Title:

M2 Selective Laser Melting GRCop-42 Recycling Study

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Executive Summary: (Purpose and Result)

A powder recycling study was conducted to examine the effects of powder reuse on physical and mechanical properties of SLM GRCop-42. Twelve sequential builds were conducted, each reusing powder from the previous build topped off with virgin powder to fill the powder chamber. Powder morphology, particle size distribution, flowability, and chemistry were examined for each subsequent build, along with the mechanical properties of tensile and LCF specimens built with each powder batch. It was found that only powder flowability demonstrated a trend with increasing reuse, with flowability and cohesiveness generally decreasing over the course of the 12 builds. Minor variation in powder chemistry was observed throughout the study, however, the variations did not correlate with increasing powder reuse.

References: (work orders, reports, etc.)

2021-0301, 2021-0302, 2021-0303, 2021-0304, 2021-0305, 2021-0306, 2021-0307, 2021-0308, 2021-0309, 2021-0310, 2021-0547, 2021-0548, 2022-0175

Background

- Powder reuse in metal additive manufacturing reduces waste and cost. In many materials, degradation of physical and mechanical properties occurs with excessive reuse of powder. The purpose of this study is to evaluate the effect of powder reuse on the properties of SLM GRCop-42 to determine if/when detrimental changes to the powder occur.
- Study considers effect of powder reuse on morphology, particle size distribution, flowability, chemistry, tensile properties, and low cycle fatigue life, specifically to determine if a decrease in mechanical properties occurs with increasing powder reuse, and if so, what physical properties of the powder may correlate to a change in mechanical properties.

Virgin Powder Content in Reuse Builds

- Virgin powder was mixed with used powder in the recycle builds to obtain the desired baseline powder mass for building the parts.
- For example, build 4 consisted of 62.05% powder originally added in build 1, 10.12% powder originally added in build 2, 12.85% powder originally added in build 3, and 14.99% virgin powder.
- From build 5 to build 12, 45-50% of the powder in the builds was either virgin powder or powder that had been reused three times or less.

Powder composition in build (%)	Residual powder content in subsequent build												
	Build 1	Build 2	Build 3	Build 4	Build 5	Build 6	Build 7	Build 8	Build 9	Build 10	Build 11	Build 12	SUM
Build 1	100												100
Build 2	85.98	14.02											100
Build 3	72.99	11.90	15.11										100
Build 4	62.05	10.12	12.85	14.99									100
Build 5	52.62	8.58	10.89	12.71	15.20								100
Build 6	44.62	7.28	9.24	10.78	12.89	15.19							100
Build 7	37.73	6.15	7.81	9.12	10.90	12.84	15.45						100
Build 8	31.98	5.22	6.62	7.73	9.24	10.89	13.10	15.23					100
Build 9	27.22	4.44	5.64	6.58	7.86	9.27	11.15	12.96	14.88				100
Build 10	23.02	3.75	4.77	5.56	6.65	7.84	9.43	10.96	12.58	15.44			100
Build 11	19.50	3.18	4.04	4.71	5.63	6.64	7.99	9.29	10.66	13.08	15.28		100
Build 12	16.65	2.71	3.45	4.02	4.81	5.67	6.82	7.93	9.10	11.17	13.04	14.63	100

Percentage of virgin powder added for a given build

Percentage of powder reused three times or fewer

Morphology

- A Malvern Morphologi G3SE analyzer was used to determine the size and shape of powder particles via optical image analysis.
- Particle size parameters include CE (circular equivalent) diameter, length, width, height, and sphere equivalent volume.
- Shape parameters include circularity, convexity, elongation, and aspect ratio.
- Analysis was performed with a 10x magnification objective lens on a 3 mm^3 sample volume.
- Powder morphology did not exhibit a trend or significant change over the course of 12 build cycles.

