Effect of minor elements on properties of C300 Maraging steel parts by SLM process

5th Edition of the Additive World Conference on industrial 3D printing
Eindhoven, March 15-16
Key Powder Manufacturing Processes and Resulting Powder Characteristics

- Powder shape and morphology
- Powder size distribution & density
- Surface Area (BET analysis)

- Powder flow rate (Hall or Carney)
- Chemistry, impurities & gas content
- Phase composition

[Images of different powders: Gas Atomized, Water Atomized, Fused & Crushed, Aggl & Sint, Sintered & Crushed, ... other processes: HDH (hydride-dehydride) and chemical purification, Sol-gel]
Feed metals are melted under vacuum and the molten stream is fed to the lower chamber. High velocity gas jets atomize the stream to form fine droplets. These droplets rapidly solidify as spherical particles.

- Versatile process with many compositions and applications
- Fully alloyed composite metal powder
- Fine microstructures are attained
- Rounded shape, good flow properties
- High purity

Typical Morphology of MetcoClad™ 21 Gas-Atomized Powder
Powder Manufacturing – R&D Atomizer

Melt stream being atomized

Pilot Atomizer

Real-time monitoring of melting and atomization
Commonality in Materials

- Several of the alloys and composites used in Laser Cladding and SLM are often similar to those used in Thermal Spray applications and/or PTA.

- However, they may or may not use a TS size fraction.
Powder Size Effects

- Better surface finish
- More robust process, best DE
- Lower powder cost
- Less porosity
- Higher deposition rate

Powder size μm

180 150 120 75 53 45
The Need for AM specific Powder Development

- **Driver:**
  - Materials for AM often based on specification from cast / forged / heat-treated materials or materials which were developed for another process

But:

- **Different cooling rates** – stress conditions different during solidification → cracks; metastable phases.; porosity
- **Element loss** e.g. due to evaporations → might require over alloying
- **Different temperature profiles / time regimes** → enough time for sintering/melting? Alloying elements?

**Powders should be optimized (chemistry, design wise) & developed for a specific process to explore its real possibilities**

**At the end component shall meet the Material spec, not the powder**
Stellite 6 type powder (-106+45 µm)

Need for Process specific designing?

Laser Clad

Dilution < 5%

PTA

Dilution 10 - 15%

In order to achieve same quality the PTA powder would benefit from parameter optimization or alloy adjustment.
SLM study: C300 Maraging Steel
Influence of powder chemistry (element variation)

- Experimental powders
  - -53+10 µm
  - Elements at pre-defined level

- Process
  - Selective laser melting

- Coupon size: 20x10x10 mm³
  55x10x10 mm³

- SLM coupon properties
  - Density
  - Impact strength
  - Hardness

<table>
<thead>
<tr>
<th>Powder</th>
<th>Element varied</th>
</tr>
</thead>
<tbody>
<tr>
<td>XM1</td>
<td>P</td>
</tr>
<tr>
<td>XM2</td>
<td>Cr</td>
</tr>
<tr>
<td>XM3</td>
<td>B</td>
</tr>
<tr>
<td>XM4</td>
<td>Nb</td>
</tr>
<tr>
<td>XM5</td>
<td>Standard</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Parameters</th>
<th>Data</th>
</tr>
</thead>
<tbody>
<tr>
<td>Equipment</td>
<td>SLM</td>
</tr>
<tr>
<td>Power</td>
<td>195 W</td>
</tr>
<tr>
<td>Scan speed</td>
<td>1200 mm/sec</td>
</tr>
<tr>
<td>Powder layer thickness</td>
<td>0.02 mm</td>
</tr>
</tbody>
</table>
# SLM study: C300 Maraging Steel

## Powder - Characteristics

<table>
<thead>
<tr>
<th>Powder</th>
<th>Apparent Density g/cc</th>
<th>Hall Flow s/50 g</th>
<th>Size (-53+10µm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>XM1 (P)</td>
<td>4.02</td>
<td>22</td>
<td>A</td>
</tr>
<tr>
<td>XM2 (Cr)</td>
<td>4.03</td>
<td>20</td>
<td>A</td>
</tr>
<tr>
<td>XM3 (B)</td>
<td>4.15</td>
<td>26</td>
<td>A</td>
</tr>
<tr>
<td>XM4 (Nb)</td>
<td>4.01</td>
<td>26</td>
<td>A</td>
</tr>
<tr>
<td>XM5 (-)</td>
<td>4.22</td>
<td>15</td>
<td>B</td>
</tr>
</tbody>
</table>

