Effect of Heat Treatment on Microstructure of Nickel-base Superalloy fabricated by Laser-Powder Bed Fusion Additive Manufacturing

Hyeyun Song and Prof. Wei Zhang
The Ohio State University

Dr. Shawn M. Kelly
EWI
Outlines

1. Objectives
2. As-built samples produced by L-PBF AM
   - Characteristics of microstructures
   - Confirmation of precipitates
   - Effect of support on hardness value
3. Post-build heat treatment
   - Homogenization
   - Aging
4. Effect of heat treatment on microstructure
5. Effect of heat treatment on surface oxidation
6. Results and future works
Objectives

- Additive manufacturing (AM) is advantageous for direct production of complex shaped components based on three-dimensional CAD.
- Requirements for better qualification of parts built by laser powder bed fusion:
  - Understanding of microstructures due to a large number of repeated heating and cooling cycles
  - Understanding of the effect of post-build heat treatment on microstructures and properties

Approaches used

- Two groups of IN718 samples:
  - Built either directly on substrate or with a grid support
  - 375 layers in building direction to a height of 15 mm
  - As-built dimensions fairly accurate
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Microstructure of as-built sample

- Size of bright precipitates: 500~800 nm
- Dendrite Arm Spacing (DAS) of gray matrix: 1~1.8μm

**EDS point analysis of sample 2 (with support)**

<table>
<thead>
<tr>
<th>Element</th>
<th>wt%</th>
<th>Literature</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fe</td>
<td>12.54</td>
<td></td>
</tr>
<tr>
<td>Cr</td>
<td>14.46</td>
<td></td>
</tr>
<tr>
<td>Ni</td>
<td>41.32</td>
<td></td>
</tr>
<tr>
<td>SUM</td>
<td>68.32</td>
<td>64</td>
</tr>
<tr>
<td>Ti</td>
<td>1.45</td>
<td></td>
</tr>
<tr>
<td>Nb</td>
<td>24.49</td>
<td></td>
</tr>
<tr>
<td>Mo</td>
<td>5.48</td>
<td></td>
</tr>
<tr>
<td>SUM</td>
<td>31.42</td>
<td>35</td>
</tr>
</tbody>
</table>

- Bright regions are enriched in Nb, Mo and Ti and depleted in Cr, Fe and Ni.
- The chemical composition matches that of Laves phase.
- NbC and delta, two other common precipitates, were not observed.
Diffraction patterns of FCC matrix and Laves phase

Matrix: FCC

Laves phase: HCP

Matrix: FCC; \( a=0.36 \) nm

B=Z=[0001]

Laves: HCP; \( a=0.48, c=0.74 \) nm

Axial ratio \( c/a = 1.54 \)
Effect of the support on the hardness value

**Hardness map of sample without support**

![Hardness map of sample without support](image)

**Hardness map of sample with support**

![Hardness map of sample with support](image)

<table>
<thead>
<tr>
<th>Avg.</th>
<th>316</th>
</tr>
</thead>
<tbody>
<tr>
<td>Max.</td>
<td>344</td>
</tr>
<tr>
<td>Min.</td>
<td>290</td>
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</table>

<table>
<thead>
<tr>
<th>Avg.</th>
<th>301</th>
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</thead>
<tbody>
<tr>
<td>Max.</td>
<td>337</td>
</tr>
<tr>
<td>Min.</td>
<td>271</td>
</tr>
</tbody>
</table>

- Small reduction (5%) in hardness value of the sample with support
Correlation of hardness variation and Laves phase

Avg. fraction of Laves phase

| Without support: 2.62% | With support: 2.64% |

2.81% volume fraction of Laves phase in the highest hardness region

• Larger fraction and coarser size of Laves phase is a likely factor for the higher hardness locally.

1.68% volume fraction of Laves phase in the lowest hardness region
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# Post-built heat treatment

- As-built microstructure does not have $\gamma''$ and $\gamma'$, the important strengthening precipitates for IN718. Hence, post-built heat treatment is still required.

