Microstructural Characterization and Modeling of SLM Superalloy 718

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Motivation for Microstructural Modeling

- A number of modeling tools are being developed to support rapid flight certification of SLM 718 components for the SLS engine under NASA’s Material Genome Initiative program.

- Post-processing heat treatment of SLM 718 components is required for consolidation and to obtain optimal mechanical properties.

Background

- Commercial software packages based on CALPHAD-based methods have been developed to predict microstructure.

- Accurate microstructural measurements are needed to “tune” these models, i.e. compare, calibrate and then validate model predictions to experimental values.
Objective

• To obtain accurate microstructural measurements that will enable a model that can predict microstructure well over a range of relevant heat treat conditions.

Approach

• $\frac{1}{2}$-inch diameter rods of superalloy 718 were fabricated using SLM on MSFC’s M2 Concept Laser.

• All section pieces were stress relieved at high temperature, cut from build plate, then hot isostatic pressed (HIP).

• The thermal history and alloy composition were used as inputs into the Pandat 2013 precipitation models.

• Detailed microstructural measurements of the precipitates were performed to verify the model predictions.
Superalloy 718

- Superalloy 718 is a great candidate for additive manufacturing
  - Used in a wide range of high temperature and aerospace applications for decades
  - Has good welding properties
  - Thermodynamic and kinetic databases are well developed
- Superalloy 718's base composition (51Ni-22Fe-19Cr-5Nb-3Mo-1Co-1Ti).
- Superalloy 718 utilizes three intermetallic precipitation phases.
  - γ' (Ni₃(Al, Ti))
  - γ'' (Ni₃Nb)
  - δ (Ni₃Nb), orthorhombic DO₉
- Precipitates along GB’s
- Due to the size and morphology of these precipitates, accurately characterizing them has been a difficult endeavor.
Computherm Pandat Modeling

<table>
<thead>
<tr>
<th>wt.%</th>
<th>Ni</th>
<th>Al</th>
<th>Co</th>
<th>Cr</th>
<th>Fe</th>
<th>Mo</th>
<th>Nb</th>
<th>Ti</th>
<th>W</th>
</tr>
</thead>
<tbody>
<tr>
<td>SLM 718</td>
<td>53.19</td>
<td>0.5</td>
<td>0.09</td>
<td>18.1</td>
<td>18.9</td>
<td>3.1</td>
<td>5.1</td>
<td>1.0</td>
<td>0.02</td>
</tr>
</tbody>
</table>

Computherm Pandat 2013 PanPrecipitation Module

- First precipitation package to allow users to apply thermal history to an initial microstructure, as well as standard homogenized alloy chemistry
- Computherm has worked closely with the Air Force Research Laboratory (AFRL) on superalloy 718 database development: PanNi_MB_2013 is their combined thermodynamic / kinetic databases.
Sample Preparation of SLM Superalloy 718

- Superalloy 718 specimens were fabricated by SLM on MSFC Concept Laser tool.

**Thermal post-processing steps – ASTM standard**

1. **Stress relief**
   - Under argon: 1950°F / 1.5hr + gas quench

2. **HIP** (optional)
   - Under vacuum: 2125°F / 1hr + gas quench

3. **Homogenization**
   - Under vacuum: 1950 or 1850 or 1700 °F 1hr + gas quench

4. **Solution heat treatment**
   - Under vacuum: 1950 or 1850 or 1700 °F 1hr + gas quench

5. **Aging**
   - Two Step Age 1 or Age 2

Age 1: 1325°F/10hr + FC to 1150°F + 1150°F/≈6hr (until total time is 18hr)
Age 2: 1400°F/10hr + FC to 1200°F + 1200°F/≈8hr (until total time is 20hr)

**Set 1 - Homogenized**

- Z41: SR + HIP + Homo + Sol 1950 + Age 1
- Z18: SR + HIP + Homo + Sol 1850 + Age 1
- Z1: SR + HIP + Homo + Sol 1850 + Age 2
- Z25: SR + HIP + Homo + Sol 1700 + Age 1

**Set 2 – Not homogenized**

- Z8: SR + HIP + Sol 1850 + Age 1
- Z3: SR + HIP + Sol 1850 + Age 2
- Z27: SR + HIP + Sol 1700 + Age 1
Microstructural Characterization – Precipitates
New Technique: HR-SEM

- New high resolution SEMs allow for direct imaging of $\gamma'/\gamma''$ precipitates when preferentially etched.

