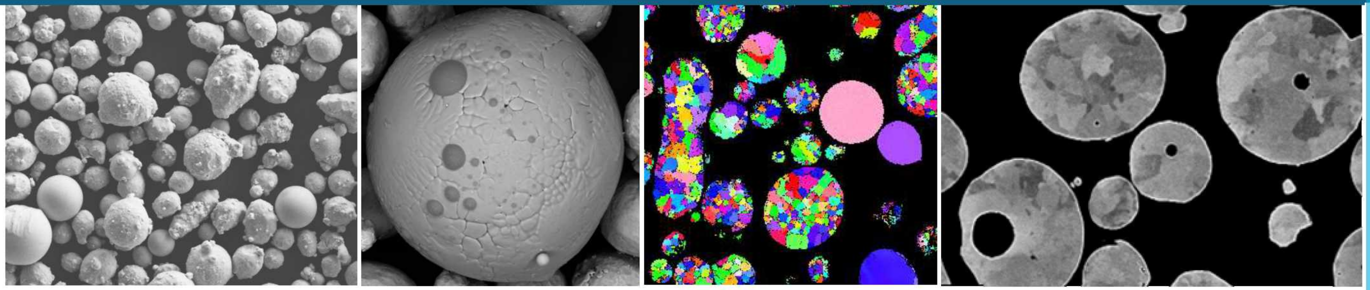
SAND2020-0464C

Pairing 316L AM Powder Feedstock with Mechanical Performance: What Really Matters



Michael Heiden, Ray Puckett, David Saiz, Bradley Jared

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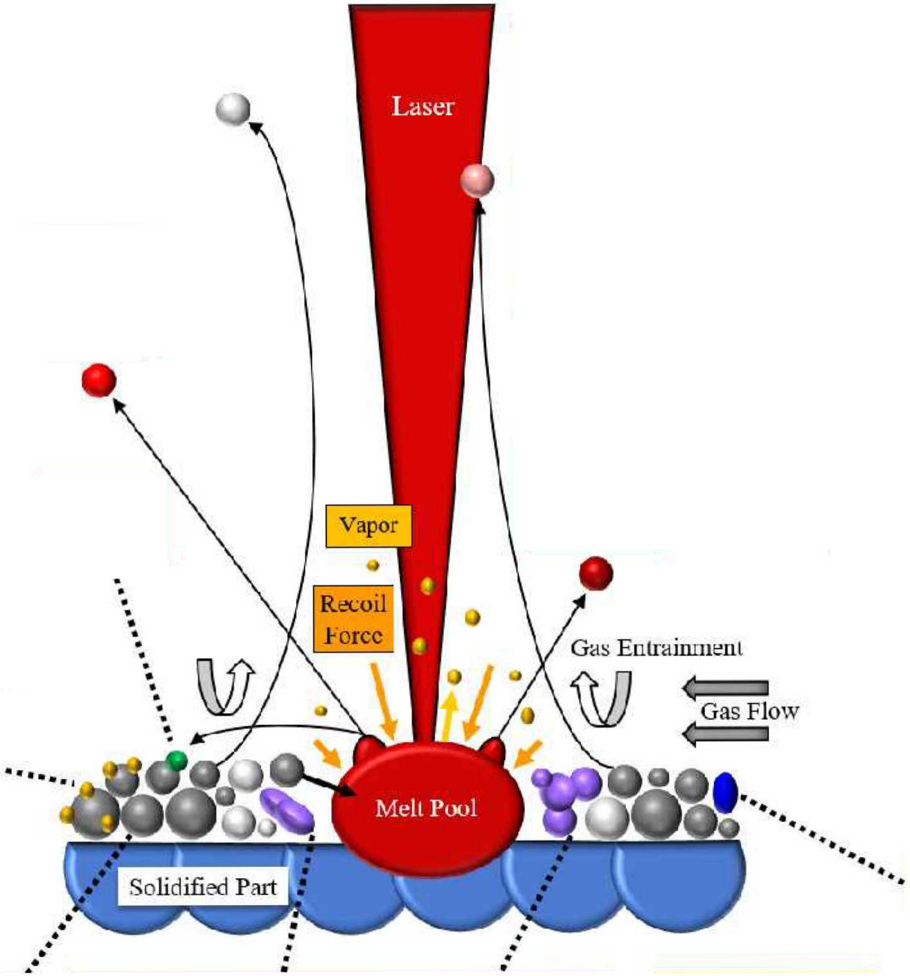
Outline

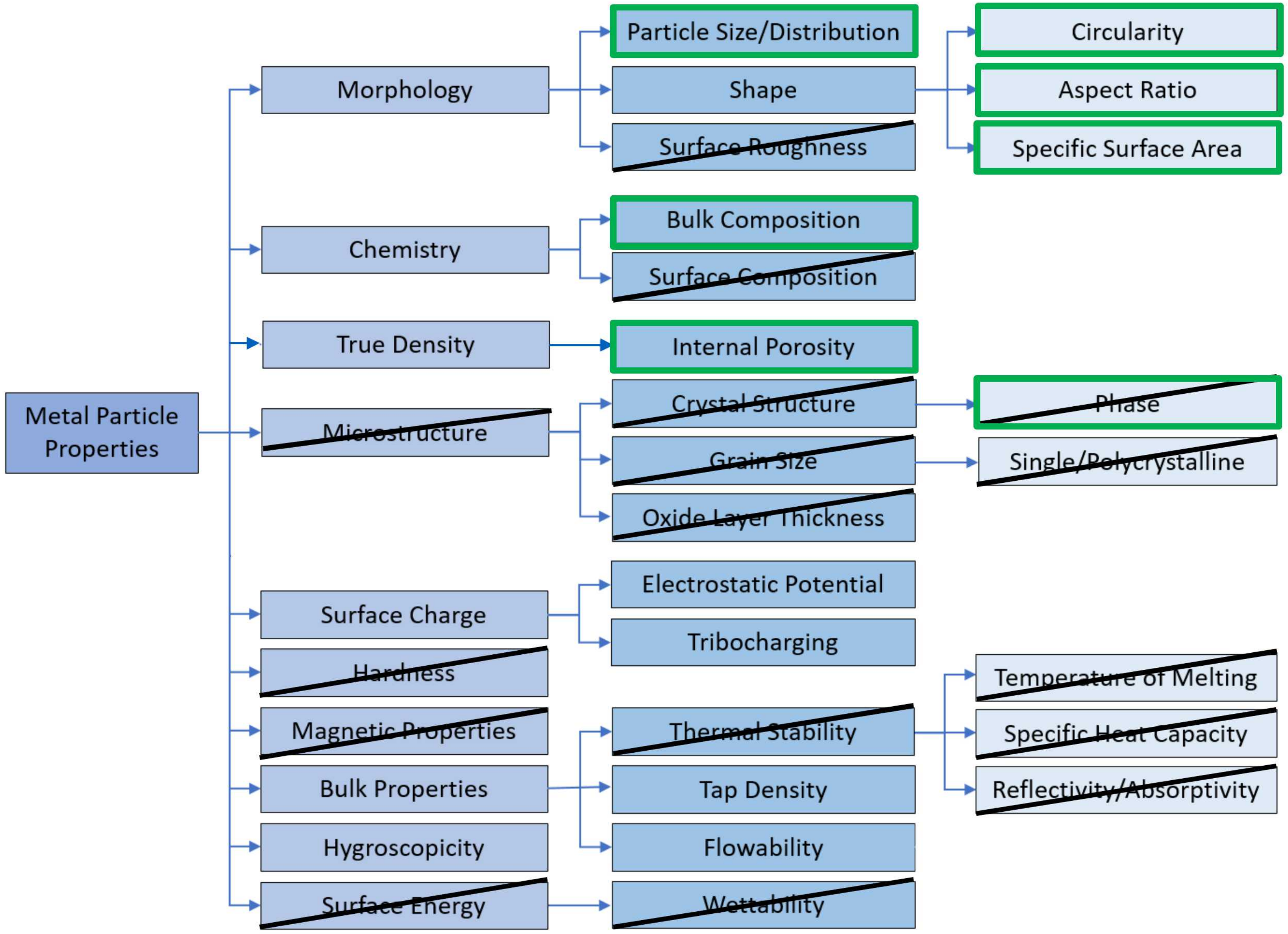
- Laser-powder interactions for 316L in L-PBF
- Powder Characteristics – Boiling Down What Matters
- Coupling Powder's Influence on Part Mechanical Properties
- Powder Reuse Effects on 316L Parts for Varying Lots

Laser-powder interactions for 316L in L-PBF



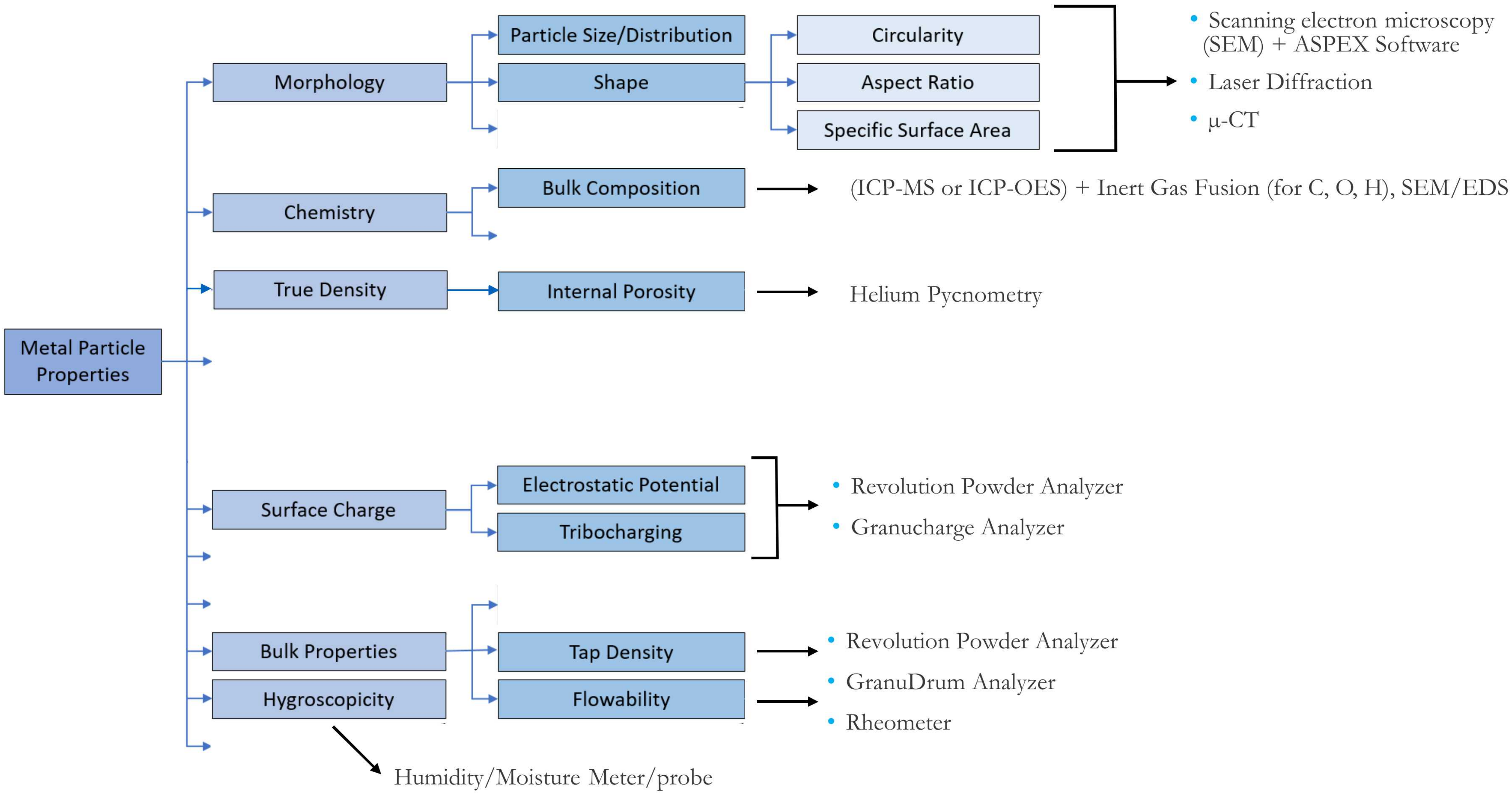
[1] Heiden, M. J., et al. (2019). "Evolution of 316L stainless steel feedstock due to laser powder bed fusion process." Additive Manufacturing **25**: 84-103.

Powder Characteristics – Boiling Down What Matters



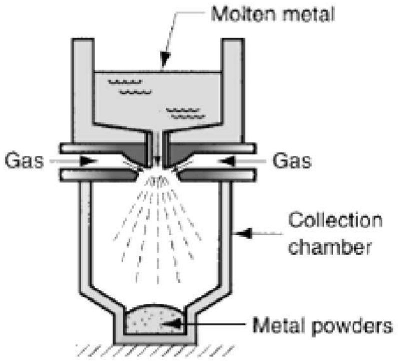
Properties crossed out are either too time intensive/expensive, shown to have no effect on part, or doesn't accurately represent whole powder lot

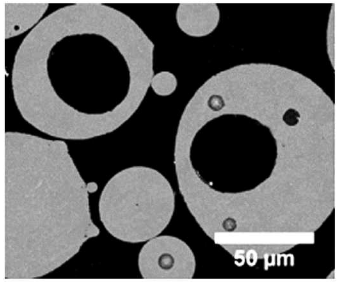
Powder Characteristics – Boiling Down What Matters



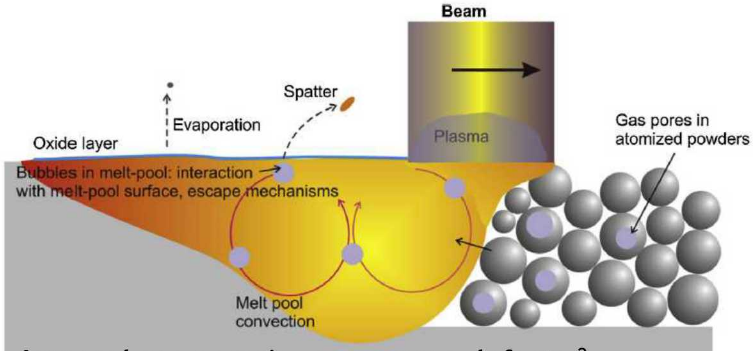
How do Powder Properties Affect AM builds?