Build Number	Circularity Mean	Aspect Ratio Mean	Elongation Mean	CE Diameter Mean (µm)
Baseline	0.97	0.88	0.12	8.44
1	0.97	0.89	0.11	8.96
2	0.97	0.89	0.11	8.02
3	0.97	0.89	0.11	7.95
4	0.97	0.89	0.11	7.62
5	0.97	0.88	0.12	7.6
6	0.97	0.89	0.11	9.07
7	0.97	0.89	0.11	7.83
8	0.97	0.88	0.12	7.89
9	0.97	0.87	0.13	7.81
10	0.97	0.89	0.12	8.24
11	0.97	0.88	0.12	8.23
12	0.96	0.87	0.13	8.03

Convex Hull Perimeter
The convex hull of a particle can be thought of as the particle surrounded by a rubber band. It is sometimes also referred to as a convex envelope or simply as an envelope. Figure 8 illustrates the difference between a perimeter, P (left) and a convex hull perimeter, P_C (right). The units of the convex hull perimeter are most often expressed in µm. Note the equivalent shapes are blue, while the respective perimeters are indicated by the black lines.

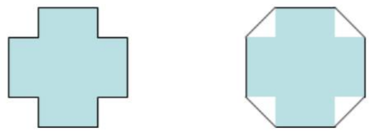


Figure 8. Perimeter (left) and convex hull perimeter (right) of an equivalent shape

Length and Width
The length is the maximum distance between any two points on the perimeter of the particle parallel to the major axis. Likewise, the width is the maximum distance between any two points on the perimeter of the particle parallel to the minor axis. The units for length and width are most often expressed in µm. The length and width are illustrated in Figure 7.

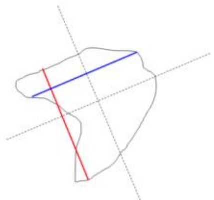
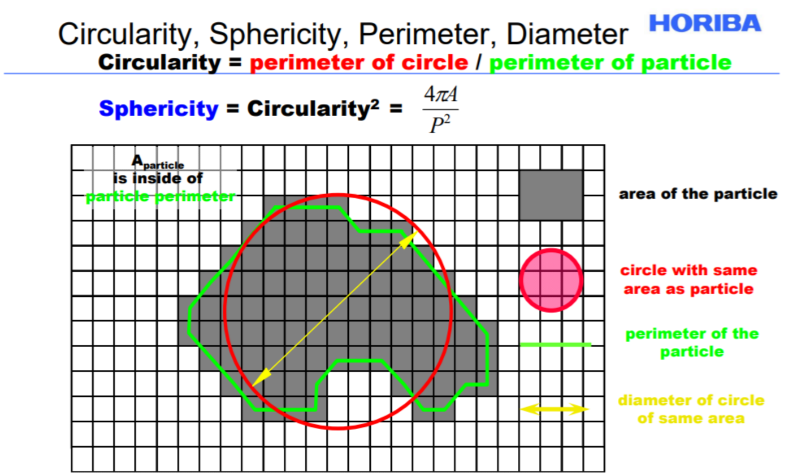


Figure 7. A particle indicating the major and minor axes (dashed lines), as well as the length (red) and width (blue)

Images copied from: Particle Technology Labs Particle Shape Factors and Their Use in Image Analysis Part 1

