Recycling and Re-Use of Materials

A COMPREHENSIVE STUDY OF IN625 POWDER REUSE

AND THE EFFECT ON ADDITIVE TENSILE PROPERTIES PRODUCED ACROSS SEVEN EOM M280 MACHINES OVER THE COURSE OF EIGHT CONSECUTIVE MONTHS.

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SENIOR PROCESS AND MANUFACTURING ENGINEER
Stratasys Direct Manufacturing is one of the largest providers of additive and conventional manufacturing solutions:

- 7 U.S. manufacturing facilities
- 12 manufacturing technologies
- 600+ employees
- Certifications: ISO 9001, AS9100
- ITAR registered
Additive Metals Production Facilities

Austin, & Belton, TX - Similar Capacity, Redundant Capability

Austin, TX

Belton, TX
Industry claims powder degrades with time and exposure. Companies are forming around the fear. Customer’s specs call for no part to be built with any powder older than 90 days.

The mechanical and material properties trended against Blend Composition will answer industry’s question of powder degradation.
What We Know

Powder Composition is in a constant state of flux.

- SDM Recycles 100% material
- The majority of powder has been recycled.
- No test method is identified to qualify recycled powder
What we’ve done

Implemented a digital system providing a resolution in powder composition unmatched by industry.

Workbench - BuildIt / Powder Report

Digital means to log and mine powder composition
Additive IN625 Property Study

**Study:** 7 EOS M280 Machines, 2 Material Providers, 8 Material Lots, >55 Builds per Data represents 385 production builds over an 8 month period.

**Testing Included:** 210 tensile bars (tests performed approx. every 4 builds), 14 Chemical, 20 Metallography

**Comparisons:**
- ✔ Build Location
- ✔ Machine to Machine
- ✔ Powder Composition
- ✔ Material Provider
Additive IN625 Property Study

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Comparisons:
- Build Location
- Machine to Machine
- Powder Composition
- Material Provider
Build Configuration

3 sacrificial cylindrical blanks, >385 builds, 7 machines
Processing – High Level

- Tensile Blanks were process alongside component
- Coupons represent additive IN625 in the stress relieved, HIP’ed, and SHT condition.
- All tests were performed at a Nadcap & A2LA accredited test house.

- **Powder Sieving**
  - (75µm)

- **Additive Manufacturing**
  - (<385 Builds)

- **Stress Relief**
  - 1925F for 120min
  - (27 separate cycles)

- **Plate Part Separation**
  - Saw

- **Machining**
  - (Nadcap facility - Per ASTM E8 cylindrical bars)

- **Testing**
  - (Nadcap facility - Per ASTM E8 cylindrical bars)

- **HIP**
  - (2200F @ 14.5ksi for 4hrs)
  - 19 separate cycles

- **Solution Heat Treatment**
  - (2175F for 60min)
  - 18 separate cycles
Powder Recycling and Re-Use

SI1476 - Vertical Tensile Properties

- UTS (ksi)
- Yield (ksi)
- Elongation (%)
- RA (%)
- % Virgin
- % Original Blend
Tensile Properties Overlaid onto Powder Composition

SI1476 - Vertical Tensile Properties

- UTS (ksi)
- Elongation (%)
- RA (%)
- % Virgin
- % Original Blend
Introduction of Separate Powder Lots

SI1476 - Vertical Tensile Properties
Microstructure Over Time

SI1849 - Vertical Tensile Properties

- **UTS (ksi)**
- **Yield (ksi)**
- **Elongation (%)**
- **RA (%)**
- **% Virgin**
- **% Original Blend**

**A**

**B**

**C**
Microstructure Over Time

A (xy) 8e-3in

B (xy) 8e-3in

C (xy) 8e-3in

Age of Oldest Powder (days)

UTS (ksi) Elongation (% Virg % Original Blend

Strength (ksi)

% Virgin

0 50 100 150 200 250 300

0% 10% 20% 30% 40% 50% 60% 70% 80% 90% 100%
Microstructure – Different Orientations

A (xy) 8e-3in
B (xy) 8e-3in
C (xy) 8e-3in

A (xz) 8e-3in
B (xz) 8e-3in
C (xz) 8e-3in
Material Chemistry Over Time

SI1849 - Vertical Tensile Properties

- UTS (ksi)
- Yield (ksi)
- Elongation (%)
- RA (%)
- % Virgin
- % Original Blend

Strength (ksi) vs. Age of Oldest Powder (days) vs. Times Recycled

- UTS (ksi)
- Yield (ksi)
- Elongation (%)
- RA (%)
- % Virgin
- % Original Blend
Material Chemistry Over Time

SI1849 & SI1991 - Chemistry

Times Recycled vs. Powder Age (days) for Cr, Mo, Fe, Nb+Ta, and Al.

