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

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

Dr. Tim Smith PhD
NASA Glenn Research Center
08/31/22
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.
Metallic Additive Manufacturing

<table>
<thead>
<tr>
<th></th>
<th></th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>Energy Source</td>
<td>Laser</td>
<td>E-Beam</td>
<td>Laser or E-Beam</td>
</tr>
<tr>
<td>Powder Bed</td>
<td>Yes</td>
<td>Yes</td>
<td>No</td>
</tr>
<tr>
<td>Power (W or kV)</td>
<td>50-1000 W</td>
<td><strong>30-60kV</strong></td>
<td>100-2000 W</td>
</tr>
<tr>
<td>Max Build Size (mm)</td>
<td>500 x 280 x 320</td>
<td>500 x 280 x 320</td>
<td><strong>2000 x 1500 x 750</strong></td>
</tr>
<tr>
<td>Material</td>
<td>Metallic Powder</td>
<td>Metallic Powder</td>
<td>Metallic Powder or Wire</td>
</tr>
<tr>
<td>Dimensional Accuracy</td>
<td>&lt;0.04 mm</td>
<td>0.04-0.2 mm</td>
<td>0.5 mm (powder) 1.0 mm (wire)</td>
</tr>
</tbody>
</table>

- 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

(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?

Inspired by Andy Jones. ODS alloy Development.
- 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?
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.
L-PBF successfully disperses the nano-scale $Y_2O_3$ particles throughout the AM build.
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

<table>
<thead>
<tr>
<th>Alloy</th>
<th>As-Built – Build direction</th>
<th>As-Built – 90° from build direction</th>
<th>HIP – Build direction</th>
<th>HIP – 90° from Built direction</th>
</tr>
</thead>
<tbody>
<tr>
<td>AM NiCoCr</td>
<td>34 ± 35</td>
<td>141 ± 96</td>
<td>-5 ± 3</td>
<td>-4 ± 6</td>
</tr>
<tr>
<td>ODS-NiCoCr</td>
<td>320 ± 51</td>
<td>185 ± 49</td>
<td>-11 ± 9</td>
<td>-12 ± 9</td>
</tr>
<tr>
<td>ODS-ReB</td>
<td>321 ± 52</td>
<td>179 ± 47</td>
<td>-8 ± 5</td>
<td>-12 ± 6</td>
</tr>
</tbody>
</table>
Oxide Dispersion Strengthened MPEA Combustor Dome
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

Over 50 simulations provided an optimized composition named GRX-810

<table>
<thead>
<tr>
<th></th>
<th>Ni</th>
<th>Co</th>
<th>Cr</th>
<th>Re</th>
<th>Al</th>
<th>Ti</th>
<th>Nb</th>
<th>Mo</th>
<th>W</th>
<th>Zr</th>
<th>C</th>
<th>B</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Old Composition</strong></td>
<td>Bal.</td>
<td>32</td>
<td>30</td>
<td>1.5</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>.003</td>
</tr>
<tr>
<td><strong>New Composition (GRX-810)</strong></td>
<td>Bal.</td>
<td>33</td>
<td>29</td>
<td>1.5</td>
<td>x</td>
<td>x</td>
<td>x</td>
<td>x</td>
<td>x</td>
<td>x</td>
<td>x</td>
<td>x</td>
</tr>
</tbody>
</table>
High resolution SEM reveals dispersed nano-oxides and grain boundary phases.
STEM-EDS analysis confirms high density of nano-scale Y2O3 particles throughout bulk. No oxide agglomeration was present. Most other elements did not react with oxides (Nb and Ti were exceptions).
GRX-810 presents higher ultimate tensile strength at room temperature while maintaining the base materials high ductility.
GRX-810 provides almost double the tensile strength and over 2x the ductility compared to AM NiCoCr.
GRX-810 Tensile Overview

- 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.
Mechanical Results – 1093°C/20MPa
Creep Rupture

![Creep Rupture Graph]

- AM 718 HIP+HT
- AM 625 HIP (14 MPa)
- Wrought H230
- HIP NiCoCr
- As-built ODS-ReB
- HIP ODS-ReB
- As-built GRX-810*
- HIP GRX-810*
• *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

Creep Strain (%)

Time (Hr)

As-built NiCoCr
HIP NiCoCr
As-built NiCoCr-ODS
HIP NiCoCr-ODS
As-built ODS-ReB
HIP ODS-ReB
As-built GRX-810
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 Resodyne 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
Contact Information

• Email: Timothy.m.smith@nasa.gov
• Phone: 216-433-2632
• Address: NASA Glenn Research Center
  21000 Brookpark Rd. Cleveland OH 44135