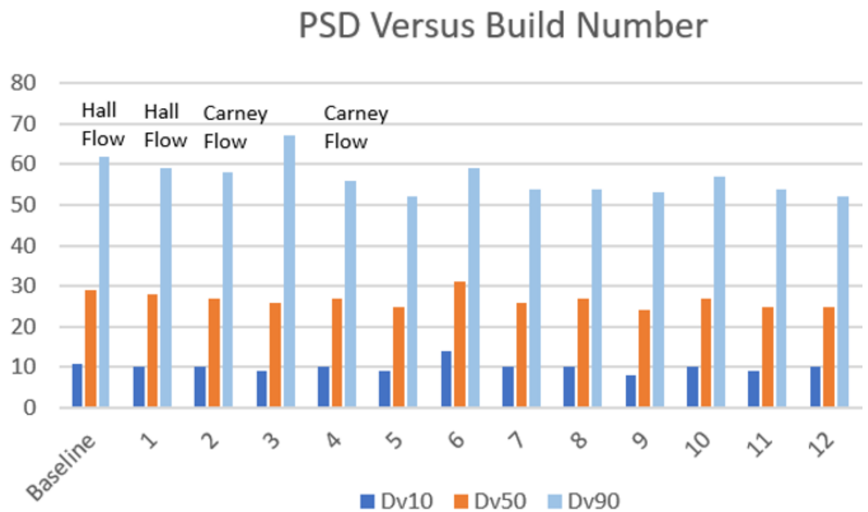


Circular Equivalent Diameter = diameter of a circle with the same area as the particle (red circle in figure above)

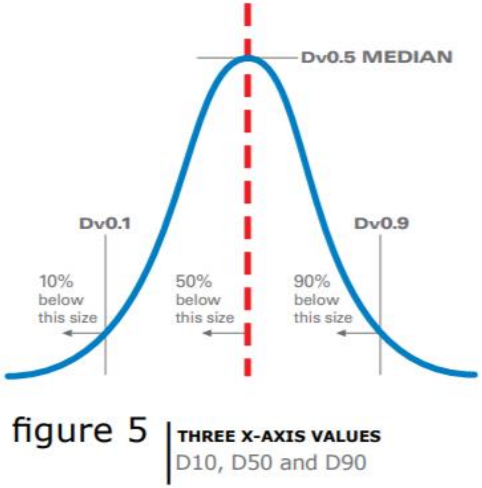
Particle Size Distribution

- Particle Size Distribution was measured using a Mastersizer 3000, which transmits red laser light and blue light through a sample dispersed in a liquid. The instrument analyzes the light scattering pattern of the particles to determine the size distribution.
- Five 30-second scans were performed on two samples for each powder. Samples were dispersed in 600 ml of distilled water.
- No trend was observed in the particle size distribution over the course of 12 build cycles.

Build Number	Dv10 (µm)	Dv50 (µm)	Dv90 (µm)
Baseline	10.7	28.8	61.8
1	10.2	28.2	59.2
2	9.8	27.1	58.1
3	8.9	26	66.9
4	10.1	26.8	55.6
5	9.5	25.2	52
6	13.8	31.2	59.1
7	9.8	26.4	54
8	10	26.6	54.1
9	8.4	24.2	52.6
10	9.8	27.3	56.9
11	9.4	24.8	54.3
12	10	25.2	52.2



Dv10 = 10% of the population lie below this value.
Dv50 = median = 50% of the population lie above this value and 50% of the population lie below this value.
Dv90 = 90% of the population lie below this value.