- Imaging at 3kV using a secondary electron detector eliminates sample thickness/overlap problems.

- Using precipitate morphology (Aspect ratio), $\gamma'$ precipitates can be separated from $\gamma''$. (Orientation dependent).

Etched with a solution of 50mL Lactic Acid 30mL Nitric Acid and 2mL HF

Z1 – Age 2

500 nm
Microstructural Characterization - EBSD

EBSD Map

SEM - Microstructure

[111] - Volume fraction analysis and γ' size analysis
[001] - γ" size analysis

Quantifying morphology distinction between precipitates…
Microstructural Characterization – Precipitates (EDS)

HAADF

Z1 – Age 2

EDS Map

Acquired from a FEI Talos (S)TEM

γ' - (Ni₃(Al, Ti))  γ'' - (Ni₃Nb)
Microstructural Characterization – Precipitates (EDS)
Z1 – Age 2

Determining Aspect Ratios

$\gamma'$ - (Ni$_3$(Al, Ti))  $\gamma''$ - (Ni$_3$Nb)

One-way Analysis of Aspect Ratio By Precipitate Type
Microstructural Characterization – Precipitates (EDS)

Z1 – Age 2

Determining Aspect Ratios

Density Map

- Age 1 cutoff ratio: 1.8
- Age 2 cutoff ratio: 2.25

Note: Presence of composite particles!
Microstructural Characterization – Precipitates
SEM Vibration/Distortion Correction

Z1 – Age 2

No Correction

Scan Corrected

At low magnifications there isn’t a noticeable difference…
Microstructural Characterization – Precipitates
SEM Vibration/Distortion Correction

Z1 – Age 2

No Correction

Scan Corrected

However, at high magnifications it is very noticeable!

*C. Ophus, J. Ciston. Ultramicroscopy 2015*
Microstructural Characterization – Precipitates

Procedure

Z1 – Age 2

Scan-corrected
Microstructural Characterization – Precipitates

Procedure

Z1 – Age 2

Normalize contrast and brightness: adaptive threshold: make binary (ImageJ)

150 nm
Microstructural Characterization – Precipitates Procedure

Watershed by hand (ImageJ)

Currently working on automating this process

Z1 – Age 2

150 nm
Microstructural Characterization – Precipitates

Procedure

Z1 – Age 2

Separate precipitates using aspect ratio cutoffs determined using EDS (ImageJ)

150 nm
Microstructural Characterization – Precipitates Procedure

Z1 – Age 2

Repair composite γ' precipitates (ImageJ)
Microstructural Characterization – Precipitates

Procedure

Z1 – Age 2

Same steps for γ'' precipitates. Merge Images. Extract statistics (Size and area fractions for both γ' and γ'') (ImageJ). Repeat until at least >500 particles from each phase is analyzed.
Microstructural Characterization – δ Precipitates

Etched Surface

Thresholded Image

<table>
<thead>
<tr>
<th>Precipitate Parameter</th>
<th>Experimental</th>
<th>Model</th>
</tr>
</thead>
<tbody>
<tr>
<td>δ area percent</td>
<td>.369 ± .24 %</td>
<td>2.0 %</td>
</tr>
<tr>
<td>δ average size</td>
<td>.03 ± .01 um²</td>
<td></td>
</tr>
<tr>
<td>δ feret dia.</td>
<td>.69 ± .15 um</td>
<td></td>
</tr>
</tbody>
</table>
XRD – Volume Fraction Validation