Internal porosity created from atomization process [1]





Porosity in gas atomized powder particles [2]

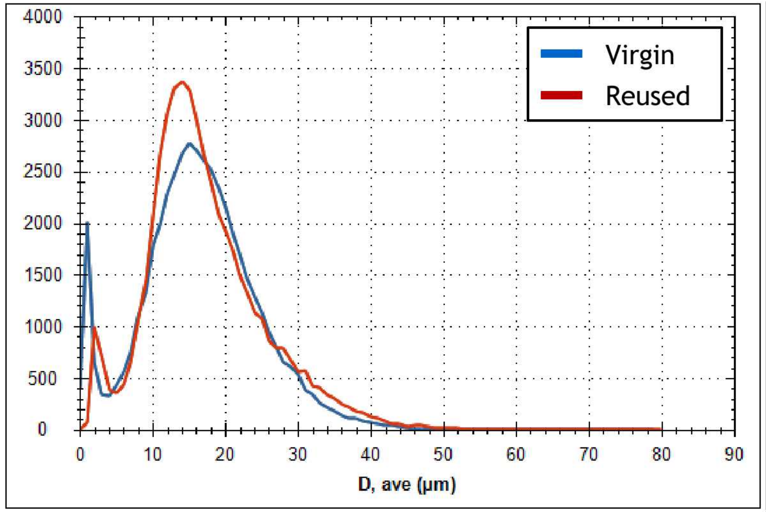


Internal gas porosity can create defects [2]

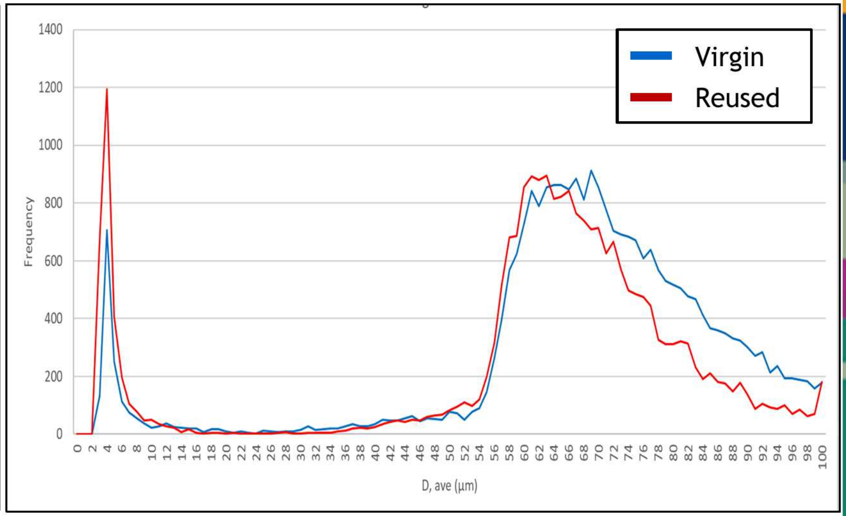
316L "bulk" chemistry changes depending on severity of machine parameters and build volume

Wt.% (Stdev)	Fe	Cr	Ni	Mo	Si	Mn	Cu	P	Co	C	S	O	N
Virgin	67.8 (0.3)	16.84 (0.34)	10.81 (0.22)	2.05 (0.20)	0.65 (0.10)	1.20 (0.12)	0.21 (0.03)	0.015 (0.002)	0.098 (0.015)	0.011 (0.002)	0.014 (0.002)	0.067 (0.010)	0.086 (0.013)
Reused (30 cycles)	67.6 (0.3)	16.91 (0.34)	10.90 (0.22)	2.02 (0.20)	0.60 (0.09)	1.27 (0.13)	0.22 (0.03)	0.016 (0.002)	0.11 (0.016)	0.016 (0.002)	0.014 (0.002)	0.095 (0.014)	0.090 (0.013)
ASTM Spec [2]	61-69	16-18	10-14	2-3	1 max	2 max	-	0.045 max	-	0.03 max	0.03 max	-	-

L-PBF reuse coarsens particles [3]



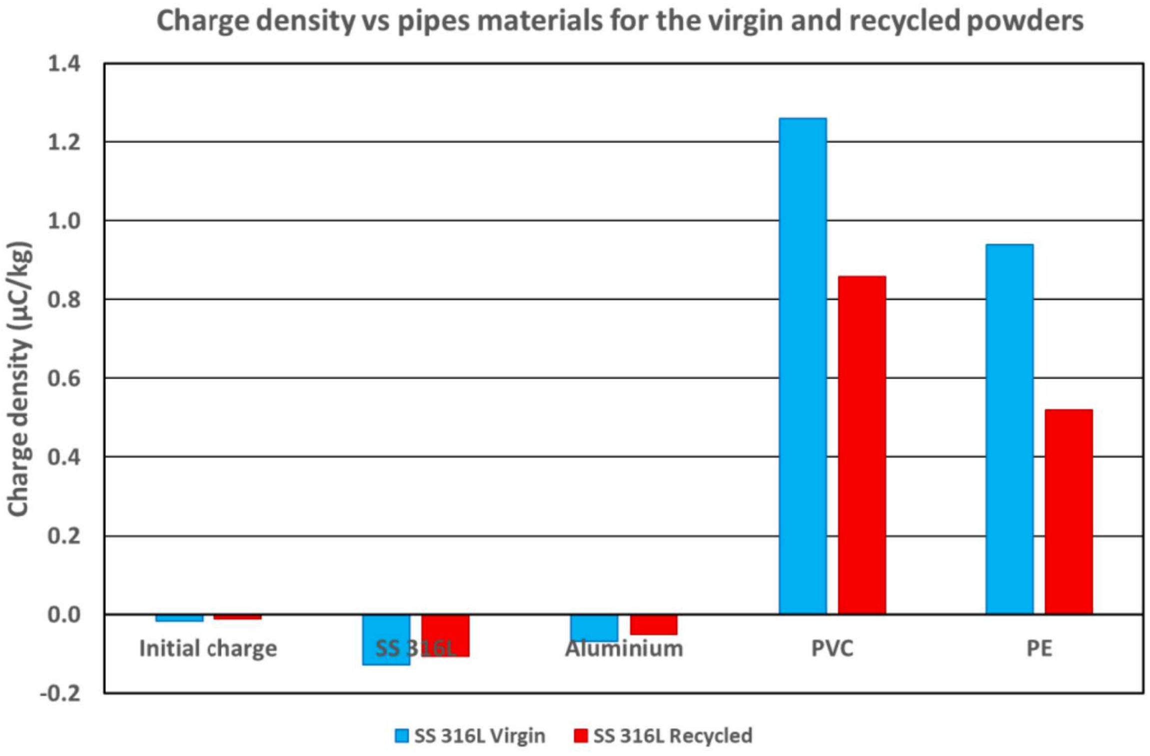
DED (LENS) reuse reduces coarse particles



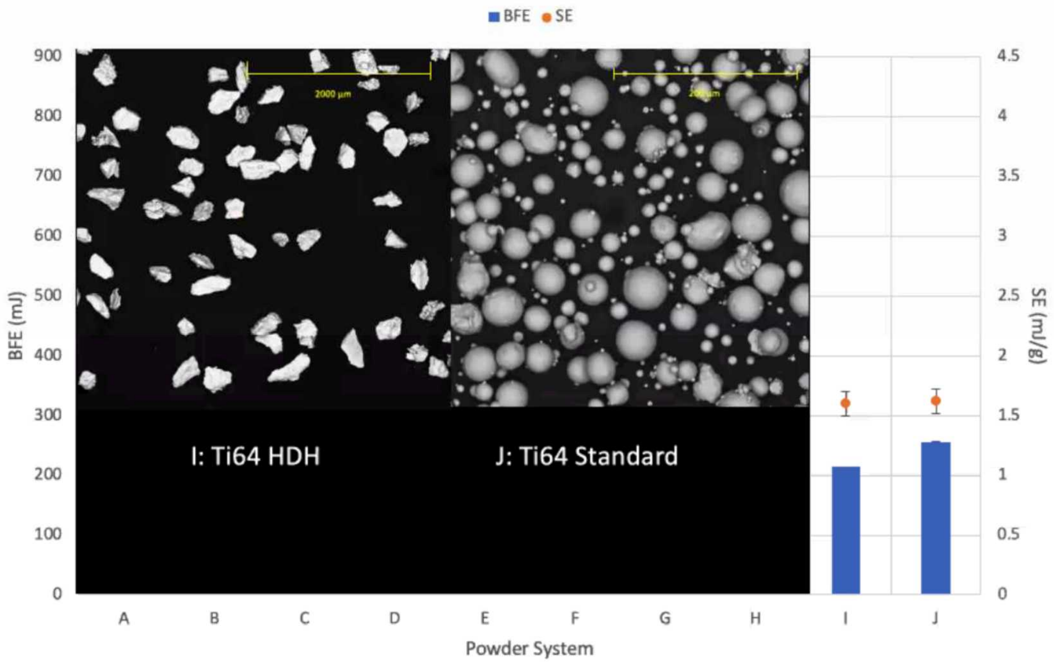
[1] Hebert, R. J. (2016). "Viewpoint: metallurgical aspects of powder bed metal additive manufacturing." Journal of Materials Science **51**(3): 1165-1175.
[2] I.E. Anderson, E.M.H. White, R. Dehoff, Feedstock powder processing research needs for additive manufacturing development, Current Opinion in Solid State and Materials Science 22(1) (2018) 8-15.
[3] Heiden, M. J., et al. (2019). "Evolution of 316L stainless steel feedstock due to laser powder bed fusion process." Additive Manufacturing **25**: 84-103.

How do Powder Properties Affect AM builds?

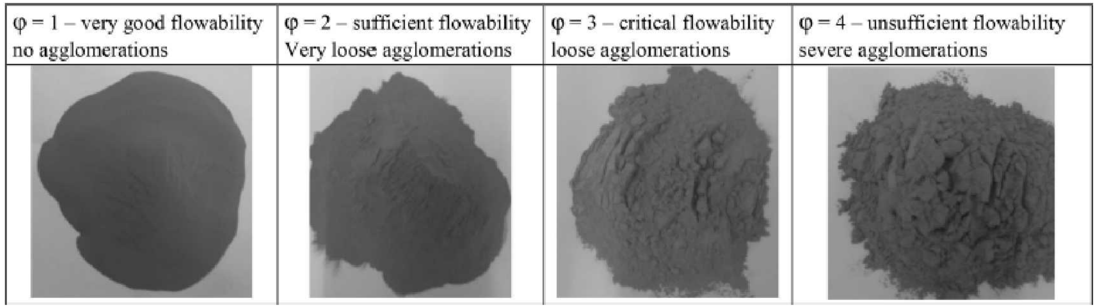
Tribocharging effects change with reuse [1]



- Triboelectric charging and charge density on particles tend to be higher for 316L virgin than reused, indicating less flowability



Surprising result: very different powder morphologies yield similar flow [4]

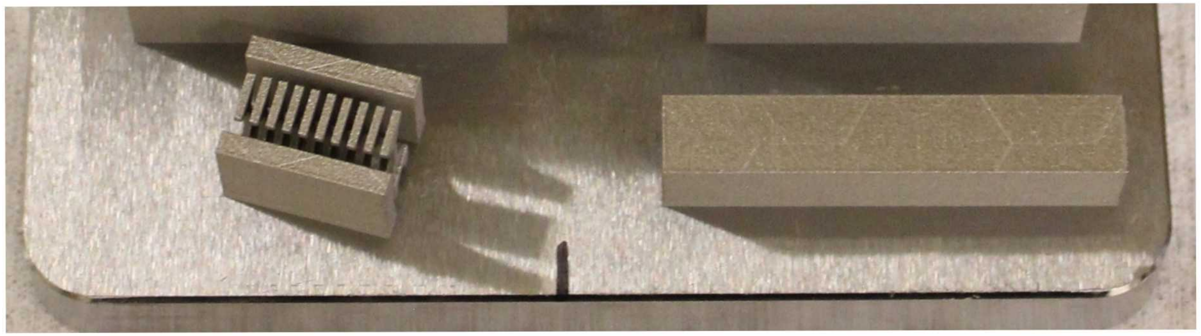


[3]

[1] Granutools. Flowability, bulk density and electrical charges - Additive manufacturing powders characterization. GranuTools Application Note. GranuTools.com (2017)
[2] Mercury Scientific Inc. AM Powder Testing. Flowability Application Bulletin 7.
[3] Spierings, A. B. Powder flowability characterization methodology for powder bed based metal additive manufacturing. Prog Addit Manuf. 1:9-20 (2016)
[4] Rollet, Tony, Carnegie Mellon University, Next Manufacturing Seminar

Coupling Powder & Mechanical Properties

+



Powder Sample

High Throughput Tensile Sample

Charpy Test Coupon

1. Do part properties change with extensive powder reuse?

2. Do any powder characteristics indicate a "bad" quality part?

Types of 316L powder tested:

- ProX 200 powder
- ProX 320 Powder
- Höganäs Virgin
- Carpenter Virgin

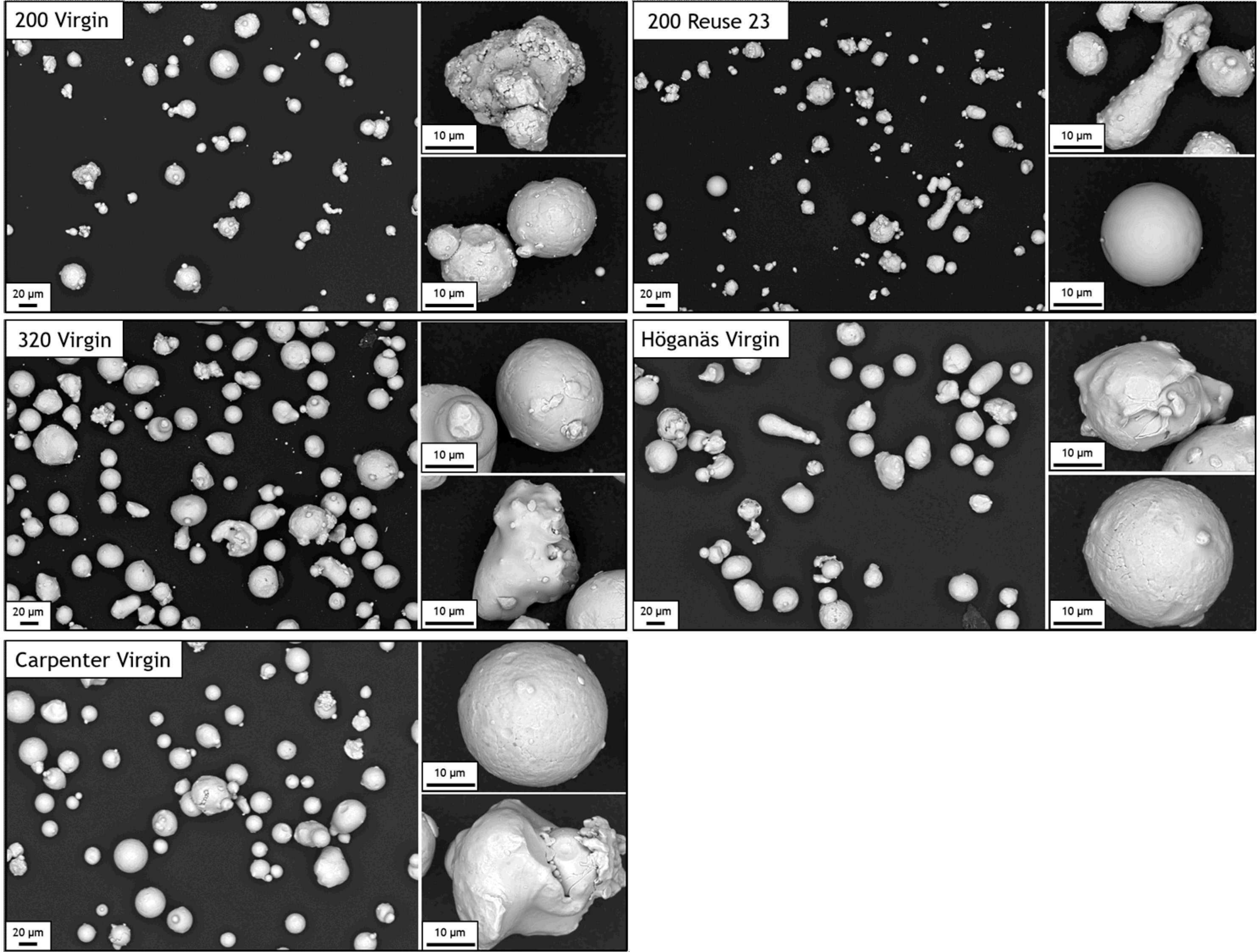
Machine parameters:

- Power: 110 W
- Mark Speed: 1400 mm/s
- Hatch Spacing: 50 µm
- Focus offset: 1.5 mm
- Style: Hexagon
- Layer Thickness: 30 & 40 µm