Copied from HORIBA Scientific, A Guidebook to Particle Size Analysis

Flowability

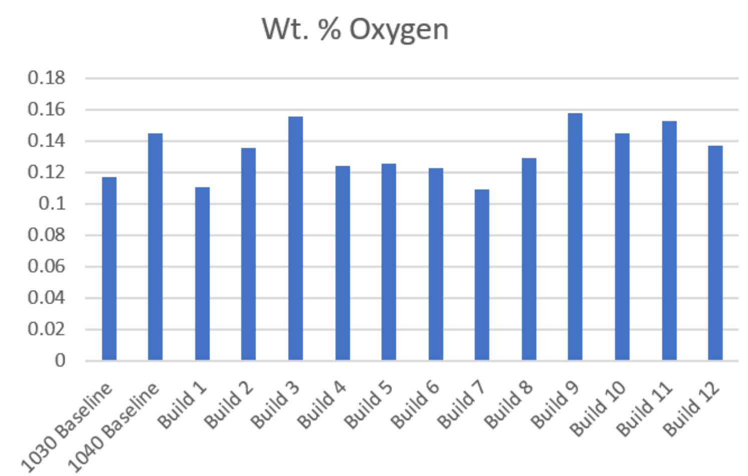
- Powder Flowability was measured using a Revolution Powder Analyzer and Hall and Carney Funnel Tests.
- The Revolution Powder Analyzer rotates a drum containing powder while imaging the response of the powder with a digital camera to determine parameters including solid/liquid ratio, avalanche energy, break energy, avalanche angle, surface fractal, and rest angle.
- The Hall Funnel measures the time for a 50 gram sample of powder to flow through a cup with a 2.54 mm orifice, while the Carney Funnel measures the time for 150 grams of powder to flow through a 5.08 mm orifice. A powder that flows through the Hall funnel is considered to be "free flowing".
- Avalanche energy, avalanche angle, and break energy generally trended upward with increasing build number, indicating decreasing flowability.
- Rest angle generally trended downward with increasing build number, indicating decreasing cohesiveness.
- S/L Ratio, Avalanche Energy, Break Energy, and Avalanche Angle were lowest for the baseline and builds 1, 2, and 4, correlating with better flowability in Hall and Carney Funnel tests.
- Powder samples with an avalanche energy less than 10.1 kJ/kg flowed through the Hall funnel. Powder samples with avalanche energy between 10.1 and 15 kJ/kg flowed through the Carney funnel.

Build Number	S/L Ratio	Avalanche Energy (kJ/kg)	Break Energy (kJ/kg)	Avalanche Angle (degrees)	Surface Fractal	Rest Angle (degrees)	Funnel Flow Time
Baseline	0.24	9	36.7	38	3.2	34.7	21.4s/50g (Hall)
1	0.24	10.1	41	39.8	4.9	36.3	22.2s/50g (Hall)
2	0.32	15	45.9	41.8	3.8	36.2	15.5s/150g (Carney)
3	0.36	17.2	46.5	42.5	2.7	35.8	No Flow
4	0.32	14.9	44.8	41.3	5	36	15.0s/150g (Carney)
5	0.42	21.5	49.6	43.6	3.1	35.6	No Flow
6	0.44	20.6	46.1	42.8	2.2	34.8	No Flow
7	0.37	16.4	43.3	42.1	2.2	35.4	No Flow
8	0.36	16.8	45.4	42	3.1	35.6	No Flow
9	0.48	26	53.2	44.9	3.1	35.1	No Flow
10	0.46	24	51	44.3	2.6	35.3	No Flow
11	0.44	21.8	48.4	43.1	2.9	34.7	No Flow
12	0.42	20.2	46.8	42.2	3.4	34.5	No Flow

Powder Chemistry

- Inductive Coupled Plasma (ICP) Spectroscopy was performed on GRCop-42 powder samples to determine the elemental composition of the powder using a Perkin Elmer AVIO 500 ICP device. Before performing ICP, samples were prepared by digesting in a nitric acid and hydroflouric acid solution.
- Oxygen analysis was performed using a LECO TCH600 Oxygen/Hydrogen/Nitrogen Analyzer following the calibration and analysis procedure developed by LECO.
- Fourteen samples were tested: two baseline control samples consisting of virgin powder, and 12 samples from subsequent builds with recycled material.
- No significant trend or change was observed in weight percentage of Chromium or Niobium over the course of the study.
- Copper weight percentage varied by up to 3% throughout the course of the study, with the lowest copper content observed for builds 6 and 8.
- Oxygen weight percentage varied by up to 0.041% throughout the course of the study.
- Despite the variation in copper and oxygen content from build to build, no consistent trend was observed in the powder chemistry throughout the study.