- Two commonly used standards are:

<table>
<thead>
<tr>
<th>Schedule</th>
<th>Homogenization</th>
<th>Solution anneal</th>
<th>Aging</th>
</tr>
</thead>
<tbody>
<tr>
<td>AMS 2773E – Casting</td>
<td>1093°C for 2 hours, AC</td>
<td>982°C for 1 hour, AC</td>
<td>718°C for 8 hours, FC to 621°C, hold for a total precipitation time of 18 hrs</td>
</tr>
<tr>
<td>AMS 2774D – Wrought</td>
<td>N/A</td>
<td>954°C for 1 hour, AC</td>
<td>10 hours at 760°C, FC to 649°C and hold for 20 hrs total precipitation time, AC</td>
</tr>
</tbody>
</table>

AMS – Aerospace Materials Specification; AC – Air cooling; FC – Furnace cooling.

- **AMS 2773E was studied at first, since the as-built microstructure is expected to be closer to cast than wrought.**

- **Questions to be answered:**
  - Can Laves phase be completely dissolved after homogenization?
  - What effect does the cooling rate for homogenization have?
  - Is the solution step necessary?
Group 1: Homogenization (and no solution annealing)

1. Peak temperature: **1100 °C or 1200 °C** (above Laves and δ solvus temperatures)
2. Heating rate: 5 °C/min
3. Cooling:
   - Air cooling
   - Force air cooling
   - Water quenching
4. Shielding gas for heating and holding steps: Ar (0.97 liter per min)

Group 2: Casting AMS 2773E (homogenization + solution)

1. Homogenization at 1093°C for 2 hours, AC
2. Solution:
   - Heating rate: 5 °C/min
   - Peak temperature: **982°C**
   - Shielding gas for heating and holding steps: Ar (0.97 liter per min)
   - Air cooling
Microstructure after homogenization

- Remaining Laves phase along the grain boundaries

Needle-shaped delta phase formed after the solution annealing step

Grain growth & reduction of Laves phase
Condition for aging

- Followed AMS (2773E) standard for casting condition:
  1. Homogenization: Holding at 1093°C for 2 hours and air cooling
  2. Aging: Holding at 718°C for 8 hours and then furnace cooling
     → 621°C for 10 hours (including cooling time from 718°C to 621°C)
  3. Shielding gas for heating and holding steps: Ar (0.97 liter per min)
TEM images of aged sample

- Distinct morphology of new precipitates formed after aging
- Evenly distribution of small pores with avg. diameter of 41.5 nm
- Avg. diameter of small precipitates ($\gamma''$): 13.5 nm
- Avg. aspect ratio for large precipitates ($\gamma'$): 0.23

**Group 1 sample: homogenization (and no solution annealing)**

- $\gamma'$: Ni$_3$(Ti,Al), FCC, spherical or cubic
- $\gamma''$: Ni$_3$Nb, BCT, disk-shaped
High resolution EDS analysis for aged sample

- $\gamma'$: Ni$_3$(Ti,Al), FCC, spherical or cubic
- $\gamma''$: Ni$_3$Nb, BCT, disk-shaped

![EDS analysis](image)
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Potential factors for hardness change:

- **Microstructure:** Precipitates (i.e., Laves phase & delta) and grain size
- **Chemistry:** Compositional homogeneity
EBSD results showing grain growth

As-built  1100°C, 8hours, WQ  1200°C, 8hours, AC

Sampling region is relatively small for the large grains. Further analysis with larger sampling region will be done to accurate measure the grain size.
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Porosities in the middle of the samples after heat treatment

- **As-built sample**
- **1200°C, 2hours, WQ**
- **1200°C, 8hours, WQ**

**Question:** Why there are severe porosities formed after high temperature heat treatment?

- Recalling the shielding gas used for heating and holding steps: Ar (0.97 liter per min)

