Small variations in powder composition lead to strong differences in part properties

Insights from an Al-Sc alloy and a Ni-base superalloy

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Outline

1. Strength variations in an Al-Sc alloy (Scalmalloy®):

2. Hot cracking in a Nickel-base superalloy: Inconel 738LC
Aluminium-Scandium Alloys

- **Strengthening** achieved by precipitation of $\text{Al}_3(\text{Sc},\text{Zr})$ nanoparticles upon ageing heat treatment
- Scalmalloy®: AlMgScZr-alloys with hypereutectoid content of scandium developed by Airbus
- **Rapid quenching** necessary to retain supersaturation: melt spinning + hot compaction or LAM can be employed

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**AlSi10Mg**

- **Ultimate tensile strength**
- **Yield strength**
- **Elongation to fracture**

produced by SLM

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The problem – unexpected drop in strength

- Same powder composition
- Similar process parameters
- Different powder atomization supplier
- Different SLM-machine vendor

- defects?
- precipitates?
- grain size?
## Sample overview – low ductility and strength?

<table>
<thead>
<tr>
<th>SAMPLE</th>
<th>2</th>
<th>3</th>
<th>5</th>
</tr>
</thead>
<tbody>
<tr>
<td>Powder atomization</td>
<td>VIGA</td>
<td>EIGA</td>
<td>EIGA</td>
</tr>
<tr>
<td>SLM machine</td>
<td>Concept Laser</td>
<td>Concept Laser</td>
<td>SLM Solutions</td>
</tr>
<tr>
<td>Post-heat treatment</td>
<td>HT</td>
<td>HT + HIP</td>
<td>HT</td>
</tr>
<tr>
<td>Yield strength / MPa</td>
<td>520</td>
<td>437</td>
<td>395</td>
</tr>
<tr>
<td>Ductility / %</td>
<td>14</td>
<td>17</td>
<td>7.4</td>
</tr>
</tbody>
</table>

**EIGA**: Electrode Inert Gas Atomization / **VIGA**: Vacuum Inert Gas Atomization

**HT**: Heat treatment (4 h@ 325°C)

**HIP**: Hot Isostatic Pressing (2h @ 325°C)
As-produced HIPed

- Unmelted powder particles and bonding defects in samples produced by the SLM Solutions machine (samples 4 – 6)
- Bonding defects are not healed by HIPing!
Low ductility – bonding defects on fracture surf.

<table>
<thead>
<tr>
<th>SAMPLE</th>
<th>3</th>
<th>5</th>
<th>6</th>
</tr>
</thead>
<tbody>
<tr>
<td>Powder</td>
<td>EIGA</td>
<td>EIGA</td>
<td>EIGA</td>
</tr>
<tr>
<td>machine</td>
<td>Concept laser</td>
<td>SLM Solutions</td>
<td>SLM Solutions</td>
</tr>
<tr>
<td>heat treatment</td>
<td>HT + HIP</td>
<td>HT</td>
<td>HT + HIP</td>
</tr>
<tr>
<td>ductility</td>
<td>↑</td>
<td>↓</td>
<td>↓</td>
</tr>
<tr>
<td>yield strength</td>
<td>↓</td>
<td>↓</td>
<td>↓</td>
</tr>
<tr>
<td>Defect area fraction</td>
<td>Close to 0%</td>
<td>13.68%</td>
<td>14.62%</td>
</tr>
</tbody>
</table>

⇒ Bonding defects are the reason for low ductility
⇒ They cannot explain low yield strength (cf. sample 3)
Influences of unbonded zones on yield stress

- Unbonded zones lead to a reduction of load-bearing area in cross section
- Decrease of apparent yield stress

But hardness measurement unaffected by bonding defects!
- Bonding defects can’t explain drop in hardness
The problem – unexpected drop in strength

- Same powder composition
- Similar process parameters
- Different powder atomization supplier
- Different SLM-machine vendor

![Graph showing yield stress comparison between reference and bad sample.]

- defects? ✓
- precipitates?
- grain size?
Coarse precipitates in AP-material and powder

- Large precipitates (20-50 µm diameter) in the SLM-produced parts are identified by EDX as $\text{Al}_3(\text{Sc,Zr})$.
- They are **only** present in powder atomized by the EIGA process.
- Presumably the time in the liquid state is not long enough to dissolve them.

courtesy of Airbus Defense and Space
Large precipitates: low strength, but high ductility

- Large precipitates apparently don’t deteriorate ductility!
- Cup-and-cone fracture around large precipitates
- Maybe they decrease strength by “consuming” solute, which then cannot lead to nm-size precipitates?
Influences of unexpected precipitates

- Large precipitates only “consume” about 0.1% of Sc!
- Does this explain the remaining difference in yield strength?