## Morphology

- **CROSS-SECTION**
- **POWDER SIZE DISTRIBUTION EFFECTS POWDER FLOW**
SLM study: C300 Maraging Steel  
Chemistry influence on Density Measurement*

* METHODOLOGY: Archimedes principle (Qualitest Electronic Densimeter MD-300S)

<table>
<thead>
<tr>
<th>#</th>
<th>XM1 (P)</th>
<th>XM2 (Cr)</th>
<th>XM3 (B)</th>
<th>XM4 (Nb)</th>
<th>XM5 (-)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>8.037</td>
<td>8.054</td>
<td>8.049</td>
<td>8.059</td>
<td>8.031</td>
</tr>
<tr>
<td>2</td>
<td>8.041</td>
<td>8.060</td>
<td>8.038</td>
<td>8.032</td>
<td>7.952</td>
</tr>
<tr>
<td>3</td>
<td>7.993</td>
<td>8.027</td>
<td>8.060</td>
<td>8.026</td>
<td>8.060</td>
</tr>
<tr>
<td>4</td>
<td>8.033</td>
<td>8.059</td>
<td>8.013</td>
<td>8.045</td>
<td>8.045</td>
</tr>
<tr>
<td>5</td>
<td>8.001</td>
<td>8.051</td>
<td>8.040</td>
<td>8.034</td>
<td>8.042</td>
</tr>
<tr>
<td>Average (gm/cc)</td>
<td>8.021</td>
<td>8.050</td>
<td>8.040</td>
<td>8.039</td>
<td>8.026</td>
</tr>
</tbody>
</table>

* Conventional C300, \( \rho = 8.1 \text{ g/cc} \)

RELATIVE DENSITY VARIED FROM 99% TO 99.5%
SLM study: C300 Maraging Steel
Chemistry influence on - Impact Strength (lb-ft)

STANDARD: ASTM E23 (Charpy Impact testing)

<table>
<thead>
<tr>
<th>Sample Set</th>
<th>XM1 (P)</th>
<th>XM2 (Cr)</th>
<th>XM3 (B)</th>
<th>XM4 (Nb)</th>
<th>XM5 (Baseline)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Specimen #1</td>
<td>12</td>
<td>35</td>
<td>11</td>
<td>42</td>
<td>27</td>
</tr>
<tr>
<td>Specimen #2</td>
<td>11</td>
<td>54</td>
<td>14</td>
<td>41</td>
<td>47</td>
</tr>
<tr>
<td>Specimen #3</td>
<td>11</td>
<td>34</td>
<td>12</td>
<td>47</td>
<td>35</td>
</tr>
<tr>
<td>Average (ft-lb)</td>
<td>11.33</td>
<td>41</td>
<td>12.33</td>
<td>43.33</td>
<td>36.33</td>
</tr>
</tbody>
</table>

Variation of Impact strength

* Conventional C300 Steel (Annealed and aged) : ~20 ft-lb

Phosphorous & Boron reduce impact strength
SLM study: C300 Maraging Steel
Comparison of XM1 (P) and XM4 (Nb)

@ 50x magnification

Micropores

Microcracks

Reduction in impact strength is coupled with Microcracks
Etched – XM1 (P) and XM4 (Nb)

Microcracks

Reduction in impact strength is coupled with Microcracks

Etchant = Nital 10%

@ 50x magnification
SLM study: C300 Maraging Steel
Chemistry influence on Hardness

Variation in Hardness (Micro/Macro)

Micro Hardness (Vickers)

Macro Hardness (HRC)

C300 Steel (Annealed and aged)
54 HRC/ 577 HV

Minimal Anisotropy observed and only minimal difference in Hardness
SLM study: C300 Maraging steel
Chemistry influence on Phase composition (XRD)

- Identical in all cases
- Diffraction planes identical to Body centered crystal structure