Sample	Cr	Nb	Fe	Al	Si	Cu	Cr:Nb Ratio
NASA-AMZ-1030 Baseline	3.08	2.66	<50 ppm	<50 ppm	<50 ppm	94.4	1.16
NASA-AMZ-1040 Baseline	3.20	2.80	<50 ppm	<50 ppm	<50 ppm	94.0	1.14
GRCOP Build 1	3.23	2.73	<50 ppm	<50 ppm	<50 ppm	94.4	1.18
GRCOP Build 2	3.22	2.72	<50 ppm	<50 ppm	<50 ppm	94.8	1.18
GRCOP Build 3	3.18	2.75	<50 ppm	<50 ppm	<50 ppm	94.4	1.16
GRCOP Build 4	3.11	2.71	<50 ppm	<50 ppm	<50 ppm	94.1	1.15
GRCOP Build 5	3.07	2.63	<50 ppm	<50 ppm	<50 ppm	94.2	1.17
GRCOP Build 6	3.20	2.70	<50 ppm	<50 ppm	<50 ppm	93.1	1.19
GRCOP Build 7	3.42	2.87	<50 ppm	<50 ppm	<50 ppm	93.8	1.19
GRCOP Build 8	3.14	2.66	<50 ppm	<50 ppm	<50 ppm	92.3	1.18
GRCOP Build 9	3.00	2.60	<50 ppm	<50 ppm	<50 ppm	94.0	1.15
GRCOP Build 10	3.27	2.69	<50 ppm	<50 ppm	<50 ppm	94.9	1.22
GRCOP Build 11	3.33	2.78	<50 ppm	<50 ppm	<50 ppm	94.8	1.20
GRCOP Build 12	3.21	2.63	<50 ppm	<50 ppm	<50 ppm	94.3	1.22
Fortified Blank % Recovery	100	100	NS*	NS*	NS*	102	

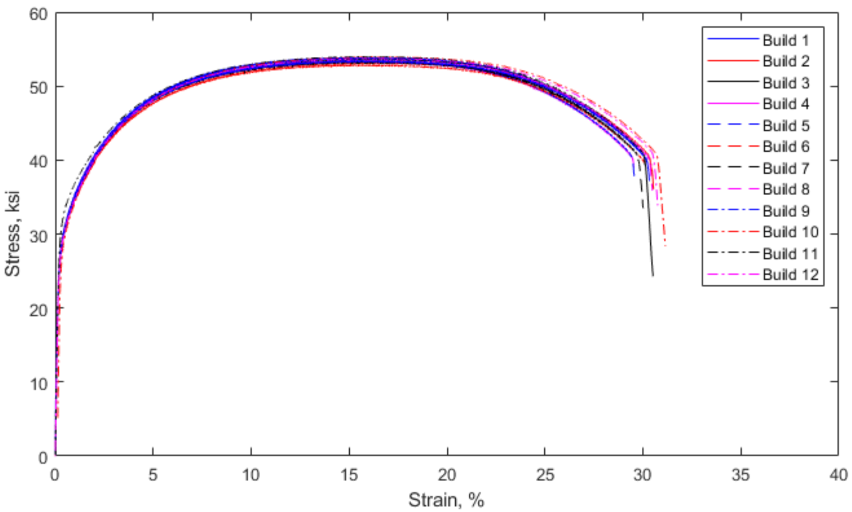


Tensile Properties

- Room temperature tensile tests were performed on machined test coupons in accordance with methods prescribed in ASTM E8. Four specimens were tested for each build number.
- No trend in tensile properties (ultimate tensile strength, yield strength, and fracture elongation) was observed, with properties remaining similar for each subsequent build.
- There does not appear to be a correlation between the tensile properties and the decreasing powder flowability trend.

Build Number	Ultimate Tensile Strength (ksi)	Yield Strength (ksi)	Elongation (%)
Baseline*	53.8	28.4	29.5
1	53.7	29.5	30.0
2	53.3	29.3	30.5
3	53.5	29.5	30.4
4	53.4	29.5	29.7
5	53.5	29.5	29.9
6	53.3	29.2	29.9
7	53.5	29.4	29.9
8	53.6	29.2	30.3
9	53.5	29.4	30.0
10	53.8	29.7	30.5
11	53.9	30.3	30.5
12	53.7	29.7	30.4

*Baseline tensile properties derived from M2 GRCop-42 Material Process Record for machined finish, Z-direction specimens.