<table>
<thead>
<tr>
<th>Elements</th>
<th>nominal composition</th>
<th>composition sample 3</th>
<th>change</th>
</tr>
</thead>
<tbody>
<tr>
<td>Mg</td>
<td>4.50</td>
<td>4.52</td>
<td>+0.02</td>
</tr>
<tr>
<td>Al</td>
<td>93.87</td>
<td>94.02</td>
<td>+0.15</td>
</tr>
<tr>
<td>Si</td>
<td>0.17</td>
<td>0.17</td>
<td>0.00</td>
</tr>
<tr>
<td>Sc</td>
<td>0.66</td>
<td>0.57</td>
<td>-0.09</td>
</tr>
<tr>
<td>Mn</td>
<td>0.50</td>
<td>0.50</td>
<td>0.00</td>
</tr>
<tr>
<td>Zr</td>
<td>0.30</td>
<td>0.23</td>
<td>-0.07</td>
</tr>
</tbody>
</table>

Matrix

<table>
<thead>
<tr>
<th>Area fraction</th>
<th>Precipitates</th>
</tr>
</thead>
<tbody>
<tr>
<td>99.69%</td>
<td>0.31%</td>
</tr>
</tbody>
</table>

Sc and Zr concentrations in at.-%

- Sc
- Zr

Graph showing Sc and Zr concentrations in at.-% for samples 1, 2, and 3.
Atom Probe Tomography of Al₃Sc

<table>
<thead>
<tr>
<th>Sample No.</th>
<th>2</th>
<th>5</th>
</tr>
</thead>
<tbody>
<tr>
<td>Number density ($\times 10^{23}/m^3$)</td>
<td>7.06</td>
<td>4.98</td>
</tr>
<tr>
<td>Average radius (nm)</td>
<td>1.57</td>
<td>1.80</td>
</tr>
<tr>
<td>Volume fraction</td>
<td>~2%</td>
<td>~2%</td>
</tr>
</tbody>
</table>

⇒ Only small influence of coarse precipitates on secondary precipitates.
Estimation of strengthening by $\text{Al}_3\text{Sc}$

Bypassing mechanism:

$$\tau_{OR} = \frac{G b \sqrt{f}}{r}$$

Cutting mechanism:

$$\tau_c = \gamma^{3/2} f \sqrt{\frac{r}{6 E_v}}$$

Precipitate strengthening follows cutting mechanism due to the size.

⇒ Sample 5 should be even a little stronger than sample 2!
The problem – unexpected drop in strength

- Bonding defects and large ppts alone cannot explain drop in strength
- Strong difference in the fraction of small grains depends on the atomization process (!)
Grain refinement by intermediate precipitates?

Different number of medium-sized precipitates (20-50 nm) – grain refinement during re-melting?
Different fractions of equiaxed grains

Area fraction of equiaxed grains depends on atomization method!
No significant influence of grain size on hardness (and hence strength)
The problem – unexpected drop in strength

- Same powder composition
- Similar process parameters
- Different powder atomization supplier
- Different SLM-machine vendor

Reduced cross sectional area due to bonding defects? ✔

Fewer nm-sized precipitates due to large precipitates? ✗

Larger volume fraction of small, equiaxed grains? ✗
Observations of precipitates free zones

- Width of PFZs is significant compared to radius of small, equiaxed grains
Precipitates free zone

Sample No. 2 5
Total width / nm 38 70
Width of ppt / nm 18 28
Width of PFZ / nm 19 42

- Soft zone around PFZ may be 10 times wider than PFZ\(^1\).
- Considering of the fine grain size (less than 1 \(\mu m\)), the PFZs may contribute significantly to low yield strength.

\(^1\)Fourmeau et al., Phil. Mag., 95 (2015) 3278
Small changes yield large differences

- Bonding defects due to poor process parameters explain difference in ductility and partially explain difference in strength
- Different atomization process leads to coarse precipitates, that in turn
  - bind Sc and Zr
  - lead to a lower number of intermediate-size precipitates
  - lead to a lower fraction of equiaxed grains
  - lead to wider PFZs

![Graph showing yield stress comparison between reference and bad sample](chart)

- Reduced cross sectional area due to bonding defects?
- Wider PFZs may contribute to lower strength

![Chart indicating yield stress comparison](chart)
Outline

1. Strength variation in an Al-Sc alloy (Scalmalloy®):

2. Hot cracking in a Nickel-base superalloy: Inconel 738LC
Problem: hot cracking in IN738LC

Cracks are oriented parallel to build direction

Intergranular microcracks

worst case sample
What type of cracking?