<table>
<thead>
<tr>
<th>Phases (Parallel to build direction)</th>
<th>%Phase</th>
<th>Phases (Perpendicular to build direction)</th>
<th>%Phase</th>
</tr>
</thead>
<tbody>
<tr>
<td>XM1 (P)</td>
<td>91.2</td>
<td>Fe$<em>{0.8}$ Ni$</em>{0.2}$</td>
<td>100</td>
</tr>
<tr>
<td>Fe$<em>{0.66}$Ni$</em>{0.34}$</td>
<td>8.8</td>
<td></td>
<td></td>
</tr>
<tr>
<td>XM5 (-)</td>
<td>94.1</td>
<td>Fe$<em>{0.8}$ Ni$</em>{0.2}$</td>
<td>100</td>
</tr>
<tr>
<td>Fe$<em>{0.66}$Ni$</em>{0.34}$</td>
<td>5.9</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

No or only minimal difference in Phase composition was found
POST BUILT AGING TREATMENT

Temperature = 480 °C
Time = 5 hrs
Atmosphere = Argon

From Literature
### SLM study: C300 Maraging Steel

#### Density after aging

<table>
<thead>
<tr>
<th>#</th>
<th>XM2-H</th>
<th>XM4-H</th>
<th>XM5-H</th>
</tr>
</thead>
<tbody>
<tr>
<td>Avg(gm/cc)</td>
<td>8.02</td>
<td>8.02</td>
<td>8.04</td>
</tr>
</tbody>
</table>

- No variation in density relative
- Relative density remained same as before

**Diagram:**

- XM2-H (Cr)
- XM4-H (Nb)
- XM5-H (-)
SLM study: C300 Maraging Steel
Impact Strength before and after Aging

Change in Impact Strength

<table>
<thead>
<tr>
<th>Sample</th>
<th>Energy (ft-lb)</th>
</tr>
</thead>
<tbody>
<tr>
<td>XM2-H</td>
<td>32.58</td>
</tr>
<tr>
<td>XM4-H</td>
<td>34.35</td>
</tr>
<tr>
<td>XM5-H</td>
<td>40.33</td>
</tr>
</tbody>
</table>

*Wrought C300 Steel (Annealed and aged): ~20 ft-lb

Increase in impact strength for powder with standard alloy composition
SLM study: C300 Maraging Steel
Micro-hardness variation after aging

Effect of heat treatment

DROP IN MICROHARDNESS AFTER AGING……..UNEXPECTED!!!
SLM study: C300 Maraging Steel
Microstructure after Aging (Etched)

XM5

XM4

SLM Coupon

(Nb)

GRAIN COARSENING
SLM study: C300 Maraging Steel
XRD PLOTS for Both Built Direction

Top Surface

(110)  (200)  (211)  (220)

Intensity (A.U.)

2-Theta

Side Surface

(110)  (200)  (211)  (220)

Intensity (A.U.)

2-Theta

Identical phases on both directions after aging
SLM study: C300 Maraging Steel
EDS and Elemental Mapping of XM4

Fe  Ni  Mo

Nb

Mass percent (%)

<table>
<thead>
<tr>
<th>Spectrum</th>
<th>Ti</th>
<th>Cr</th>
<th>Fe</th>
<th>Co</th>
<th>Ni</th>
<th>Nb</th>
<th>Mo</th>
</tr>
</thead>
<tbody>
<tr>
<td>Area 2667</td>
<td>0.79</td>
<td>0.27</td>
<td>56.39</td>
<td>10.83</td>
<td>17.84</td>
<td>0.37</td>
<td>4.61</td>
</tr>
<tr>
<td>Area 2668</td>
<td>0.88</td>
<td>0.34</td>
<td>57.61</td>
<td>10.32</td>
<td>17.06</td>
<td>0.22</td>
<td>4.77</td>
</tr>
<tr>
<td>Area 2669</td>
<td>0.84</td>
<td>0.34</td>
<td>56.98</td>
<td>10.70</td>
<td>16.95</td>
<td>0.41</td>
<td>4.68</td>
</tr>
</tbody>
</table>

 Element segregation was not observed in any sample
Summary SLM study: C300 Maraging Steel

As build
- **Toughness** of as built and aged coupon obtained by SLM is **well above** C300 wrought alloy
- **Hardness** of C300 coupons obtained by SLM is **well below** that of wrought alloy - may impact wear resistance
- **Reduction in impact strength** of B and P containing alloys is attributed to **micro cracks**