**Procedure:** Exam surfaces using optical microscopy and SEM
Preparation of the samples

Surface appearance after heat treatment

* Original surface on which SEM images were taken

Sample cutting

* New surface for observing surface oxidization
* Center of this new surface not exposed to shielding gas

Cross section of sample
Confirmation of the oxidation layer

- Cracking and porosity features observed in all the exterior surfaces exposed to shielding gas
- Such features not observed in the unexposed interior
- Therefore, it is determined to be oxidation formed during high-temperature heat treatment
Comparison with literature data

**This study**

- 1200°C, 8hours, WQ
- 1200°C, 2hours, FAC

**Literature**

- Cr$_2$O$_3$
- γ (Ni-Cr-Fe) matrix

1. External oxidation scale
2. Internal oxidation zone: thread like oxides

- 1. Intergranular oxides penetrations

- Wrought


- Further analysis ongoing to understand the severity of oxidation with improved shielding condition (e.g., higher flow rate of Ar)
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Summary and conclusions

1. Characterization of as-built microstructures:
   - Fine dendrites with continuous network of the Laves phase
   - NbC and delta, two other common precipitates, were not observed

2. Post-built heat treatment:
   - Laves phase dissolved more at higher temperature and longer hold time. There was still a small amount of Laves remaining after homogenization at 1200°C for 8 hours.
   - Significant grain growth took place.
   - Delta phase appeared in casting condition (1093°C 2hrs AC + 982°C 1hr AC), which could have a detrimental effect on the mechanical properties.
   - Oxidation layers occurred on the exposed surfaces.

3. Microstructure after aging
   - Hardness comparable to wrought condition
   - Formation of strengthening precipitates (γ” and γ’)

Plans for future work

1. Characterization for heat treatment samples
   - Homogenization to completely dissolve the Laves phase

2. Creep test for aging conditions of INCONEL 718
Thank you
Effect of heat treatment on microstructures

• **Homogenization:**
  - Decreased hardness value (30%) for the higher peak temperature (1100°C and 1200°C)
  - Decreased hardness value (20%) for casting condition (1093°C 2hrs AC + 982°C 1hr AC)
  - Increased grain size (> 200%) for the higher peak temperature (1100°C and 1200°C)
  - Control factors of hardness value: holding time and peak temperature

• **Homogenization + aging (without solution annealing):**
  - Formation of strengthening precipitates (γ’ and γ’’’) even though Laves phase was not completely eliminated in the homogenization step
Oxidation process at high temperature atmosphere

- Oxygen molecules can collide with the sample surface, breaking into oxygen atoms

- Chemical absorption by interaction between the free electrons of the base alloy and the oxygen atoms

- The nucleated oxides grow perpendicular to the surface of the base alloy
  → Pores can act as the crack initiators due to stress concentrations
**Peak temperature and time**

Remaining Laves phase fraction as function of homogenization temperature and time

\[ R = \exp \left[ \frac{-2.48 \times 10^{-19} \times t}{\exp(-0.036 \times T)} \right] \times 100 \]

<table>
<thead>
<tr>
<th>Time (hour)</th>
<th>Temp. (°C)</th>
<th>Remaining Laves phase fraction (%)</th>
<th>Time (hour)</th>
<th>Temp. (°C)</th>
<th>Remaining Laves phase fraction (%)</th>
<th>Time (hour)</th>
<th>Temp. (°C)</th>
<th>Remaining Laves phase fraction (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>1100</td>
<td>96.16</td>
<td>1</td>
<td>1140*</td>
<td>84.78</td>
<td>1</td>
<td>1200</td>
<td>23.88</td>
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<td>1100</td>
<td>92.47</td>
<td>2</td>
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<td>2</td>
<td>1200</td>
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<td>1200</td>
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<tr>
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<td>0</td>
<td>60</td>
<td>1140*</td>
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</tr>
</tbody>
</table>