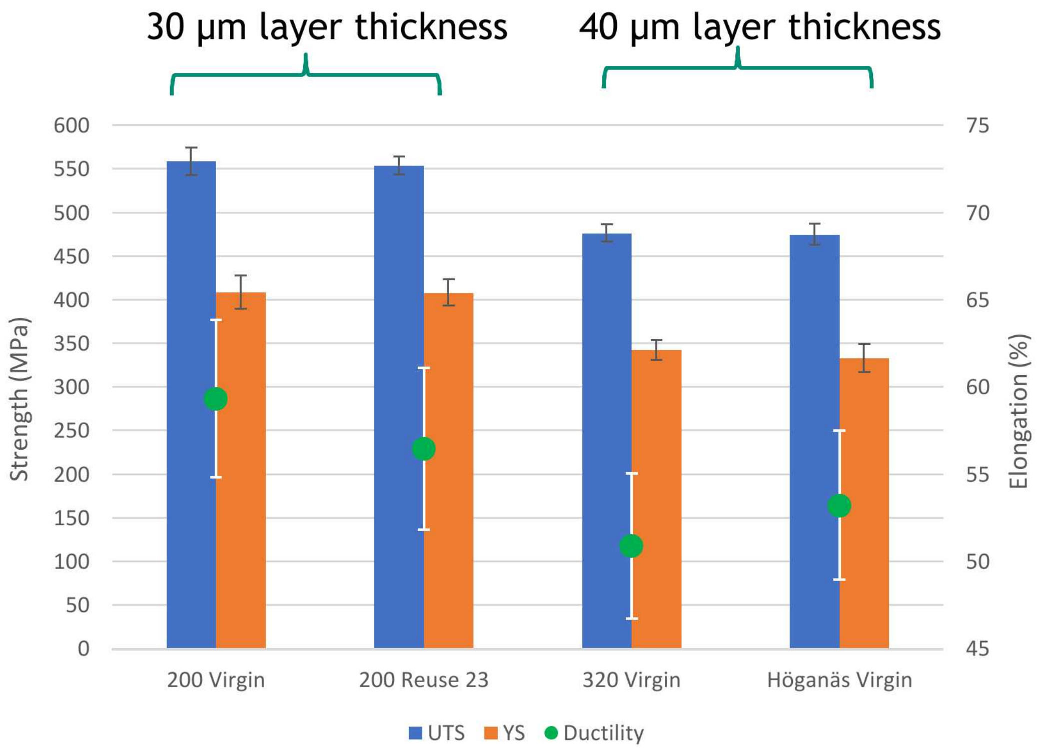
Properties tracked for each build:

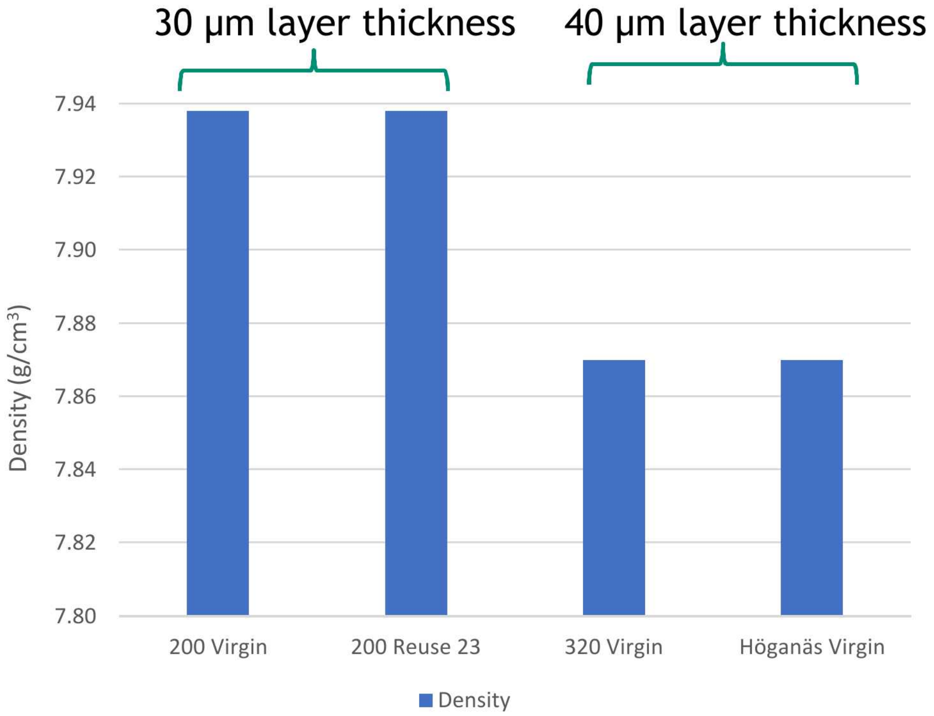
- Powder Morphology
  - Average Diameter
  - Size distribution: D10, D50, D90
  - Aspect Ratio
- Powder Bulk Chemistry
  - EDS
  - ICP-MS/OES + LECO
- Part Mechanical Properties
  - UTS
  - YS
  - % EL
  - Density
  - Fracture Toughness
  - Hardness

Starting Powders

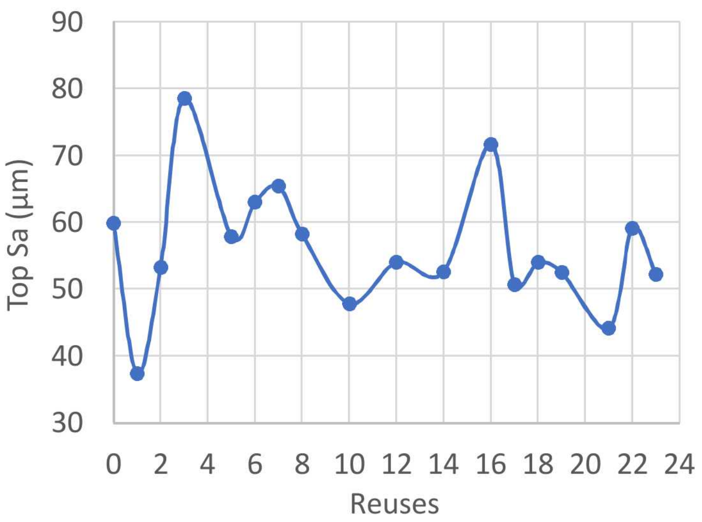


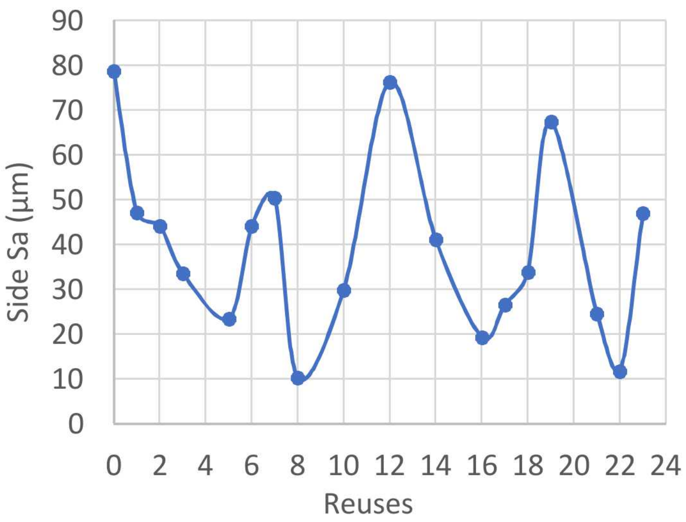
Initial powder quality is an indicator of resulting mechanical properties



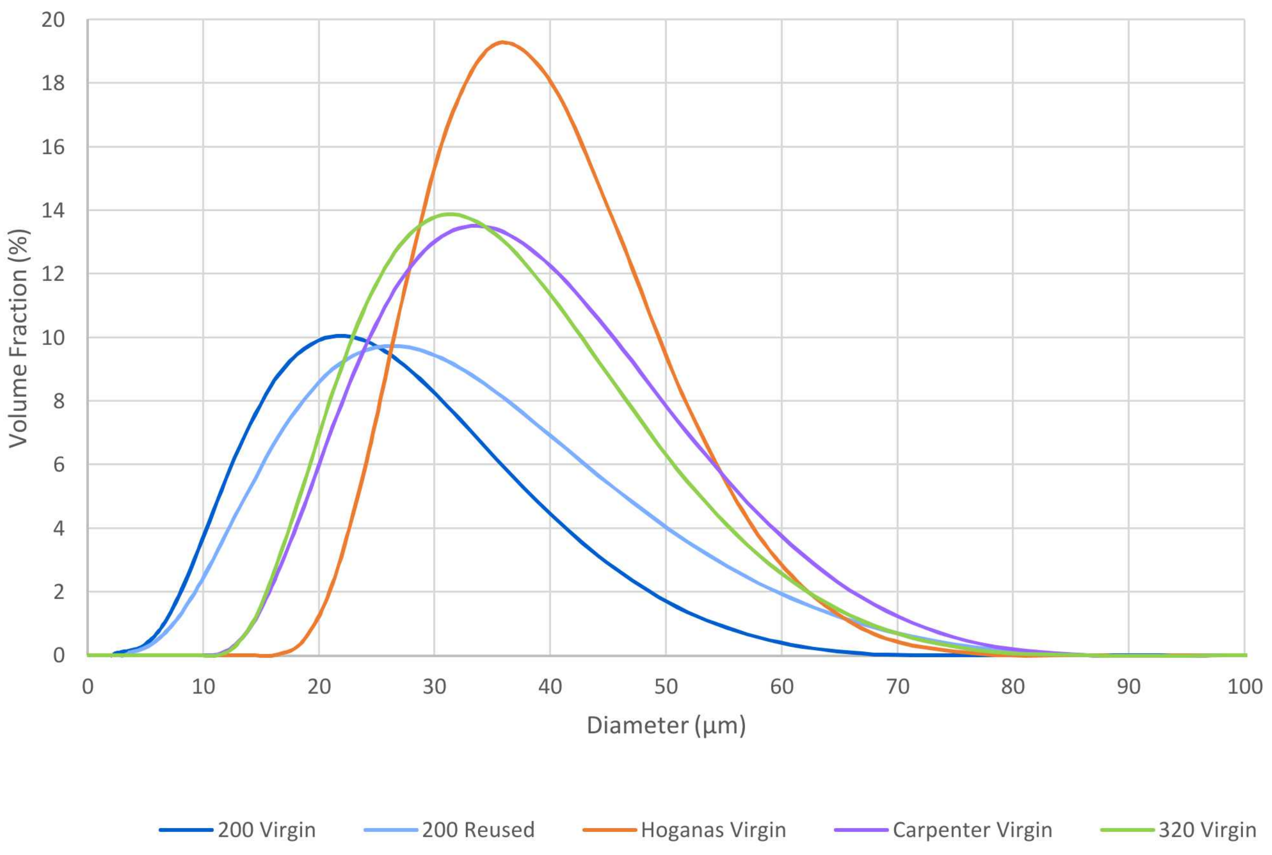


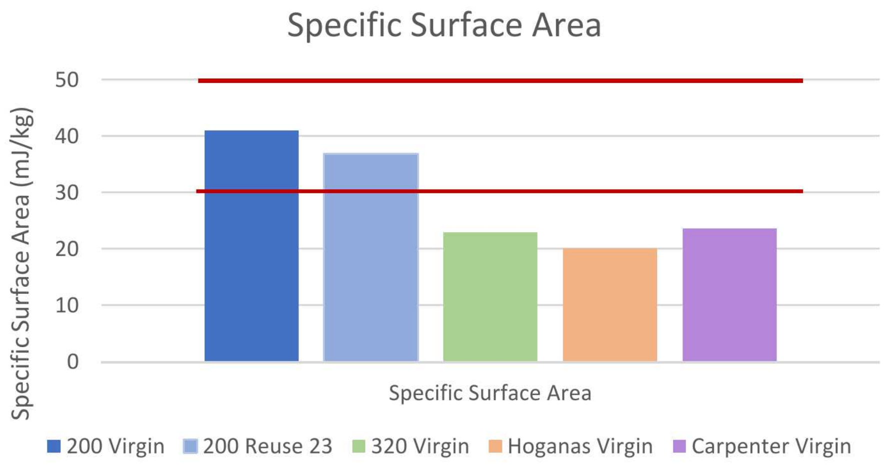
- Mechanical properties for parts built at 30 µm layer thickness best at these process settings
- UTS, YS, ductility & density change **very little** with reuse
- Part surface roughness is unaffected by powder reuse

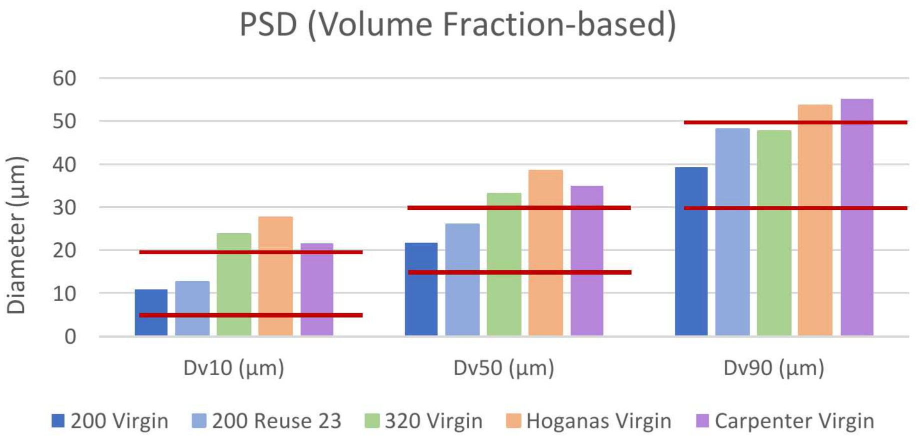


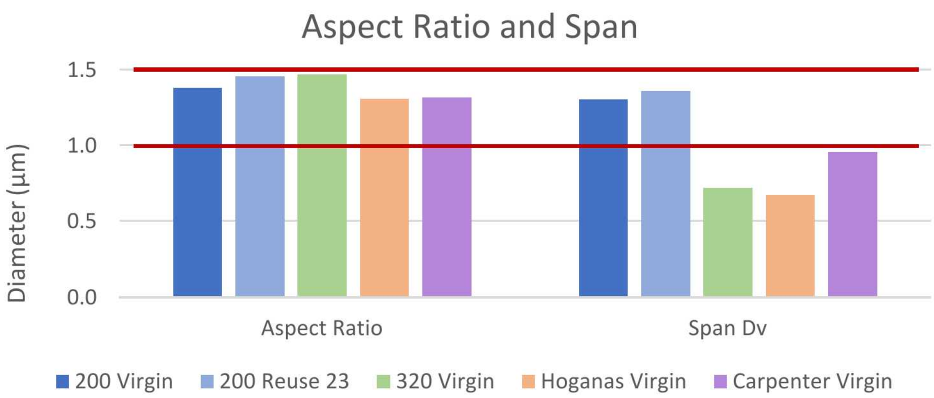


Tracking powder morphology





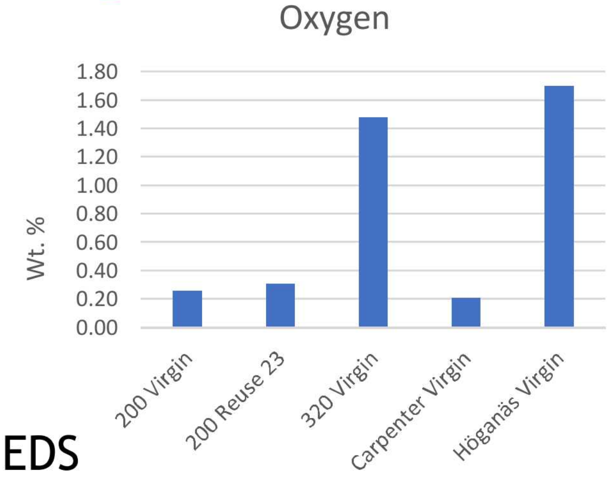


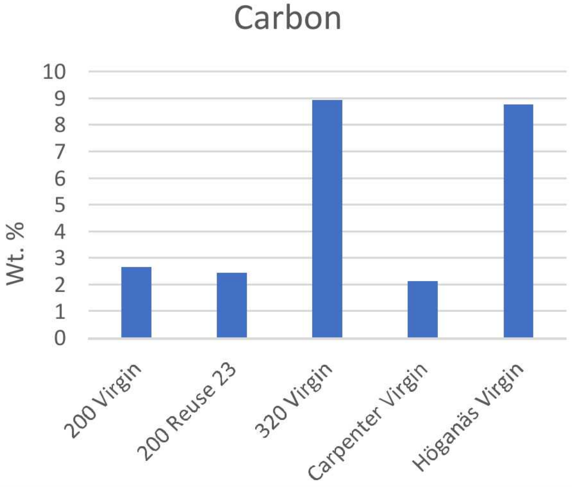


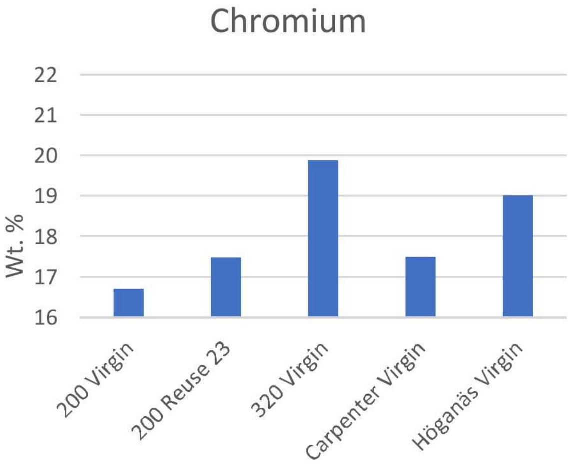
- Large variety of PSD distributions
- Virgin 320 and Höganäs powders fairly similar
- Reuse shifts distribution to the right and span widens