After heat treatment
- **Increase** in toughness for sample with **standard chemistry** after aging
- **Reduction** in toughness of **Cr and Nb** containing alloys by \(~20\%\) after ageing
  - Can be attributed to inclusions or grain/precipitate coarsening
- **Drop in micro-hardness** by \(~15\%\) after aging for all the candidates
  - May be due to **over aging** or **improper heat treatment** OR
  - Due to **insufficient alloying** required for the process

Future work
- Powder **chemistry to be modified to obtain the required hardness**
- **Modify ageing** treatment
- Properties to be evaluated: UTS / YS / Elongation / Modulus of elasticity / Dimensional Stability
Laser Cladding Study: Inconel 625 type Powder

Influence of powder chemistry (element variation)

- Experimental powders
  - -106+45 µm
  - Elements at pre-defined level, varied from Standard IN 625

- Evaluated properties
  - Density
  - Dilution
  - Hardness

<table>
<thead>
<tr>
<th>Powder</th>
<th>Element varied</th>
</tr>
</thead>
<tbody>
<tr>
<td>SM1</td>
<td>Si</td>
</tr>
<tr>
<td>SM2</td>
<td>Mn</td>
</tr>
<tr>
<td>SM3</td>
<td>Iron</td>
</tr>
<tr>
<td>SM4</td>
<td>Nitrogen</td>
</tr>
<tr>
<td>SM5</td>
<td>Baseline</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Parameters</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Laser source</td>
<td>ND-YAG</td>
</tr>
<tr>
<td>Substrate</td>
<td>Stainless Steel</td>
</tr>
<tr>
<td>Laser spot size</td>
<td>3.5 mm</td>
</tr>
<tr>
<td>Feed rate</td>
<td>7 to 9 gm/min</td>
</tr>
<tr>
<td>Preheating</td>
<td>None</td>
</tr>
</tbody>
</table>

Gas atomized; SM Troy

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Laser cladding study: Inconel 625 type powder

Resulting Laser clad structure

SM1 (Si)
SM2 (Mn)
SM3 (Fe)
SM4 (N)

Baseline

Powders with Fe and N variations from IN625-Baseline led to less porosity.

But, at the same time the N-variation showed insufficient clad bond (means this powder requires more heat input)
Laser Cladding Study: Inconel 625 Type Powder
Effect on Hardness & Dilution

Effect of element variation on Macro (Surface) Hardness
[SM5 = Baseline; Standard IN 625]

Effect of element variation on Dilution
Note: SM4 = insufficient clad; more heat required
**MetcoAdd™ 718 A/B**  
Ni-Based Superalloy for Powder Bed Additive Manufacturing

**Chemical composition:**

<table>
<thead>
<tr>
<th>Product</th>
<th>Weight Percent (nominal)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Ni</td>
</tr>
<tr>
<td>MetcoAdd™ 718A/B</td>
<td>Balance</td>
</tr>
</tbody>
</table>

**Particle size distribution:**

<table>
<thead>
<tr>
<th>Product</th>
<th>Nominal Range μm</th>
<th>D90 μm</th>
<th>D50 μm</th>
<th>D10 μm</th>
<th>Hall Flow s/50 g</th>
</tr>
</thead>
<tbody>
<tr>
<td>MetcoAdd™ 718A</td>
<td>–45 +15</td>
<td>45.95</td>
<td>29.91</td>
<td>17.91</td>
<td>&lt; 18</td>
</tr>
<tr>
<td>MetcoAdd™ 718B</td>
<td>–53 +20</td>
<td>51.8</td>
<td>37.2</td>
<td>27.5</td>
<td>&lt; 16</td>
</tr>
</tbody>
</table>

For the nominal range, particle size analysis 45 μm or above measured by sieve (ASTM B214), analysis below 45 μm by laser diffraction (ASTM C 1070, Microtrac). Fractional analysis (D90, D50, D10) by laser diffraction.

Aero Engine bore-scope boss made from MetcoAdd™ 718 using Laser Additive manufacturing.  
Image: Courtesy of MTU Aero Engines, Munich

Typical cross-section of MetcoAdd™ 718A gas-atomized powder showing its very dense inner structure.
Conclusion

- Materials for AM are often based on specification from cast, forged, heat-treated materials or other processes.
- The performance of an AM part is strongly dependent on the interaction of material, equipment & parameters and post treatment.
- Development of AM optimized materials is only possible if the whole production chain is considered.
Thank you.