<table>
<thead>
<tr>
<th>Phase ID (4)</th>
<th>Chemical Formula</th>
<th>PDF-#</th>
<th>Space Group</th>
<th>Vol% (esd)</th>
<th>Wt% (esd)</th>
<th>a (esd)</th>
<th>c (esd)</th>
</tr>
</thead>
<tbody>
<tr>
<td>γ'/γ&quot;</td>
<td>IN$_7$T$_8$</td>
<td>98-000-1033</td>
<td>cFm-3m (225)</td>
<td>89.34 (3.11)</td>
<td>88.30 (2.26)</td>
<td>3.59588 (17)</td>
<td>3.59588 (17)</td>
</tr>
<tr>
<td>γ&quot;</td>
<td>IN$_7$T$_8$</td>
<td>98-000-1036</td>
<td>tI4/mmm (139)</td>
<td>10.57 (0.63)</td>
<td>11.62 (0.63)</td>
<td>3.61425 (63)</td>
<td>7.42937 (132)</td>
</tr>
<tr>
<td>NbC</td>
<td>NbC</td>
<td>04-001-1554</td>
<td>cFm-3m (225)</td>
<td>0.09 (0.02)</td>
<td>0.09 (0.02)</td>
<td>4.43000 (0)</td>
<td>4.43000 (0)</td>
</tr>
<tr>
<td>δ</td>
<td>IN$_7$T$_8$</td>
<td>98-000-1035</td>
<td>oPmnn² (59)</td>
<td>0.00 (0.00)</td>
<td>0.00 (0.00)</td>
<td>5.14100 (0)</td>
<td>4.53400 (0)</td>
</tr>
</tbody>
</table>

Refinement Failed (R/E=13.92), Round=4, Iter=6, P=31, R=9.0% (E=0.65%, EPS=0.5)

Crystal structure of γ and γ' phases are to similar to separate in XRD

<table>
<thead>
<tr>
<th>Precipitate Parameter</th>
<th>SEM</th>
<th>XRD</th>
</tr>
</thead>
<tbody>
<tr>
<td>γ' volume fraction</td>
<td>5.1 ± 0.8 %</td>
<td>N/A</td>
</tr>
<tr>
<td>γ&quot; volume fraction</td>
<td>11.1 ± 0.9 %</td>
<td>10.6 ± 0.6</td>
</tr>
<tr>
<td>δ volume fraction</td>
<td>.37 ± .24 %</td>
<td>≈ 0 %</td>
</tr>
</tbody>
</table>
### Phase Extraction

<table>
<thead>
<tr>
<th>Precipitate Parameter</th>
<th>Experimental</th>
<th>Phase Extraction</th>
</tr>
</thead>
<tbody>
<tr>
<td>$(\gamma' / \gamma'' / \delta)$ volume fraction</td>
<td>16.6 ± 1.2 %</td>
<td>15.7 %</td>
</tr>
</tbody>
</table>

Can not separate $\gamma'/\gamma''/\delta$ phase due to similar chemistries

### XRD and Phase Extraction Combined

<table>
<thead>
<tr>
<th>Precipitate Parameter</th>
<th>SEM</th>
<th>XRD + PE</th>
<th>Model</th>
</tr>
</thead>
<tbody>
<tr>
<td>$\gamma'$ volume fraction</td>
<td>5.1 ± 0.8 %</td>
<td>5.1 ± 0.6</td>
<td>2 %</td>
</tr>
<tr>
<td>$\gamma''$ volume fraction</td>
<td>11.1 ± 0.9 %</td>
<td>10.6 ± 0.6</td>
<td>14 %</td>
</tr>
<tr>
<td>$\delta$ volume fraction</td>
<td>.37 ± .24 %</td>
<td>0 %</td>
<td>2 %</td>
</tr>
</tbody>
</table>

The XRD + PE analysis validates the new SEM characterization technique!
Microstructural Analysis – Results
Gamma Prime Phase

**γ' Area Fractions**

![Bar chart showing γ' area fractions for different samples with error bars indicating 95% confidence interval.]