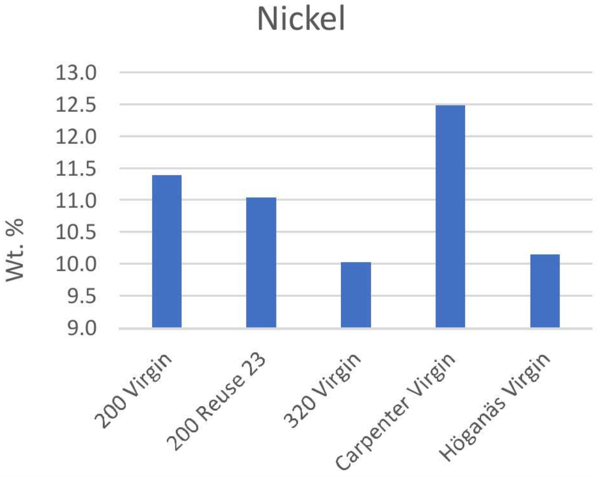
EDS not accurate enough for use in specifications

EDS

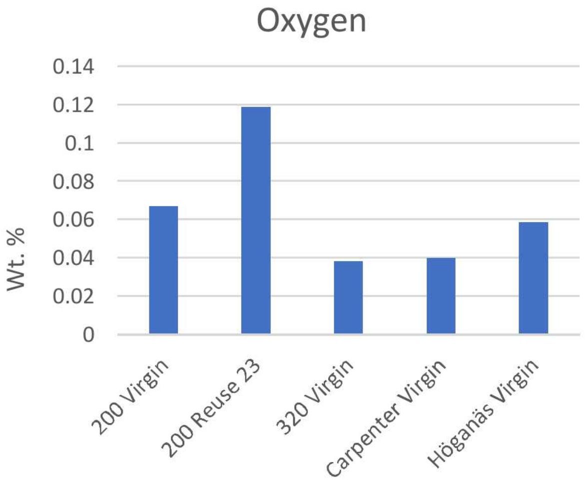


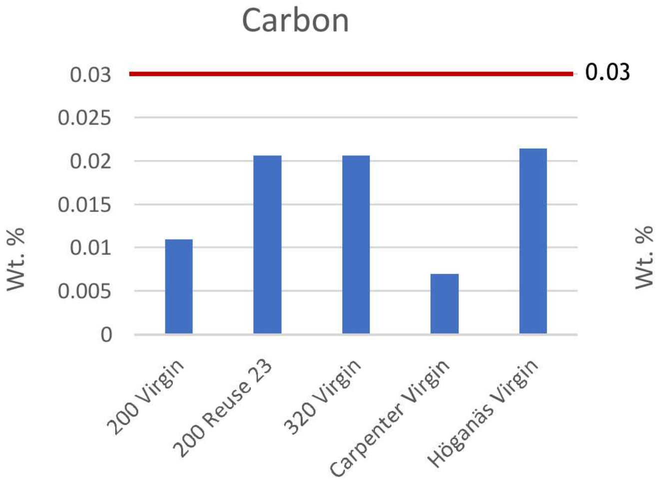


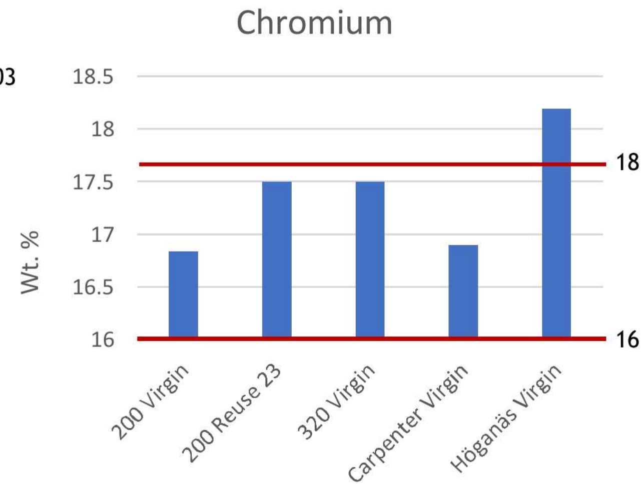


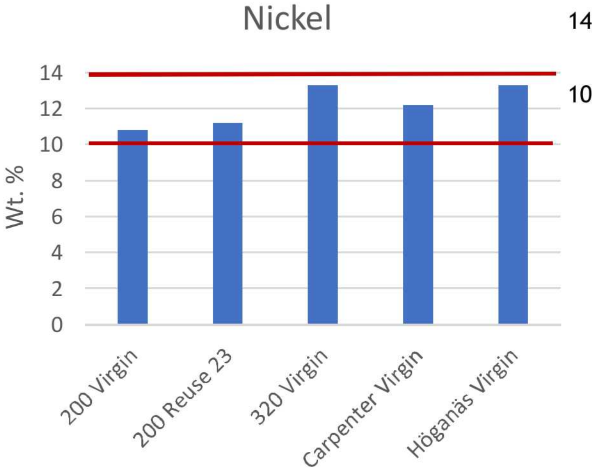


ICP-MS + IGF



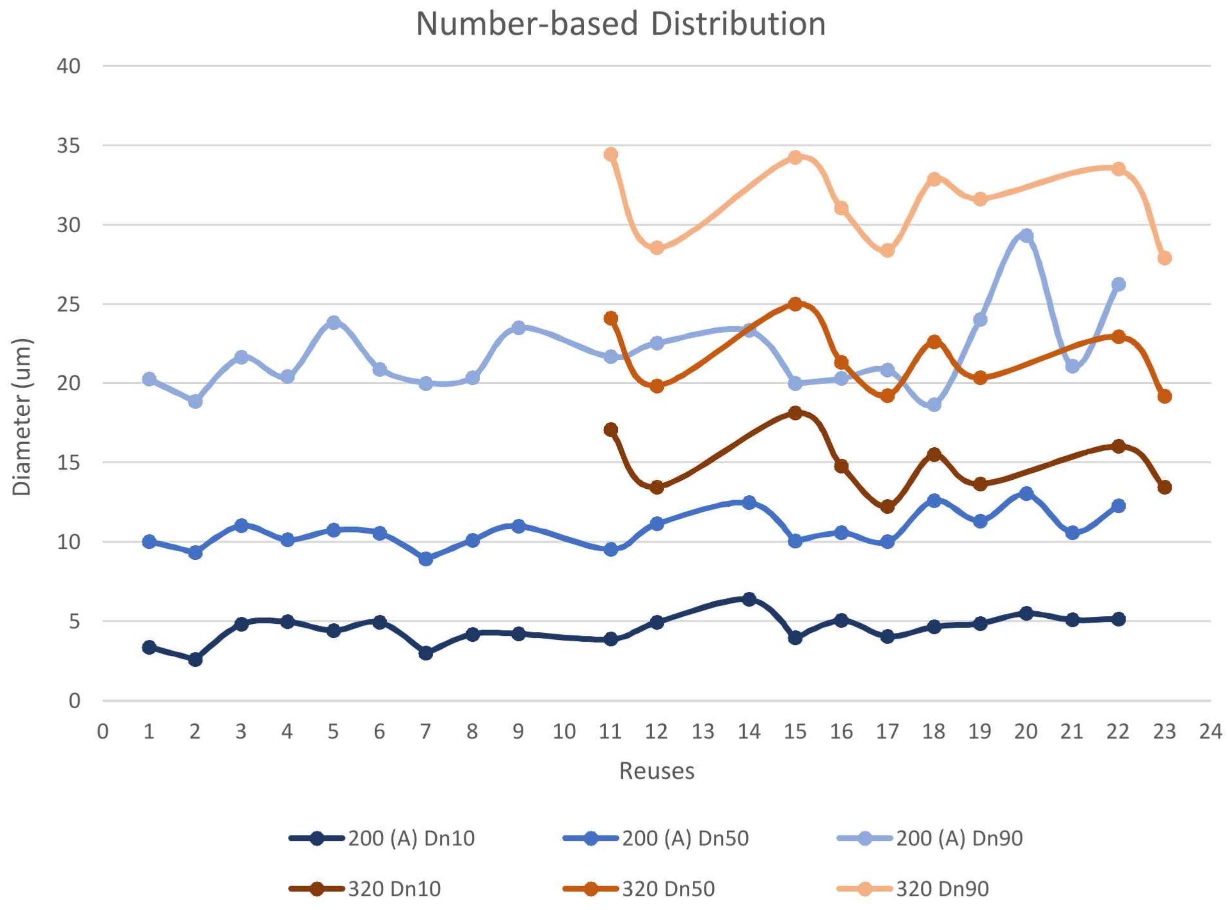


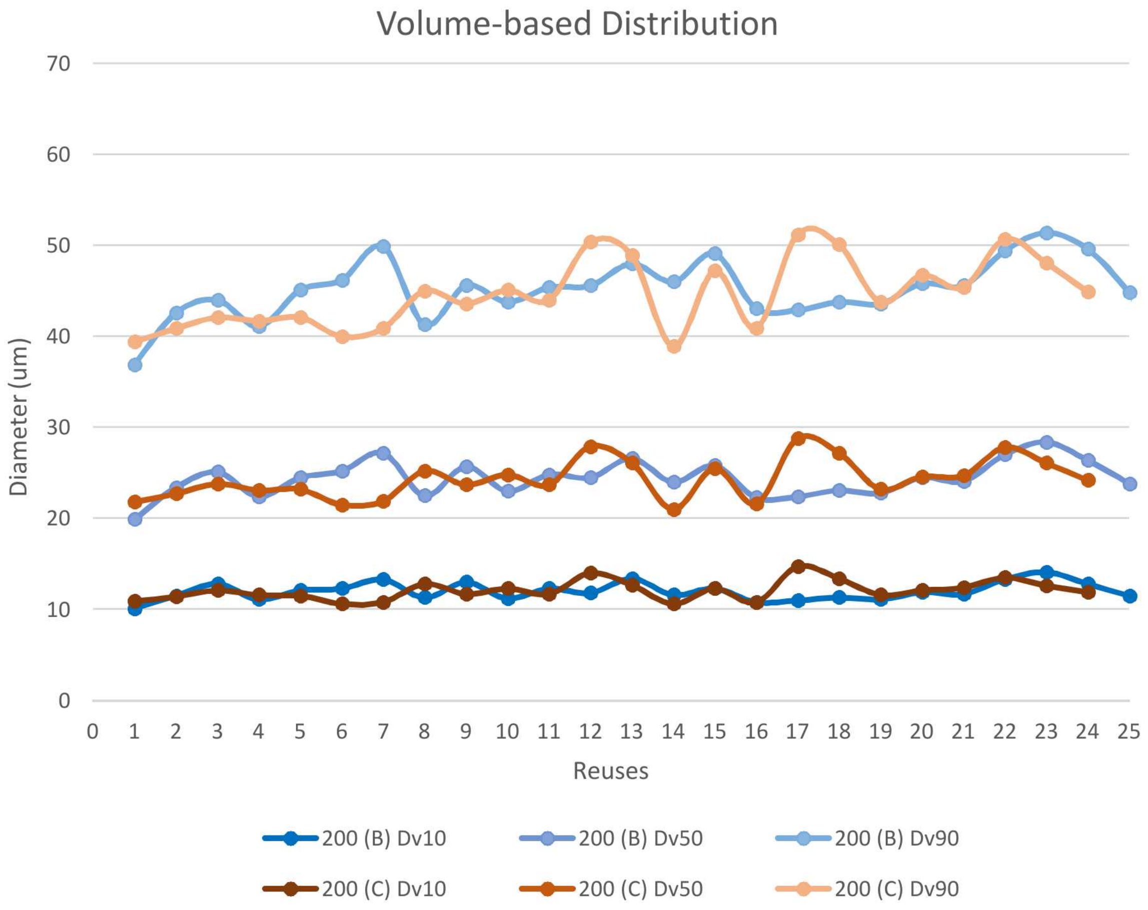




- Oxygen currently has no ASTM limit – should set limit at 0.1 wt. %. Beyond has unknown effect on 316L parts
- According to existing ASTM standard for 316L SS, Höganäs would be out of Cr tolerance
- Powder with higher oxygen % does not necessarily produce weaker mechanical properties

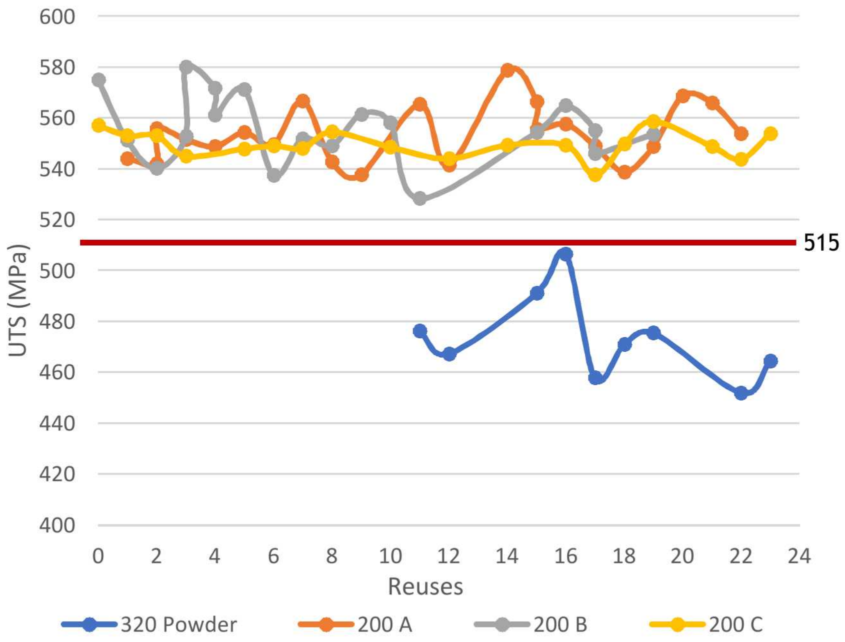
Particle Size Distribution (PSD) Increases with Reuse

