- Metrology, measurements of Gao blocks
- Tensile/creep testing at elevated temperature (1000 °C or higher)

Compare mechanical properties to Inconel MA754

Built Sample Performance

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[1] Dreshfield, R. L. "The Continuing Battle against Defects in Nickel-Base Superalloys." 2-6 Mar. 1986, United States, 1986. https://ntrs.nasa.gov/search.jsp?R=19860022229.

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[4] Kok, Y., X. P. Tan, P. Wang, M. L. S. Nai, N. H. Loh, E. Liu, and S. B. Tor. 2018. "Anisotropy and Heterogeneity of Microstructure and Mechanical Properties in Metal Additive Manufacturing: A Critical Review." *Materials & Design* 139 (February): 565–86. https://doi.org/10.1016/j.matdes.2017.11.021.

[5] ASTM International. *ASTM B213-17 Standard Test Methods for Flow Rate of Metal Powders Using the Hall Flowmeter Funnel*. West Conshohocken, PA; ASTM International, 2017. doi: https://doi.org/10.1520/B0213-17.

Acknowledgements

This research was funded under NASA grant # 18-Fellows'18-0009.

This presentation was made possible, in part, through financial support from:
- CWRU School of Graduate Studies
- Case School of Engineering Faculty Investment Fund

The authors would also like to thank Tim Smith and David Scannapieco, from NASA Glenn Research Center, for their support and assistance.