- Cr: Weight (%)
- Mo: Weight (%)
- Fe: Weight (%)
- Nb+Ta: Weight (%)
- Al: Weight (%)

Graph shows the weight percentage of each element over time for each recycling cycle (0 to 300 days).

Legend:
- SI1991 Sup. 1
- SI1849 Sup 1
- Limit

Graphs indicate stability in weight percentage for these elements over multiple recycling cycles.
Material Provider Comparison

Yield Strength Material Provider Comparison, Yts

- **Provider 1 YTS**, Avg = 53.3ksi, $\sigma = 0.769$ksi
- **Provider 2 YTS**, Avg = 53.0ksi, $\sigma = 0.756$ksi

Ultimate Strength Material Provider Comparison, Uts

- **Provider 1 UTS**, Avg = 124.0ksi, $\sigma = 0.731$ksi
- **Provider 2 UTS**, Avg = 127.2ksi, $\sigma = 0.577$ksi

Material Provider 1, n = 180
Material Provider 2, n = 30
Material Provider Comparison

Material Provider - Mechanical Property Comparison

- UTS: Supplier 1 - 0.61%, Supplier 2 - 0.59%
- Yield: Supplier 1 - 1.37%, Supplier 2 - 1.09%
- Modulus: Supplier 1 - 11.41%, Supplier 2 - 4.72%
- Elongation: Supplier 1 - 6.47%, Supplier 2 - 1.71%
- RA: Supplier 1 - 5.51%, Supplier 2 - 4.73%

Sample sizes:
- UTS: n = 180, Yield: n = 30

Percentiles for tensile properties:
- Strength (ksi, Msi), Percent
- Tensile Property, Percent
Machine to Machine Consistency

Material Strength vs. Times Recycled, 1 machine
Machine to Machine Consistency

Material Strength vs. Times Recycled, 2 machines
Machine to Machine Consistency

Material Strength vs. Times Recycled, 3 machines

Strength (ksi) vs. Times Recycled for 3 machines.
Machine to Machine Consistency

Material Strength vs. Times Recycled, 4 machines
Machine to Machine Consistency

Material Strength vs. Times Recycled, 5 machines

- Chart shows the relationship between strength (in ksi) and times recycled.
- Data points indicate consistency across multiple machines.
- Machine to machine consistency is evident from the close grouping of data points.
Machine to Machine Consistency

Material Strength vs. Times Recycled, 6 machines
Machine to Machine Consistency

Material Strength vs. Times Recycled, 6 machines, 2 atomizers
Material Provider Comparison

The different chemistries produced by different suppliers produced statistically different ultimate tensile strength.

<table>
<thead>
<tr>
<th>Material Chemistry</th>
<th>Supplier Provider Comparison</th>
</tr>
</thead>
<tbody>
<tr>
<td>Supplier 1 (% by Weight)</td>
<td>Supplier 2 (% by Weight)</td>
</tr>
<tr>
<td>Al</td>
<td>0.14</td>
</tr>
<tr>
<td>Cr</td>
<td>21.54</td>
</tr>
<tr>
<td>Mo</td>
<td>9.1</td>
</tr>
<tr>
<td>Nb+Ta</td>
<td>3.73</td>
</tr>
<tr>
<td>Fe</td>
<td>3.90</td>
</tr>
</tbody>
</table>
Material Provider Comparison

IN625 Chemistry

- Cr
- Mo
- Fe
- Nb+Ta
- Al

Graph showing the weight (%) of IN625 chemistry over powder age (days) for different material providers.
Material Provider Comparison

IN625 Chemistry

- **Cr**: Comparison of Cr content over Powder Age (days) for different material providers and limits.
- **Mo**: Comparison of Mo content over Powder Age (days) for different material providers and limits.
- **Fe**: Comparison of Fe content over Powder Age (days) for different material providers and limits.
- **Nb+Ta**: Comparison of Nb+Ta content over Powder Age (days) for different material providers and limits.
- **Al**: Comparison of Al content over Powder Age (days) for different material providers and limits.