- **Z8**: 2.4
- **Z3**: 2.0
- **Z27**: 2.1
- **Z41**: 2.4
- **Z18**: 2.3
- **Z1**: 2.6
- **Z25**: 2.2

- **Error bars**: 95% confidence interval
- **Legend**:
  - Red: Experimental
  - Pink: Model
Microstructural Analysis – Results
Gamma Double Prime Phase

\( \gamma'' \) Area Fractions

<table>
<thead>
<tr>
<th>Sample</th>
<th>Experimental</th>
<th>Model</th>
</tr>
</thead>
<tbody>
<tr>
<td>Z8</td>
<td>0.7</td>
<td>1.0</td>
</tr>
<tr>
<td>Z3</td>
<td>0.7</td>
<td>1.4</td>
</tr>
<tr>
<td>Z27</td>
<td>0.8</td>
<td></td>
</tr>
<tr>
<td>Z41</td>
<td>0.8</td>
<td></td>
</tr>
<tr>
<td>Z18</td>
<td>0.7</td>
<td></td>
</tr>
<tr>
<td>Z1</td>
<td>0.8</td>
<td></td>
</tr>
<tr>
<td>Z25</td>
<td>0.7</td>
<td></td>
</tr>
</tbody>
</table>

Error bars = 95% confidence interval
Microstructural Analysis – Results
Gamma Double Prime Phase

γ″ Size

Length (nm)

Sample

Z8  Z3   Z27  Z41  Z18  Z1   Z25

Error bars = 95% confidence interval

Experimental
Model
Methodology – 3D Size distributions

γ'' Size Analysis: [001] oriented grains
γ' Size Analysis: Any orientation

Using the measured area size distributions of each precipitate, the numerical volumetric size distributions were calculated using the equation below assuming a spherical particle*. This works for γ' for all orientations. For γ'' precipitates, it must be performed only on the two edge-on variants of γ'' in [001] oriented grains.

\[
(N_v)_j = \frac{1}{\Delta} \sum_{i=j}^{k} \alpha_i (N_A)_i
\]

Where \(N_A\) is the experimentally obtained area number densities, \(D_{\text{max}}=k\Delta\), and \(k\) equals the total number of size groups. \(\alpha\) is a pre-determined coefficients associated with the probability of the polish surface plane cutting a sphere as revealed below.

\[
P_{i,j} = \frac{1}{r_{\text{max}}} \left[ \sqrt{(r_{\text{max}}^2) - (r_{i-1})^2} - \sqrt{(r_{\text{max}}^2) - (r_i)^2} \right]
\]

*Stereoology and Quantitative Metallography, ASTM, STP 504
γ' precipitates possess a mostly normal size distribution.
γ" Size Distributions

γ" precipitates do not possess a normal size distribution.
Discussion

**Experimental**
- Composite particles are not completely separated (esp. Age 1 samples).

**Assumptions:**
- Perfectly etched samples
- \( \gamma' \) are spherical, \( \gamma'' \) are circular plates.
- No subsurface features are imaged.

**Future work:** further automate post-processing procedure and find more accurate ways to separate \( \gamma'/\gamma'' \) composite particles.

**Model**
- Carbides/Oxides were suspended to simplify calculations
- Inter-particle interactions not well established.

**Tuning Parameters:**
- Compatible thermodynamic database
- Compatible mobility database
- \( \Delta E \) – phase energy shift for equilibrium phase fractions
- \( D_{\text{scale}} \) – Diffusivity correction factor
- Molar volume for each phase
- Coherent surface energy (mJ/m\(^2\))
- Lattice misfit energy (mJ/m\(^2\))
- Incoherent surface energy (mJ/m\(^2\))
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- Molar volume for each phase
- **Coherent surface energy** (mJ/m$^2$)
- Lattice misfit energy (mJ/m$^2$)
- Incoherent surface energy (mJ/m$^2$)
Conclusions

- A new method using high resolution scanning electron microscopy combined with advanced processing techniques allows for unprecedented microstructural characterization of additively manufactured superalloy 718.

- XRD and Phase extraction support the findings from the SEM analysis.

- Differences in γ" and γ' size distributions are currently unexplained.

- Currently, the precipitation models predict the microstructural trends resulting from different post-processing heat treatment steps.

- Calibrating future precipitation models using results from this new technique will further improve their accuracy.
Acknowledgments: Microstructural Characterization and Modeling of SLM Superalloy 718

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Analytical Chemistry
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- Metallography
- X-ray Diffraction
- Computed Tomography

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Questions?