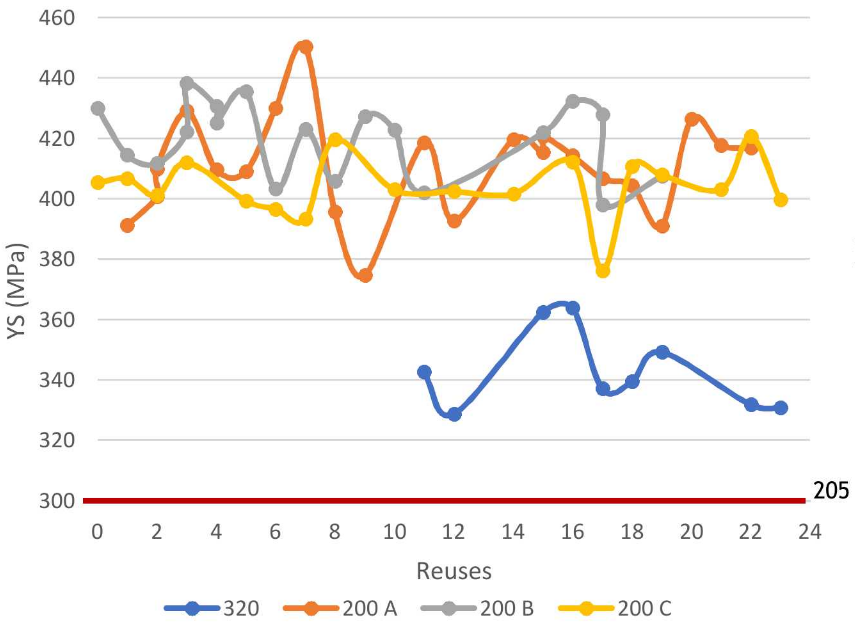


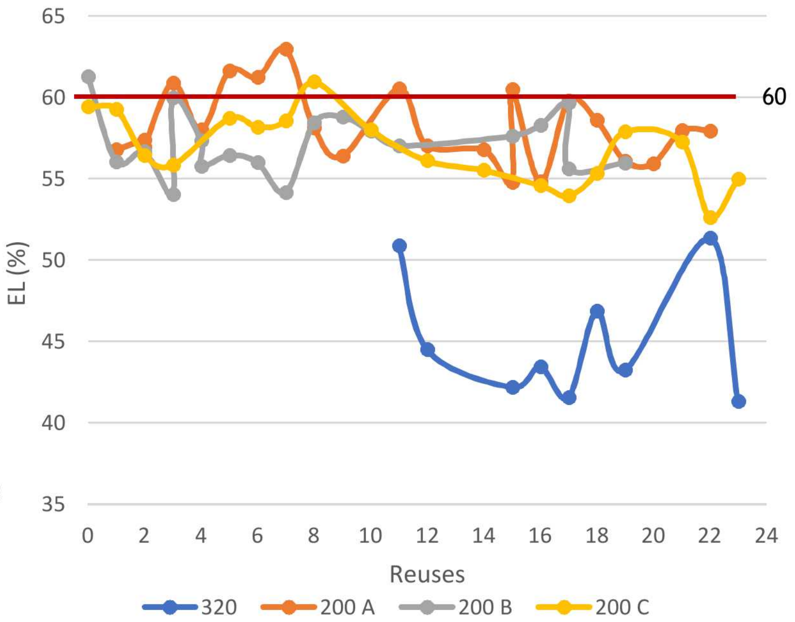


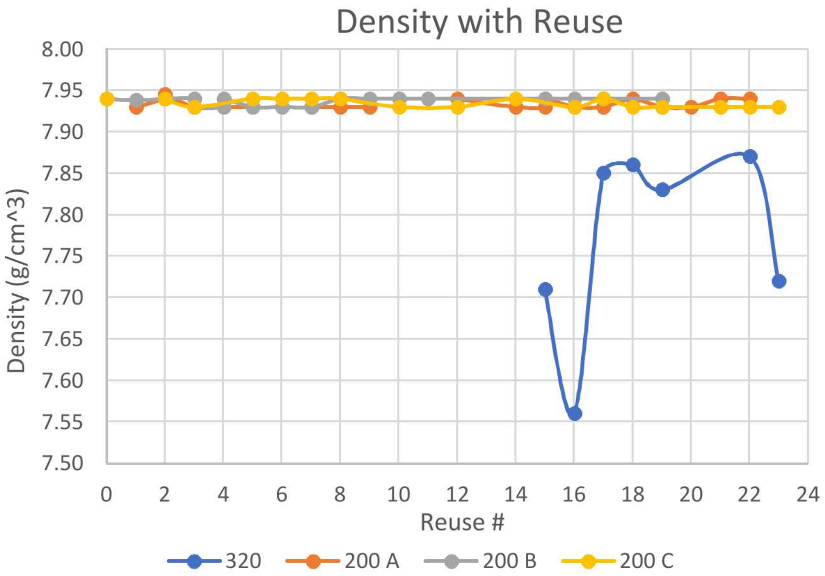
- Particle size distributions trend slightly upward to larger diameters with reuse
- Some variation throughout – may be due to sampling

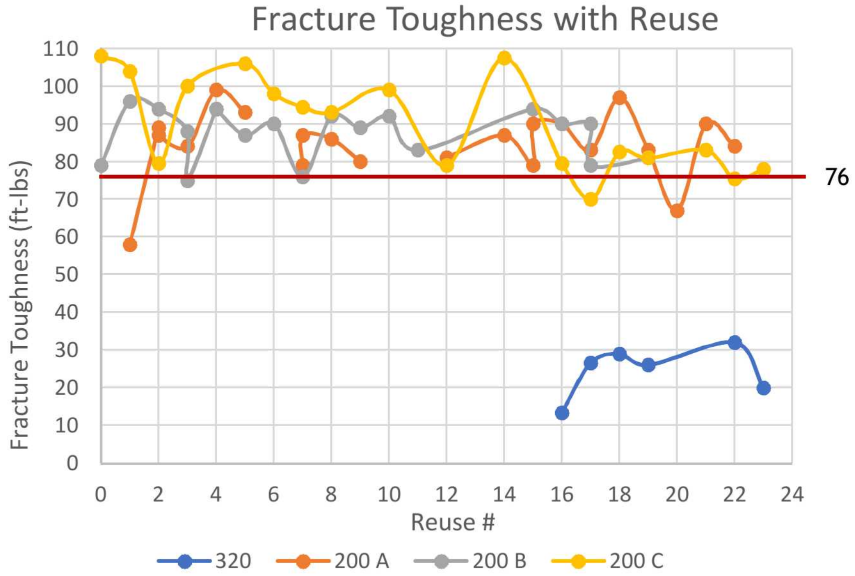
No trends in AM 316L mechanical property variation with reuse

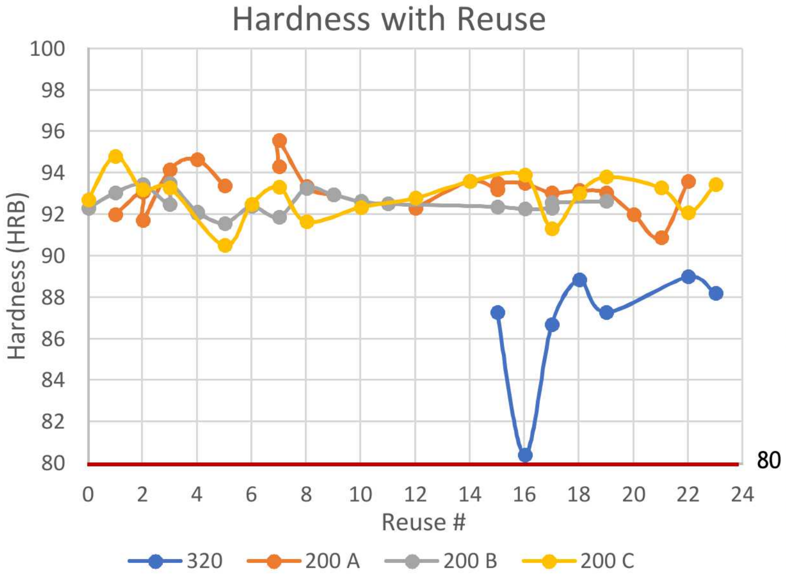




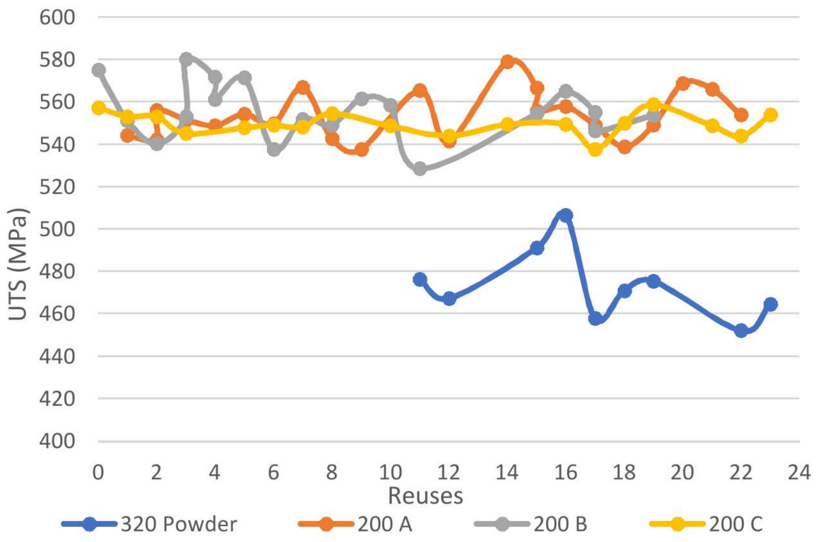


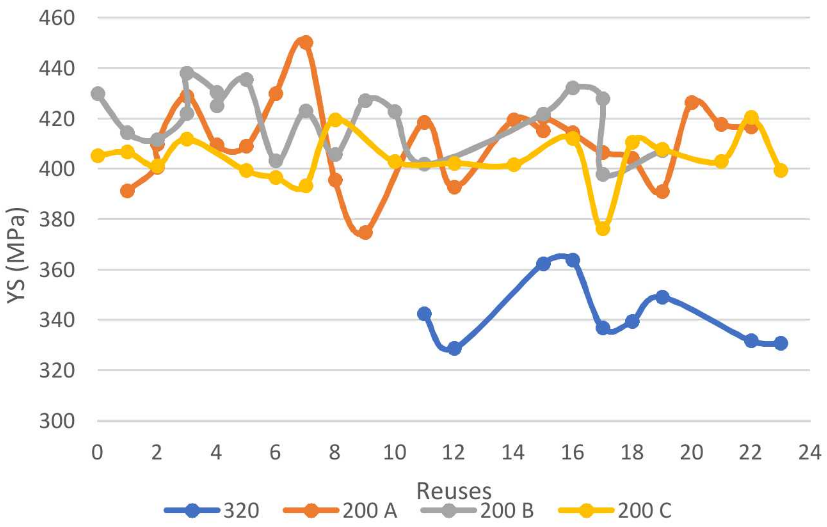


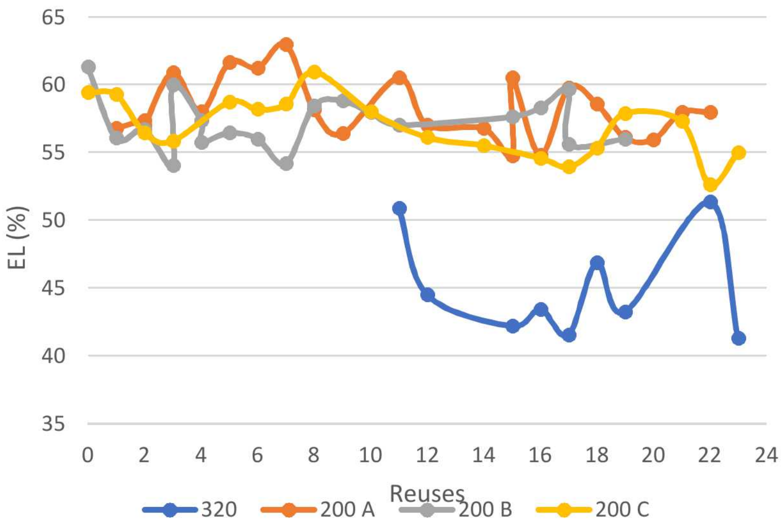




AM 316L mechanical properties insensitive to powder reuse



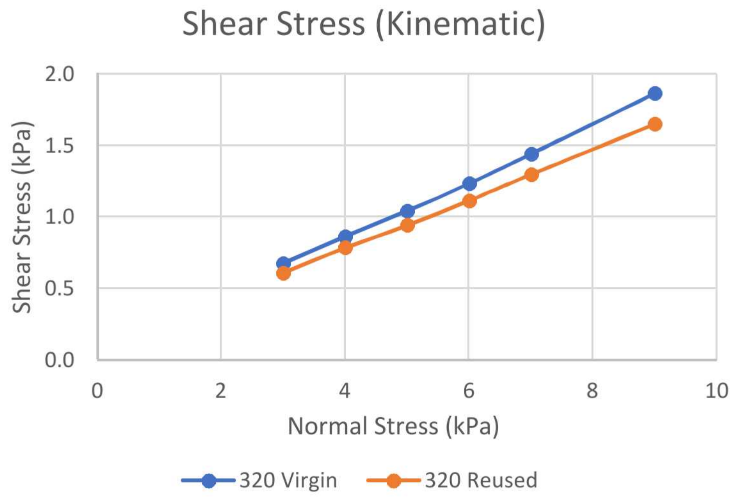


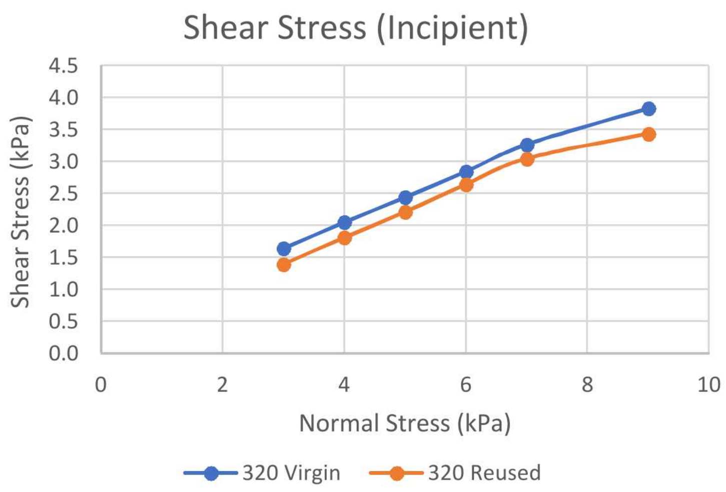


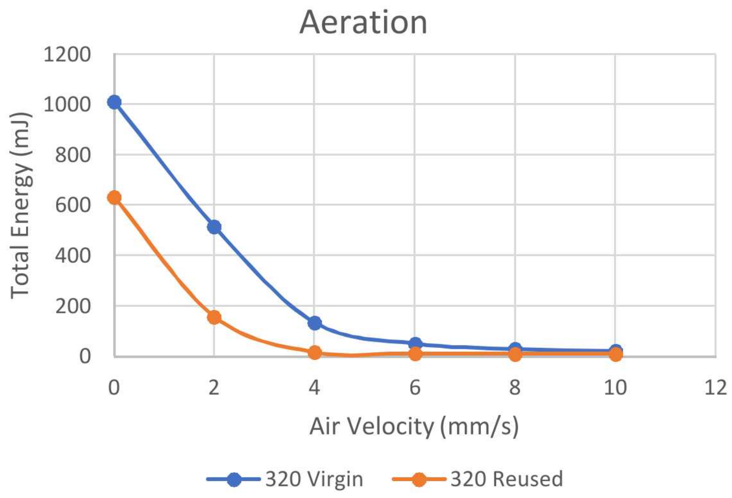
- Lots of powder characteristics change with reuse, but properties don't reflect that change
- Mechanical property variability may be primarily due to machine process and build location variability, <u>not powder</u>
- Can any flowability metrics be used to assess powder quality?