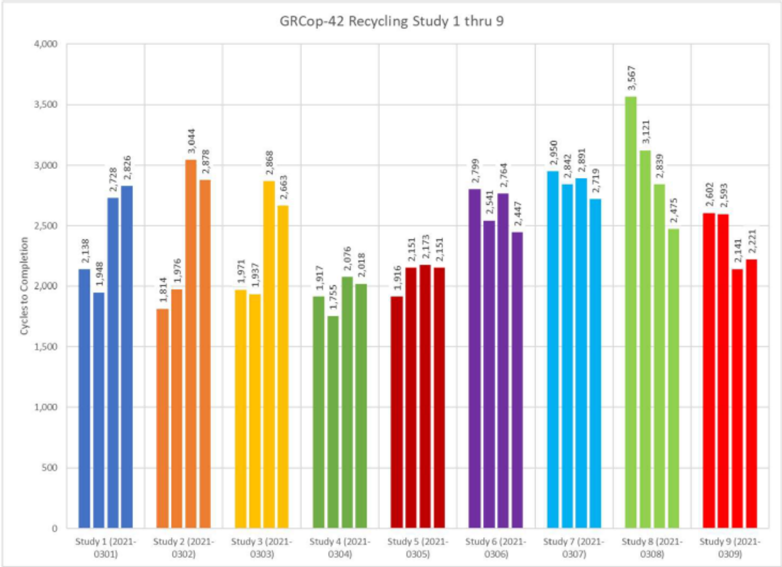


Representative stress-strain curves for each build demonstrating consistent mechanical properties despite repeated powder reuse.

Low Cycle Fatigue Life

- Low Cycle Fatigue (LCF) testing was conducted in accordance with ASTM E606 to determine the effects of powder reuse on LCF life.
- Cyclic loading was applied at 0.5 Hz in strain control using a triangular waveform. Four specimens were tested for each build.
- All specimens were tested to a total delta strain of 1.0% with a stress ratio (R) of 0.1.
- Tests were run until peak cyclic load decreased by 90% from the peak load of a reference cycle.
- Reported cycles to completion are cycles to 50% stress drop or cycles to specimen failure, whichever occurred first.
- To date, LCF testing has been performed on specimens from builds 1-9. Specimens for builds 10-12 are currently being machined.
- No trend is apparent in correlating LCF life to the number of times the powder has been reused. Two sample t-tests comparing the first build against each subsequent build failed to show a statistically significant difference in the mean number of cycles to completion between the first build and subsequent builds.

Build Number	1% Strain LCF Mean Cycles to Completion
1	2410
2	2428
3	2360
4	1942
5	2098
6	2638
7	2851
8	3001
9	2389



Results Summary

- Only powder flowability characteristics show any specific trend corresponding to the number of times GRCop-42 powder was reused, with flowability (as measured by avalanche energy, avalanche angle, and break energy) and cohesiveness (as measured by rest angle) decreasing with repeated powder reuse. This is the likely contributing factor that increased the difficulty the machine operator experienced during the recycling study.
- Slight variability was observed in the powder chemistry throughout the different builds, with copper content varying by up to 3% over the course of the study, and oxygen content varying by up to 0.041% over the course of the study. Other alloying elements (including chromium and niobium) experience minimal build-to-build variation in content. Despite the variation in copper and oxygen content, no correlation between repeated reuse and powder chemistry was apparent.
- Remaining physical (morphology and particle size distribution) and mechanical (tensile and LCF) properties exhibited minimal variation between individual builds.
- The mixing of virgin powder with the recycled powder to obtain the desired total powder mass may have contributed to the minimal variation from build to build; Nearly 50% of the powder in builds 5-12 was either virgin powder or powder that was used three times or less.