1. Liquation cracks?
2. Strain age cracks?
3. Solidification cracks?

- no low-melting phases
- cracking without presence of $\gamma'$

Hot cracks are probably solidification cracks
Solidification cracking

Tensile stresses develop upon solidification and cooling shrinkage

\[ \downarrow \]

Liquid film persists to low temperatures

\[ \downarrow \]

Liquid film ruptures

\[ \downarrow \]

cracking

\[ \Rightarrow \]

Microsegregation during solidification exacerbates the problem by lowering solidus temperature
Crack chemistry part I

Auger Electron Spectroscopy

Cracked region

Non-cracked region

Atom Probe Tomography

Impact in analysis chamber

Si and Zr enrichment on crack surface
Solidification cracking theory: Rappaz/Boettinger

- attractive, i.e., LAGB
  - at $T=T_m$
  - $\gamma_{GB} < 2\gamma_{SL}$
  - at $T < T_m$
  - liquid film thickness
  - $\gamma_{gb}$
  - $2\gamma_{sl}$
  - $\Rightarrow$ below melting point, liquid film is unstable

- repulsive, i.e., HAGB
  - at $T=T_m$
  - $\gamma_{GB} > 2\gamma_{SL}$
  - at $T < T_m$
  - liquid film thickness
  - $\gamma_{gb}$
  - $\Rightarrow$ there is a stable liquid film thickness even below $T_m$

Undercooling to make liquid film unstable: $\Delta T_b = \frac{\gamma_{gb} - 2\gamma_{sl}}{\Delta S_f} \frac{1}{\delta}$

Cracked GB misorientation

Only HAGBs show cracking, as predicted by theory.
Crack chemistry part II

There is a lot of Zr on HAGBs but none on LAGBs.
Mystery: Why is Zr high on HAGBs?


Fig. 7—Evolution of the average composition of solid and liquid as a function of temperature as predicted with the sharp interface model and the parameters of the left column of Table I ($D_L = 10^{-13} \text{m}^2\text{s}^{-1}$, $\gamma_{sl} = 0.1 \text{Jm}^{-2}$, $\gamma_{gb} = 0.3 \text{Jm}^{-2}$, $\lambda/2 = 10 \mu\text{m}$, and $\delta = 1 \text{nm}$). The dashed line corresponds to the coalescence line.

Fig. 8—Influence of the grain-boundary energy on the deviation from the liquidus line during the last stage of solidification, as predicted with the sharp interface model and the parameters of the left column of Table I for three types of interfaces: attractive ($\gamma_{gb} = 0$), neutral ($\gamma_{gb} = 2\gamma_{sl}$), and repulsive ($\gamma_{gb} = 3\gamma_{sl}$).

➡️ Model that correctly predicts misorientation-dependence of cracking predicts **opposite** behaviour of Zr $\mu$-segregation

attractive, i.e. LAGB
repulsive, i.e. HAGB
in our case: Zr conc.
Samples with low Zr/Si content?

**Experiments**

- Cracking vanishes **completely** when Zr & Si are reduced (0.04% difference!)
- Cracking intensifies when C is reduced (probably due to less binding of Zr & Si in carbides)
- Solidus temperature increases by \( \sim 25 K \) when excluding Zr, supporting \( \mu \) segregation as reason for solidification cracking

**Thermocalc Simulation (TTNi8)**

<table>
<thead>
<tr>
<th></th>
<th>high Zr, Si</th>
<th>low Zr, Si</th>
</tr>
</thead>
<tbody>
<tr>
<td>Zr concentration</td>
<td>0.06 wt.-%</td>
<td>0.02 wt.-%</td>
</tr>
<tr>
<td>Si concentration</td>
<td>0.07 wt.-%</td>
<td>0.01 wt.-%</td>
</tr>
<tr>
<td>liquidus</td>
<td>1346°C</td>
<td>1348°C</td>
</tr>
<tr>
<td>solidus – equilibrium</td>
<td>1267°C</td>
<td>1282°C</td>
</tr>
<tr>
<td>solidus – Scheil</td>
<td>1058°C</td>
<td>1083°C</td>
</tr>
</tbody>
</table>

\( \rightarrow +2K \) \( \rightarrow +15K \) \( \rightarrow +25K \)
Conclusions

Small variations in
- powder composition,
- powder atomization,
- process parameters,
- post-processing

can lead to large changes in
- defect density,
- precipitation,
- grain size,
- yield strength and ductility.

Two examples:
- Unexpected drop of yield strength in an Al-Sc alloy
- Elimination of hot cracking in a Ni-base superalloy by Zr & Si reduction
Thank you for your attention.

E. A. Jägle, L. Wu, F. Palm, D. Raabe, *in preparation*
E. A. Jägle, L. Lu, J. Risse, D. Raabe, *in preparation*

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