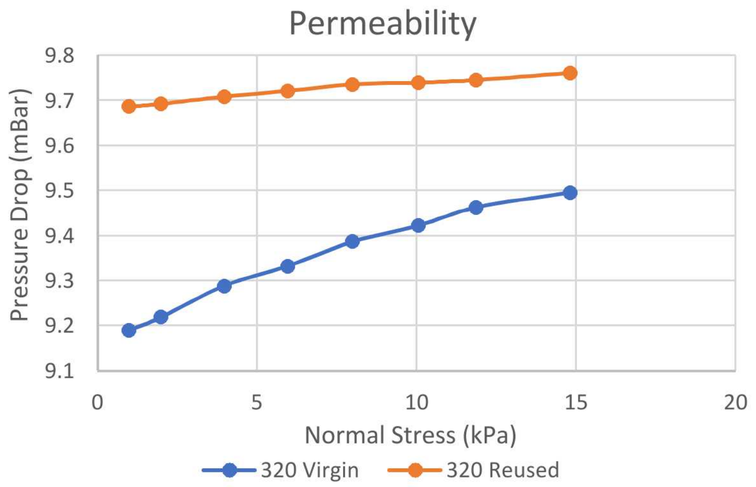
> - UTS variation: ± 60 MPa
> - YS variation: ± 70 MPa
> - Ductility variation: ± 16%
> - Density variation: ± 0.02 g/cm^3
> - Hardness variation: ± 6 HRB
> - Fracture Toughness variation: ± 45 ft-lbs

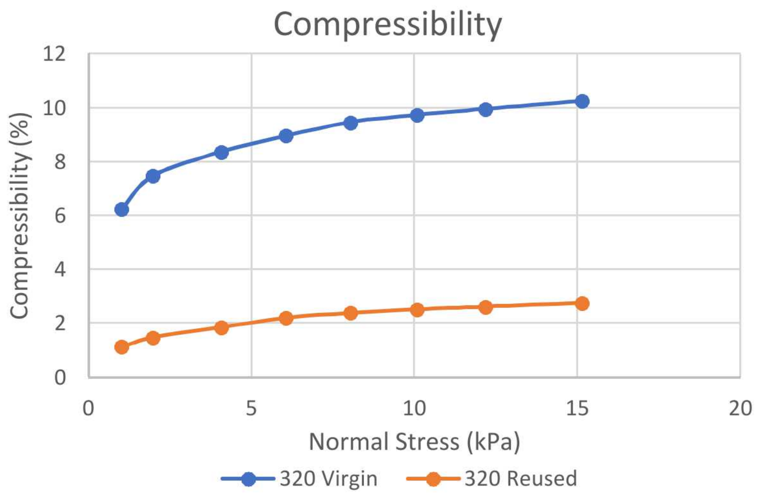
FT4 Rheometry of 320 powder reuse







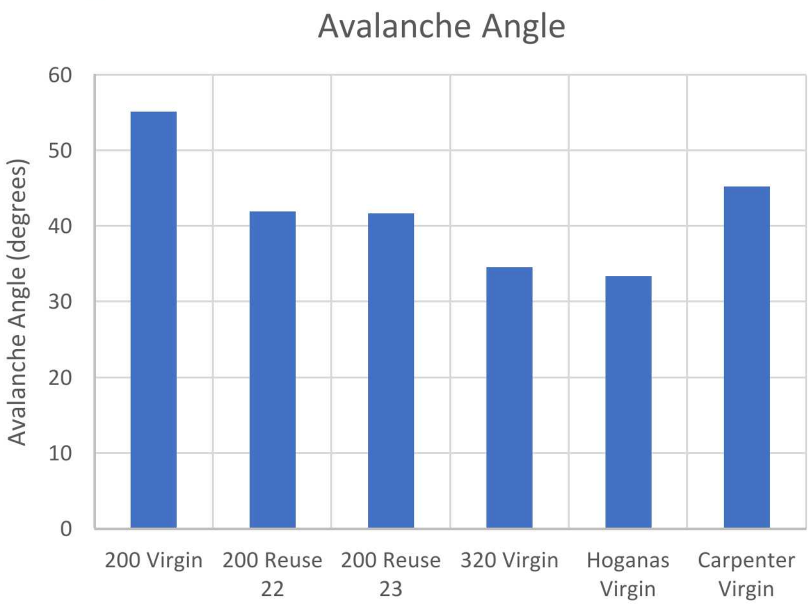


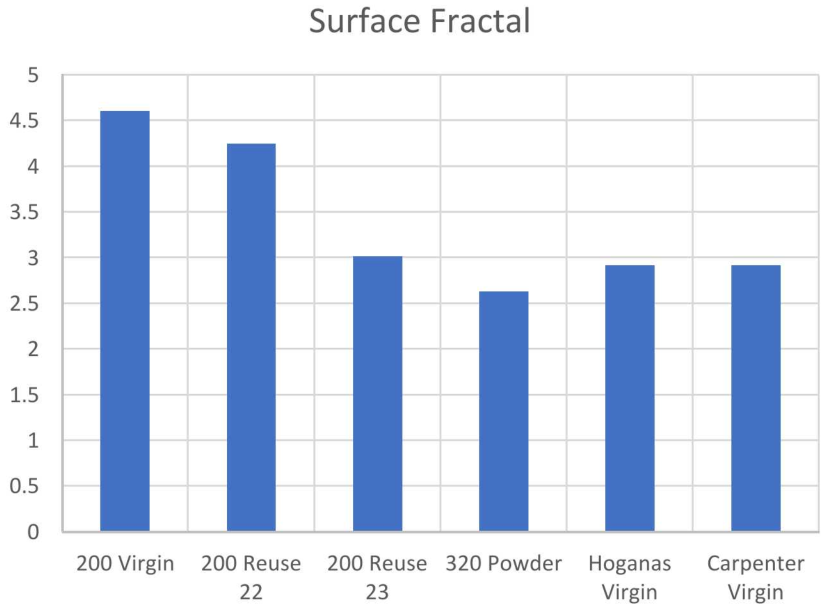


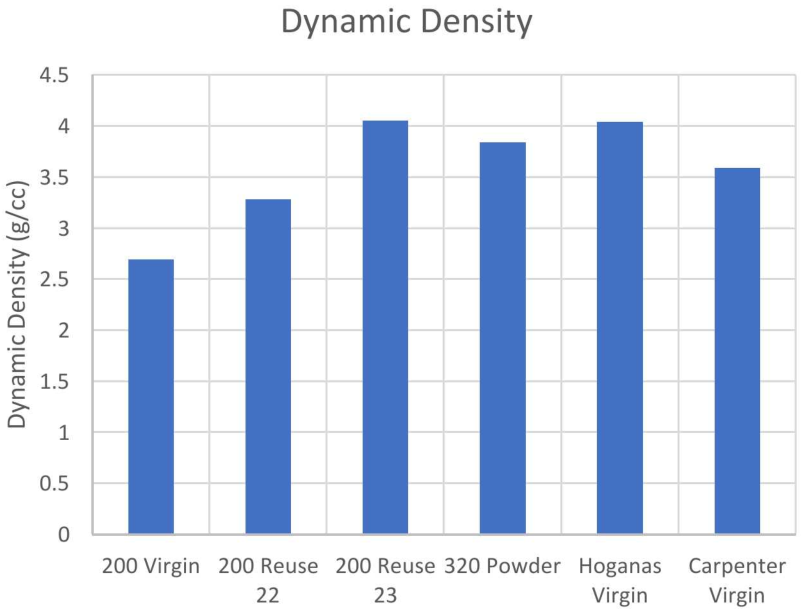
Metric	320 Virgin	320 Reuse 23
BFE (mJ)	991.76	1077.37
SI	0.97	1.00
FRI	1.04	1.13
SE	3.50	2.86
CBD	4.55	4.72
BDtap	4.84	4.66

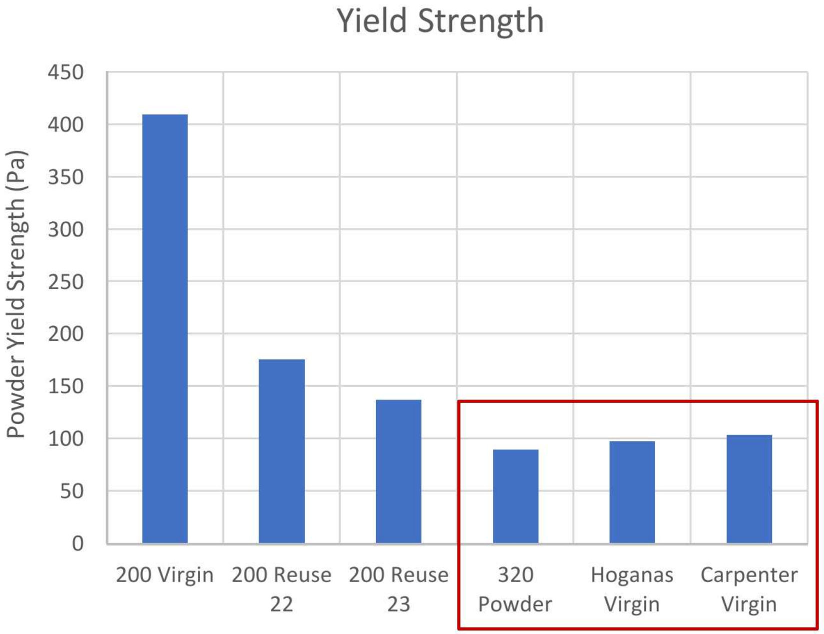
- Virgin powder: higher tap bulk density
- Reused powder: better packing ability, less entrapped air and better ability to release trapped air, less cohesive and lower tendency to agglomerate, lower mechanical interlocking, lower resistance/shear stress to flow, and more stable