Comparison of hardness maps for homogenized samples

Homogenization process: 1100°C, 2hrs, AC

<table>
<thead>
<tr>
<th>Depth (μm)</th>
<th>Width (μm)</th>
<th>Micro Hardness (VHN)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>0</td>
<td>200</td>
</tr>
<tr>
<td>10</td>
<td>5000</td>
<td>280</td>
</tr>
<tr>
<td>20</td>
<td>5000</td>
<td>260</td>
</tr>
<tr>
<td>30</td>
<td>5000</td>
<td>240</td>
</tr>
<tr>
<td>40</td>
<td>5000</td>
<td>220</td>
</tr>
</tbody>
</table>

Load: 300g

Avg. 248
Max. 273
Min. 204

Homogenization process: 1100°C, 2hrs, Force-AC

<table>
<thead>
<tr>
<th>Depth (μm)</th>
<th>Width (μm)</th>
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</tr>
</thead>
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<td>240</td>
</tr>
<tr>
<td>40</td>
<td>5000</td>
<td>220</td>
</tr>
</tbody>
</table>

Load: 300g

Avg. 262
Max. 278
Min. 241

1100°C, 2hrs and 953°C 1hr, AC

<table>
<thead>
<tr>
<th>Avg.</th>
<th>Max.</th>
<th>Min.</th>
</tr>
</thead>
<tbody>
<tr>
<td>251</td>
<td>292</td>
<td>200</td>
</tr>
</tbody>
</table>

The same scale: 200 ~ 290 HVN
Comparison of hardness maps for homogenized samples

The same scale: 200 ~ 290 HVN

**Homogenization process: 1100°C, 2hrs, AC**

<table>
<thead>
<tr>
<th>1100°C, 2hrs, AC</th>
</tr>
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<tbody>
<tr>
<td>Avg.</td>
</tr>
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<td>Max.</td>
</tr>
<tr>
<td>Min.</td>
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</tbody>
</table>

**Homogenization process: 1100°C, 2hrs, Forced-AC**

<table>
<thead>
<tr>
<th>1100°C, 2hrs, Forced-AC</th>
</tr>
</thead>
<tbody>
<tr>
<td>Avg.</td>
</tr>
<tr>
<td>Max.</td>
</tr>
<tr>
<td>Min.</td>
</tr>
</tbody>
</table>

**Homogenization process: 1100°C, 8hrs, WC**

<table>
<thead>
<tr>
<th>1100°C, 8hrs, WC</th>
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<tbody>
<tr>
<td>Avg.</td>
</tr>
<tr>
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</tr>
<tr>
<td>Min.</td>
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</table>
Comparison of hardness maps for homogenized samples

The same scale: 210 ~ 360 HVN

Homogenization process: 1100°C, 2hrs, WQ

<table>
<thead>
<tr>
<th>Depth (µm)</th>
<th>Width (µm)</th>
<th>Micro hardness (VHN)</th>
</tr>
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<tbody>
<tr>
<td>0</td>
<td>0</td>
<td>Load: 300g</td>
</tr>
<tr>
<td>2</td>
<td>2</td>
<td>12x10³</td>
</tr>
<tr>
<td>4</td>
<td>4</td>
<td>10</td>
</tr>
<tr>
<td>6</td>
<td>6</td>
<td>8</td>
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<table>
<thead>
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Homogenization process: 1100°C, 8hrs, WQ

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<tr>
<td>Min.</td>
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</tbody>
</table>
• The grain growth as the higher homogenization temperature
• Increased grain diameter (> 50%) on water quenched condition for the same peak temp.
The reduced hardness values show the higher misorientation angles. However, little difference in the misorientation angles all peak temperatures.
Difficult to decide the effect of high amount of misorientation angles due to small number of grains in heat treated conditions
Schematic of non-uniform precipitation

- **Nb rich PPT.**
- **Nb depleted area**

As-received condition

- **Nb diffusion along G.B.**
- **Very slow lattice diffusion**

At temp., prior to deformation

- **PPT. along prior G.B.**
- **Newly recrystallized grains (Nb depleted area)**

Aging response, after deformation

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