The graphs show the weight percentage of each element over the Powder Age in days, with different material providers indicated by various markers and colors.
Conclusion

Stratasys Direct Manufacturing’s mission to control, track, and develop the AM production process has lead to **vertical integration of multiple post process operations and inspection techniques** providing the industry with:

- An efficient and economic powder management process
- Increased confidence in DMLS material/part quality
- Increased customer confidence
- Insight into powder lifecycle
- Evidence process variation at the additive system level is reduced via certified heat treatments
THANK YOU

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Out of the 210 Bars, 3 Appeared “Different”

SI1989 - Vertical Tensile Properties

<table>
<thead>
<tr>
<th>Build</th>
<th>Build</th>
<th>Times Recycled</th>
<th>Times Recycled</th>
</tr>
</thead>
<tbody>
<tr>
<td>50814</td>
<td>51334</td>
<td>0</td>
<td>30</td>
</tr>
<tr>
<td></td>
<td></td>
<td>10</td>
<td>40</td>
</tr>
<tr>
<td></td>
<td></td>
<td>20</td>
<td>50</td>
</tr>
<tr>
<td></td>
<td></td>
<td>30</td>
<td>60</td>
</tr>
<tr>
<td></td>
<td></td>
<td>40</td>
<td>70</td>
</tr>
</tbody>
</table>

- **UTS (ksi)**
- **Elongation (%)**
- **% Virgin**
- **% Original Blend**

**Strength (ksi)**

**Age of Oldest Powder (days)**

**Yield (ksi)**

**Elongation (%)**

**% Virgin**

**% Original Blend**
Tensile Results, Build 50814

![Graph showing tensile results for Build 50814 with stress and strain axes labeled.]
Tensile Results, Build 51334 & 54747
Metallographic Comparison, Builds 51334 & 54747

Stress Relieved, Additive IN625 (100x)

Solution Heat Treated, HIP’ed, Stress Relieved, Additive IN625 (100x)
What We’re Doing
Ongoing Additive Alloy Consistency Studies

✓ **IN625** (Status: Complete)
  7 EOS M280 Machines, 2 Material Providers, 8 Material Lots,
  >55 Builds per Machine, Data represents 385 production builds over an 8 month period.

☐ **CoCr** (Status: Sampling Coupons)
  8 EOS M280 Machines, 2 Material Providers, 12 Material Lots,
  Averaged 46 Builds per Machine, Data represents 369 production builds over an 10 month period.

☐ **IN718** (Status: Executing Build)
  3 Different Machines (M280, M290, M400). 2 Material Providers, 3 Virgin Powder Builds,
  6 Highly Recycled Powder Blends, Results will be compared against API Standard 6A718

☐ **AlSi10Mg** (Status: Building and Collecting Bars)
  4 Different M290’s, Multiple Orientations, 3 Months of “Random” Production
BUILD LOCATION COMPARISON

Build Plate Location - Tensile Property Comparison

- UTS (ksi)
- Yield (ksi)
- Modulus (Msi)
- Elongation (%)
- Reduction of Area (%)

BL: 1.05%, 1.13%, 1.10%
FL: 1.29%, 1.17%, 1.42%
FR: 1.29%, 1.17%, 1.42%

n = 66, 74, 70

Strength (ksi, Msi)
MACHINE TO MACHINE COMPARISON

Machine Comparison - Tensile Property Comparison

- UTS
- Yield
- Mod
- Elogation
- RA

Strength (ksi, Msi)

Percent

0.48%  0.60%  0.54%  0.48%  0.26%

0.48%

0.60%

0.54%

0.48%

0.26%

0.83%  0.80%  0.76%  0.21%  0.12%

0.87%

0.80%

0.76%

0.21%

0.12%

5.89%  6.76%  6.34%  5.30%  7.97%

6.79%

6.34%

5.30%

7.97%

3.35%  3.35%  2.82%  2.01%  1.12%

3.35%

2.82%

2.01%

1.12%

1.35%  2.86%  2.35%  2.82%  1.72%

2.86%

2.35%

2.82%

1.72%

3.16%  3.78%  4.26%  3.85%  4.62%

3.78%

4.26%

3.85%

4.62%

n= 30, 29, 20, 30, 30, 27, 14
Stratasys Direct & SAE AMS Additive Manufacturing (AMSAM)

4 Sponsored Specifications – in development:


AMS 7001  Nickel Alloy, Corrosion and Heat-Resistant, Powder for Additive Manufacturing, 62Ni – 21.3Cr – 9.0Mo – 3.65Nb

AMS 7002  Powder Manufacturing

AMS 7003  Laser Powder Bed Fusion Process

SAE contacted Stratasys Direct and requested the submission of the data to establish industry spec. minimum tensile properties for IN625.