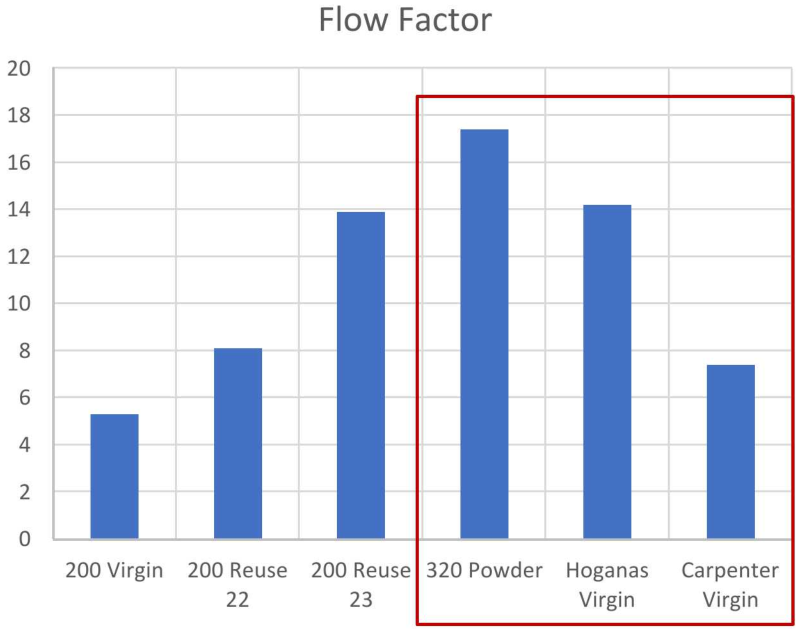
Revolution Powder Analysis

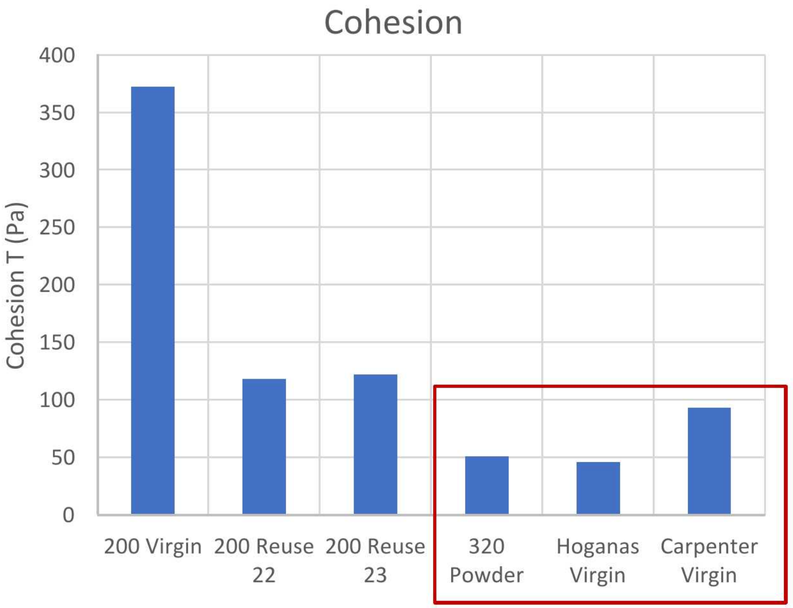




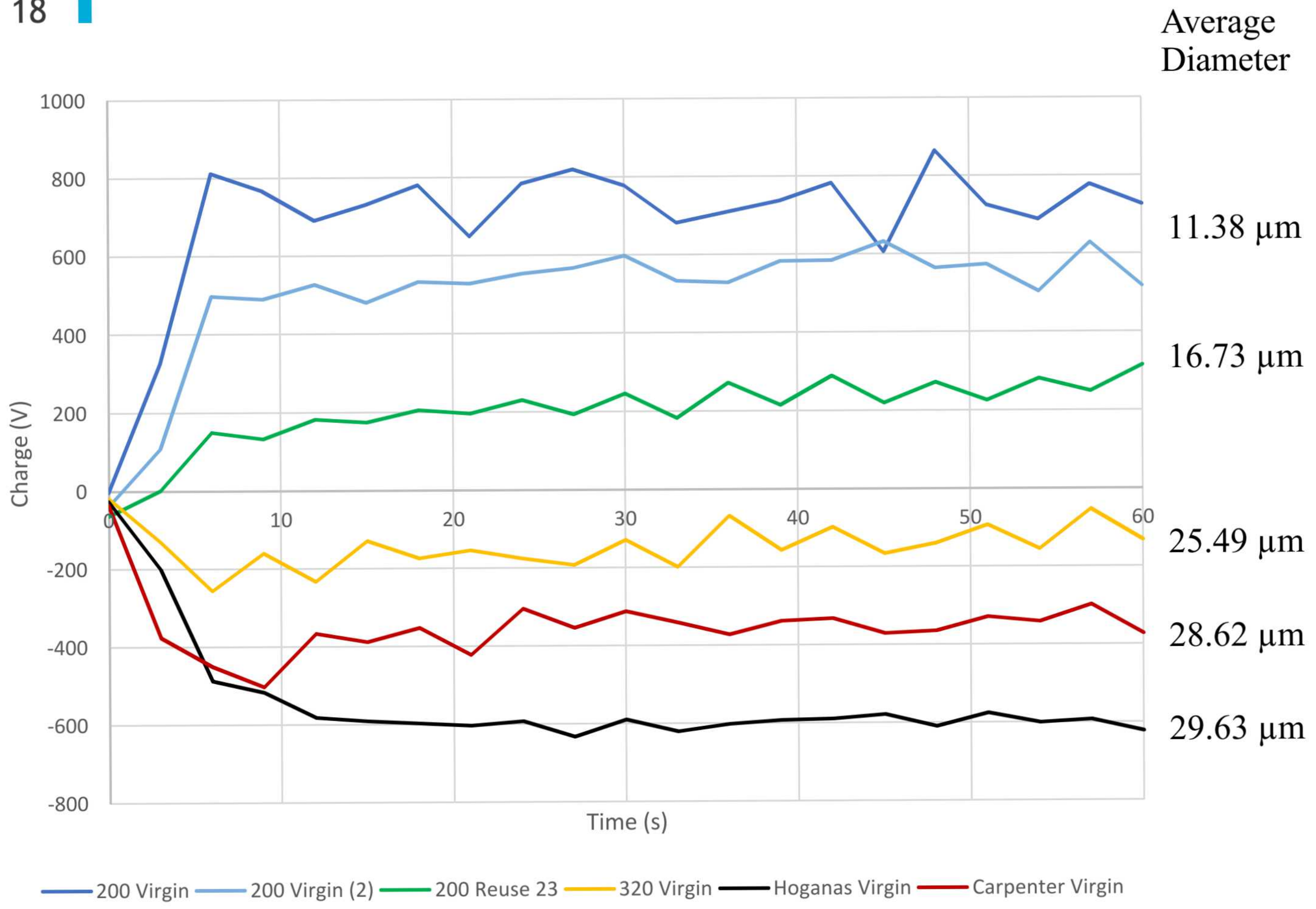


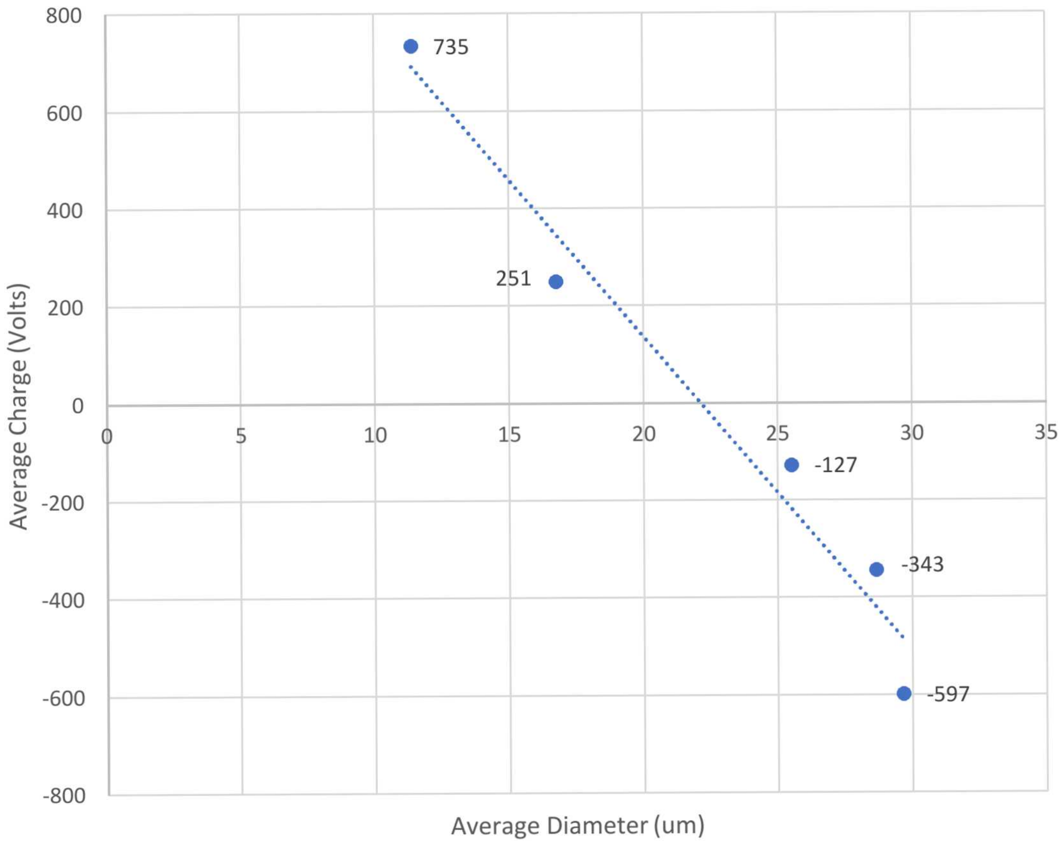






Charge Density Variation with Powder Lot

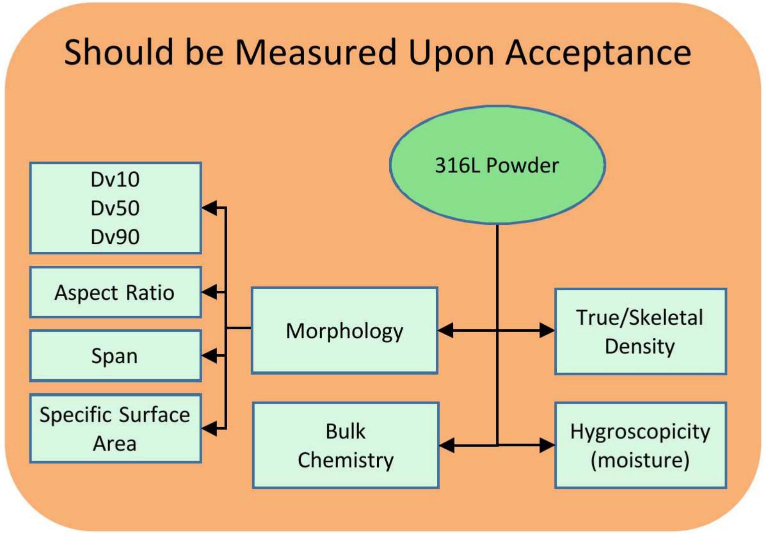


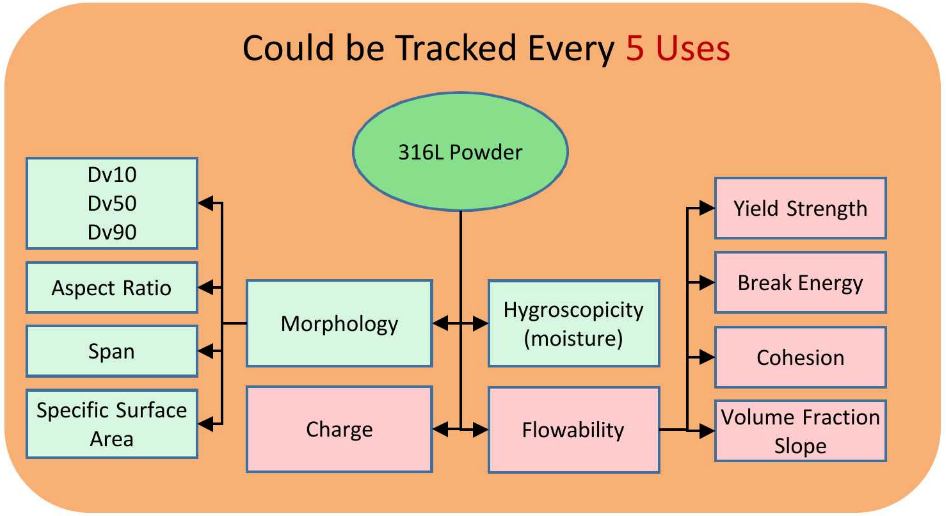


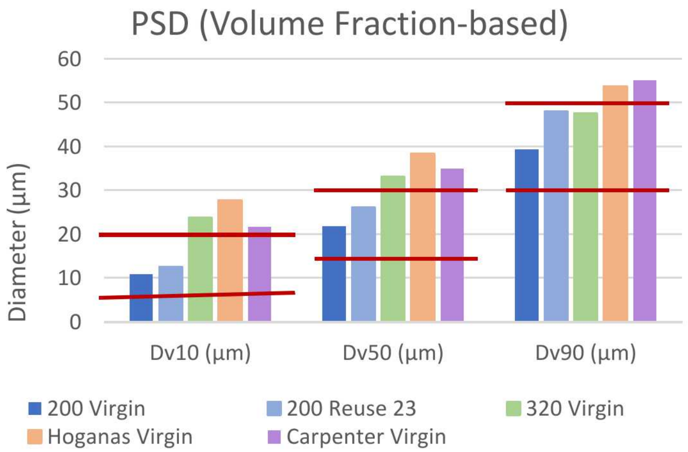
- Powders with largest avg. diameters require larger layer thickness, reducing mechanical properties at same parameters
- Larger powder sizes switch their plateaus to a negative charge
    - Reason unknown, size range unknown, effects on resulting parts unknown

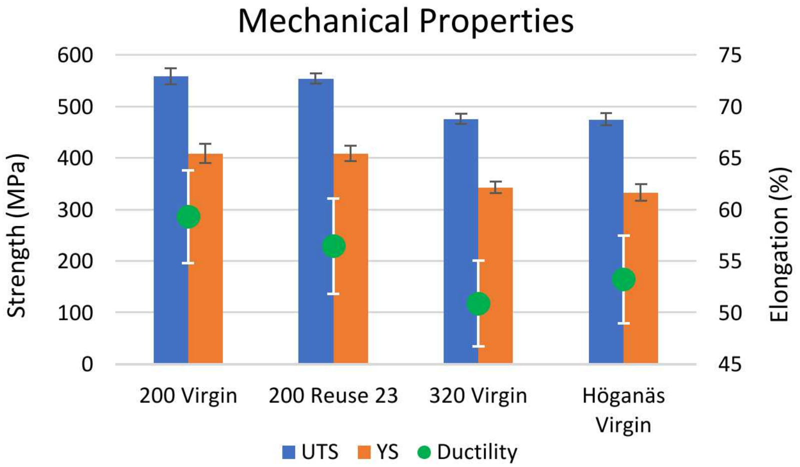
Final Remarks: One Specification per Alloy/AM-machine Combination

- Type of powder used depends on part requirements & vendor supply
- Essential to tailor machine parameters to powder PSD
- 316L mechanical properties relatively insensitive to powder reuse, as long as D_v50 remains below the machine layer thickness
- Property variability primarily due to AM machine process conditions
- Different metal alloys will require more stringent limits
- Difference in powder interaction for blade vs roller, L-PBF vs EBM, so PSD may need to be tied to specific machine types to assure reliability









Acknowledgements

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Dan Tung

Sara Dickens

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Significant increase in C and O in built parts with 30x reuses

Built Parts

Wt.% (Stdev)	Fe	Cr	Ni	Mo	Si	Mn	Cu	P	Co	C	S	O	N
Virgin	67.7 (0.3)	16.70 (0.33)	10.75 (0.26)	2.07 (0.21)	0.62 (0.09)	1.11 (0.11)	0.24 (0.04)	0.020 (0.003)	0.11 (0.02)	0.14 (0.02)	0.014 (0.002)	0.21 (0.03)	0.080 (0.012)
Reused	67.8 (0.3)	16.77 (0.33)	10.68 (0.21)	2.02 (0.20)	0.55 (0.08)	1.25 (0.12)	0.25 (0.04)	0.022 (0.003)	0.13 (0.02)	0.19 (0.03)	0.011 (0.002)	0.14 (0.02)	0.090 (0.013)

Initial Powder

Virgin	67.8 (0.3)	16.84 (0.34)	10.81 (0.22)	2.05 (0.20)	0.65 (0.10)	1.20 (0.12)	0.21 (0.03)	0.015 (0.002)	0.098 (0.015)	0.011 (0.002)	0.014 (0.002)	0.067 (0.010)	0.086 (0.013)
Reused	67.6 (0.3)	16.91 (0.34)	10.90 (0.22)	2.02 (0.20)	0.60 (0.09)	1.27 (0.13)	0.22 (0.03)	0.016 (0.002)	0.11 (0.016)	0.016 (0.002)	0.014 (0.002)	0.095 (0.014)	0.090 (0.013)
ASTM Spec [2]	61-69	16-18	10-14	2-3	1 max	2 max	-	0.045 max	-	0.03 max	0.03 max	-	-

- Carbon and Oxygen both increased with reuse in powder. However, only Carbon increased with reuse in the actual parts. Oxygen content decreased.
- Carbon content is actually out of ASTM spec on the part themselves

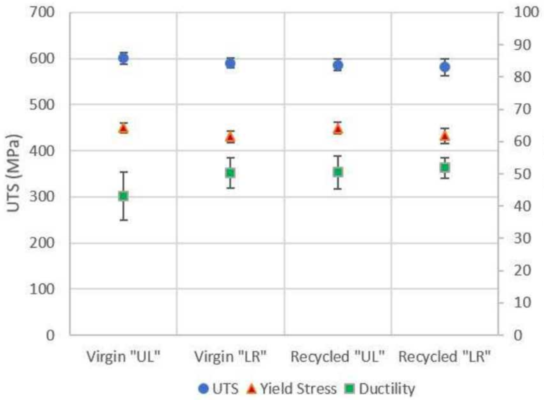
[2] ASTM F3184-16 Standard Specification for Additive Manufacturing Stainless Steel Alloy (UNS S31603) with Powder Bed Fusion, ASTM International ASM Handbook (2016) 1-9.

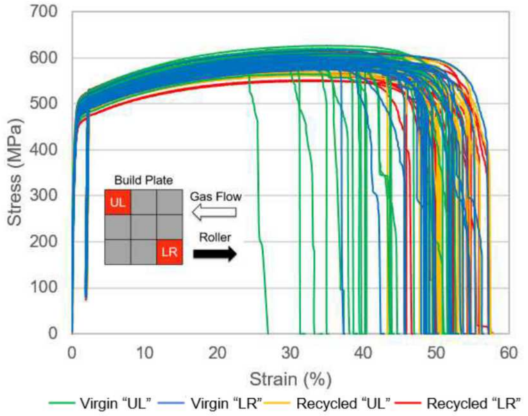
Appendix

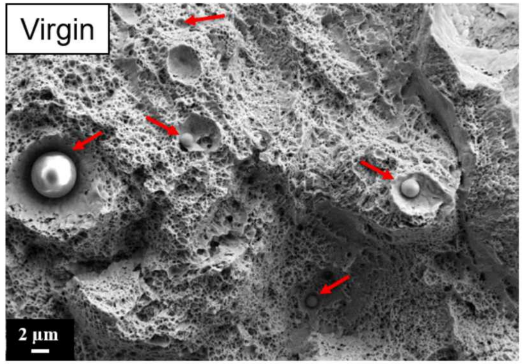
Flowability Criteria

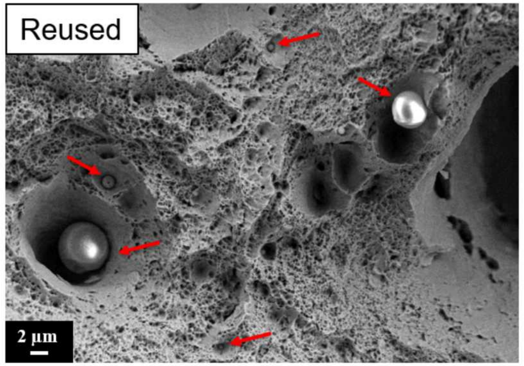
	Criterion	FT4 indices
Powder bed density	Good packing ability	↑Conditioned bulk density (ρ_c)
		↓Compressibility (CI)
	Low entrapped air	↑Conditioned bulk density (ρ_c)
		↓Compressibility (CI)
	Good ability to release entrapped air	↑Permeability (↓PD)
Powder bed uniformity	Low tendency to agglomerate	↓Aeration energy (AE)
	Low mechanical interlocking	↓Specific energy (SE)
		↓Cohesion coefficient (c)
Minimal disturbances	Low resistance to flow	↓Specific energy (SE)
		↓Basic flow energy (BFE)

↑: index to maximize, ↓: index to minimize

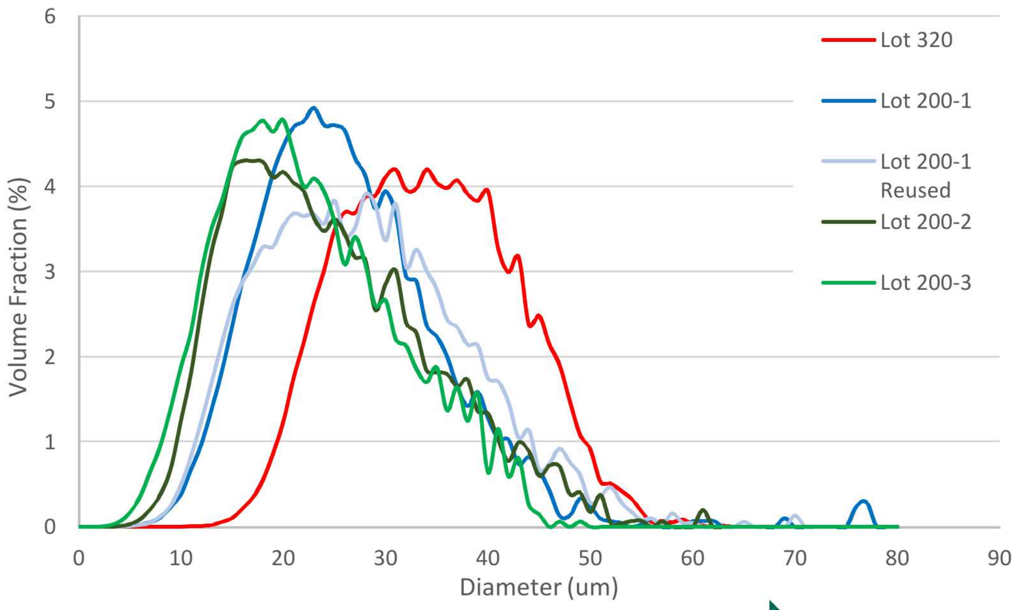


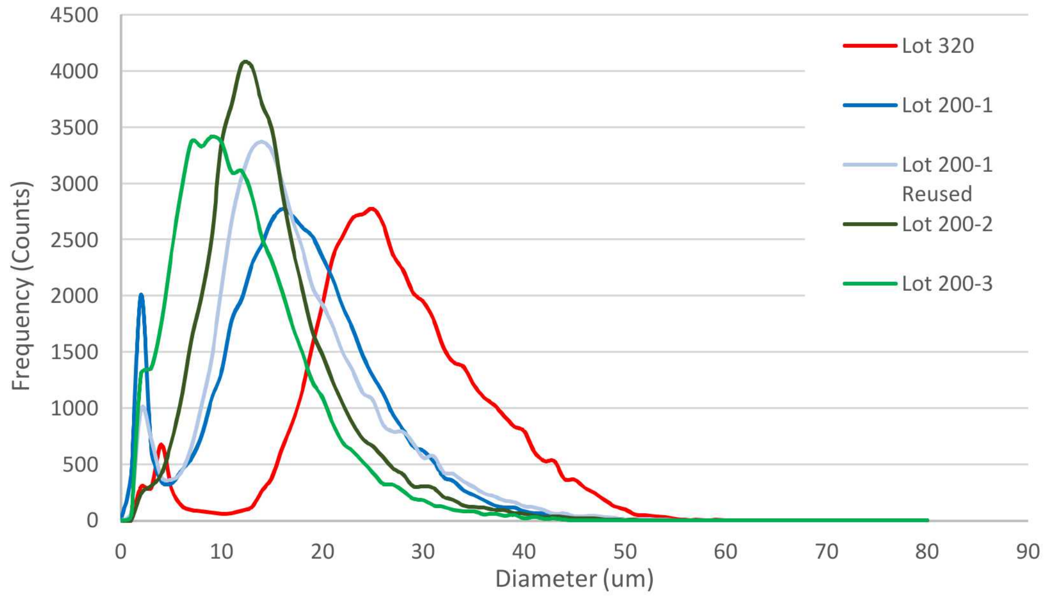


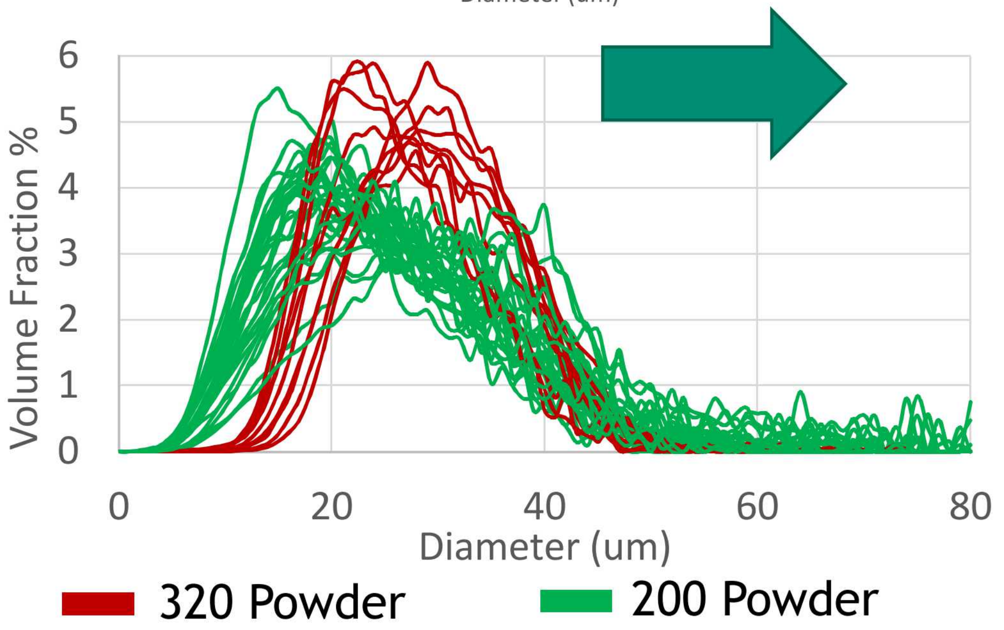




Particle Size Distribution Comparisons

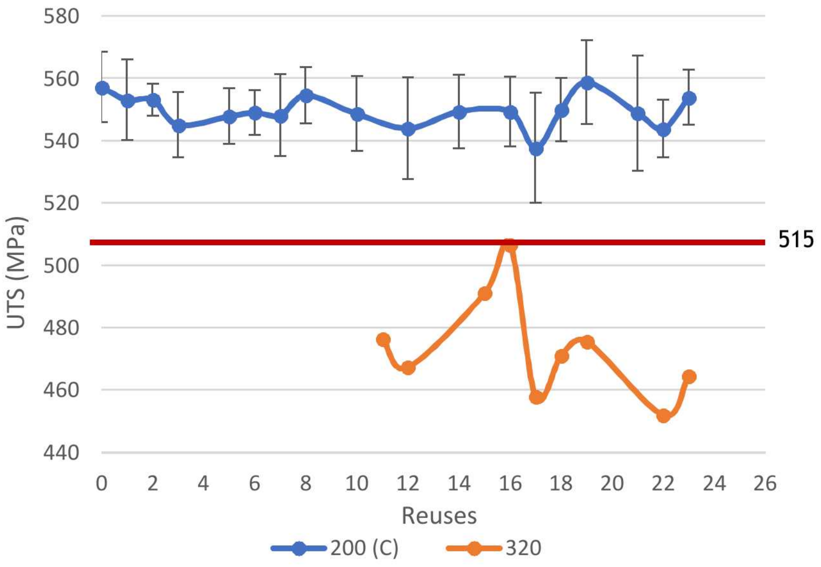


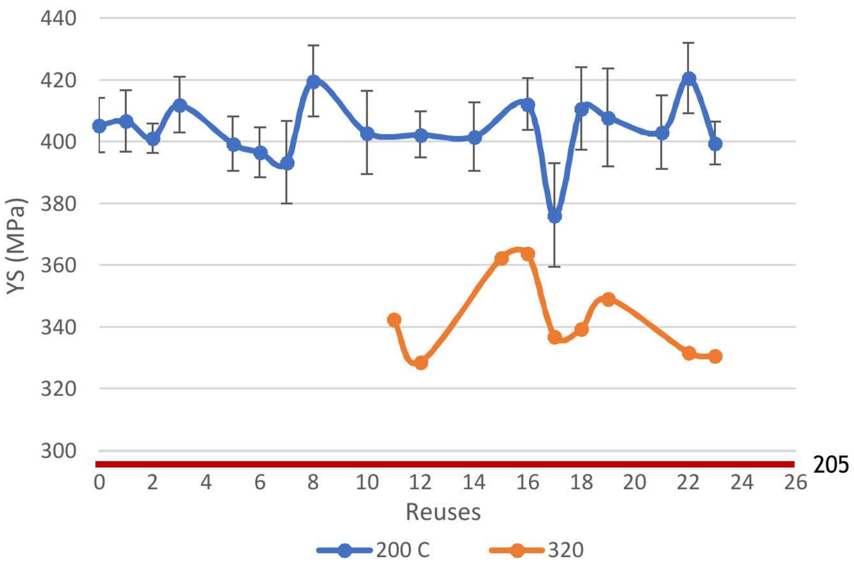


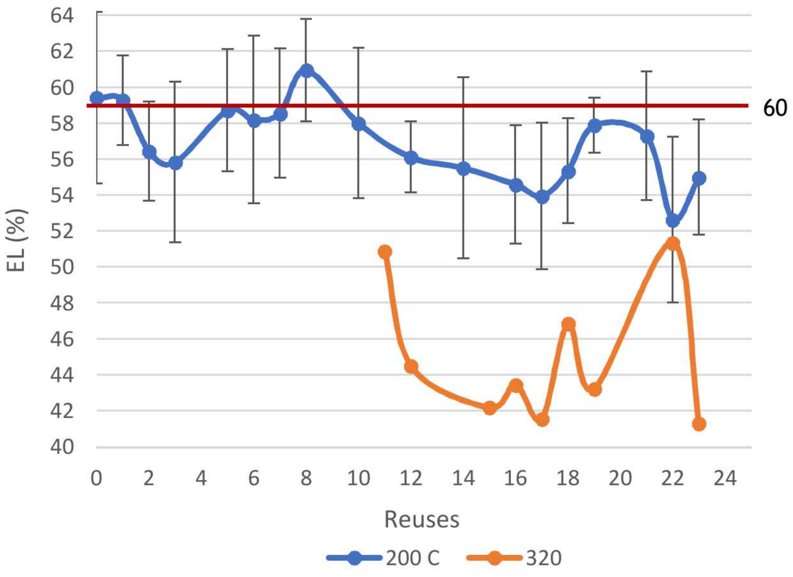


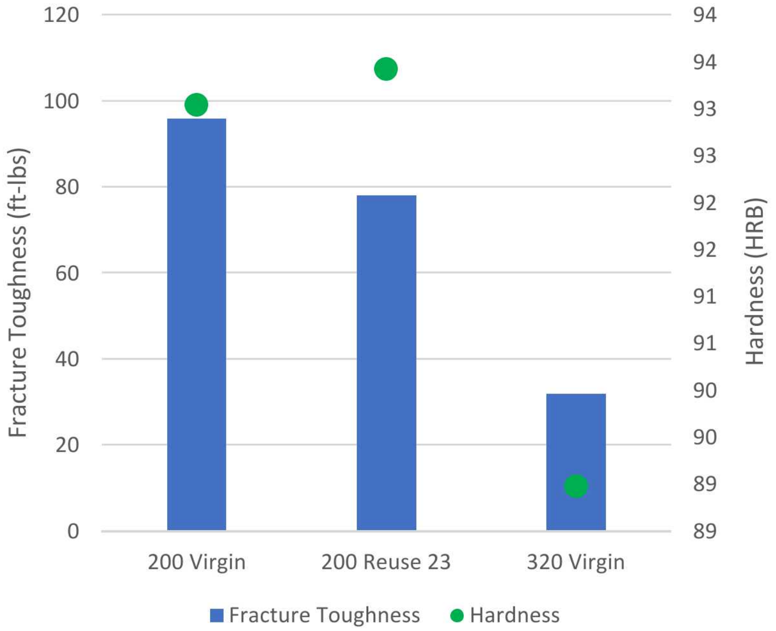
Lot	Average Diameter (μm)	Aspect Ratio	D_n10	D_n50	D_n90
320	27.5	1.4	17.4	25.7	36.8
200-A	16.9	1.5	7.0	16.9	26.5
200-A Reused	16.7	1.5	8.2	15.4	27.5
200-B	14.4	1.4	7.5	13.3	22.9
200-C	11.6	1.5	4.2	10.6	20.2

Deviation consistent across reuse cycles

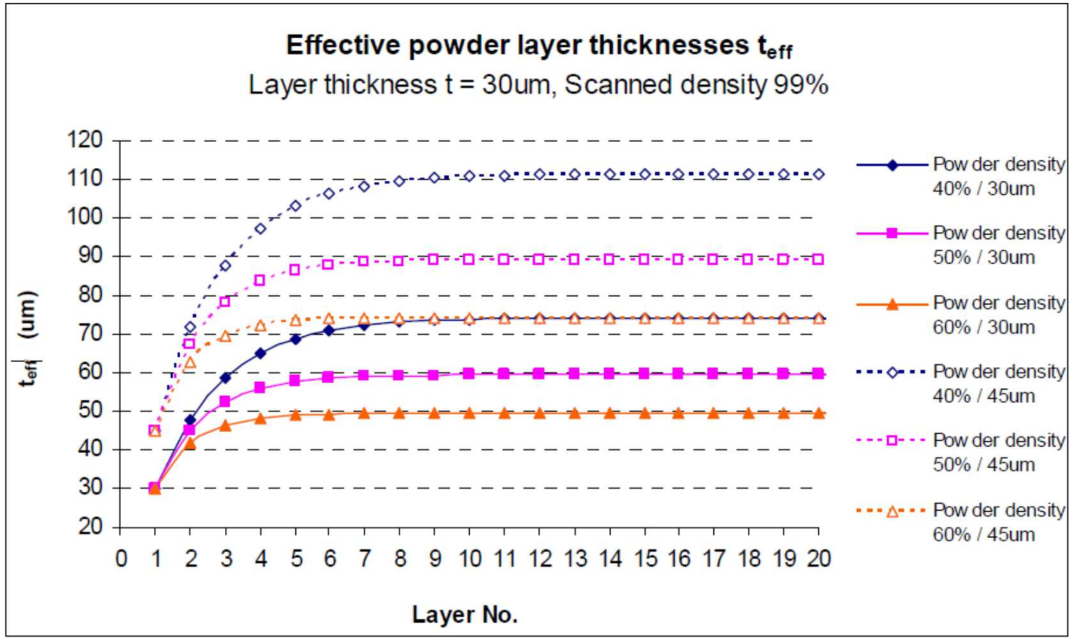








Influence of Powder Size on Laser Interactions



Suggestion:

$10\ \mu m < D_{50} < 20\ \mu m$

General Feedstock Suggestions

$$\frac{t_{eff}}{D_{90}} \approx 1.5$$

- The real (teff) powder layer thickness should be at least 50% higher than the diameter of 90% of powder particles

- Hoganas: 1.25 320 powder: 1.66 200 powder: 2.5

Suggestion: $2 < \frac{t_{eff}}{D_{90}}$

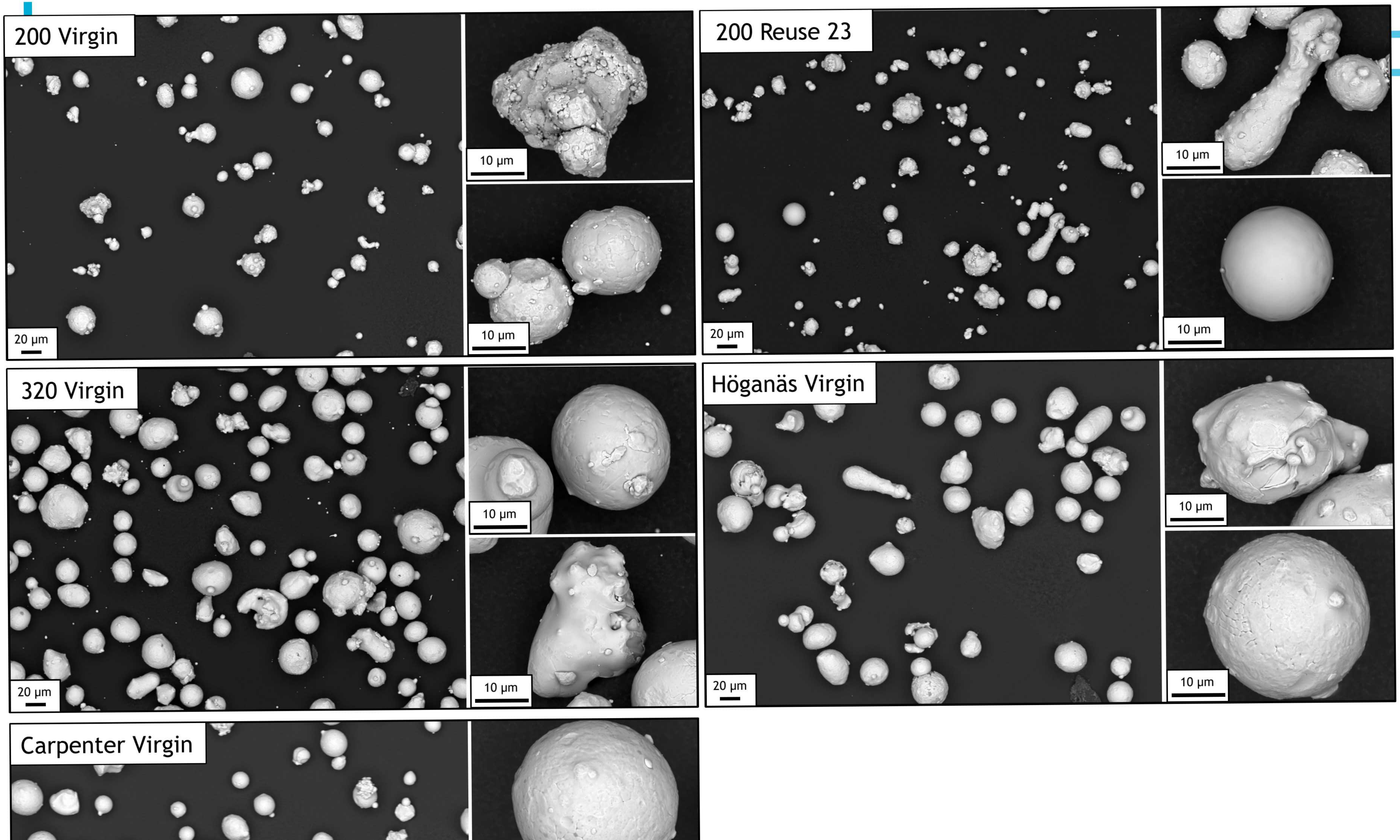
$$\frac{D_{90}}{D_{10}} \approx 5$$

- Suggests there needs to be a sufficient amount of fine particles in the distribution to fill voids between coarser particles

- Hoganas: 1.91 320 powder: 2.10 200 powder: 4.24

Suggestion: $4 < \frac{D_{90}}{D_{10}} < 5$

Spierings, A. B. (2011). "Influence of the particle size distribution on surface quality and mechanical properties in AM steel parts." Rapid Prototyping Journal **17**(3): 195-202.
Spierings, A. B. (2009). "Comparison of density of stainless steel 316L parts produced with selective laser melting using different powder grades." SFF Symposium 2009, 342-353


200 Virgin
20 µm
10 µm
10 µm
200 Reuse 23
20 µm
10 µm
10 µm
320 Virgin
20 µm
10 µm
10 µm
Höganäs Virgin
20 µm
10 µm
10 µm
Carpenter Virgin